

The Interaction of HVEM Generated
Point-Defects with Dislocations
in
Fe-Ni-Cr Alloys



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Abstract

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The climb of dissociated dislocations in FCC materials is known to be complex: Observations of climb under HVEM irradiation in CuAl suggest that it proceeds via the nucleation of interstitial loops directly onto individual partials. In silver, however, dissociated dislocations appear to constrict at an early stage in the irradiation, and dense vacancy cluster damage is seen to form in their vicinity.

This thesis presents results and analysis of a study aimed at the determination of the interaction of HVEM generated interstitials and vacancies with pre-existing dislocations in a range of Fe-17Cr-Ni ternary alloys (with the nickel content varying between 15 and 40%). Two quaternary alloys (Fe-15%Ni-17%Cr-1%Si and Fe-15%Ni-17%Cr-2%Mo) are also studied. As with earlier studies in CuAl and Ag, pre-existing dislocations in a (111) orientated foil were first characterized at sub-threshold voltages employing the weak-beam technique, then irradiated with 1MeV electrons at temperatures in the range 400-430°C and finally returned to the low voltage microscope for post-irradiation characterization of the observed damage.

Analysis of the post-irradiation microstructures indicates that interstitial climb only occurs at particularly favourable sites, such as pre-existing jogs: For the ternary alloys, constrictions are removed along edge and mixed dislocations, whilst zig-zagging of screw and near-screw dislocations may also be attributable to jog climb. After the annihilation of constrictions evidence of climb is not seen and pipe diffusion is thought to be occurring. The precipitation of small clusters, many of which are identifiable as vacancy SFT, is reminiscent of observations in silver.

The addition of silicon to the matrix apparently leads to the creation of favourable sites for interstitial climb, as evidenced by the formation of high densities of new jogs after irradiation. Loops are seen to precipitate close to dislocations in the Mo-doped material. The origin of these loops is unclear at this stage. The relevance of the results to the phenomenon of void swelling is discussed.

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Time is rather short since this thesis must be at the binders in 97 minutes, so...

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Chapter 1

INTRODUCTION & BACKGROUND

1.1 The problem

Austenitic (γ -phase) stainless steels are widely employed within reactor design; their principal uses being for fuel element cladding, supports and spacers, and control rod guide tubes within the core of fast reactors. Concomitant with these applications, however, have come a variety of induced or enhanced radiation damage effects.

Almost all these effects are detrimental, steels suffering particularly from those of void swelling, irradiation creep and embrittlement. These seriously limit the useful life of reactor components necessitating costly and difficult renewal. In the hostile environment of a commercial fast reactor, for example, in which core structures are subject to dose-rates up to 40 displacements per atom (dpa)/year at temperatures in the range 500-600°C, the void swelling problem, first observed by **Cawthorne & Fulton (1967)** in 316 stainless steel cladding, is particularly severe (up to 10% per year). This can lead eventually to buckling and weakening of core components, making their removal difficult and expensive. TEM observations of these swollen steels, under a variety of irradiation conditions have been very great in number (e.g. **Nelson & Mazey, 1969**; **Mazey et al, 1971**) and show that a complicated microstructure of voids and dislocation networks has evolved. In spite of over 20 years of further research, however, many aspects of the phenomenon of void swelling remain unexplained.

1.2 Void Swelling

Sir Alan **Cottrell (1972)** once described void swelling as “*a fortunate ... combination of fundamental scientific problems and practical needs*”. Whilst many in nuclear engineering may consider the swelling of irradiated steels to be singularly unfortunate, there is little doubt that the phenomenon encompasses a vast range of basic concepts, metallurgical variables and irradiation sensitive effects (**Brailsford & Bullough, 1972, 1981**).

Interstitials and vacancies are created in equal numbers by irradiation. In Fe-Ni-Cr alloys, and 316 stainless steel, interstitials and vacancies generally become ‘mobile’ at $\sim 100\text{K}$ and $\sim 300\text{K}$ respectively (**Nakata et al, 1985**). Hence, at reactor operating temperatures, both defect species may diffuse very rapidly and will migrate to defect sinks.

In order for a material to swell, vacancies and interstitials must not all migrate to the same sink. It was recognized as early as 1959 (**Greenwood et al**) that a tendency for dislocations to show a preference for interstitials over vacancies may play a crucial rôle, allowing excess vacancies to cluster.

This tendency is expected because interstitials have a larger strain field than vacancies and so have a bigger elastic interaction with line dislocations. This preference was formalized in terms of a dislocation ‘bias’¹ for interstitials in 1975 (**Heald**). Vacancy dislocation loops are not expected to be stable in the bulk because they themselves would act as biased sinks for interstitials. Voids may however nucleate and grow under certain conditions. These are: that the temperature is high enough for vacancy mobility ($T \gtrsim 0.3 T_m$); that the temperature is not so high that vacancy emission exceeds net vacancy absorption ($T \lesssim 0.55 T_m$); and that gas (e.g., He or O) is present leading to internal pressure which stabilizes void nuclei against collapse to loops. If these conditions are satisfied there will exist at any temperature a minimum critical cavity size for growth (e.g., **Katz & Wiedersich**, 1971), when the probabilities for vacancy emission and absorption are equal, and this is dependent on nearly all materials and irradiation variables (e.g., **Mansur**, 1978). Void embryos which achieve this size will be stable and grow.

An alternative, and possibly complementary theory behind the growth of vacancy clusters has been advanced (**Mansur & Wolfer**, 1978; **Mansur**, 1978) which considers the effects of solute segregation to form ‘coated voids’. These coated voids will possess different elastic moduli from the matrix and it is proposed that interstitial defects, by reason of their larger first-order elastic strain field, are more strongly repelled than vacancies.

The amount by which austenitic steels swell is dependent on a great many factors. Most notable amongst these is the effect of altered nickel concentration, in which increasing nickel levels are found to strongly decrease swelling (**Johnston et al**, 1974) until, after nickel concentrations of the order of 40-60% are attained, further additions slightly increase swelling (see figure 1.1). Ferritic steels exhibit very low swelling.

High dislocation densities introduced by cold work have also been found to affect swelling;

¹The ‘**bias**’ of a sink is the ratio of interstitial to vacancy defects which arrive. A sink is said to be a ‘**biased sink**’ if this ratio differs from unity. Another term used to describe defect sinks is the ‘**sink efficiency**’, as given by (e.g., **Balluffi**, 1975):

$$\eta = \frac{\text{actual rate of defect destruction}}{\text{hypothetical rate of defect destruction if the rate is diffusion limited}}$$

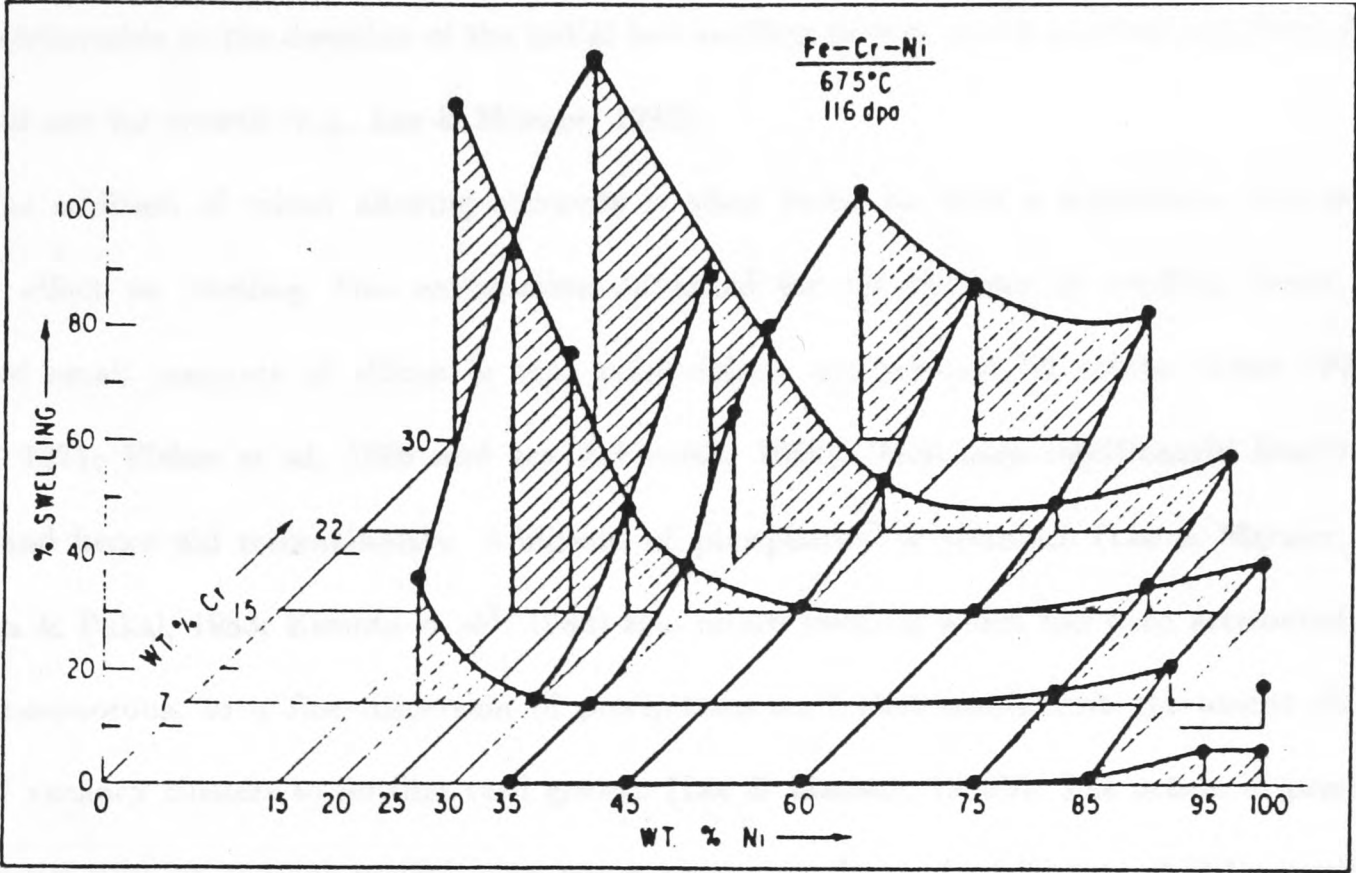
If a sink is a ‘**perfect sink**’, defect destruction is ‘**diffusion rate-controlled**’ ($\eta = 1$) otherwise it is ‘**assimilation rate-controlled**’. In the former case, the defect ‘**super-saturation**’ (C_i / C_i^0) at the sink will be zero, whilst in the latter case it will be greater than zero (under irradiation, the concentration (C_i) of defect species i can never be less than the thermal equilibrium value (C_i^0)).

If the defect concentration is depressed below the thermal equilibrium value (by raising the temperature), ‘sinks’ act as defect ‘sources’ (e.g., **Manning**, 1968, p.195).

Figure 1.1

**The Dependence of Void Swelling on the Composition of
Fe-Ni-Cr alloys after Irradiation with 5 MeV Ni⁺ ions
to 116 dpa at 675°C**

after Bates & Johnston (1977)



Leffers et al (1983) report that a small amount of cold work initially increases swelling in copper, whilst cold working to >50% substantially reduces swelling. It is possible that this reduction in swelling may be due to the action of a high density of dislocations as a completely dominant sink if recombination occurs at dislocations (see **Lee & Mansur**, 1990b). Increases in swelling for low levels of cold work have been modelled employing a basic rate theory approach.

Swelling behaviour may often be divided into two regimes (**Garner & Wolfer**, 1984; **Walters**, 1985), namely a transient regime of little or no swelling, followed by a regime of essentially constant swelling rate. There is some evidence to support the contention (**Garner & Wolfer**, 1984) that under neutron irradiation the swelling rate is constant ($\sim 1\%/dpa$) within the latter regime, irrespective of composition and temperature. Employing this model, the difference between low and high swelling alloys is attributable to the duration of the initial low swelling period, which is related to the minimum critical void size for growth (e.g., **Lee & Mansur**, 1985).

The addition of minor alloying elements is often found to have a significant, and generally beneficial, effect on swelling. One explanation advanced for the decrease in swelling found on the addition of small amounts of silicon is that point-defects are trapped by solute atoms (**Fisher & Williams**, 1971; **Fisher et al**, 1980 and see **Ashworth**, 1989). This may significantly hinder defect diffusion and hence aid recombination. Additions of phosphorus or titanium (**Lee & Mansur**, 1990b; **Hishinuma & Fukai**, 1983; **Kimoto et al**², 1986) also retard swelling which has been attributed, in the case of phosphorous, to a fine dispersion of precipitates such that insufficient gas atoms diffuse to individual vacancy clusters to fertilize void growth (**Lee & Mansur**, 1990b). The action of precipitates as preferential sites for vacancy precipitation was evidenced in the work of Kimoto et al by significantly enhanced void number densities. Conversely oxygen is found to enhance swelling since it may act to pressurize cavities (**Lee & Mansur**, 1990a).

Additions of oversize solutes, such as manganese (**Garner et al**, 1987) or molybdenum (**Gessel & Rowcliffe**, 1977) have also been found to reduce swelling, presumed to arise from defect trapping, although the effect is considerably less pronounced than that seen after P or Si additions.

Attempts have been made to correlate experimental observations of swelling with a very large variety of possible variables. In Table 1.1, a list of some of these variables has been kept as brief as possible. Those marked with a † are considered, although sometimes only in passing, in this volume. The selected references given often deal with more than one possible origin for their observations.

²Void Formation on twin boundaries was seen by **Norris** in 1971.

Table 1.1

The minimum in swelling observed at intermediate Ni concentrations has been related to:	
• Phase Stability	(Harries, 1975b; Stubbins, 1987; Garner et al, 1987)
† Changes in defect properties (particularly of diffusivities) of the alloying elements with composition	(Venker & Ehrlich, 1976; Marwick, 1978; Rothman et al, 1980; Garner & Wolfer, 1984; Dimitrov & Dimitrov, 1984)
• Shear modulus	(Bates, 1979)
† Changes in the minimum cavity size for bias driven growth	(Mansur & Coghlan, 1983; Lee & Mansur, 1985, 1990a);
† The composition dependent effects of major alloying element segregation effects on void bias	(Garner & Wolfer, 1984; Muroga et al, 1990)
Explanations for effects of minor element additions:	
† The effect of minor alloying element segregation on void bias	(Mansur, 1978; Wolfer et al, 1982)
† Point defect trapping by solutes	(Schilling & Schröder, 1975; Fisher et al, 1980; Watanabe et al, 1989)
† Changes in cavity density due to precipitation	(Hishinuma & Fukai, 1983; Kimoto et al, 1986; Lee & Mansur, 1986, 1990b)
The following variables also affect swelling behaviour:	
† Increased dislocation density introduced by cold work	(Leffers et al, 1983)
† P.k.o. recoil spectrum	(Jung, 1990; English, 1985, 1990; Mansur et al, 1985)
• Time dependent effects on the dislocation bias due to;	
temperature and dose rate	(Bullough & Quigley, 1983)
climb	(Rauh & Bullough, 1985)
network evolution by loop nucleation	(Wolfer & Glasgow, 1985)

A full literature review of all experimental and theoretical approaches aimed at explaining macroscopic swelling behaviour is unlikely to compress into a single volume. Various researchers have reviewed particular fields of interest, however, and the reader is referred initially to the papers, and references, of **English** (1990), **Brailsford & Bullough** (1981), **Fisher et al** (1980), **Dimitrov et al** (1987), **Stubbins** (1987), **Mansur** (1978), **Mansur & Coghlan** (1983) and **Lee & Mansur** (1985).

We conclude this section by noting the results of **Perks** (1988), who observed a minimum in segregation for a Fe-45%Ni-15%Cr, and **Tappin et al** (1990), who observed a minimum in defect yield after heavy-ion irradiations in the same alloy. In both studies the nickel content of the alloys covered the range from 15 to 70%, whilst chromium content was maintained at 13 and 15%. Perks explains his results in terms of a minimum in the numbers of freely migrating defects. Final results from the work of D.K.Tappin should shortly be available³.

1.3 This Study

There have been numerous observations of voids (e.g., **Norris**, 1971; **Rothaut et al**, 1983; **Lee & Mansur**, 1990b) and high densities of both interstitial and vacancy loops (e.g., **Urban**, 1971; **Jackson et al**, 1978; **Muncie et al**, 1985, 1987; **Kiritani**, 1987; **Jenkins et al**, 1987) close to irradiated dislocations, suggesting that dislocations can act as sinks for both interstitials and vacancies. Precise determination of the nature of all the damage and its relation to the line was, however, often beyond the resolution of the techniques employed.

The objective of this study was to isolate ways in which HVEM generated point-defects interact with pre-existing line dislocations in a range of Fe-Ni-Cr alloys. Owing to the fine-scale nature of damage often seen to form close to dislocations, the weak-beam technique (**Cockayne** and co-workers, 1969-1973; see, e.g., **Thomas & Goringe**, 1979) is employed. It was hoped that by studying these interactions light would be shed on the sink behaviour of dislocations, possibly leading to a better understanding of the importance of dislocations to the phenomenon of void swelling.

The advantages of HVEM irradiation are many, affording a high dose rate, a reasonable degree of control over temperature, specimen position and orientation, and making experiments repeatable and relatively quick. Another important factor, bearing in mind the current radiation damage climate, is the low cost when compared with other irradiation sources.

A major disadvantage of HVEM irradiation, however, is its poor simulation of reactor conditions: Whilst the application of results obtained with one irradiation source to predict or explain

³*Ph.d. Thesis, University of Liverpool - in preparation.*

results obtained with another is a not a trivial exercise, a systematic study such as is undertaken here may highlight any differences in behaviour intrinsic to the material. The correlation of electron, heavy-ion and neutron damage has been extensively considered in the literature (e.g., Bullough & Quigley, 1983; Foreman & Makin, 1979) and therefore is not discussed further here.

A similar increase in the incubation period required to produce swelling as nickel content is raised has been found for heavy-ion (e.g., Johnston et al, 1974), neutron (e.g., Bates & Johnston, 1977) and electron (Hishinuma et al, 1978, 1981; Walters, 1985) irradiations.

1.3.1 Materials:

The three pure ternary alloys chosen for study are as follows (at.%):

Fe-17%Cr-15%Ni

Fe-17%Cr-25%Ni

Fe-17%Cr-40%Ni

These compositions span a large range in swelling behaviour. In addition two quaternary alloys were selected, consisting of additions of 1%Si and 2%Mo respectively to the 15%Ni ternary alloy. Silicon and Molybdenum exhibit different segregation behaviour in Fe-Ni-Cr (see chapter 8), such that one (Si) is expected to migrate ^{towards} ~~with~~, and the other (Mo) away from defect sinks. These levels of Si and Mo additions are typical of technological steels.

Over the last 10 years or so, the Dimitrovs at Vitry-sur-Seine have studied diffusion behaviour in 17%Cr base Fe-Ni-Cr ternary alloys via a series of resistivity & recovery experiments. Since some information is known, therefore, about point-defect parameters for this system, chromium content was maintained at 17%. Swelling behaviour is sensitive to chromium content, such that the amount of swelling increases with increased chromium concentration (Bates & Johnson, 1977). The effect is considerably less marked than for comparable changes in nickel content, however (Garner et al, 1987).

At a recent conference, Dimitrov et al (1987), in reviewing their experiments of the previous 6 years, were able to present an empirically derived relation for the compositional dependence of interstitial migration enthalpy. Perks & Murphy (1987) have also further extrapolated from Dimitrov's data to produce relations for vacancy migration and formation enthalpies for the range 20-80% Ni and 5-20% Cr. The formulae are as follows (in eV):

$$H_m^V = 1.044 + 0.44 N_{Ni}$$

$$H_m^I = 0.96 + 0.65 N_{Cr} - 0.8 N_{Ni}$$

- where H_m^V and H_m^I are the migration enthalpies for vacancies and interstitials respectively; N_d is the fractional composition of element d,

and, $H_f^V = 1.81 + 2.1 N_{Cr} - 0.95 N_{Ni}$

- where H_f^V is the vacancy formation enthalpy.

These equations assume linear dependencies on composition. These equations do not always lead, however, to values in good agreement with experiment, particularly for H_m^V (see **Dimitrov & Dimitrov, 1988**).

1.4 The climb of dissociated dislocations:

1.4.1 Early theoretical models:

Ever since the postulation by **Heidenreich & Shockley (1948)** that in FCC structures dislocations are dissociated into two partials separated by a region of stacking fault, researchers have attempted to isolate the mechanism(s) by which these dissociated dislocations climb. Undoubtedly the simplest solution was that proposed by **Stroh (1954)**, namely that constriction along a length of dislocation must occur prior to climb. He noted, however, that a high activation energy is likely to be required. A variety of other theoretical models soon followed⁴ in which jog nucleation directly onto the partials, nucleation onto the stacking fault and climb motion of parts of the stacking fault were considered.

The model of **Thomson & Balluffi (1962)** has received considerable further attention and is shown in figure 1.2. Calculations performed by **Grilhé et al (1977)**, aimed at determining the activation energies for the operation of this mechanism, are detailed in chapter 8.

1.4.2 Studies of the Climb of Dissociated Dislocations by Weak-Beam Imaging:

The experimental techniques employed in all climb studies by weak-beam imaging and HVEM irradiation are fundamentally similar to that given by **Cherns et al (1980)**. These techniques are also employed in this study (see chapter 2).

1.4.2.1 CuAl

Weak-beam imaging was first employed to study irradiated dissociated dislocations by **Carter et al (1978)** in the low stacking fault energy (SFE) Cu-13% & 15% Al alloys. This preliminary report confirmed the fine-scale of damage formed close to irradiated dislocations and found loops to have nucleated onto the partials themselves. On completion of a later, more detailed, study, **Cherns et al (1980)** were able to deduce that climb in Cu-13%Al, at both room-temperature and elevated temperatures (200°C) was proceeding via the nucleation of interstitial perfect prismatic dislocation loops (or Frank loops which subsequently unfault) directly onto the partials, the partial of greater edge character generally being favoured. As distinct from the **Thomson & Balluffi** mechanism, nucleated

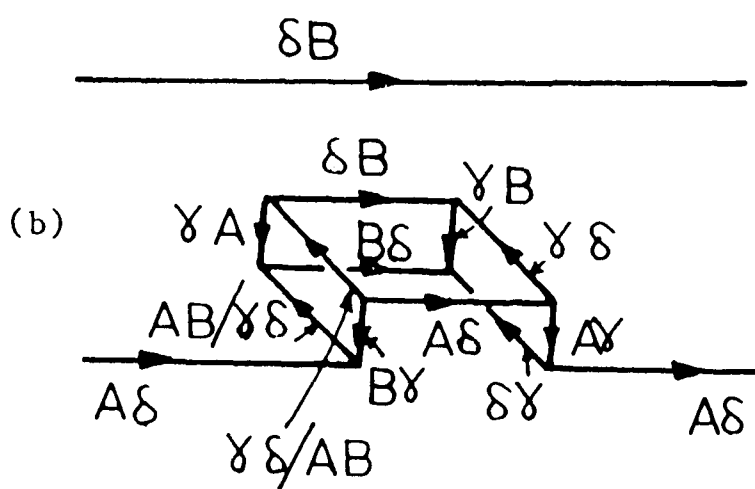
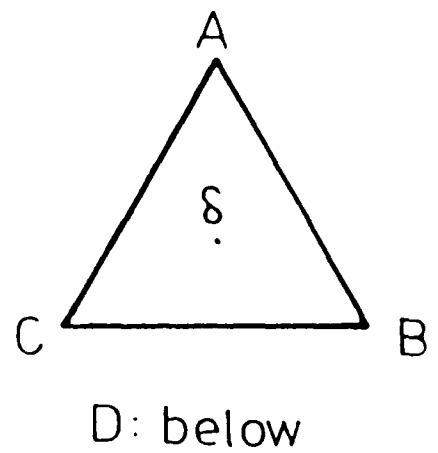
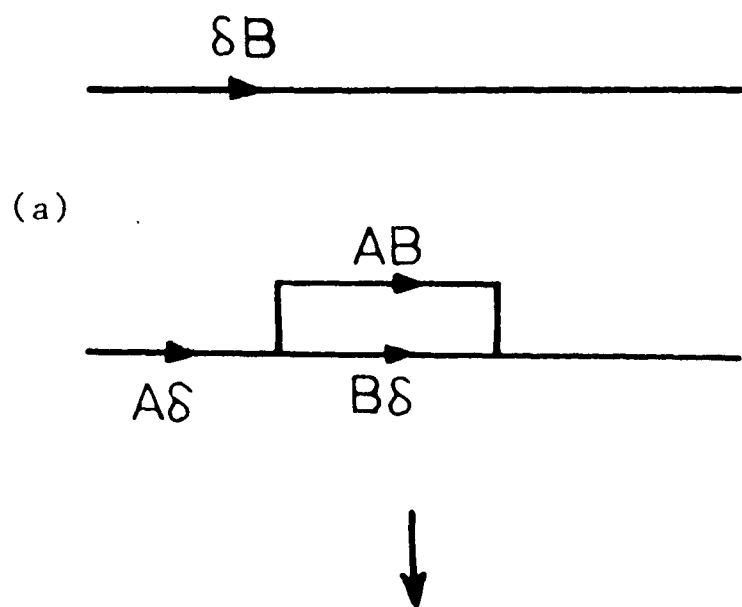
⁴**Kimura et al, 1959;**

Thomson & Balluffi, 1962; Escaig, 1963.

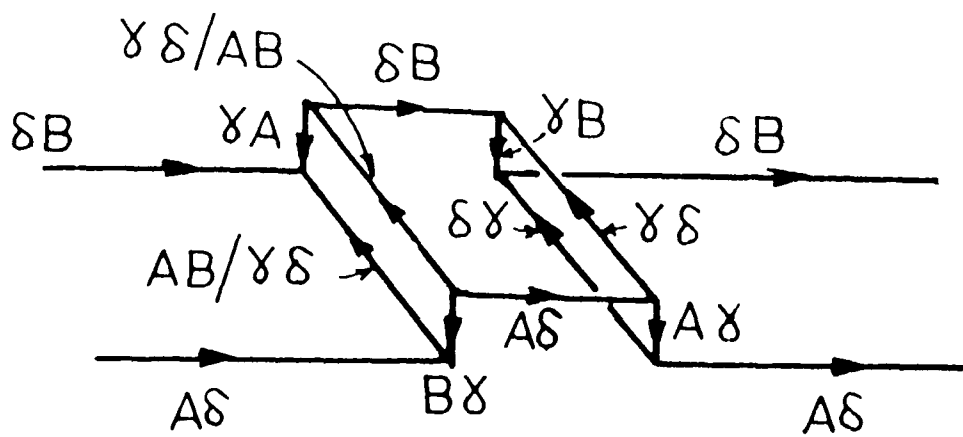
Figure 1.2

The Thomson & Balluffi (1962) Climb Mechanism

- (a)* Nucleation of perfect prismatic loop with the same Burgers vector and character as the total dislocation;
- (b)* Dissociation to equilibrium configuration;
- (c)* Extended double-jog formation by glide across the stacking fault.



- (c)
- (a) Nucleation of perfect prismatic loop of same character as total dislocation.
 (b) Dissociation across stacking fault to equilibrium configuration.
 (c) Extended double jog formation by glide across stacking fault.



loops were not necessarily of the same Burgers vector as the parent dislocation and the final configurations frequently contained a number of Shockley dipoles. Dislocation dipoles may be recognized by their characteristic contrast, as shown in figure 1.3. The room-temperature mechanism of Cherns et al (1980) is shown in figure 1.4 for the case of a 60° dislocation.

At elevated temperature (figure 1.5) there was an assumed interaction between the configurations attached to each partial such that very different configurations from those seen after room-temperature irradiation evolved. The smaller pre-irradiation partial separation of screw dislocations was believed responsible for further increasing the influence of the second partial, such that at elevated temperatures evidence only for the nucleation of loops with the same Burgers vector as the total dislocation was seen.

Lastly Cherns et al (1980) hypothesized that increasing the SFE, and hence reducing the partial separation, of the system studied may also affect the Burgers vectors of nucleated loops, possibly favouring operation of the Thomson & Balluffi (1962) mechanism.

Hardy (1985) attempted to test this hypothesis by studying the higher SFE Cu-10%Al alloy. Behaviour entirely consistent with that of Cu-13%Al was seen however, and similar mechanisms to those isolated by Cherns et al (1980) were thought to be operating.

Similar, nucleation based, mechanisms leading to dipole formation have been proposed in electron irradiated silicon (Ourmazd et al, 1981) and GaAs (Cherns & Feuillet, 1985) and, under vacancy super-saturation, in Cu-13%Al (Décamps et al, 1983). Climb studies performed up to 1984 have been reviewed by Cherns (1984). The observations in silicon are of interest in that an origin is suggested for the formation of interstitial climb debris close to irradiated dislocations. In the higher SFE GaAs, Frank loops were often observed along irradiated dislocations. This may suggest that prismatic loops nucleated onto individual partials originally nucleate as Frank loops. Note that the glide of two Shockley partials is required to unfault an extrinsic Frank loop (e.g., Nabarro, 1967, p.203).

1.4.2.2 Silver

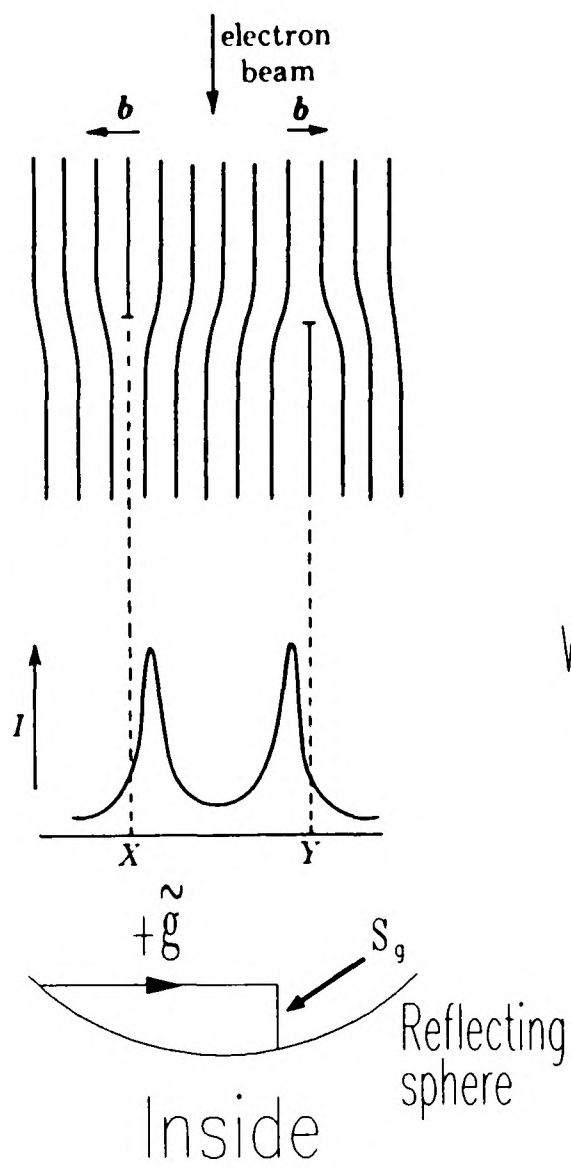
Hardy (1985) also embarked upon a study of the mechanisms of dissociated dislocation climb in silver. It was hoped that by studying a pure material in tandem with Cu-10%Al, any possible segregation effects in the binary alloy might be highlighted. Silver also has a similar SFE, to that of Cu-10%Al (Cockayne et al, 1971; Ray, 1971), although the partials are more narrowly dissociated, and hence if the mechanism of dislocation climb is dependent on, or strongly related to SFE, both materials

Figure 1.3

**Contrast characteristic of dislocation dipoles
see Cherns et al (1980)**

Inside/Outside Contrast

– from a dipole



Weak-beam
image
intensity

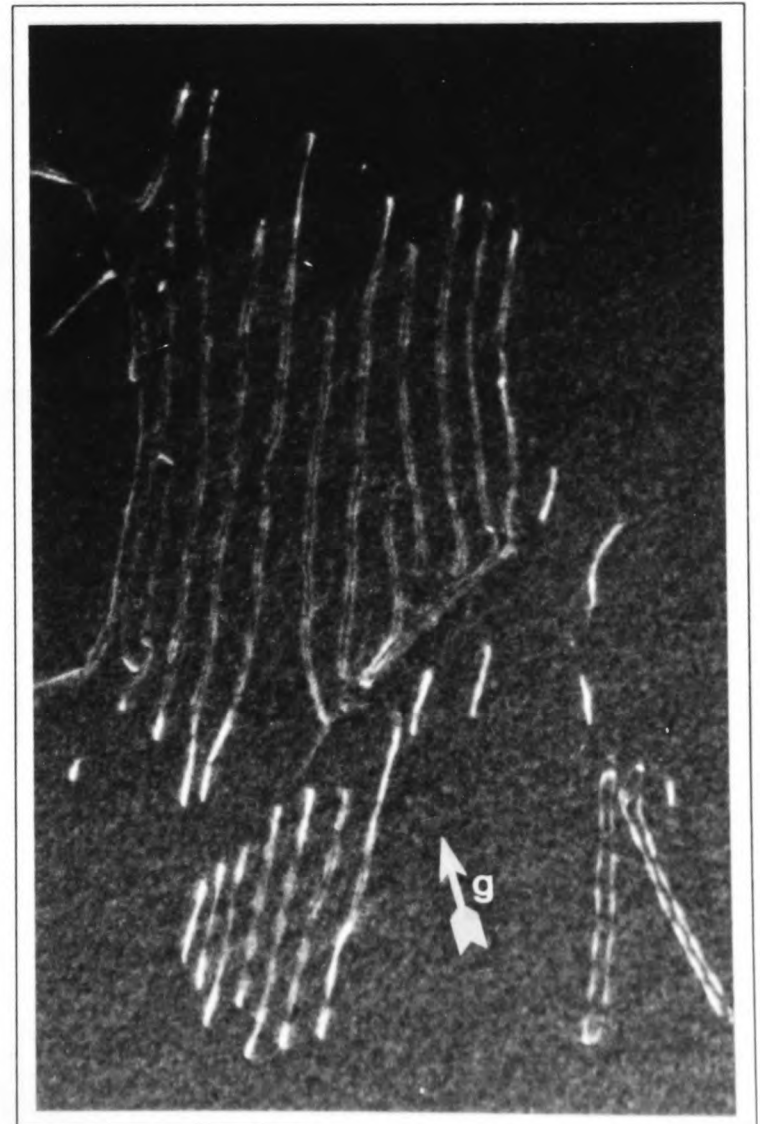
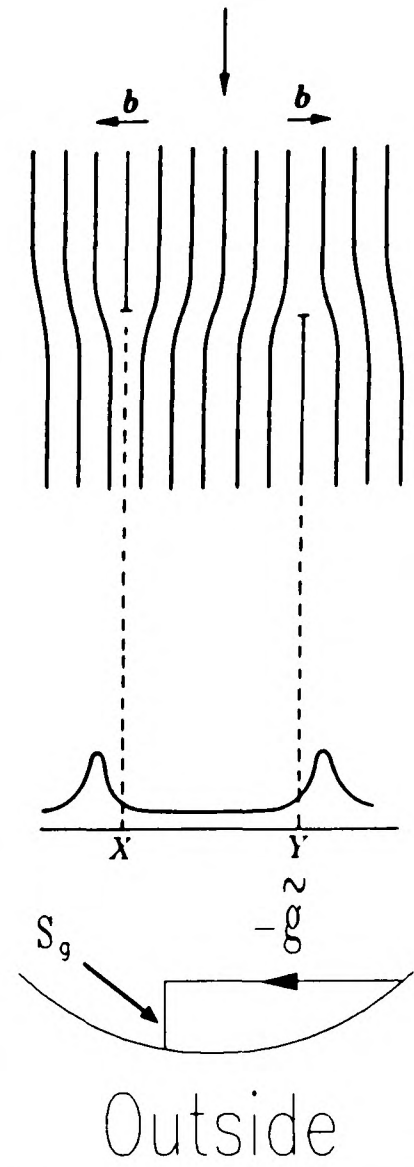


Diagram essentially from Cherns et al (1980)

might have been expected to behave in much the same manner. In silver, however, no evidence was seen for dipole formation, whilst copious vacancy cluster damage formed close to irradiated dislocations. These observations are summarized, and discussed, within chapter 8.

The SFE is strongly influenced by nickel content in the Fe-Ni-Cr system (e.g., **Bampton et al**, 1978; **Miodownik**, 1978). Hence another reason behind the selection of the Fe-Ni-Cr system for research becomes apparent, since by varying nickel content the effect of varying partial separation may be studied.

Table 1.2

Some Stacking Fault Energies for Fe-Ni-Cr Ternary Alloys

Alloy (wt.% Cr/Ni)	d_e (edge dislocation; in Å)	SFE (mJ.m^{-2})
21/14	126 ± 20	18 ± 4
16/14	100 ± 20	23 ± 5
(Ag	~ 80	$16 \pm 2)$
16/22	73 ± 15	31 ± 5
21/22	69 ± 12	33 ± 5
26/22	65 ± 10	35 ± 5

- data from Bampton et al (1978), except for silver (Cockayne et al, 1971), from weak-beam imaging of isolated dislocations.



Figure 1.4

Room-temperature Climb Mechanism for a 60° Dislocation in Cu-13%Al (Cherns et al, 1980)

(a) Before irradiation.

(b) 'Loop-jog' formation via prismatic loop nucleation. Cherns et al (1980) hypothesize that these loops may have formed originally as interstitial Frank loops which subsequently unfault such that their Burgers vectors lie in the plane of the foil. These prismatic loops need not have the same Burgers vector as the total dislocation. Nucleation is expected to be greater on the partial of greater edge character ($\vec{A}\delta$) and is subject to the following criteria:

- 1) The interaction energy of the nucleated loop and the osmotic climb force (e.g., Hirth & Lothe, p.558) must be maximized; i.e., the nucleated loop must be as close to edge orientation as possible;

- 2) The activation energy for reaction with the parent partial must be as low as possible; i.e., the nucleated loop must react with the parent partial such that a Shockley dislocation is produced. In Thompson's (1953) notation this is equivalent to saying that the nucleated interstitial loop and parent partial share the Roman letter.

(c) The 'loop-jogs' now dissociate on the higher plane δ . The climb force acts to twist them towards edge orientation. In the microscope a configuration such as that shown in (d) is seen. Areas of stacking fault are separated by small regions of perfect crystal. This region of perfect crystal is bounded by a Shockley dipole which exhibits characteristic contrast. Configurations on neighbouring partials are not expected to interact for these widely dissociated dislocations.

(e) In the final stage of this mechanism, secondary nucleation, prompted by the rotation of line sections adjacent to 'loop-jogs' has occurred. Employing the nucleation criteria 1 and 2 above, alternate loops will have differing Burgers vectors.

After nucleation and dissociation of the secondary loops, a continuous length of partial $\vec{A}\delta$ now lies on the upper plane δ . This length may straighten and glide away from climb debris on the upper plane δ . In the study of Cherns et al the dissociation on the upper plane δ generally remains relatively narrow.

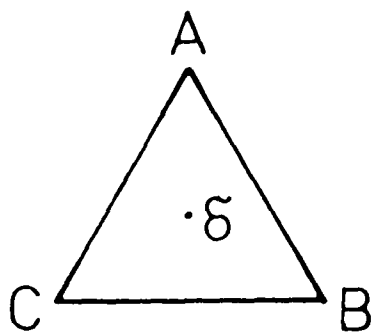
For other dislocation orientations all room temperature observations could be explained in terms of this mechanism: For the case of a total dislocation of pure screw character, for example, the mechanism predicts that nucleation and climb should proceed at the same rate on each partial but in opposite directions. This was consistent with experimental observations.

δB

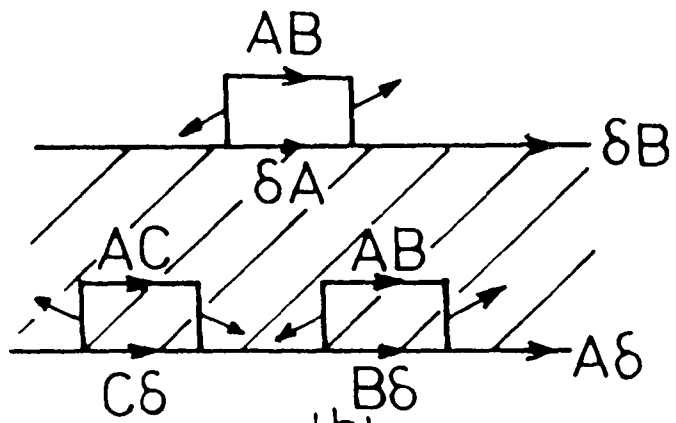
$A\delta$

(a)

Nucleation

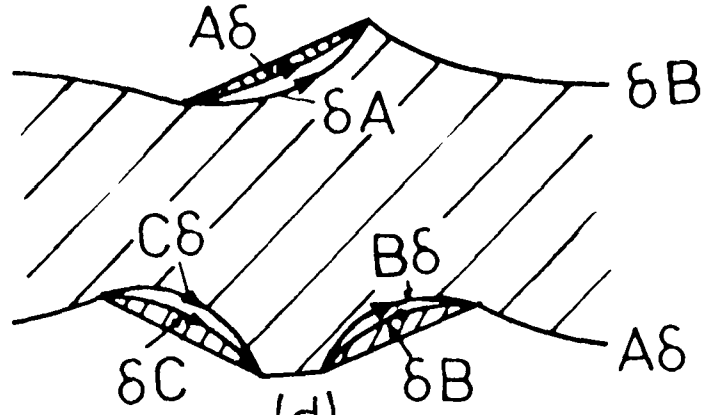


D below



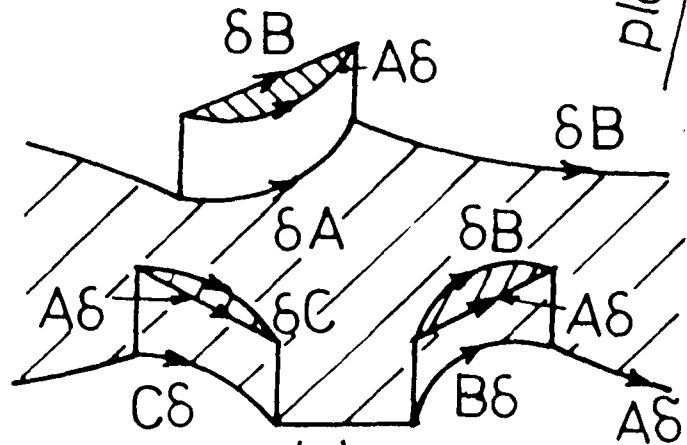
(b)

Dissociation



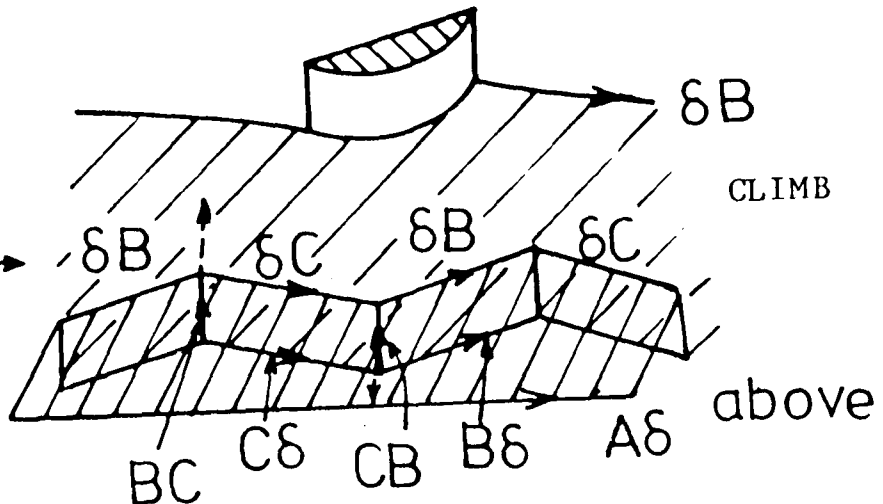
(d)

Secondary Nucleation



(c)

plan view



(e)

Climb force acts to twist nucleated loops to edge orientation

Figure 1.5

High temperature mechanism for a 60° Dislocation in Cu-13%Al (Cherns et al, 1980)

At elevated temperature (200°C), dislocations assume very different configurations from those of the room temperature irradiations. These are explained by Cherns et al in terms of the room-temperature mechanism, with the proviso that configurations on neighbouring partials may interact. A notable characteristic of the high temperature irradiations is the much lower dipole densities on the partials than seen at room temperature, explained by greater losses of point defects to other sinks. Cherns et al therefore thought it necessary to consider only nucleation on the partial of greater edge character, except for the cases of pure edge or pure screw dislocations where both partials are of equal character.

(a) and (b) proceed in similar fashion to the room-temperature case: The loop-jogs have dissociated and the climbed partial $\vec{A}\delta$ has straightened on a lower plane δ . Cherns et al then suggest that the action of the climb force pulls dipoles $\vec{B}\delta/\delta\vec{B}$ across the stacking fault ribbon such that opposing segments $\vec{B}\delta/\delta\vec{B}$ on the original plane may annihilate. This results in the configuration shown in (c) (The climb force increases linearly with temperature).

Further rotation of the climbed partials $\vec{A}\delta$ and $\delta\vec{B}$, under the action of the climb force, will tend to cause the jogs across the dislocation to be extended, leaving the cusped dipole $\vec{C}\delta/\delta\vec{C}$ associated with the partial of greater screw character.

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^a*Denotes the number of pages in each figure.*

Chapter 2

EXPERIMENTAL

2.1 Basic Technique

The technique employed for the Fe-Ni-Cr alloys studied here is as follows:

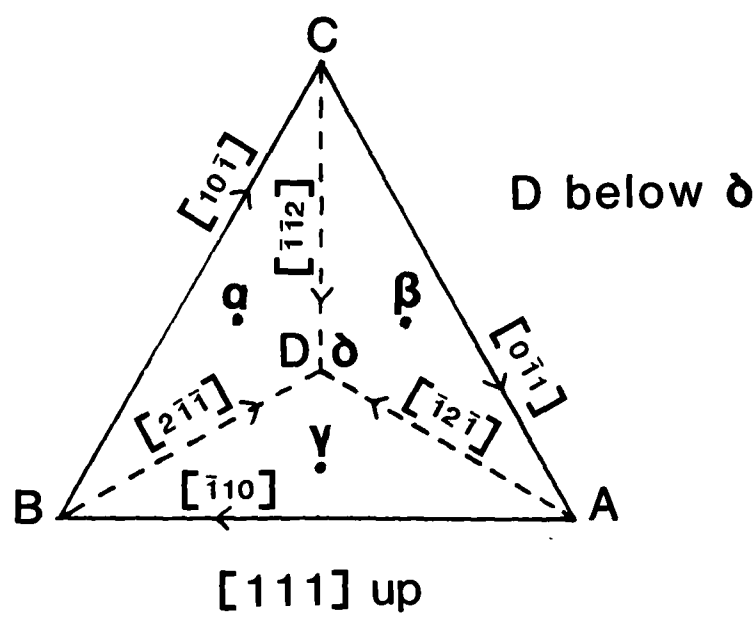
Single crystals were provided by Metals, Crystals & Oxides Ltd. of Cambridge. These had been grown by the Bridgman method and were found to contain an ideal dislocation density as received (see §2.2 below). These crystals were then orientated by Laue back-diffraction prior to being sectioned parallel to a (111) slip plane by spark machining. Discs of 3mm. diameter were then spark cut from these sections and electron transparent foils were prepared by double-jet electro-polishing at 28-30V in a solution of 4% perchloric acid in anhydrous methanol at -55°C. Perforation was detected by eye and the specimens immediately washed twice in anhydrous ethanol and twice in anhydrous methanol.

The pre-irradiation characterization was generally performed in a Philips CM30 TEM, although an EM430 microscope was also employed. Ideally long curved dislocations were chosen, to provide a range of orientations, which were characterized by weak-beam imaging at 200kV (a voltage at which sub-threshold damage was acceptable) employing the familiar $\tilde{g} \cdot \tilde{b} = 0$ invisibility criterion (Howie & Whelan, 1962). The stacking fault between partial dislocations was assumed to be intrinsic in nature. Hardy (1985) provides a discussion of the validity of this assumption. The nature of all stacking faults between partials was found to be the same throughout this study.

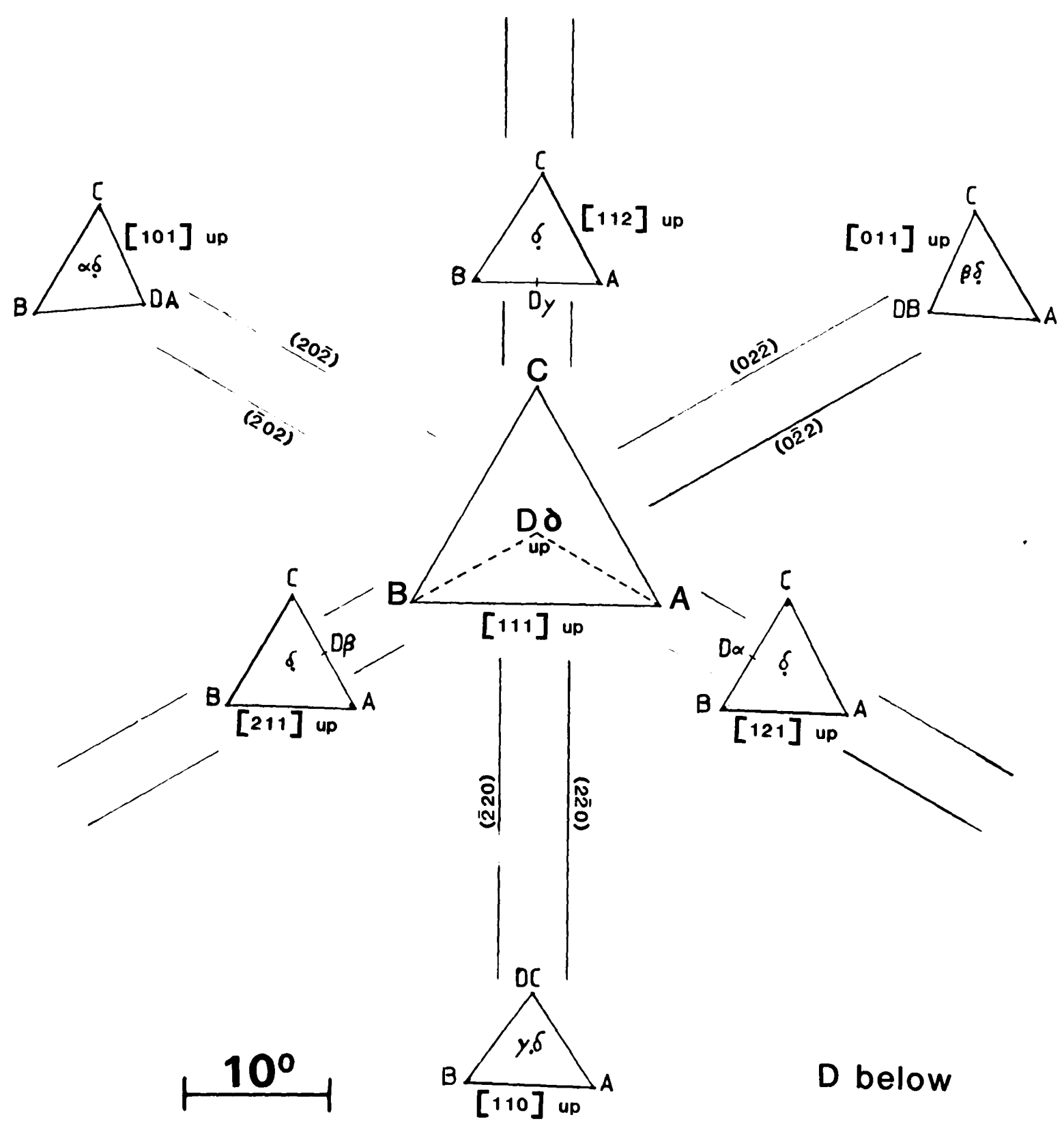
These dislocations were then irradiated with 1MeV electrons in the Argonne HVEM. The displacement threshold (E_d) has been measured to be ~ 18.5 eV ($\langle 110 \rangle$) in 316SS (Makin et al, 1977). The electron density was measured using a Faraday cup placed at the HVEM beam centre from which the level of displacement damage was calculated using a value for the displacement cross-section of 70 barns (Makin et al, 1977 - attributed to Oen, 1965). Irradiations were performed at 50kX magnification with a fully focussed beam. A typical beam current was 1nA which corresponds to a dose rate of 10^{-3} dpa/sec.. It was endeavoured to maintain a constant dose rate for all the experiments. In the event this did not prove possible and specimens were actually irradiated at dose rates ranging from $6 \cdot 10^{-4}$ to $1.3 \cdot 10^{-3}$ dpa/sec.. Beam heating is expected to be only a few degrees ($< 10^\circ\text{C}$ from Fisher, 1970). The Argonne heating stage was calibrated with an external thermocouple in a dummy column but experimental problems encountered in this procedure indicate that the uncertainty in temperature may be as high as $\pm 50^\circ\text{C}$.

The post-irradiation characterization was generally performed in a Philips EM430, although

Figure 2.1



THOMPSON TETRAHEDRON



the CM30 was also used. The area of reciprocal space within which post-irradiation characterizations were performed is shown in figure 2.1. Occasionally micrographs were taken in every pole shown, although it was generally found that micrographs in $\pm \tilde{g}$ of all $\langle 2\bar{2}0 \rangle$ -type $\left((111) \text{ pole} \right)$ and $\langle 11\bar{1} \rangle$ -type $\left(\{112\} \text{ poles} \right)$ reflections were adequate. Only a small percentage of these micrographs is shown in this thesis. Where possible true weak-beam conditions were used (e.g., **Cockayne**, 1972) but occasionally the deviation parameter (S) was reduced below $2 \times 10^{-2} \text{ \AA}^{-1}$ because a surface contaminant layer, resulting from the HVEM irradiation, reduced the already weak image intensities. This problem was particularly prevalent in irradiations of the 15% nickel alloy. The nature of loops were determined employing characteristic ‘inside/outside’ contrast (see figure 2.2 and, for example, **Loretto & Smallman**, 1975, p.77). The line direction is assigned to be clockwise around a loop. Both the FS/RH (**Bilby et al**, 1955; see **Hirth & Lothe**, p.20) convention and **Thompson’s** (1953) notation are employed throughout.

2.2 Dislocation Geometries

The main thrust of research in this volume is aimed at a study of isolated dislocations in thin films. The presence of neighbouring defect sinks, which may be biased^S_A, would be likely to alter the ratio of interstitials to vacancies arriving at dislocations. The behaviour of irradiated dislocations would then also be dependent upon the nature of surrounding sinks and thus may not be representative of the material under consideration. Since thin films are used the surfaces are likely to be one of the more dominant sinks. Surfaces, by reason of their low strain field interaction with point-defects, are generally assumed to be neutral sinks and thus their effect will be to alter only the total number of defects arriving at dislocations and not the ratio of interstitials to vacancies.

Almost all dislocations in the as-received material were found to be glissile in the plane of the foil. In a few instances dislocation densities were increased by slight bending around the stem of a small glass funnel prior to thinning. It was found that this introduced lengths of sessile dislocation where a pre-existing glissile dislocation crossed a slip trace (e.g. see figure 4.5).

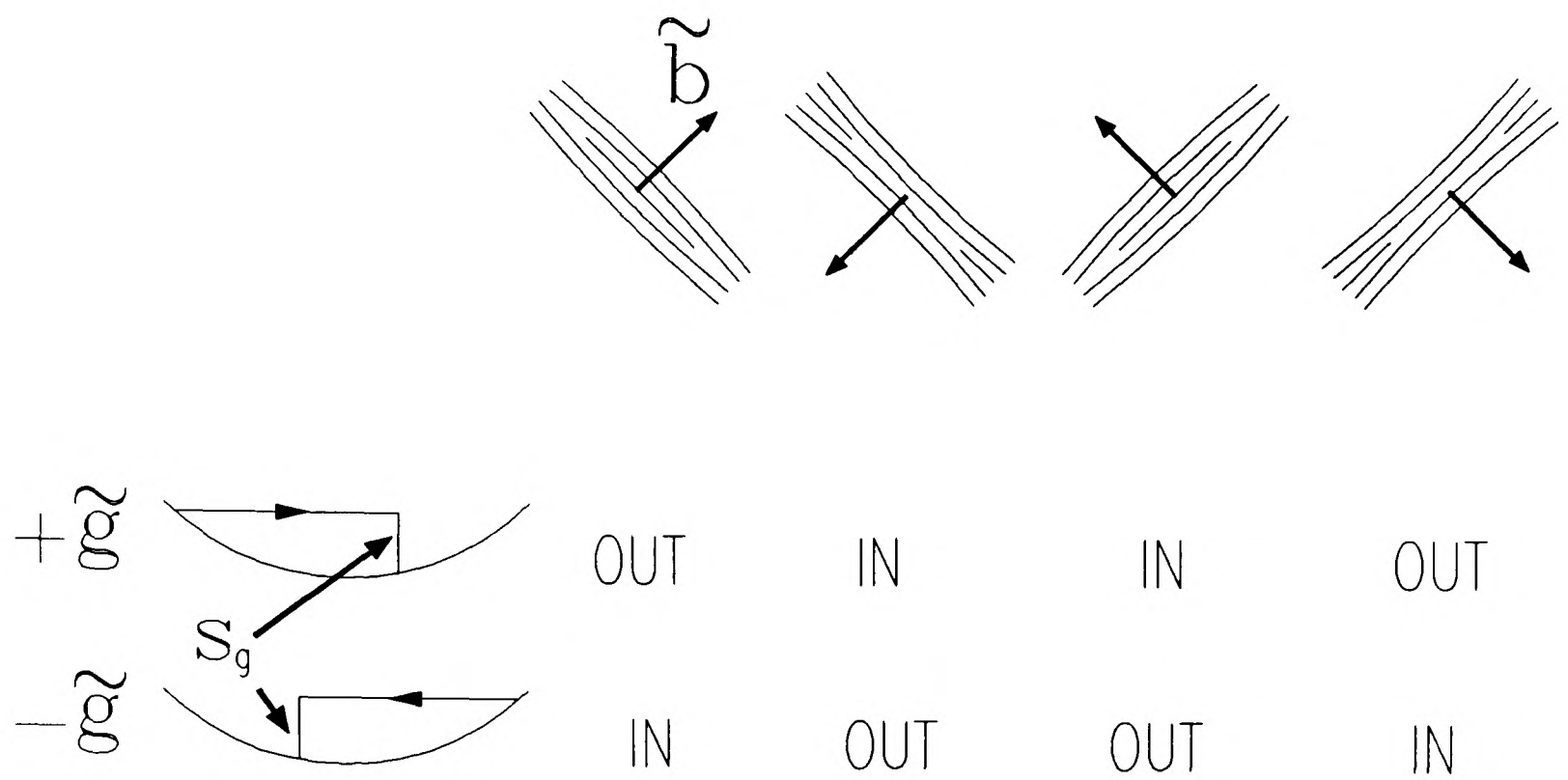
2.3 Irradiation Temperature

A scoping study was performed in the Argonne HVEM in 1987, before single crystal specimens were yet available, to determine an ideal temperature, or temperature range within which a reasonable density of resolvable defects which had interacted with the dislocation was seen. In many cases in-situ observations were made, imaging at 1MeV under bright-field kinematical (BFK) conditions. The temperatures studied were: Room temperature (20°C); $\sim 160^\circ\text{C}$; $\sim 280^\circ\text{C}$; $\sim 390^\circ\text{--}430^\circ\text{C}$; $\sim 475^\circ\text{C}$;

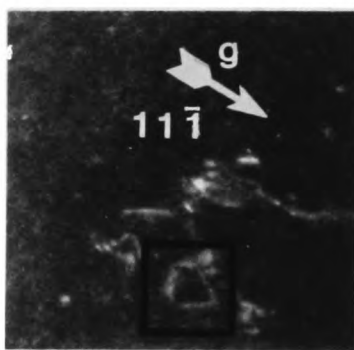
Figure 2.2

**Characteristics and Origins of 'Inside/Outside' Contrast
Shown by Dislocation Loops**

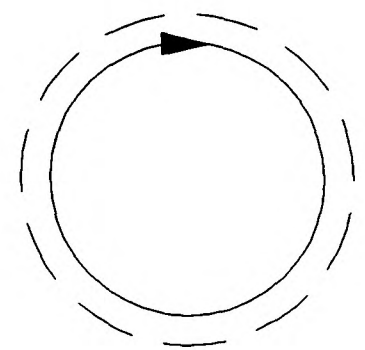
Inside/Outside Contrast



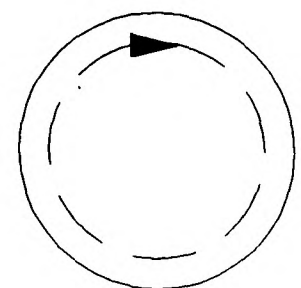
Viewed
from
above



$$(\vec{g} \cdot \vec{b}) s_g > 0$$



$$(\vec{g} \cdot \vec{b}) s_g < 0$$



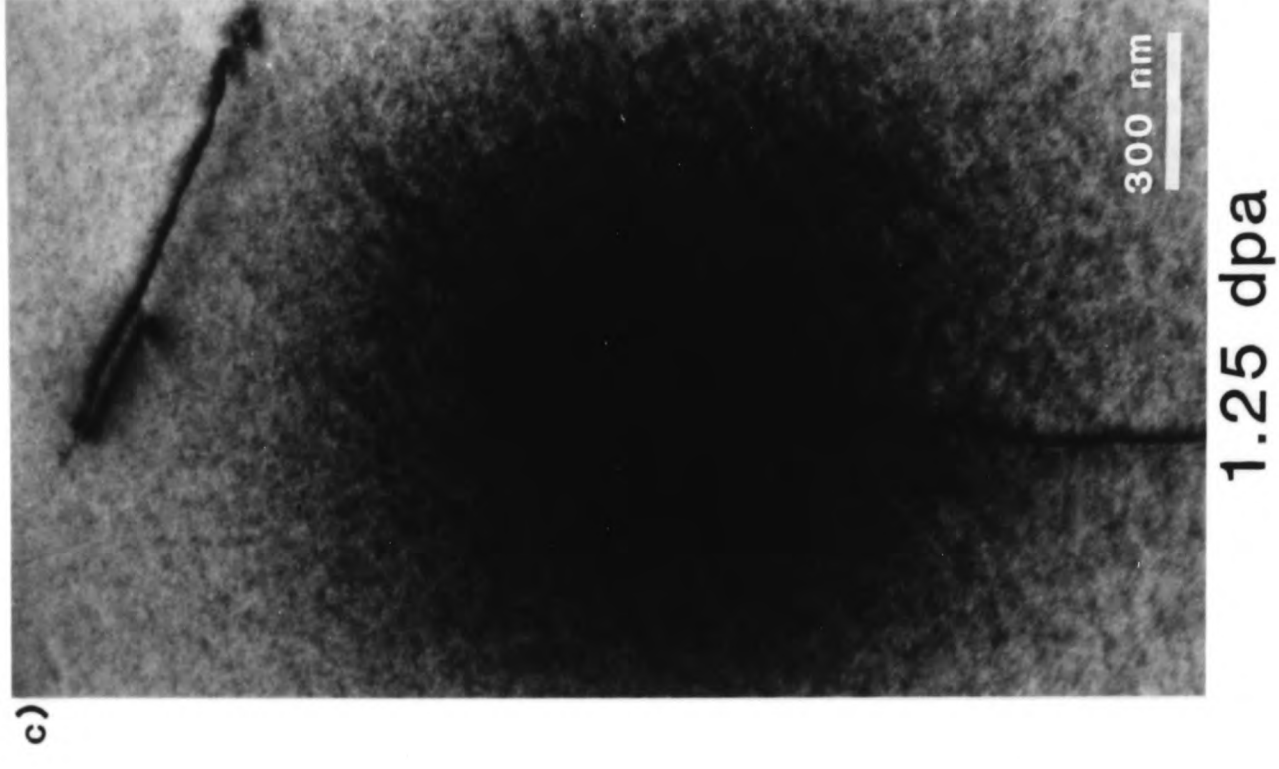
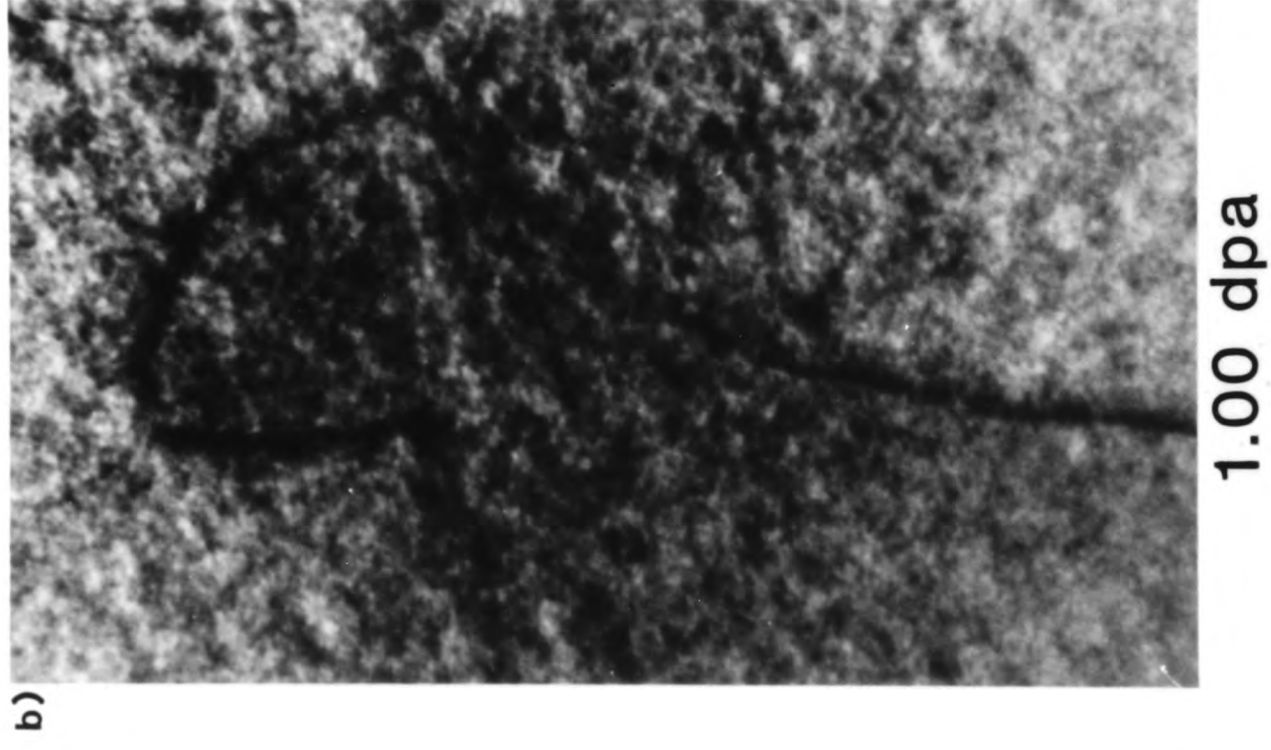
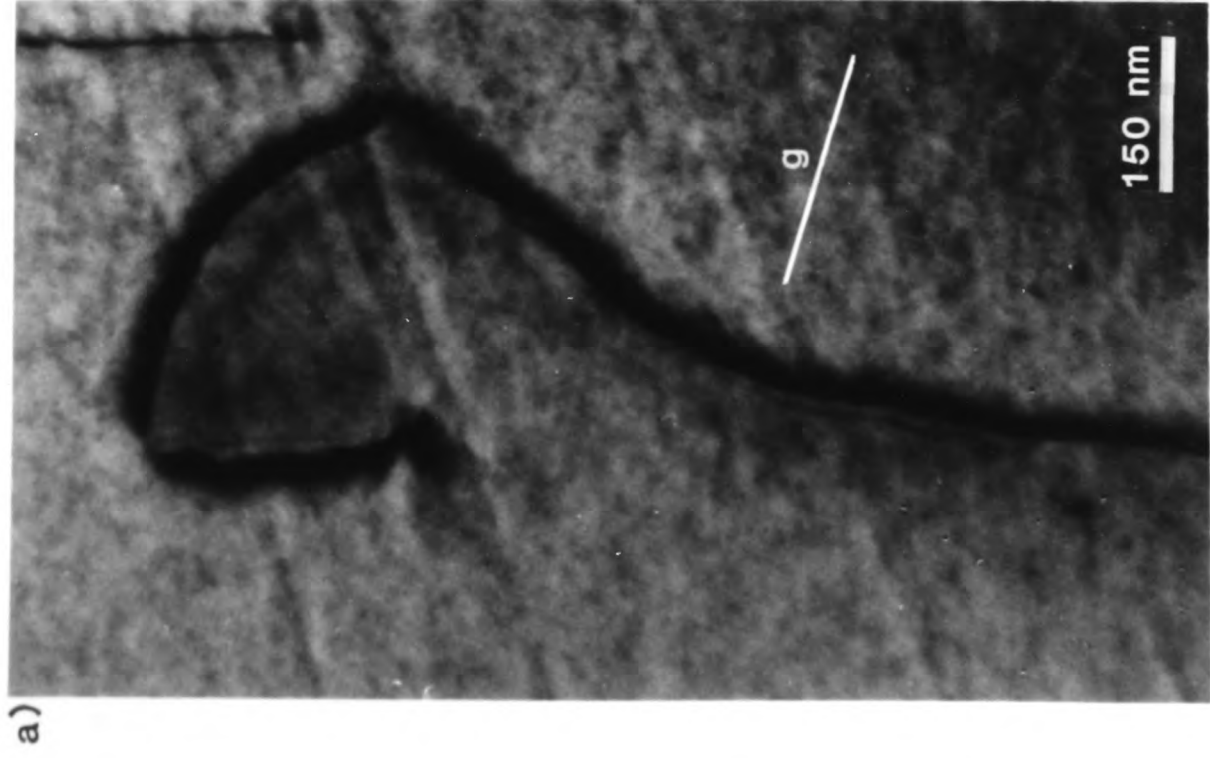
respectively. In micrograph (b) it is possible that some small changes in dislocation configuration can be seen and the irradiation was stopped at this point.

The specimen was then transferred to an EM430 for a higher resolution weak-beam study (micrographs (c) to (f)). Dislocation 'A' in micrograph (c) appears to have a complex zig-zag configuration, whilst dislocation 'B' is fairly straight. Dislocation 'C' also appears to show some features along its length (e.g. 'K'). It is also possible ((f)) that the cluster density close to the dislocations is greater than in the bulk. The differences between pre- and post-irradiation dislocation configurations are, however, slight when compared with the great density of background clusters visible in the weak-beam image (f). These clusters effectively prohibit detailed analysis. It was concluded, therefore, that the irradiation temperature must be increased still further to both reduce the background loop density and accelerate any interaction of defects and line.

In contrast with all room temperature irradiations the background clusters appear larger in size and fewer in number. In micrograph (d) many of them are visible as short bright streaks lying along the trace of planes β . In micrograph (e) the short bright streaks lie along the trace of planes γ . This is consistent with these defects being interstitial Frank loops, with Burgers vectors $\tilde{b}_L = \overrightarrow{\beta B}$ and $\overrightarrow{\gamma C}$ showing 'inside' contrast in $\tilde{g} = [20\bar{2}]$ and $\tilde{g} = [\bar{2}02]$ respectively. The formation of vacancy loops is not apparent.

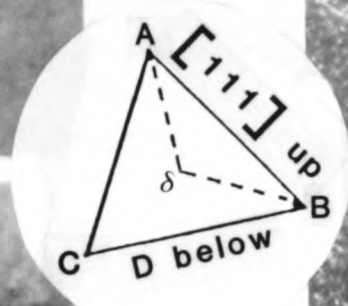
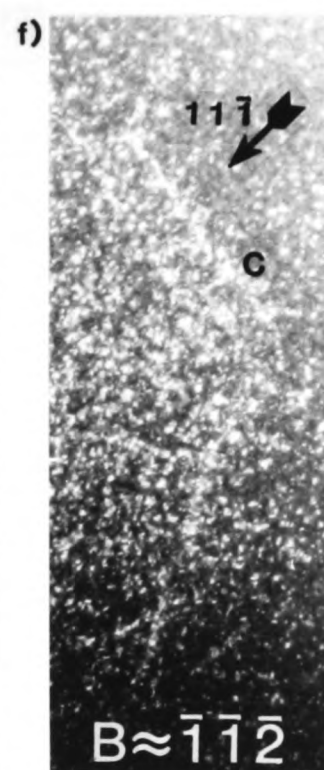
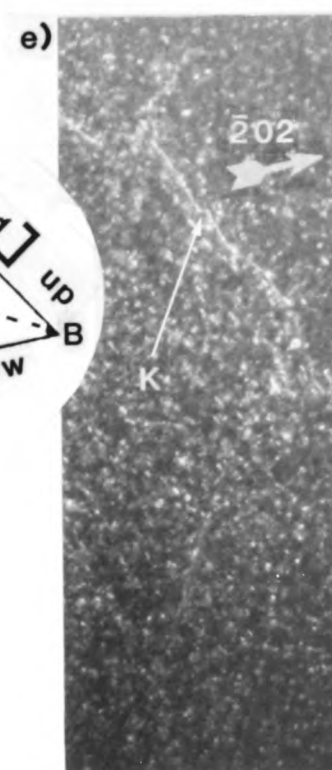
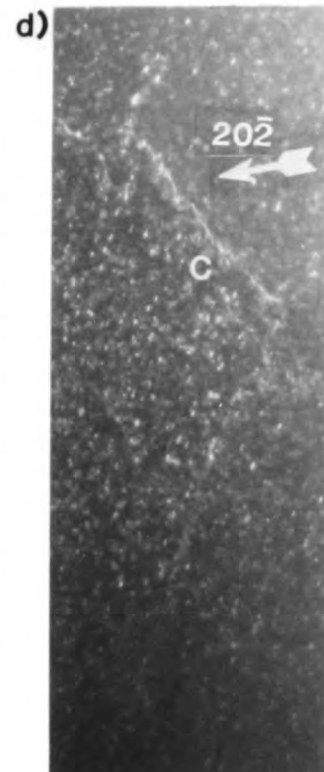
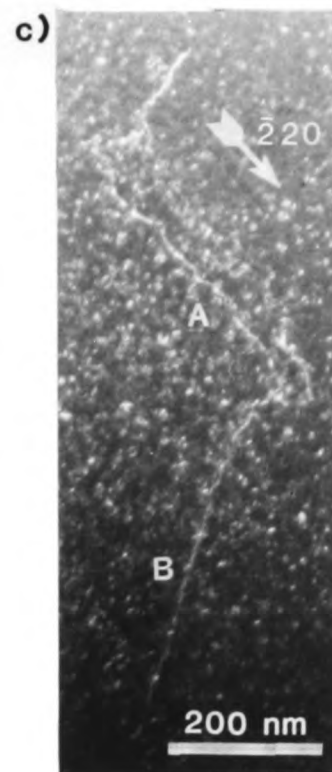
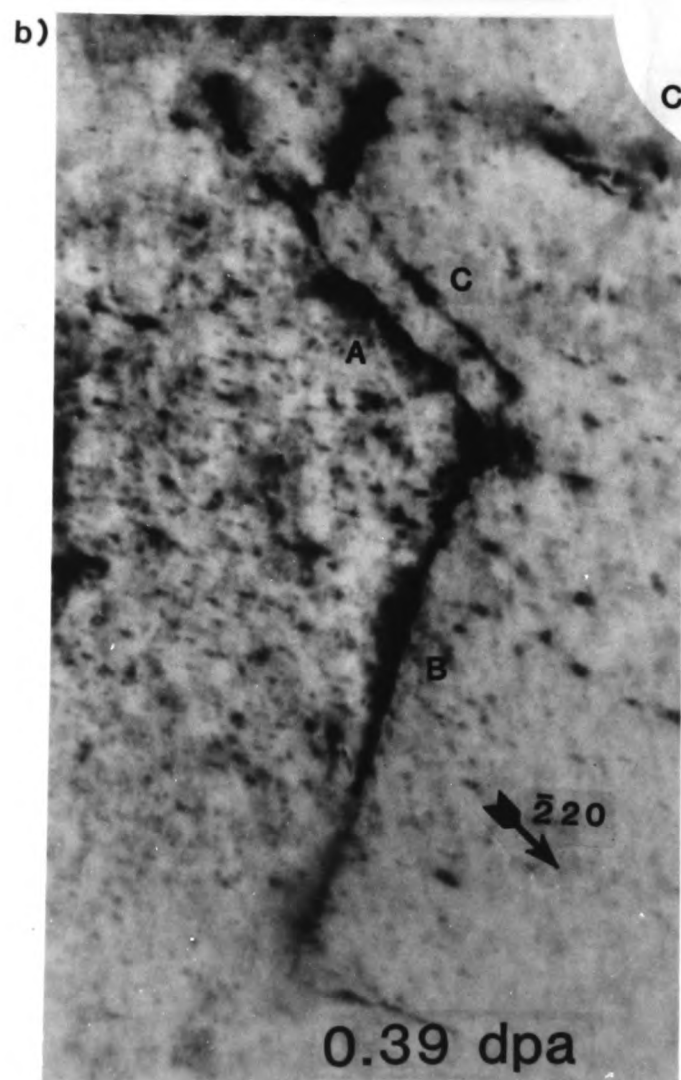
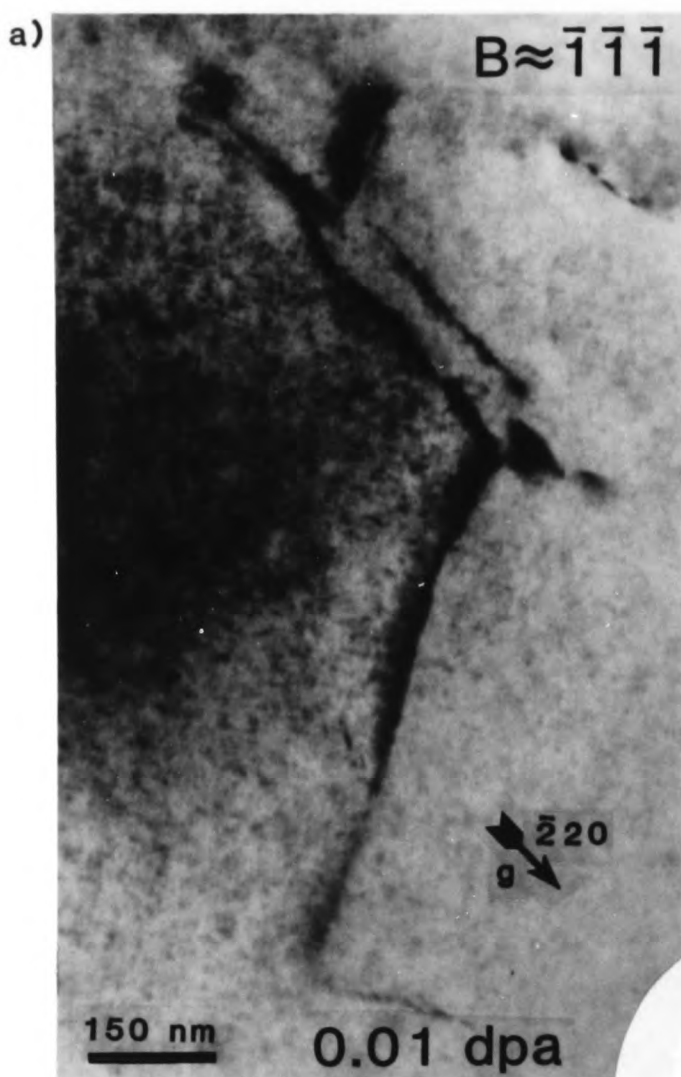


Figure 2.3



Room Temperature Irradiation of Dislocation in Fe-15%Ni-17%Cr-0.5%Si (at.%)

Figure 2.4



Pure Screw and 60° Dislocations
in Fe-15%Ni-17%Cr-0.5%Si (at.%)

0.39 dpa at $\sim 280^\circ\text{C}$

Introduction to Materials Chapters

The following five chapters each deal with a single material, and consist of detailed presentation and discussion of figures. Each figure, which is usually a montage of micrographs, is considered in turn. Text relating to each figure, or group of figures, is usually sub-divided into **‘Observation’** and **‘Interpretation’** sections. Each chapter is then concluded with a **‘Points of Discussion’** section and an overall **Summary**.

‘Observation’: As the title indicates, these sections describe experimental observations made from a series of micrographs, part or all of which are shown in the corresponding figure. Where a contrast analysis can be performed with reasonable surety it is included within these sections. In the mass of scientific literature, “*results*” normally consist of these sections alone.

‘Interpretation’ of contrast from individual features seen in a series of micrographs is central to this work. As a result occasionally lengthy consideration must be given to assessing the likelihoods of possible configurations before and after irradiation. This rather tedious process, if left entirely to the discussion chapter (chapter 8) would render this thesis extremely difficult to follow; the reader continually having to check back to the relevant results section and figure.

‘Points of Discussion’: Aspects of the discussion which relate particularly to observations made on a given material are contained within these sections. As the reader progresses through the five chapters it is hoped, therefore, that (s)he will gain a graduated impression of the fundamentally similar mechanisms by which interstitials and vacancies interact with pre-existing dislocations in each alloy. This allows the discussion chapter (chapter 8) to concentrate on more general aspects arising from this work.

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Chapter 3

Irradiation of Fe-15%Ni-17%Cr alloy

Figure: 3.1; 50°-70° Dislocations in Fe-15%Ni-17%Cr; 0.05 dpa at ~ 400°C.

Observations:

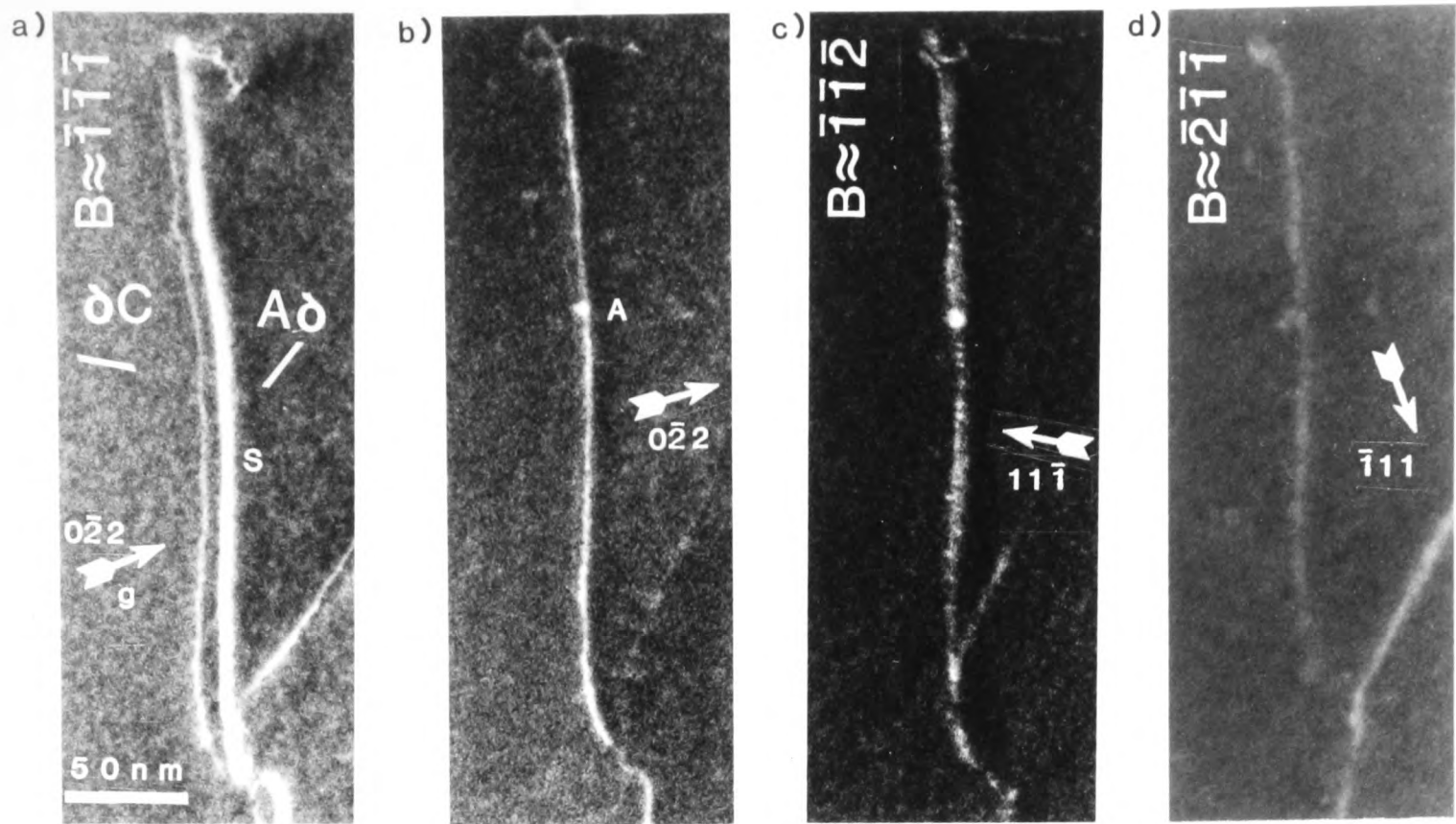
In common with almost all other figures in this thesis the micrographs shown here are selected from a larger contrast series. Two regions, which are highlighted in the low magnification print at lower right, have been enlarged. The right-hand region (1), containing a 70-80° dislocation section with total Burgers vector $\tilde{b}_T = \vec{AC}$, is shown in micrographs (a) to (d). Micrographs (a) and (b) show $|\tilde{g} \cdot \tilde{b}_T| = 2$ for the dislocation before and after irradiation to 0.05 dpa at ~ 400°C respectively. Micrographs (c) and (d) are taken after irradiation. In (c) the near edge partial and stacking fault are imaged whilst (d) has $\tilde{g} \cdot \tilde{b}_T = 0$ and shows the stacking fault only. Before irradiation ((a)) the dislocation appears evenly and widely dissociated. The partial image separation at 'S' (70°) is ~ 105Å.

Micrographs (e) to (h) show the second, left-hand region (2) which contains a near 60° dislocation with $\tilde{b}_T = \vec{BC}$. Micrographs (e) and (g) are taken before, and (f) and (h) after, irradiation. All four micrographs have $|\tilde{g} \cdot \tilde{b}_T| = 2$. Before irradiation the left hand (edge) partial $\vec{\delta C}$ is heavily constricted (see (e)), possibly indicating the presence of jogs (Carter & Ray, 1974).

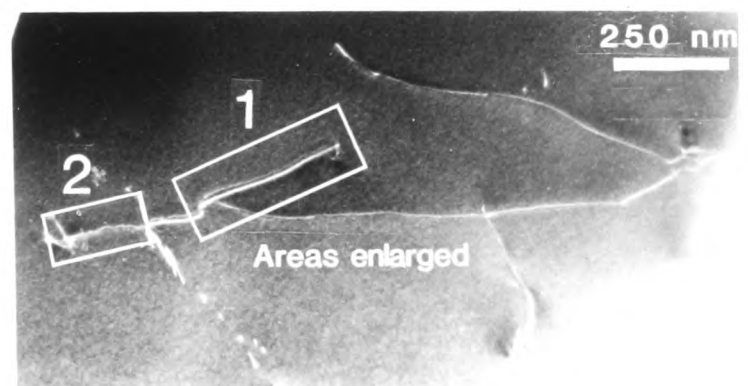
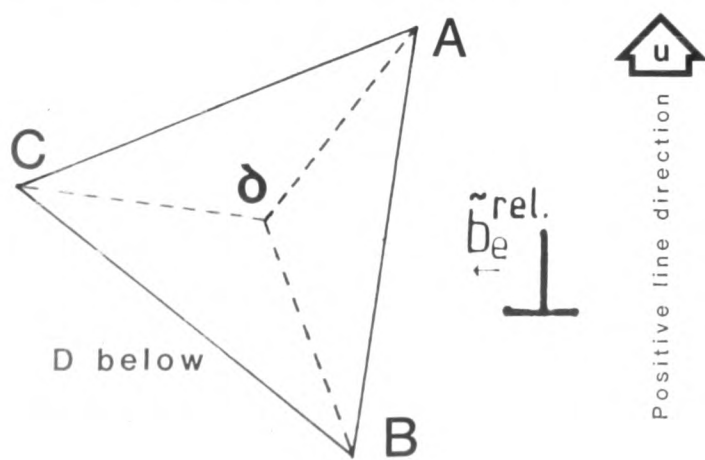
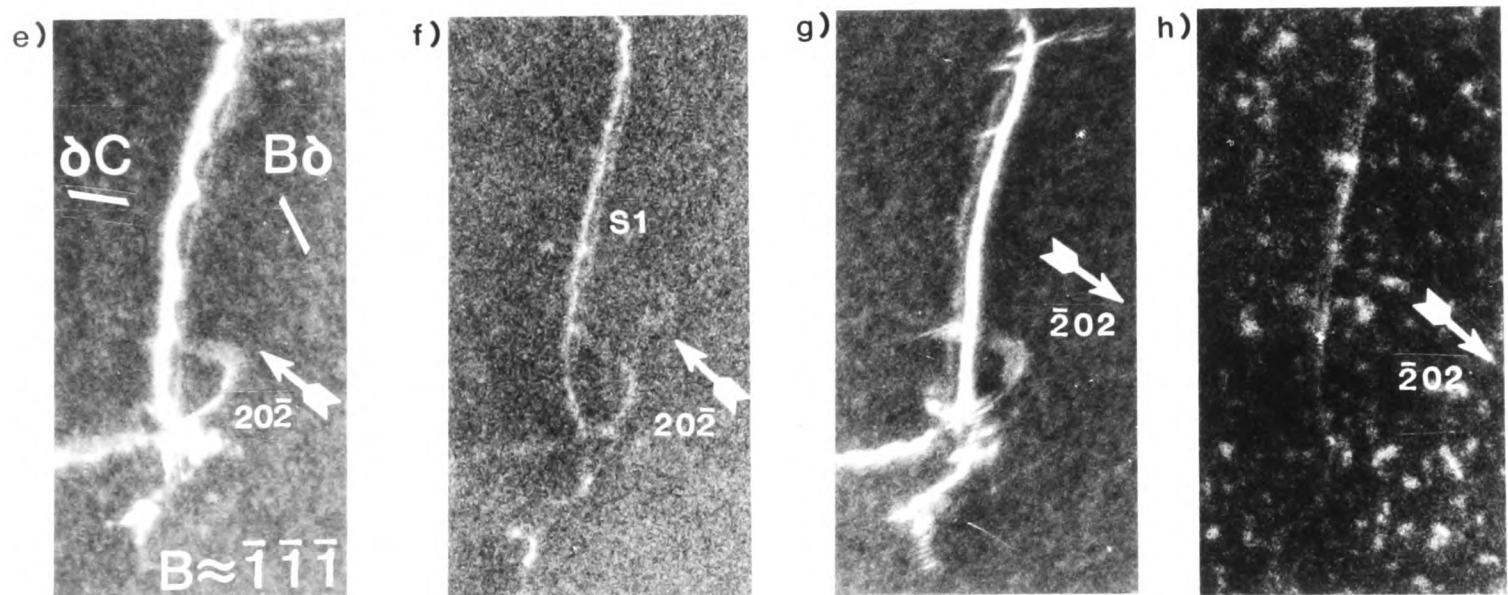
After irradiation both dislocations have a narrower partial separation but remain dissociated. In micrograph (b) it is difficult to see the continuing, even dissociation of dislocation 1, however the presence of even and unbroken, if weak, stacking fault contrast (SFC) in micrograph (d) suggests this to be the case. This result is confirmed by other (not shown) reflections in the full contrast series which show no sign of constrictions along the near edge partial $\vec{A\delta}$. For dislocation (2) the continuing dissociation is plainly visible in micrograph (f). The partial image separation at point 'S1' (60°) is ~ 40Å. It is noticeable that constrictions seen along this dislocation before irradiation have disappeared. No new constrictions have formed along either dislocation and there is no evidence for major climb motion of the lines.

In micrograph (b) a large cluster ('A') is imaged. This cluster is visible in all $\tilde{g} = \pm \langle \bar{1}11 \rangle$ (not all shown) type reflections suggesting that it is faulted. It is also visible in $\tilde{g} = \pm [20\bar{2}]$ but is apparently out-of-contrast in $\tilde{g} = \pm [2\bar{2}0]$ (not shown). This suggests a Burgers vector $\pm \vec{\gamma C}$. However, notice firstly that on tilting the foil to bring planes γ vertical ($\tilde{B} \approx [\bar{1}\bar{1}\bar{2}]$; (c)) feature 'A' does not appear as a thin line, suggesting that it is not confined to a plane γ . Secondly, this feature apparently shows 'inside' contrast in $\tilde{g} = [0\bar{2}2]$ ($\Rightarrow \tilde{b} = \vec{\gamma C}$) and $\tilde{g} = [11\bar{1}]$ ($\Rightarrow \tilde{b} = \vec{C\gamma}$). Contrast exhibited by this

Figure 3.1



50°–70° and 70°–80° dislocations in Fe–15%Ni–17%Cr (at.%)
0.05 dpa at ~400°C



feature therefore appears inconsistent with any simple Frank or perfect loop variant.

In micrograph (c) the formation of a line of very fine scale cluster damage is evident. These tiny clusters are seen very close to the stacking fault although the foil has been tilted through $\sim 20^\circ$ to $\tilde{B} \approx [\bar{1}\bar{1}\bar{2}]$. Feature 'A' also shows no substantial displacement from the dislocation image. Similar tiny clusters are also seen along dislocation (2) in other reflections (not shown) of the full contrast series. In both cases these clusters could not be characterized directly using either the $\tilde{g} \cdot \tilde{b} = 0$ invisibility criterion or characteristic inside/outside contrast (see chapter 2).

Figure: 3.2; 65° Dislocation segment in Fe-15%Ni-17%Cr; 0.07 dpa at $\sim 420^\circ\text{C}$.

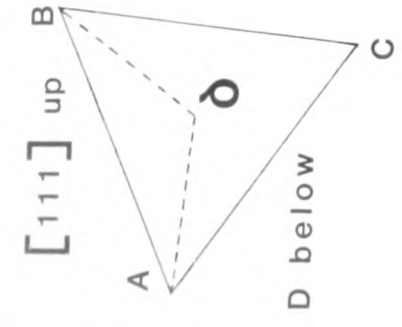
Observations:

The formation of another large cluster along a near 60° dislocation, after a slightly higher dose, is shown in this figure. Micrograph (a) shows $|\tilde{g} \cdot \tilde{b}_T| = 2$ before irradiation and micrographs (b) to (e) are taken after irradiation. Before irradiation the dislocation contains a large number of constrictions, as evidenced by the heavily cusped appearance of the near-edge partial $\vec{A}\delta$ in (a). The partial image separation at 'S' (60°) is $\sim 85 \text{ \AA}$. After irradiation this partial now appears smoother (see (b) and (c)) suggesting the disappearance of these constrictions. In micrograph (b) it is difficult to discern a separate image from the left-hand partial $\vec{\delta B}$, however contrast arising from the stacking fault separating the two partials is plainly visible in (d). The partial separation has narrowed but the dislocation is still dissociated. There is no evidence for major climb motion of the line.

Micrographs (b) to (e) all show the presence of feature 'A'. This feature is not visible in $\tilde{g} = \pm [0\bar{2}2]$ or in $-\tilde{g}$ of the reflections of the above micrographs which is consistent with an interstitial Frank loop of Burgers vector $\vec{\beta B}$. However, micrograph (e) apparently shows this feature as a thin line. This micrograph is taken with planes α close to the vertical ($\tilde{B} \approx [\bar{1}\bar{2}\bar{1}]$) and might suggest that feature 'A' lies on a plane α . Therefore, whilst the intensities of contrast arising from this feature are consistent with an interstitial $\tilde{b} = \vec{\beta B}$ Frank loop its shape is not. Contrast intensity exhibited by this feature is not consistent with either a $\tilde{b} = \pm \vec{A}\alpha$ Frank loop (it is visible in $\tilde{g} = [20\bar{2}]$) or with any possible product ($\pm \vec{A}\vec{D}$, $\vec{A}\vec{C}$ or $\vec{A}\vec{B}$) from the unfauling of such a Frank loop. It should be noted that this feature lies very close to the dislocation although it has not apparently reacted with the line (see (b)). In this respect it is very similar to the large cluster ('A') on dislocation (1) of figure 3.1.

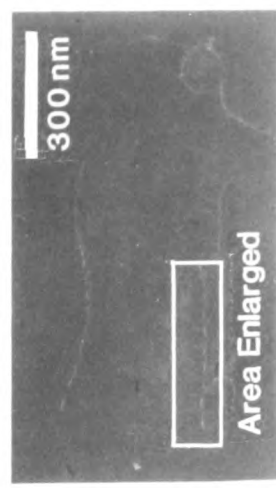
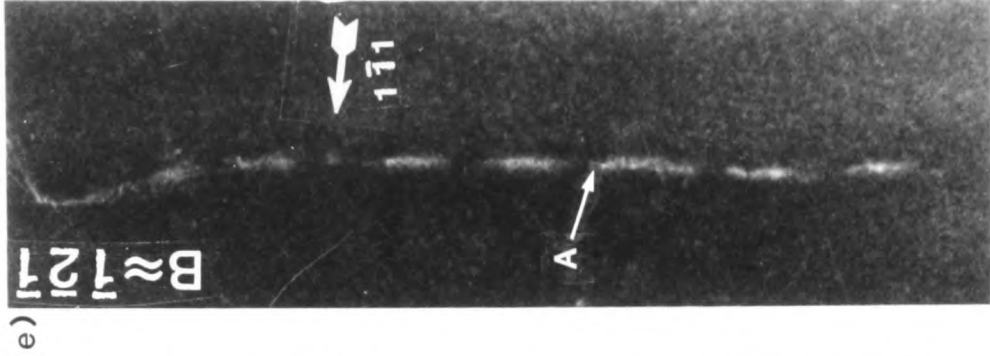
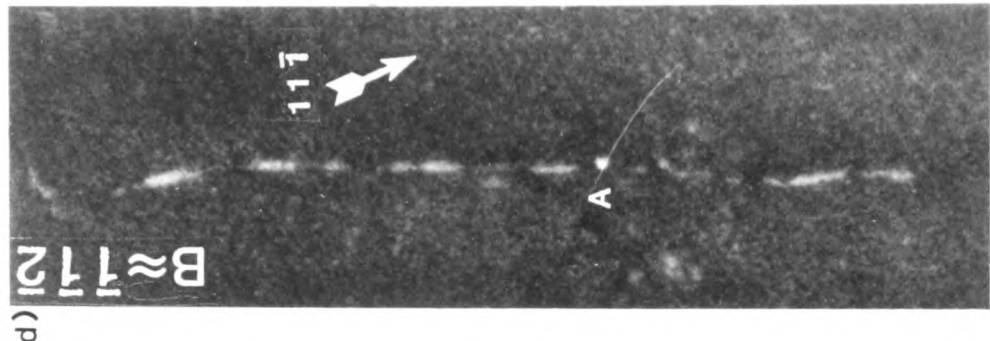
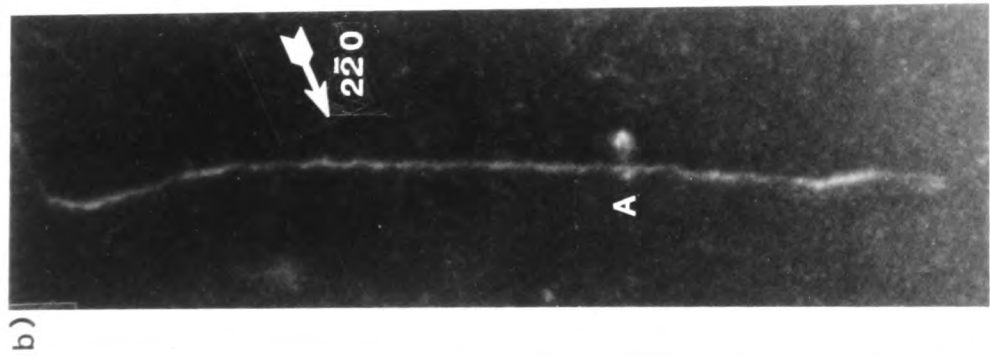
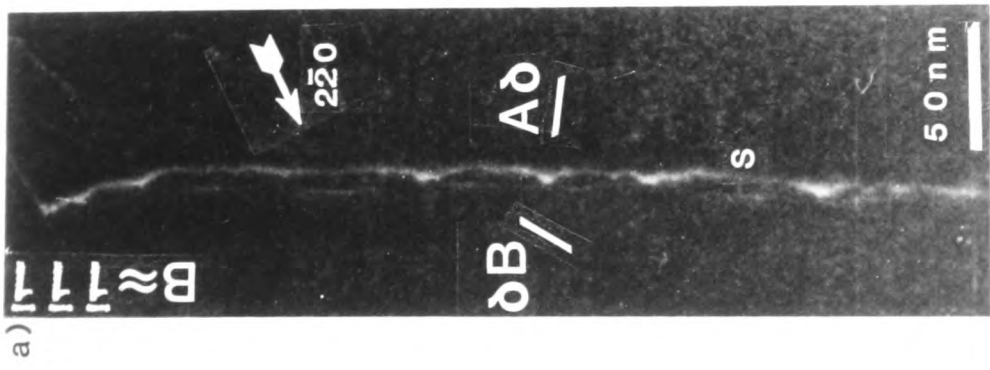
Finally, also in common with figure 3.1, the formation of fine scale cluster damage very close to the stacking fault is suggested by micrograph (e), particularly in the region just below 'A'.

Figure 3.2



Positive line direction

$$\vec{T} \rightarrow \vec{b}_e^{rel.}$$



65° Dislocation segment in Fe-15%Ni-17%Cr (at.%)

0.07 dpa at ~420°C

Figure: 3.3; Dislocations in Fe-15%Ni-17%Cr; 0.07 dpa at $\sim 420^\circ\text{C}$.

Observations:

In figure 3.3 a complex network of dislocations is present before irradiation ((a)), consisting mainly of dislocation 'A'. This dislocation contains a number of constrictions and frequently showed inside/outside contrast typical of a dipole (see figure 1.3). Its detailed pre-irradiation configuration has not been determined.

After irradiation ((b)-(d)), dislocation 'A' is dramatically smoother in appearance. The upper portion of the line seems to have annihilated whilst the lower portion has straightened from its highly curved pre-irradiation configuration. Most constrictions have been removed and a large cluster ('B') is seen on the line.

In common with the large clusters seen in the preceding two figures, it was not possible to characterize this feature using standard techniques ($\tilde{g}\cdot\tilde{b}=0$ and inside/outside contrast). However, when this cluster is viewed at extremely high magnification, as in micrograph (d), arrowhead contrast typical of vacancy stacking fault tetrahedra (SFT) is suggested in some reflections. Notice here that the out-of-contrast edge is parallel to the $\langle\bar{2}20\rangle$ type $\tilde{g}$ as expected for SFT. Micrograph (c) also shows evidence for fine scale cluster formation close to, or on the stacking fault. This is particularly evident along segment 'C' but may also be seen at other points along the line. It should be noted that segment 'C' lies closest to the pre-irradiation dislocation position. The partial separation has decreased at 'C' but the dislocation remains widely dissociated. The discontinuity in SFC along section 'C', seen in micrograph (c), is not echoed by any other reflection of the full contrast series; it is believed that this is an artifact of the weak-beam conditions employed for this micrograph. This micrograph was selected because it best shows the fine scale cluster formation.

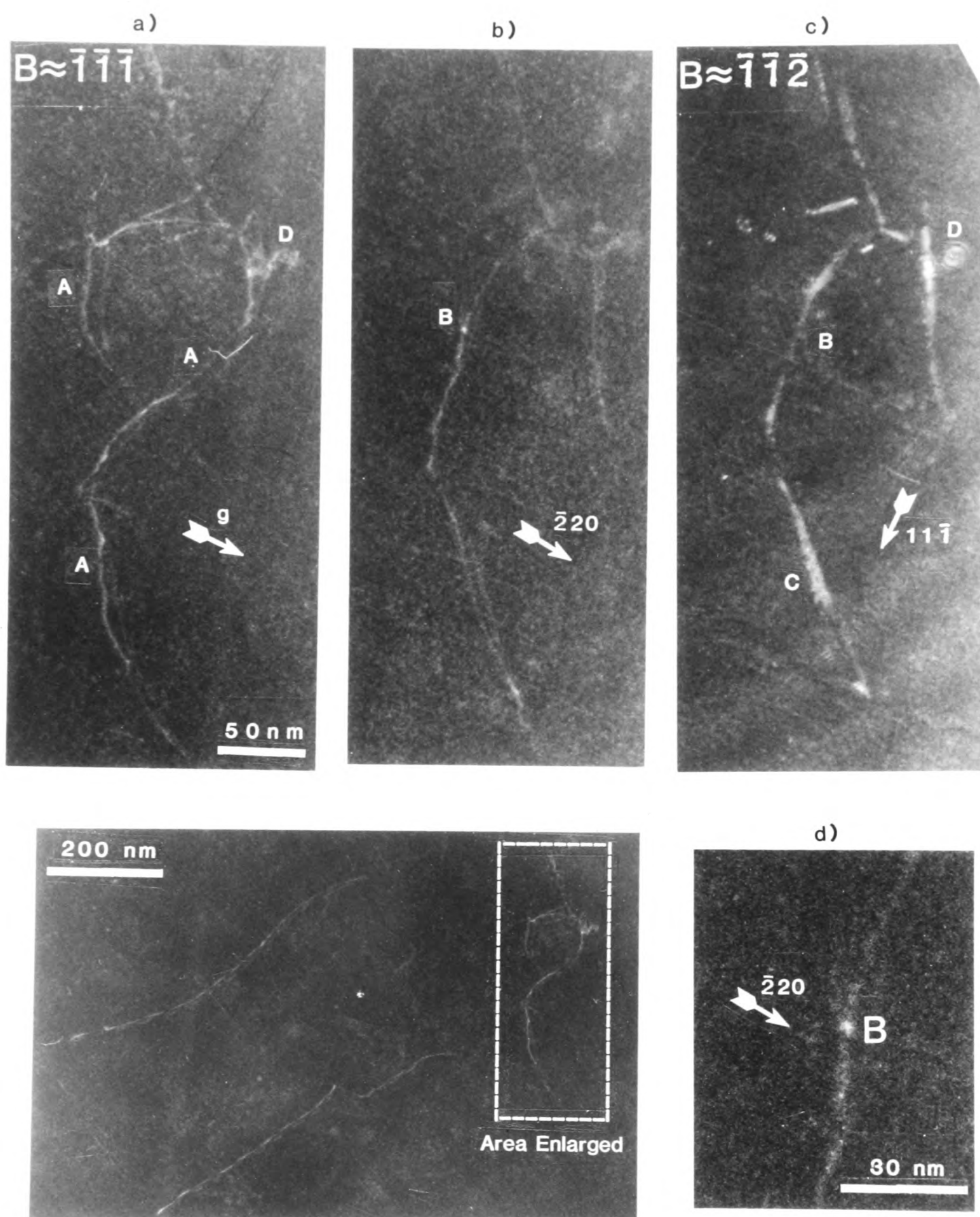
Lastly, feature 'D', part of which is visible before irradiation ((a)), has grown to assume a perfectly round shape. This loop showed contrast consistent with an interstitial Frank loop, with Burgers vector $\tilde{b}_L = \vec{D}\delta$.

Figures: 3.1, 3.2, 3.3

Interpretation:

Features 'A' and 'B' of the above figures appear similar: They all lie very close to their parent dislocations and exhibit contrast inconsistent with any Frank, or perfect loop variety. Their proximity to the line suggests that they have nucleated within the strain field of the dislocation core. The characterization of feature 'B' of figure 3.3 above as a three dimensional vacancy cluster may thus possibly be representative of most or all these features. In that case sufficient vacancies have

Figure 3.3



Dislocations in Fe-15%Ni-17%Cr (at.%)
0.07 dpa at $\sim 420^\circ\text{C}$

agglomerated for the feature to be recognized as a complete, or nearly complete, SFT. Other small features, which cannot be identified unambiguously as SFT, may also be vacancy clusters: Very similar features have previously been seen to grow to become identifiable as SFT during an in-situ irradiation experiment in silver (Jenkins et al, 1987). The characteristic of features 'A' is that they lie on pure 60° sections of dislocation. They may also be vacancy clusters which have not developed into recognizable SFT. However (indirect) evidence to be presented later (see chapter 8) allows the possibility that in this material larger clusters formed on 60° dislocations may actually be interstitial in nature.

The direct determination of the nature of these clusters, which is of course desirable, is unfortunately complicated by the proximity of the dislocation image, and possibly also the depth of the defect in the foil: In many cases the superimposition of cluster and line images may lead to incorrect determination of inside or outside contrast whilst the sensitivity of weak-beam images to defect depth may lead to artificially weak contrast. This latter effect is of particular importance when defect sizes are $\lesssim$ the effective extinction distance (ξ_{eff} , see, e.g., Hirsch et al, 1965).

The formation of a line of fine scale cluster damage lying parallel to mixed, and near-edge dislocations is a very common observation throughout this volume. Complete and semi-formed SFT are frequently interspersed within these lines of cluster damage. However, these clusters apparently do not form close to screw dislocations, as is illustrated next.

Figure: 3.4; Screw Dislocation in Fe-15%Ni-17%Cr; 0.05 dpa at $\sim 400^\circ\text{C}$.

Observations:

A right-handed screw dislocation containing a number of pre-existing constrictions, some of which are indicated ('J'), is shown in micrograph (a). These constrictions are likely to indicate the presence of jogs (Carter et al, 1974 & 1977). After irradiation ((b)-(l)) the dislocation has assumed a sharply pointed zig-zag configuration. The spacing between the zig-zags appears very similar to that between pre-existing jogs ($\simeq 40-85\text{ nm.}$). Micrographs (d) and (e) show $|\tilde{\mathbf{g}} \cdot \tilde{\mathbf{b}}_{\text{T}}| = 2$ for the dislocation after irradiation and evidence that some parts of the line are still dissociated is confirmed by SFC seen in micrograph (f). Elsewhere the dislocation appears to be constricted. It is very noticeable that the dislocation is now composed of straight sections. In many reflections the dislocation shows a simple, periodic, zig-zag structure. However, careful study of almost any zig-zag reveals a threepenny bit structure; 'zig' and 'zag' being separated by very short ($\sim 10-20\text{ nm.}$) segments. On tilting the foil away from $\tilde{\mathbf{B}} \approx [\bar{1}\bar{1}\bar{1}]$ projections of these short segments sometimes apparently lengthen, suggesting that they are inclined in the foil. In general segments at the top of a zig-zag (as the figure is viewed) appear long for $\tilde{\mathbf{B}} \approx [\bar{2}\bar{1}\bar{1}]$ and extremely short for $\tilde{\mathbf{B}} \approx [\bar{1}\bar{1}\bar{2}]$. Segments at the bottom of each zig-zag

show similar, but opposite behaviour indicating that they are inclined in the opposite sense. It may be confirmed that the apparent presence of connecting segments is not an artifact of the weak-beam image by noting that they (the connecting segments) remain visible in both $\pm \tilde{g}$ of any particular reflection (see segments at 'J' in (d) and (e), for example).

Micrograph (k) images a steeply inclined segment 'Y'. A directional analysis, discussed below, showed this segment to be glissile on γ and to lie along $[\bar{2}\bar{1}\bar{3}]$. It is uncertain whether this segment was present before irradiation. The foil orientation for micrographs (k) and (l) is shown to their right.

The evolution of the dislocation to form perfectly straight sections suggests alignment along low-index crystallographic directions. However, 'parallel' sections often appear slightly misaligned. Individual misalignments vary as the foil is tilted but are typically $\sim 5-10^\circ$.

Several other points should also be noted here:

- Most 'zigs' and 'zags' appear dissociated. All connecting segments appear constricted;
- No evidence is seen either for the formation of large clusters along the line, or for the very fine scale cluster formation, on, or very close to the stacking fault, as seen after irradiations of mixed dislocations (e.g. see figure 3.3 (c));
- The Burgers vector of the dislocation ($= \vec{AB}$) appears unaltered after irradiation.

A high density of loops are seen to have nucleated in the background. Two of each loop type ('A', 'B', 'C', 'D') are labelled. Loops 'A' are visible in $\tilde{g} = \pm [0\bar{2}2]$, $[\bar{2}20]$, $[11\bar{1}]$ and $[\bar{1}11]$. They are out-of-contrast in $\tilde{g} = \pm [20\bar{2}]$ and $[1\bar{1}1]$. These loops are also aligned along $[10\bar{1}]$ and exhibit "inside" contrast in $\tilde{g} = [02\bar{2}]$, $[\bar{2}20]$, $[11\bar{1}]$ and $[\bar{1}11]$. This contrast is entirely consistent with a planar interstitial Frank loop of Burgers vector $\vec{b}_L = \vec{\alpha A}$. These loops are not visible in $\tilde{g} = \pm [1\bar{1}1]$ because the foil is tilted to bring planes α to the vertical; the loops are therefore viewed edge-on and displacements due to their opposite sides exactly cancel for each column through the crystal. Features 'B', 'C' and 'D' are similar interstitial Frank loops with Burgers vectors $\vec{\beta B}$, $\vec{\gamma C}$ and $\vec{D\delta}$ respectively. None of these background loops appears to have unfaulted. In micrograph (d) one of the loops 'A' appears to lie on the dislocation; reference to micrographs (f) and (g) show, however, that this loop has not reacted with the line and actually lies some distance below it. Background interstitial Frank loops were also seen after the irradiation of figure 3.1 ($\sim 400^\circ\text{C}$) although very few are contained within the area of the

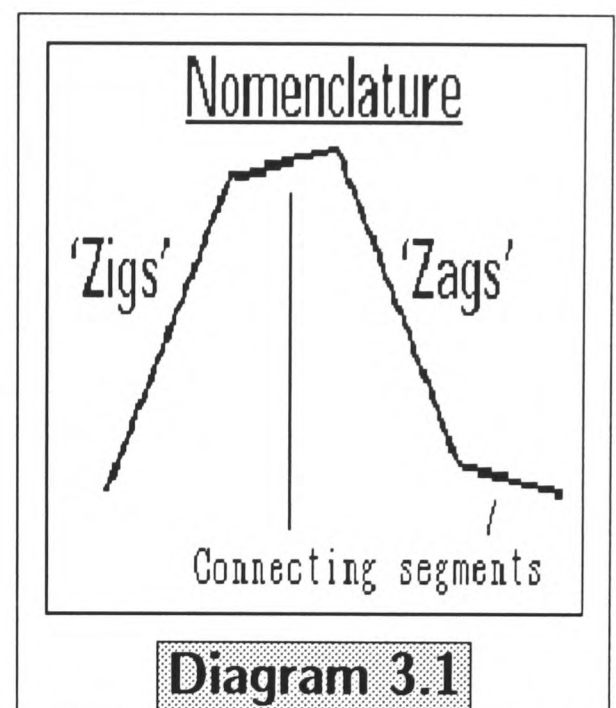
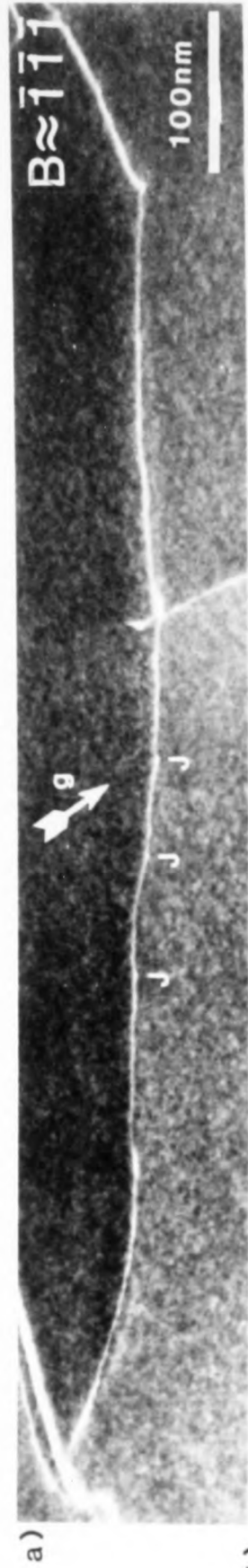
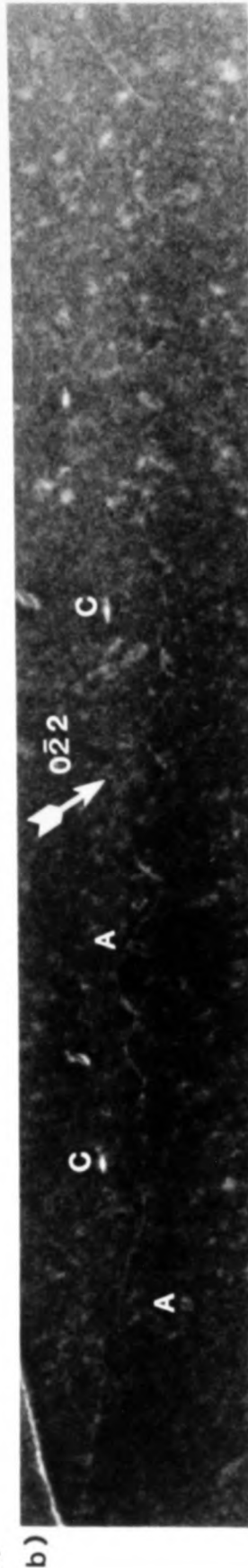


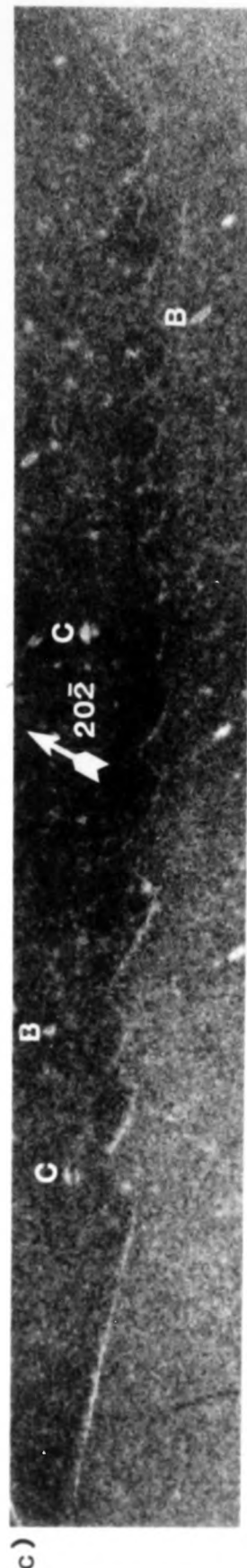
Figure 3.4
(3 pages)



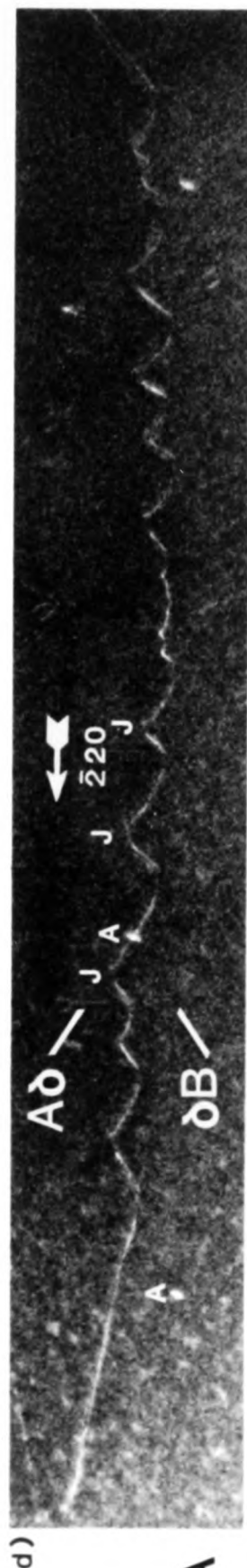
a)



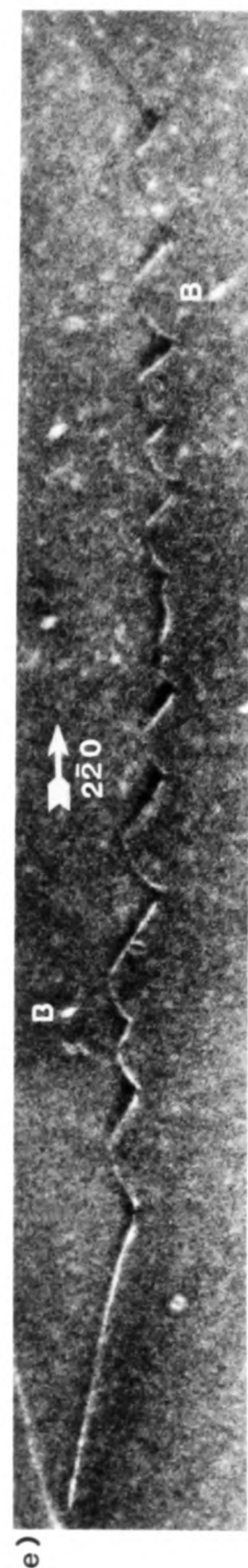
b)



c)

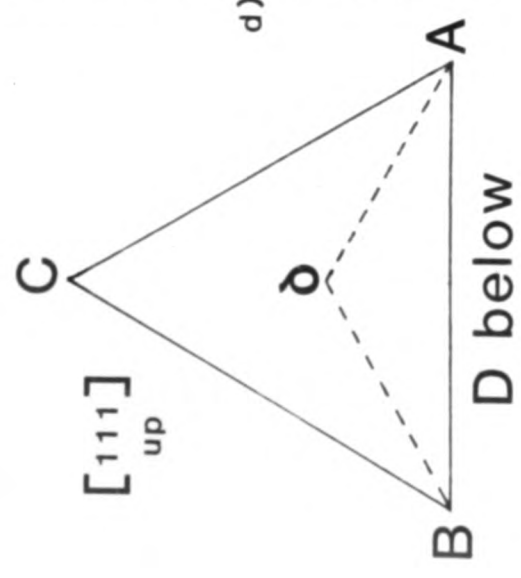


d)



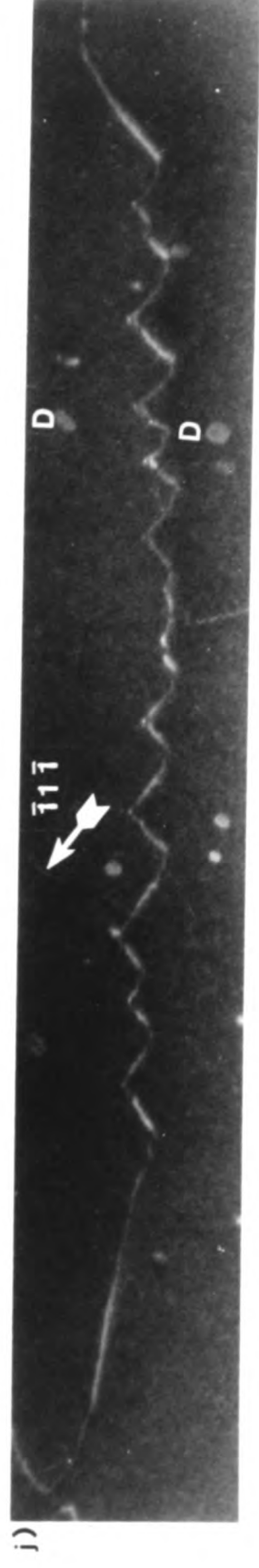
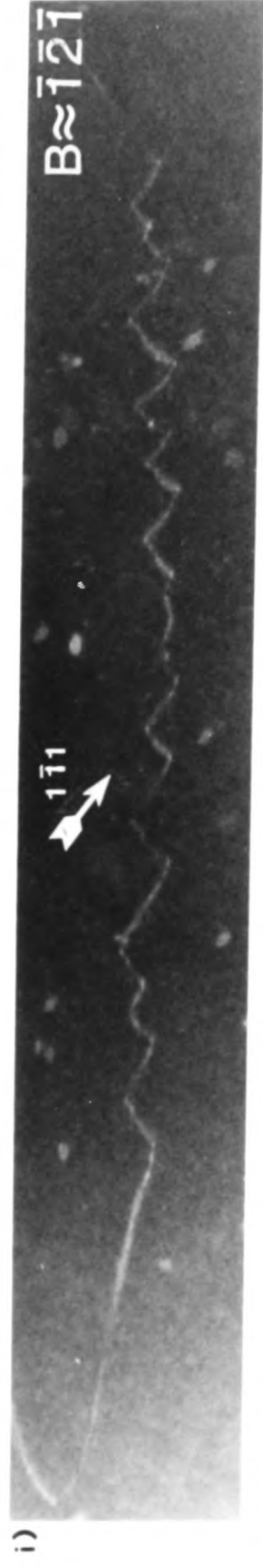
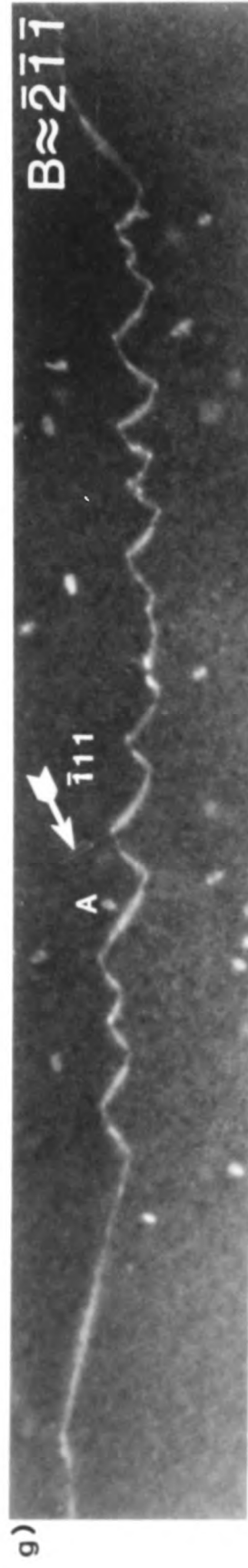
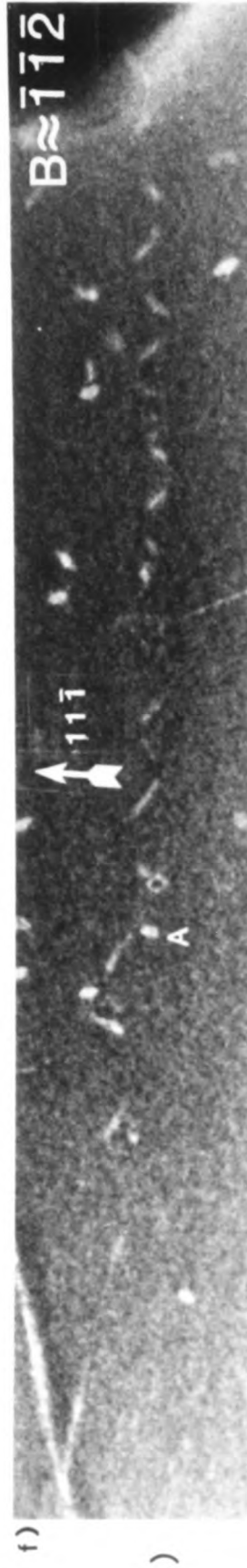
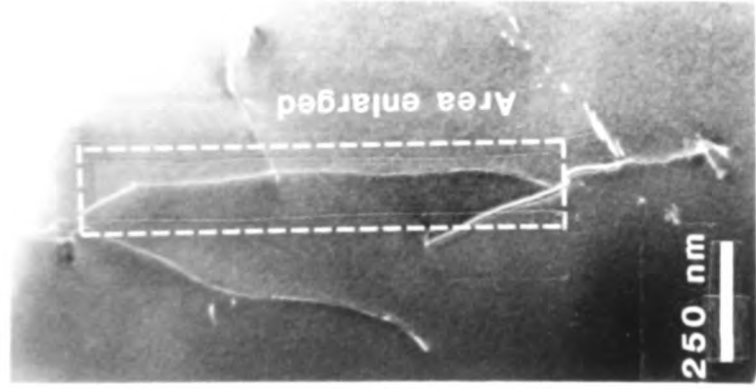
e)

Screw Dislocation in
Fe-15%Ni-17%Cr (at.%)
0.05 dpa at ~400°C



 Positive line direction

Screw Dislocation in
Fe-15%Ni-17%Cr (at.%)
0.05 dpa at ~400°C



0.05 dpa at $\sim 400^{\circ}\text{C}$

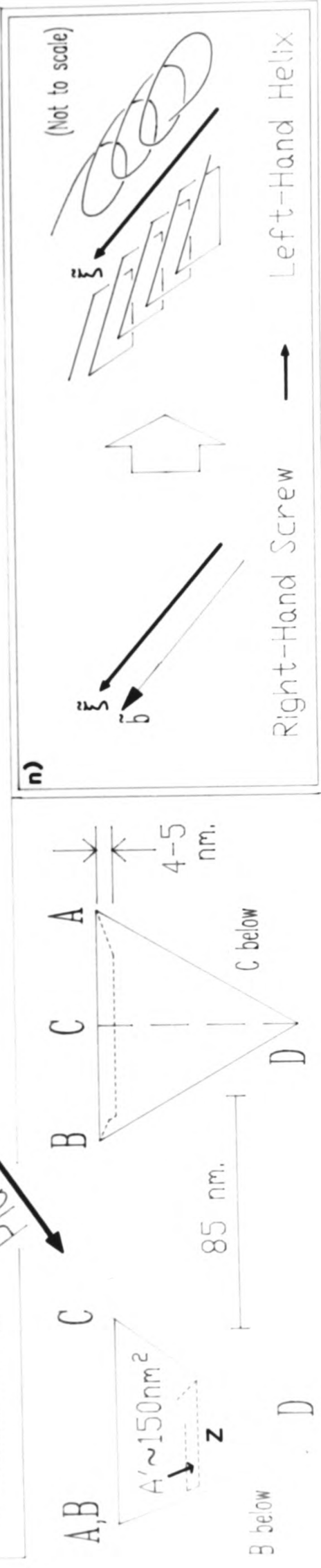
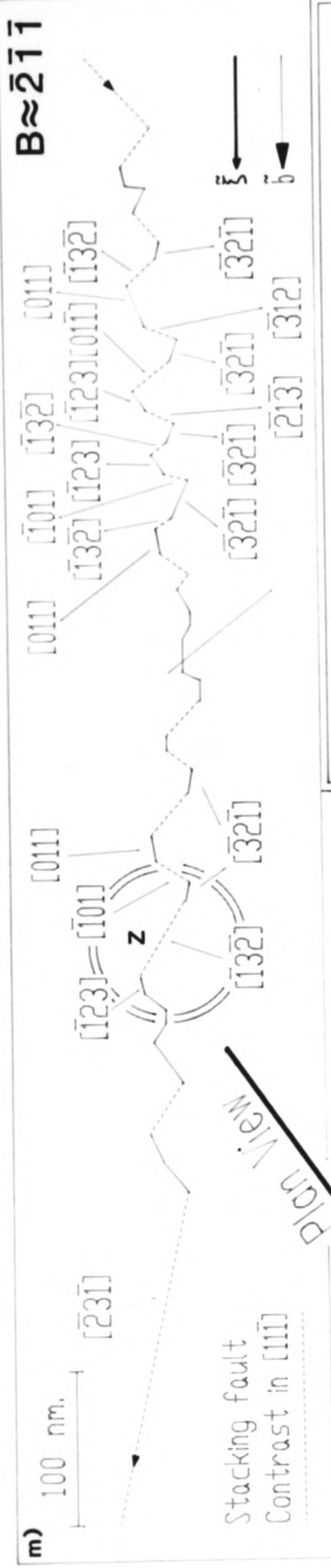


figure. Almost no new background loops were seen to nucleate during the irradiations of figures 3.2 and 3.3 ($\sim 420^\circ\text{C}$).

Finally it should be noted that formation of the zig-zag structure is suppressed in the region where a straight glissile dislocation with $\tilde{\mathbf{b}} = \overrightarrow{\text{AC}}(\beta)$ has intersected, but not apparently reacted, with screw dislocation $\overrightarrow{\text{AB}}$ towards the centre of each figure.

Interpretation:

The shape of this dislocation after irradiation, consisting of sharp points and straight line segments, does not appear to correspond to a ‘classical’ circular equilibrium helix, examples of which were first seen many years ago (e.g., Bontinck & Amelinckx, 1957; Thomas & Whelan, 1959). It is thought instead that this configuration is *principally* two dimensional for the following reasons;

- For an equilibrium circular helix, both its pitch and radius are likely to be roughly similar (Hirth & Lothe, p.634; Grilhé, 1966; Hellström & Vingsbo, 1975; Thomas & Whelan, 1959 (and references therein)). The pitch of the configuration observed here is up to 85 nm., little less than the thickness of the foil (~ 100 nm.). Depth oscillations, which would be separated by $\sim 50\text{\AA}$ under the conditions operating, are not evident in the weak-beam images, however.
- Regions of the dislocation are still dissociated. Whilst the mechanism of Cherns et al (1980; their figure 15) results in a helical form consisting of alternate dissociated and constricted dislocation sections, the nucleation of a high density of interstitial loops directly onto the line, and of a different Burgers vector to the line, must be invoked. There is, as yet, no evidence to support the occurrence of any such nucleation.

Figure 3.4 (*m*) is a schematic diagram of the projected shape of the post-irradiation dislocation configuration for $\tilde{\mathbf{B}} \approx [\bar{2}\bar{1}\bar{1}]$ (micrograph (*g*)). Dotted sections are those sections of the line which exhibit SFC in $\tilde{\mathbf{g}} = \pm [11\bar{1}]$. Solid sections appear constricted. A directional analysis was performed, using projections onto (111), (121), (211), (112) and (110) planes, on all line segments greater than about 10 nm. in length. It was found that the misalignment of “parallel” segments could be explained by assuming that many lie along $\langle 123 \rangle$ directions, whilst others lie along $\langle 110 \rangle$. Six $\langle 123 \rangle$ directions are contained in each $\{111\}$ plane. They lie approximately 11° either side of every $\langle 112 \rangle$. In projection $\langle 123 \rangle$ directions appear misaligned by only a few degrees with $\langle 110 \rangle$ or other $\langle 123 \rangle$ directions.

If the error in plotting the diffraction vector, $\tilde{\mathbf{g}}$, onto a micrograph is $\sim 5^\circ$ (although a more realistic estimate is probably $\sim 3^\circ$), a slim possibility is allowed that a particular segment, apparently

lying along a line direction $[\bar{3}2\bar{1}]$ for example, may actually lie along $[\bar{3}1\bar{2}]$ when seen in (111) or (121), but not in (110) projections. Its sense of inclination in the foil is the same for both, however. Fore-shortening or lengthening exhibited by segments on tilting provides their senses of inclination in the foil. Further information on diffraction vector direction may be obtained by noting that on tilting the foil to bring planes α , β or γ vertical, all $\langle 123 \rangle$ directions in those planes are projected onto $\langle 110 \rangle$. Also dislocation segments with Burgers vector $\tilde{b}_T = \vec{AB}$ and lying along $\langle 123 \rangle$ directions in α or β may not be dissociated. Segments which exhibit SFC must therefore lie on planes γ or δ . Interestingly SFC is only seen from segments in δ . Directions shown in (m) were therefore deduced in this lengthy cyclic manner. Small errors in these directions are feasible but their senses of inclination in the foil are accurate.

The longest period of the dislocation, extending ~ 85 nm. between points 'Z' (see (l)) is sketched in horizontal and vertical plan views at lower left. Although these drawings are nominally to scale they are based on measurements of sections only a few nanometres in length and errors may be up to 50%. However, it can be seen that the dislocation has assumed a left-handed 'flat-helical' configuration. A more tightly wound left-handed helix composed of straight segments is sketched schematically in (n), adjacent to a circular helix for comparison. Helices composed of straight sections along $\langle 123 \rangle$ and $\langle 110 \rangle$ directions have previously been seen after quenching in low SFE Cu-4 at.%Si, Cu-6 at.%Si and Cu-13.43 at.%Al alloys (Décamps & Condat, 1980, 1985;). The formation of similar segmented helices in aluminium has also been reported by Segall & Shoaib (1970) and referred to by Décamps et al (1983). Décamps & Condat (1980) reference other incidences when dislocations lying along $\langle 123 \rangle$ directions have been observed (see also, Wilson, 1978), whilst Segall & Shoaib (1970) list a number of papers, produced between 1959 and 1961, in which segmented helices may have been seen. Finally it should be noted that the area (A') perpendicular to $\tilde{b}$ of turn 'Z' is somewhere in the region of 100-300 nm² (150 nm²). This corresponds to only a few thousand (~ 4000) atoms along this 85 nm. stretch of dislocation⁵. It is also interesting to note that the vertical radius of this coil is ~ 5 nm.; the ratio $85/5 \left(= \frac{\lambda}{r} = \frac{\text{slope}}{2} \right)$ happens to correspond very closely to the limit of 6π proposed on p.635 of Hirth & Lothe for a loosely wound helix constrained by straight dislocation segments.

The earlier characterization of clusters formed in lines close to dislocations of appreciable edge character as predominantly vacancy in nature (figures 3.1, 3.2 and 3.3) is reinforced by their absence

⁵Unfortunately, the quantitative approach of Thomas & Whelan (1959) and Hellström & Vingsbo (1975), employed to estimate the local defect super-saturation, cannot be employed here since the helix is neither defined through the foil, nor is it possible to estimate the local density of helices.

close to the screw dislocation of figure 3.4. This is consistent with the small hydrostatic strain field of screw and near screw dislocations. If vacancies have not migrated in any great number to the screw dislocation then interstitials are likely to be responsible for the formation of the zig-zag configuration above (see ‘*Points of Discussion*’ towards the end of this chapter).

Axiom 17.1 on page 624 of Hirth & Lothe states: “*The reaction ... of a right-handed screw into a left-handed helix involves vacancy emission*” (or interstitial absorption). The former case, in which the dislocation is climbing via vacancy emission, refers to the climb of dislocations to form helices at high temperatures (see, e.g., Mizuno & Kino, 1987). In this case dislocations will act as vacancy sources (as do surfaces and other ‘sinks’) to establish an equilibrium concentration of vacancies (see, for example, Manning, 1968, p.195). Such a mechanism is unlikely under conditions of high supersaturations of interstitials, as is expected to be the case at dislocations under HVEM irradiation (Heald, 1975; Brailsford & Bullough, 1972).

The primary origin of this ‘flat-helix’, and of zig-zag structures exhibited by screw dislocations throughout this volume, is considered within the ‘*Points of Discussion*’ section towards the end of this chapter, which concerns itself solely with the latter explanation; that this ‘flat-helical’ configuration is formed by the absorption of a net excess of interstitials.

Figure 3.5; 90°-0° Dislocation in Fe-15%Ni-17%Cr (at.%); Irradiated at ~400°C.

Observations:

In figure 3.5 we see the onset, and evolution of a zig-zag configuration as the dislocation tends from edge to screw character. Micrograph (a) shows dislocation $\overline{AB}$ prior to irradiation with $|\tilde{g} \cdot \tilde{b}_T| = 2$. This dislocation tends from pure edge, on the left hand side, to pure screw on the right hand side of the figure. Black numerals along the line refer to the nearby total dislocation character (ϕ). The many constrictions point to the existence of a number of pre-existing jogs along the line, otherwise the dislocation is widely dissociated. Background clusters evident in this micrograph are probably vacancy in nature and result from sub-threshold 300kV irradiation (see, e.g., Sigle et al, 1988; Shepherd, 1987), at which voltage this pre-irradiation characterization was conducted⁶. Many of these clusters are recognisable as SFT in $\tilde{g} = \langle \bar{1}11 \rangle$ type reflections taken before irradiation (not shown) and were most common in thin regions near the hole. Their depths within the foil are not known but the majority are probably near the lower foil surface (Cherns et al, 1975, 1977).

During irradiation in the Argonne HVEM, the voltage was reduced to 200kV after a dose of

⁶Later pre- and post-irradiation characterizations were conducted at 200kV to reduce this vacancy damage.

approximately 0.03 dpa and micrograph (b) was taken at temperature *in-situ*. The deviation parameter, s , for this micrograph is $\simeq 1.85 \cdot 10^{-2} \text{ \AA}^{-1}$. The accelerating voltage was then re-increased to 1 MeV for continuation of the irradiation. Whilst the resolution is too poor for detailed analysis this micrograph admirably shows the rationale behind conducting experiments in the Argonne HVEM.

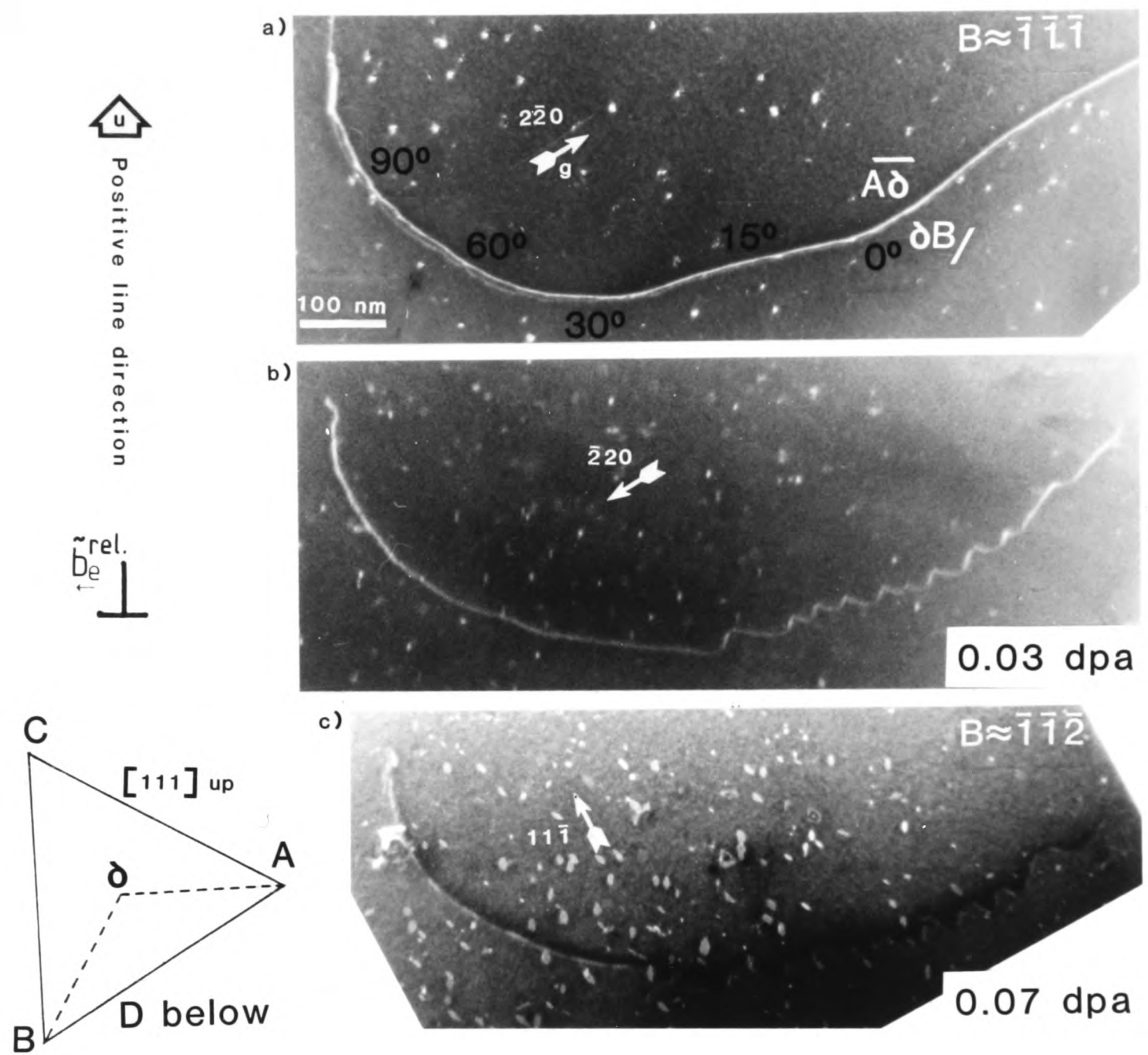
In this micrograph ((b)) we see the onset of zig-zagging of the line as the dislocation character passes from edge through 30° . The period of the zig-zag is approximately 25–40 nm. This zig-zag configuration is roughly symmetric, and most developed, about $\phi = 0^\circ$. As the dislocation approaches the 30° orientation either side of this point the period of the zig-zag lengthens and it becomes a succession of long and short segments; the longer segment being those which lie more nearly parallel to the initial dislocation position. Many of these segments appear to lie close to 20° or 40° orientations, consistent with $\langle \bar{1}\bar{2}3 \rangle$ directions on δ . Notice particularly that only a few of the zig-zags show evidence for the existence of inclined connecting segments; most zig-zags appear to meet at a point. Small loops have nucleated in the background whilst vacancy clusters present before irradiation have disappeared. There is no correlation between the positions of new loops and pre-existing vacancy clusters.

Micrograph (c) was taken at 200 kV in a Philips CM30 EM after completion of the irradiation. The dislocation had now received a total dose of approximately 0.07 dpa. An area of this micrograph is shown at higher magnification in (d).

In micrograph (c) (and (d)) it is immediately evident that the zig-zag configuration is now much more developed than at the intermediate dose point. Many ‘parallel’ dislocation segments, which appeared slightly misaligned after 0.03 dpa, now actually appear to *be* parallel. It is best seen in (d) that ‘zigs’ and ‘zags’ lie accurately along $[10\bar{1}]$ and $[01\bar{1}]$. (This result is confirmed by comparing these directions with those of stacking fault fringes exhibited within background loops). Almost all ‘zigs’ and ‘zags’ are visible in this reflection, for which $\tilde{g} \cdot \tilde{b}_T = 0$, suggesting that they are dissociated. Many regions between ‘zigs’ and ‘zags’ appear entirely constricted, however. The full contrast series suggests that these connecting segments lie on a plane γ and are shallowly inclined in the foil.

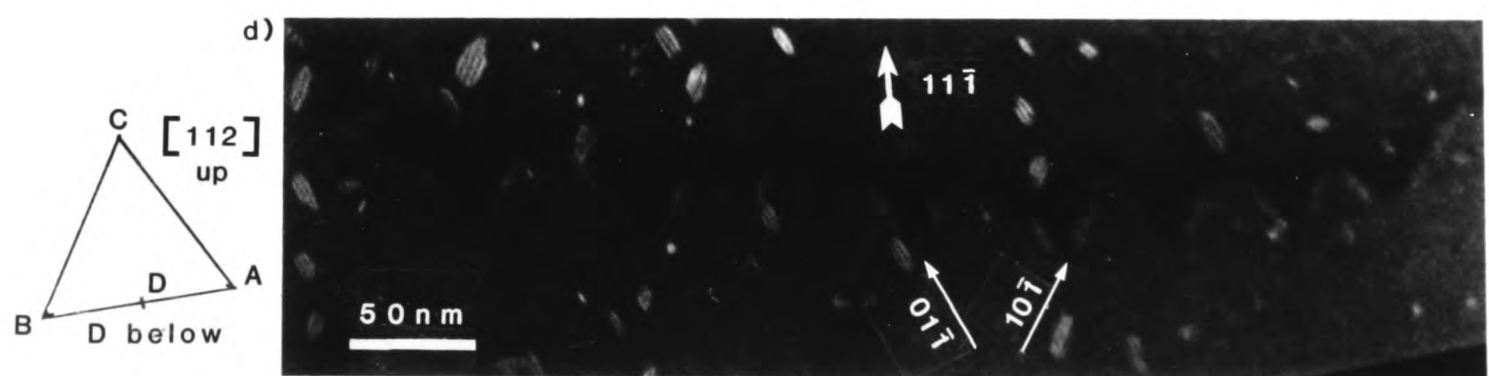
Reflections of the full contrast series, including $\tilde{g} = [11\bar{1}]$ shown here, display very many background loops. There are fewer loops in the vicinity of the 60° to 90° section of this dislocation than elsewhere. Many of these background loops can be correlated with small features visible at the intermediate dose point. All were found to be interstitial in nature and almost all are still faulted. Micrograph (c) shows loops with $\tilde{b} = \alpha\bar{A}$ and $\beta\bar{B}$ in strong contrast.

Figure 3.5



90°–0° Dislocation in Fe–15%Ni–17%Cr (at.%)

Irradiated at ~400°C



Interpretation:

This specimen was maintained at elevated temperature in the HVEM for a considerable time (a few hours) whilst voltages were changed and micrographs taken at the intermediate dose point. During this time a thick contaminant layer formed on the surfaces which was found, on inserting the specimen into a lower kV instrument, to have degraded weak-beam images beyond acceptable limits⁷. In consequence a full characterization, leading to a determination of line directions of short segments, or even of the number of segments, did not prove possible. Much must therefore be inferred from other irradiations, specifically that of figure 3.4.

It would seem likely that the configuration of 'flat-topped' zig-zags at the intermediate dose point is very similar to that of figure 3.4, with most 'zigs' and 'zags' lying along $\langle \bar{1}\bar{2}3 \rangle$ directions in δ separated by short inclined segments. It is believed that those zig-zags which appear to meet at a point after 0.03 dpa do not have inclined connecting segments between 'zig' and 'zag'. As such their likely configuration is close to that of figure 3.7(c). At the higher dose point, most 'zigs' and 'zags' show SFC, which, from the evidence of figure 3.4, is only shown by segments lying on δ . These zig-zags are therefore in 60° orientation, at which one partial has $\phi = 90^\circ$, suggesting that the effect of individual partials, rather than the total dislocation, is important. The alignment of zig-zags along $\langle 123 \rangle$ directions in δ may thus represent only an intermediate stage in the formation of the higher dose (0.07 dpa) configuration of figure 3.5, in which zig-zags lie along $\langle 110 \rangle$. Theory suggests (Bullough et al, 1964) that $\langle 123 \rangle$ directions may correspond to shallow energy minima for glissile, rhombus-shaped $\tilde{b} = \langle 110 \rangle$ loops in Al-1%Mg. These loops would then lie on (012) habit planes as occasionally observed (Makin & Hudson, 1963). It must be said, however, that a more rigorous treatment, incorporating anisotropy (Bacon et al, 1970) does not produce such good agreement with experiment, although conditions are found whereby perfect loops in Cu and Al, of particular sizes, may still lower their energies by glide away from edge orientation.

Finally, there appears to be an interstitial loop depleted zone (LDZ) near to the 60° - 90° section of this dislocation. The background interstitial loop density is unaffected near the lower hydrostatic strain field of the screw, and near-screw section of the dislocation.

⁷As alluded to above, this was an early experiment. The study of damage evolution by analysing the same area by weak-beam imaging at successive dose points was later abandoned. Contamination was independent of irradiated area, but was proportional to specimen time in the HVEM.

Figure 3.6; Glissile and Sessile Dislocations in Fe-15%Ni-17%Cr; 0.07 dpa at $\sim 420^\circ\text{C}$.

Observations:

Micrographs 3.6 (a) and (b) show a complex dislocation prior to irradiation. The dislocation to the left hand side has Burgers vector $\vec{AB}$ and is glissile on δ . It is in 60° orientation and, as depicted at centre left of the figure, its extra half-plane is below. In $\tilde{g} = [2\bar{2}0]$ (micrograph (a)), $|\tilde{g} \cdot \vec{b}_T| = 2$ for the total dislocation and both the lower (90°) partial, $\vec{A}\delta$, and upper (30°) partial, $\vec{\delta}B$, are visible. Dislocation 'L' to the right hand side of 'X' is visible in $\tilde{g} = \pm [02\bar{2}]$ (not shown) and $[2\bar{2}0]$ but is out-of-contrast in $\tilde{g} = \pm [20\bar{2}]$ (see (b)). This is consistent with Burgers vector $\vec{AD}$. This dislocation has probably been formed via the reaction of a section of the inclined dislocation $\vec{BD}$ with $\vec{AB}$ during slight deformation of the foil prior to thinning⁸. Dislocation $\vec{AD}$ lies along the intersection of planes α and δ and is sessile in both. Micrograph (a) shows this dislocation not to be completely straight, however, and may, in fact, be imaging partial $\vec{A}\delta$ of a Lomer-Cottrell lock $\vec{A}\delta + \vec{\delta}\alpha + \vec{\alpha}D$ (see Hirth & Lothe, p.799). At 'C', dislocation $\vec{AB}$ appears to have cross-slipped onto γ . This section is close to pure screw in character. In micrograph (b) the stacking fault is imaged as a bright straight line. Dislocations $\vec{AB}$ and $\vec{BD}$ have not reacted along this length⁹.

The low magnification micrograph (c) highlights the area shown at high magnification in (d) and (e). These latter two micrographs are taken after irradiation to approximately 0.07 dpa. The glissile dislocation $\vec{AB}$ at left is shown after irradiation in figure 3.2.

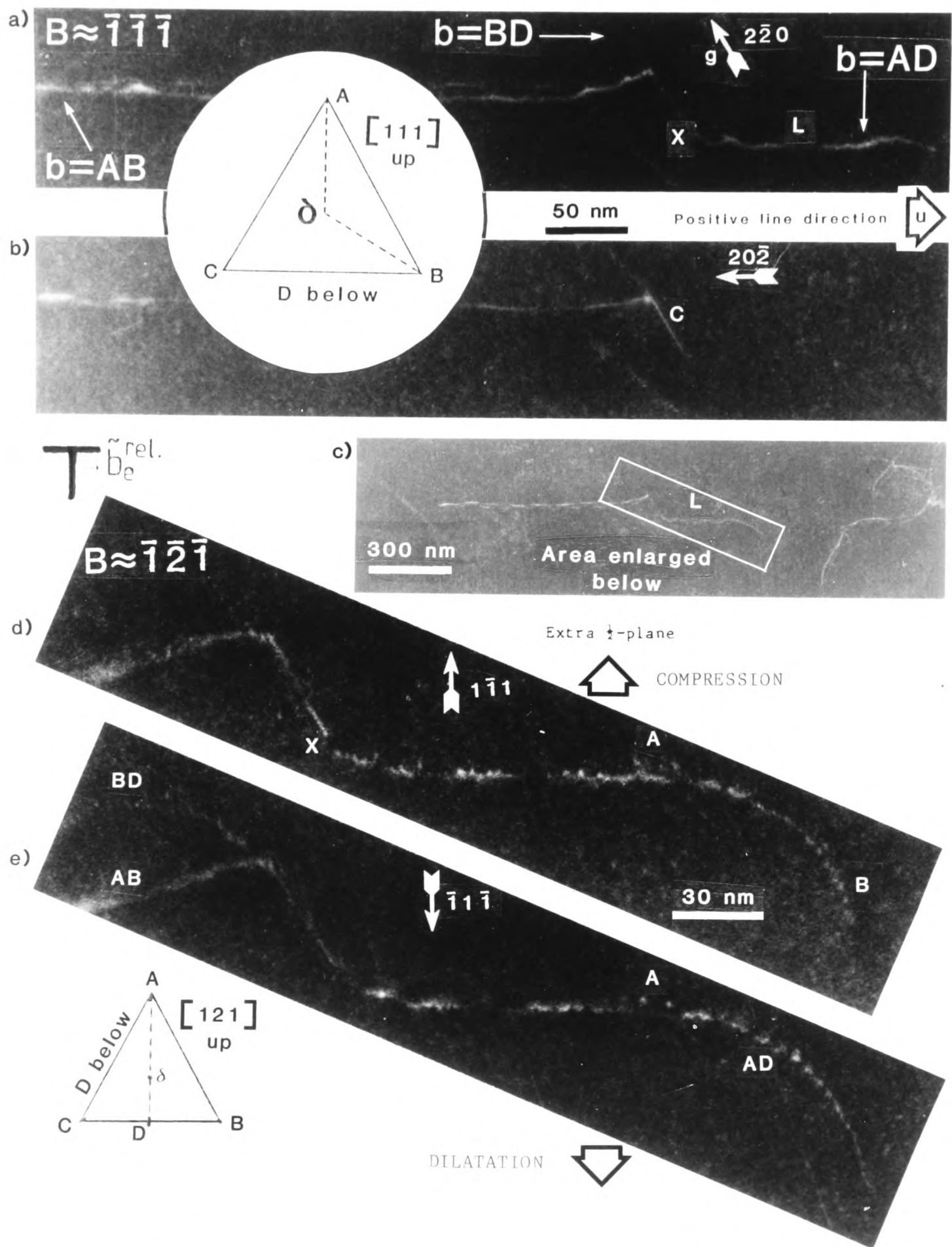
It can immediately be seen from these micrographs that dislocation $\vec{AD}$ now contains a great many tiny cusps. These cusps are $\sim 3-5$ nm. across; approximately an order of magnitude smaller than the zig-zags of the screw dislocation of figure 3.4. Close inspection suggests that the sharp points of the cusps all extend in the same direction away from the line. In the reflections $\tilde{g} = \pm [1\bar{1}1]$ shown here, both dislocations $\vec{A}\delta$ and $\vec{\delta}\alpha$ would be visible. The Lomer-Cottrell barrier therefore appears to have constricted to form a perfect prismatic dislocation. This result is confirmed by other reflections in the series. The straight line dislocation at 'C' appears unaffected by the irradiation.

Some small clusters have formed at 'A'. These clusters could not be characterized using standard techniques. It is possible that the two clusters more brightly visible in (e) possess arrow-head contrast. At 'B' the dislocation tends towards screw orientation and becomes glissile on γ ; this section

⁸ Initially dislocation $\vec{BD}$ was probably in pure screw orientation, gliding on γ . Intersection with $\vec{AB}$ then prompted easy cross-slip onto α . Reaction then occurs of $\vec{AB}(\delta) + \vec{BD}(\alpha) \rightarrow \vec{AD}(\delta/\alpha)$. Diagram A.10 (Appendix) depicts a similar mechanism.

⁹ Reference to figure 22-11 of Hirth & Lothe, and/or consideration of half-plane symmetry show the formation of an extended-dislocation barrier to be unlikely here.

Figure 3.6



Glissile and Sessile Dislocations in Fe-15%Ni-17%Cr (at.%)

0.07 dpa at $\sim 420^\circ\text{C}$

is inclined in the foil and exhibits thickness oscillations. Note also that as soon as the dislocation becomes glissile on γ it ceases to exhibit the fine scale cusping.

Interpretation:

Whilst cusps and constrictions on glissile dislocations disappear under irradiation, this sessile dislocation exhibits opposite behaviour. It is believed that this is attributable to the particular core structure of Lomer dislocations resulting from dislocation intersection (see below).

The extra half-plane of this dislocation extends both downwards, and to one side as indicated by the large arrows. It can easily be seen, therefore, that the clusters formed at 'A' lie to its compressional strain field side and are hence likely to be vacancy in nature. The observation that they do not exhibit contrast consistent with any perfect or Frank loop variety, coupled with the suggestion of 'arrow-head' shapes, indicates that these clusters may be extended in three dimensions.

At foil orientations employed here, the fine scale cusping may result from climb or $\{100\}/\langle 110 \rangle$ glide (e.g., Seeger et al, 1957). When considering the latter possibility it is worth noting, however, that dissociated glissile dislocations, for which the process is easier (Hirth & Lothe, p.273), show no evidence of glide here. It is also evident that the vacancy clusters at 'A' are removed substantially further from the line, in a sense consistent with interstitial climb, than those in preceding figures (see figures 3.1 and 3.2).

Finally, the author is rather surprised by the assertion on page 801 of Hirth & Lothe that there have been "*numerous observations of barrier (1)*" (a type '1' Lomer-Cottrell lock) as drawn in their diagram 22-12, which shows the Lomer dislocation, itself, to be dissociated. If dislocation $\overline{AD}$ is actually dissociated before irradiation, as suggested in micrograph (a), this would appear to constitute only the *third* such *direct* observation of a dissociated Lomer dislocation. As far as the author is aware, the *only*¹⁰ previous such observations are in Ag (Korner & Karnthaler, 1981) and in Cu-10%Al (Karnthaler & Wintner, 1975, their figure 2). Notice that in the low SFE alloy (SFE $\simeq 11.5 \text{ mJ.m}^{-2}$, Ray, 1971) the Lomer dislocation is dissociated by very much *less* than the reactant glissile

¹⁰Holmes et al (1979), employing the weak-beam technique, ascribe small fluctuations seen along a 'type 1' Lomer-Cottrell dislocation in Cu to a possible narrow dissociation of very short segments. Extensive calculations led them to predict that all three partials of 90° dissociated Lomer dislocations may have a negative line tension and that the dissociated form is only $5.0 \pm 1.3\%$ (see Korner & Karnthaler, 1981) lower in energy than the undissociated form. Bonneville & Douin (1990), however, employing the same technique, find that Lomer dislocations in Cu are constricted, although it is arguable whether weak-beam images provide sufficient resolution for a positive determination. Lomer dislocations in Ni are not expected to dissociate.

dislocations, contrary to the predictions of Jøssang et al (1965). Neither of the other two references given by Hirth & Lothe (Strunk, 1970; Lindroos & Mikk-oja, 1967), nor any of the references given therein, provides evidence for the *dissociation* of Lomer dislocations, although the formation of Lomer-Cottrell locks is often seen. There have, however, been numerous HREM observations of constricted Lomer dislocations (Bourret & Colliex, 1982 (Si and Ge); Mills & Stadelmann, 1989 (Al); Korner et al, 1987 (Si); Elkajbaji et al, 1988 (Si)). Thibault-Desseaux et al (1989) postulate that the apparently energetically favourable dissociation of Lomer dislocations does not occur because the $\frac{1}{6}\langle 110 \rangle$ stair-rod has a compound core structure, leading to a very high core energy. This is a prime demonstration of the importance of core structure determinations *prior* to widespread acceptance of results computed from elasticity theory. Finally, figures 3.6 and 4.7 of this thesis provide indirect evidence that the dissociation of *one of the reactant* dislocations at the lock itself may be greater than that of dissociated glissile dislocations in the plane of the foil.

Points of Discussion

- *Jog Climb.*
- *Dislocations and Interstitials: Pipe Diffusion?*
- *Origin of the Configuration of Figure 3.6.*
- *Background Loops.*

Jog Climb

The correlation of pre-existing jogs and the final zig-zag structure of figure 3.4 is important in considering the mechanisms whereby interstitials may be interacting with the line, as is the observation that, for this 'flat-helix' structure, dissociated dislocation segments on δ have not apparently climbed very far. Were climb to be proceeding via the nucleation of copious numbers of jogs under irradiation, say via the nucleation of small loops, the final zig-zag structure is likely to bear little relation to the pre-existing jog positions. Pre-existing jogs therefore seem important as the sites at which this process is initiated, and at which most climb has occurred. For these narrowly dissociated screw dislocations pre-existing jogs may be constricted, in which case they are substantially more favourable as sites for interstitial precipitation, leading to climb of the jog, than are dissociated straight line segments. Jog climb in a sense consistent with interstitial absorption was first directly observed, employing the weak-beam technique, by Carter (1980b).

Consideration of the directions in which jogs climb leads to an explanation for the

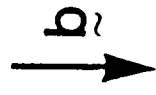
Figure 3.7

**Schematic Representation of Climb Forces acting
on Pre-existing Jogs**

JOG CLIMB

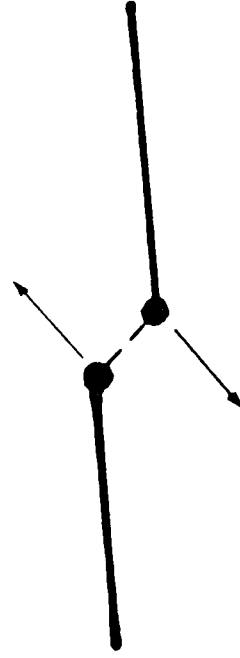
a)

90°



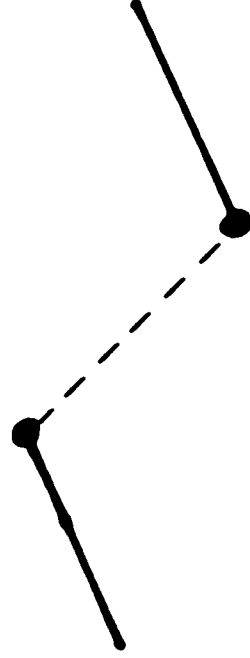
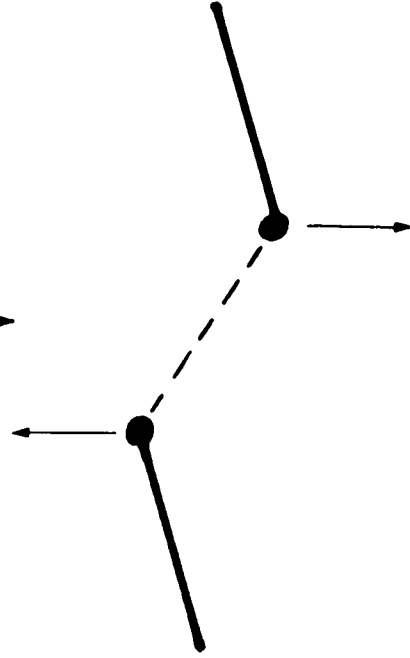
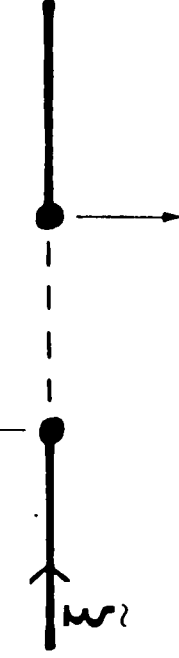
b)

Mixed



c)

0°



disappearance of constrictions after irradiation along mixed dislocations (e.g. see figure 3.1; region (2)) and possibly for the primary origin of the screw dislocation zig-zag structure. As is indicated in figures 3.7 (a) and (b), climb forces on a pair of equal, and opposite, jogs (labelled “Up” and “Down”) in a mixed or edge dislocation will be such as to cause the jogs to climb together and annihilate under, say, a net interstitial super-saturation for the sense of the jogs as shown. Essentially this is similar to the argument put forward by **Carter & Ray** in 1974. For the mixed dislocations of figures 3.1(2) and 3.2 above, where there are a great many jogs, the direction of motion of an individual jog is unimportant.

As dislocations approach screw orientation the climb forces on the jogs will tend away from the line direction and, presumably, a point is reached beyond which these forces will overcome the line tension forces which tend to keep the dislocation straight. Jogs will now not climb together and annihilate but will instead give rise to a *principally* two dimensional zig-zag configuration as shown in figure 3.7 (c). For the screw dislocation of figure 3.4 climb forces act perpendicular to the line. The numbers 1, 2 and 3 in figure 3.7 refer to initial, intermediate and final configurations respectively. Small arrows represent the directions of climb forces on the jogs whilst $\vec{b}$ is the Burgers vector and ξ the positive line direction.

Finally, it is thought that pre-existing jogs are also important in the final generation of ‘flat-helical’ configurations on screw dislocations. This is discussed within chapter 8.

Dislocations and Interstitials: Pipe Diffusion ?

Given the observations of vacancy clusters forming close to mixed and edge dislocations (e.g. figure 3.1(1)) the fate of the more mobile interstitials generated during irradiation must be considered. A greater number of interstitials than vacancies are expected to migrate to dislocations (see chapter 1). The growth, by climb, of interstitial Frank loops during the irradiation of figure 3.5, coupled with evidence for a LDZ and for the climb, in an interstitial sense, of pre-existing jogs on screw dislocations, suggests that significant numbers of interstitials arrive at glissile mixed and edge dislocations. However, the above figures provide no evidence for climb of the line itself and, although this possibility cannot be excluded in the (111) foils used, it would seem unlikely that dislocations would climb so uniformly for no sign to be visible at any specimen tilt. Furthermore there is no evidence for the nucleation of interstitial loops along glissile dislocations. A full explanation of these results may involve the core diffusion of irradiation generated interstitials: In figure 3.1(1) the dislocation is initially jog-free, possibly leading to easy core diffusion to the surface. Pre-existing jogs in screw dislocations act as favourable sites for interstitial absorption and hence it is supposed that the sense of jog-climb in figures

3.1(2) and 3.2 is interstitial. These pre-existing jogs complicate the extra half-plane of a dislocation and lead to a discontinuity in the stacking fault ribbon. As such they will impede core diffusion. Interstitials migrating to the dislocation after annihilation of these jogs do not lead to the formation of new jogs and may now core diffuse to other sinks unimpeded. Core diffusion also provides a ready explanation for the observed suppression of the zig-zag configuration towards the centre of figure 3.4; interstitials migrating to this region may diffuse along the intersecting inclined glissile dislocation, which isunjogged, rather than attach to the screw dislocation.

For the Lomer dislocation of figure 3.6, pipe (a.k.a. core) diffusion may be significantly impeded. The intersection of $\vec{AB}$ and $\vec{BD}$, via which mechanism this length of Lomer dislocation was likely created, produces jogs in both dislocations (see **Hirth & Lothe**, p.800). Perhaps intersection over a long length, and subsequent constriction either of an intermediate Lomer-Cottrell barrier, or other, configuration, may thus produce a copious number of pure screw *kinks* along the length of the newly formed dislocation $\vec{AD}$.

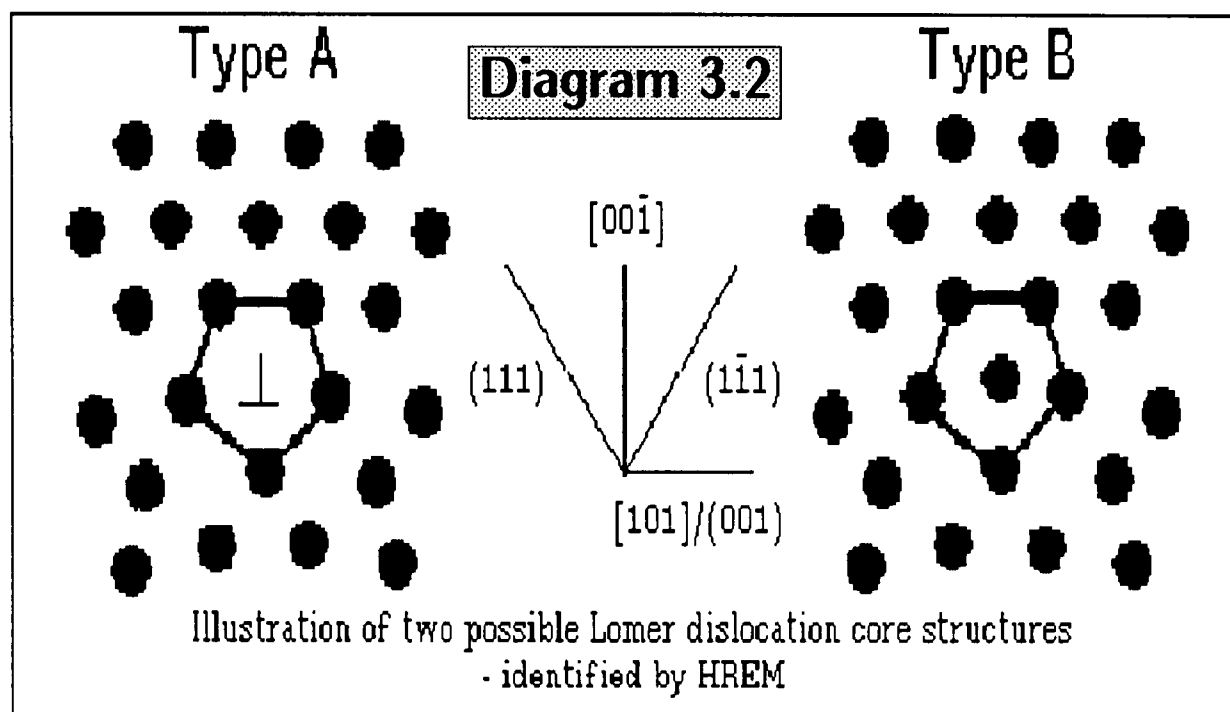
In common with the pre-existing jogs seen on glissile dislocations, such kinks may similarly act as barriers to pipe diffusion. Studies of the structure of Lomer dislocations by HREM (see above) indicate that they are constricted, with cores which are much more compact than those of glissile 60° dislocations. **Mills & Stadelmann** (1989) identified two core structures in Al: “Type ‘A’” showed a hollow pentagonal structure, of internal diameter $\sim 2\tilde{b}$, whilst “type ‘B’” placed an atom at the centre. “Type ‘A’” was believed to be the ‘true’ core structure whilst they attributed “type ‘B’” to the existence of an intersection kink towards the middle of their 6 nm. length of dislocation.

In high SFE materials it has been found experimentally (e.g. in Cu, Ni and Ag by **Karnthaler**, 1978; in Ni_3Fe by **Korner & Karnthaler**, 1980; and possibly in Si by **Korner et al**, 1987) that constricted dislocations, with $\tilde{b} = \frac{a}{2} \langle 110 \rangle$, may glide on $\{100\}$ planes under high stresses. The observations of **Carrard & Martin** (1988) of $\langle 110 \rangle / \{001\}$ glide in Al were of pure-screw $\tilde{b} = \frac{1}{2} \langle 110 \rangle$ dislocations, generated by dislocation multiplication on their $\{001\}$ glide planes (see **Carrard & Martin**, 1987 for details), rather than a **Lomer** (1951) intersection. Dislocations with $\tilde{b} = \frac{1}{2} \langle 110 \rangle$ in the Lomer orientation, which were observed by **Mills & Stadelmann** (1989) to undergo $\{100\}$ glide in Al and all of which had “type ‘A’” structures (free of kinks), had also not been formed by a ‘Lomer’ intersection. It may be significant that the “type ‘B’” Lomer dislocation, which *had* been formed by a conventional Lomer reaction was not observed to glide. If the individual cusps (seen in figure 3.6 to be about 3-5 nm. long) represent distances between kinks (and if this value is roughly typical of intersection produced Lomer dislocations) then their observation that the only Lomer dislocation produced by dis-

location intersection contained an “intersection kink” along its 6 nm. length is hardly surprising¹¹.

It therefore seems likely that the Lomer dislocation of figure 3.6, which has probably been formed via dislocation intersection, has a “type ‘B’” core structure. The ramifications for pipe diffusion are obvious from the diagram below (Diagram 3.2):

A second factor, aside from the core configuration, which may further affect pipe diffusion is the observation that cores of Lomer dislocations are very compact; if the Smoluchowski (1952) model of pipe diffusion¹² may be employed then a reduction in the size of this ‘hollow’ core may also inhibit core diffusion.



essentially from Mills and Stadelmann, 1989.

Origin of the Configuration of Figure 3.6

We have seen above that dislocations are attracting interstitials; for glissile dislocations this probably leads to the climb of pre-existing jogs, followed by core diffusion along mixed and edge dislocations once these jogs have been annihilated. This intersection produced Lomer dislocation is pure edge in character and will have a large hydrostatic strain field. It is thus also likely to attract point-defects, the majority of which are expected to be interstitial in nature. Core diffusion seems extremely

¹¹The detailed mechanism by which such intersection kinks may form is uncertain. It may be related to the continuous nature of the Burgers vector density as a function of distance from the dislocation core (e.g., *Huang et al*, 1989) and will probably require detailed atomistic calculations to be performed, employing complex many-bodied potentials, before the probabilities either of the existence of these kinks, or possible intermediate configurations, can be isolated. The state of research in this field has apparently altered little since 1982, at which time it was graphically expressed by *Hirth & Lothe* (their figure 22-1, p.793).

¹²Point-defects migrating down a hollow core.

unlikely and therefore interstitials must be accommodated at the line. They are not seen to precipitate out as clusters. Whilst $\{100\}$ glide may yet explain the presence of the fine scale cusps, it is a conservative process. As such it does not accommodate point-defects. It is therefore supposed that the cusping evidences interstitial climb of the dislocation, which appears constricted after irradiation. It may therefore climb via the direct absorption of individual defects. Loops of the same Burgers vector as the dislocation (perhaps formed from interstitial Frank loops via the reaction $\vec{\alpha}\vec{B} + \vec{\alpha}\vec{C} + \vec{\alpha}\vec{A} \longrightarrow \vec{D}\vec{A}$) may also nucleate and react. The sharp points of the cusps, which hence lie nearer to the initial dislocation position, may correspond to the intersection produced pure screw kinks. Nucleation on these sections would not seem to be likely.

Background Loops

It is apparent that different irradiations have led to very different levels of interstitial background loop formation. For example; after irradiation to 0.07 dpa at $\sim 420^\circ\text{C}$ (e.g. figure 3.3) very few background loops are evident, whilst, after nominally the same dose, in figure 3.5 a very high density of background loops is seen. This irradiation was performed at $\sim 400^\circ\text{C}$. The irradiation to 0.05 dpa shown in figure 3.4 was also nominally at 400°C ; the density of background loops here is similar, or possibly slightly lower than at the 0.03 dpa dose point in figure 3.5, however.

With a possible error of $\pm 50^\circ\text{C}$ on temperature measurement it is extremely difficult to ascribe differences in total background loop area to any temperature variation. Correlation is, however, much improved when comparing background loop density with the time for which each specimen was in the HVEM: c.f.; the irradiated specimen shown in figures 3.3, 3.2 and 3.6 was at temperature in the HVEM for only about 3 minutes whilst the earlier irradiations of figures 3.4 and 3.5 were at temperature for ~ 2 and 6 hours respectively. These lengths of time led to correspondingly heavy contamination layers, and it seems likely that injection of impurities (probably C, N and O) from this layer by 1 MeV electrons has led to the observed higher background loop densities. A 20°C (or possibly greater) temperature difference may also lead to lower loop densities since interstitials may be more likely to diffuse to the surfaces of the foil. It would seem surprising, however, that this effect should be quite so marked.

Finally, evidence of climb at pre-existing interstitial Frank loops, such as 'D' of figure 3.3, suggests that the observed differences in background loop density are more likely to be attributable to nucleation than climb rate. Faster diffusion of interstitials to the surfaces of the foil will obviously decrease climb rates.

Summary

Observations:

Irradiated dislocations in this material exhibit the following phenomena:

- Narrowing of partial separations;
 - Disappearance of constrictions;
 - Zig-zag configurations;
 - Vacancy clusters very near line;
 - Large cluster at 60° orientation;
 - Interstitial Frank loop depleted zone (LDZ);
 - Straightening of dislocations; some glide (7a);
 - Fine scale cusping;

... 1 ...

... 2 ...

... 3 ...

... 4 ...

... 5 ...

... 6 ...

... 7 ...

... 8 ...

These are shown in the following figures: (Table 3.1)

Figure	Character	Dose (dpa)	Temperature (°C ± 50°)	Observations (see above)
3.1 (1)	~ 75°	0.05	400	1, 4, 5, 7;
(2)	~ 60°	0.05	400	1, 2, (4, 5), 7;
3.2	~ 60°	0.07	420	1, 2, 4, initially straight;
3.3	Mixed	0.07	420	(1), 2, 4, 7, 7a;
3.4	~ 0°	0.05	400	3, interstitial Frank loops formed;
3.5	30°- 90°	0.03 & 0.07	400	(1), 2, 6 (60°- 90°);
	0°- 30°	0.03 & 0.07	400	3
3.6	~ 0°	0.07	420	unaffected;
	90°(sessile)	0.07	420	1, 4, 8;

The narrowing of partial separations is most noticeable for edge and mixed dislocations for which the initial separation is wider. It appears to occur very soon after commencement of the irradiation. It is difficult to determine whether screw dislocations have similarly constricted.

The disappearance of constrictions along edge and mixed dislocations, and the initial zig-zagging of screw, and near-screw dislocations are both consistent with the climb of pre-existing jogs. In the case of screw dislocations the intermediate zig-zag configuration is particularly complex, forming a ‘flat-helical’ structure. Segments often lie along <123> directions. Very few interstitials are required for creation of the ‘flat-helical’ structure.

The zig-zagging is suppressed close to intersection with an inclined glissile dislocation (figure

3.4). An initiallyunjogged screw dislocation ('C' of figure 3.6) is unaffected by irradiation. Screw dislocations also do not show evidence for nearby vacancy cluster nucleation or for an interstitial LDZ.

Irradiations of edge and mixed dislocations do lead to vacancy cluster nucleation. These vacancy clusters, some of which are occasionally resolvable as SFT, appear to be three dimensional. Lines of these clusters lie *very* close to the dislocation core. When a high density of interstitial background loops is present (figure 3.5) there is evidence for a depleted zone. These background loops are almost all still faulted.

Large scale glide is only evident for the highly curved initial configuration of figure 3.3. Long dislocations are frequently observed to have straightened between two points but glide, in the absence of jogs, is uncommon.

Irradiation of a sessile Lomer dislocation leads to its constriction and the formation of a unique structure of fine-scale cusps. Cusping ceases to be evident as soon as the orientation of the dislocation alters such that it may be glissile on an inclined plane.

Mixed and edge dislocations show no evidence for the formation of new jogs. This is true for both initially jog-free segments and segments along which jogs have been annihilated. The situation is less certain for screw dislocations, however. At no time is there any evidence for interstitial loop nucleation directly onto perfect or partial dislocations; Burgers vectors of all dislocations are unchanged by irradiation.



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Chapter 4

Irradiation of Fe-25%Ni-17%Cr alloy

Figure 4.1; $0^\circ - 90^\circ$ Dislocation in Fe-25%Ni-17%Cr; Irradiated at $\sim 430^\circ\text{C}$.

Figure 4.1 shows a glissile dislocation which varies from screw to edge orientation from left to right of the figure. Large white numerals refer to the nearby total dislocation character and all micrographs shown are taken with the beam direction $\tilde{B} \approx [\bar{1}\bar{1}\bar{1}]$. Small arrows indicate projections of labelled directions. In $\tilde{g} = [2\bar{2}0]$ both partials are in contrast such that $|\tilde{g} \cdot \tilde{b}_T| = 2$ for the total dislocation of Burgers vector $\tilde{b}_T = \vec{AB}$. Upper and lower partials have Burgers vectors $\tilde{b}_p = \vec{\delta B}$ and $\tilde{b}_p = \vec{A\delta}$ respectively; screw sections of the dislocation are therefore left-handed. The lower partial is the brighter in micrograph (a) which is taken before irradiation.

This dislocation was then subjected to two separate, but closely spaced irradiations. The approximate extents of the HVEM beam for these irradiations are shown in the post-irradiation, bright-field micrograph (d). 'X' marks the HVEM beam centre.

Micrographs (c) and (d) are taken under semi-weak-beam dark-field conditions after irradiation, with deviation parameter $s \simeq 1.7 \cdot 10^{-2} \text{ \AA}^{-1}$. The foil thickness, which increases from $\sim 100 \text{ nm}$. to $\gtrsim 200 \text{ nm}$. from right to left, renders true weak-beam images too dim.

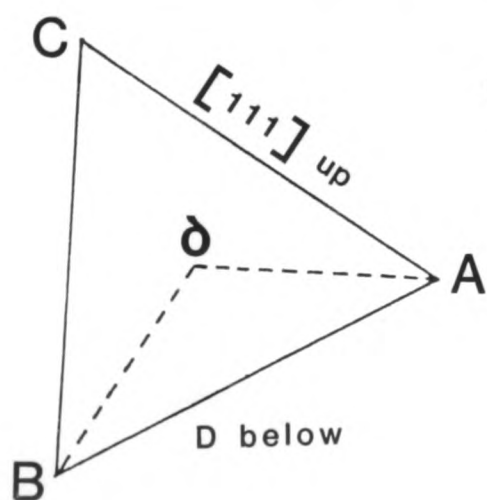
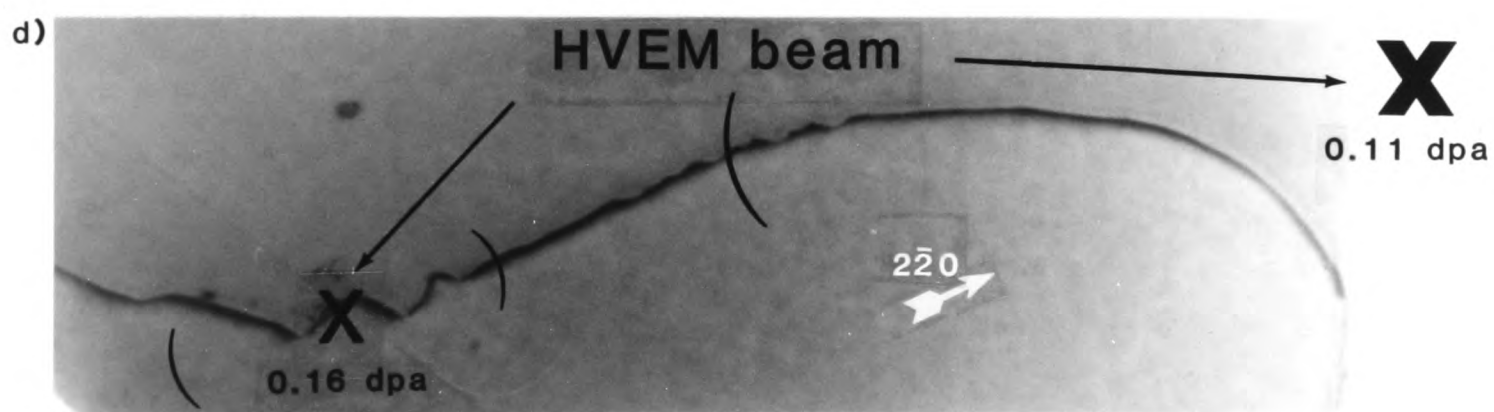
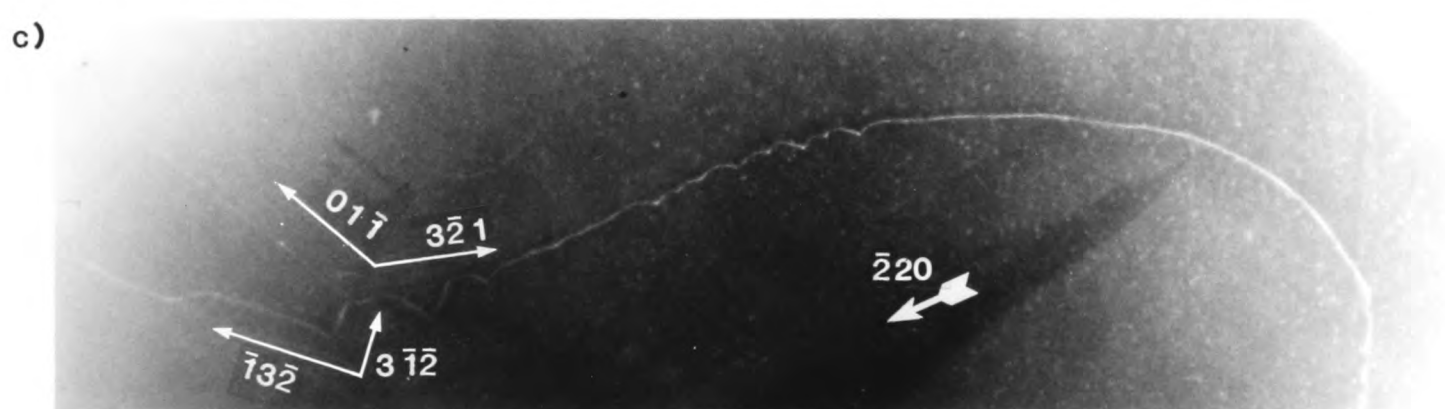
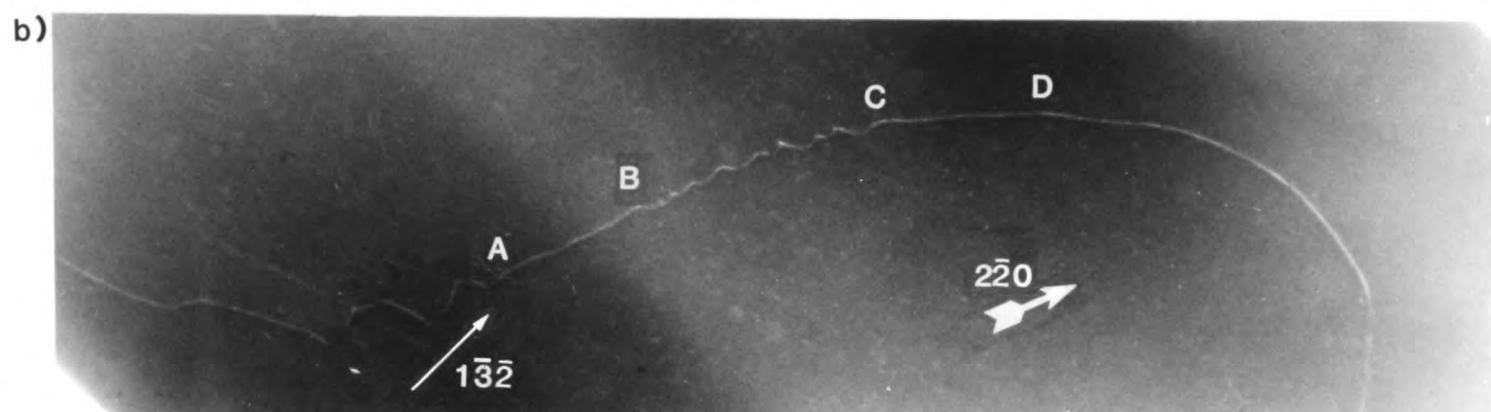
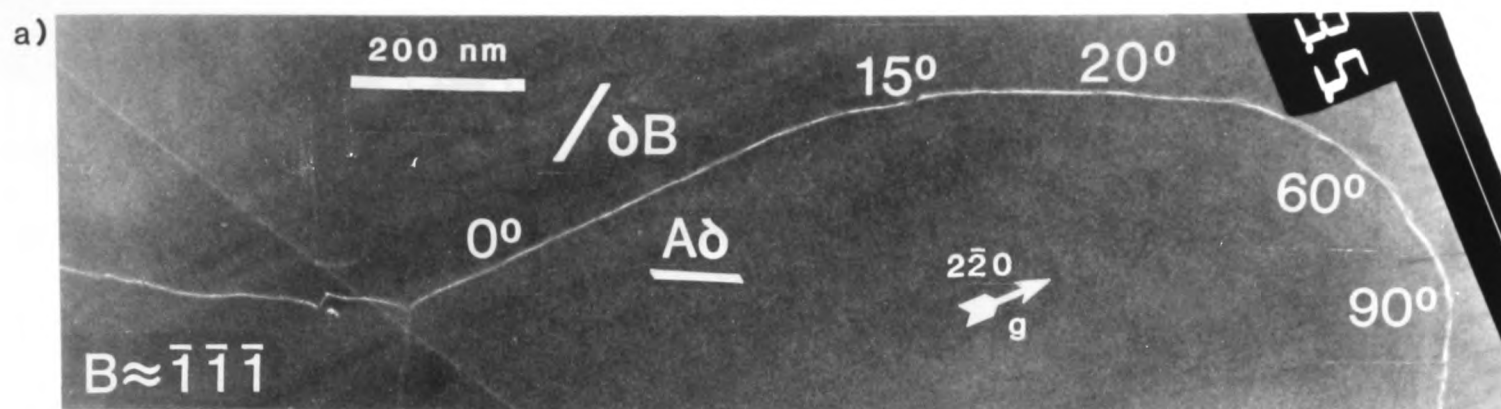
Observations:

The post-irradiation configuration of this dislocation shares many features with that of figure 3.5; constrictions have apparently been annihilated along mixed and edge dislocation sections¹³ and the partial separation may have decreased, although it is difficult to be certain, whilst screw sections have zig-zagged. In the higher dose irradiated area (0.16 dpa) the zig-zagging is most pronounced; some points of the zig-zag appear to correspond with pre-existing features, which may be jogs. Line directions of 'zigs' and 'zags' are often consistent with $[3\bar{1}\bar{2}]$ and $[\bar{1}3\bar{2}]$ directions in δ respectively. Line directions of connecting segments do not appear to lie along low-index, or $\langle \bar{1}\bar{2}3 \rangle$ directions in δ , nor along any $\langle 110 \rangle$ direction. They are consistent with the projections of $[1\bar{3}\bar{2}]$ and $[3\bar{2}1]$ directions in γ , however.

The lesser zig-zagging towards the centre of these micrographs lies at the periphery of the lower dose irradiation. It decays gradually as distance from the centre of this irradiation increases, eventually ceasing entirely near 'B'. Length 'AB' is not straight and lies very close to pure screw orientation. The zig-zagging also apparently ceases abruptly, near 'C', when the orientation of the total dislocation passes from 0° through approximately 20° . Length 'CD' may be associated with the zig-zag; it is

¹³ The right-hand side of this figure is contained within the area of figure 4.5.

Figure 4.1



$0^\circ-90^\circ$ Dislocation in Fe-25%Ni-17%Cr (at.%)

$T \rightarrow \tilde{b}_e^{rel.}$ Positive line direction $\rightarrow u$

Irradiated at $\sim 430^\circ\text{C}$

0.11 dpa: HVEM spotsize 10

0.16 dpa: HVEM spotsize 16

straight and lies accurately along $[2\bar{3}1]$ in δ . Careful study, in (111), (121), (211) and (112) poles, suggests that ‘zigs’ and ‘zags’ are contiguous near ‘B’, whilst towards the right-hand side (‘C’) connecting segments are only $\sim 5-10$ nm. in length and probably lie on γ . Individual segments were found to be too short for the accurate determination of line direction and hence this is not shown here, although longer ‘zags’, near ‘C’, apparently lie along $[1\bar{3}2]$. Notice that even towards the low dose end of this configuration, whilst the period of the zig-zag decreases, line directions of comparable segments (i.e. compare ‘zig’ with ‘zig’) appear to remain parallel. Finally, the dislocation configuration appears generally similar to that shown after the intermediate (0.03 dpa) dose of figure 3.5 (b).

Interpretation:

Unfortunately, the full contrast series, taken over a large angle of tilt, did not include the high dose irradiated area, which is in a very thick (~ 200 nm.) foil region. Hence, the line directions of particular, short segments cannot be determined with precision. However, it can still be seen that directions are generally consistent with those of figure 3.4, except that, in this case, a right-handed ‘flat-helical’ structure seems to have evolved. One of the ‘zags’ may lie along an inclined $\langle 123 \rangle$ direction (specifically $[1\bar{2}3]$ in β) as occasionally found in figure 3.4; this is still consistent with the helical structure being both right-handed and flattened, however.

As mentioned above, the area towards the centre of the micrographs received a graduated dose. The decay of the zig-zag configuration, along a dislocation section of almost constant (screw) character, is consistent with a gradual reduction in dose from right to left, as expected (see (d)). Where the zig-zag ceases, to the left hand side, may thus represent an effectively un-irradiated area. In consequence we see the evolution of the zig-zag structure, as a function of dose, from left to right.

Observations here seem to confirm that the initial stage in the formation of a ‘flat-helical’ configuration involves jog climb. The absence of connecting segments as the zig-zag fades towards ‘B’ suggests that it is purely two-dimensional in this region. It is therefore similar to the configuration of figure 3.7 (c). The fact that connecting segments only become resolvable towards the right hand extent of this zig-zag, which received a higher dose, suggests that jog climb must occur prior to their formation.

Since the zig-zag is only formed within irradiated areas, HVEM irradiation is responsible for its formation rather than any annealing effects. By virtue of the expected dislocation bias for interstitials, we would therefore expect a ‘flat-helical’ structure, formed from a left-handed screw dislocation, to be right-handed; evolving via the absorption of interstitials (see axiom 17.1 of Hirth & Lothe and the

‘*Interpretation*’ section of figure 3.4).

Figure 4.2; Dislocations in Fe-25%Ni-17%Cr; 0.1 dpa at $\sim 430^\circ\text{C}$.

In the low magnification, bright-field (BF) micrograph (c) almost the entire irradiated area is shown before irradiation. The two dislocations shown in this figure are sketched schematically in drawing (f). The shorter (left-hand) dislocation is shown before irradiation in (a) whilst the longer dislocation is shown before irradiation in (d). All micrographs on the first page are taken with beam direction $\tilde{\mathbf{B}} \approx [\bar{1}\bar{1}\bar{1}]$ and $\tilde{\mathbf{g}} = [2\bar{2}0]$ as indicated at centre right. Micrographs (a), (b), (d) and (e) are all at the same magnification. Both dislocations have Burgers vector $\tilde{\mathbf{b}}_{\text{T}} = \overline{\mathbf{A}\mathbf{B}}$ and vary in orientation from near-screw to $\sim 40^\circ$. The senses of individual partials, as well as areas shown in the second page of the figure, are indicated in (f).

Observations:

The shorter dislocation is shown before and after irradiation in (a) and (b) respectively. After irradiation, it is most evident along the near-screw section that many new cusps have formed, segmenting the line. This has also occurred in micrographs (d) and (e), which show most of the longer dislocation before and after irradiation respectively. Dipoles lying accurately along $[0\bar{1}1]$, seen towards the centre of (d), have disappeared after irradiation. The narrowing of the partial separations, which has occurred along the entire length of both dislocations, is best seen towards the top of (d) and (e). At the bottom of micrograph (e) zig-zagging typical of pure screw dislocations is visible.

A central region of the longer dislocation is shown at higher magnification in the second page of the figure. Micrograph (g) is taken before irradiation whilst (h) and (i) are after irradiation. All micrographs on this page are at the same magnification (scale bar at centre left) with beam directions as indicated. These micrographs are part of a full contrast series taken in all principal reflections of the (111), (121), (211) and (112) poles.

Points ‘A1’ and ‘E1’ in the pre-irradiation micrograph ((g)) correspond to constrictions. The right-hand partial ($\overrightarrow{\mathbf{A}\delta}$) is the brighter in this reflection. After irradiation two, straight, *discrete* lines of clusters can be seen; one towards the bottom of micrograph (h) and another towards the top of micrograph (i). These lines of clusters appear to lie very close to the initial dislocation position. Cusps ‘A’, ‘E’ and ‘F’ have apparently moved away, in directions indicated by small arrows, from these discrete lines of clusters. These clusters could not be characterized either by the $\tilde{\mathbf{g}} \cdot \tilde{\mathbf{b}}$ invisibility criterion or by looking for characteristic inside/outside contrast and are probably three dimensional. The direction of motion of these cusps is consistent with the rotation of the long intervening sections away

from screw orientation. Straight sections of the line between cusps exhibit SFC in $\tilde{g} = \pm [11\bar{1}]$ (not shown) and are found to lie accurately along $[3\bar{2}1]$ ('FE') and $[1\bar{2}1]$ ('BA').

Neither of points 'B' and 'D' seem much affected by the irradiation. The precise line directions of dislocation sections between them is uncertain, however. The full contrast series (not shown) suggests that some segments may lie on a plane γ although apparently not along $\langle 110 \rangle$, $\langle 112 \rangle$ or $\langle 123 \rangle$ directions. None of these segments is seen to exhibit SFC. Cusp 'C' is also slightly anomalous in that its motion appears to have twisted neighbouring line sections *towards* screw orientation. This region ('DB') is very complex before irradiation, there being a number of nearby dipoles and other, unidentified, features close to the line.

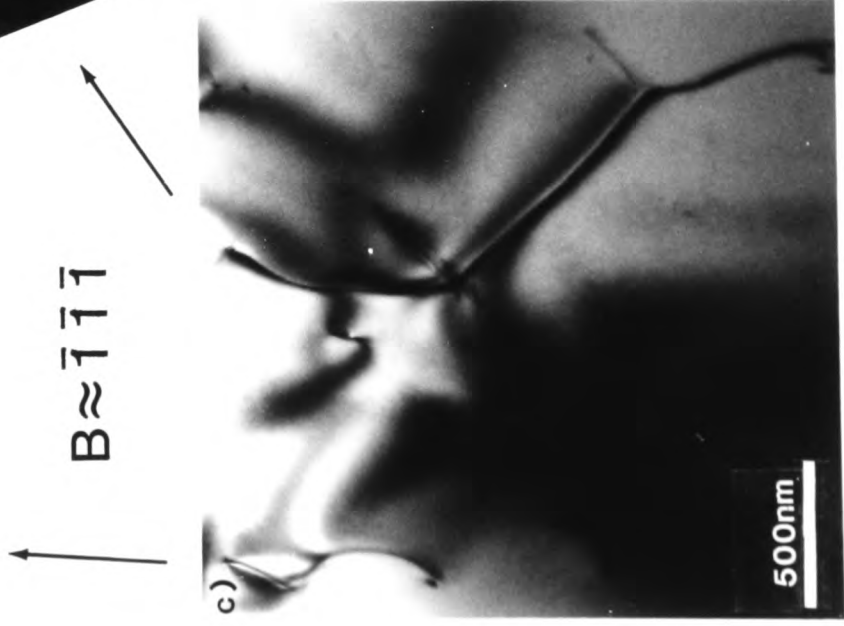
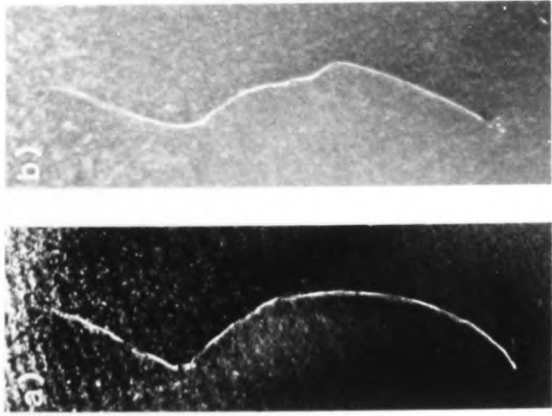
In micrographs (j), (k) and (l) the lower part of the shorter dislocation of micrograph (c) is shown. Micrograph (j) is taken before irradiation whilst (k) and (l) are after irradiation. The extra-half plane lies below this section of the dislocation. Cusp 'G' appears to have behaved similarly to 'A', 'E' and 'F' above, in that the neighbouring long dislocation section 'HG' has been twisted away from screw orientation to lie in 40° orientation along $[1\bar{3}2]$. This section ('HG') shows SFC in $\tilde{g} = [11\bar{1}]$ (not shown), which is also visible very faintly in $\tilde{g} = [\bar{1}\bar{1}1]$ ((l)). Both micrographs show a *discrete* line of clusters to have formed. This line of clusters is not straight and accurately follows the pre-irradiation dislocation position. Individual clusters could not be characterized. Micrograph (l) has been selected, rather than its opposite, because, by virtue of its very weak SFC, it enables the presence of a few clusters along the new dislocation position to be seen. No clusters are seen between the new and old dislocation positions.

On tilting from (211) to (112) poles the dislocation image is seen to move to the left with respect to the line of cluster image. With the tetrahedron orientated as shown it can be seen that this relative motion is consistent with the clusters lying below, and hence to the compressional strain field side of, the dislocation, suggesting that they are vacancy in nature. The amount of this motion is consistent with the clusters lying ~ 20 nm. below the dislocation, however, without calculating precise dislocation positions the potential error on this measurement is large.

Interpretation:

Constrictions 'A1' and 'E1' may represent the pre-irradiation positions of jogs 'A' and 'E'. As can be seen from figure 3.7 (b), climb forces on jogs on mixed dislocations do not act perpendicular to the line. It is possible therefore, particularly for a near-screw mixed dislocation, that a jog may climb both along and perpendicular to the line. This motion, and a possible mechanism for its occurrence, is

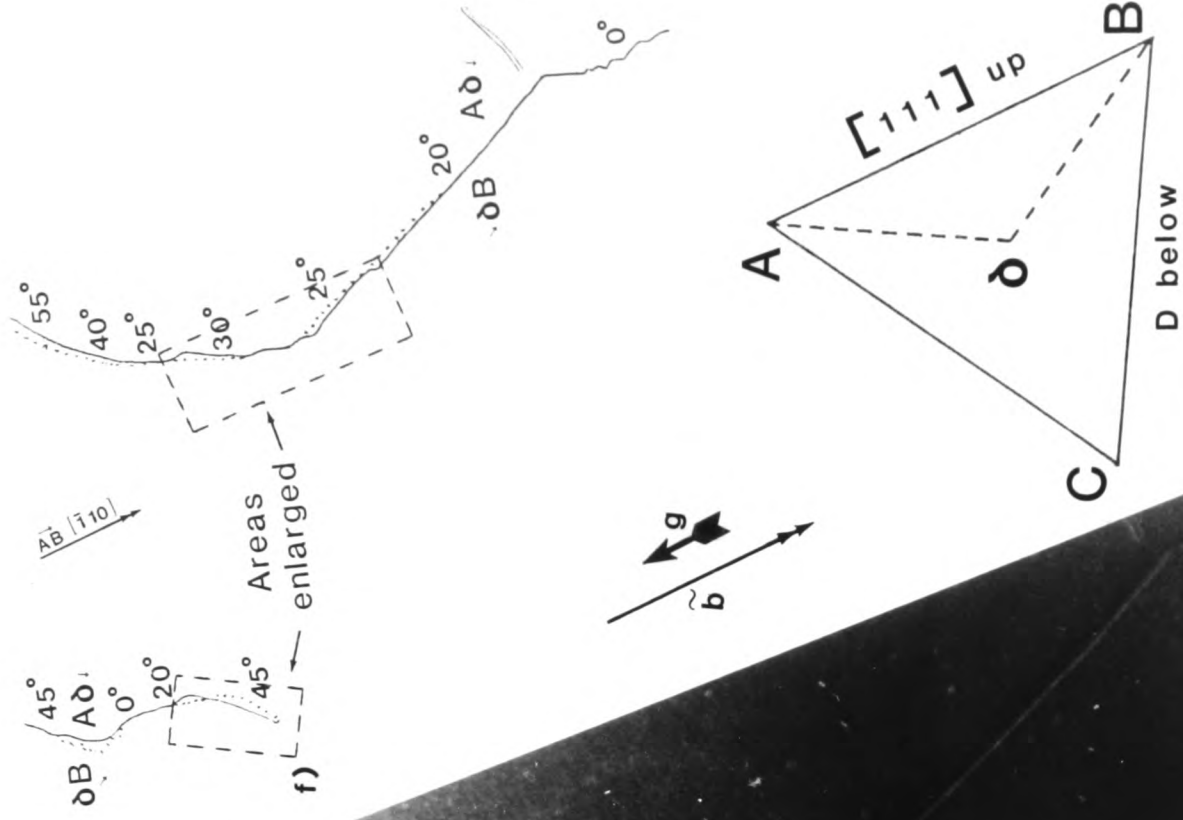
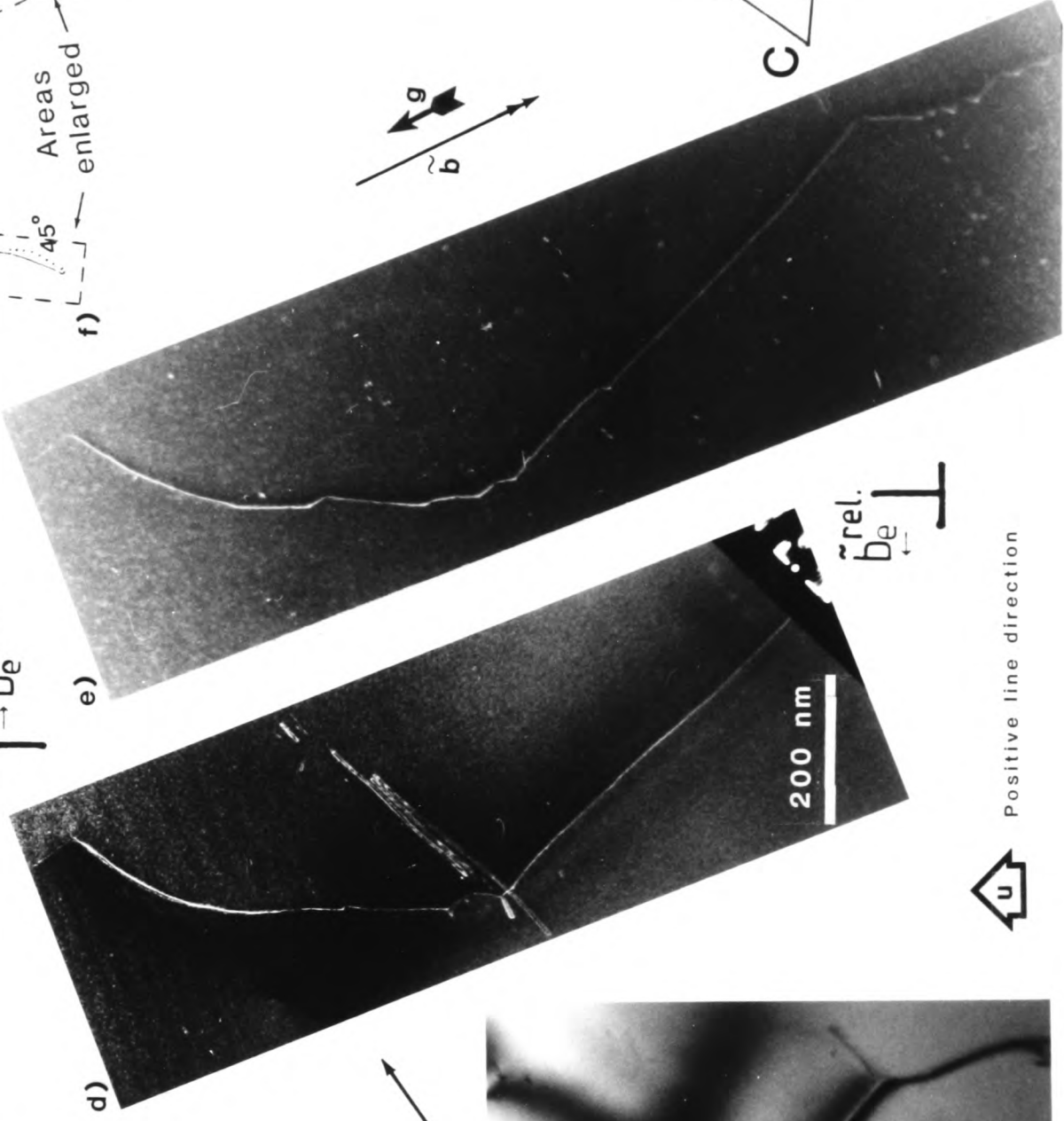
Figure 4.2
(2 pages)

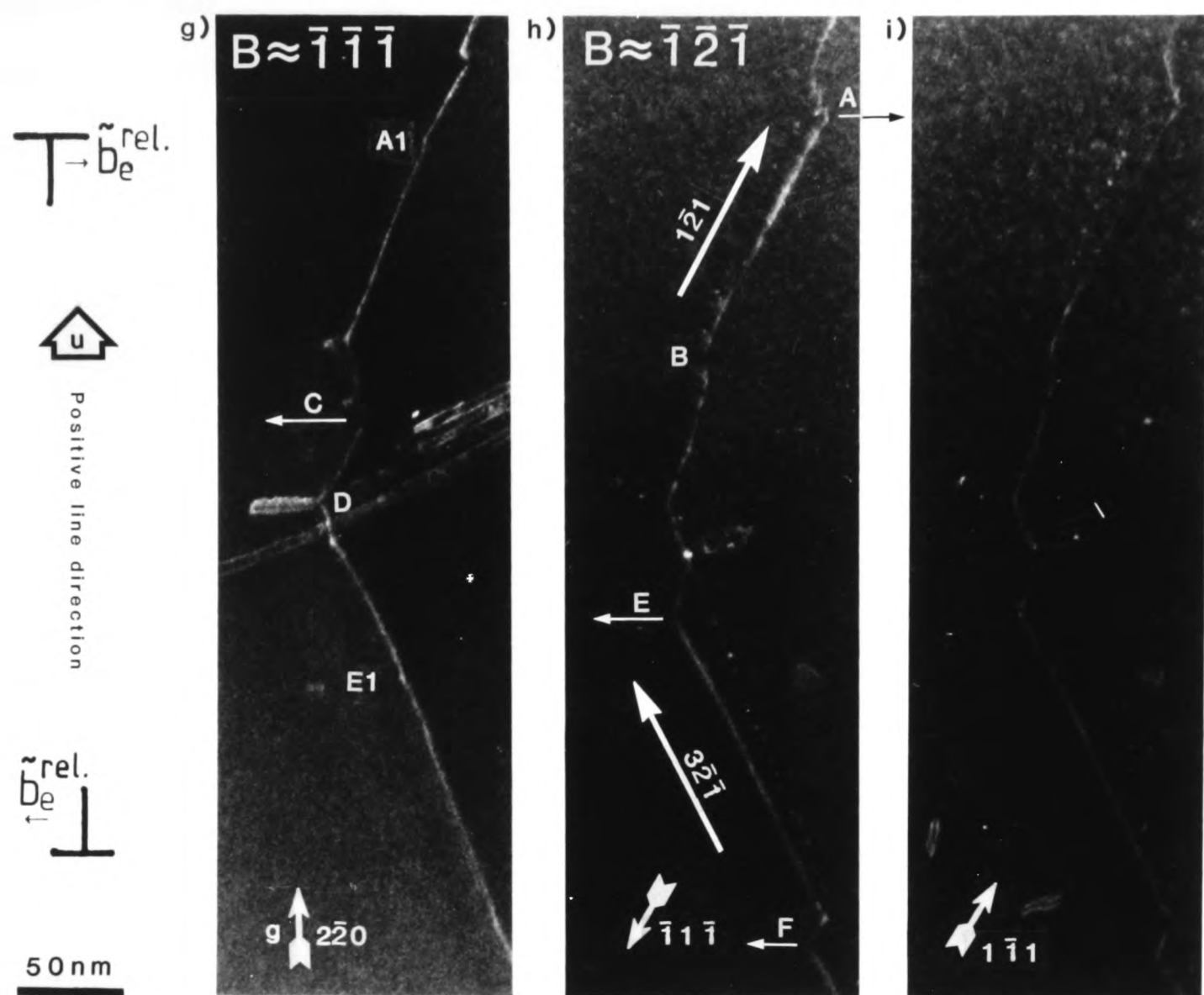


Dislocations in Fe-25%Ni-17%Cr (at.%)

0.1 dpa at $\sim 430^{\circ}\text{C}$

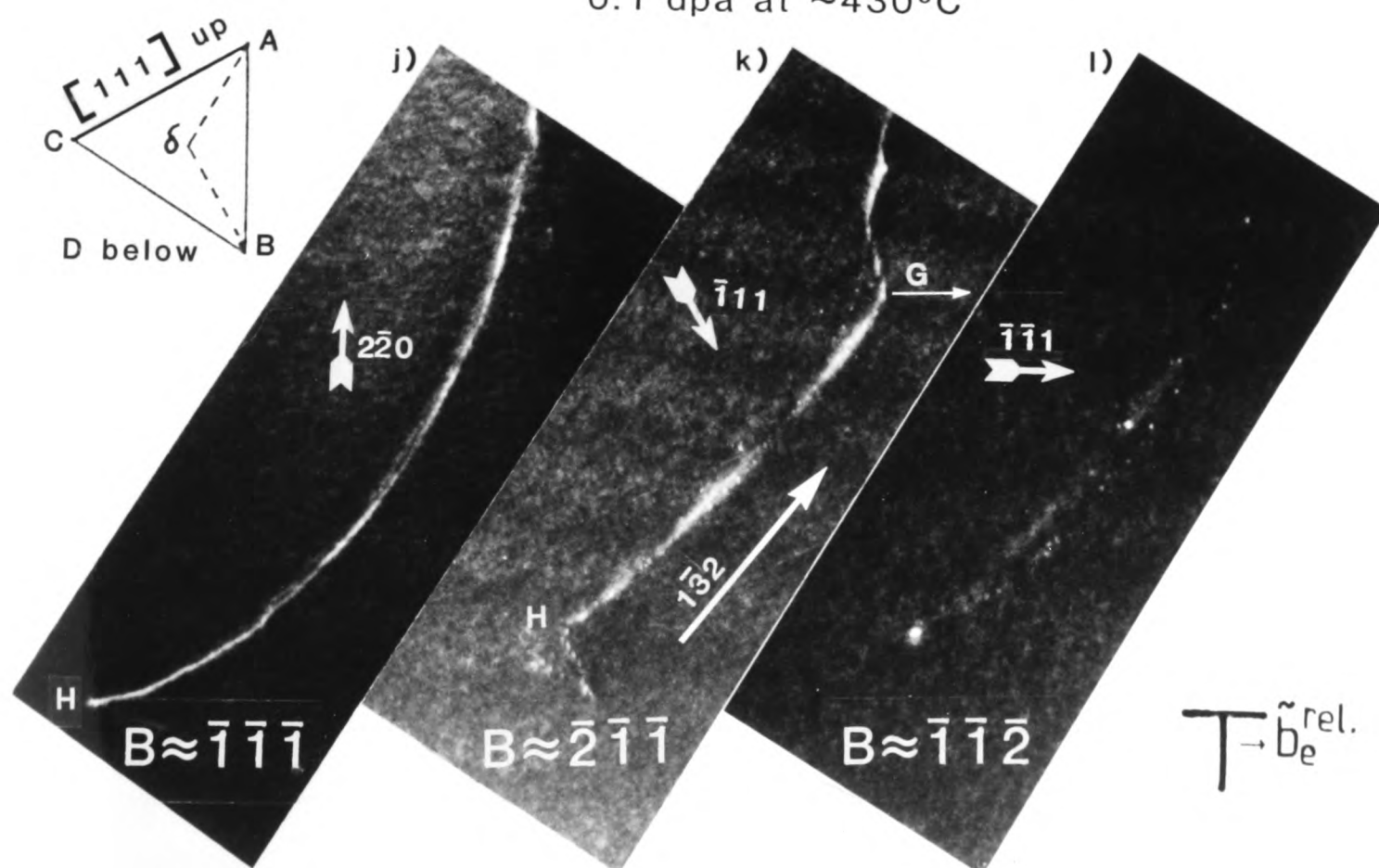
$\vec{T} \rightarrow \vec{b}_e^{\text{rel.}}$





Dislocations in Fe-25%Ni-17%Cr (at.%)

0.1 dpa at $\sim 430^\circ\text{C}$



discussed later. A mechanism for the formation of discrete lines, rather than a continuous cloud, of clusters is also advanced.

The disappearance of dipoles is consistent with observations of the climb of background loops (e.g. figure 3.5); if such climb occurs at these dipoles, probably by net interstitial absorption, it may easily result in their mutual annihilation (see, for example, **Carter**, 1980b). The near-screw central section of the longer dislocation, containing cusp 'C', lies very close to many of these dipoles; whilst the cusping at 'A', 'E' and 'F' seems likely to be due to interstitials, by reason of the dislocation (interacting with HVEM generated local super-saturations and its expected) bias for interstitials, the central region 'DB' of this dislocation may be within an area of very low super-saturations. No line of clusters has formed near this section of the dislocation. Motion of the line here may thus not be representative of the behaviour of isolated dislocations under irradiation; thermal effects, or possibly even the effect of an artificially high net vacancy super-saturation if the dipoles act as particularly efficient sinks for interstitials (although the absence of any vacancy clusters does not confirm this), may be dominant.

The detailed configuration of the shorter dislocation is more complex than it first appears, in that several short sections apparently lay on a plane γ prior to irradiation (e.g. 'below 'H'); climb may have occurred at the inter-connecting stair-rods, although certain conservative glide processes leading to bowing out and annihilation may result in an identical configuration.

Figure 4.3; 60° Dislocation in Fe-25%Ni-17%Cr; 0.11 dpa at $\sim 430^\circ\text{C}$.

Observations:

Micrographs (a) and (b) are taken before, and (c) to (g) after, irradiation to 0.11 dpa. This 60° dislocation contains a very large number of constrictions along its length before irradiation, as shown by the two $|\tilde{\mathbf{g}} \cdot \tilde{\mathbf{b}}_{\text{T}}| = 2$ images, (a) and (b). It is intersected by two other dislocations at a node, which is the subject of a later figure (figure 4.7).

After irradiation ((c)-(g)), all constrictions to the left of the node have disappeared (see (c) and (d)) leaving a now evenly dissociated line. The partial image separation at 'S'(60°) is $\sim (30 \pm 5) \text{Å}$. This does not appear to have altered appreciably from before irradiation, although the pre-irradiation separation is very variable and probably far from equilibrium.

Constrictions have also been annihilated to the right of the node, except along a section of the dislocation, 'K' (see (d)), which has bowed out on δ ; this section never appears straight on tilting to (112), (211) and (121) poles as shown on the second page of the figure (micrographs (e), (f) and (g)) and thus the bowing out is not a result of climb on α , β or γ . Surviving constrictions (jogs) along this

section may glide perpendicular to $\tilde{\mathbf{b}}$ on δ , as seen by **Carter & Hirsch (1977)** in low-SFE Cu alloys.

Micrograph (e) has $\tilde{\mathbf{g}} \cdot \tilde{\mathbf{b}}_T = 0$ for the dislocation. This image shows fairly even SFC along most of the dislocation's length, reinforcing the observation that the dislocation is still dissociated¹⁴. This image also shows the presence of several small clusters very close to the line, two of which are ringed.

On tilting to the (211) pole ((f)) these clusters are seen to be displaced downwards (as the figure is viewed) with respect to the dislocation image. This motion is in the same sense as point 'D' of the Thompson Tetrahedron shown at right. In micrograph (f), the *lower* partial $\overrightarrow{\delta\mathbf{B}}$ and stacking fault are imaged whilst in micrograph (e) only the stacking fault is imaged; this suggests that measurements of the magnitude of this motion by comparing relative cluster and line positions are likely to be underestimates for a dissociated dislocation. However, the plane bending criterion, which leads to an overestimate (probably $\sim 20\text{\AA}$) of the magnitude of this shift in micrograph (f), and possible random errors (a few $\text{\AA}$ - see **Cockayne, 1970**) prohibit the accurate use of these measurements, as guides to the actual vertical displacements of clusters from the dislocation, without detailed image simulations.

Nevertheless, with the partial image separation of $\sim 20\text{-}25\text{\AA}$ here, the magnitude of the observed shift ($\sim 40\text{\AA}$) is sufficiently large to conclude that these clusters lie up to 20 nm. beneath the dislocation. As indicated on the first page of the figure, the extra half-plane is below this dislocation and hence these clusters are probably vacancy in nature.

This result is seemingly confirmed by an opposite shift of the clusters with respect to the line image in micrograph (g). This shift is also consistent with the motion of 'D' of the tetrahedron. One of these clusters ('T' - arrowed) exhibits arrow-head contrast typical of vacancy SFT.

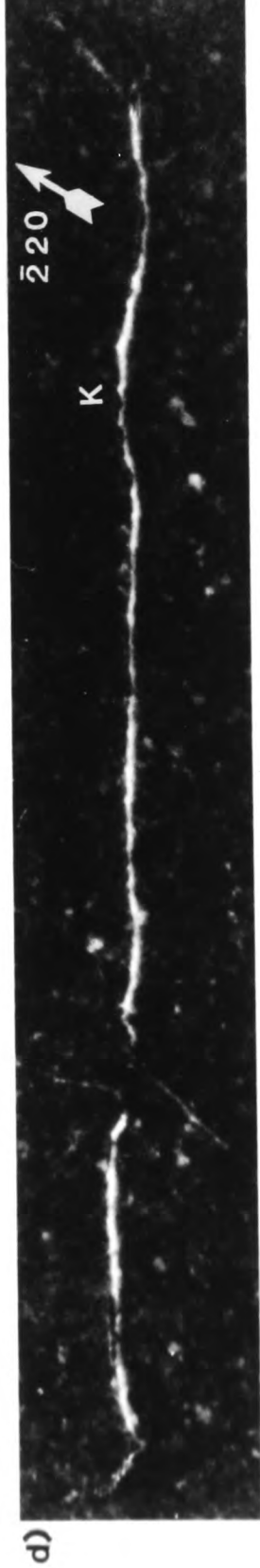
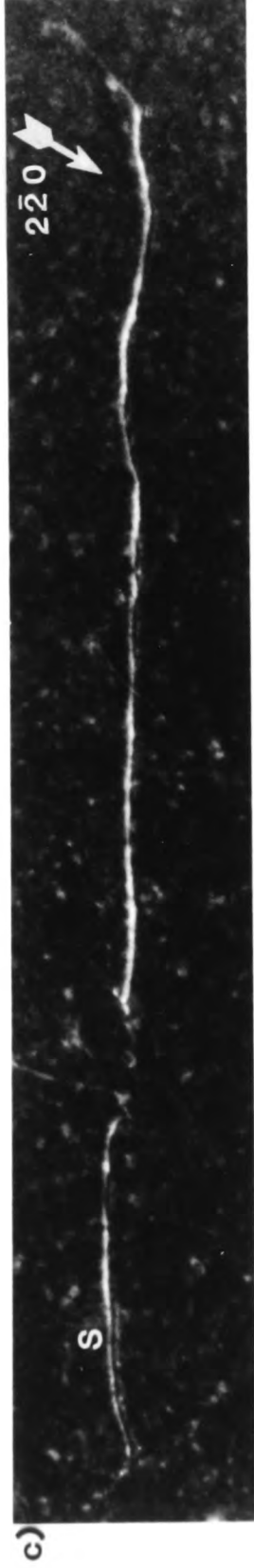
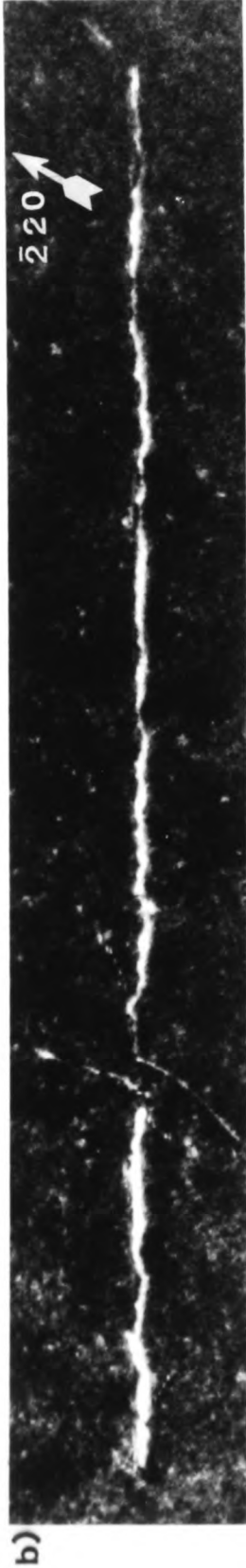
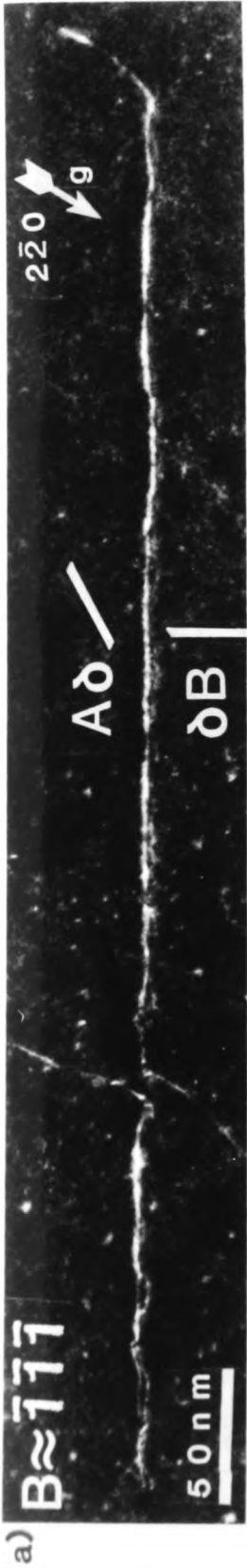
Interpretation:

Most of this 60° dislocation seems to have reacted to irradiation in a way consistent with other observations: Constrictions have been annihilated leaving an even dissociation and vacancy clusters have formed close to the line; dissociated, straight line sections have not apparently climbed and the Burgers vector of the dislocation is unchanged. No loops are seen to have nucleated directly onto partials. Mechanisms already outlined therefore seem to be operating.

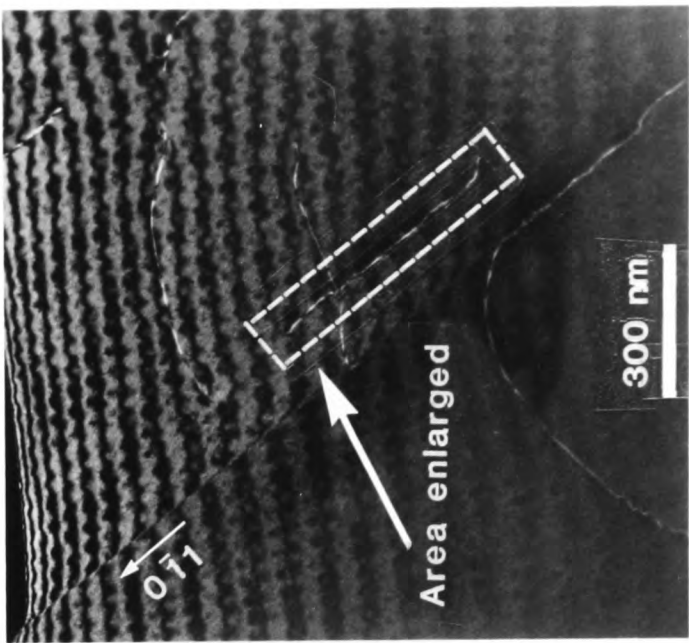
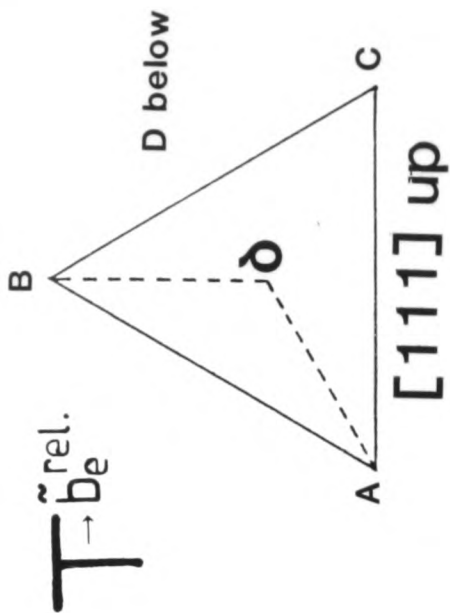
The survival of some constrictions may be relevant to any explanation of the glide of a section of this dislocation *away* from 60° orientation; they may indicate that, for whatever reason, this area did

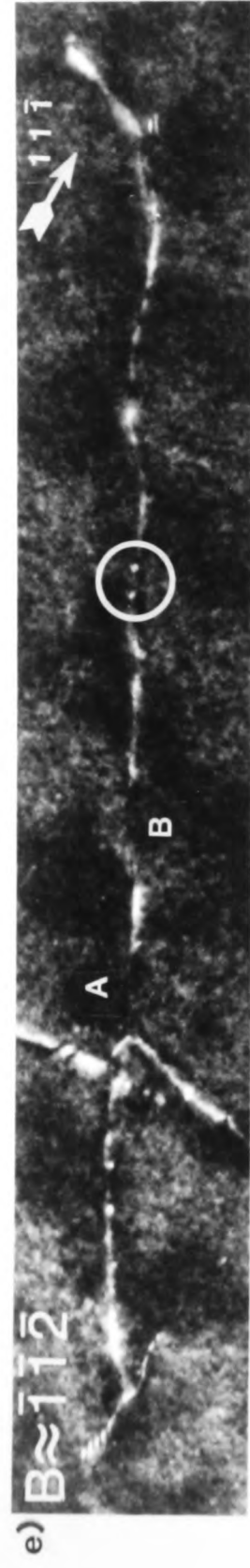
¹⁴*SFC is not evident along sections 'A' and 'B'; in region 'A' an inclined dislocation has intersected and reacted to create a very complex configuration, whilst at 'B' the image of dislocation $\tilde{\mathbf{b}}_T = \overrightarrow{\mathbf{AB}}$ is thought to have been affected by this nearby inclined dislocation. The region at 'B' can be seen to be dissociated in micrograph (d) whilst region 'A' is considered at length in figure 4.7.*

Figure 4.3
(2 pages)

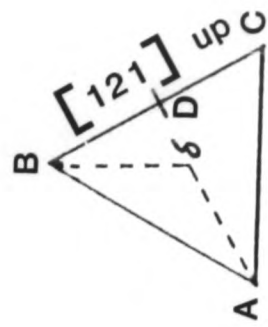
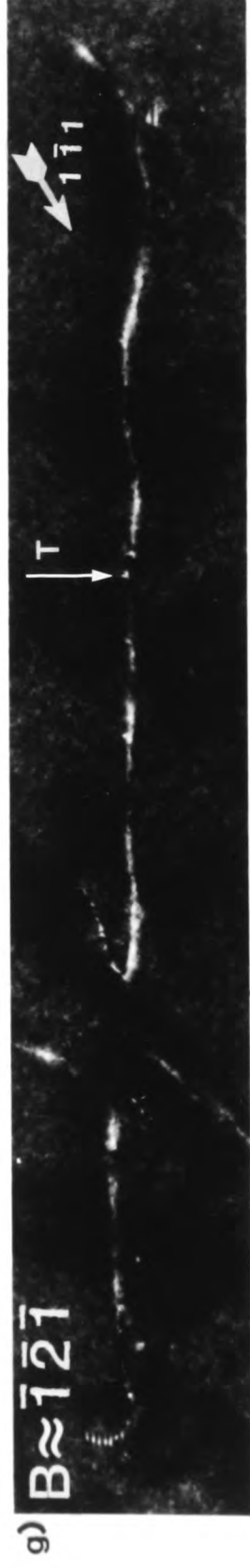
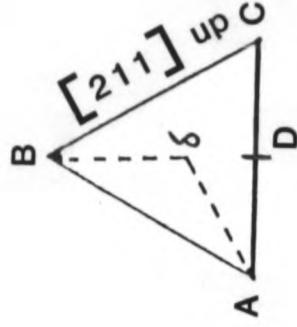
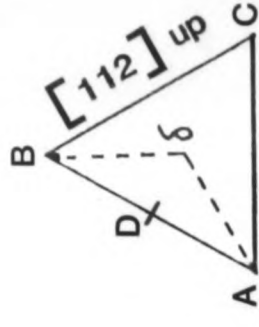


60° Dislocation in Fe-25%Ni-17%Cr (at.%)
0.11 dpa at ~430°C





Direction of tilt
from (111) pole



60° Dislocation in Fe-25%Ni-17%Cr (at.%)

0.11 dpa at ~430°C

D below

not receive a high defect flux during irradiation (although it was not very far removed from the HVEM beam centre), suggesting, perhaps, that the origins of this glide are thermal. An alternative explanation is, of course, that jogs along this section are all of the same sign, although the absence of *any* clusters here might suggest the former explanation. This observation was never duplicated elsewhere in this material, however, and its cause remains uncertain.

Figure 4.4; 65° Dislocation in Fe-25%Ni-17%Cr; 0.12 dpa at ~ 430°C

Observations:

A single micrograph has been selected, which provides clear evidence of the nature of the cluster damage along dislocations. This area is taken from the extremely long dislocation of figure 4.6, extending to the left from point 'E' shown at the bottom of the first page of that figure.

The extra half-plane lies below this near 60° dislocation, which has total Burgers vector $\tilde{b}_T = \vec{AB}$. In this reflection ($[\bar{1}\bar{1}1]$) the lower partial, which has $\tilde{b}_p = \vec{\delta B}$, is imaged. This partial lies close to edge orientation. The beam direction $\tilde{B} \approx [\bar{2}\bar{1}\bar{1}]$, as represented by the tetrahedron at lower right, separates cluster and line images in a sense consistent with their lying below the dislocation. All clusters close to the line are displaced in the same sense on tilting to the (211) pole.

Confirmation of vacancy nature is provided by the observation that very many of these clusters can be seen to be SFT. Other smaller features exhibit contrast inconsistent with any perfect or Frank loop variety and are probably also three-dimensional and vacancy in nature. Small features, very similar to these, have previously been seen to grow to become identifiable as SFT during an in-situ irradiation experiment in silver (**Jenkins et al**, 1987).

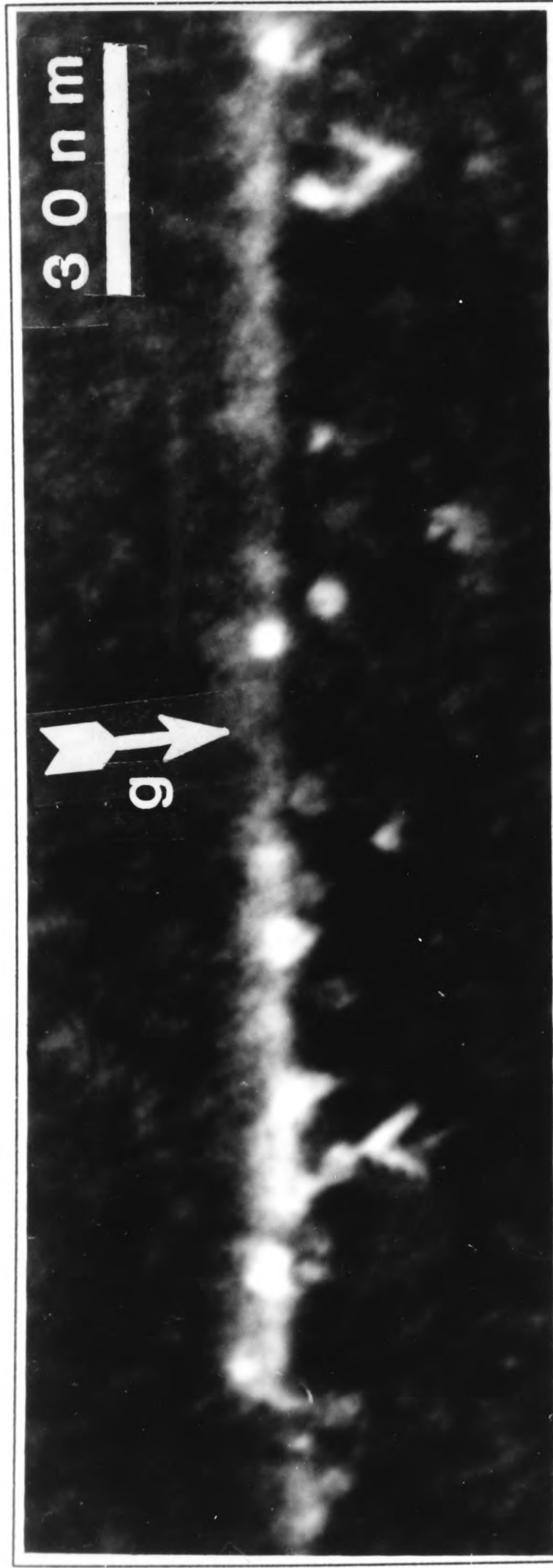
Figure 4.5; Glissile and Sessile Dislocations in Fe-25%Ni-17%Cr;

Page 1: Before Irradiation; Page 2: 0.11 dpa at ~ 430°C.

Observations: Initial Configuration;

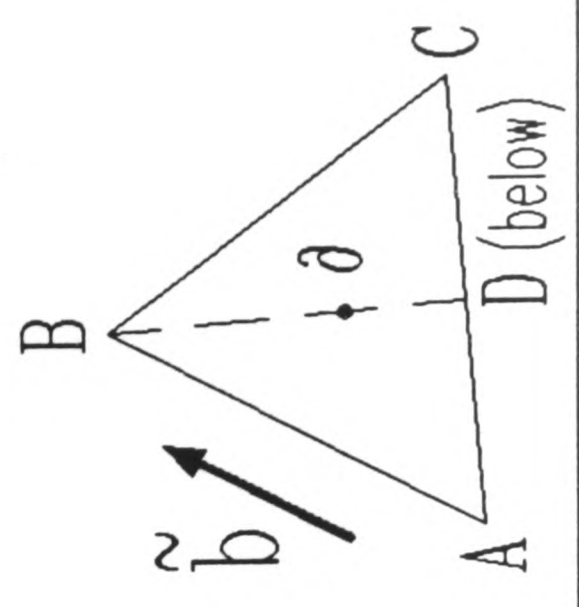
In this figure a section of a glissile dislocation with Burgers vector $\tilde{b}_T = \vec{AB}$ on δ , seen to the left-hand side, has intersected with a few of many dislocations $\tilde{b}_T = \vec{BD}(\alpha)$, which are short and steeply inclined, to form segments of the sessile Lomer dislocation $\tilde{b}_T = \vec{AD}(\alpha/\delta)$. These inclined dislocations may have been frozen in position when the stress leading to their glide was relieved. Glide of a large number of these dislocations $\vec{BD}(\alpha)$, as well as others (see below), along the slip trace visible from lower right to upper left in the low magnification micrograph (a), probably occurred during deformation of the foil prior to thinning to result in the formation of the step at the edge of the foil. The reaction product $\vec{AD}$ is sessile in both α and δ and should therefore lie exactly on the plane α defined by this trace. Lengths of dislocation $\vec{AD}$ (e.g. '1') are out-of-contrast in $\tilde{g} = [202]$ ((d)) and

Figure 4.4

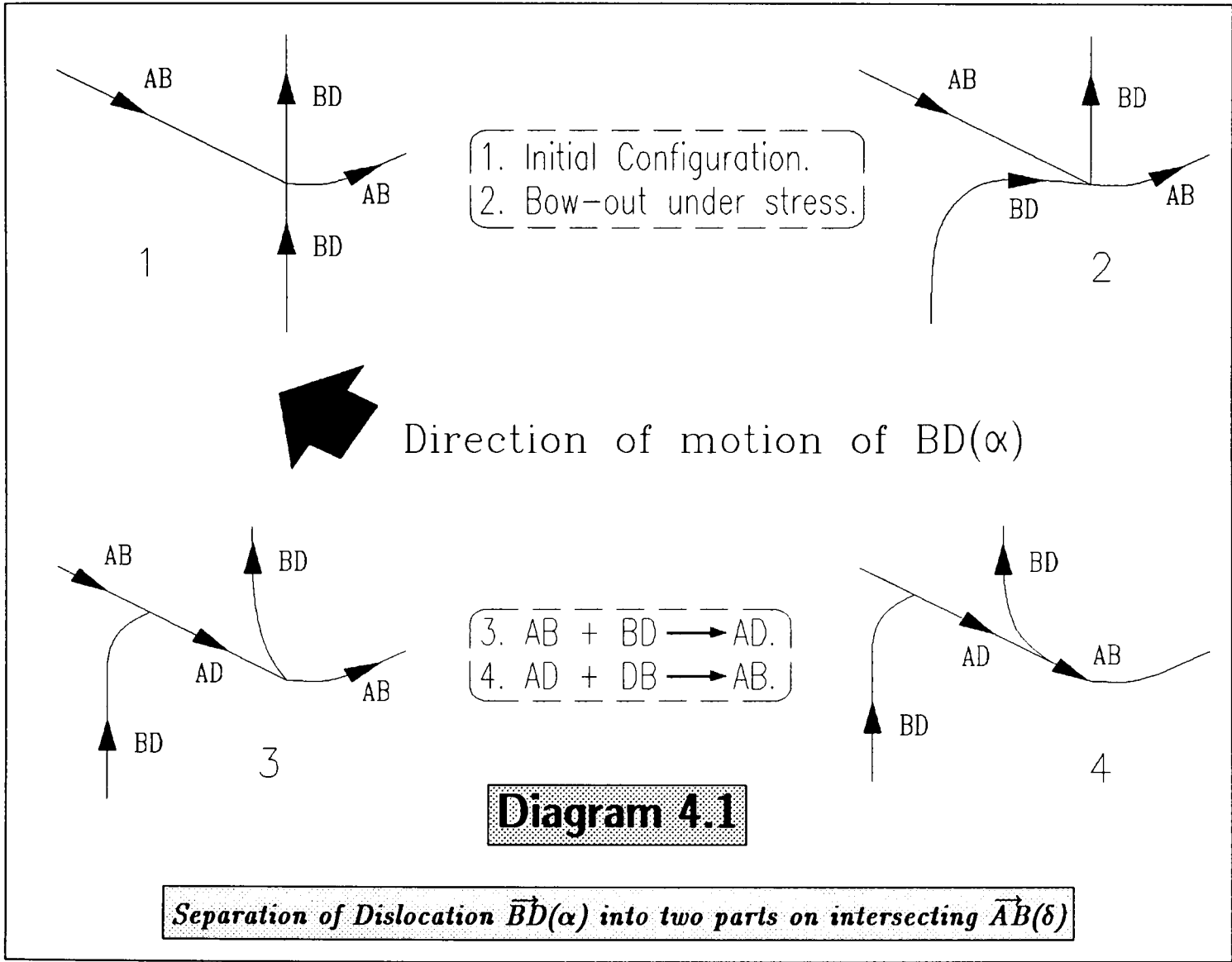


65° Dislocation in Fe-25%Ni-17%Cr (at.%)
 0.12 dpa at ~430°C

$$\tilde{B} \approx [2\bar{1}1] \quad \tilde{g} = [\bar{1}\bar{1}1]$$



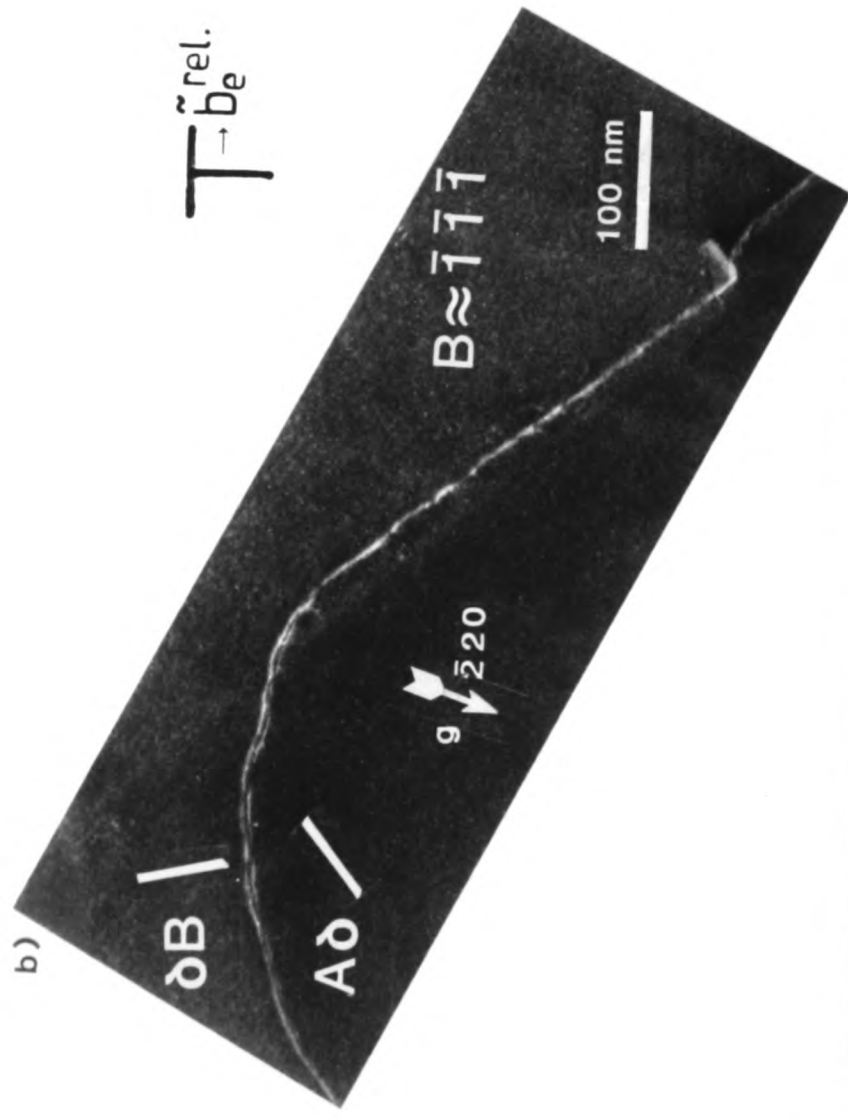
appear completely constricted. Short sections of line, labelled ‘A’ and ‘B’, are believed to be two ‘halves’ of a single dislocation $\vec{BD}(\alpha)$. It is expected that the intersection of $\vec{AB}(\delta)$ with $\vec{BD}(\alpha)$ will proceed via an intermediate configuration requiring the splitting of dislocation $\vec{BD}$ into two segments, either as shown in diagram 4.1, or via a mechanism such as that shown in diagram A.10 (appendix). Both sections of dislocation $\vec{BD}(\alpha)$ show SFC in $\tilde{g}=[0\bar{2}2]$ ((c))¹⁵; the lower section (‘B’), which extends downwards on plane α and is in roughly 30° orientation, appears to be very widely dissociated. This wide dissociation may be stabilized by the creation of a Lomer-Cottrell ‘type 1’ barrier (see below and Hirth & Lothe, p.801). A large jog (‘J’) has formed in dislocation $\vec{AD}$ near its intersection with the upper section of $\vec{BD}(\alpha)$. This jog may lie along $[0\bar{1}0]$. Notice that the entire length of dislocation between ‘A’ and ‘B’ appears fairly straight (and constricted) before irradiation.



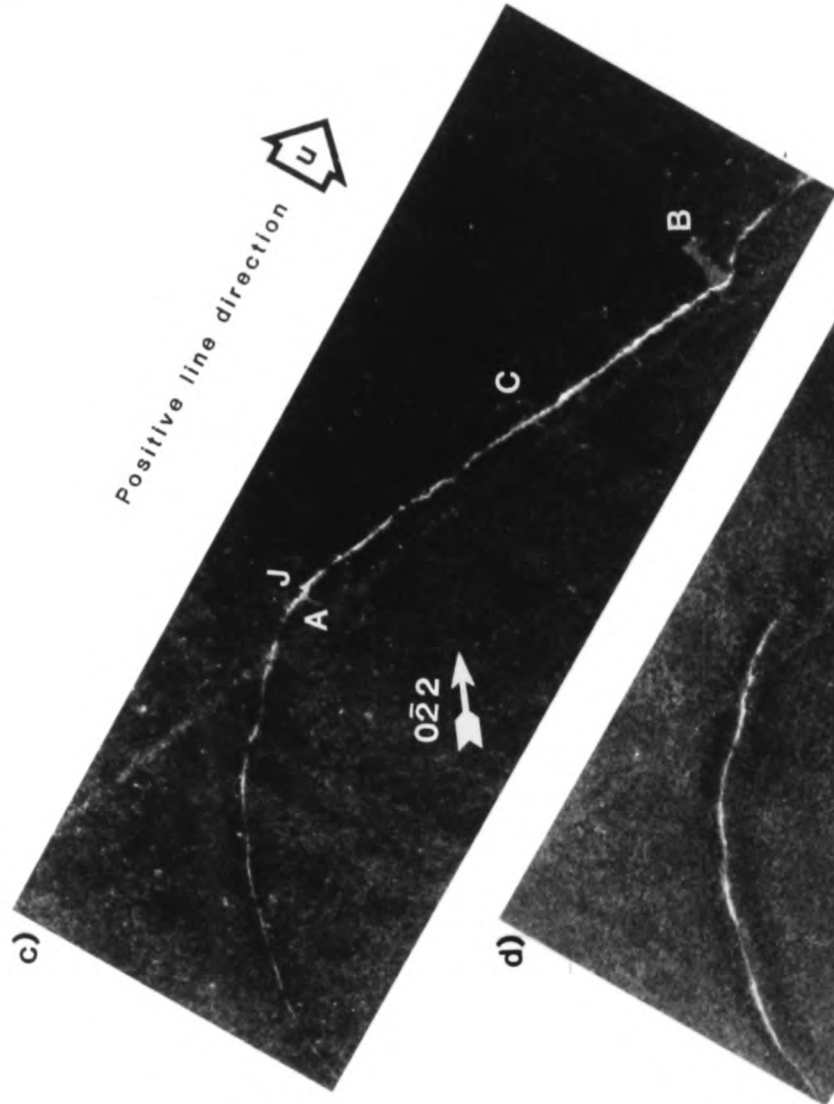
An opposite dislocation $\tilde{b}_T = \vec{DB}(\alpha)$ appears to be intersecting towards the middle of this length. This has re-created a short segment of Burgers vector $\tilde{b}_T = \vec{AB}$ (near ‘C’) which is not well resolved prior to irradiation but is readily visible in the post-irradiation micrograph (h). Numerals ‘1’ and ‘2’ refer to the sections of line, which have *total* Burgers vector $\vec{AD}$, above and below this central portion respectively. The lower portion (‘2’) is complex: The line of contrast faintly visible in $\tilde{g}=[\bar{2}02]$ ((d)) is a Shockley partial $\vec{\delta C}$, created by the intersection of $\vec{AD}$ with inclined dislocations $\vec{DC}(\alpha)$.

¹⁵ Dislocation $\vec{BD}(\gamma)$ would not show SFC in this reflection.

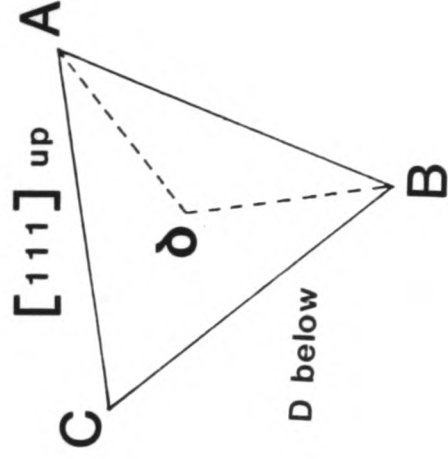
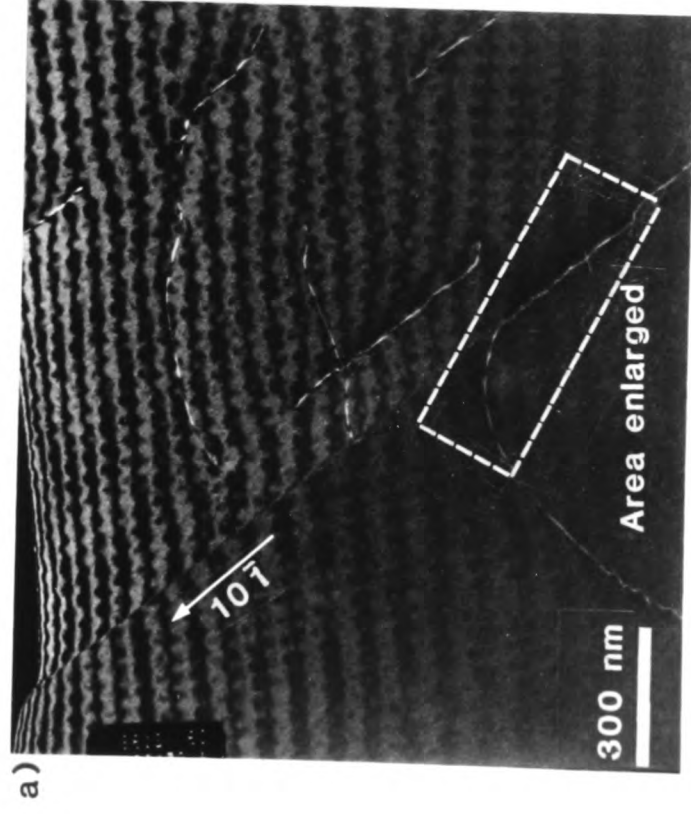
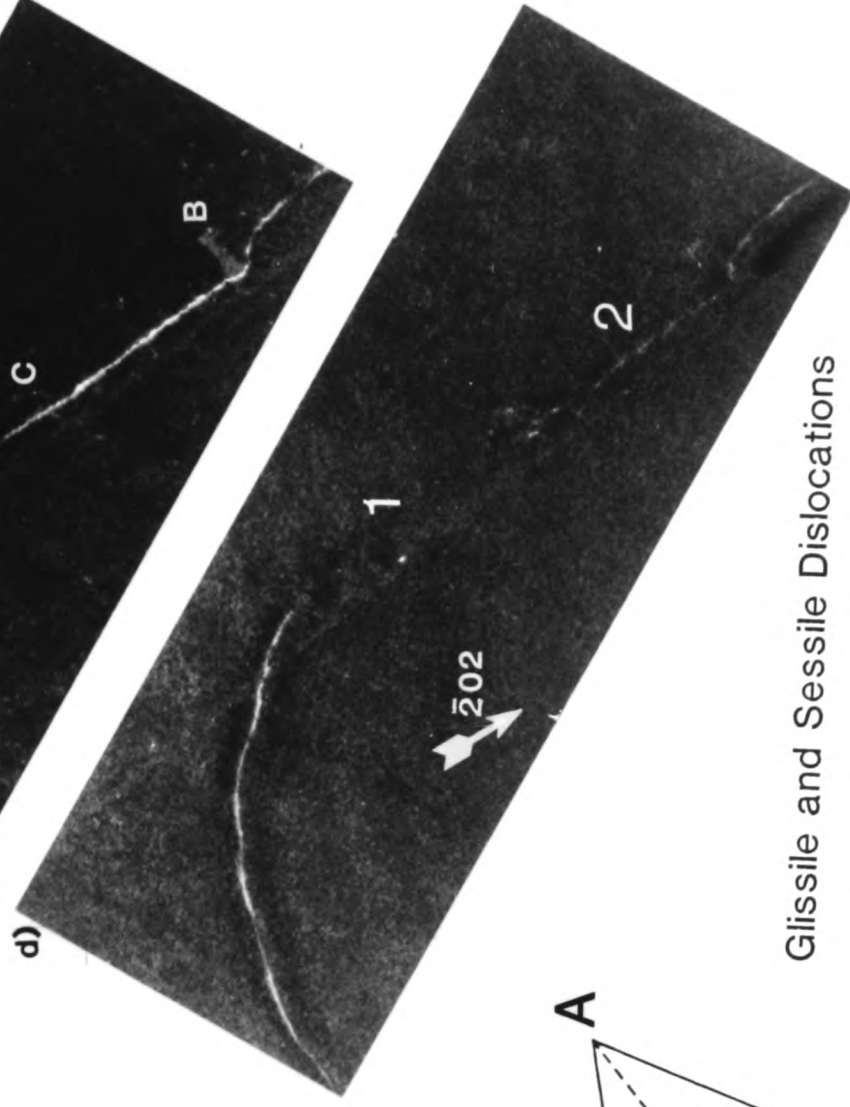
Figure 4.5
(2 pages)



$$\vec{T} \rightarrow \vec{b}_e^{\text{rel.}}$$



Positive line direction 



Glissile and Sessile Dislocations
in Fe-25%Ni-17%Cr (at.%)

Before Irradiation

These dislocations were probably also gliding along the slip trace. Hence, section '2' consists of alternate sessile-glissile-sessile segments. The apparent configuration of section '2' is sketched in diagram 4.2.

It should be noted that these inclined dislocations are very difficult to see. It is possible, therefore, that more of them may be present, resulting in yet further 'segmentation' of section '2'.

In drawing (e), the pre-irradiation configuration is outlined. A large part of section '2' has Burgers vector $\vec{AC}(\delta)$ although its *total* Burgers vector is $\vec{AD}$. This drawing is rotated slightly with respect to the mic-

rographs and the small tetrahedron refers to it alone; the tetrahedron shown in the first page of the figure is relevant to the micrographs.

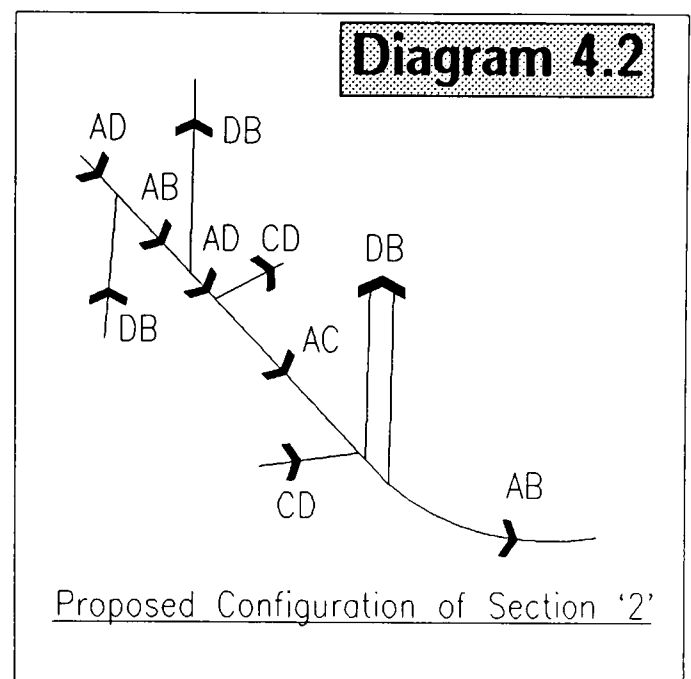
The large discrepancy in dissociation widths of the two halves ('A' and 'B') of $\vec{BD}(\alpha)$ is easily explained by consideration of the geometry of the situation. It can be shown that the obtuse intersection of the top portion of dislocation $\vec{BD}(\alpha)$ with $\vec{AB}(\delta)$, with line directions and Burgers vectors as shown, would result in the creation of a 'type 4' extended dislocation barrier. Although the existence of this barrier is theoretically favourable, it requires the formation of the relatively high-energy, $\frac{1}{3}\langle 110 \rangle$ stair-rod $\vec{b} = \vec{A\alpha}/\delta D$ and is very rarely seen. Were it to have formed here its dissociation width would be small (see Hirth & Lothe, p.801, for details).

However, the acute intersection of $\vec{BD}(\alpha)$ with $\vec{AB}(\delta)$ may lead to the formation of a 'type 1' Lomer-Cottrell lock. This requires only the formation of the low energy, $\frac{1}{6}\langle 110 \rangle$ stair-rod $\vec{b} = \vec{\delta\alpha}$ and has a significantly wider dissociation width. It is possible that this lock may have formed, where $\vec{BD}(\alpha)$ (part 'B') intersects section '2', artificially widening the partial separation.

Beyond this the pre-irradiation configuration has not been further determined.

Observations: Post-Irradiation;

Glissile and sessile dislocations have responded distinctively to irradiation. Along the glissile dislocation $\vec{AB}(\delta)$ at left constrictions have disappeared and it now appears as a smooth line. In $|\vec{g} \cdot \vec{b}_T| = 2$ images taken after irradiation (see figure 4.1) both partials could not be individually resolved; these images were, however, of poor quality and whilst the partial separation has seemingly narrowed this cannot be determined with surety. Reflections of the full contrast series confirm its Burgers vector to be



unchanged. The part of section '2' with Burgers vector $\vec{AC}$ also appears little affected by the irradiation. Partials can be seen to be dissociated in the $|\vec{g} \cdot \vec{b}_T| = 2$ image ((f)), whilst SFC is visible in micrograph (h) ($\vec{g} \cdot \vec{b}_T = 0$). The partial separation at 'B' of dislocation $\vec{BD}(\alpha)$ can be seen to have narrowed by comparing micrographs (c) and (f).

Section '1' of the sessile dislocation $\vec{AD}$ is now sharply cusped. These cusps vary from ~ 4 to ~ 18 nm. in length. In micrograph (g) the foil is tilted to bring planes α to the vertical. Notice that dislocation $\vec{AD}$ is now cusped *to the left* of the original trace along which it was formed. Sharp points of the cusps lie closer to the original dislocation position. Both dislocations $\vec{AB}(\delta)$, at left, and $\vec{AD}(\delta/\alpha)$ have promoted the formation of small cluster damage in their vicinity. This cluster damage is particularly clearly visible in (h), from which it can also be seen that it lies to the compressional strain field side of both dislocations. Several of these clusters show arrow-head contrast typical of vacancy SFT at higher magnification. Dislocation $\vec{AB}(\delta)$ also seems to have glided downwards on δ , away from this line of clusters whilst jog 'J' has disappeared.

Notice that the clusters have mainly formed in a discrete line which lies accurately parallel to the position of the glissile dislocation prior to irradiation. Near the Lomer dislocation the line of these clusters does not appear so discrete, however. Section '2' lies some distance from the centre of the irradiation, which may explain the absence of these vacancy clusters here.

Interpretation:

For the case of the sessile Lomer dislocation of section '1', it may definitely be established that the sense of motion of the line is consistent with interstitial climb: As is shown in drawing (e), the extra half-plane(s) of this dislocation lies both downward and to its right in these (111) foils. Hence, the observation that cusping of the line is to the left of the slip trace along which it was formed is consistent with interstitial climb. The possibility that this dislocation is actually moving by $\{001\}/\langle 110 \rangle$ glide must also be considered but arguments put forward in the preceding chapter relating to figure 3.6 are equally applicable here. The conclusion that the Lomer dislocation of section '1' is climbing by the absorption of interstitials is reinforced by the observations that vacancies are still seen to cluster along this dislocation, and that sharp points of the cusp lie towards the compressional strain field, i.e. the direction from which the dislocation has come, as was seen in figure 3.6. The disappearance of the jog 'J' is also consistent with interstitial precipitation along the neighbouring sessile section of dislocation $\vec{AD}$; if dislocation $\vec{AB}(\delta)$ has glided downward, as appears to be the case, this jog would be lengthened unless annihilated by climb of the Lomer dislocation.

Finally, the formation of a cloud, rather than a discrete line, of vacancy clusters near the Lomer dislocation suggests that its motion has been more gradual than that of the glissile dislocation $\vec{AB}(\delta)$ (which may have been ‘pinned’ by these clusters - see ‘*Points of Discussion*’ towards the end of this chapter). This may further indicate that glide processes are not dominant here.

Figure 4.6; 60° Dislocation Segment in Fe-25%Ni-17%Cr; 0.12 dpa at $\sim 430^\circ\text{C}$.

Observations:

As is shown by the low magnification micrograph at the bottom of the first page of this figure, a short segment from a very long ($> 2\mu\text{m.}$) dislocation has been enlarged. Included in the area shown are two background loops (‘A’) and a loop which lies very close to the dislocation (‘B’). The dislocation has Burgers vector $\tilde{b}_T = \vec{AB}$ and lies exactly in the 60° orientation here.

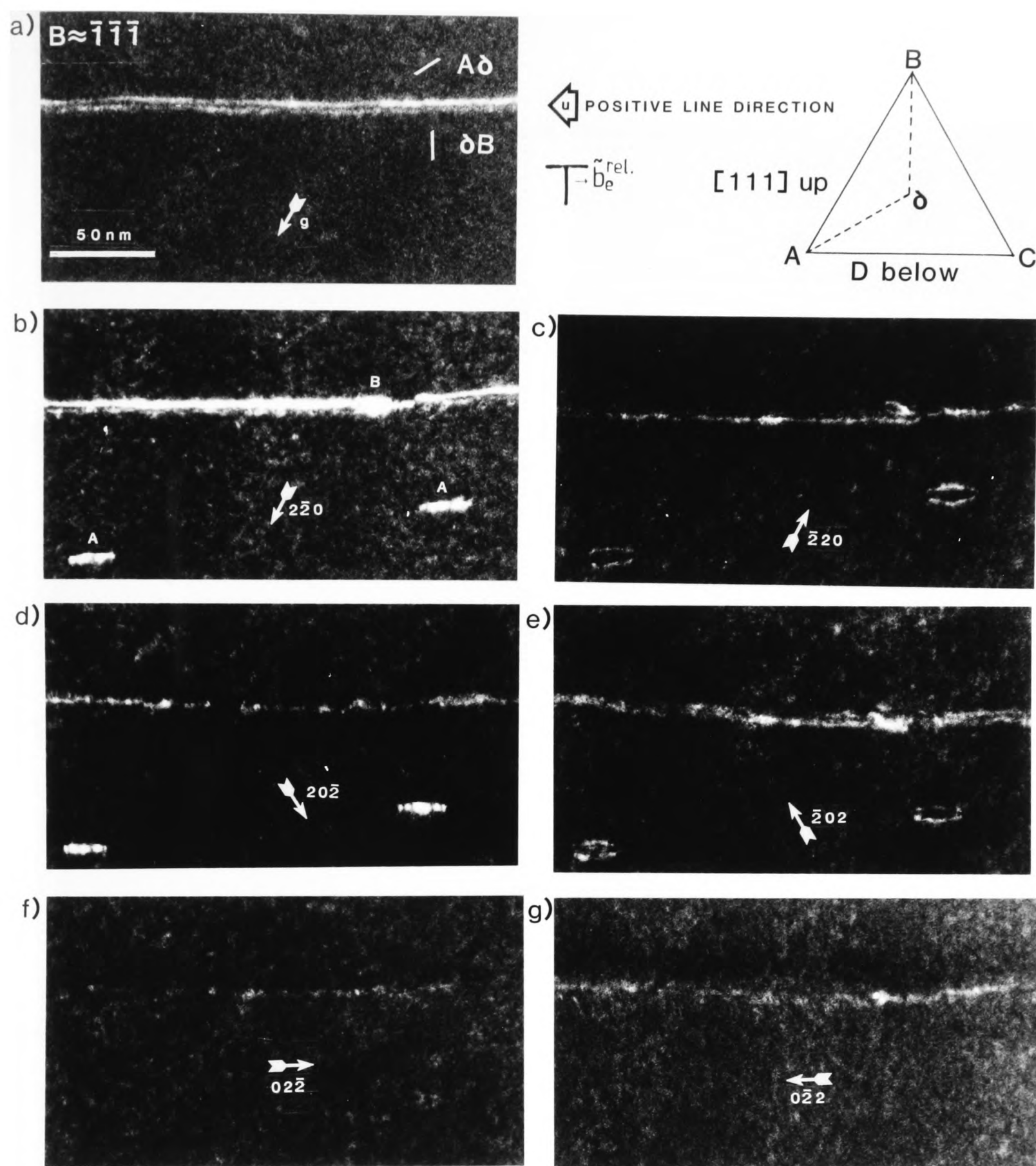
Micrograph (a) was taken before irradiation, whilst micrographs (b)-(w), and the low magnification locator, are taken after irradiation. The post-irradiation micrographs are shown roughly in the chronological order in which they were taken. Before irradiation the dislocation is very evenly dissociated, with a partial image separation of $\sim 40\text{ \AA}$.

The most striking features seen after irradiation are the two background loops ‘A’ and loop ‘B’, which apparently lies *on* the dislocation. The line itself can be seen to be still dissociated along much of its length in (b) and (c) except near a new constriction which has formed close to loop ‘B’, at which one section of the line is displaced with respect to the other. This is confirmed by SFC exhibited in the $\tilde{g} \cdot \tilde{b}_T = 0$ image (s). This dissociation may have narrowed slightly after irradiation, but, if so, it is not a large effect.

Loops ‘A’ show ‘inside’ contrast in $\tilde{g} = [2\bar{2}0]$ and $[20\bar{2}]$. They also show SFC in $\tilde{g} = [1\bar{1}1]$ and are out-of-contrast in $\tilde{g} = [02\bar{2}]$. This is consistent with interstitial Frank loops of Burgers vector $\tilde{b}_L = \vec{\beta B}$. Loops labelled ‘A’, ‘C’ and ‘D’ in the low magnification micrograph at the bottom of the first page of the figure were also found to be interstitial Frank loops, with Burgers vectors $\vec{\beta B}$, $\vec{\gamma C}$ and $\vec{D\delta}$ respectively.

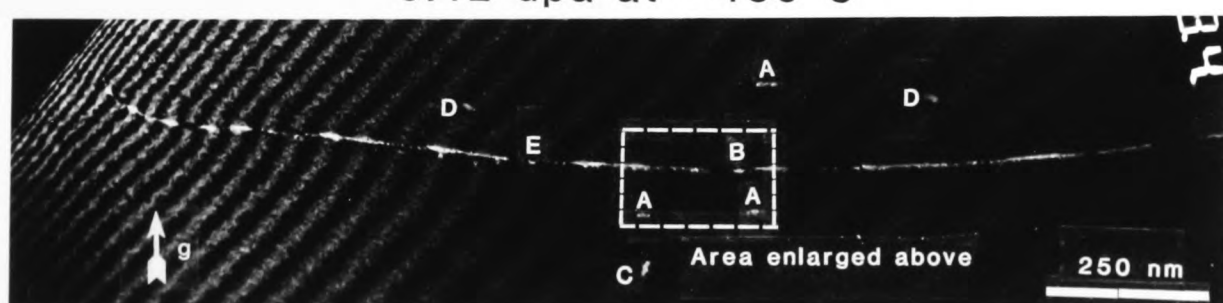
A large amount of cluster damage is evident along the line. It is most clearly resolved in the $\tilde{g} \cdot \tilde{b}_T = 0$ images, (m) and (p)-(s). In $\tilde{g} = [1\bar{1}\bar{1}]$ ((k)), for example, many clusters show arrow-head contrast typical of vacancy SFT. Almost all others of these clusters exhibited arrow-head-like contrast in at least one of the micrographs taken of this area¹⁶. Additionally, this line of cluster damage is displaced downward, and upwards, with respect to the line on tilting to $\tilde{B} \approx [2\bar{1}\bar{1}]$ and $\tilde{B} \approx [0\bar{1}\bar{1}]$ respectively. Direct measurement of the magnitude of this motion suggests that these clusters lie $\sim 15\text{ nm}$. below the dislocation, i.e. to its compressional strain field side. It is very important to notice

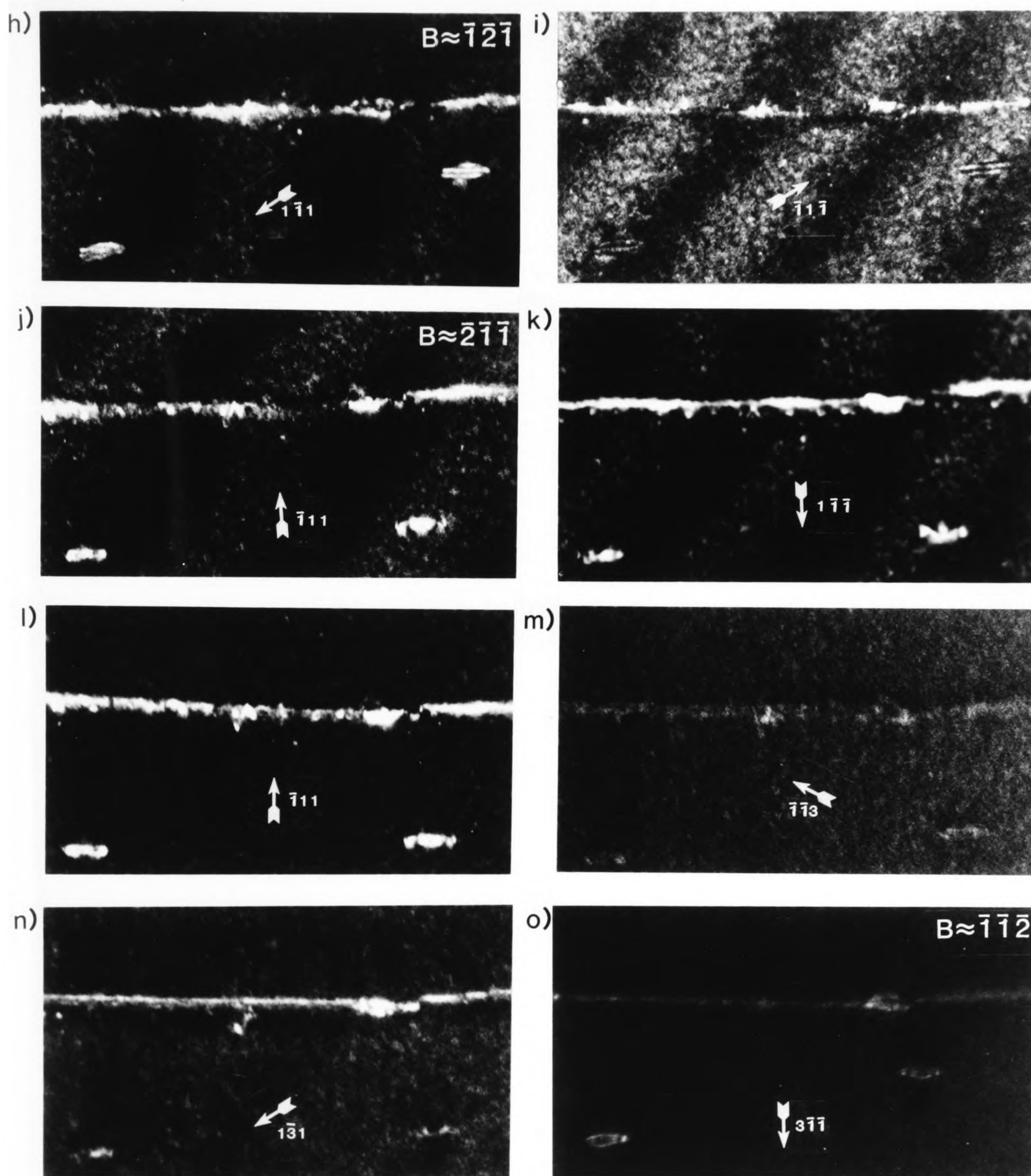
Figure 4.6
(3 pages)



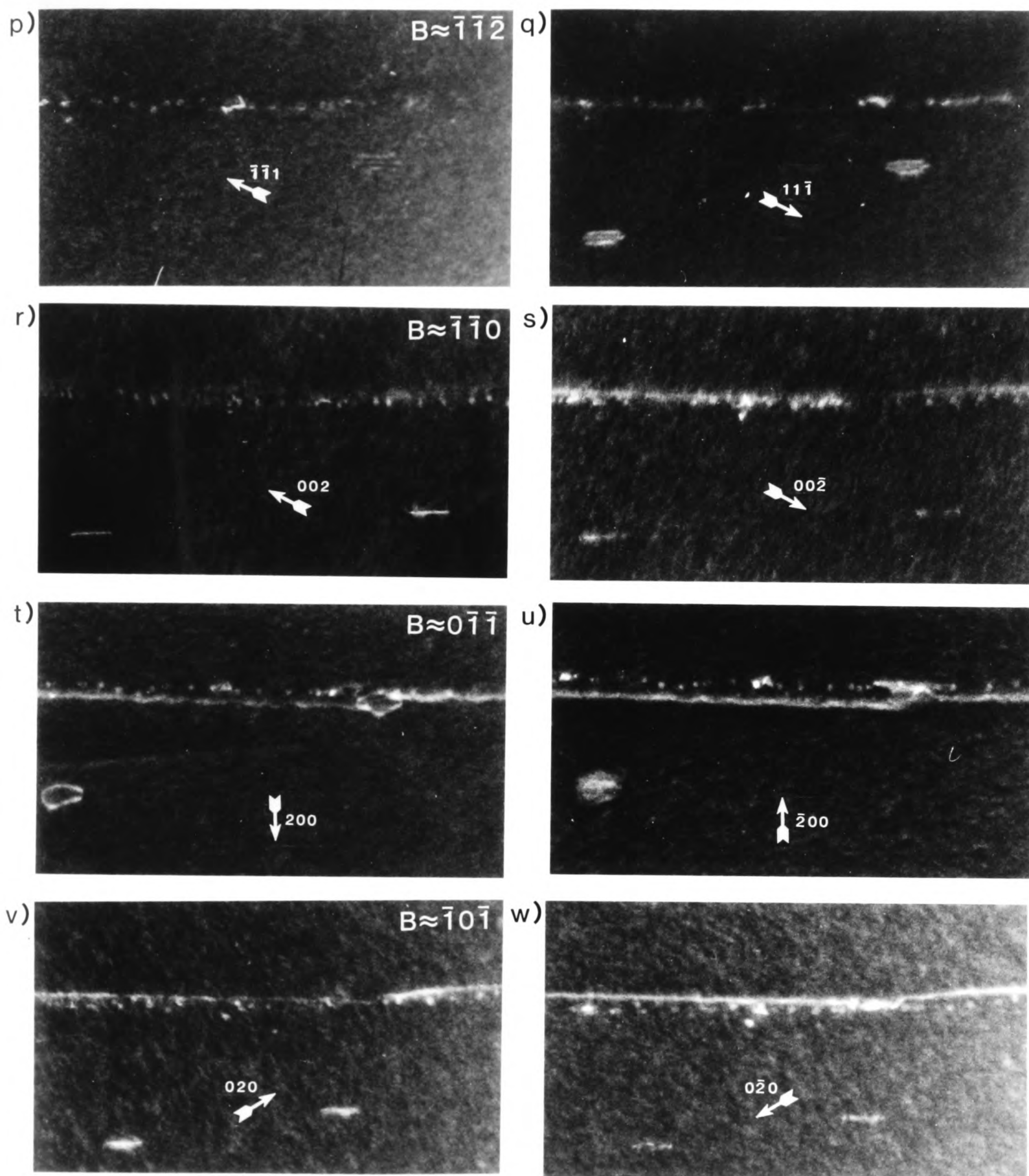
60° Dislocation segment in Fe-25%Ni-17%Cr (at.%)

0.12 dpa at ~430°C





60° Dislocation segment in Fe-25%Ni-17%Cr (at.%)
0.12 dpa at ~430°C



60° Dislocation segment in Fe-25%Ni-17%Cr (at.%)
 0.12 dpa at ~430°C

that this line of cluster damage is displaced in the same sense, and by the same amount with respect to *all* sections of dislocation $\overrightarrow{AB}$ on tilting to *any* pole.

Loop 'B' is a particularly interesting feature: In the first page of the figure, this loop shows 'outside' contrast in $\tilde{g} = [\bar{2}20]$ ((c)) and appears very similar to the background, $\tilde{b}_L = \overrightarrow{\beta B}$, loops 'A'. In micrographs (j), (k) and (l) ($\tilde{g} = \pm [\bar{1}11]$), loop 'B' also appears very similar to loops 'A'. Notice, however, that 'B' appears rotated very slightly with respect to the background loops in (c). On the second page of the figure, loop 'B' can also be seen, showing faint contrast, to be rotated slightly in $\tilde{g} = [\bar{1}\bar{1}3]$ ((m)). This reflection has $\tilde{g} \cdot \tilde{b}_T = 0$ for dislocation $\overrightarrow{AB}$. Other differences between loops 'A' and 'B' also become apparent on the second page of the figure: In $\tilde{g} = \pm [\bar{1}\bar{1}1]$ ((h) and (i)), loops 'A', but not 'B', show SFC in both reflections, as expected from faulted Frank loops. SFC is also not shown by 'B' in micrographs (o), (p), (q) and (s), in all of which $\tilde{b}_L = \overrightarrow{\beta B}$ loops can be seen to be faulted.

A small faulted region does, however, appear to be visible near the top of feature 'B', showing solid contrast in $\tilde{g} = [\bar{1}\bar{1}\bar{1}]$ ((i)). It is possible that part of an SFT is visible within this region. This SFT may be visible as two faint lines above the upper edge of loop 'B' in (j) and (l), and may also be visible to the left of 'B' in micrograph (n).

On the third page of the figure the micrographs are shown exactly in chronological order. Loops 'A' are visible, both showing SFC, in $\tilde{g} = \pm [\bar{1}\bar{1}1]$ ((p) and (q)). In micrograph (q) the small region to the top of 'B' is also visible.

In $\tilde{g} = \pm [200]$, the foil is tilted such that planes β are brought nearer to the horizontal (they are actually at an angle of $\sim 35^\circ$ with the horizontal). The right-hand background loop is no longer visible. Both the remaining loop 'A' and loop 'B' now appear very similarly larger. Micrograph (u) shows that loop 'A' is still faulted and must, therefore, still lie on a plane β ; thus, it appears that loop 'B' also lies on, or close to a plane β . When viewed in 'outside' contrast ($\tilde{g} = [200]$, (t)), the shape of both loops is, however, slightly more complex than might be expected: One edge of loop 'A' has now straightened and lies along a direction consistent with the projection of $[1\bar{2}3]$ (β), whilst loop 'B' apparently has a rhomboid shape.

Micrograph (t) appears to show that the constriction, seen close to 'B' in micrograph (c), actually lies where the line is intersected by loop 'B'. It also suggests that the two 'halves' of dis-

¹⁶It is often useful to take a series of micrographs, at different values of deviation parameter (*s*), as an aid to the identification of SFT (see *Jenkins, 1973* for a full discussion): Micrographs (j) and (l) are taken at (*g*, 9*g*) and (*g*, 8*g*) ($\equiv s = 2.35$ and $2.06 \cdot 10^{-2} \text{\AA}^{-1}$) respectively and many clusters are recognizable as SFT in only one of these reflections.

location $\overrightarrow{AB}$ attach to this loop at different points. Notice, however, the presence of at least one, and possibly two, faint line(s) crossing loop 'B' from one 'half' of dislocation $\overrightarrow{AB}$ to the other.

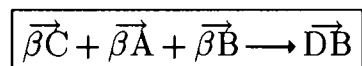
In micrograph (u), the region around 'B' has a very complex appearance: The small faulted region seen in micrograph (q), for example, now appears as a long, bright strip. It apparently lies within the line of cluster damage. Dislocation $\overrightarrow{AB}$ is now visible as a continuous, kinked line, superimposed on which loop 'B' may be visible as a fuzzy haze.

In the final two reflections taken of this area, both background loops 'A' are again visible. The foil is now tilted such that planes β are vertical (as are planes γ), and both loops are seen as thin, straight lines. Notice that the line of cluster damage is displaced by the same amount with respect to the entire dislocation on tilting from (011) to (101) poles.

Interpretation:

The disappearance of the right-hand background interstitial loop in $\tilde{g} = \pm [200]$ suggests that it has the *perfect* Burgers vector $\tilde{b}_L = \overrightarrow{DB}$. However, this same loop is visible, showing SFC, in $\tilde{g} = [1\bar{1}1]$ ((q)), in which reflection loop $\overrightarrow{DB}$ has $\tilde{g} \cdot \tilde{b}_L = 0$, and otherwise exhibits contrast consistent *only* with the *faulted* Frank loop $\tilde{b}_L = \overrightarrow{\beta B}$. As mentioned above, the micrographs on the third page are shown exactly in chronological order; the ordering on the first two pages is not so precise but *all* were taken before *any* of the micrographs of the third page.

It therefore seems as is the right-hand loop has unfaulted *during* the time that this specimen was in the microscope, and specifically between the taking of micrographs (q) and (t), via the reaction:



Large background Frank loops in these materials are generally observed to be round (see, for example, 'D' of figure 3.3). It is possible, therefore, that the truncated appearance of the left-hand background loop in micrograph (t) may indicate that unfaulting is in progress, with one of the unfaulting partials possibly lying along $[1\bar{2}3]$.

The action of a similar unfaulting reaction may explain the complexity of loop 'B': In many ways contrast from this feature resembles the interstitial Frank loops below it. This loop plainly shows 'inside' contrast in $\tilde{g} = [1\bar{1}1]$ and 'outside' contrast in $\tilde{g} = [\bar{2}20]$ as do the Frank loops below it. In addition, most of it appears to lie on, or close to, a plane β . It therefore seems very likely that it originated as an interstitial Frank $\tilde{b}_L = \overrightarrow{\beta B}$ loop. It is not thought to be coincidence that this loop species is aligned with the dislocation line, which is in precisely 60° orientation here. The absence of SFC points strongly towards this loop having been unfaulted, however. Of the possible products from

such a reaction, *the best fit* to the observed contrast is Burgers vector $\vec{AB}$: Loop $\vec{CB}$ would be out-of-contrast in $\tilde{g}=[1\bar{1}1]$ whilst $\vec{DB}$ would be out-of-contrast in $\tilde{g}=[200]$. The observed rotation of loop ‘B’ with respect to loops $\vec{\beta B}$ is also consistent with the rotation of a loop $\vec{AB}$ towards edge orientation, explaining the roughly rhomboid shape seen in micrograph (t). This loop is however very complex: It plainly does not lie on a $(\bar{1}10)$ plane since it does not appear as a thin line when these planes are vertical; all of this loop also does not lie *exactly* on a plane β as evidenced by its ‘fat’ image when planes β are vertical.

A possible explanation for this may be that feature ‘B’ actually lies between the two planes. The apparent reluctance of unfaulted loops to glide to edge orientation is seemingly confirmed by the observation that the newly formed loop $\vec{DB}$ still shows as a very thin line in the final pair of micrographs ((v) and (w)). Loop $\vec{DB}$ is therefore still at, or very close to its original orientation.

The weak contrast seen from ‘B’ in $\tilde{g}=[\bar{1}\bar{1}3]$, and perhaps the faint haze in $\tilde{g}=[\bar{2}00]$, may point to it being only partially unfaulted and hence consisting of elements with Burgers vectors $\vec{AB}$ and $\vec{\beta B}$. The configuration of this feature does not easily make itself known but certain points are apparent:

- It neither lies in the plane of the foil (δ , compare (c) and (t)) nor in a plane γ ;
- It does not have Burgers vector $\vec{DB}$ or $\vec{CB}$;
- If it has Burgers vector $\vec{AB}$, it has probably formed from a $\vec{\beta B}$ Frank loop and thus accommodates interstitials.

Obviously loop ‘B’ and the dislocation have interacted. The dislocation may have prompted the formation of loop $\vec{AB}$, rather than another variant from the unfaulting of a $\vec{\beta B}$ loop. The exact details of the interaction of loop ‘B’ and the dislocation line are difficult to interpret, however. Nevertheless, certain facets of this interaction should be considered:

The suggestion is made, by micrograph (t), that the constriction is due to the occurrence of some reaction of dislocation $\vec{AB}$ with loop ‘B’, since the two ‘halves’ of dislocation $\vec{AB}$ apparently attach to this loop. Comparison of the images of loop ‘B’ in micrographs (c) and (t) demonstrate that loop ‘B’ *definitely* does not lie on a plane δ , and thus, the seemingly unavoidable implication is that the two ‘halves’ of dislocation $\vec{AB}$ are at different heights in the foil, i.e. we are seeing climb of one part of the dislocation with respect to the other. The key to this, however, is the line of cluster damage, which appears completely straight, as did the dislocation prior to irradiation. Similar lines of cluster damage have been seen to form parallel to pre-irradiation dislocation positions, probably at an early stage in the irradiation (see, for example, figures 3.1 and 4.2). Before irradiation this dislocation was evenly dissociated, and therefore jog-free, along its entire $2\mu\text{m}$. length, including the region shown in

the above figure. Hence, all clusters within the line of cluster damage should lie, at least approximately, within the same plane δ .

Thus, we can see from the fact that the line of cluster damage is displaced in the same sense, and by *exactly the same amount*, on tilting from the (011) to the (101), or *any* other, pole, that all sections of dislocation $\vec{AB}$ lie *at the same height* above this line of clusters. The constriction seen near 'B', which is shown in all reflections, therefore corresponds to a kink in the line. Any climb of the line, almost all of which is still dissociated, must therefore have been completely homogeneous along its entire length, including the kinked section. This would appear a little unlikely. The one (or possibly two) faint line(s), visible crossing loop 'B' in micrograph (t), may be (partials $\vec{A\delta}$ and $\vec{\delta B}$ of) dislocation $\vec{AB}(\delta)$, therefore.

It would appear that the bright strip of contrast, seen at the top of 'B' in micrograph (u) amongst others, is vacancy in nature. Contrast is often confused from this region and the fault vector $\vec{R}$ cannot be determined directly. Notice, however, that it appears to lie close to, or within, the line of vacancy cluster damage in micrograph (u) and that some reflections seem to show one, or more SFT associated with it. It would seem likely, therefore, that it is a faulted vacancy defect of some kind, which is possibly extended in three dimensions. It is possible that the shape of this vacancy cluster has been dictated by the nearby interstitial loop 'B' and/or the dislocation.

As is shown by the low magnification micrograph, there is a very low density of background loops here. Thus, the probability that features 'B' should have formed so close to the dislocation by chance seem very slim. The question as to why apparently associated interstitial and vacancy clusters should have formed so close to a dislocation of precisely 60° character is addressed **later**.

The important observations to be drawn from this figure are summarized below: this figure shows the apparent precipitation of point-defects, at a dislocation which lies in precisely 60° orientation. This does not appear to have led to climb of the line, but is likely to be related to the existence of a newly formed kink. The interstitial loop is most likely to have formed from a Frank loop, which was *exactly* aligned with the 60° dislocation.

Figure 4.7; Dislocation Node in Fe-25%Ni-17%Cr,

Page 1: Before Irradiation; Page 2: Schematic; Pages 3 & 4; 0.11 dpa at $\sim 430^\circ\text{C}$

To test the hypothesis that pipe diffusion is an important mechanism by which point-defects interact with dislocations, a dislocation node, which might be expected to slow pipe diffusion, is analysed in detail. This node is that shown at lower magnification in figure 4.3. It is suggested as a result that interstitials may precipitate at points such as this node, by reason of its complex

configuration.

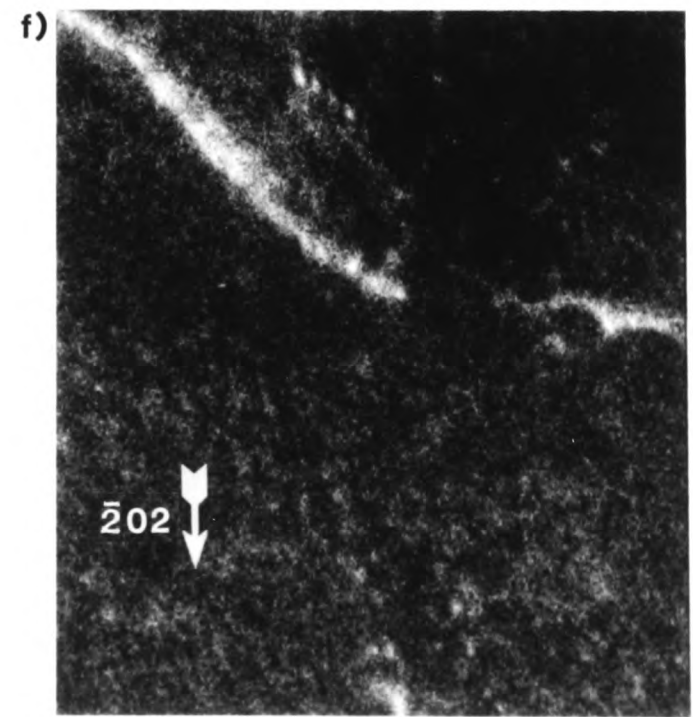
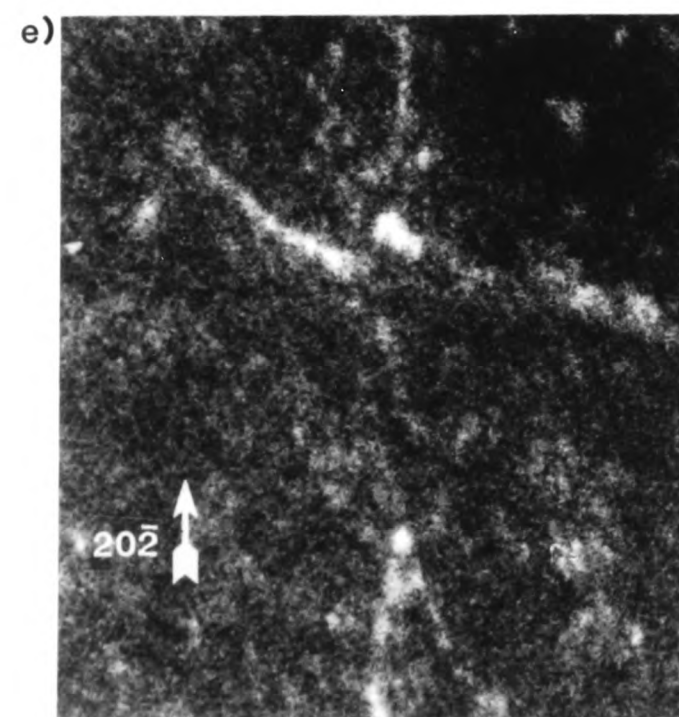
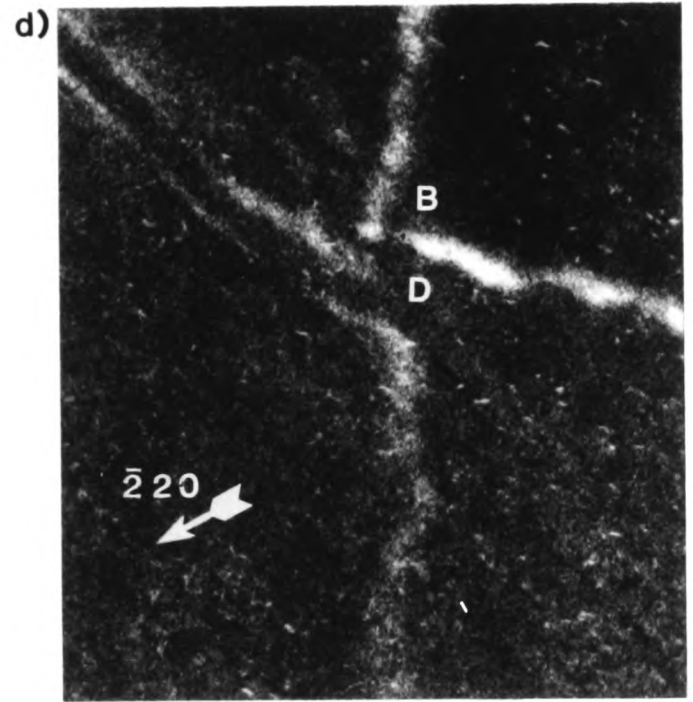
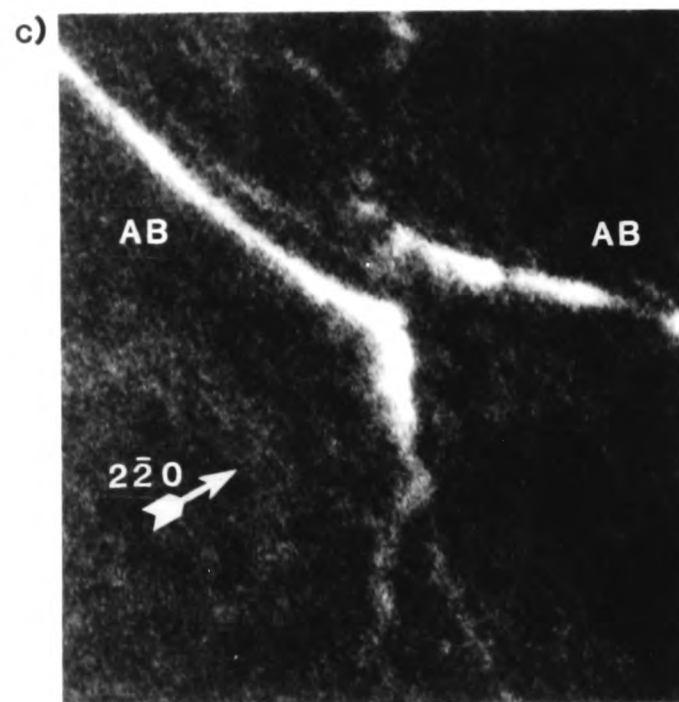
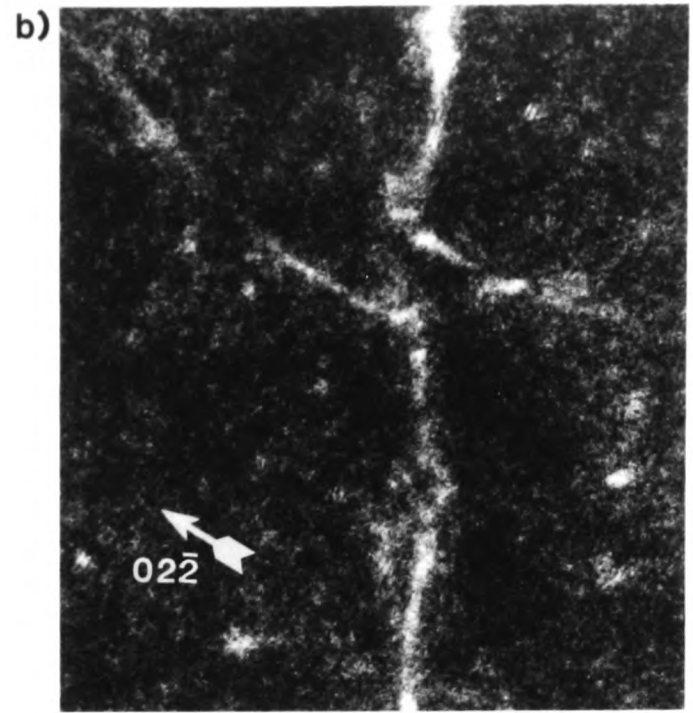
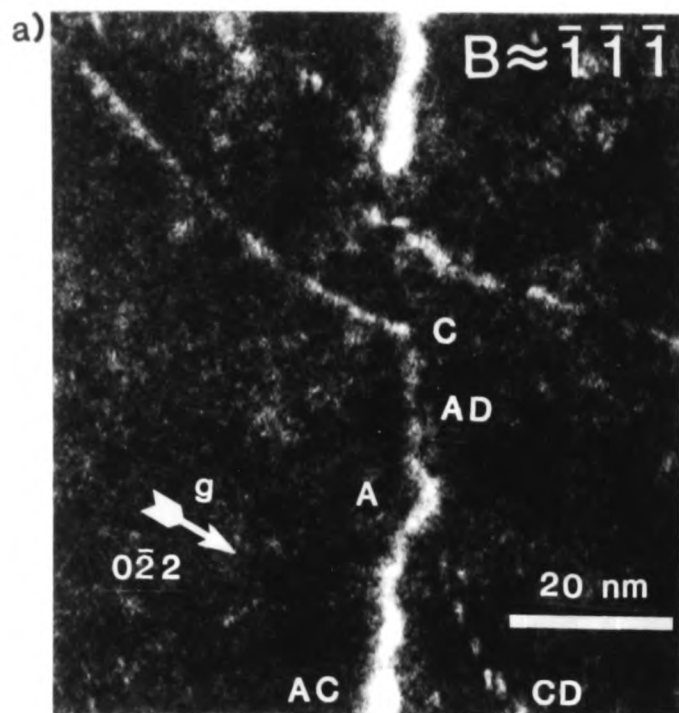
Observations: Initial Configuration;

This node is formed by the intersection of at least three dislocations, with Burgers vectors $\tilde{b}_T = \vec{AB}(\delta)$, $\vec{AC}(\delta)$ and $\vec{CD}(\alpha)$. The pre-irradiation configuration that can be deduced from the reflections taken is represented by the schematic diagram (g) and the orientation of the Thompson tetrahedron is shown on the third page of the figure. Dislocation $\vec{AC}$ is in exactly 60° orientation, parallel to the trace of plane α and is the same dislocation as shown in figure 4.3 (there indexed as $\tilde{b}_T = \vec{AB}$). Dislocation $\vec{AB}$ is also very close to the 60° orientation. Between points 'A' and 'C' the line is out-of-contrast in $\tilde{g} = \pm [20\bar{2}]$ suggesting that it has Burgers vector $\tilde{b}_T = \vec{AD}$. Consideration of the conservation of Burgers vectors at point 'A' dictates that dislocation 'CD' has Burgers vector $\tilde{b}_T = \vec{CD}$, the lower section of which may be in pure screw orientation. It shows SFC in $\tilde{g} = [0\bar{2}2]$ and so is dissociated on a plane α . This dislocation ('CD') is apparently visible in micrograph (c), in which reflection it should have $\tilde{g} \cdot \tilde{b} = 0$. It is possible that other reflections were excited when this micrograph was taken giving rise to a spurious image. All these micrographs appear to show dislocations $\vec{AB}$, $\vec{AC}$ and the upper section of $\vec{CD}$ coming together very close to one another at point 'B'. Micrograph (d) seems to show a very small gap between point 'B' and the upper partial $\delta\vec{B}$ of dislocation $\vec{AB}$ entering from the left. Compiling this information leads to the configuration sketched schematically in (g), in which dashed lines represent supposed dislocation positions within the node core.

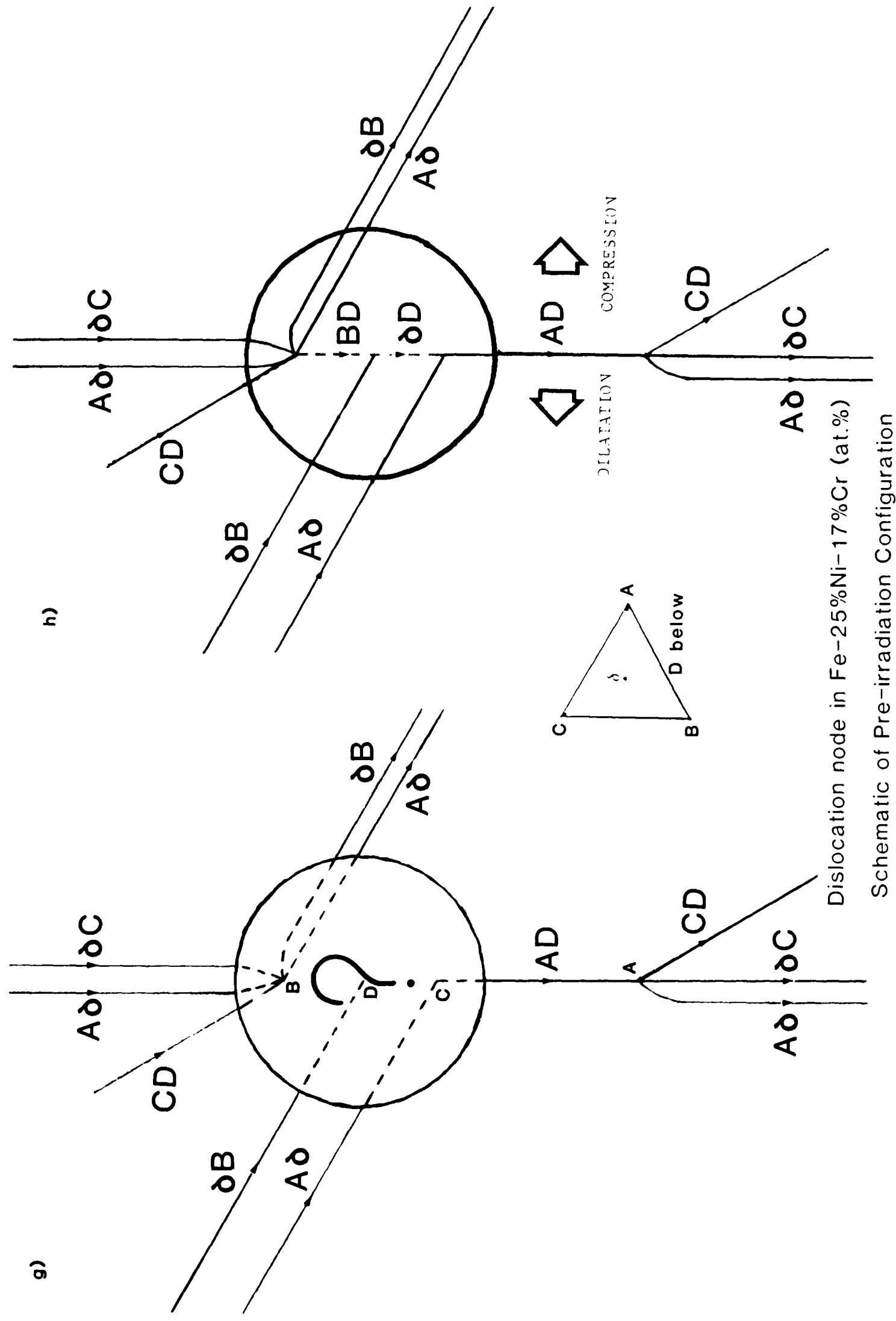
Applying the conservation of Burgers vectors to points 'B', 'C' and 'D' yields dislocations with Burgers vectors $\tilde{b}_T = \vec{BD}$ and $\delta\vec{D}$ as shown in drawing (h). These are represented as dashed lines and are not apparently visible in micrographs (a) to (f). Dislocation $\vec{BD}$ is believed to be very short and also steeply inclined to the plane of the foil and, as such, is unlikely to be visible. Dislocation $\delta\vec{D}$ would not be visible in any of these $\langle\bar{2}20\rangle$ -type reflections. The sense of the extra half-plane, lying to the right, of dislocation $\vec{AD}$ is also indicated in (h). Line directions are shown by small arrow-heads. It should be noted that this initial configuration, a mechanism for the formation of which is propounded in the appendix , is hypothetical. The real configuration may in fact be much more complex, containing an even greater number of dislocations, although Burgers vectors of dislocations between points 'B', 'C' and 'D' must sum to the Burgers vectors of those dislocations indicated by dashed lines. In particular, as is shown in ^{the} ₁ appendix (stage 6), the wide separation of dislocation $\vec{AB}$ may be stabilized by the formation of a Lomer-Cottrell lock.

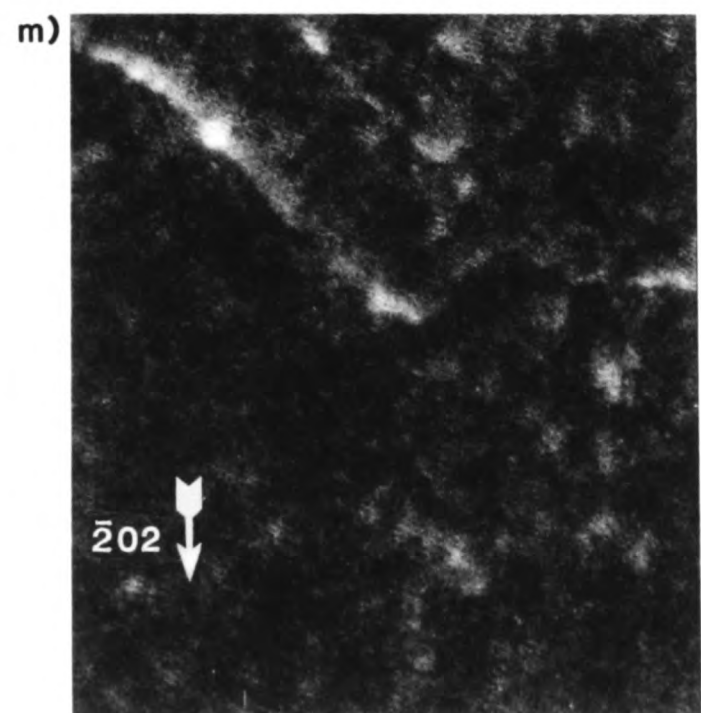
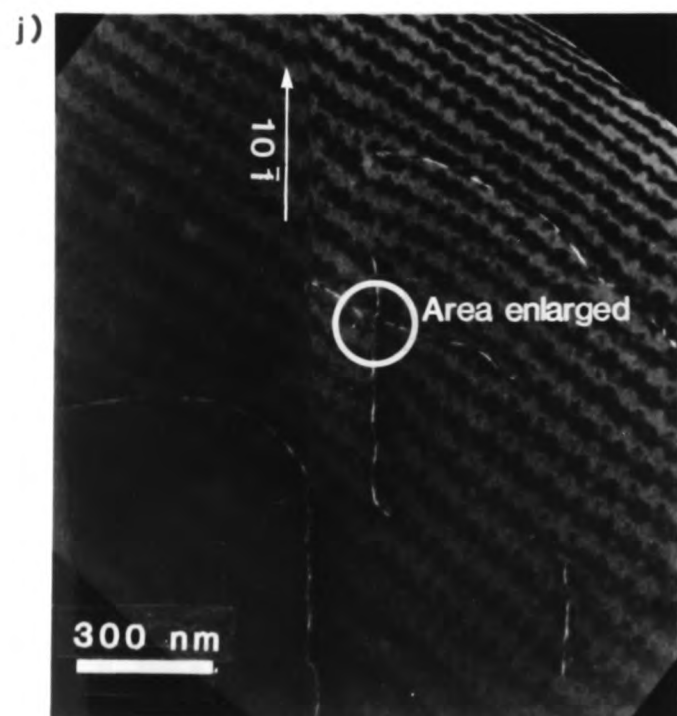
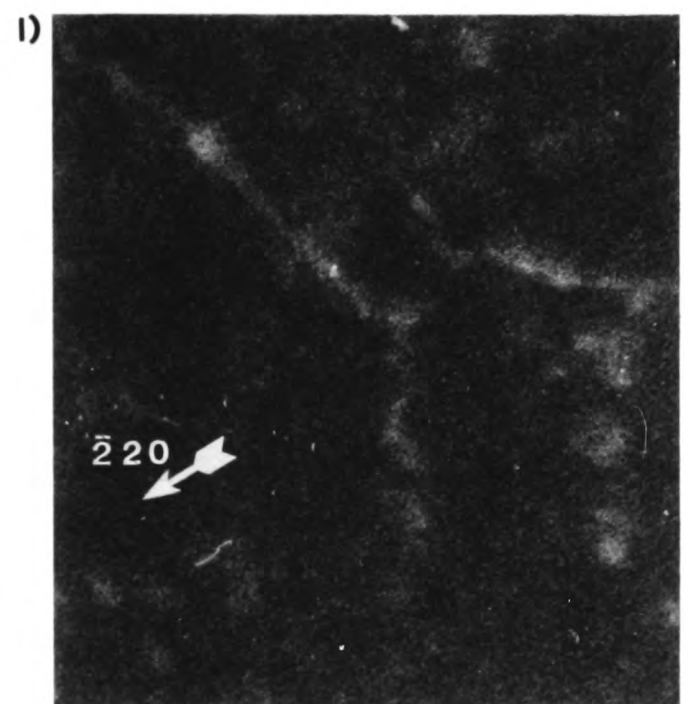
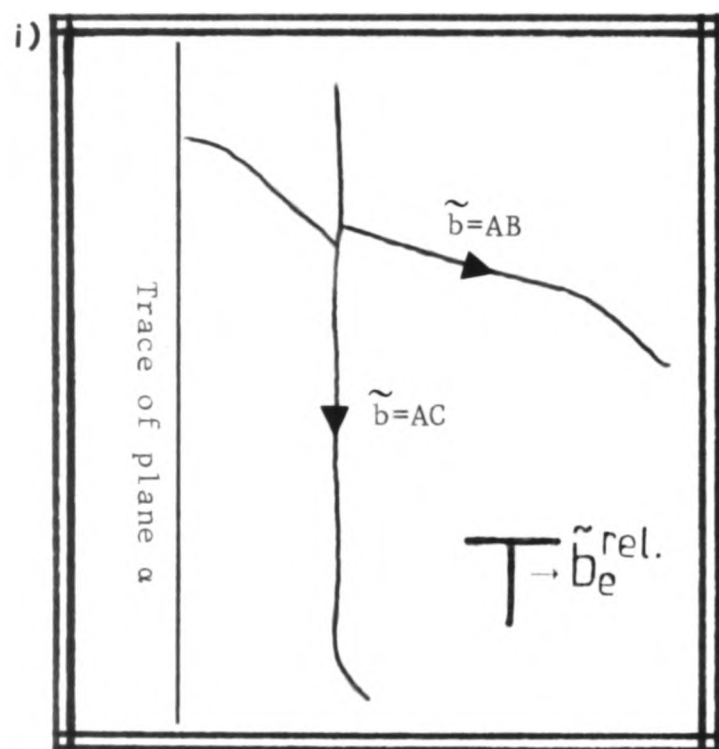
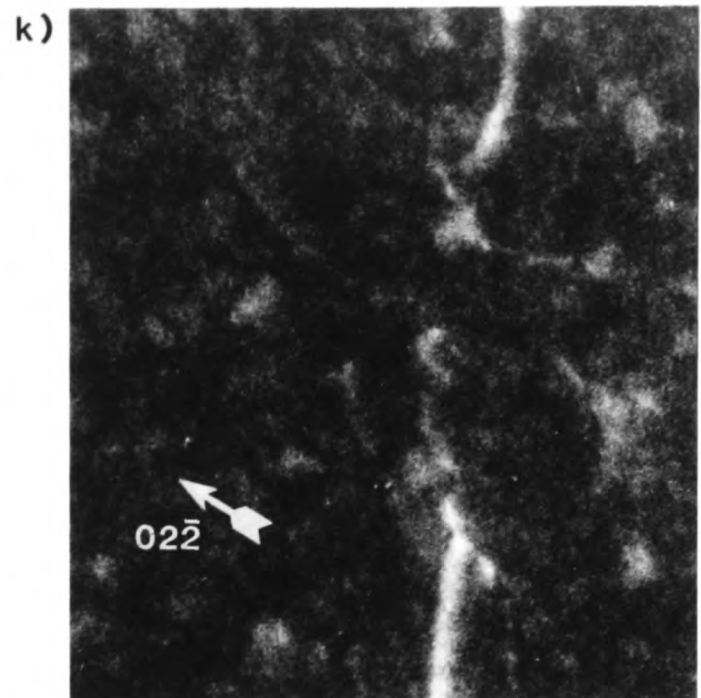
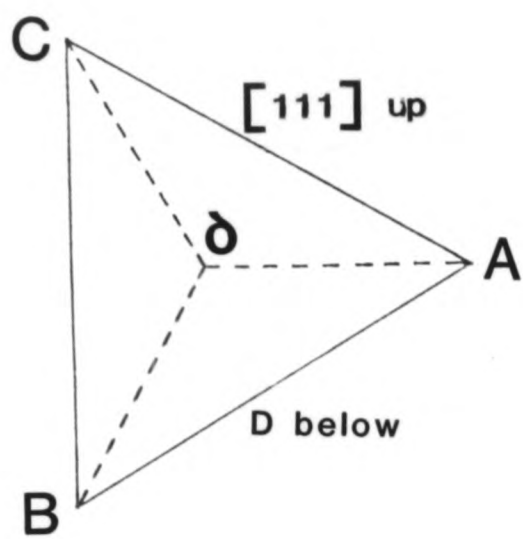
In the third page of the figure; drawing (i) shows the approximate positions of dislocations $\vec{AB}$

Figure 4.7
(4 pages)



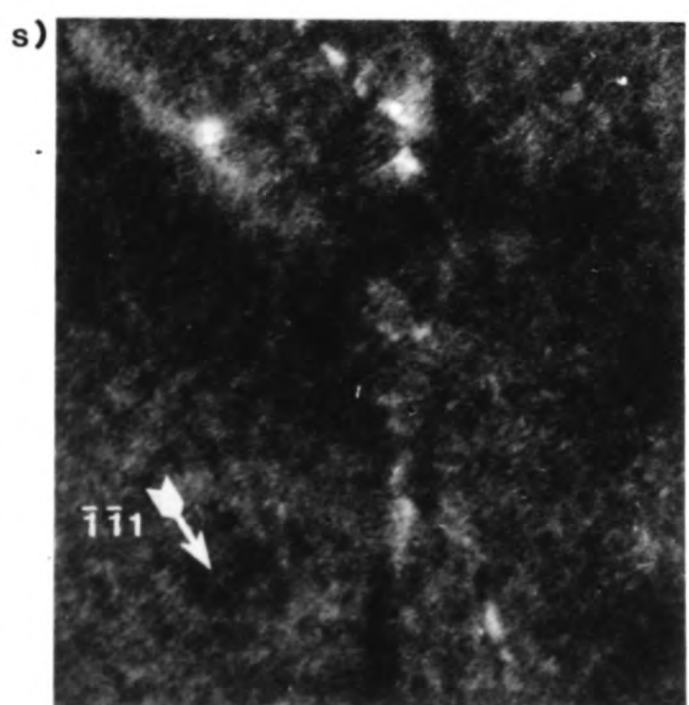
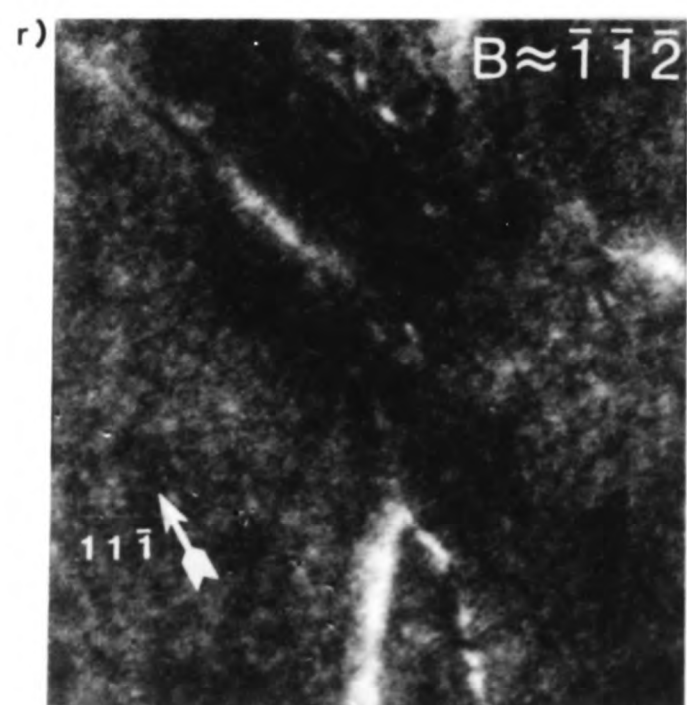
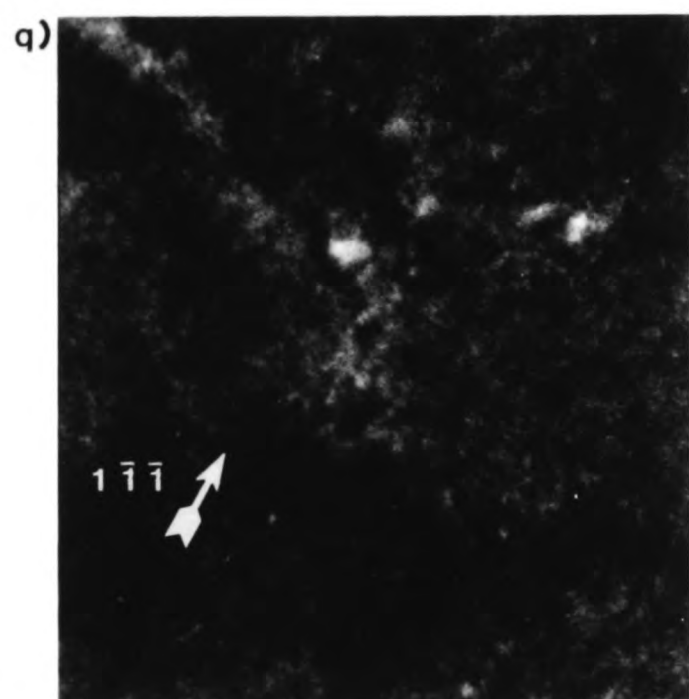
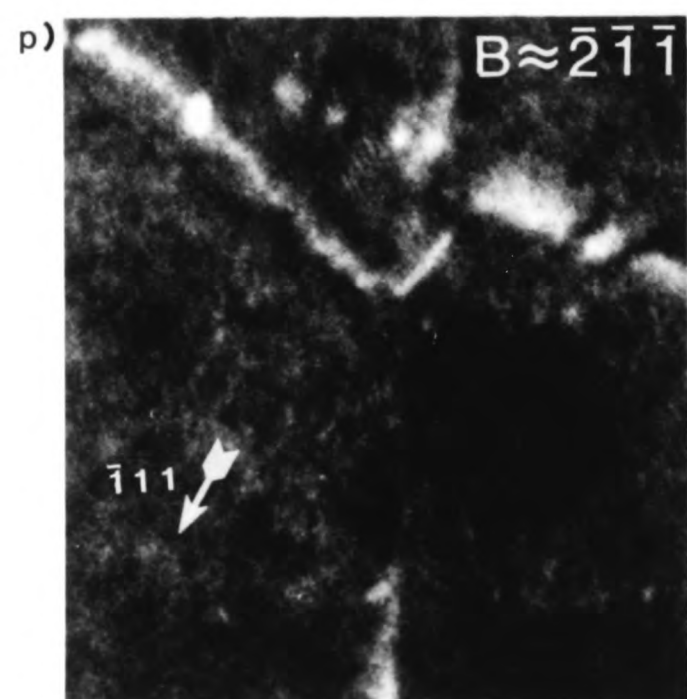
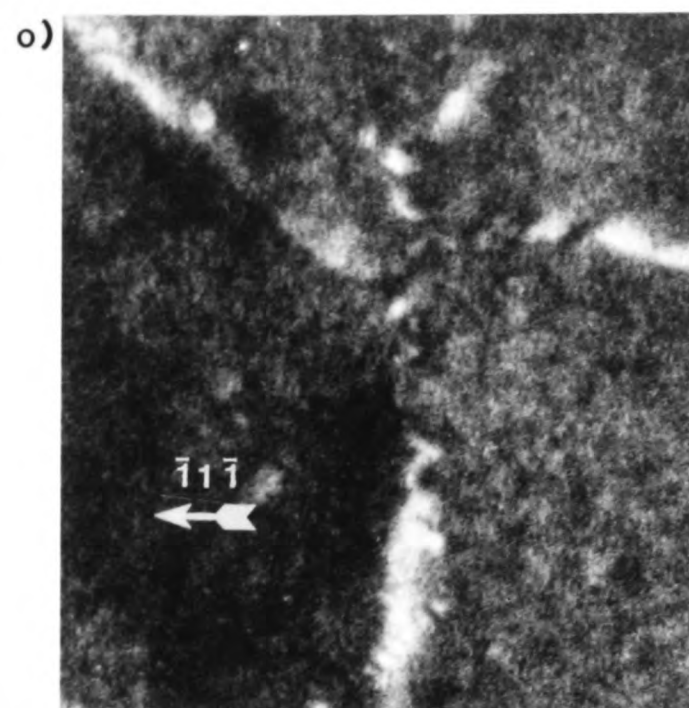
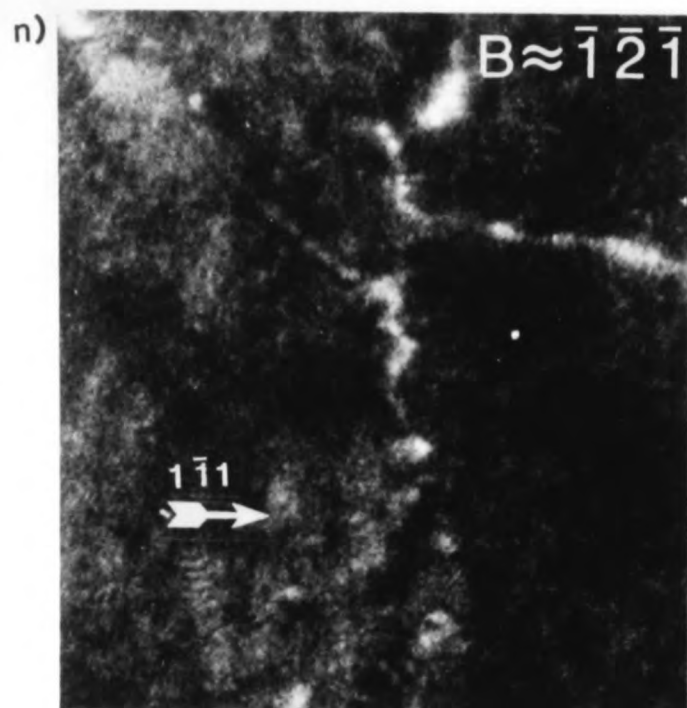
Dislocation node in Fe-25%Ni-17%Cr (at.%)
Before Irradiation





Dislocation node in Fe-25%Ni-17%Cr (at.%)

0.11 dpa at $\sim 430^\circ\text{C}$



Dislocation node in Fe-25%Ni-17%Cr (at.%)

0.11 dpa at $\sim 430^\circ\text{C}$

and $\overrightarrow{AC}$, from both of which the extra half-plane extends downwards, whilst micrograph (j) shows, at low magnification, the entire irradiated area and the area enlarged in the rest of the figure.

Observations: Post-Irradiation;

Micrographs (k)-(s) on the third and fourth pages of the figure are taken after irradiation to 0.11 dpa at approximately 430°C. In micrograph (k) dislocation $\overrightarrow{AD}$ is visible. This dislocation shows the fine scale zig-zag structure, or cusping, characteristic of sessile Lomer dislocations. This cusping is best shown by micrographs (n) and (o), which are taken with planes α vertical and appears identical to that seen along dislocations $\overrightarrow{AD}$ of figures 4.5 and 3.6. The spacing between cusps, at ~ 5 nm., is also very similar to these examples. Cusps appear to lie to the left, separated by sharp points which lie towards the compressional side of the dislocation strain field.

In micrograph (l), $|\tilde{g} \cdot \tilde{b}_T| = 2$ for dislocation $\overrightarrow{AB}$. Before irradiation the left hand side of this dislocation was very widely dissociated, however the partial separation appears to have narrowed after irradiation such that individual partials can no longer separately be resolved. This narrowing has apparently occurred by movement of the upper (edge) partial $\overrightarrow{\delta B}$ such that the total dislocation $\overrightarrow{AB}$ now lies close to the initial position of partial $\overrightarrow{A\delta}$.

In micrograph (o) there would appear to be a very faintly visible zig-zag line connecting the two sides of dislocation $\overrightarrow{AB}$ (best seen by studying the print from an angle). This is similar in form to another very faintly visible zig-zag line running somewhere between points 'B' and 'C' in micrograph (q). These zig-zag structures appear very similar to those seen to decorate sessile dislocations.

Micrographs (r) and (s) highlight the complexity of the post-irradiation configuration. Whilst dislocations are observed within the node region in other reflections, these micrographs show no contrast arising from this area. It would seem unlikely that no dislocation within the core show any contrast in $\tilde{g} = \pm [11\bar{1}]$ and it is thought that the apparent lack of contrast is instead due to complex strain-field interactions from a maze of short dislocation sections.

Interpretation:

The first point to be made, from an overall view of the post-irradiation micrographs, is the apparent extreme complexity of the post-irradiation configuration. It is believed that a very large number of new dislocation lines have formed within a very small region. Strain fields associated with these lines overlap, giving rise to artificially strong, or weak image contrast, sometimes to the extent where the entire region sometimes appears dislocation-free (micrograph (r) for example). It has, therefore, not proved possible to develop a post-irradiation configuration, akin to that proposed before

irradiation. Instead certain interesting features are highlighted and some more general observations, concerning the expected behaviour of dislocation nodes under irradiation, are made both here and within the discussion.

Dislocation $\vec{AD}$ is behaving in a way entirely consistent with the irradiated sessile dislocations of figures 3.6 and 4.5; yet again the minimum spacing between cusps is ~ 5 nm., possibly suggesting some latent similarity between these dislocations, and the cusps lie towards the dilatational strain field in a sense consistent with interstitial climb. It is supposed, therefore, that interstitial climb is responsible for the cusping in common with previous examples. Hence, it would appear that fine-scale zig-zag formation is a recognizable characteristic of the interstitial climb of Lomer dislocations. This strongly suggests that the faint zig-zags, which also have a spacing of ~ 5 nm., result from the climb of sessile dislocations.

At this stage ^{the} appendix should be consulted. This details a proposed three-dimensional pre-irradiation configuration, and mechanism for its formation, via the contraction of two double extended super-jogs when intersected by the pure-screw dislocation $\vec{CD}(\beta \rightarrow \alpha)$. In brief, however, the essential points raised by this treatment are first; that the entire configuration may have been generated principally by the operation of Lomer-Cottrell (Cottrell, 1952) type mechanisms, with some restrictions as to initial dislocation positions, and second; that inherent in the contraction is the generation of very many favourable sites (the number has been minimized in the particular mechanism shown) for interstitial precipitation. In the anti-penultimate diagram of the series (number 8) the potential for the precipitation at these sites of interstitials as Frank loops is suggested, using the criteria, suggested by figure 4.6, that such loops should align parallel with the parent dislocation.

Such a mechanism provides a ready explanation for the extreme complexity of the node core after irradiation, via the unfauling and intersection of these loops. As more interstitials are precipitated, and this may occur either by direct nucleation or via the build-up of pipe diffused interstitials, a very dense network of characteristically zig-zagged Lomer dislocations will evolve by both climb and loop glide processes.

The glissile dislocations $\vec{AB}(\delta_1)$ and $\vec{AC}(\delta_2)$ lie in the plane of the foil. HVEM generated point-defects are, hence, more likely to be trapped at these dislocations than at the inclined screw dislocation $\vec{CD}(\alpha)$. In figure 4.3, vacancies are seen to form three dimensional clusters close to dislocation $\vec{AC}(\delta_2)$ whilst most constrictions disappear; vacancies also cluster near dislocation $\vec{AB}(\delta_1)$ which reacts similarly to the irradiation. Mechanisms similar to those previously outlined therefore also seem to be operating here. Therefore, if interstitials are pipe diffusing along these dislocations, after the

annihilation of constrictions, they will migrate both towards the surfaces and towards the node.

It can be seen from the pre-irradiation configuration that pipe diffused interstitials, arriving from the left along the glissile dislocation $\overrightarrow{AB}(\delta_1)$ and along dislocation $\overrightarrow{AC}(\delta_2)$ would be funnelled into the core of the node. Some of these interstitials may transfer to the steeply inclined dislocations $\overrightarrow{BD}(\alpha)$ and $\overrightarrow{CD}(\alpha)$ and thence be lost at the surfaces. Otherwise their exits from the node core, via pipe diffusion, appear blocked by super-jogs and sessile dislocations.

Evidence of interstitial climb along the Lomer dislocation $\overrightarrow{AD}$, and cusped lines consistent with new climbing sessile dislocations, suggests that the transfer of interstitials to, and possibly also their diffusion along, the inclined screw dislocation $\overrightarrow{CD}$ is not a particularly efficient process. This may lead to a locally higher interstitial super-saturation level than normally found along smooth, dissociated glissile dislocations in these specimens. However, the evidence for interstitial climb also points to a lower interstitial super-saturation level here than in the bulk (interstitial loops are seen to nucleate in the bulk), since this region is acting as a sink for interstitials.

One possibility is that regions within the node are behaving in a similar fashion to the dislocation of figure 4.6, leading to the precipitation of pipe diffused interstitials as Frank loops when a certain threshold super-saturation level is attained. Examination of the proposed pre-irradiation configuration, employing the criterion that a parent dislocation may not be rotated to accommodate precipitated Frank loops, provides a very great number of possible sites for these loops (see diagram A.8). Subsequent climb, glide, rotation and intersection of these loops may then lead to the maze of dislocations suggested above.

Also note that the proposed pre-irradiation configuration shown does not immediately produce sites for the precipitation of interstitials as $\tilde{b}_L = \gamma\overrightarrow{C}$ loops; perhaps this may explain the lack of contrast from the node core in $\tilde{g} = \pm [11\bar{1}]$. It should be noted, however, that if the configuration of diagram 8 is developed further any number of such sites may be generated. Finally, consideration of triple and quadruple extended super-jogs, using exactly the same procedures as applied in appendix A, leads to an even greater variety of these sites, and correspondingly greater scope for interstitial precipitation.

In the absence of greater experimental evidence it is not possible to be certain about the precise initial or final configurations. However, without the precipitation of point-defects within the node region, which defects seem likely to be interstitial in nature in the case of dislocation $\overrightarrow{AD}$, it is very hard to see any explanation for the formation of new zig-zagged dislocations or for the complexity of the contrast. It therefore appears as if this dislocation node is acting as a preferential site for interstitial precipitation, probably by reason of its complex configuration. These interstitials probably arrive

principally via pipe diffusion along neighbouring glissile dislocations. Bulk diffusion directly to the core will also contribute but is not expected to be a large effect since the core covers only a small area of the specimen.

Points of Discussion

- *The Formation of Discrete Lines of Vacancy Clusters.*
- *The Orientation Dependence of Pipe Diffusion.*
- *Motion of a near-screw dislocation to one side only of its initial position.*

The Formation of Discrete Lines of Vacancy Clusters

The apparent preference shown by dislocations to lie along particular crystallographic directions, notably $\langle 123 \rangle$'s, may lead to an explanation for the formation not only of *discrete* lines of clusters seen in figure 4.2 but also of the observation that even towards the low dose end of the zig-zag of figure 4.1, comparable segments appear parallel. We have seen that, for near-screw dislocations, zig-zagging of the line may be explained by the lateral climb of jogs. It is uncertain whether these jogs are climbing via the absorption of single interstitials or via the assimilation of interstitial clusters. It seems likely, however, whatever the mechanism, that the lateral climb of jogs will be a 'gradual' process. In effect, therefore, climbing jogs exert a gradually increasing torque on neighbouring line sections, prompting their glide.

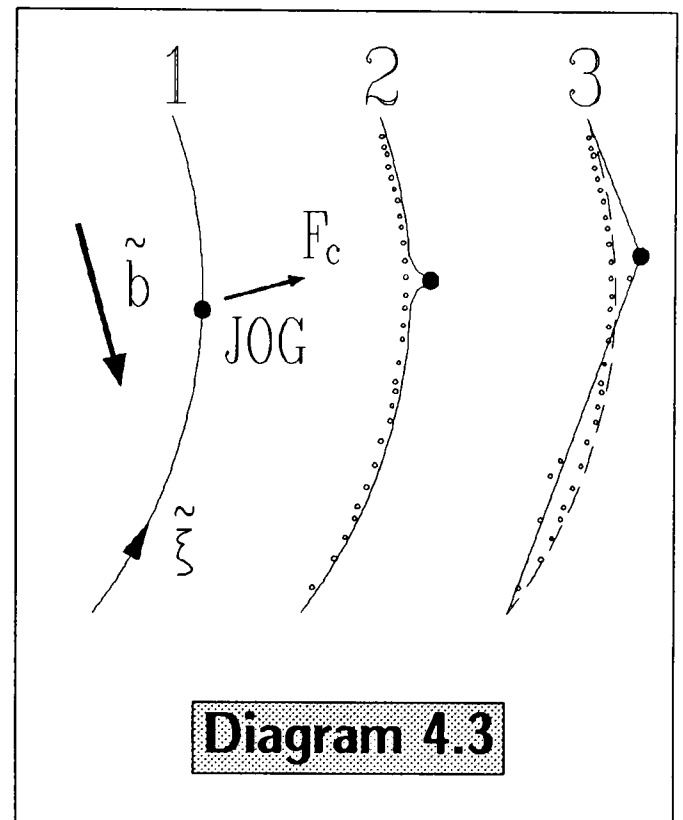
The formation of a discrete line of clusters suggests, however, that a force exists to resist the glide of a dislocation section away from its original orientation. After some time, during which clusters have nucleated and grown within the strain field of the dislocation at its initial position, this force is *suddenly* overcome; the dislocation then glides *very quickly* to its next orientation (which is often the final orientation in these low dose experiments), allowing insufficient time for cluster formation at intermediate positions.

The *preferential* nucleation of vacancy clusters near a dislocation must relax the strain field of the dislocation core; this will exert a force to resist dislocation glide away from its original position since core energies must increase when such glide occurs. If, however, the jog has climbed sufficiently such that, after gliding, the dislocation may lie along a $\langle 123 \rangle$ direction, it is possible that the corresponding reduction in core energy may be sufficient to negate any increase required to dislodge the dislocation from its original position.

For the shorter dislocation of figure 4.2, small clusters now appear to be forming at the new dis-

location position (see micrograph (l)). This has obviously occurred late in the irradiation. Alignment, and straightening, along $[1\bar{3}2]$ has resulted in the dislocation crossing over the line of clusters which were pinning it at its original position. This is sketched schematically in diagram 4.3. The new position of the dislocation is likely to be particularly stable against further glide, since inherently low core energies, owing to the orientation of the dislocation, are further lowered by the formation of new clusters. There does appear to be a materials dependence in the relative stability of certain crystallographic directions. Consideration of any differences between materials is left to chapter 8.

Finally, the observation that the very small zig-zags in figure 4.1 lie parallel to one another is consistent with the suggestion that these zig-zags are not formed *unless* the dislocation may lie along a low index direction. This situation is less clear here, however, since ‘pinning’ vacancy clusters are not evident although, in this case, line tension, which is increased by the formation of a zig-zag, will have the same effect. An increase in line tension is clearly not the origin of the discrete line of clusters near the shorter dislocation of figure 4.2 since the line length of the dislocation is *reduced* after irradiation (see micrographs (a) and (b)).



The Orientation Dependence of Pipe Diffusion

Many experimental observations in this volume may be explained, either wholly or in part, by postulating an orientation dependence of dislocation pipe diffusion. Such a dependence has previously been observed in NaCl and for dissociated dislocations in Ni (Canon & Stark, 1969). In both these cases, and others (see the review by Atkinson & LeClaire, 1984) pipe diffusion is found to be slower along screw than along edge dislocations. Canon and Stark hypothesize that this is simply due to the lower binding energy of a vacancy with a screw, rather than an edge, dislocation. Such a mechanism would appear equally applicable to interstitials, however.

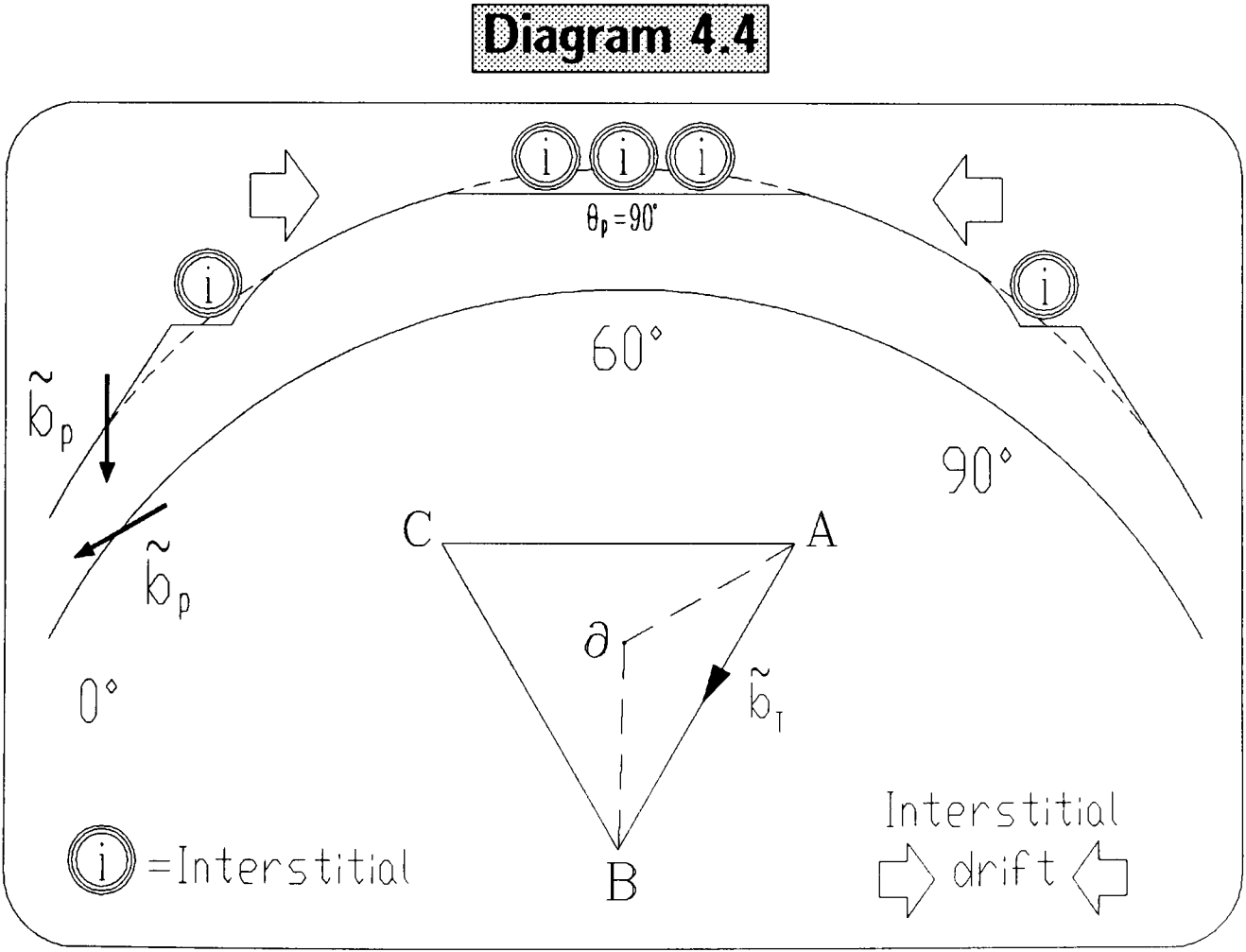
Hirth & Lothe (p.569) consider a point-defect diffusing along a dislocation as an “incipient double jog”. Whilst the sides of this jog are always in edge orientation, the character of the ‘top’ of the jog (as drawn in their diagram 15-23) is dependent on the orientation of the nearby dislocation. Under the influence of any local super-saturation the ‘top’ of a ‘jog’ (i.e. a point-defect) will endeavour to

twist towards edge orientation. If the line itself is not in pure-edge orientation this action will be resisted by line tension.

In effect, therefore, the diffusing point-defect may lower its interaction energy, with both the local super-saturation and with the dislocation carrier, by migrating towards regions of the line where it may lie in pure edge orientation. This will result in a preferential *drift* of defects. It also suggests an explanation for the hypothesis that defects have a lower binding energy with screw than with edge dislocations.

For the case of a dissociated dislocation, it is reasonable to assume that the partial nearest edge in character, which will have the greatest interaction with the irradiation induced super-saturation, is dominant. Defects¹⁷ are therefore expected to migrate towards regions where the total dislocation is in the 60° orientation and one partial is pure-edge in character.

Diagram 4.4 represents this mechanism schematically, for the case of interstitials diffusing along a curved, dissociated dislocation. Individual interstitials are represented as twisting a short section of the partial towards edge orientation as they migrate towards $\beta = 60^\circ$. This mechanism should be equally applicable to vacancy diffusion.



As is shown in diagram 4.4, such a mechanism will result in a build-up of defects along 60° sections of the dislocation. In the case of the seemingly linked interstitial and vacancy defects ‘B’ of

¹⁷If defects migrate as clusters, effectively forming small Frank loop nuclei, it follows more directly that these clusters should migrate to sections of a dislocation of precisely 60° character.

figure 4.6, it is supposed that the super-saturation reaches a level sufficient for the precipitation of interstitials and vacancies into visible clusters before back-diffusion becomes significant. These clusters will lie initially parallel to the dislocation, on close-packed $\{111\}$ planes, to minimize their interaction energies with both the locally high super-saturation and the line. Hence, the Burgers vectors of these loops are expected to be dependent primarily on the orientation of the parent dislocation rather than its Burgers vector.

Other observations of the formation of relatively large clusters near to dislocations at precisely 60° orientation, such as the uncharacterized clusters of figures 3.1 and 3.2 seem consistent with this mechanism. These clusters are considered further in chapter 8.

Motion of a near-screw dislocation to one side only of its initial position

The preferential drift of defects may also shed light on an aspect of the post-irradiation configuration of figure 4.2, micrographs (g) to (i). In this figure it appears as if the dislocation has moved away from its original position (as indicated by a line of vacancy defects) to one side only. This is in apparent disagreement with the basic jog climb mechanism proposed so far, under the operation of which a dislocation should be displaced to either side of its original position by the climb of both sides of a double jog (see figure 3.7). One possible explanation is that jogs along this dislocation are all of the same sign. This possibility cannot be excluded, but seems unlikely since the presence of double jogs is consistent with many other experimental observations. Double jogs should also be the result of a purely thermal generation mechanism (Hirth & Lothe, p. 495).

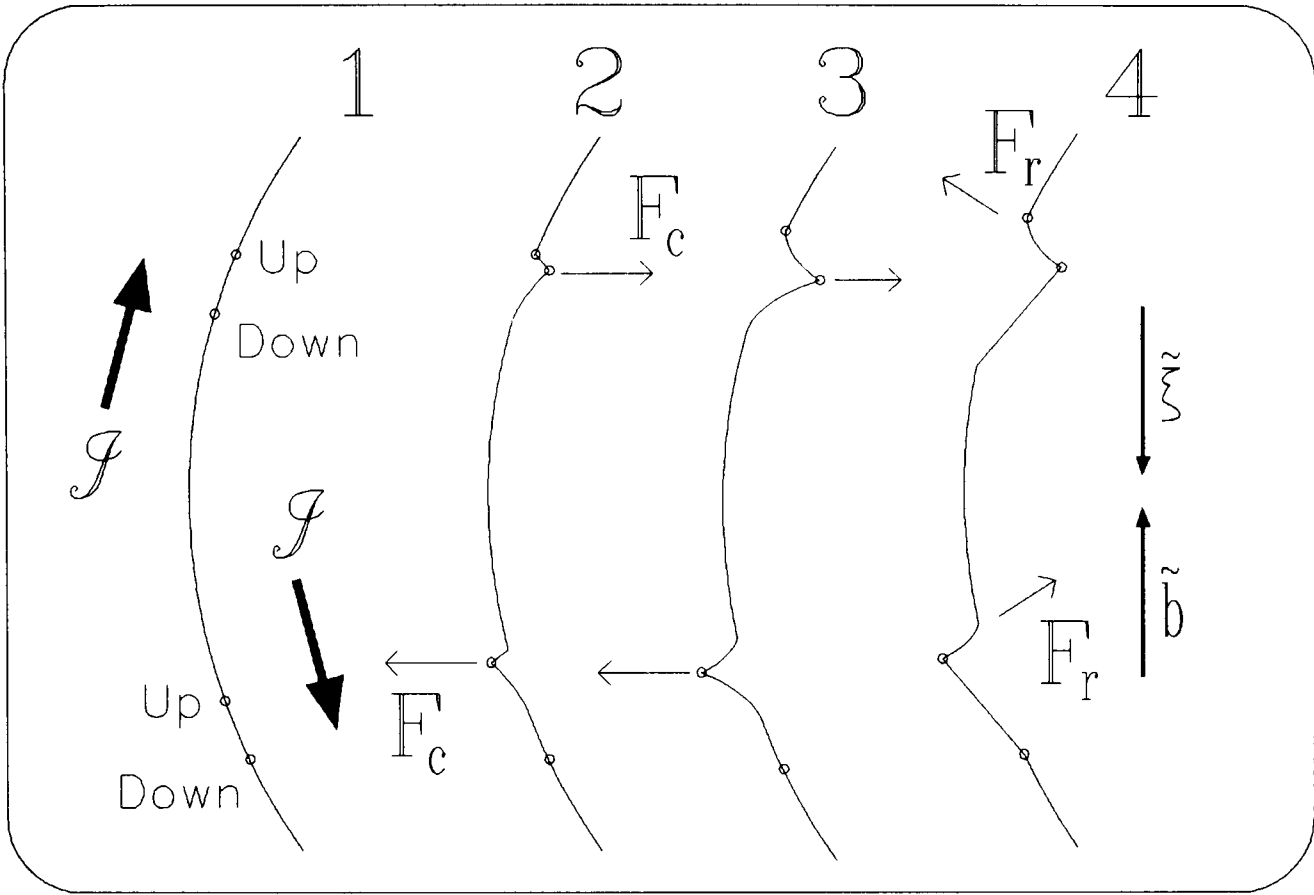
An alternative, and highly speculative, explanation is provided below, in which only one side of a double jog is climbing: Notice that the dislocation of figure 4.2 is curved; passing through screw orientation. As depicted in diagram 4.4, pipe diffusing defects, which are likely to be interstitial, will therefore drift away from the central screw section in both directions along the dislocation. If jogs are efficient assimilators of interstitials (and evidence presented here seems to suggest that they are), jogs nearest the screw section will therefore receive a greater flux of defects¹⁸.

This is represented schematically in diagram 4.5. Numbers '1' and '4' refer to initial and

¹⁸We neglect here the secondary effect of the increased hydrostatic strain field for nearer edge sections the other side of a double-jog. This will slightly reduce the net imbalance of defects absorbed at each side of a double-jog via greater bulk diffusion of defects to these sections. For the configuration shown in figure 4.2, however, sections either side of jogs (e.g. jogs 'A' and 'F') are at very similar, near screw, orientations; a bulk diffusion imbalance may be only a small effect, therefore. This factor must, however, be taken into consideration in any attempt to quantify uneven climb rates exhibited by jogs as a function of dislocation configuration.

‘final’ configurations respectively. In ‘1’, ‘Up’ and ‘Down’ refer to the senses of individual jogs whilst “ $\mathcal{J}$ ” refers to the preferential drift of interstitials described above. “ F_c ” refers to the climb force, which acts in the direction indicated, experienced by the climbing jog. In the final stage (‘4’), “ F_r ”, a force resisting further climb of the jog, arises from a combination of the effects of line tension, core energies and any new vacancy cluster nucleation as outlined in the first section above.

Diagram 4.5



The particular arrangement of pre-existing jogs (i.e. the senses of ‘Up’ and ‘Down’ jogs) shown in this diagram is purely speculative, but is suggested by the direction of motion of the dislocation of figure 4.2. Essentially it is unimportant, however, since it can easily be shown that very similar shapes will evolve via this mechanism from any pre-irradiation configuration.

Summary

Observations:

The following phenomena are shown by irradiated dislocations in this alloy:

- Two dimensional zig-zag configuration (no connecting segments); ... 1 ...
- ‘Flat-helix’, three dimensional zig-zagging; ... 2 ...
- Straight dislocation sections along <123> or <112> directions; ... 3 ...
- Kink formation; ... 4 ...

- Vacancy cluster/SFT formation; as discrete line at pre-irradiation position (5a); ... 5 ...
- Removal of constrictions; ... 6 ...
- Fine-scale cusping; ... 7 ...
- Interstitial precipitation at 60° orientation; unfaulting of interstitial Frank loop; ... 8 ...
- Narrowing of partial separations?; ... 9 ...

As shown in the following figures: (Table 4.1)

Figure	Character	Dose (dpa)	Temperature (°C ± 50°)	Observations (see above)
4.1	~ 0°	0.16	430	2, 3;
	0°-20°	< 0.11	430	1-2, 3, 4;
4.2	0°-40°	0.1	430	3, 4, 5a, 9;
4.3	60°	0.11	430	5, 6, 9;
4.4	65°	0.12	430	4, 5, 5a;
		0.11	430	5a, 6, 9;
4.5	60°- 90°	0.11	430	5, 7;
	90°(sessile)	0.11	430	
4.6	60°	0.12	430	4, 5, 5a, 8, 9;
4.7	NODE	0.11	430	7, (8), 9 and as figure 4.3;

The narrowing of partial separations is not as evident in this alloy as in the 15%Ni pure ternary. For a 60°, evenly dissociated, dislocation the partial image separation prior to irradiation was ~ 40Å. This may have narrowed to ~ 30-35Å after irradiation although neither value is determined with surety.

Apart from the new kinks formed towards the middle of an extremely long dislocation, the only other kinking was associated with the alignment along <123> or <112> directions of near-screw dislocations. For the near-screw dislocations, kinking is likely to be associated with the formation of a zig-zag. One kink on an exactly 60° segment of the extremely long dislocation of figure 4.6 appears associated with point-defect precipitation. Also apart from figure 4.6, almost all constrictions disappeared along dislocations of ≥ 30° in character.

Burgers vectors were never seen to be altered after any irradiation.

Small clusters, often identified as SFT and always lying towards the compressional strain field side of the dislocation, were seen close to all dislocations of ≥ 20° in character. Where measurements can be made, these clusters apparently lie ~ 15-20 nm. from the dislocation.

All sessile Lomer dislocations exhibited fine-scale cusping. Cusps are typically ~ 5 nm. across

and always extend into the dilatational strain field of the dislocation. In the final figure (4.7) new finely-cusped lines, their cusps being ~ 5 nm. across, are believed to arise from new sessile dislocations.

Bulk interstitial loops are seen, but are fairly rare in these irradiations. Specimens were at temperature in the HVEM for times of 8 (figures 2, 4 and 6) and 9 (figures 1, 3, 5 and 7) minutes. Many small background loops were seen to have formed, after only ~ 0.03 dpa, in another specimen which was at temperature for several hours (not shown).



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Chapter 5

Irradiation of Fe-40%Ni-17%Cr alloy

To study the interaction of HVEM generated point-defects with dislocations in a higher SFE material, a single crystal of an Fe-40%Ni-17%Cr alloy was ordered. A year later, shortly prior to visiting Argonne in 1988, a polycrystalline cast was received. As a result it did not prove possible either to finely control specimen orientation or to carry out as many experiments as desired in this material.

Some preliminary experiments, on near- $\{100\}$ foils, were conducted during the Argonne visit. These experiments were employed to give an overview of behaviour in this material; a full analysis not being undertaken. Further experiments were conducted in the Oxford HVEM, with foils as close to $\{111\}$ as possible. Many difficulties were encountered in the operation of the Oxford instrument.

This chapter is therefore broadly separated into two sections, consisting of Argonne and Oxford irradiations. The behaviour of dislocations irradiated in both microscopes is found to be mutually consistent although differences in background loop formation are very evident. Comment is made on the comparability of Oxford and Argonne irradiations.

Argonne Irradiations:

Figures 5.1^{a)} & 5.2^{b)}; Dislocations in Fe-40%Ni-17%Cr; 0.04^{a)} & 0.06^{b)} dpa at $\sim 400^\circ\text{C}$.

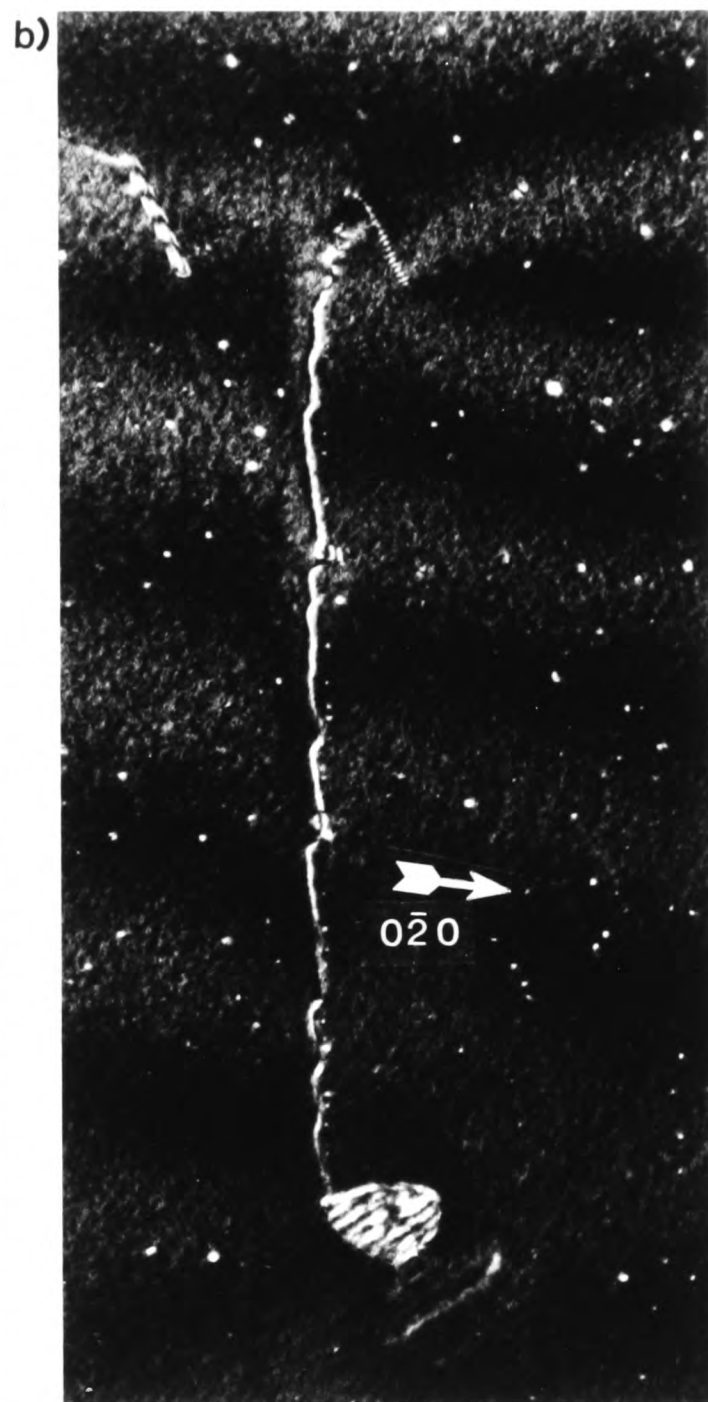
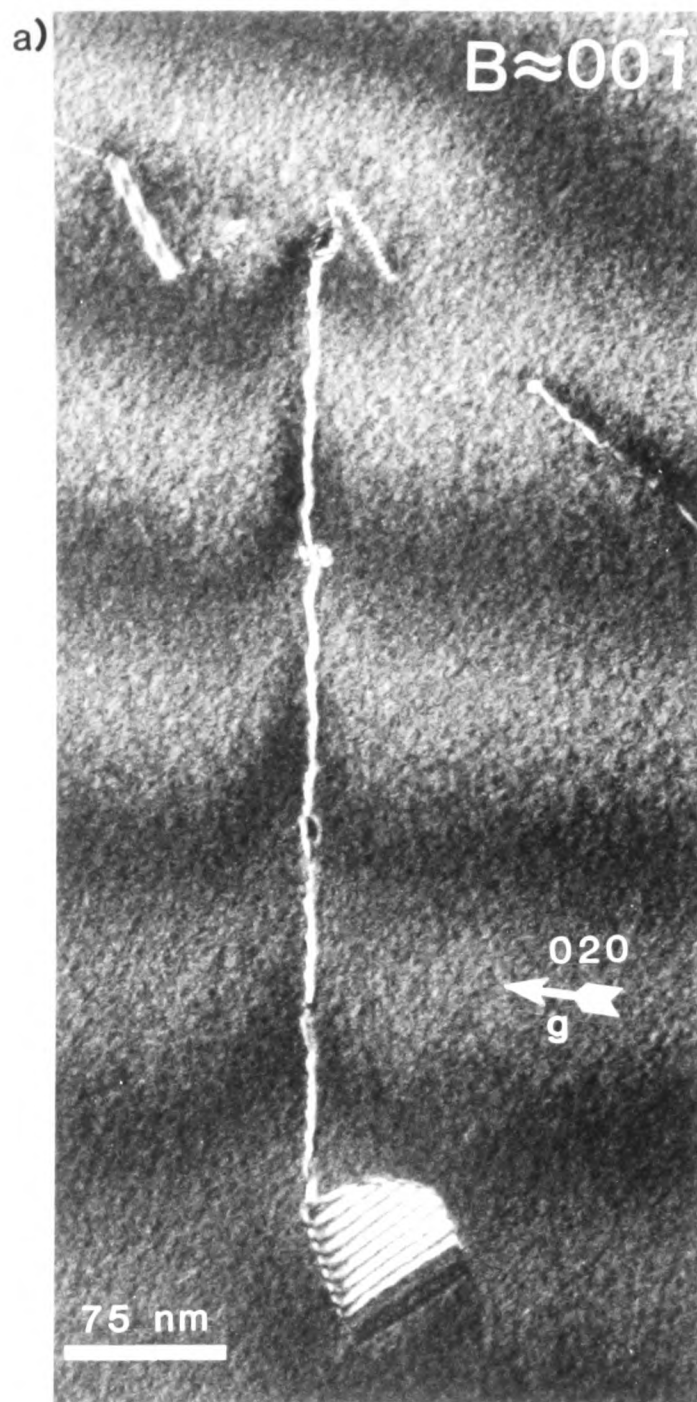
Figures 5.1 and 5.2 show dislocations which were irradiated in the Argonne HVEM. The specimen orientation is close to $\{001\}$. Micrographs taken in the 8 reflections ($\pm \tilde{g}$ of the 4 nearest spots) around this $\{001\}$ pole do not always enable unique Burgers vector characterization but are sufficient to give a general overview.

Observations:

In figure 5.1, a dislocation is shown before irradiation in micrograph (a). The dislocation is apparently heavily jogged but continuous and relatively straight. This dislocation exhibited $\tilde{g} \cdot \tilde{b} = 0$ (stacking-fault) contrast in $\tilde{g} = \pm [200]$ (not shown) suggesting that it is dissociated and has a Burgers vector of $\vec{AC}$ or $\vec{DB}$. The configuration of this dislocation is discussed below. Several large faulted regions, such as is shown at the lower end of this dislocation, were seen throughout this specimen prior to irradiation.

After irradiation ((b)) a number of cusps have formed, where the line has apparently moved to one side away from a line of cluster damage close to its original position. The general appearance of this dislocation is similar to irradiated sessile dislocations in other materials, except that the spacing between cusps, which varies up to 50 nm., is roughly an order of magnitude greater. A low density of

Figure 5.1



Dislocation in Fe-40%Ni-17%Cr (at.%)

0.04 dpa at $\sim 400^\circ\text{C}$

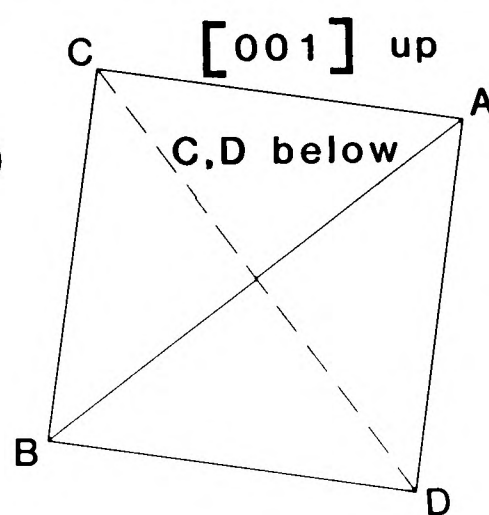
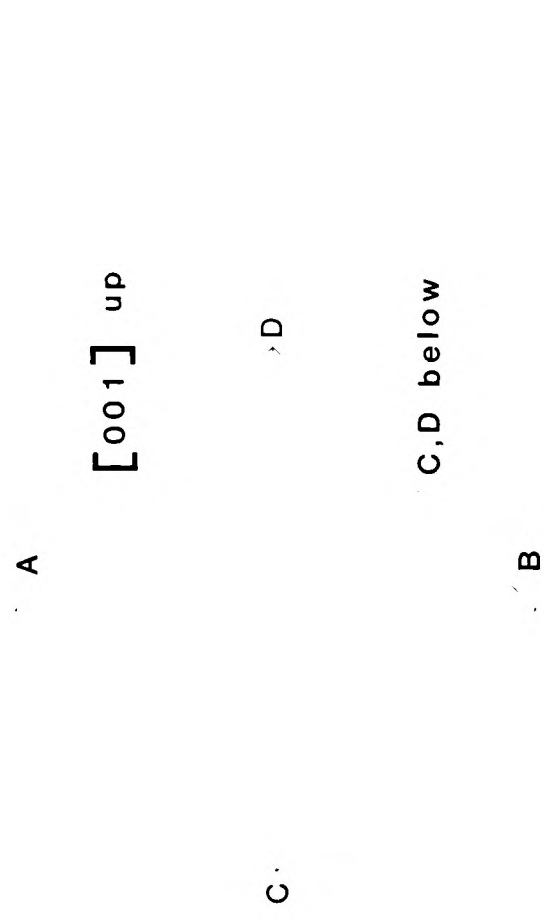
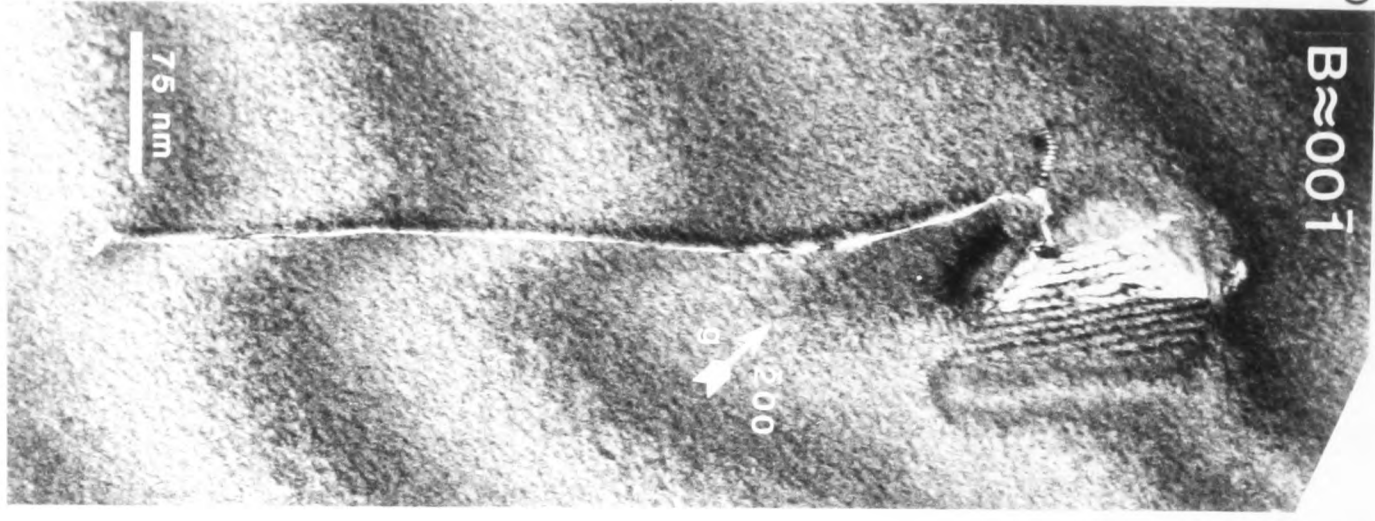
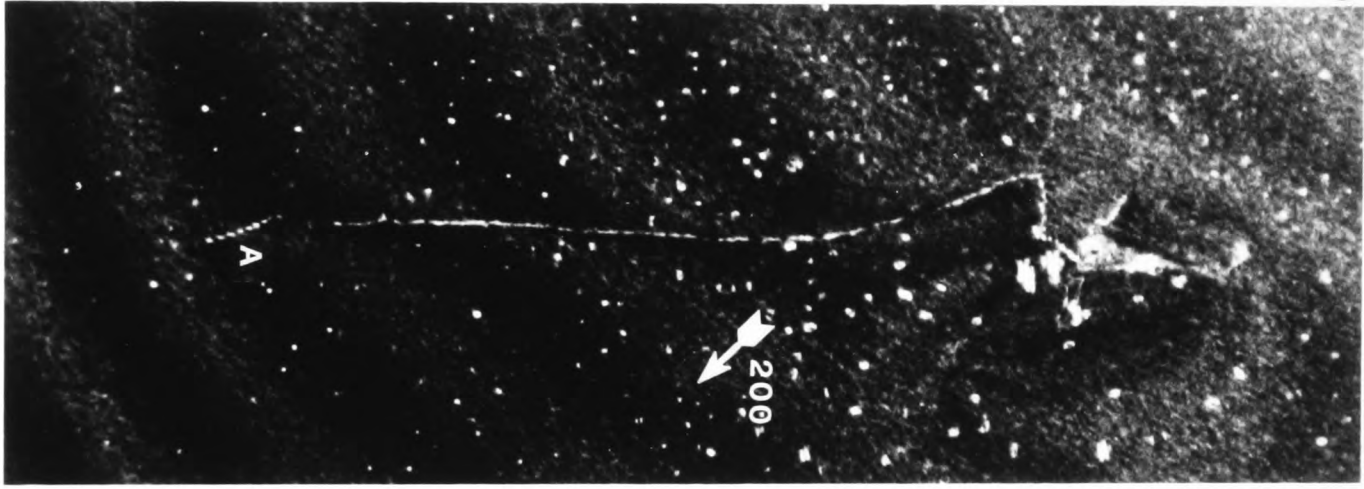
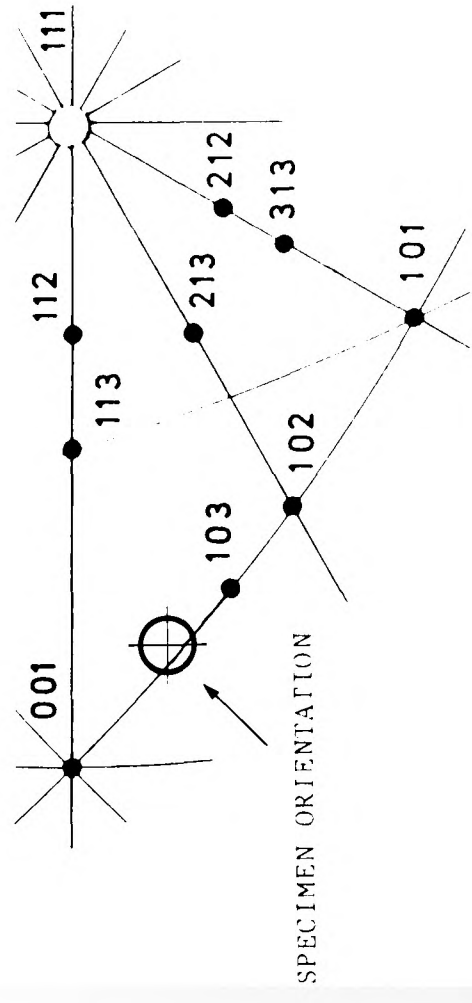


Figure 5.2



Dislocation in Fe-40%Ni-17%Cr (at.%)
0.06 dpa at ~400°C



perfect dislocation loops is seen in the bulk. The nature of these loops cannot be determined from the incomplete contrast series taken. Finally, the large faulted region at the base of the dislocation has shrunk after irradiation.

A similar large faulted region is also observed to have shrunk after irradiation in figure 5.2. Before irradiation ((a)) this dislocation exhibited $|\tilde{g} \cdot \tilde{b}_T| = 2$ contrast in $\tilde{g} = \pm [\bar{2}20]$ and $\tilde{g} \cdot \tilde{b}_T = 0$ contrast in $\tilde{g} = \pm [\bar{2}\bar{2}0]$ (not shown) consistent with Burgers vector $\tilde{b}_T = \vec{AB}$. It is therefore close to pure-screw orientation.

After irradiation (micrograph (b)) a high density of small background loops is evident. Very few of these loops are seen close to the large faulted region and there also appears to be a loop depleted zone (LDZ) near to the dislocation. No line of clusters close to the pre-irradiation dislocation position is seen in any reflection. The dislocation has moved slightly to the right.

Finally it should be noted that background loops formed away from the dislocation after the 0.06 dpa irradiation of figure 5.2 are significantly larger, and greater in number, than those formed after the 0.04 dpa irradiation of figure 5.1.

Interpretation: Pre-irradiation Configuration:

The dislocation of figure 5.1 appears continuous in all reflections and is ~ 450 nm. long in projection. Since the foil is only ~ 100 nm. thick here, the dislocation cannot be inclined at greater than $\sim 12.5^\circ$, averaged over its length, to the foil surfaces. Shown at lower right in figure 5.2 is a schematic diagram giving the approximate foil surface orientation. Of the four $\{111\}$ glide planes, the (111) plane, inclined at $\sim 45^\circ$ and on which a dislocation $\vec{AC}$ would be glissile, lies nearest to the surface. Hence, this dislocation must consist of either short 60° glissile segments, separated by large jogs of the same sign, or be completely sessile.

The situation for the dislocation of figure 5.2 is different. This dislocation appears to lie parallel to its Burgers vector and is ~ 530 nm. long in projection. It may not, therefore, be inclined at greater than $\sim 10.5^\circ$ to the surfaces. Since the foil orientation makes an angle of only $\sim 7^\circ$ with the $[\bar{2}20]$ Kikuchi band, this dislocation may lie in pure-screw orientation and be free of jogs. The short inclined section at 'A', which may be a glissile mixed segment of dislocation $\vec{AB}$ on δ or γ , connects one end of the screw section to a foil surface.

Post-Irradiation:

The cusping exhibited by the dislocation of figure 5.1 may originate from either glide or climb of a $\{111\}$ habit plane dislocation, or climb of a sessile dislocation. The fourth possibility, of

$\langle 110 \rangle / \{100\}$ glide is excluded by noting that, with possible Burgers vectors of $\vec{AC}$ or $\vec{DB}$, this dislocation is not 'glissile' in the (001) plane of the foil.

In the case of a dislocation comprised of glissile segments, motion of the line away from the clusters may result from the lateral climb of jogs (see figure 3.5), simultaneous with the glide of intervening segments. Climb of the segments themselves cannot be excluded here, although ^{for the ternary alloys} glissile, 60° dissociated dislocations have not been observed to climb elsewhere in this study. Consideration of other irradiations in this volume suggests that the small clusters are more likely to be vacancy, than interstitial, in nature.

The possibility that we are seeing climb of a straight sessile dislocation seems unlikely for three reasons: First, previous observations of sessile dislocations have shown characteristic fine-scale cusping after irradiation (see, for example, figure 4.5). The period of these cusps was ~ 5 nm. Second, if the dislocation were, effectively, a straight line before irradiation, its line direction would be close to $[\bar{2}11]$ or $[\bar{1}\bar{2}1]$ (or their neighbouring $\langle 123 \rangle$ directions). Similar directions have been seen after, but never before, irradiation and it is hard to imagine how a sessile dislocation lying along these directions may be formed. Finally, with possible Burgers vectors of $\vec{AC}$ or $\vec{DB}$ the dislocation may not be dissociated; hence the apparent stacking fault contrast seen in " $\tilde{g} \cdot \tilde{b}_T = 0$ " images must be incorrect.

The screw dislocation of figure 5.2 will have little, or no hydrostatic strain field. The lower misfit vacancies are thus only very weakly attracted to the line and are not seen to have coalesced in sufficient numbers to form visible clusters. This is consistent with observations of other irradiated screw dislocations in this volume (e.g. figures 3.4 and 6.1). The dislocation is apparently still acting as a sink for point-defects, however, as evidenced by the formation of a LDZ nearby. The nature of individual loops seen in the above two figures cannot be characterized here. The absence of a zig-zag configuration, often seen after the irradiation of other screw dislocations, may be attributable to a lack of pre-existing jogs.

The behaviour of both these dislocations is discussed further at the end of the chapter.

Oxford Irradiations:

The Oxford HVEM has given much sterling service over the years, and was the irradiation source employed in many references in this volume. More recently, however, the extreme age of the instrument demands considerable patience, whilst keeping both beam and desired area on the screen simultaneously can be an exacting, and occasionally impossible, task.

In consequence, successful experiments were rare and it did not prove possible to determine the

irradiation conditions. Doses are, therefore, rough estimates based on Argonne HVEM data, which is an identical instrument. These doses may be over-estimates by up to a factor of ~ 2 , and under-estimates by up to a factor of 3. The mean value from which the dose is calculated is the observed Argonne mean dose rate ($10^{-3} \text{ dpa} \cdot \text{sec}^{-1}$), whilst the potential spread is also empirically deduced from dose rates seen with various filaments and bias settings in the Argonne instrument¹⁹.

Finally, extended high temperature conditioning of the heating stage made no apparent difference to the very rapid build-up of contamination. This contamination layer formed independently of irradiation. With the experimental difficulties outlined above, specimens were in the Oxford HVEM for a considerably longer time than was normal at Argonne. Two of the three experiments were aborted when contamination proved impenetrable. It is hoped, therefore, that the reader will excuse the correspondingly poor quality of post-irradiation (semi-)weak-beam micrographs. The Oxford HVEM hot-stage calibration is believed accurate to within $\sim 20^\circ\text{C}$.

The following two figures (5.3 and 5.4) are taken from an area irradiated for 33 seconds, which, at the Argonne mean dose rate, equates to 0.033 dpa.

Figure 5.3; 60° Dislocation in Fe-40%Ni-17%Cr; Irradiated at $\sim 400^\circ\text{C}$.

Observations:

A long 60° section of a dislocation is shown in this figure. The whole dislocation is shown at lower magnification in the pre-irradiation micrograph 5.3 (g). Micrograph (a), for which $|\tilde{\mathbf{g}} \cdot \tilde{\mathbf{b}}_T| = 2$, is taken before irradiation, whilst (b) to (f), and micrograph (h), are taken after irradiation. Before irradiation the dislocation contains few constrictions and appears very evenly dissociated. The partial image separation at 'S' (60° orientation) is $\sim 30 \text{ \AA}$.

After irradiation the shape of the dislocation is little altered. However, the line is more complex in appearance, being apparently decorated, particularly towards the left hand side which is near to the surface of the foil, and by feature 'G' on a 60° section. Individual partials are no longer separately resolved in the $|\tilde{\mathbf{g}} \cdot \tilde{\mathbf{b}}_T| = 2$ images (b) and (c), however, the presence of SFC in micrograph (e) suggests that most of the dislocation is still dissociated on δ .

Throughout the full contrast series feature 'G' exhibits similar contrast to loop 'A' which has Burgers vector $\tilde{\mathbf{b}}_L = \alpha \vec{\mathbf{A}}$ (interstitial) and is out-of-contrast in $\tilde{\mathbf{g}} = \pm [20\bar{2}]$ (not shown). In the case of feature 'G', however, self-consistent strong/weak contrast cannot be isolated and it is not possible to assign a unique Burgers vector direction by this method with surety. This confusing contrast may

¹⁹For the Argonne irradiations it was endeavoured to keep the dose rate constant. Specimens were actually irradiated at dose rates in the range $(0.6 - 1.3) \times 10^{-3} \text{ dpa} \cdot \text{sec}^{-1}$ (see chapter 2).

result from the proximity of feature 'G' and the line. In common with 'A', feature 'G' is visible in $\tilde{g}=[11\bar{1}]$, suggesting that it is faulted, and $\tilde{g}=\pm[\bar{3}11]$ (not shown). However, whilst 'A' is often seen as an oval shaped loop, feature 'G' always appears as a very straight line (e.g. micrograph (e)).

Two SFT (circled in (d) & (e) and shown at higher magnification in (f)) were seen to form close to the dislocation. These SFT move away from the line as the specimen is tilted from $\tilde{B}=[\bar{1}\bar{1}\bar{2}]$ to $\tilde{B}=[\bar{1}\bar{2}\bar{1}]$ suggesting that they lie to the compressional strain field side of the line. The two SFT may also be displaced laterally from the dislocation by a small amount. Notice that these two SFT appear fairly isolated; they do not form part of a continuous line of cluster damage as often seen lying parallel to 60° dislocations in other materials (see, for example, figures 4.3 to 4.6, 6.4, 6.7, 7.2 and 7.10).

The observation that feature 'G', and other small clusters such as 'H', also appear displaced from the dislocation by the same amount as the SFT in micrograph (d), suggests that they too are vacancy in nature. This result appears to be confirmed by micrograph (h), for which the foil has been tilted through $\sim 50^\circ$ from (111) to the $(\bar{1}33)$ pole²⁰: Feature 'G' is now displaced from the line image by an amount suggesting that it lies ~ 25 nm. directly below the dislocation, i.e. to its compressional strain field side. Clusters to the left-hand side of the dislocation are similarly displaced.

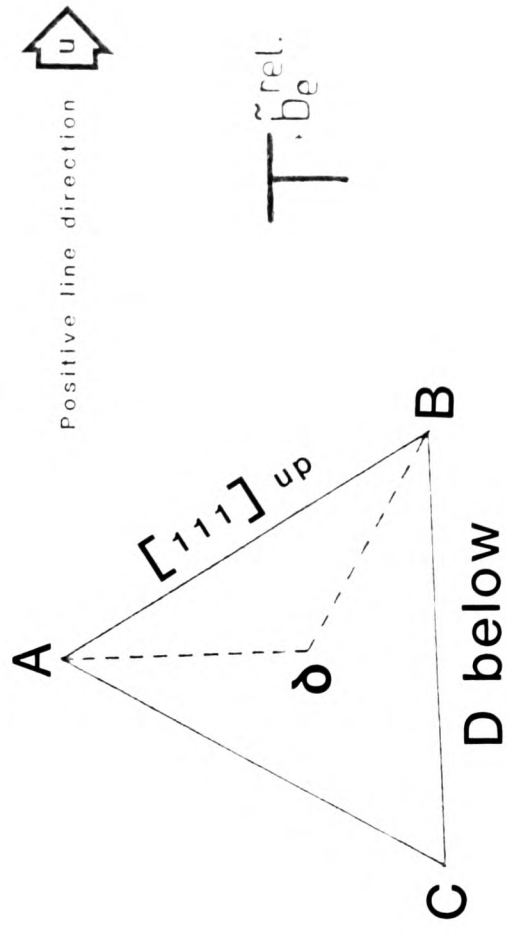
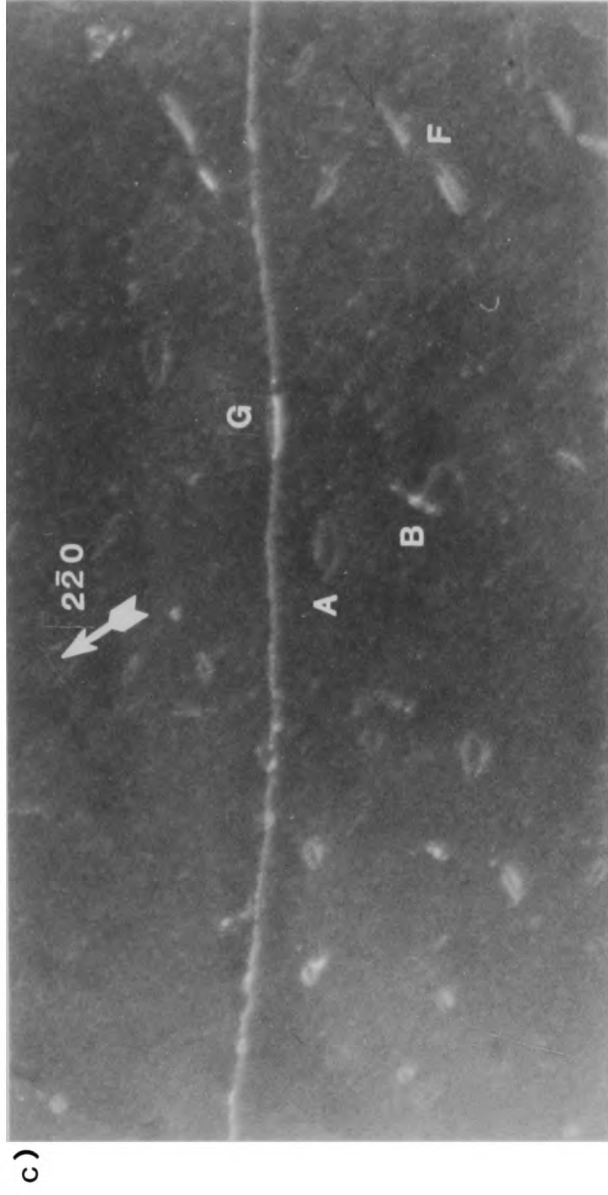
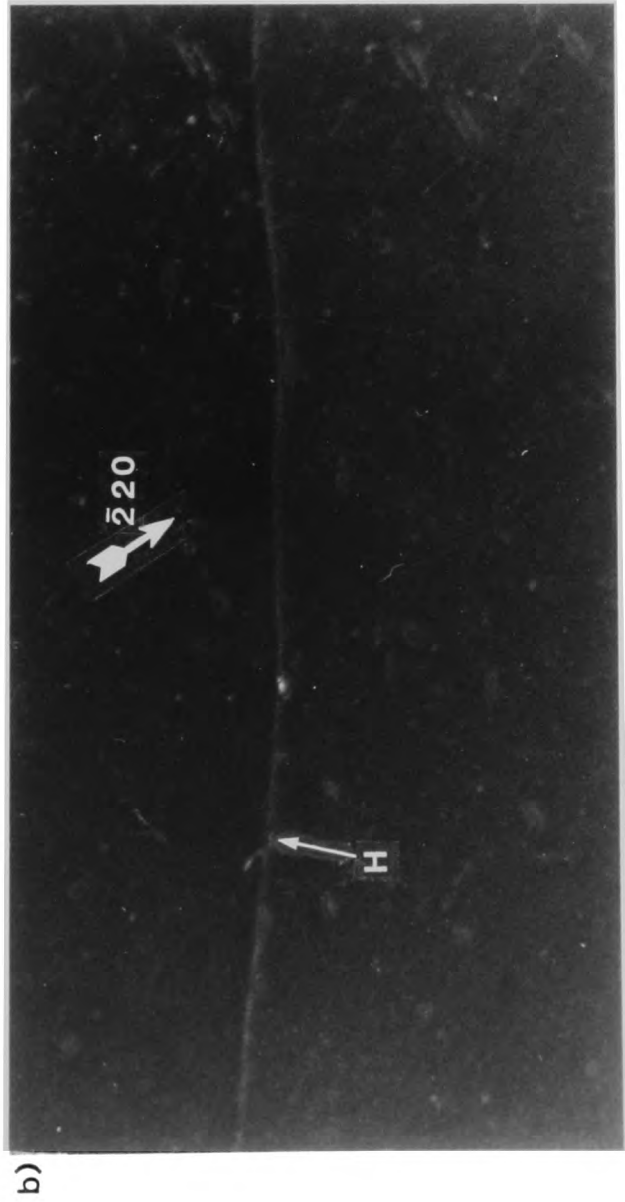
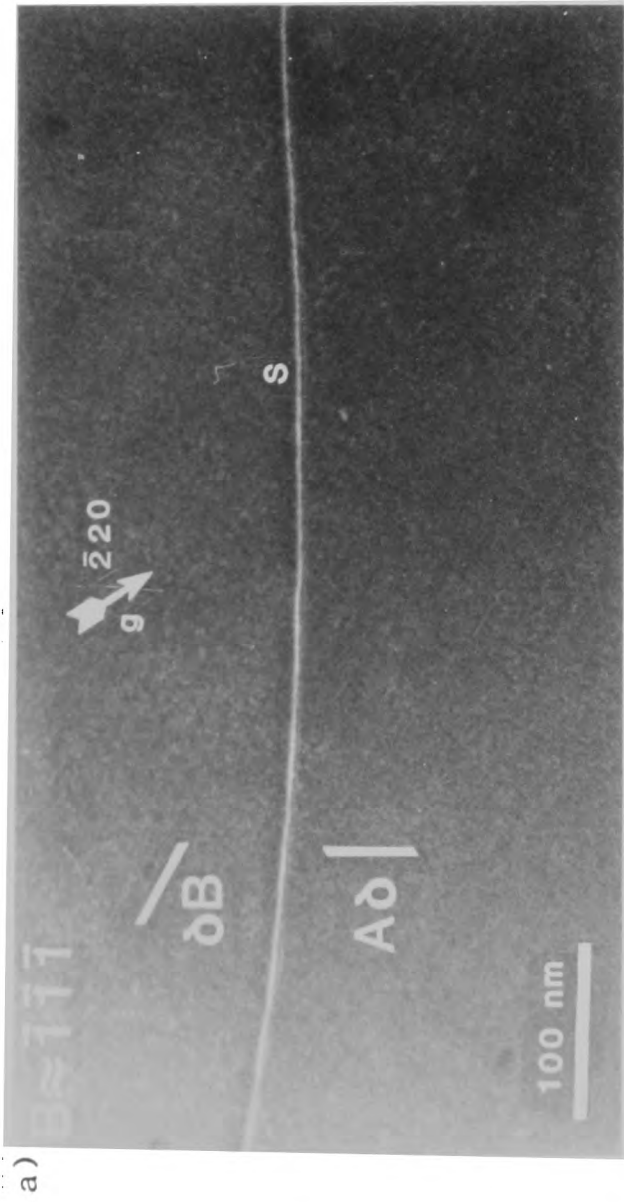
In micrograph (d), there appears to be a gap in the line close to 'G'. The implication that a reaction has taken place between cluster 'G' and the dislocation is not borne out by micrograph (h), however, in which the lower, edge, partial $\overrightarrow{A\delta}$ can be seen to be continuous at this point. In common with all other irradiations, therefore, the Burgers vector of this dislocation does not appear to have altered after irradiation. There is also no evidence for climb of the, still dissociated, line.

A high density of large background loops is seen to have formed. It is possible that the area contained within these background loops is lower in a region close to the dislocation (see micrograph (e)) than in the bulk; this is not a dramatic effect, however, and would require a detailed statistical analysis for verification. Many of these loops were characterized using standard techniques and all were found to be interstitial in nature. Burgers vectors of these loops are: 'A'= $\overrightarrow{\alpha A}$; 'B'= $\overrightarrow{\beta B}$; 'C'= $\overrightarrow{\gamma C}$; 'D'= $\overrightarrow{D\delta}$.

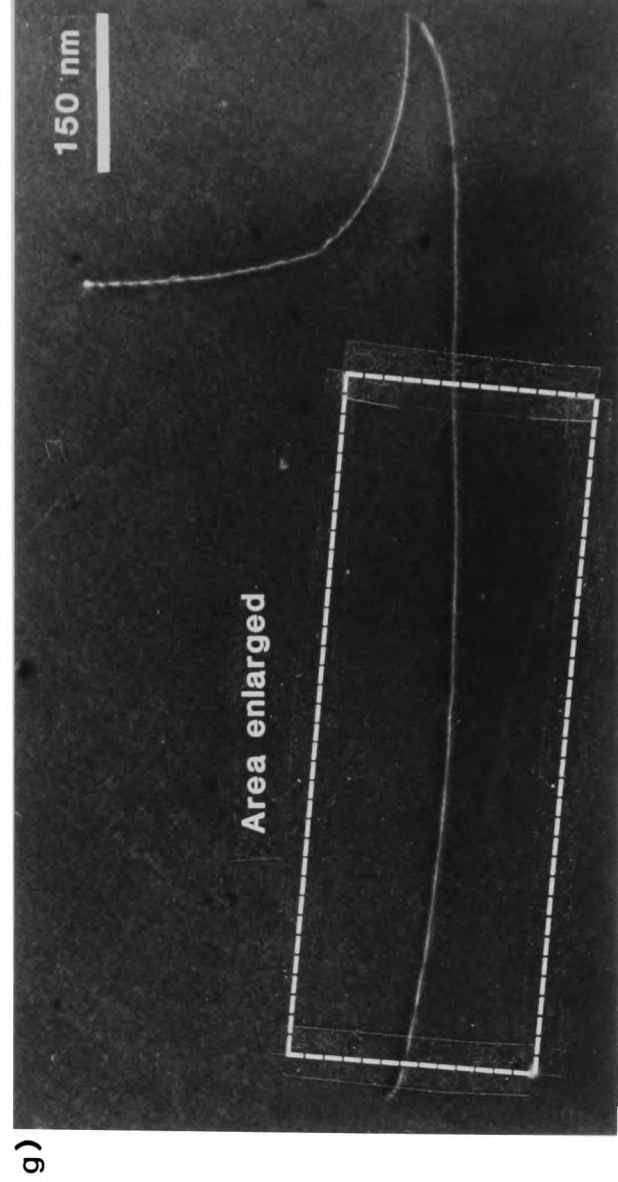
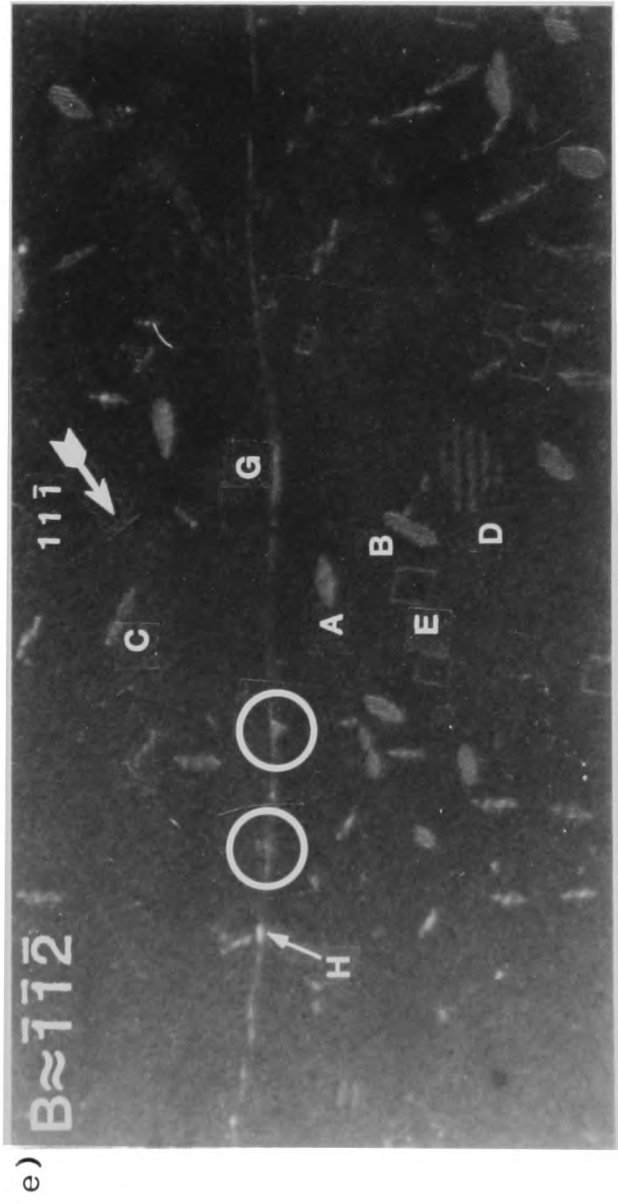
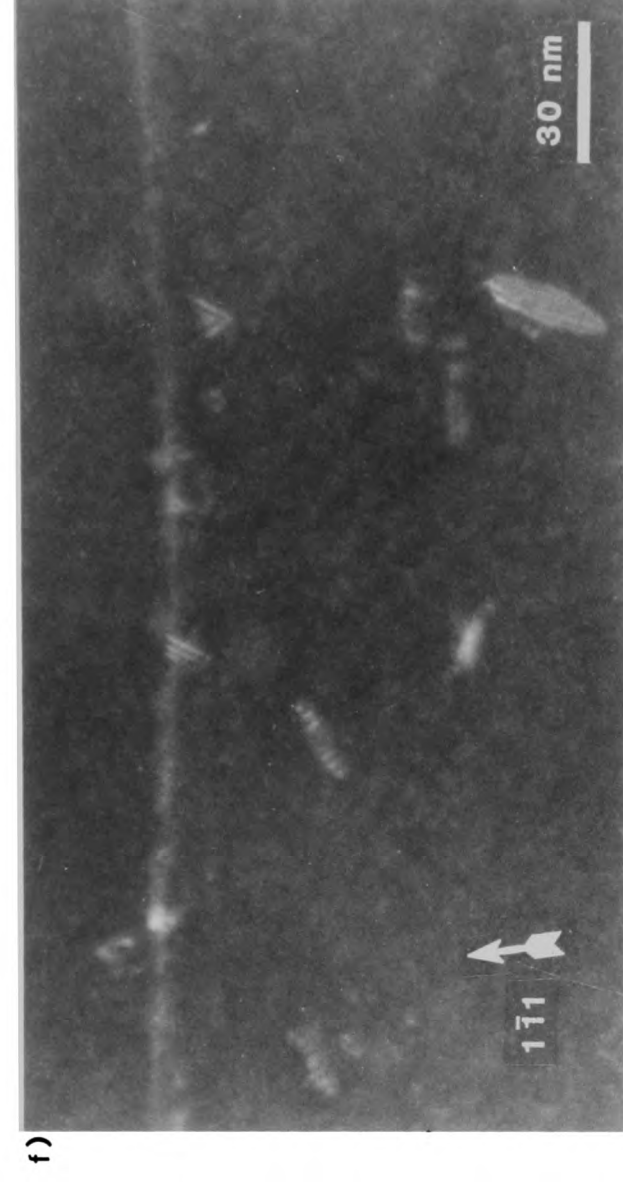
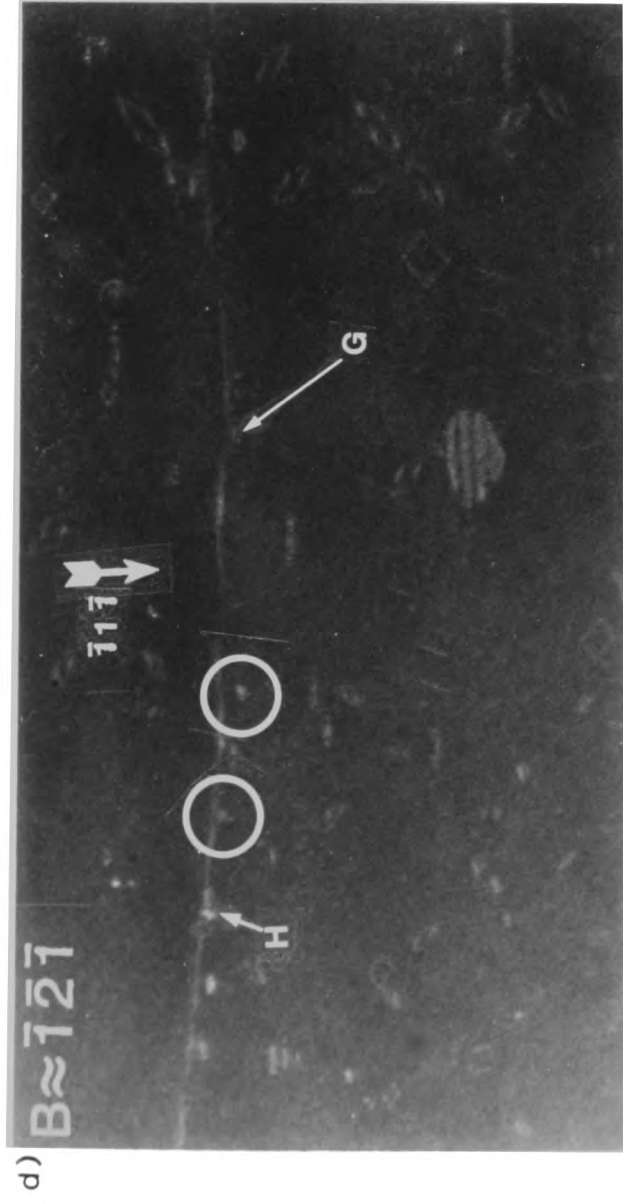
Interstitial perfect prismatic loops are also commonly seen, such as 'E' ($\tilde{b}_L=\overrightarrow{DC}$) and 'F' ($\tilde{b}_L=\overrightarrow{AB}$). It is interesting to note that whilst very many faulted $\tilde{b}_L=\overrightarrow{\alpha A}$, $\overrightarrow{\beta B}$ and $\overrightarrow{\gamma C}$ loops are visible, only one loop ('D') could be conclusively identified as having Burgers vector $\overrightarrow{D\delta}$. It seems likely that this is a thin foil effect, $\overrightarrow{D\delta}$ loops being unfaulted more easily than are loops on inclined planes. It is

²⁰Maximum tilt along $[0\bar{2}2]$ Kikuchi band. Employed for analysis of near-screw section shown in the following figure (figure 5.4).

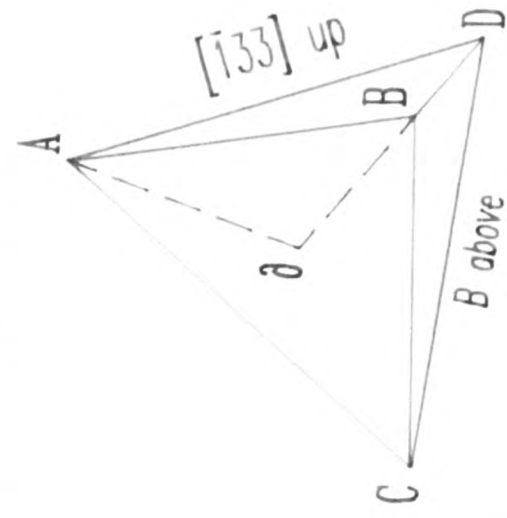
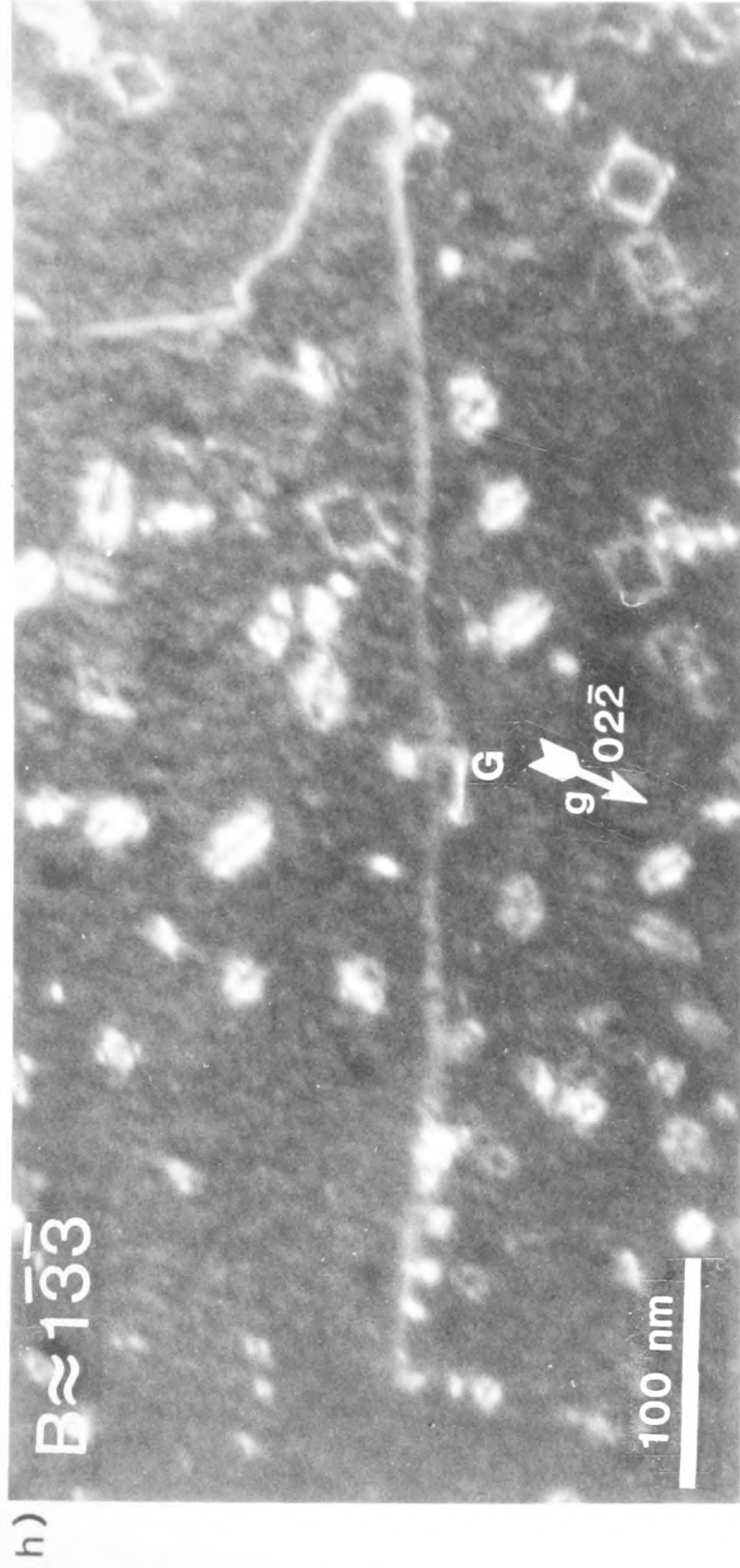
Figure 5.3
(3 pages)



60° Dislocation in Fe-40%Ni-17%Cr (at.%) Irradiated at $\sim 400^\circ C$



60° Dislocation in Fe-40%Ni-17%Cr (at.%) Irradiated at ~400°C



60° Dislocation in
Fe-40%Ni-17%Cr (at.%)

Irradiated at $\sim 400^\circ\text{C}$

dramatically evident that a large disparity exists between numbers of visible interstitials and vacancies; very few vacancies form visible clusters.

Interpretation:

It seems likely that feature 'G' is similar in nature to the strip of stacking fault seen in figure 4.6, micrograph (u) (near 'B'). The presence of feature 'G' at a point where the dislocation is exactly at the 60° orientation may similarly result from the preferential drift of defects (vacancies in this case) towards a region where one partial is in pure edge orientation.

The population of small vacancy clusters close to the section of dislocation nearest to a foil surface may result from action of the surface as an efficient sink for interstitials. Such interstitial depletion may create a sufficiently large net super-saturation of vacancies to enable the survival, and growth, of visible clusters (e.g., Kiritani, 1975).

A lower density of interstitial loops near the dislocation would suggest that the dislocation is acting as a sink for interstitials, in common with the screw dislocation of figure 5.2. However, the fact that the dislocation still appears to be dissociated on a *single* plane, δ , suggests that major climb motion of the line has not taken place. The hypothesised operation of the Thomson & Balluffi (1962) mechanism in high SFE materials (Cherns et al, 1980) cannot be excluded here, but seems unlikely since extended double jog nucleation would have had to have occurred homogeneously along the entire length of the dislocation. It would appear, therefore, since no interstitial clusters are seen anywhere along this section of the dislocation, that interstitials have diffused along the dislocation to be lost at the foil surface at left.

It is possible that point-defects may also have diffused towards the screw section at right. The fate of this section is enlarged upon in the following figure:

Figure 5.4; Dislocation in Fe-40%Ni-17%Cr; Irradiated at $\sim 400^\circ\text{C}$.

Observations:

Micrographs (a) and (b) are $|\tilde{\mathbf{g}} \cdot \tilde{\mathbf{b}}_{\text{T}}| = 2$ images taken before irradiation. Several pre-existing constrictions are evident in micrograph (a) whilst the dissociation of the dislocation is more clearly imaged in (b). The partial image separation at points 'S', at which the dislocation is in 60° orientation, is $\sim 30 \text{ \AA}$. As the dislocation tends towards screw orientation the partials are no longer individually resolved; the expected partial separation²¹ would be about $18 \text{ \AA}$, below the computed resolution limits of the weak-beam technique (Cockayne, 1970, 1972). The extra half-plane is above the dislocation

²¹Employing isotropic elasticity and setting $\nu = 1/3$ (see Hirth & Lothe, p. 315, for example).

along segment 'EF' and below it elsewhere.

After irradiation (micrographs (c)-(i)) there has been a large change in the shape of the line. Segments 'EF' and 'FA' appear slightly straighter and the sharp cusp at 'F' has been removed. In micrograph (c) the cusp at 'A' is seen to be very much sharper than before irradiation, and pronounced cusps have also formed at other points ('B', 'C', 'D', 'G'). These cusps remain visible on tilting to either the (121) or (112) pole, or close to the (211) pole (micrograph (g)). This specimen was sectioned a few degrees away from (111) orientation, making it impossible to reach the (211) pole.

The entire length of the dislocation remains visible in all three independent $\tilde{g} = \langle 2\bar{2}0 \rangle$ reflections in the (111) pole (only 2 shown here) after irradiation. It is also visible in $\tilde{g} = \pm [1\bar{1}1]$ and $[3\bar{1}\bar{1}]$. Its Burgers vector ($= \vec{AB}$) is therefore unaffected by the irradiation. Individual partials are no longer separately imaged after irradiation in either $|\tilde{g} \cdot \tilde{b}_T| = 2$ image (1 only shown). In the $\tilde{g} \cdot \tilde{b}_T = 0$ images (e.g. (f)), plainly visible SFC associated with the 60° dislocation segment 'EF', and weak SFC associated with segments 'FA' and 'BG' suggest, however, that these segments are still dissociated on δ . The SFC from these regions is more strongly visible in the opposite reflection ($\tilde{g} = [\bar{1}\bar{1}1]$, not shown). Segment 'CD' shows an isolated region of particularly strong SFC in micrograph (f), also suggesting the presence of a faulted region; this faulted region is however only weakly visible in the opposite reflection. Segments 'AB' and 'GC' apparently shows no SFC in either reflection.

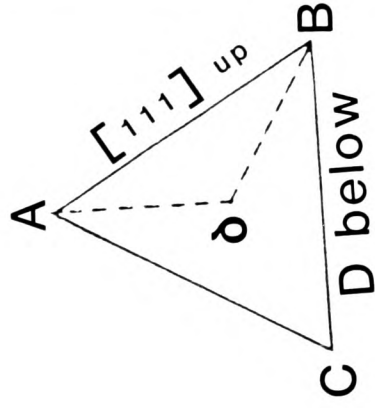
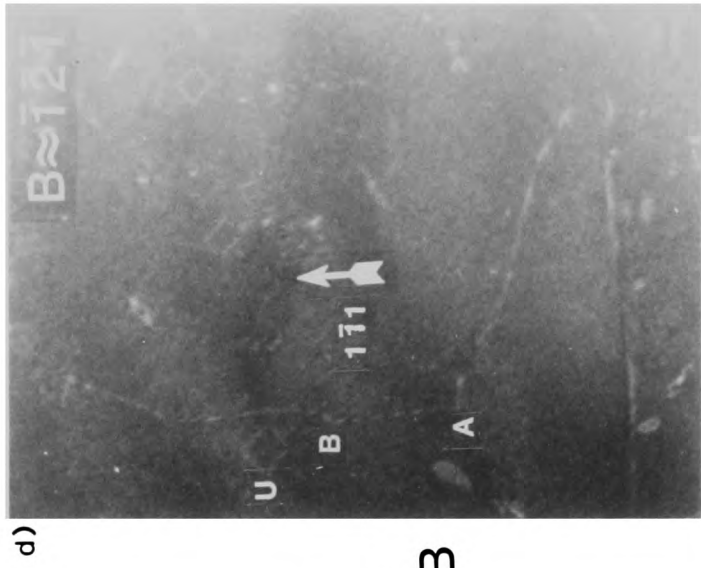
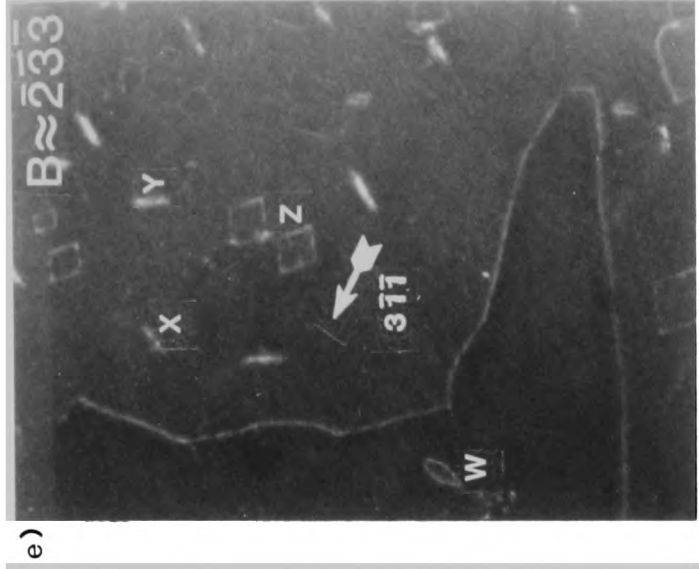
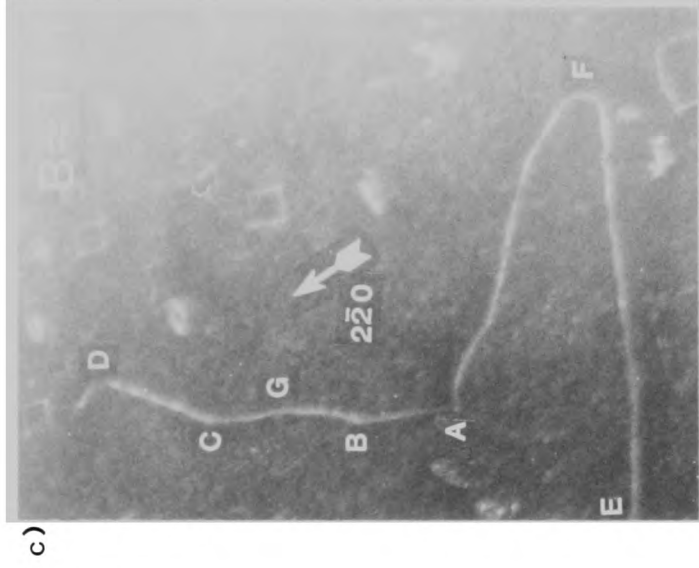
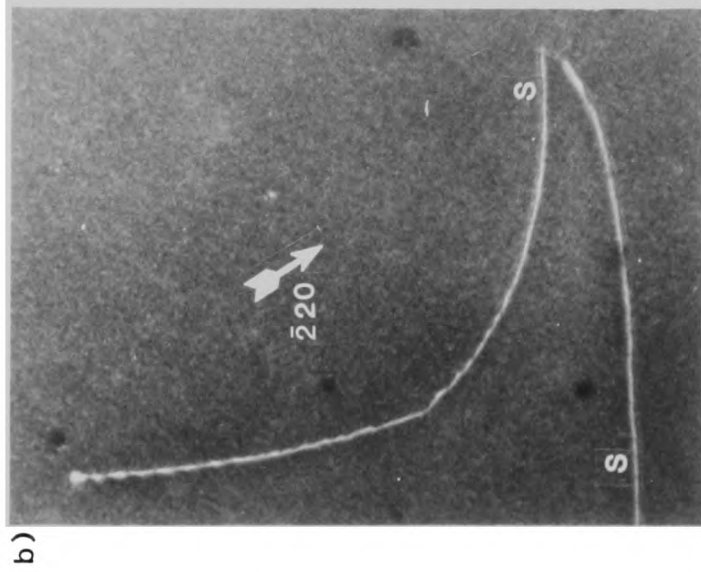
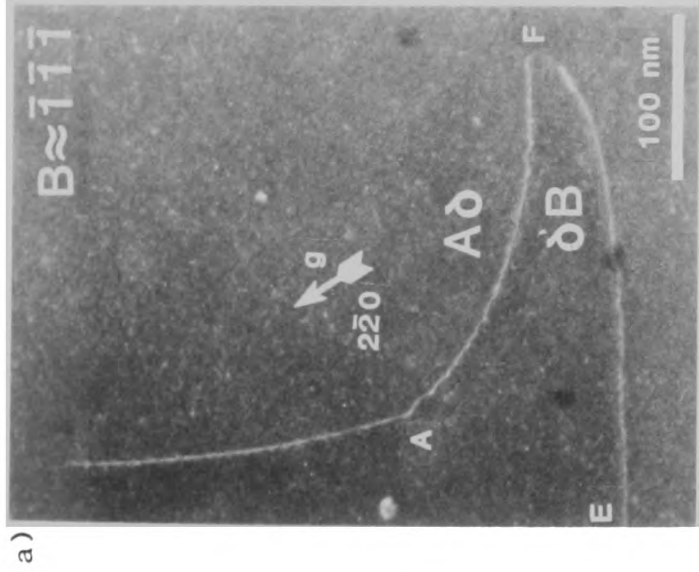
This dislocation is no longer confined to a single plane δ . Looking at micrograph (d), for example, segment 'AB' appears longer than in (c) suggesting that 'A' is below 'B'. In the same pair of micrographs segment 'CD' appears shorter when the foil is tilted to $\tilde{B} \approx [\bar{1}\bar{2}\bar{1}]$, suggesting that 'C' is above 'D'. These results are confirmed by the large angle tilt experiment shown on the second page of this figure, in which segments of the dislocation are seen to rotate clockwise, or anti-clockwise and change in length on tilting towards $\tilde{B} \approx [1\bar{3}\bar{3}]$. Directional analyses performed on these segments yielded some unusual results, however:

Segments 'EF' and 'FA' of the dislocation, lying nearly perpendicular to the tilt axis ($[0\bar{1}1]$), exhibit marked foreshortening, echoed by the relative sizes of micrographs (g), (h) and (i), on tilting either side of $\tilde{B} \approx [\bar{1}\bar{1}\bar{1}]$. This suggests that these segments are still principally confined to a plane δ .

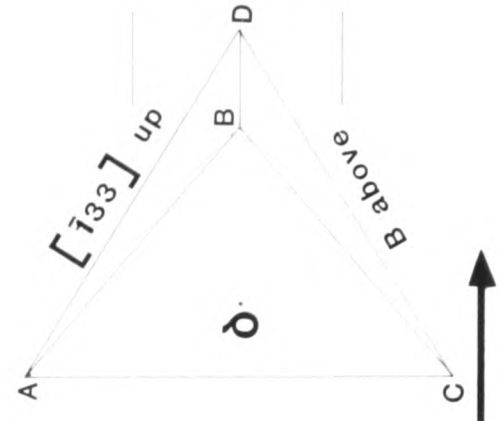
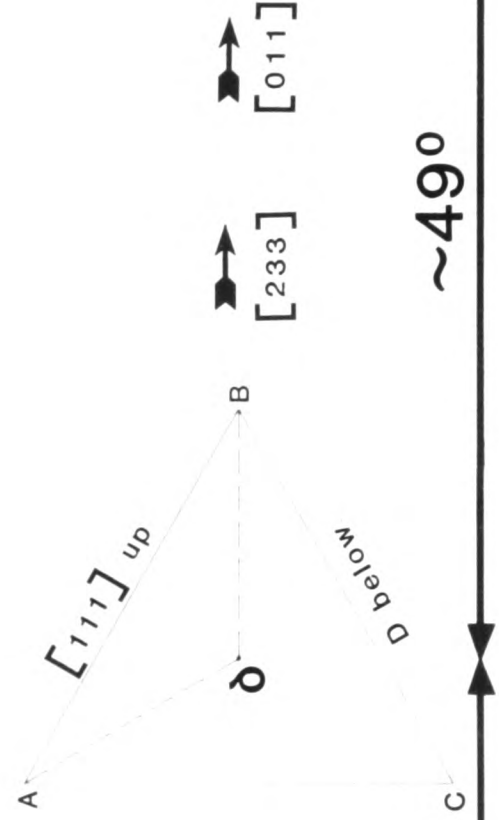
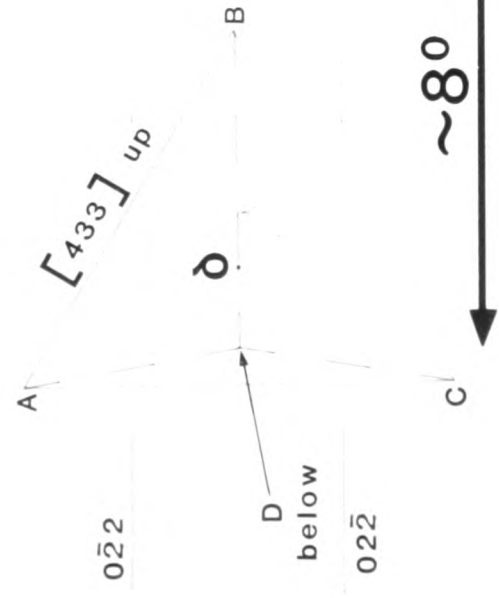
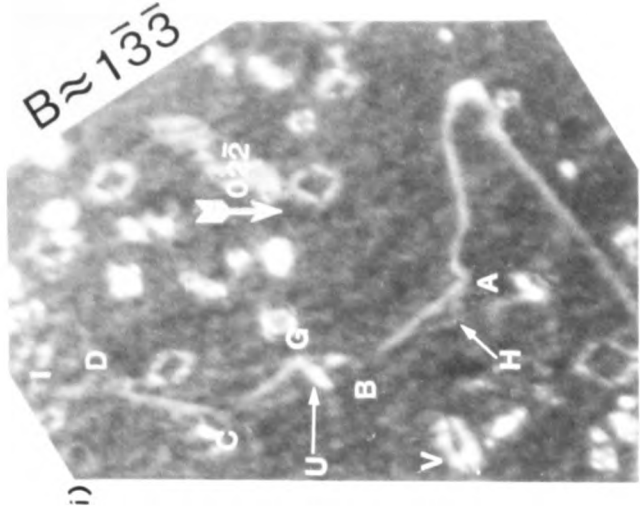
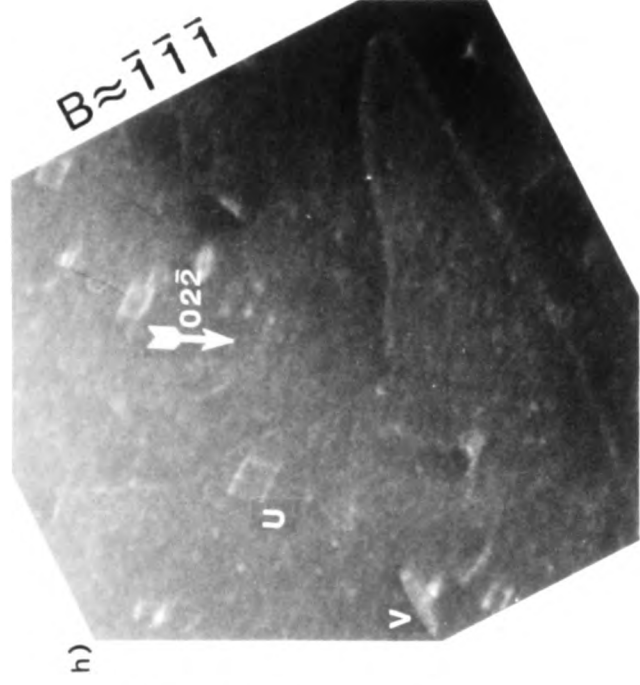
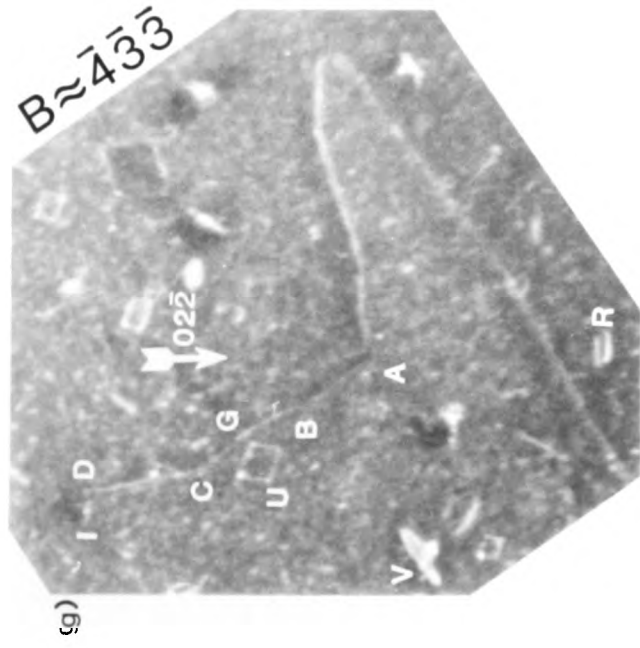
At $\tilde{B} \approx [\bar{1}\bar{1}\bar{1}]$, segment 'AB' makes an angle of $35 \pm 2^\circ$ with $[0\bar{1}1]$ whilst at $\tilde{B} \approx [1\bar{3}\bar{3}]$ the same angle is $40 \pm 2^\circ$. Solving the above, and checking the result with similar data from $[\bar{2}\bar{3}\bar{3}]$, $[\bar{1}\bar{2}\bar{1}]$ and $[\bar{1}\bar{1}\bar{2}]$ beam directions, yields the result that this segment is inclined at about 22° to planes δ , with line direction within $\sim 5^\circ$ either side of $[1\bar{1}1]$. It does not appear, therefore, to lie on a close-packed plane.

The neighbouring segment 'BG' appears to lie on δ , whilst 'GC' is rotated and lengthened by a

Figure 5.4
(2 pages)



Dislocation in Fe-40%Ni-17%Cr (at.%) Irradiated at $\sim 400^\circ\text{C}$



$\sim 49^\circ$

$\sim 8^\circ$

Dislocation in Fe-40%Ni-17%Cr (at.%) Irradiated at $\sim 400^\circ\text{C}$

similar amount in the same sense as segment 'AB' on tilting towards $\tilde{B} \approx [1\bar{3}\bar{3}]$. It seems likely, therefore, that 'GC' also lies along a direction close to $[1\bar{1}1]$.

Segment 'CD' is observed to rotate markedly clockwise. A similar analysis to that performed on segment 'AB' above shows that it is inclined at about 21° to planes δ , with line direction $\sim 20^\circ$ away from the horizontal, close to $[\bar{1}\bar{6}3]$ ($\pm 5^\circ$; 'C' above 'D').

Above point 'D' there is a very short line segment ('DI'). It appears almost perpendicular, and almost parallel to the tilt axis in (g) and (i) respectively. The line direction of this segment has not been determined precisely but is consistent with it being very steeply inclined in the foil, with point 'I' above 'D'. No SFC is visible from this section in micrograph (f) and it does not appear to lie on a close-packed plane.

Owing to the large angle at which the foil is tilted in micrograph (i), and to the presence of a particularly heavy contamination layer from the Oxford HVEM, it was found necessary to strengthen the weak-beam conditions used elsewhere in this thesis, reducing the deviation parameter (s) to $\sim 0.7 \cdot 10^{-2} \text{\AA}^{-1}$. As a result what may be cluster damage along the line 'DG' (which may extend to points 'H' and 'I' at either extremity of the line) is faintly visible in this micrograph. This line is not imaged in other reflections. This section of dislocation was close to the 30° orientation before irradiation, its extra half-plane lying beneath. In micrograph (i) the clusters are seen to the right of the dislocation from 'D' to 'G', which is consistent with their lying below it (i.e. to its compressional strain field side). Point 'H' may correspond to the pre-irradiation position of cusp 'A'.

Several background features have been marked in micrographs (d), (e) and (g). These were characterized, using standard techniques, as follows:

'Z' is a perfect prismatic interstitial loop on a (011) plane, with Burgers vector, $\tilde{b}_L = \vec{DB}$ in pure edge orientation (see figure 7.6 for details of these loops).

Several pure edge prismatic loops with Burgers vectors in the plane of the foil are seen in both figures from this irradiated area. Three such loops are 'X', 'Y' and 'R', lying on the near vertical ($\bar{1}10$), ($\bar{1}01$) and ($0\bar{1}1$) planes with Burgers vectors $\vec{AB}$, $\vec{CB}$ and $\vec{CA}$ respectively. It is probable that these are also interstitial type defects, in common with all other background features observed here, having formed from $\tilde{b}_L = \vec{\beta B}$ and $\tilde{b}_L = \vec{\alpha A}$ interstitial Frank loops.

'W' is an interstitial Frank loop with $\tilde{b}_L = \vec{\beta B}$. All loops of species 'W', 'X', 'Y' and 'Z' are showing strong 'inside' contrast in micrograph (e) ($\tilde{g} = [3\bar{1}\bar{1}]$).

Loop 'U' is also a perfect prismatic loop in pure edge orientation, with Burgers vector $\tilde{b}_L = \vec{DA}$. It is exhibiting 'outside' contrast in $\tilde{g} = [1\bar{1}1]$ and is also seen in the tilt series (g) to (i). In micrograph

(i) the foil is tilted such that the (101) planes, on one of which this loop is lying, are very steeply inclined in the foil. Loop ‘U’ thus appears as a fairly thin line, which should not be confused with the nearby dislocation image. This loop is visible in ‘inside’ contrast throughout the tilt series.

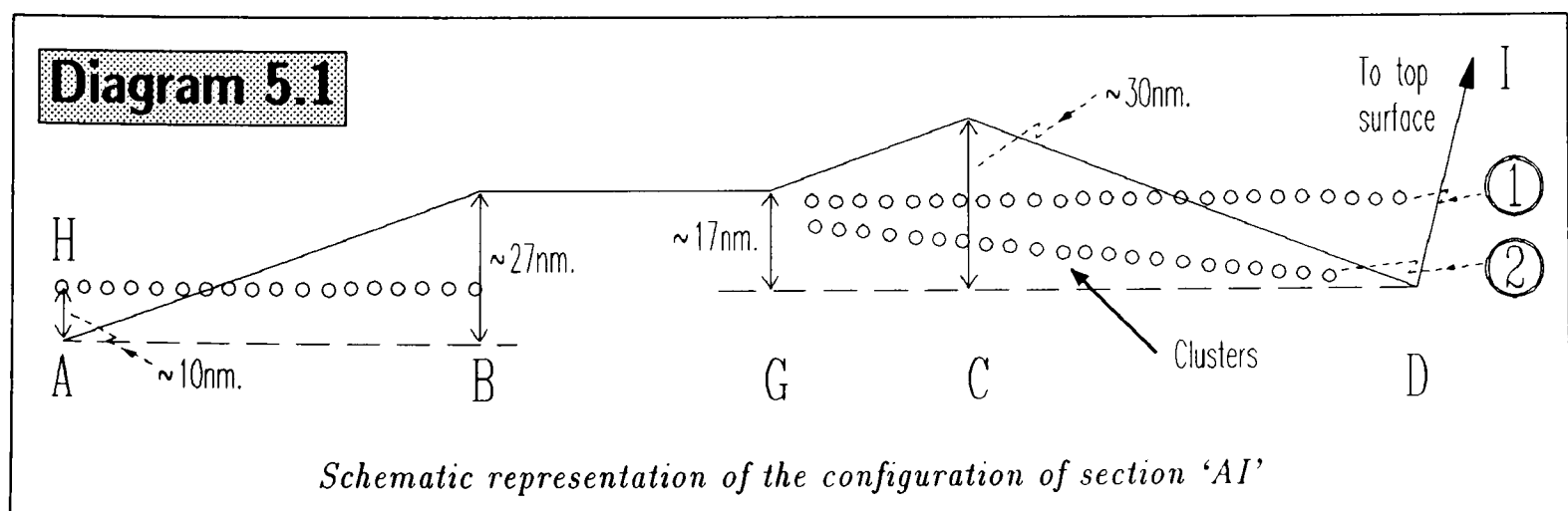
Loop 'V' is outside of the area shown in the first page of this figure but lies along $[\bar{1}01]$ in $\bar{B} \approx [\bar{1}\bar{1}\bar{1}]$ images. It shows contrast and inside/outside behaviour consistent with $\tilde{b}_L = \alpha \vec{A}$. In micrographs (g) and (h) direction $[\alpha \vec{A}]$ is upwards in the foil; in micrograph (i) the foil is tilted such that direction $[\alpha \vec{A}]$ now points downwards. This leads to the expected contrast reversal from loop 'V'; showing 'inside' contrast in (g) and (h) and 'outside' contrast in (i) (e.g., Loretto & Smallman, 1975).

The stronger conditions employed for micrograph (i) clearly show the formation of an interstitial loop depleted zone (LDZ), extending perhaps 50-100 nm., around the dislocation (particularly near point 'A').

Interpretation:

The exact results of directional analyses performed are very susceptible to small measurement errors. Such errors would have to be unrealistically large, however, for the upward, or downward senses of line segments analysed to have been determined incorrectly. It was frequently possible to plot diffraction vectors with considerable accuracy ($\sim 2^\circ$) by making use of features with known, low-index, crystallographic directions.

The apparent 3D configuration of the 0-30° line section ‘AI’ is sketched in diagram 5.1. Two possible positions (‘1’ and ‘2’) are given for the line of clusters. These correspond to the line of clusters extending from point ‘G’ to either point ‘I’ (‘1’) or point ‘D’ (‘2’). The exact positions of these (vacancy) clusters cannot be determined since they are only visible in a single reflection of the series. Some vertical displacements are also indicated in the diagram. These are estimated from measured projected line lengths and angles made by line sections and a horizontal (111) plane. Errors on the magnitudes of these displacements are potentially large and their values should be treated with caution.



The location of lines of clusters, which are assumed to lie close to the pre-irradiation position of

the dislocation, will dictate which of two possible interpretations is more likely to be correct; either position is consistent, however, with the essential result shown by this figure, namely that this dissociated dislocation has climbed via the absorption of interstitials:

Position '1': Both segments 'AB' and 'GI' are now approximately evenly distributed about their pre-irradiation positions. This corresponds to a right-handed helical configuration. Since this has formed from a left-handed screw dislocation, interstitial absorption, or vacancy emission, is required. As pointed out in figure 3.4 (*'Interpretation'* section), vacancy emission under HVEM irradiation is extremely unlikely. In addition, the presence of a LDZ around the dislocation, suggesting that significant numbers of interstitials have migrated to the dislocation, points to interstitial absorption being the dominant mechanism.

Position '2': The extra half-plane lies below section 'GI'. Since this section now lies above its original position, and does not lie on a plane γ , interstitial climb is indicated.

Of the two positions, '1' would seem the most likely since it corresponds to the configuration of segment 'AB', and indicates that the line of clusters from 'G' to 'I' may lie on a (111) plane. Point 'I' is probably where the dislocation meets the top surface of the foil.

The apparent line directions of the segments 'AB' and 'CD' provide clues to a mechanism via which this configuration could have evolved, by noting that, looking down $[\bar{1}\bar{1}\bar{1}]$, direction $[1\bar{1}1]$ (segment 'AB') lies directly above $[1\bar{2}1]$ (δ) and direction $[\bar{1}\bar{6}3]$ (segment 'CD') lies, almost, directly above $[0\bar{1}1]$ (δ). Both directions are inclined at $\sim 20^\circ$ to the horizontal.

It would appear, therefore, that these segments have glided, probably under the influence of the jog climb mechanism previously detailed, to lie along $[1\bar{2}1]$ and $[0\bar{1}1]$. Further glide of the line, or climb of the jogs, is then resisted by core forces (see '*Points of Discussion*' in chapter 4). In the case of the lower cluster position ('2'), operation of the **Thomson & Balluffi** (1962) mechanism may then be prompted; climbing the line vertically upwards by the absorption of precipitated interstitial loops with the same Burgers vector as the dislocation²². Alternatively, climb may be proceeding via a mechanism slightly different to that of **Condat & Décamps** (1985). Both mechanisms are discussed in chapter 8. It is not fully understood why climb should be favoured towards the middle of section 'AI'; the presence of efficient sinks for interstitials at either end, such as the surface, and possibly the glissile section 'AF' may be a factor, as might any orientation dependence of core diffusion.

In the case of the more likely cluster position '1', the absorption of a few interstitials, possibly

²²Subject to the nucleation criteria derived by **Grilhé et al** (1977).

via the mechanisms of Thomson & Balluffi or Condat & Décamps, is still required to form the helical structure, however, the entire configuration may evolve principally via core diffusion, simply via atomic redistribution; for section 'CD' this would correspond to the diffusion of vacancies towards the surface (point 'I'), away from the pure screw section of the dislocation. This would seem to be more likely than the opposite diffusion of interstitials towards the screw section and away from the surface and may suggest that vacancy pipe diffusion is efficient here. The situation is slightly less clear for section 'AB', at one end of which the dislocation turns sharply through screw orientation, but the apparently fairly even vertical distribution of this segment about the line of clusters 'HB' also suggests that core diffusion is dominant. The very evident formation of an interstitial LDZ indicates that both interstitial and vacancy core diffusion mechanisms are likely to be involved. The unpinning of the pre-existing sharp cusp 'F', and (associated) formation of the small jog visible in micrograph (i), may indicate climb, which is likely to be interstitial by reason of the expected dislocation bias.

The facts that segments 'AB' and 'CD' are of approximately equal length, and inclined at about the same angle in the foil, may indicate that defect re-distribution (and/or absorption) is roughly constant along the entire dislocation, suggesting that pipe diffusion is indeed responsible.

Finally, the reason why the inclined segment 'CD' should show SFC in $\tilde{g}=[11\bar{1}]$ is not understood; the reversal of strong/weak contrast in $\tilde{g}=\pm[11\bar{1}]$ from this segment, with respect to dislocation sections dissociated on δ , may indicate either that it is an extrinsically faulted region or that it has a fault vector $\tilde{R}=\pm[\bar{1}11]$; different from the rest of the dislocation. In either case, this may constitute either the first direct observation of the operation of the **Thomson & Balluffi** (1962) mechanism or a high SFE variation of the **Condat & Décamps** (1985) mechanism. Operation of either mechanism may also explain the slight deviation of the projection of segment 'CD' onto δ from $[0\bar{1}1]$, if clusters with $\tilde{b}=\pm\vec{AB}$ and habit plane β , have attached to the dislocation inhomogeneously along its length.

Alternatively, bearing in mind that this segment ('CD') presumably lies very close to the foil surface, this contrast may simply be an artifact of the weak-beam image, perhaps arising from the excitation of a reflection too distant to be seen within the area of reciprocal space visible on the microscope screen.

The essential points from this figure may be summarized as follows:

- The dislocation is acting as a net absorber of interstitials. For the more likely case of cluster position '1', however, and for segment 'AB', the imbalance is not likely to be great.
- It is likely (given position '1') that the core diffusion of both interstitials and vacancies has been a

very efficient process here, as might be expected for a high SFE alloy (see chapter 8, §8.2.3). For either of positions '1' or '2', interstitials, for which the dislocation is a strong sink (formation of LDZ), have apparently attached to the dissociated dislocation, leading to its climb, probably via an assimilation rate-controlled mechanism, which may be similar to those proposed by **Thomson & Balluffi** (1962) and **Condat & Décamps** (1985). For position '2', core diffusion is still suggested along segment 'AB', although whether vacancies, interstitials, or both are involved is unclear.

Points of Discussion

- *The Rôle of Pipe Diffusion in the Argonne Irradiations.*
- *Background Loops: Oxford vs. Argonne Irradiations.*

The Rôle of Pipe Diffusion in the Argonne Irradiations

The characterization of the nature of large background loops seen after irradiation in the Oxford HVEM as interstitial is thought to be likely to represent the character of the smaller background loops of the Argonne irradiation. This is in agreement with observations of interstitial background loops in other alloys.

The formation of an LDZ near the screw dislocation of figure 5.2, and the lack of any evidence for climb, suggests that pipe diffusion of interstitials is occurring here. This is apparently confirmed by the observations that the very large faulted region, likely to be intrinsic in nature, at the end of this dislocation has been effectively annihilated whilst other irradiated faulted regions (not shown), not connected to a dislocation, shrank to a lesser extent after irradiation. This is analogous to the relative decreases in sizes of 'connected' and 'unconnected' voids in Al seen on annealing by Volin et al (1971). Both vacancies (figure 5.3 and probably 5.4) and interstitials (notably figure 5.2) are core diffused. The decrease in size of unirradiated faulted regions was noticeable but even less marked. Unirradiated large faulted regions are likely to have shrunk by vacancy emission on heating to the irradiation temperature.

The faulted region at the end of the heavily jogged dislocation of figure 5.1 has only shrunk slightly after irradiation. Jogs therefore seem to act as effective barriers to core diffusion, which otherwise appears to be efficient in this material. Since the core diffusion of interstitials along the pure screw dislocation of figure 5.2 is seemingly very effective, the supposition that this dislocation was initially jog-free, thereby explaining the lack of zig-zagging, is apparently confirmed. The behaviour of this dislocation is therefore consistent with mechanisms outlined in the preceding two chapters, and with the near screw dislocation of figure 5.4, along which pre-existing constrictions are seen.

Background Loops: Oxford vs. Argonne Irradiations

It can be seen from the above that the behaviour of both Oxford and Argonne irradiated dislocations is generally consistent, taking into account differences in pre-irradiation configurations. However, the briefest of glances at the figures reveals marked differences in the sizes, if not the density, of background loops. It is thought that these differences are directly attributable to the use of different microscopes, whilst the behaviour of the lines themselves are substantively unaffected.

It is possible that the greater area of interstitial loops seen after irradiation in the Oxford HVEM is simply due to a higher dose. However, it seems more likely than not that dose rates in Oxford and Argonne instruments are similar, in which case the Oxford irradiation is to a slightly lower dose (0.033 dpa) than either Argonne irradiation.

Perhaps a more likely origin for background loop differences lies in a probable, large, difference in microscope vacuums and contaminant concentrations, a typical vacuum gauge reading for the Oxford HVEM being $\sim 2.5 \times 10^{-5} \tau$, whilst the Argonne microscope regularly achieved 10^{-6} - $10^{-7} \tau$.

Quantitative comparisons are obviously difficult between non-identical vacuum gauges located at different points in different columns, however it is expected that the Argonne microscope regularly achieved a far better vacuum than its Oxford sibling. The build-up of a contaminant layer is also substantially quicker in the Oxford instrument. Incident 1MeV electrons will effectively implant atoms from this contaminant layer into the specimen, which may create sites for interstitial precipitation more quickly than otherwise. The large size of the many loops, after the nominally low dose Oxford HVEM irradiation, indicates that once a sessile loop nucleus has formed it may climb, and unfault, very quickly. This observation is apparently confirmed by the qualitative observation that background loops seen after the Argonne irradiations shown are significantly larger after 0.06 dpa, than after 0.04 dpa. A similar effect of contamination on the sizes of background loops was noted at the end of chapter 3.

Finally, the use of different hot stages may also be a factor. Although both hot stages were calibrated, there may still have been a significant temperature difference between the two irradiations.



Summary

Observations:

The following behaviour is exhibited by irradiated dislocations in this material:

• (Interstitial) loop depleted zone;	... 1 ...
• Shrinkage of large faulted regions;	... 2 ...
• New cusps formed along the line;	... 3 ...
• (Vacancy) clusters near line;	... 4 ...
• Large vacancy cluster at 60° orientation;	... 5 ...
• Helix formation / Interstitial climb;	... 6 ...

These are shown in the following figures: (Table 5.1)

Table 5.1

Figure	Character	Dose (dpa)	Temperature (°C)	Observations (see above)
5.1	see text	0.04	400 ± 50	1, 2, 3, 4;
5.2	~ 0°	0.06	400 ± 50	1, 2, possible glide;
5.3	60°	see text	400 ± 20	(1, 3,) 4, 5;
5.4	0°-30°	see text	400 ± 20	1, (4), 6;

It is extremely difficult to tell whether the partial image separations are decreased by irradiation. The level of contamination encountered after Oxford irradiations (only 1 shown) degraded the resolution of the weak-beam images to an extent such that even if the dissociation width had *increased* slightly the partials would still not have been separately imaged. The geometry of the Argonne specimens (only 1 shown), in which long dislocations were not found, also prohibited this determination.

The lack of evidence for climb of the dissociated, glissile dislocation of figure 5.3 suggests that jog climb, rather than climb of the intervening segments, is the primary origin of the new cusps seen along the dislocation of figure 5.1.

Burgers vectors of irradiated dislocations are unaffected by irradiation. Operation of the Thomson & Balluffi (1962), or Condat & Décamps (1985) mechanisms may have been seen.

The following observations suggest that pipe diffusion involves both defect species, and is comparatively efficient in this alloy:

- Greater annihilation of ‘connected’ large faulted region of figure 5.2.
- Formation of LDZ’s.
- Precipitation of large vacancy cluster at 60° orientation in figure 5.3.
- Probable large scale atomic redistribution along near-screw dislocation of figure 5.4.
- No zig-zags seen along screw dislocation which may have been jog-free.



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Chapter 6

Irradiation of Fe-15%Ni-17%Cr-1%Si alloy

The addition of a small amount of silicon to the 15%Ni pure ternary alloy is seen to have an effect on the configurations of irradiated dislocations. Generally it is often found, however, that observations may be understood in terms of mechanisms proposed in the preceding three chapters.

Figure 6.1; 0° - 20° Dislocation in Fe-15%Ni-17%Cr-1%Si; 0.09 dpa at ~ 400°C.

Observations:

Micrographs (a) and (b) on the first page of this 5 page figure show a long dislocation prior to irradiation. This dislocation curves through pure-screw orientation to the centre of each micrograph and is in approximately 25° orientation at either end. It is one of two which intersect to form a $K_u P_u$ node pair (see Hirth & Lothe, p.729 and Carter, 1980a) shown at left. The other dislocation is also shown in the low-magnification, post-irradiation, bright-field micrograph at lower right and is the subject of the following figure (figure 6.2). The P_u node is extended whilst the K_u node is apparently constricted. The extension of K-type nodes is generally expected to be small (see Hirth & Lothe, p.727).

Both partials are visible in micrograph (a), which has $|\tilde{g} \cdot \tilde{b}_T| = 2$ for dislocation $\tilde{b}_T = \vec{AB}$. Individual partials are labelled and the dislocation line direction (normally $\tilde{u}$ in this thesis) is from right to left. The screw section of this dislocation is therefore left-handed. Both micrographs on the first page show this dislocation to be evenly dissociated and free of constrictions. The partial image separation varies from $\sim (35 \pm 10) \text{ \AA}$ to $\sim (55 \pm 10) \text{ \AA}$ at 0° and 25° orientations respectively; errors are based both on possible estimates of the centre of each partial image and the spread of measurements for a particular orientation.

Both dislocations were irradiated to a dose of approximately 0.09 dpa in the Argonne HVEM. The irradiation was centred on the node, at spot-size 10, such that the periphery of the HVEM beam lay very close to the right-hand side of these micrographs.

Almost a full set of post-irradiation micrographs is shown (micrographs (c) to (o)). From these micrographs, and from $\langle 11\bar{3} \rangle$ type reflections (not shown) taken at the various $\{112\}$ poles, it can be seen that the dislocation has behaved similarly to other screw, and near-screw dislocations analysed in the preceding three chapters. There is little point, therefore, in duplicating what is written therein and we concentrate instead on observable differences in behaviour:

The first, and most important, observation is that a previously evenly dissociated, jog-free dislocation has now become complex, and zig-zagged, along its entire length. Zig-zagging is most evident

where the dislocation crosses pure-screw orientation but is generally not very advanced. To the right-hand side of the dislocation (near 'B' as labelled on micrograph (o)) the zig-zagging is faint; this region lay close to the periphery of the HVEM beam and probably received only a low dose. In the higher dose region ('D', see (o)) the dislocation has moved only to one side from its original position, with the longer segments of a zig-zag being those which lie nearer to edge orientation. This behaviour, similar to the 20° dislocation of figure 4.2, is exemplified by the 25-30° dislocation of the following figure (6.2) and so is not discussed further here.

The zig-zag configuration, where the line passes pure-screw orientation, has only formed over a very short length of dislocation and is particularly complex. The spacing of the zig-zags, at $\sim 30-40 \text{ \AA}$, is similar to those seen previously (see figure 3.4 for example). At least one section ('C', see micrograph (o) for labelling) of the dislocation can be seen to be widely dissociated in the $|\tilde{g} \cdot \tilde{b}_T| = 2$ micrograph (d). The confusing absence of SFC from this section in either $\tilde{g} \cdot \tilde{b}_T = 0$ image (micrographs (i) and (j)) is thought to be an artifact of the weak-beam image. The partial image separation along section 'C' is about $35 \text{ \AA}$; apparently unchanged from its pre-irradiation value²³. The line direction of section 'C' is close to $[\bar{1}\bar{3}2]$, however, it can be seen that the partials are not exactly parallel.

The only obvious formation of an inclined connecting segment (see diagram 3.1 for nomenclature) is of segment 'A'. In micrograph (o) this segment appears very short and is aligned with $[1\bar{1}0]$. It probably lies on a plane γ . Care must be taken in attempting to assign a line direction to this segment, since the dislocation is not constricted; only images of the same partial may be compared.

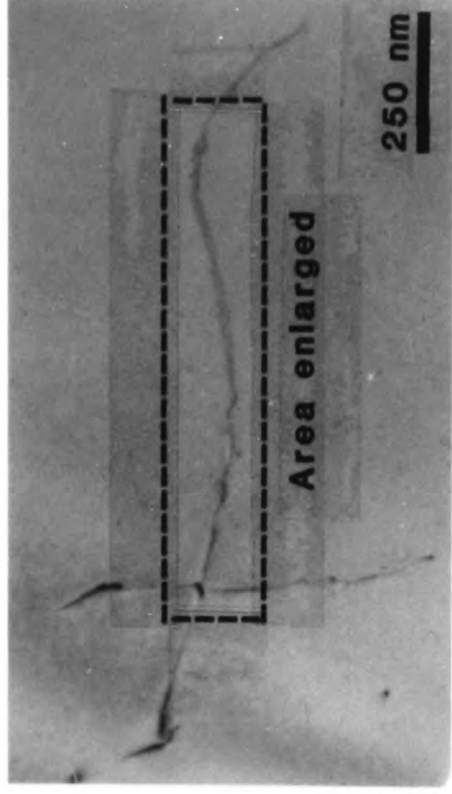
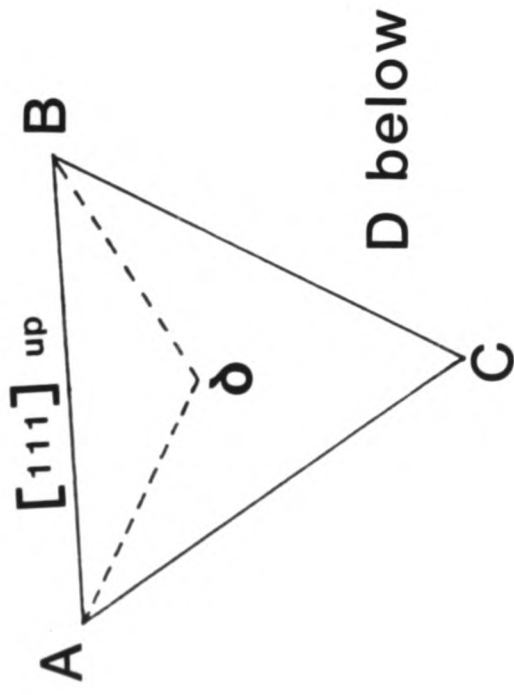
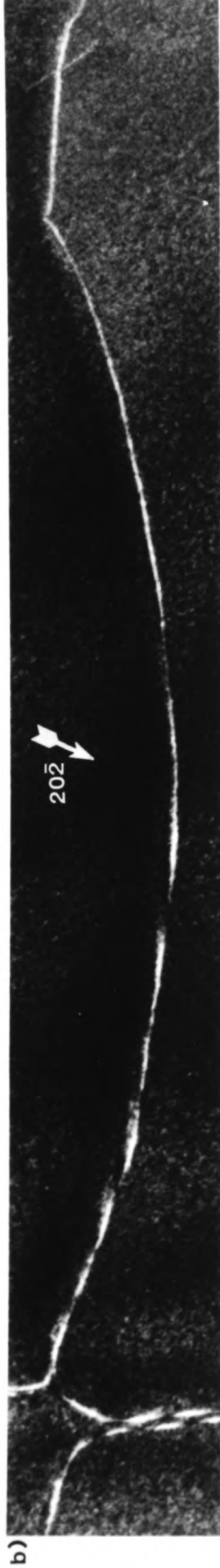
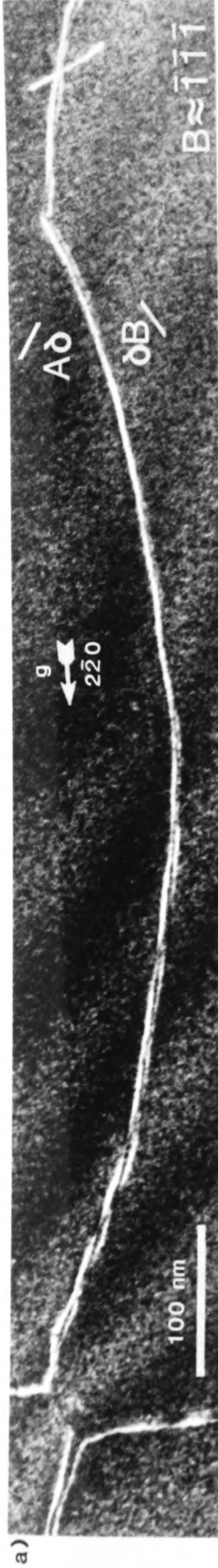
In micrographs (g) and (h), which are taken with beam direction $\tilde{B} \approx [\bar{1}\bar{1}\bar{1}]$, only the upper partial $\tilde{b}_p = \vec{A}\delta$ is visible. The same partial is visible, and connecting segment 'A' appears longer, in micrographs (m) and (n), which are taken with beam direction $\tilde{B} \approx [\bar{1}\bar{2}\bar{1}]$. This is consistent with segment 'A' being inclined upwards in the foil (looking along the line direction). The evident foreshortening of this segment (compare micrographs (c) and (o)) on tilting to $\tilde{B} \approx [\bar{1}\bar{1}\bar{2}]$ suggests that it is quite steeply inclined, and a comparison of all these micrographs, and others, indicates that the segment probably lies along $[3\bar{1}2]$. The complexity of the dislocation introduces some uncertainty in this measurement, however, and direction $[2\bar{1}1]$, or anywhere between the two, is also feasible.

Since the upwardly inclined segment 'A' lies in a region of the dislocation which was initially in pure-screw orientation, it indicates the onset of formation of a right-handed helix.

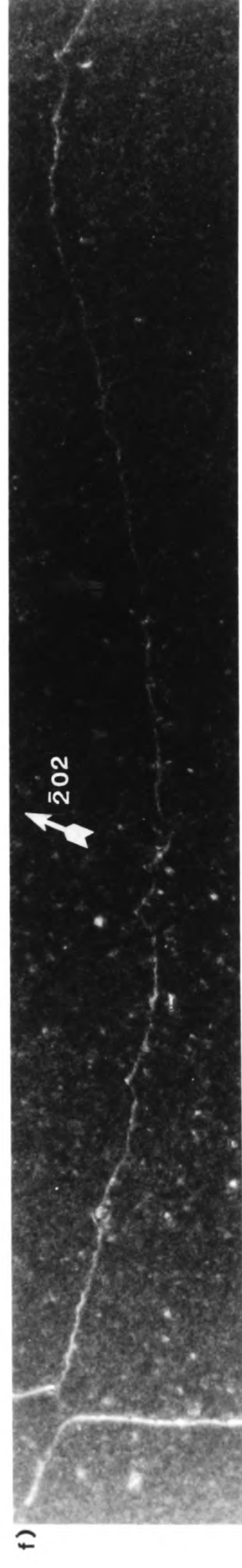
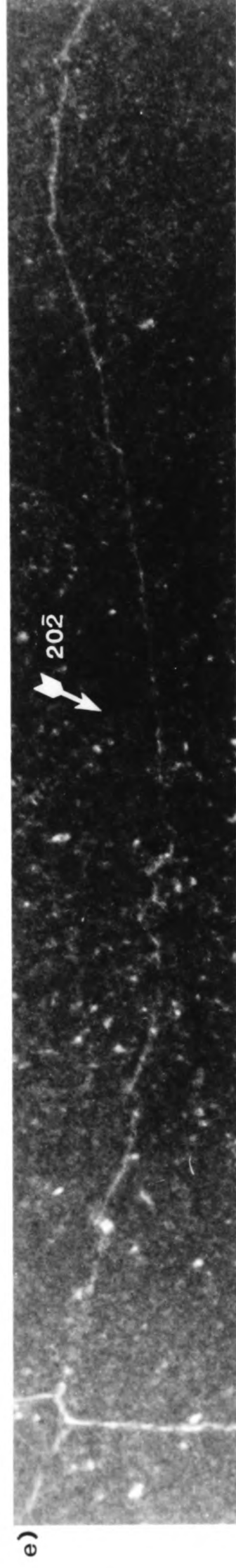
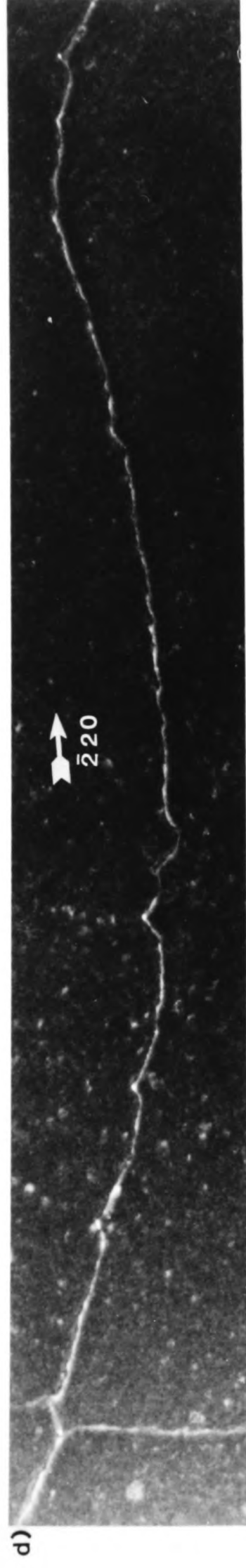
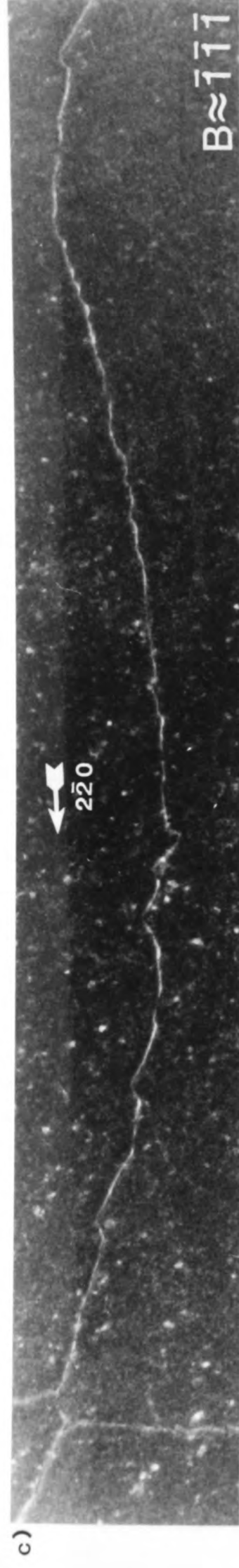
It is possible that one other segment of the dislocation also lies on a plane γ . A detailed study of the largest zig-zag, near 'B' (micrograph (o)), reveals a short, very straight segment which may also

²³The partial separation has apparently narrowed along near-screw sections.

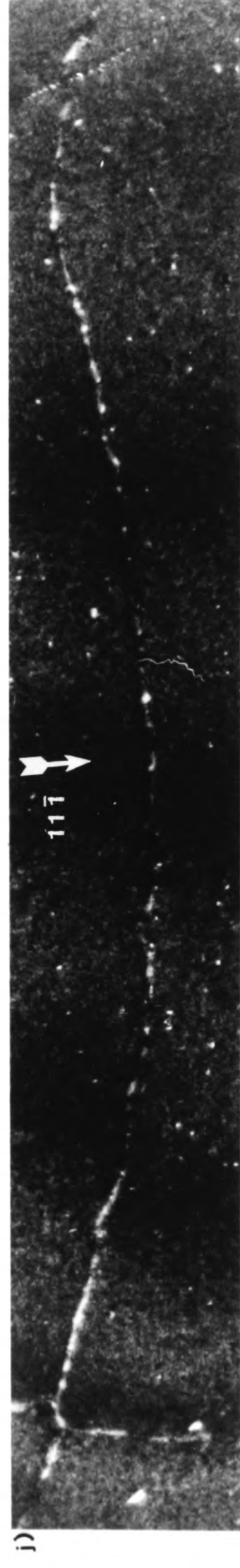
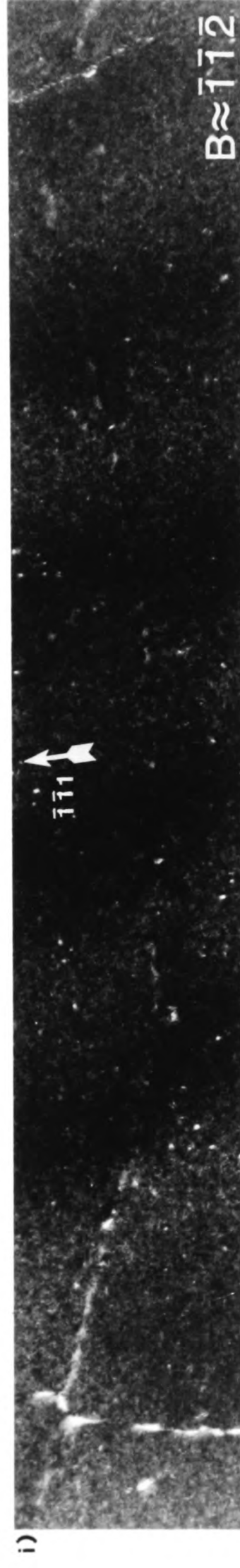
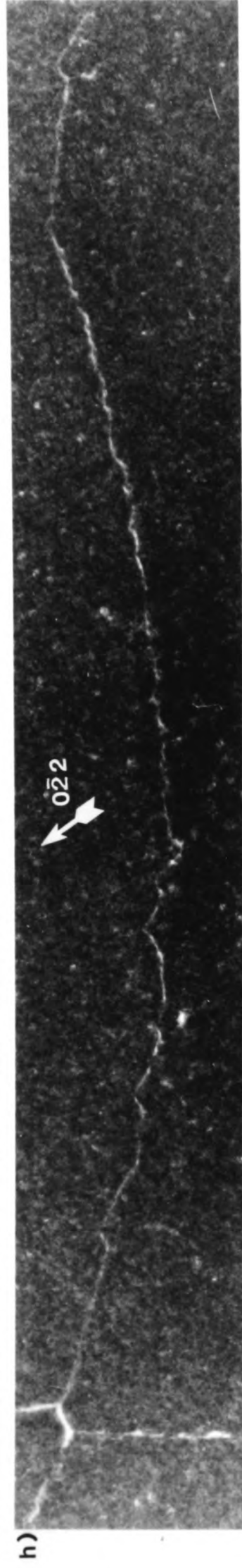
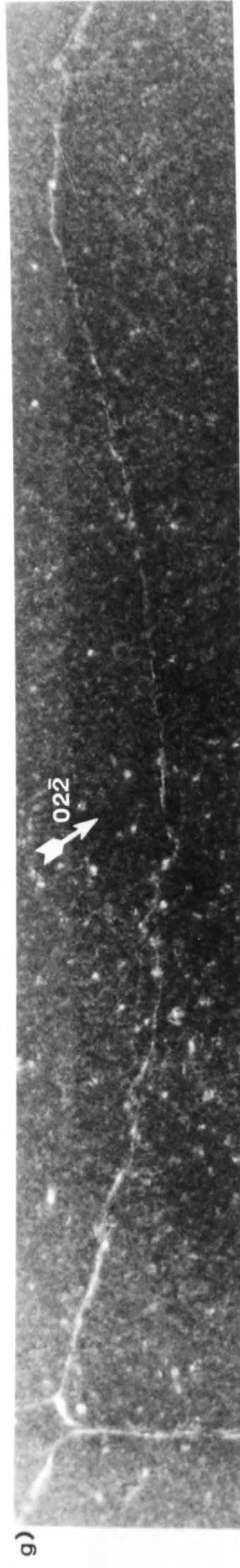
Figure 6.1
(5 pages)



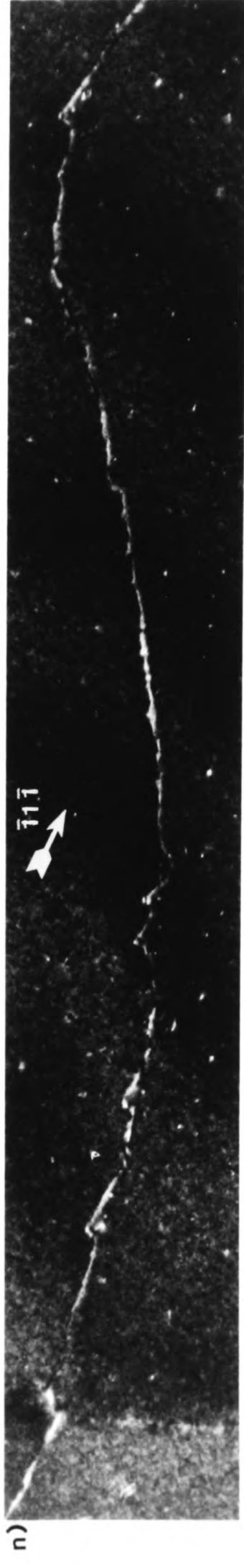
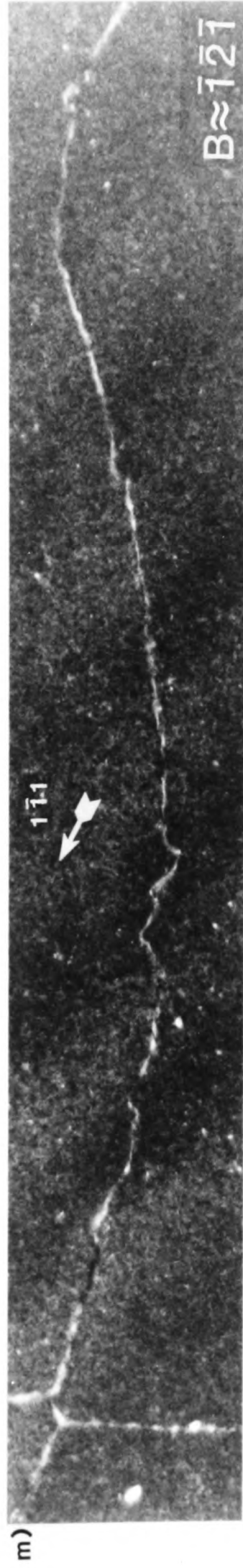
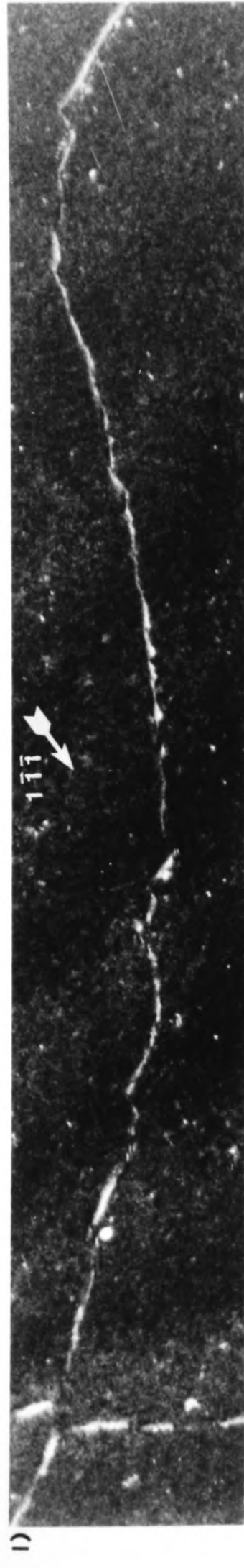
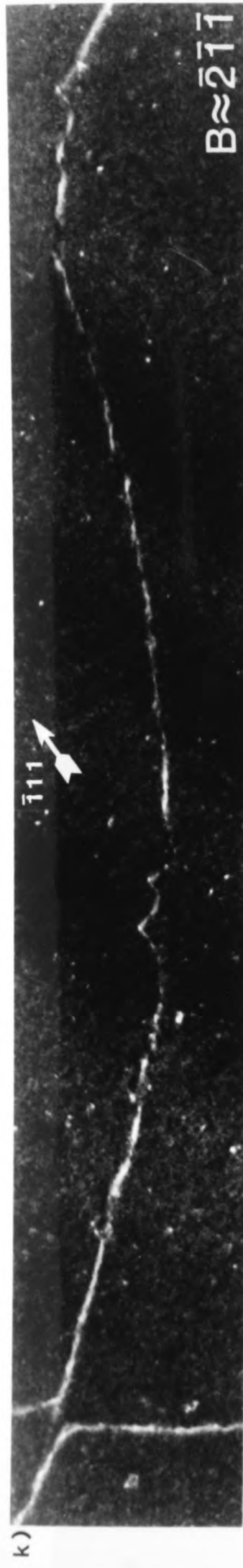
0° - 20° Dislocation in Fe-15%Ni-17%Cr-1%Si (at.%)



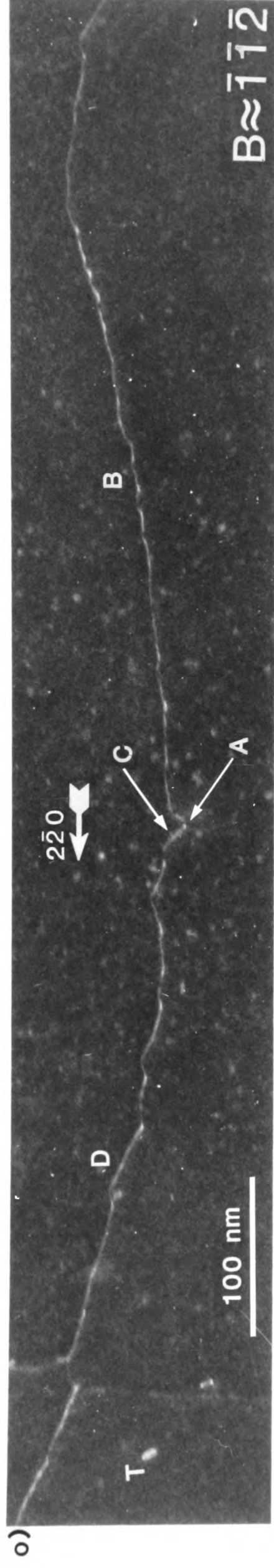
0° – 20° Dislocation in Fe-15%Ni-17%Cr-1%Si (at.%) 0.09 dpa at ~400°C



$0^\circ \sim 20^\circ$ Dislocation in Fe-15%Ni-17%Cr-1%Si (at.%) 0.09 dpa at $\sim 400^\circ\text{C}$



0° - 20° Dislocation in Fe-15%Ni-17%Cr-1%Si (at.%) 0.09 dpa at ~400°C



0° – 20° Dislocation in Fe-15%Ni-17%Cr-1%Si (at.%) 0.09 dpa at ~400°C

lie along $[1\bar{1}0]$. Comparison with micrograph (h) , for example, allows the possibility that this segment may be inclined, although its sense of inclination cannot be determined. Notice the substantially lesser extent of the zig-zagging along the entire length of the dislocation when seen in micrograph (o) , as compared with any other micrograph. At this tilt ($\tilde{B} \approx [\bar{1}\bar{1}\bar{2}]$), the fore-shortening of dislocation segments of mixed character lying on a plane δ is greater than for any other micrograph in which they are visible, suggesting that, for the majority of the dislocation at least, zig-zagging is confined to a plane δ .

Another difference between this dislocation and irradiated screw dislocations in other materials is the apparent formation of clusters close to the line. Neither the nature, nor the Burgers vectors of these clusters can be conclusively determined from the micrographs by standard techniques; notice, however, that the large cluster formed near 'D' is visible as an arrow-head in $\tilde{g} = [\bar{1}1\bar{1}]$ $((n))$. Other clusters near the 30° section of the dislocation at far right also show contrast suggesting that they are SFT.

The final point concerns cluster 'T' at far left. This cluster can easily be seen in every reflection of the contrast series apart from $\tilde{g} = \pm [02\bar{2}]$ (micrographs (g) and (h)). Initially this might suggest a Burgers vector of $\tilde{b}_L = \pm \tilde{\beta}\tilde{B}$. However, further study of the micrographs reveals, first, that it does not show inside/outside contrast consistent with a planar loop; second, since it is visible when the foil is tilted to bring planes β vertical $((k)$ and $(j))$ it is not confined to a plane β ; third, the onset of SFT formation is suggested by the shape of the cluster in micrographs (i) , (j) and (n) and fourth, whilst this feature is obviously very dim in micrographs (g) and (h) , it does not appear to be completely out-of-contrast in $\tilde{g} = [02\bar{2}]$ $((g))$. It seems likely, therefore, that this cluster is actually a vacancy SFT, in the process of forming via the dissociation onto inclined planes of a triangular, or hexagonal, $\tilde{b}_L = \tilde{B}\tilde{\beta}$ vacancy Frank loop. This unfauling process may be affected by nearby defect sinks (particularly surfaces, see **Jenkins**, 1973, for a full discussion).

Careful study of most other of the small background clusters visible in these micrographs also suggests that they are SFT. Interstitial background loops were not identified in this irradiation.

Figure 6.2; 25° Dislocation in Fe-15%Ni-17%Cr-1%Si; 0.09 dpa at $\sim 400^\circ\text{C}$.

Observations:

In this three page figure the second dislocation crossing at the node is shown. The majority of the dislocation is in 25° orientation and has Burgers vector $\tilde{b}_T = \tilde{B}\tilde{C}$, with line direction and extra half-plane as indicated on the second page of the figure. Micrograph (a) shows $|\tilde{g} \cdot \tilde{b}_T| = 2$ before irradiation, whilst all other micrographs are taken after irradiation. The pre-irradiation partial image separation at

'S' (25°) is $\sim (45 \pm 4) \text{ \AA}$. Dislocation 'M' is visible in all three $\langle 2\bar{2}0 \rangle$ type reflections (only 1 shown) in the (111) pole before irradiation and therefore has a Burgers vector lying in δ . In $\pm \tilde{g}$ of two of the three reflections this dislocation exhibited strong/weak contrast consistent with a dipole arrangement (see figure 1.3). Its individual components were never separately resolved and hence its Burgers vector remains unknown. The Burgers vector of the 25° dislocation $\tilde{b}_T = \vec{BC}$ appears unaffected by intersection with dislocation 'M'. To the left-hand side of the figure the complex multiple dislocation arrangement ('M1') is similarly indeterminate, although it must have total Burgers vector $\tilde{b}_T = \vec{BC}$.

As mentioned in text relating to the preceding figure, behaviour exhibited by the dissociated dislocation $\vec{BC}$ after irradiation is generally similar to that of other 20°-30° dislocations in the preceding three chapters. Contraction of the P_u node, consistent with the often seen narrowing of partial separations in chapter 3, appears to have resulted in, or resulted from, the glide of adjacent dislocations as indicated schematically in drawing (d). Dipole 'M' has apparently been annihilated and the simplification of section 'M1', to leave a single dissociated dislocation with Burgers vector $\tilde{b}_T = \vec{BC}$, is probably also the result of mutual annihilation.

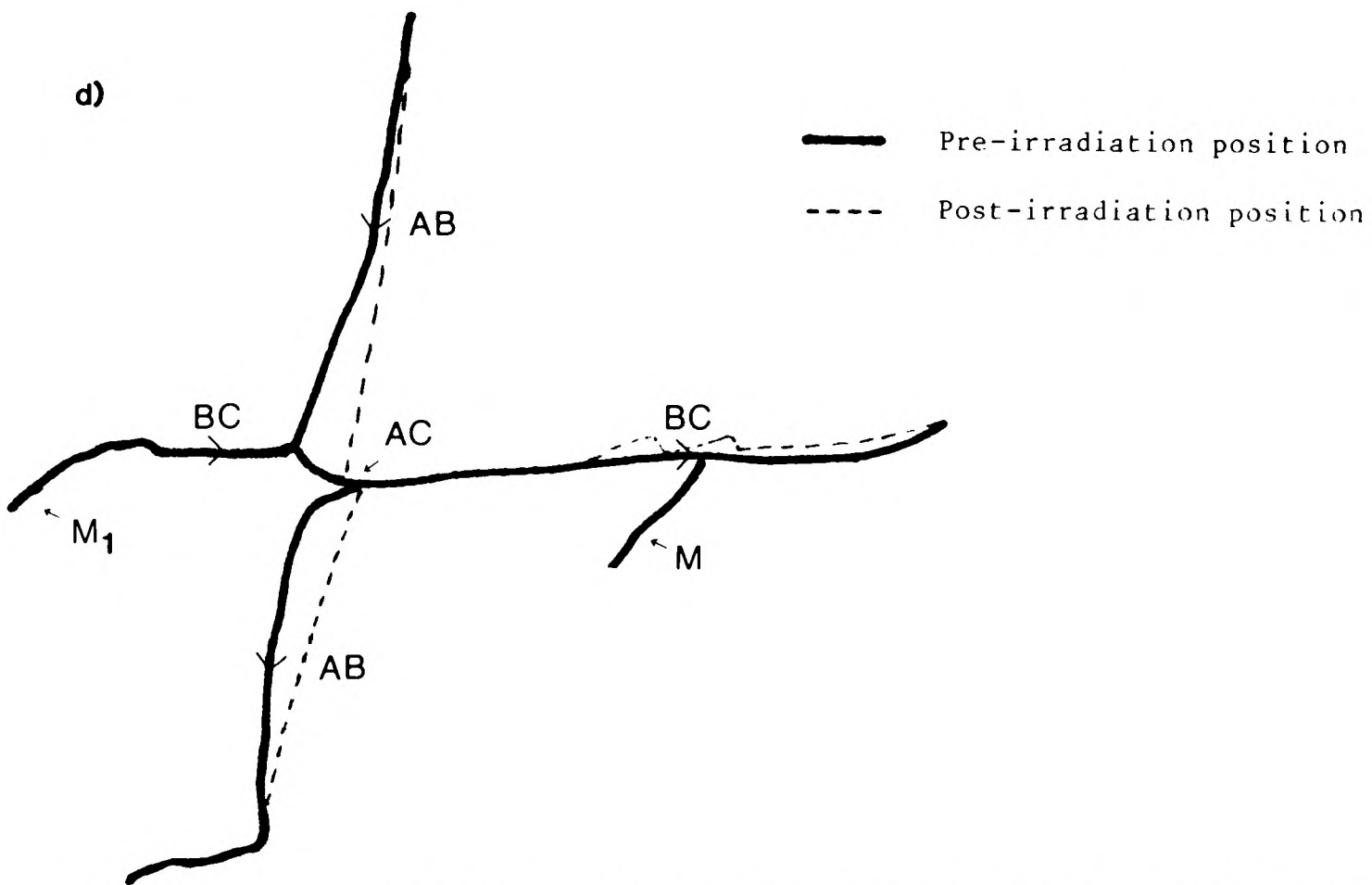
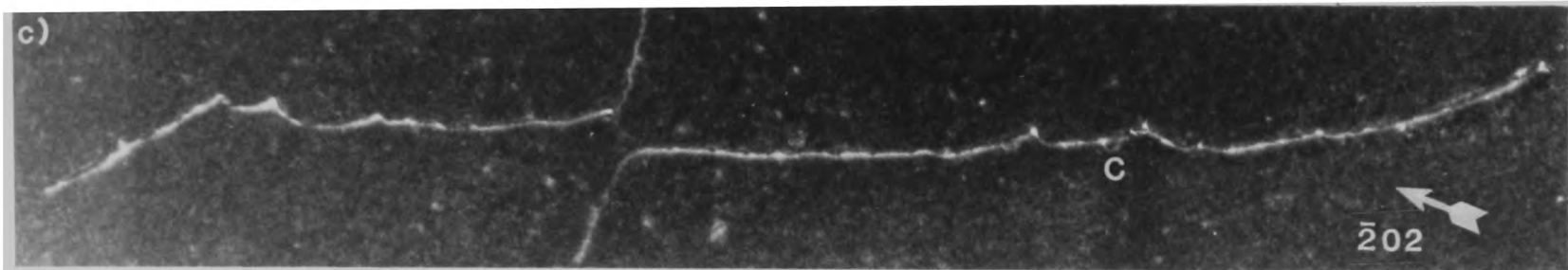
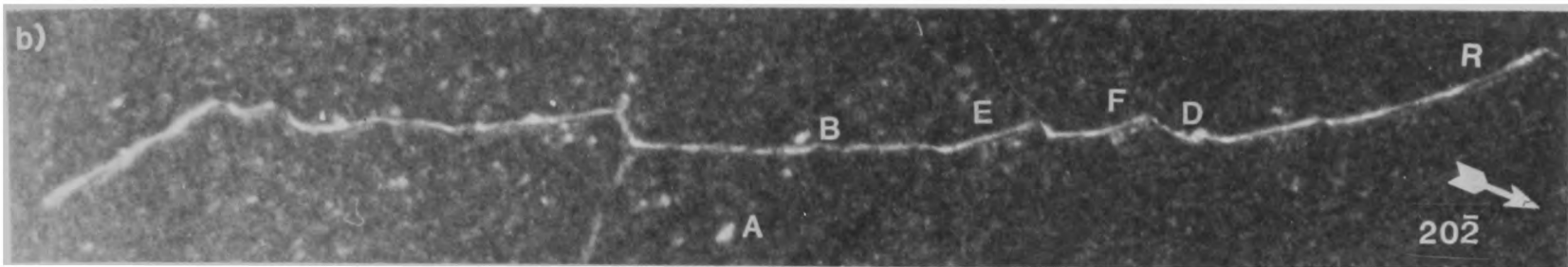
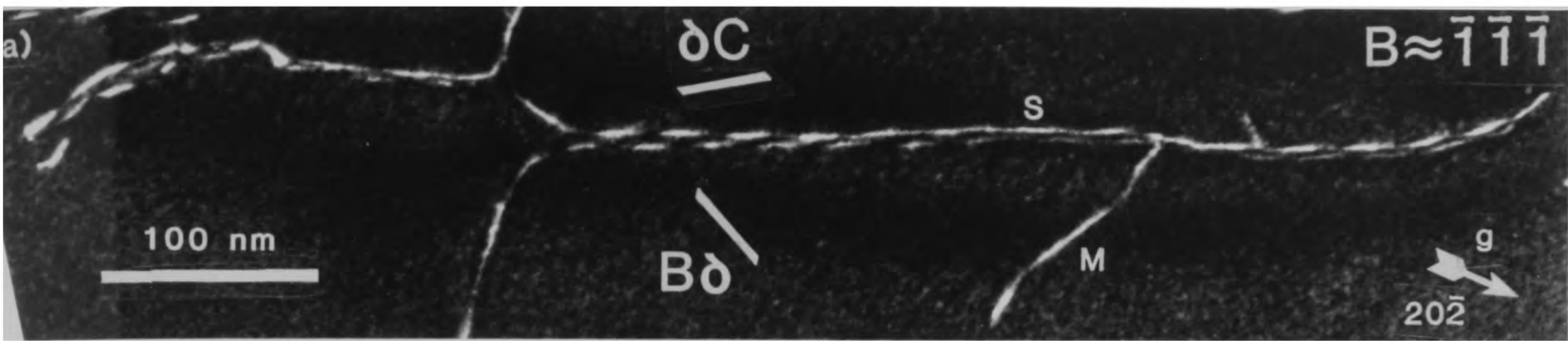
Dislocation $\tilde{b}_T = \vec{BC}$ can be seen to be still dissociated along almost all of its length in the post-irradiation $|\tilde{g} \cdot \tilde{b}_T| = 2$ images ((b) and (c)). This is confirmed by SFC in the $\tilde{g} \cdot \tilde{b}_T = 0$ image (m). The partial image separation has apparently narrowed, however, the formation of many new cusps, along an erstwhile evenly dissociated dislocation, prevents any measurement of the new equilibrium separation near these cusps. The post-irradiation partial image separation at 'R' (45°) is $\sim (30 \pm 4) \text{ \AA}$, less than the measured values of pre-irradiation separation for 25° line sections.

In common with figure 4.2, and many others, vacancy clusters are seen close to the pre-irradiation dislocation position. In contrast with that figure, however, these clusters do not seem to lie in as discrete a line; their configurations being somewhat 'messier'.

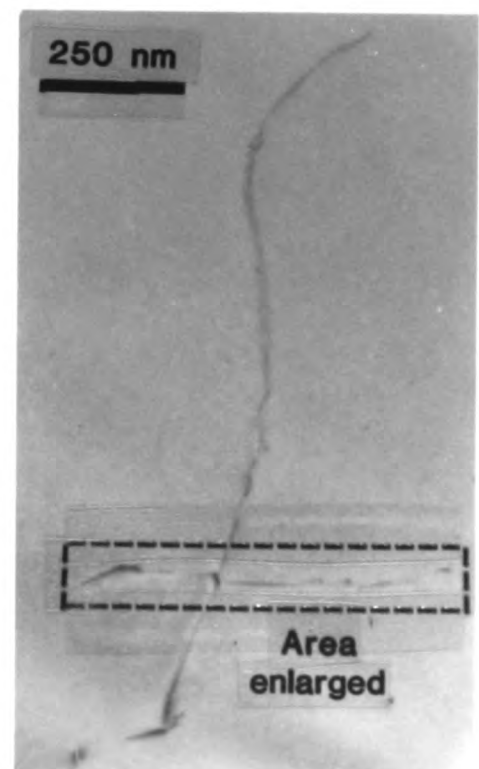
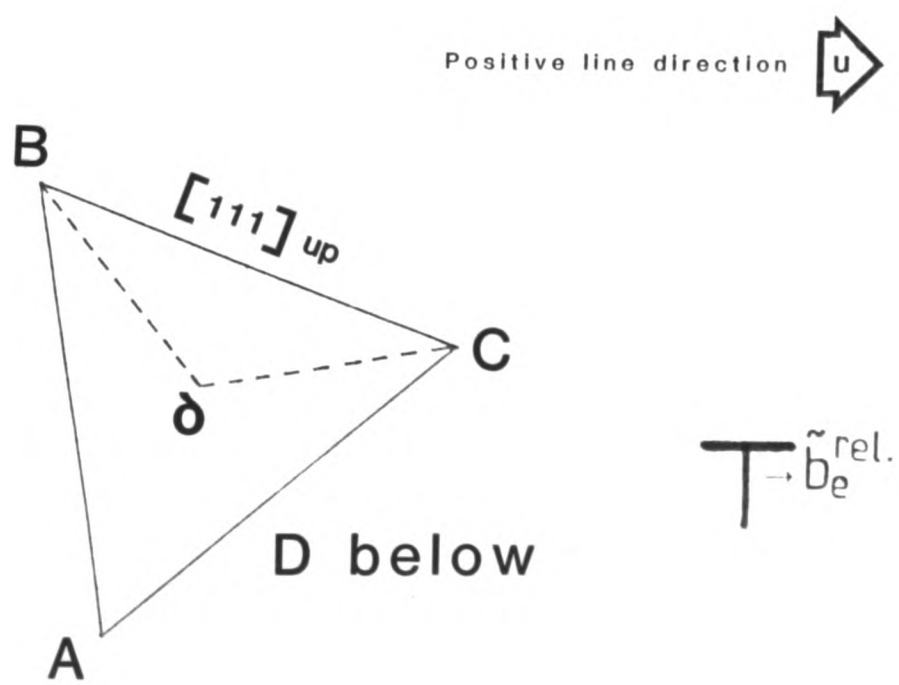
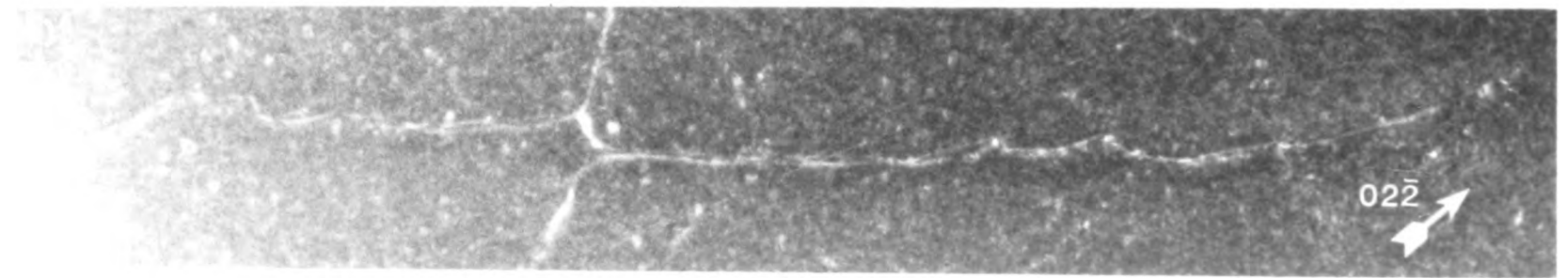
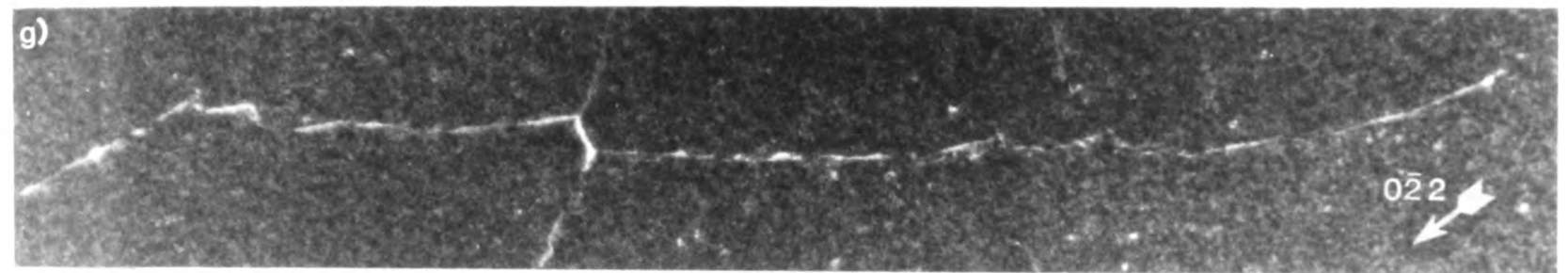
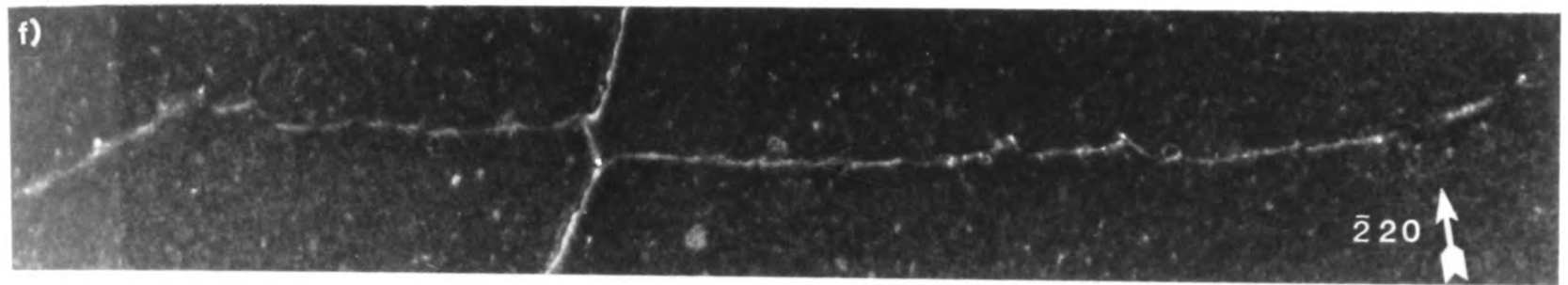
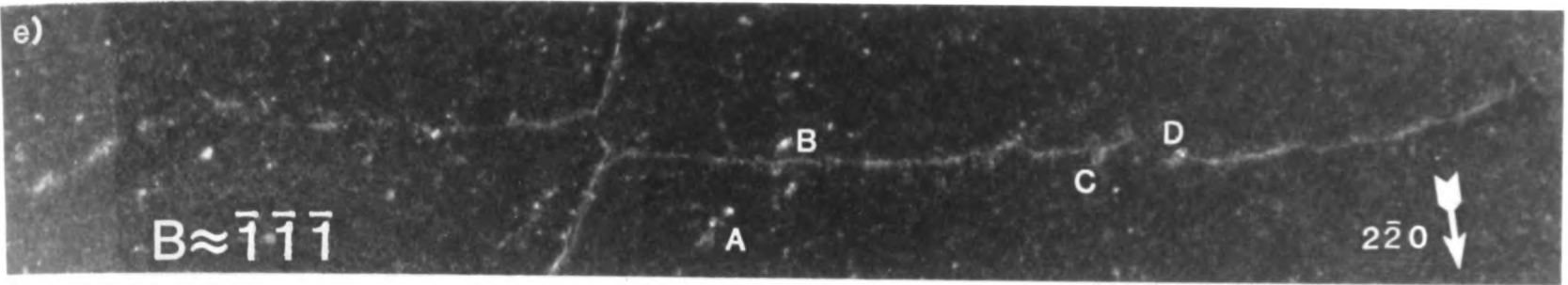
In figure 4.2, straight line dislocation sections, which had given rise to discrete lines of clusters, were found to lie *accurately* along $\langle 123 \rangle$ and $\langle 112 \rangle$ directions. This also appears to be the case for the upper partial $\vec{\delta C}$ at segment 'E', which appears to lie along, or very close to ($\pm 2^\circ$) the $[12\bar{3}]$ direction. Notice, however, that segment 'F' does not lie *exactly* parallel to segment 'E', although the misalignment is $< 5^\circ$. Both segments appear dissociated, and give rise to SFC (see (m)) and therefore lie on a plane δ . This leads to the conclusion that *one, the other, or neither* segment lies *exactly* along a $\langle 123 \rangle$ direction.

Micrograph (n) is a $\tilde{g} \cdot \tilde{b}_T = 0$ image which does not show SFC from dislocation $\vec{BC}$. This enables clusters forming close to the dislocation to be separately resolved. Notice that the clusters have

Figure 6.2
(3 pages)

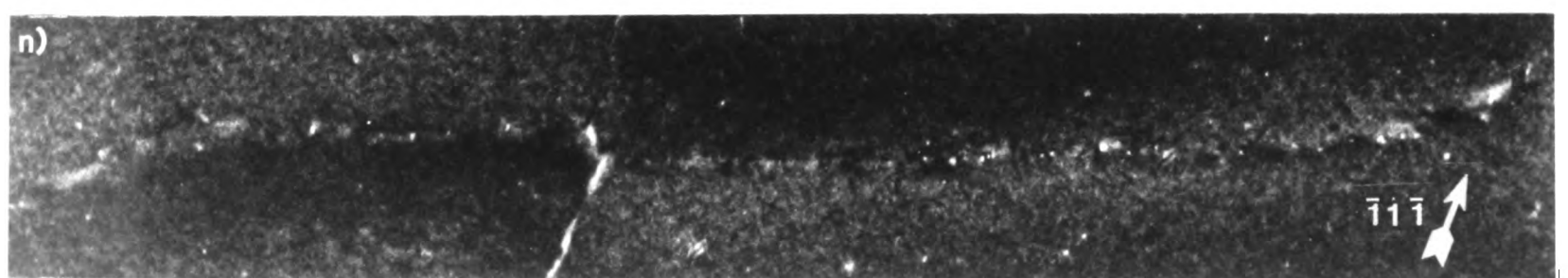
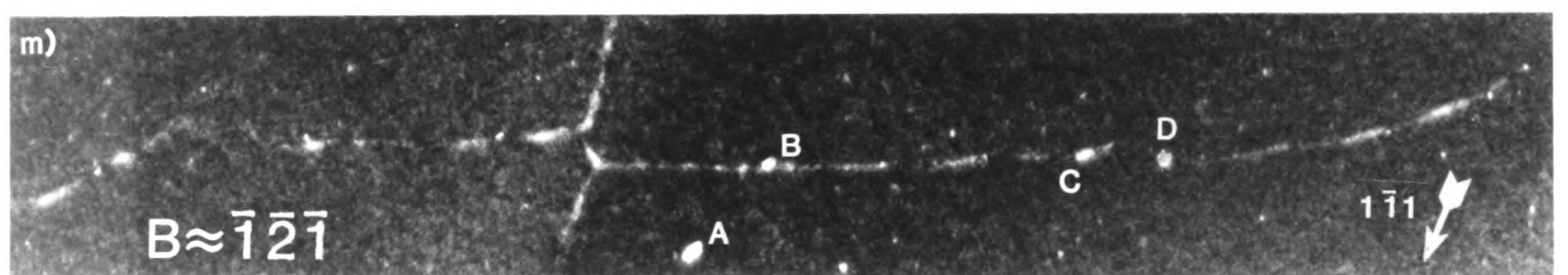
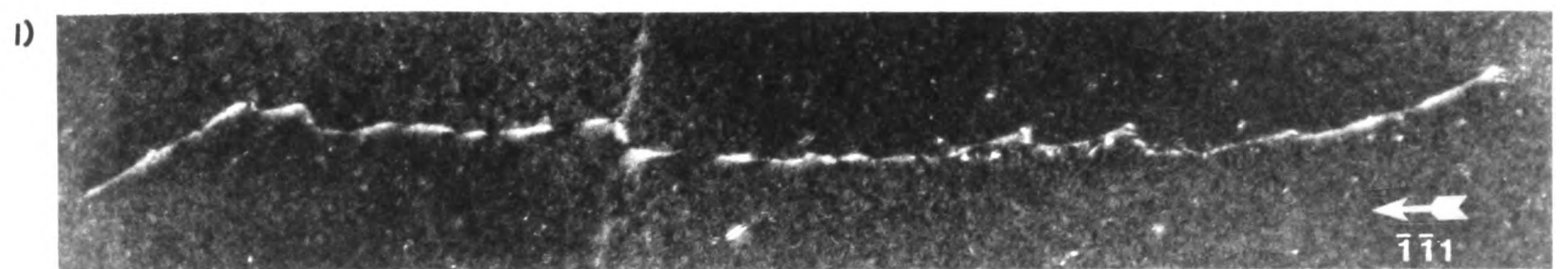
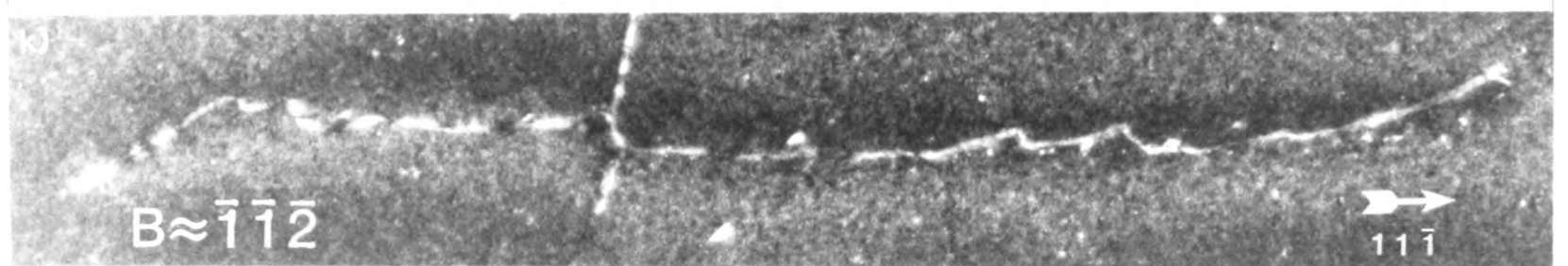
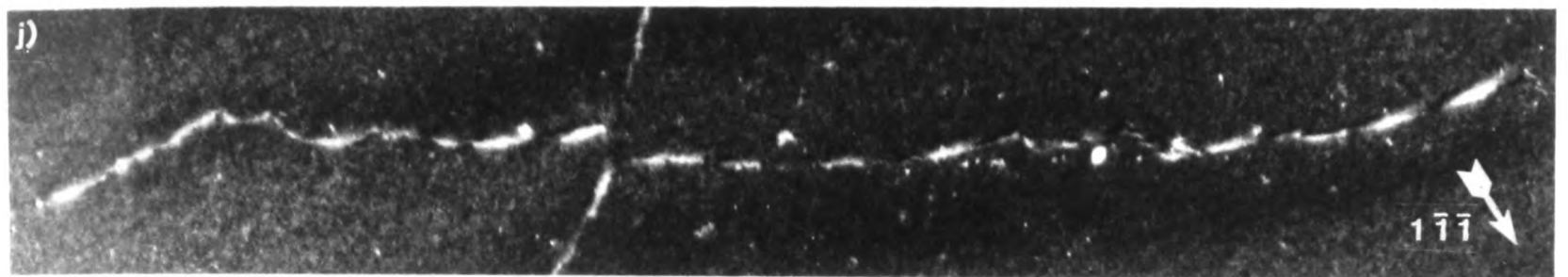
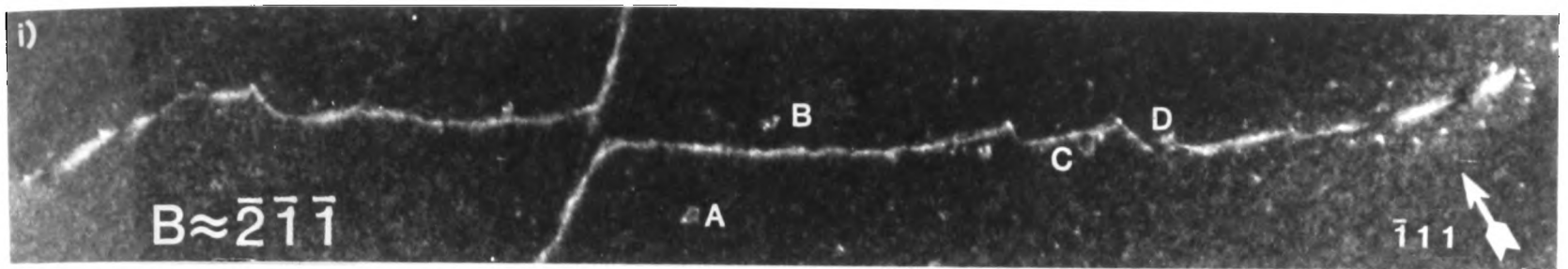


25° Dislocation in Fe-15%Ni-17%Cr-1%Si (at.%)
0.09dpa at ~400°C



25° Dislocation in Fe-15%Ni-17%Cr-1%Si (at.%)

0.09dpa at $\sim 400^\circ\text{C}$



25° Dislocation in Fe-15%Ni-17%Cr-1%Si (at.%)

0.09dpa at ~400°C

formed preferentially near to the section of dislocation $\overline{BC}$ which has zig-zagged.

In common with figure 6.1 all clusters are found to be vacancy in nature: Cluster 'A' is the same as that labelled 'T' (SFT) in figure 6.1. Clusters 'B', 'C' and 'D' also show contrast inconsistent with any planar perfect, or Frank loop variety and are occasionally visible as SFT (see, e.g. clusters 'B' and 'C' in micrograph (l); 'D' in (n)). Many of the small clusters also show 'arrow-head' contrast in at least one reflection.

Most of the clusters lying very close to the dislocation are small ($\sim 20-40 \text{ \AA}$ or so), whilst those away from the dislocation are generally larger. On tilting the foil (compare (f) and (j)) the larger cluster 'C' and the row of small clusters apparently lie very close below the dislocation ($< 10 \text{ nm}$. to its compressional strain field side). The large background cluster 'B', which is laterally displaced from the dislocation, is actually found to lie between 20 nm . and 30 nm . *above* the line.

Finally, no evidence is seen, either in the reflections shown or in $\langle 113 \rangle$ and $\langle 2\bar{2}0 \rangle$ type reflections taken in the various $\{112\}$ poles, for the existence of any inclined connecting segments separating 'zigs' and 'zags'. A $\tilde{g} = [\bar{2}02]$ reflection (not shown) taken in the (121) pole confirms the zig-zagging to be confined to a plane δ .

Figures: 6.1 and 6.2

Interpretation:

For these screw dislocations, the processes by which irradiation generated point-defects are interacting with the line appear similar to those already outlined. Some differences in behaviour are evident, however, the most notable of which is the formation of zig-zag configurations where no pre-existing jogs are seen. It would appear, therefore, that new jogs are being nucleated prior to the operation of the jog climb mechanism of figure 3.7. There may be a link between the nucleation of new jogs and the observation that, in general, formation of the zig-zag configurations is not very advanced.

The presence of vacancy clusters, and the lack of identifiable interstitial clusters, in the bulk is at odds with observations in the preceding three chapters; it would appear, in this case, that most interstitials are being lost from the foil at the surfaces. Some may be absorbed at the pre-existing dipoles 'M' and 'M1' and at new jogs on the dislocation, resulting in the onset of the right-handed helix seen to the centre of the micrographs of figure 6.1. The loss of most interstitials to foil surfaces may also explain the observation that zig-zag configurations are apparently in an early stage of formation.

The possibility that bulk SFT result from sub-threshold damage, in spite of the operation of the microscope at 200kV, either via processes outlined in chapter 3 or via the primary generation of a silicon atom PKO (see Zaluzec et al, 1985) may be discounted since they are not seen prior to HVEM

irradiation. SFT formation has been seen previously in irradiated austenitic steels (e.g., Muroga et al, 1988b, and see the lists in Stoller & Odette²⁴, 1988, and Zinkle et al, 1987) but appears to be a comparatively rare observation²⁵.

The observation that both of the two long kinks of figure 6.2 ('E' and 'F') do not lie exactly along a $\langle 123 \rangle$ direction may explain the 'messy' appearance of the row of nearby vacancy clusters. If $\langle 123 \rangle$ directions correspond only to shallow energy minima, or no minima at all, then the glide of the dislocations from initial to final positions mechanism may not be as rapid as depicted in diagram 4.3. Relaxation of the dislocation core by the nucleation of clusters within its strain field will may still prompt the formation of fairly discrete lines of clusters, however. The fact that one of these segments ('E') does appear to lie along $\langle 123 \rangle$ may suggest that these directions do actually still correspond to shallow energy minima.

Finally, the formation of small vacancy cluster damage appears to be linked with motion of the line; being densest where jogs have formed and the line has zig-zagged away from its pre-irradiation position. This is considered further later in the chapter ('Points of Discussion').

*Figure 6.3; Dislocations and Node in Fe-15%Ni-17%Cr-1%Si; Before Irradiation;
Introduction:*

Three dislocations meeting at a P_s node are shown, at low magnification, in figure 6.3. Three areas are enlarged from this region in the following three figures (figures 6.4, 6.5 and 6.6), containing 50° - 80° , 60° and 70° dislocations respectively. Each area includes the node as a reference point and each dislocation is indexed with the Thomson tetrahedron as shown. The dislocation of area 1 has Burgers vector $\tilde{b}_T = \vec{AB}$ such that both partials $\vec{A}\delta$ and $\delta\vec{B}$ are visible in this reflection ($|\tilde{g} \cdot \tilde{b}_T| = 2$). Partial $\delta\vec{C}$ is out-of-contrast. The Burgers vectors of individual partials, and dislocation line directions are shown in the inset at lower right. After irradiation, all three areas are again shown together for comparison in figure 6.9. During irradiation the HVEM beam was centred on the node, such that the furthest extent shown of area 2 lay at the beam periphery.

²⁴*Stoller & Odette attribute SFT seen in the bulk as resulting from their neutron irradiation. They do not appear to consider, however, the possible effect of sub-threshold vacancy injection, seen to lead to bulk SFT formation at room temperature (see figure 3.5 and related text), caused by their inspection of irradiated specimens at microscope operating voltages up to and including 300 kV.*

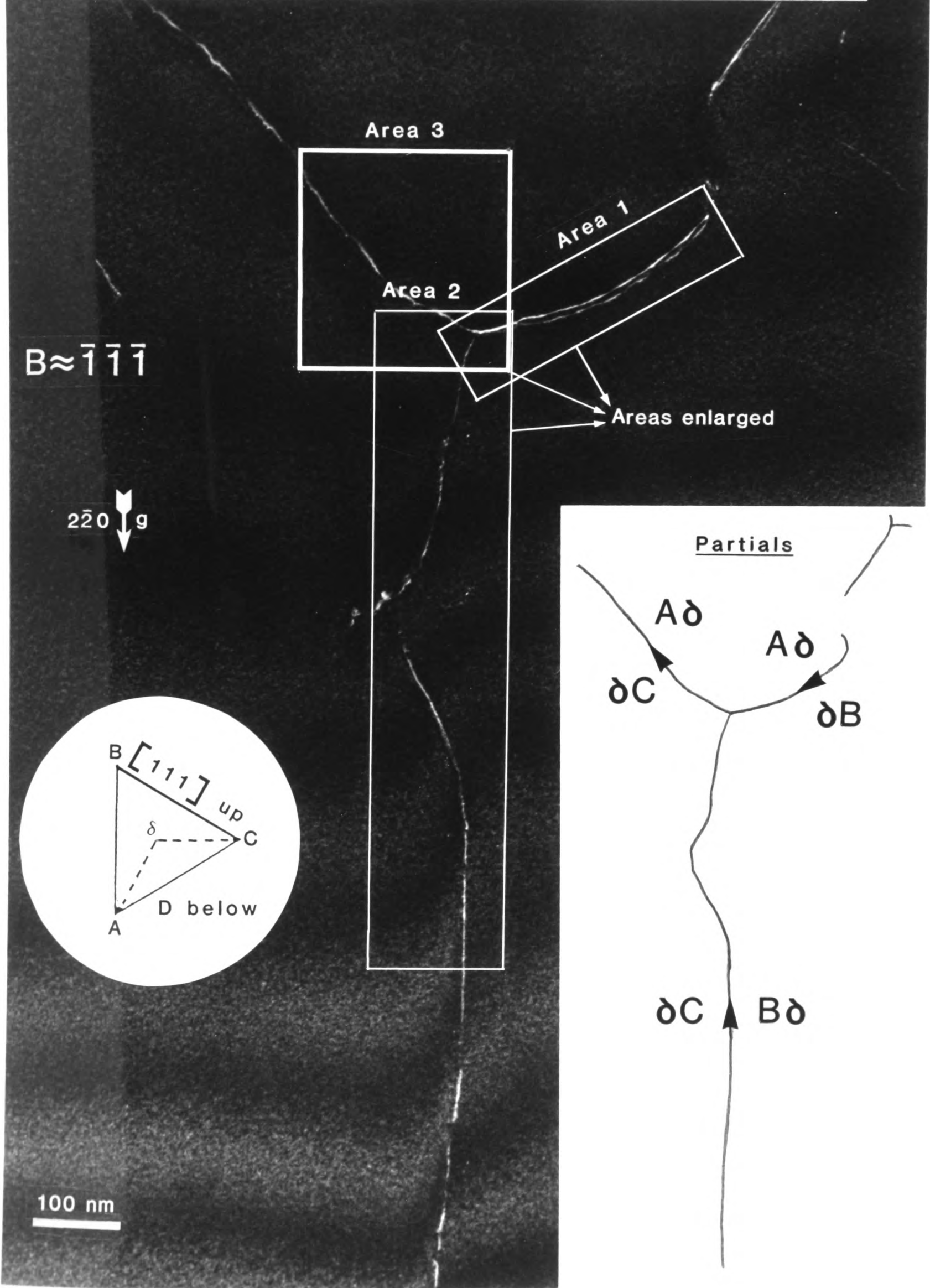
²⁵*Zinkle et al suppose the relative rarity of observations of SFT to arise from the presence of impurities, such as oxygen, which favour void, rather than SFT, formation. Under neutron irradiation, helium atoms may have a similar effect; this is mentioned briefly in chapter 8.*

Figure 6.3

Dislocations meeting at a Node

Before Irradiation

Dislocations and Node in Fe-15%Ni-17%Cr-1%Si (at.%)
Before Irradiation



‘Area 1’: Figure 6.4; 50-80° Dislocation in Fe-15%Ni-17%Cr-1%Si; 0.13 dpa at ~ 400°C.

Observations:

In this two page figure, both partials are visible in the pre-irradiation micrograph (a) which has $|\tilde{g} \cdot \tilde{b}_T| = 2$ for dislocation $\tilde{b}_T = \vec{A}\vec{B}$. All other micrographs ((b)-(n)) are taken after irradiation.

Most of the dislocation can be seen to be widely dissociated before irradiation ((a)). The partial image separation at ‘S’ (75°) is $\sim (78 \pm 4) \text{ \AA}$. The presence of a number of constrictions is illustrated by the cusped appearance of the left-hand (near-edge) partial $\vec{\delta}\vec{B}$. The large constriction at ‘T1’, at which the two sections of partial $\vec{\delta}\vec{B}$ at either end of this constriction do not meet at a point, is thought to be a super-jog (see below). The configuration of the dislocation at ‘T1’ is very reminiscent of observations of super-jogs along dissociated dislocations in low SFE materials (Carter, 1979 - see his figure 6a; 1990 and, with Hirsch, 1977)

After irradiation, the dislocation can clearly be seen to be still dissociated in micrograph (b). This dissociation has apparently decreased, with the partial image separation at ‘S’ now being only $\sim (47 \pm 4) \text{ \AA}$. Along most of the dislocation, pre-existing constrictions have been annihilated except near the node where the formation of new constrictions cannot be excluded. In the second page of the figure, a large number of small clusters are seen to have formed close to the evenly dissociated section of the line below ‘T1’. Many of these clusters may be seen as SFT in $\langle 11\bar{1} \rangle$ type reflections. Micrographs (h) and (i), taken with beam direction $\tilde{B} \approx [\bar{2}1\bar{1}]$, show this cluster damage to lie below the dislocation, and hence to its compressional strain field side. The magnitude of this displacement is consistent with most clusters lying approximately 15-20 nm. below the dislocation.

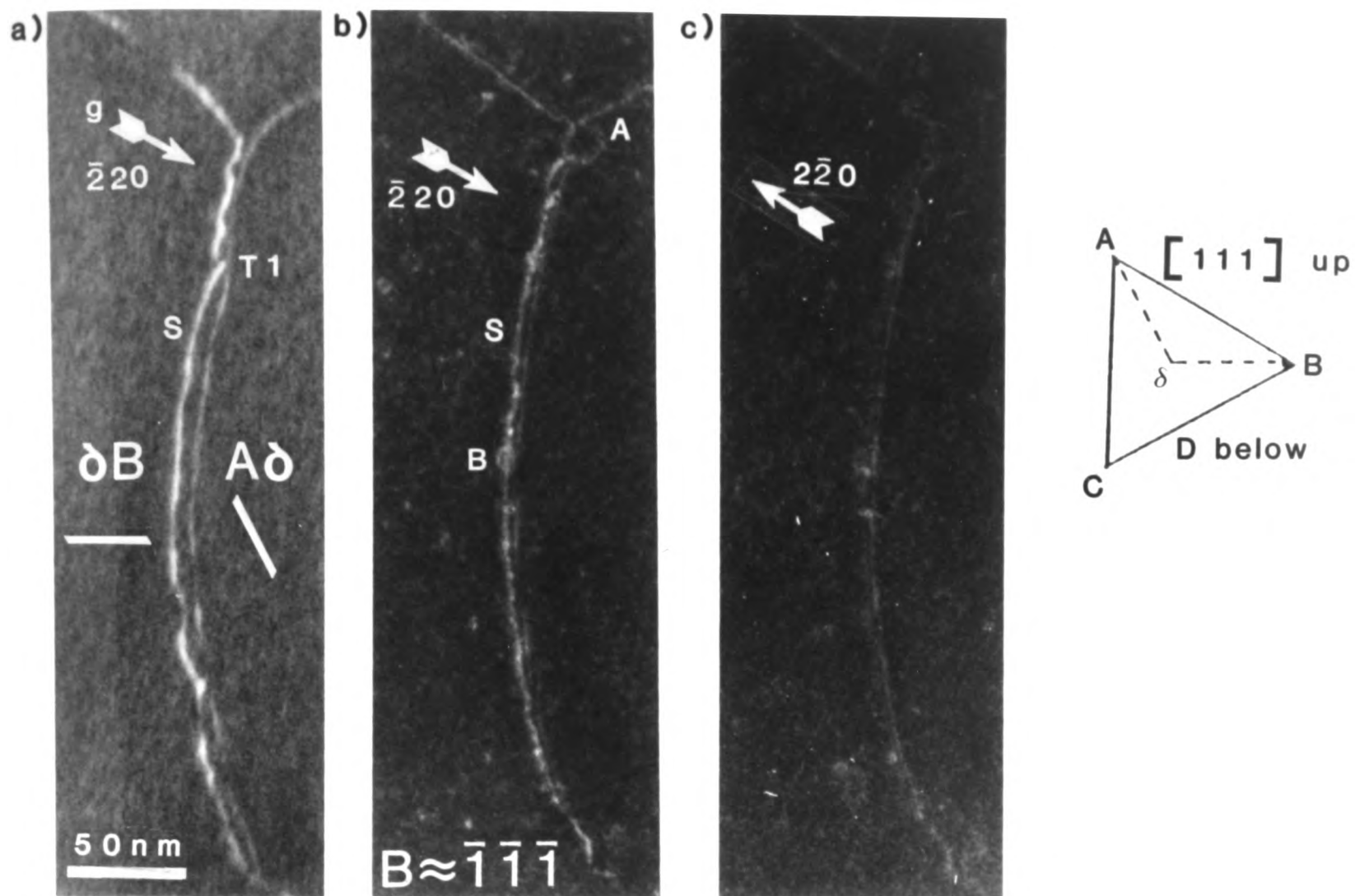
Two large SFT, at positions ‘T1’ and ‘T2’, are particularly in evidence in micrographs (h) and (i). These micrographs (particularly (i)) also show the presence of a large jog at ‘T2’. This jog is $\sim 5\text{-}6 \text{ nm}$. long in projection and is not visible in reflections taken with beam direction $\tilde{B} \approx [\bar{1}\bar{1}\bar{1}]$ suggesting that its line direction is along, or close to, $[\bar{1}\bar{1}\bar{1}]$. The lower part of the dislocation, as the figure is viewed, lies above the upper section (which includes the node).

At ‘A’ a short section of dislocation appears to have bowed out. This dislocation is visible in $\tilde{g} = [\bar{2}20]$ ((b)) and $\tilde{g} = [0\bar{2}2]$ ((g)) and may have Burgers vector $\tilde{b}_p = \pm \vec{A}\vec{\delta}$. It bounds a large faulted region which is visible in all $\tilde{g} = \langle 1\bar{1}\bar{1} \rangle$ type reflections.

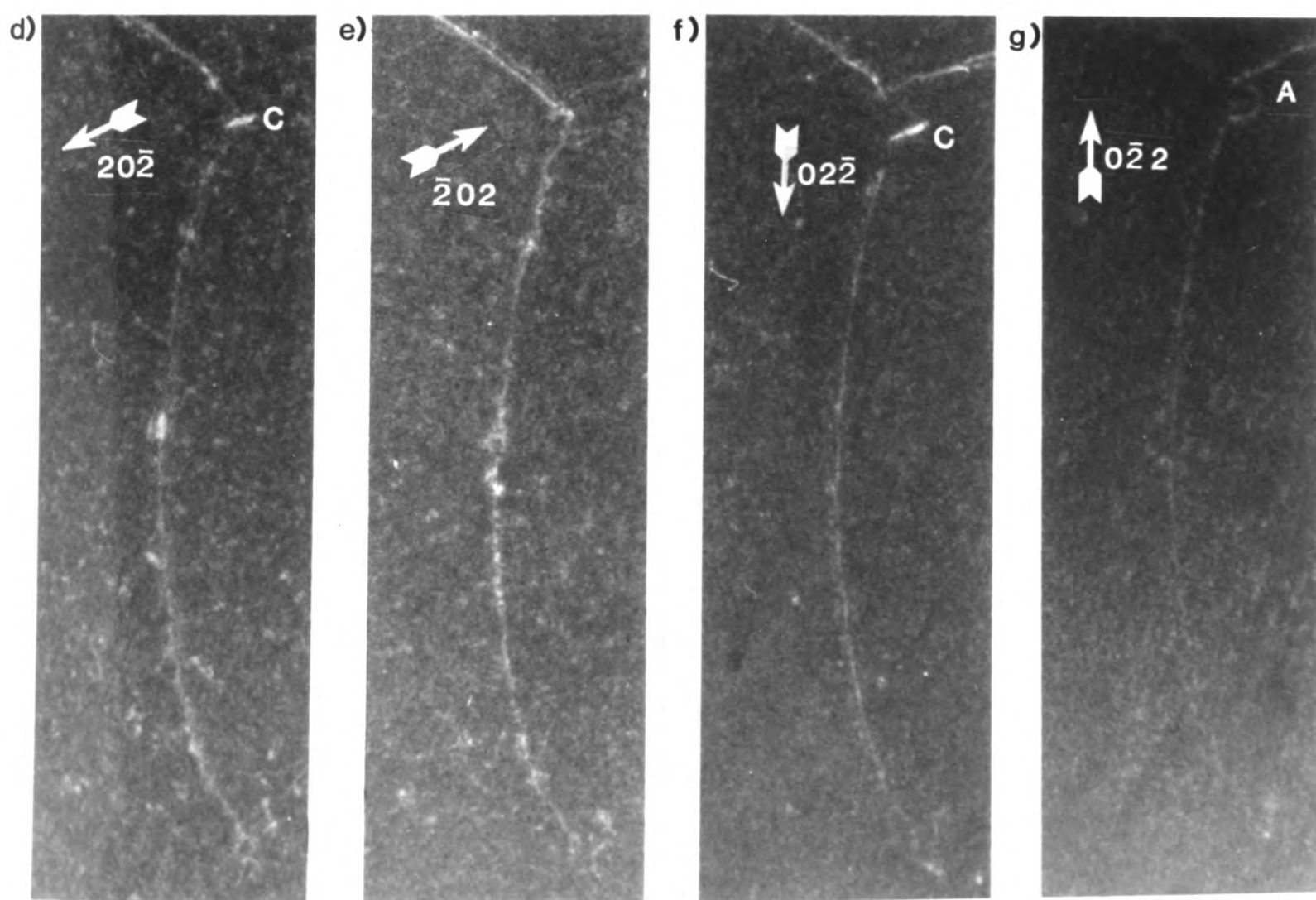
Feature ‘C’ appears to be associated with this bowed out section (‘A’) and exhibits contrast consistent with a small loop, or dipole (particularly on the first page of the figure). It is out-of-contrast in $\tilde{g} = \pm [\bar{2}20]$ and may also be out-of-contrast in $\tilde{g} = \pm [\bar{1}11]$ and $[1\bar{1}\bar{1}]$. It shows ‘inside’ contrast in $\tilde{g} = [20\bar{2}]$, $[02\bar{2}]$, $[11\bar{1}]$ and $[11\bar{3}]$. Stacking fault contrast is apparently absent within the region

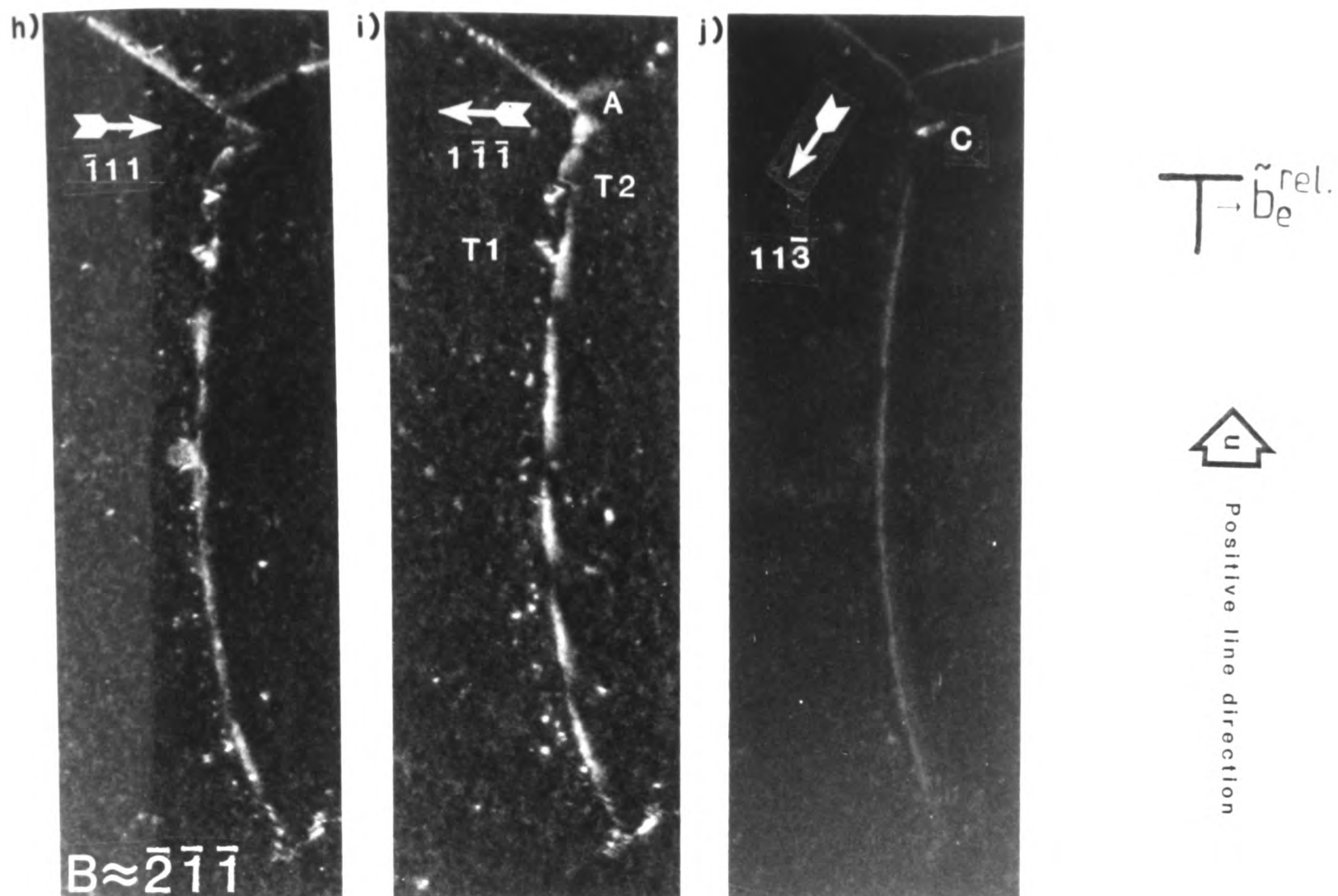
Figure 6.4
(2 pages)

‘Area 1’



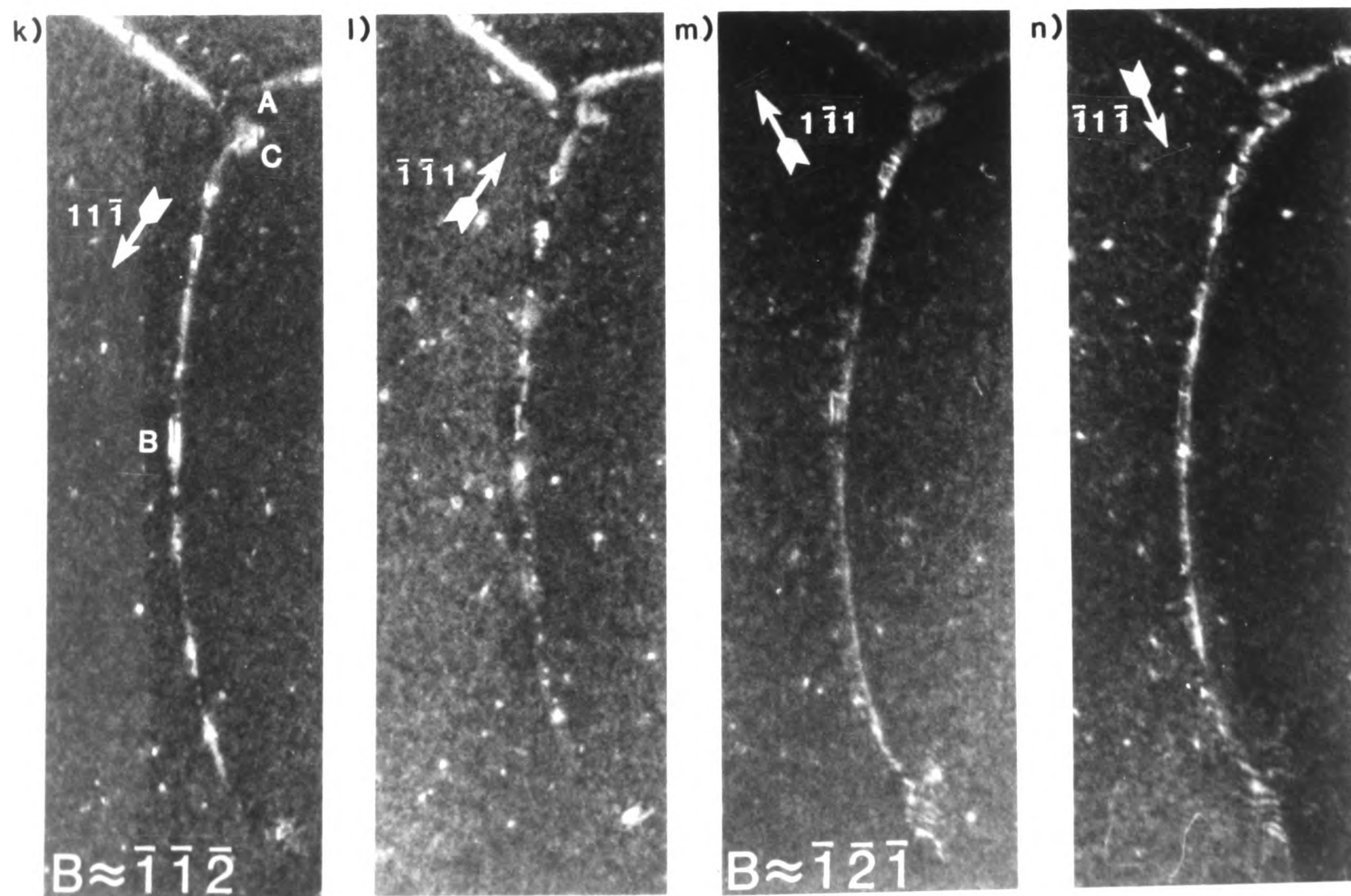
50–80° Dislocation in Fe–15%Ni–17%Cr–1%Si (at.%)
0.13 dpa at $\sim 400^\circ\text{C}$





50–80° Dislocation in Fe–15%Ni–17%Cr–1%Si (at.%)

0.13 dpa at $\sim 400^\circ\text{C}$



encompassed by this feature in micrograph (*k*), although it is difficult to be certain on this point. This behaviour is reminiscent of $\tilde{b} = \vec{C}\delta/\delta\vec{C}$ Shockley dipoles seen to form along HVEM irradiated dislocations in Cu-13%Al (Cherns et al, 1980).

In common with many previous figures, a large cluster has formed along a section of the line which is exactly in 60° orientation ('*B*'). This cluster appears to exhibit two kinds of contrast:

- 1) Stacking fault fringe-like contrast in micrographs (*k*) to (*n*);
- 2) Loop-like contrast in (*b*), (*d*) and (*h*).

Feature '*B*' is completely out-of-contrast in $\tilde{g} = \pm [02\bar{2}]$, suggesting a Burgers vector $\tilde{b} = \pm \vec{B}X$, where *X* is β , δ or D. It may be characterized by analysing the apparently separate components of contrast listed above:

1) The fringe-like contrast appears very similar to the SFT to be found elsewhere along the dislocation, particularly at '*T1*' in micrograph (*k*) and '*T2*' in micrograph (*m*). These fringes appear to result from a faulted region on a plane β , which lies parallel to the dislocation here.

2) The loop-like contrast, being visible in $\tilde{g} = [\bar{2}20]$, $[20\bar{2}]$ and $[\bar{1}11]$ and otherwise out-of-contrast, is consistent with Burgers vectors $\tilde{b} = \pm \vec{B}\bar{D}$ or $\vec{B}\delta$. Two observations suggest the latter Burgers vector ($\vec{B}\delta$) to be more likely, namely that this feature apparently shows SFC in micrograph (*h*), and that it is apparently out-of-contrast in micrograph (*j*), which has $|\tilde{g} \cdot \tilde{b}_T| = 1$ for $\vec{B}\bar{D}$ and $|\tilde{g} \cdot \tilde{b}_T| = \frac{2}{3}$ for $\vec{B}\delta$.

On tilting the foil normal (upward) from $[111]$ to $[211]$, feature '*B*' is apparently displaced to the left by a similar amount to the line of small vacancy clusters. Feature '*B*' is therefore probably a complex 3D vacancy cluster, part of which is forming into a large SFT from a planar loop on δ . This loop may be bounded by a Shockley partial dislocation with $\tilde{b} = \pm \vec{B}\delta$. A full discussion of weak-beam contrast arising from SFT in various stages of formation was given by Jenkins (1973 - his figure 6.16; 1974).

Finally, contrast from background clusters, which are only a few nm. in size, was found to be too unreliable for their natures to be determined via the inside/outside contrast method. Contrast from these features may be further complicated by depth effects, causing them to appear misleadingly faint, or completely out-of-contrast. A few of the larger background clusters, contained within the irradiated area shown by figure 6.3 and as covered by this series of three figures, apparently showed arrow-head contrast.

Interpretation:

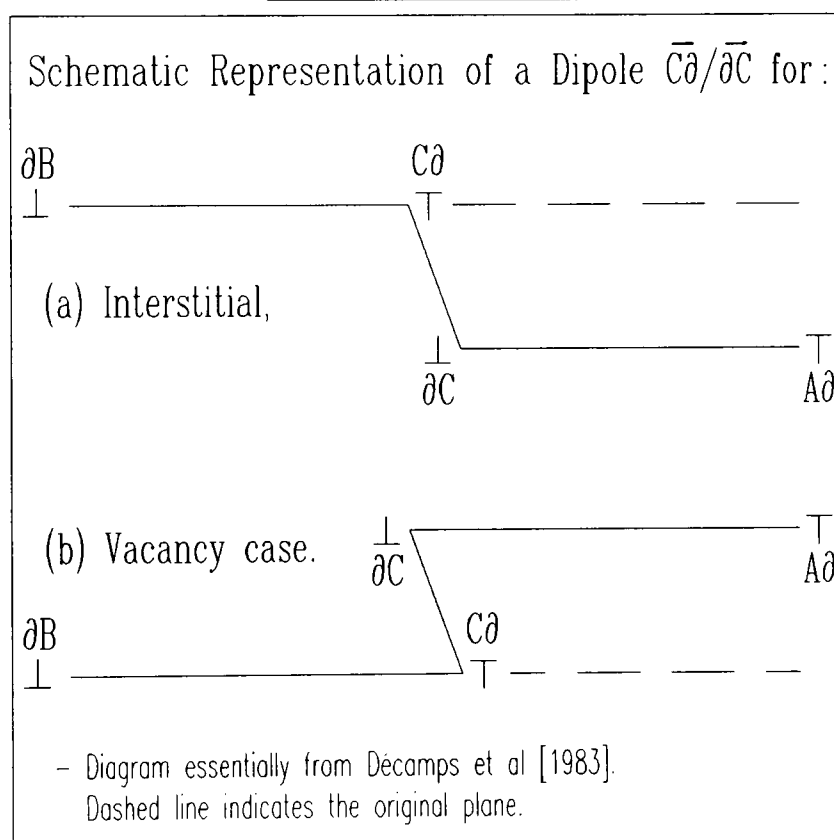
It would appear plausible that positions '*T1*' and '*T2*', at which large SFT have formed,

represent the pre- and post-irradiation positions respectively of the large super-jog. Since 'T2' is closer to the node than 'T1' this is consistent with interstitials attaching to the super-jog, causing its climb along the line. Climb of the super-jog may also have aided constriction of the node (Carter, 1980a). Unfortunately the super-jog is not directly imaged prior to irradiation, however, if one makes the assumptions that it lies vertically in the foil, and that its height (~ 15 nm.) is unaffected by the irradiation, one finds that a few thousand atoms (6000) are required to account for the super-jog's apparent motion. These few thousand atoms have attached to a $\sim (25 \pm 5)$ nm. section of dislocation. The figure of 6000 is, of course, extremely approximate but should represent an order of magnitude estimate.

The presence of the large vacancy clusters at 'T1' and T2' may indicate that the super-jog, which may be constricted (see $\tilde{g} \cdot \tilde{b}_T = 0$ images, particularly (l)), is acting as an efficient sink for interstitials. This efficient interstitial sink may then allow sufficient vacancies to aggregate to form these large clusters. This point is considered further, in the context of other irradiations performed in this material, within the 'Points of Discussion' section towards the end of this chapter.

Interstitials may also play a rôle in the formation of cluster 'C'. This cluster does not appear to overlap the stacking fault of the parent dislocation (see micrograph (l) and contrast this with feature 'B' in micrograph (b)). Consideration of geometry alone therefore suggests interstitial nucleation (see diagram 6.1). As outlined above, contrast exhibited by this feature appears most consistent with a $\vec{C}\delta/\delta\vec{C}$ dipole; this may be formed by the operation of the nucleation-rate controlled mechanism isolated by Cherns et al

Diagram 6.1



(1980) in HVEM irradiated Cu-13%Al. For the particular configuration shown in these micrographs, the operation of this mechanism also apparently leads to a plausible explanation, depicted in diagram 6.2 (see 'Points of Discussion'), for the bowing-out of the *locally nearer edge*, right-hand partial $\vec{A}\delta$.

The individual partials, near cluster 'B', were widely separated before irradiation. The nearer edge partial ($\vec{\delta B}$) may therefore be expected to predominate. It would seem unlikely, however, that the presence of a vacancy cluster as large as 'B' should result solely from bulk diffusion: Whilst partial $\vec{\delta B}$

will have the largest interaction with bulk diffused vacancies where it is pure-edge in character, it will also have a significantly large interaction in, say, the 85° orientation. Therefore, in the absence of core diffusion, large vacancy clusters might be expected to form along, say, 55° and 65° sections of dislocation $\vec{AB}$ as well. Hence, bulk diffusion alone does not appear to offer an adequate explanation of why such a large cluster should form *only* where dislocation $\vec{AB}$ is *precisely* in 60° orientation. The core diffusion drift mechanism of diagram 4.4, substituting vacancies for interstitials, therefore appears to be operating here.

Finally, it is believed to be significant that pre-existing constrictions have apparently been annihilated along most of the dislocation, and in this section no interstitial but many vacancy clusters are seen, whilst near the node, which has constricted and where pre-existing constrictions have not been annihilated, evidence consistent with interstitial precipitation is seen. This is considered further within the ‘*Points of Discussion*’ section.

‘Area 2’: Figure 6.5; 60° Dislocation in Fe-15%Ni-17%Cr-1%Si; 0.13 dpa at $\sim 400^\circ\text{C}$.

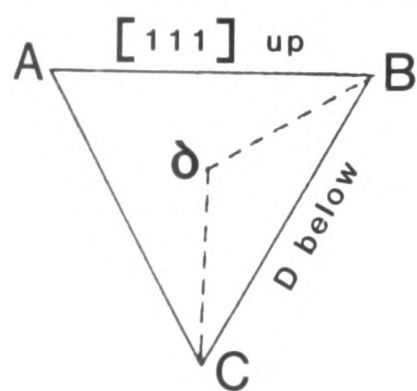
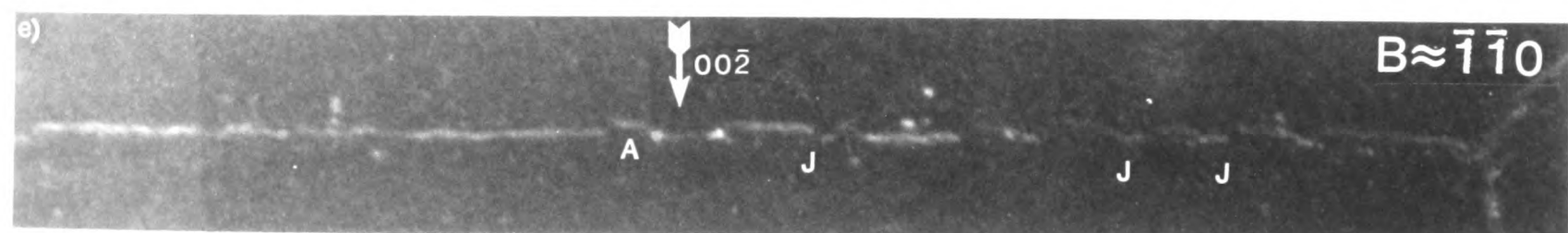
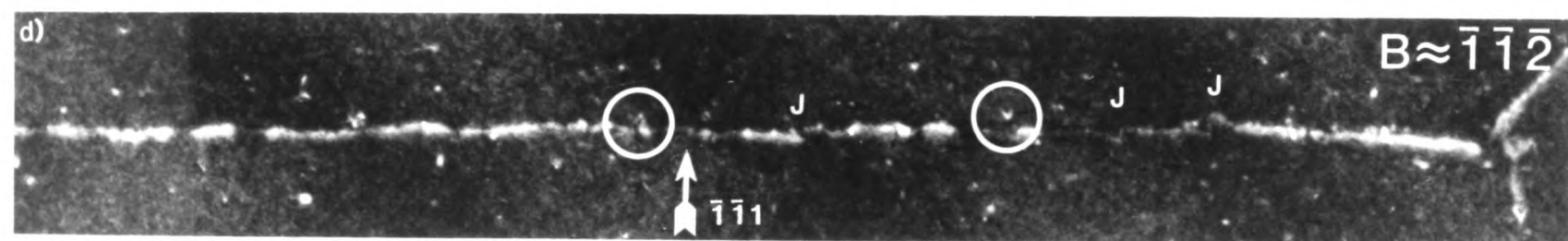
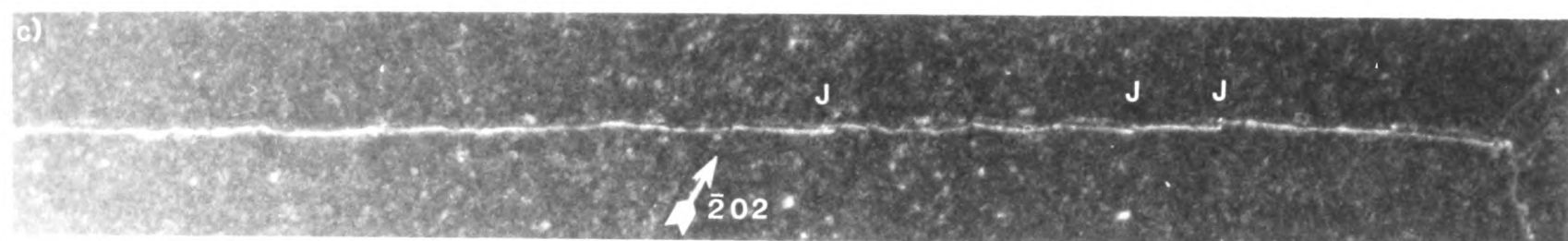
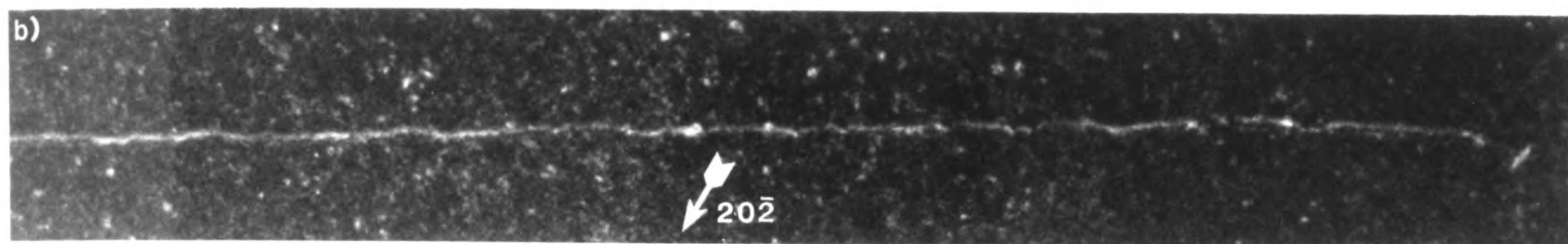
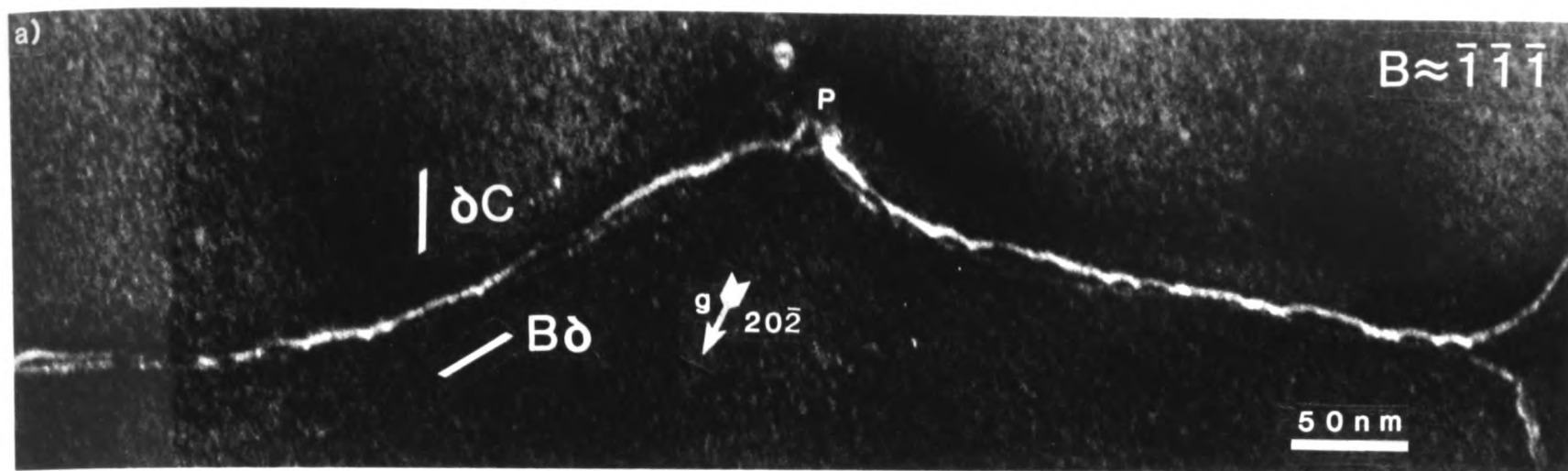
Observations:

The second area highlighted in figure 6.3 is shown. Micrograph (a) is taken before, whilst (b), (c) and (d) are taken after, irradiation. Micrographs (a), (b) and (c) all have $|\tilde{g} \cdot \tilde{b}_T| = 2$ for dislocation $\tilde{b}_T = \vec{BC}$. The node is shown at far right.

Before irradiation ((a)) the dislocation is constricted at many points along its length, and is apparently pinned at point ‘P’. In the same reflection after irradiation ((b)) it is obvious that large scale motion of the line has occurred. The dislocation is now approximately straight and lies close to the 60° orientation. This has apparently occurred via the downward motion (as the figure is viewed) of the centre section.

In the post-irradiation $|\tilde{g} \cdot \tilde{b}_T| = 2$ images (b) and (c), many constrictions are evident, although these seem to be fewer in number than before irradiation. The presence of these surviving constrictions makes any measurement of the new equilibrium separation unreliable, although the average partial image separation has apparently decreased. On tilting the foil away from (111) to (112) or (110), it may be seen that constrictions, particularly at the right-hand side (which was initially nearer edge orientation and which received a higher dose) actually represent jogs. The separation between jogs along this dislocation is generally $\sim 30\text{-}50\text{ nm}$. Three of the jogs are labelled (‘J’). In micrograph (d), the two right-hand jogs ‘J’ appear to be of the same sign; on tilting the foil to $\tilde{B} \approx [\bar{1}\bar{1}0]$, however, it may be seen that the section of dislocation between these, and many other jogs, actually lies above both the node and other sections of the line (particularly at left at left). In both (112) and (110)

Figure 6.5
‘Area 2’



Positive line direction



$$\vec{T} \rightarrow \vec{b}_e^{\text{rel.}}$$

60° Dislocation in Fe-15Ni-17Cr-1Si (at %)

0.13 dpa at ~400°C

projections individual jogs appear $\sim (10 \pm 5)$ nm. high. Since these jogs are both visible, and appear ‘higher’ when planes γ are vertical they indicate climb of the dislocation. Several sections of the dissociated dislocation therefore appear to have climbed nearly vertically in the foil.

A low density of clusters, which clusters are often recognizable as SFT (ringed) and so are likely to be vacancy, is seen to form close to the dislocation. These clusters lie within the compressional strain field of the dislocation, and often appear to lie close to a jog although jogs are seen without clusters. The density of vacancy clusters here is significantly lower than that seen near the dislocation of figure 6.4.

Finally, large vacancy clusters, such as ‘B’ of figure 6.4, are not seen along 60° sections. The Burgers vector of the dislocation appears to be unaffected by the irradiation.

Interpretation:

The possibility that these, apparently constricted, super-jogs should have been present prior to irradiation is shown to be unlikely in the following figure (figure 6.6). The jog climb mechanism of figure 3.7, in which jogs climb together to annihilate, therefore does not seem to be operating here. Instead, it would appear either that jogs have coalesced to form super-jogs, spaced at roughly 50 nm. and approximately 10 nm. in height, or that nucleation, leading to climb, has occurred on sections of the dissociated dislocation. The coalescence of jogs to form super-jogs would be, seemingly, a very surprising result since super-jogs are not expected to be stable unless dissociated (see **Hirth & Lothe**, pp. 265 and 577). Dissociation of the jogs is not seen either in the $|\tilde{g} \cdot \tilde{b}_T| = 2$ ((b) & (c)) or $\tilde{g} \cdot \tilde{b}_T = 0$ images (not shown), although they may be extended by an amount beyond the resolution limits of the weak-beam technique (effectively ~ 30 Å here). Since evenly spaced partials are seen to be dissociated by only ~ 40 -50 Å after irradiation (see figure 6.4), extension of the super-jogs, which has not been imaged, would appear possible²⁶.

Lastly, one section of the dislocation which appears to have climbed (near ‘J’ at centre) apparently corresponds to that section which was pinned at ‘P’. Unpinning of the dislocation may therefore have occurred via the climb of this line section around the pinning obstacle. The dislocation may then straighten by glide.

²⁶Alternatively, notice that these super-jogs are most in evidence to the right of ‘P’ where the dislocation was initially closer to edge orientation. Jogs along this section of line will, hence, experience the highest interaction with any local super-saturation, perhaps aiding them to overcome jog-jog repulsion forces (**Hirth & Lothe**, p. 264). A possible absence of core diffusion along this section of dislocation, alluded to within the ‘Points of Discussion’ section towards the end of this chapter, may have a significant bearing on the stability of these super-jogs (see **Hirth & Lothe**, p.577).

These micrographs alone do not enable the sense of dislocation climb to be definitely determined, since it is not known whether the ‘higher’ sections have climbed up, or the ‘lower’ sections have climbed down. The observation that several sections now lie above the planar P_s node might suggest interstitial climb, whilst the theoretical preference for vacancy precipitation at extended super-jogs (see **Hirth & Lothe**, p.582 and **Argon & Moffat**, 1981) might suggest the opposite. This question is considered within the ‘*Points of Discussion*’ section.

‘Area 3’: *Figure 6.6; 70° Dislocation in Fe-15%Ni-17%Cr-1%Si; 0.13 dpa at ~ 400°C.*

Observations:

The final dislocation intersecting at the node of figure 6.3 has Burgers vector $\tilde{b}_T = \vec{AC}$. The node is shown at the lower right in each micrograph. In micrograph (a) (before irradiation), $|\tilde{g} \cdot \tilde{b}_T| = 2$ and the partials are seen to be fairly evenly, and widely, dissociated. The partial image separation at ‘S’ (70°) is $\sim 70 \text{ \AA}$. A few constrictions, such as ‘A’, are also evident but super-jogs are apparently absent. Micrographs (b) to (i) are taken after irradiation.

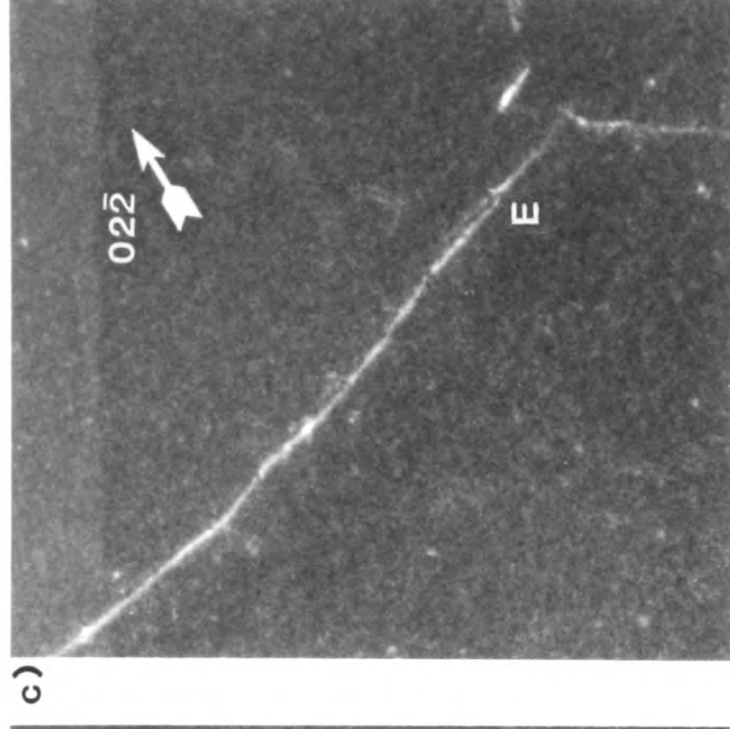
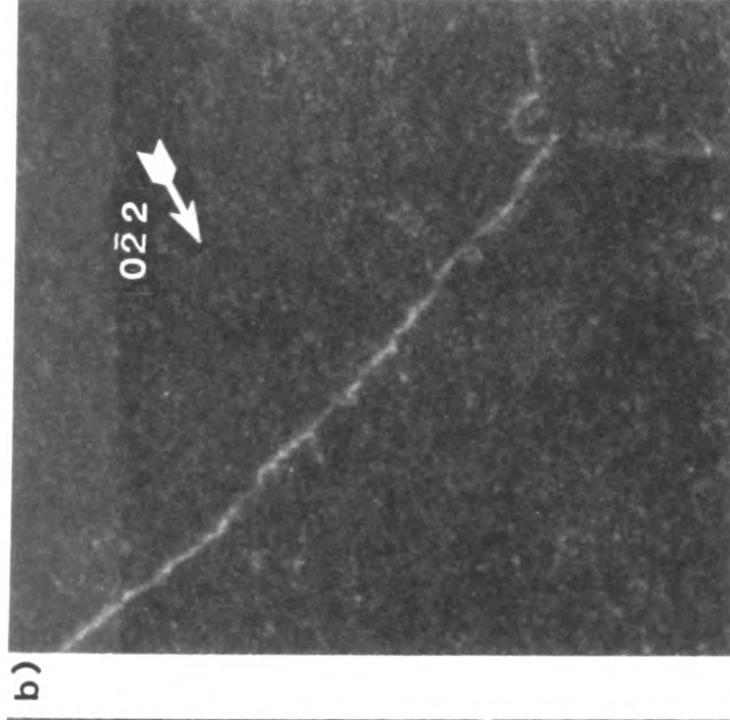
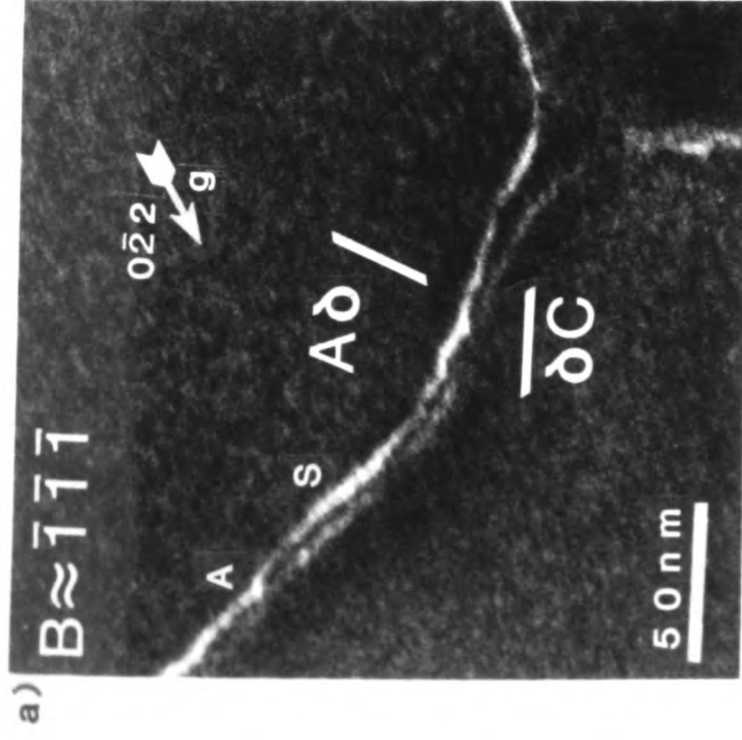
In micrograph (b), the upper (near-edge) partial $\vec{A\delta}$ is the brighter. This partial now appears heavily cusped, indicating the formation of many new constrictions. The dislocation has straightened and now lies close to, or in the 60° orientation. In micrograph (c), a large constriction ‘E’ is marked; above this point both partials $\vec{A\delta}$ and $\vec{\delta C}$ are apparently visible along a short length of the dislocation. This constriction apparently corresponds to a gap in the stacking fault contrast of micrograph (d); notice that SFC appears along much of the dislocation in (d), suggesting that many sections are dissociated.

The $\tilde{g} \cdot \tilde{b}_T = 0$ images (d) and (e) clearly show the presence of a low density of vacancy cluster damage close to the line. Significantly fewer vacancy clusters are visible here than are seen close to the evenly dissociated section of the dislocation of figure 6.4. Two SFT are arrowed; these SFT appear to lie close to large jogs visible in other reflections (such as (f)). Other SFT are also shown in (d) and (e): ‘T’ appears to lie some distance from the line, but may actually indicate the pre-irradiation position of the upper partial $\vec{A\delta}$; similarly two clusters visible at ‘A’, one of which is recognizable as an SFT, may lie close to the position of the pre-existing jog ‘A’. Cluster ‘T2’ is the same SFT as that shown in figure 6.4.

The nature of the jogs is clearly shown in the second page of the figure, in which the tilt between micrographs is $\sim 55^\circ$ and in which jogs are labelled ‘E’, ‘F’, ‘G’ and ‘H’. Micrographs (h) and (i) apparently suggest that sections ‘EF’ and ‘GH’ lie above the rest of the dislocation, and above the node. These sections, particularly ‘EF’, also exhibit strong/weak contrast in $\pm \tilde{g}$ which may be

Figure 6.6
(2 pages)

‘Area 3’

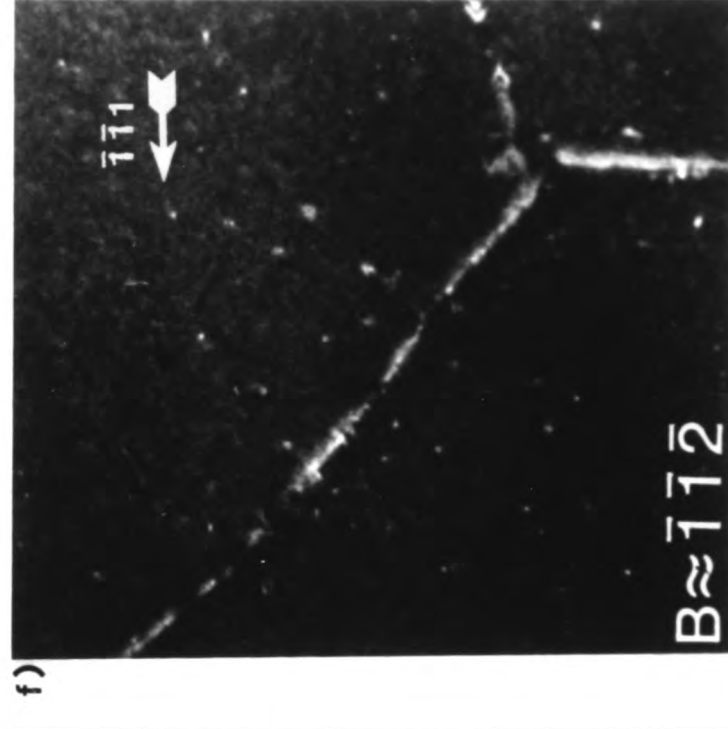
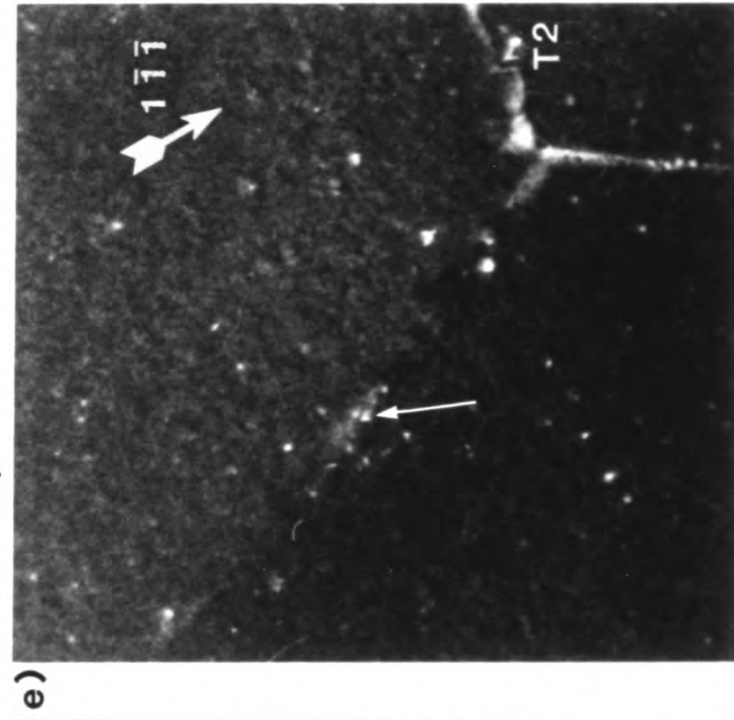
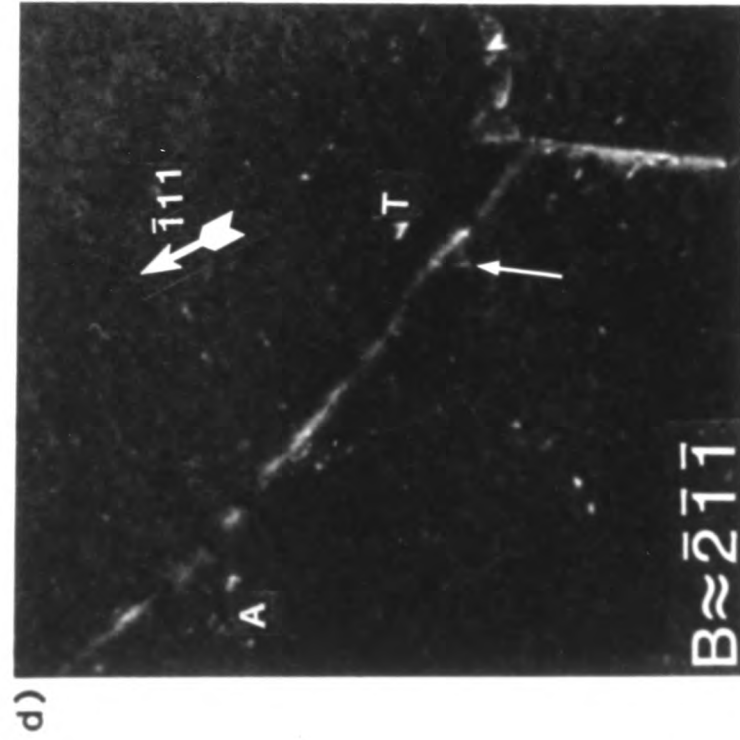


70° Dislocation in Fe-15%Ni-17%Cr-1%Si (at.%)

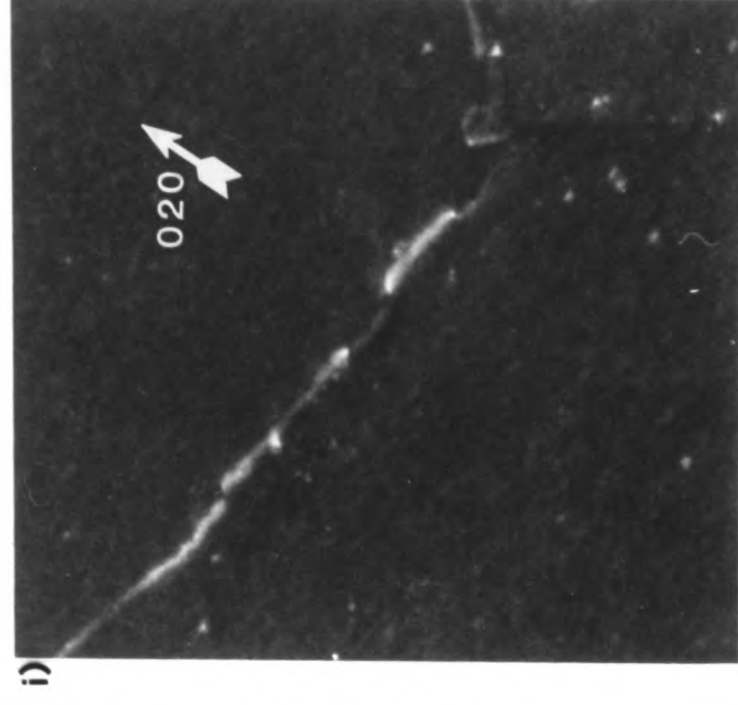
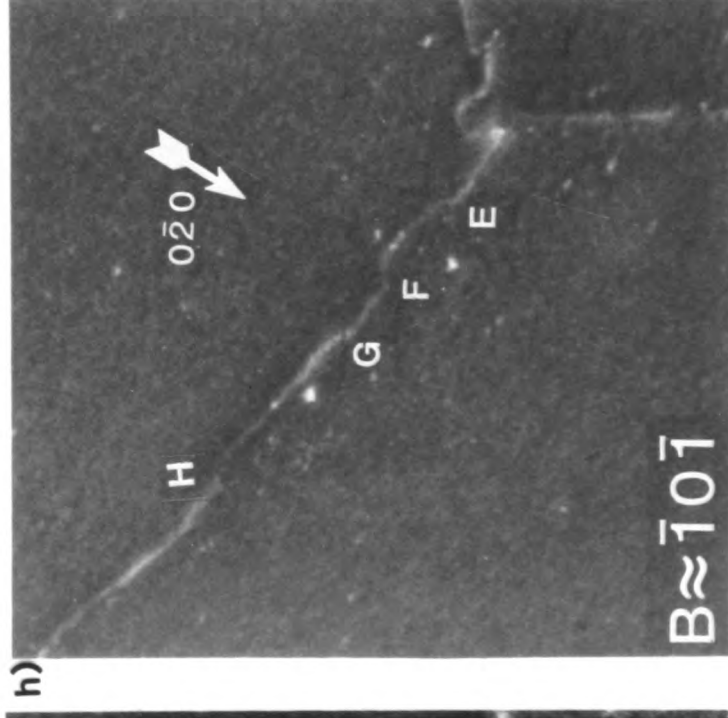
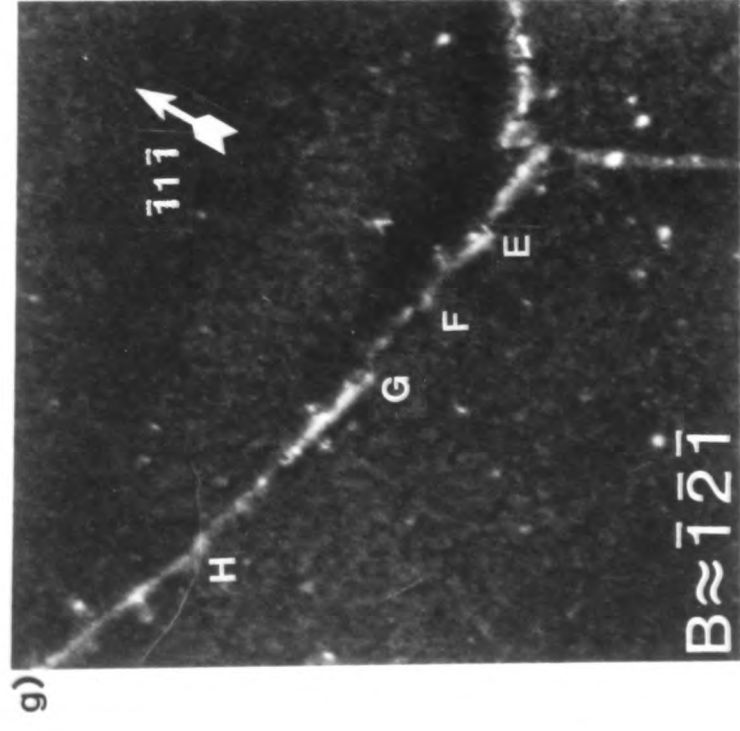
0.13 dpa at $\sim 400^\circ\text{C}$



Positive line direction



$$T \rightarrow \tilde{b}_e^{\text{rel.}}$$



70° Dislocation in Fe-15%Ni-17%Cr-1%Si (at.%)

0.13 dpa at ~400°C

indicative of a dipole or small loop. Since the Burgers vector of this dislocation does not appear to have altered after irradiation, a Burgers vector of $\vec{CA}$ is suggested by the sense of the strong/weak contrast.

Interpretation:

In common with the long dislocation of figure 6.5, short sections of the dislocation appear to have climbed. The amount of climb in either figure is not great, being only $\sim 10\text{ nm}$. The ends of climbed line sections are bounded by super-jogs; in the case of figure 6.5, a very high density of pre-existing constrictions were present and it is uncertain whether these super-jogs are formed by the coalescence of pre-existing jogs or by new nucleation onto the dislocation. In the case of figure 6.6, however, new nucleation would appear to be the most likely possibility, since the jog (constriction) density is considerably lower before irradiation. The observations, in all figures from this area, that the Burgers vectors of dislocations are unchanged by irradiation (apart from at 'C' in figure 6.4), indicates that nucleation is proceeding only by the assimilation of loops with the same Burgers vector as the parent dislocation. This is apparently occurring along dissociated dislocations, possibly indicating operation of the **Thomson & Balluffi** (1962) mechanism.

Since it cannot definitely be determined from these micrographs whether, say, section 'EF' or 'FG' lies closer to the original plane of this dislocation, the direction of climb must be inferred from considerations of both the geometries of all three dislocations intersecting at this node and of the likelihoods of the two possibilities; namely, interstitial or vacancy climb. This is left for the '*Points of Discussion*' section towards the end of this chapter.

0.2 dpa ('High Dose') Irradiations:

The following two figures show two closely spaced areas from a specimen irradiated to approximately 0.2 dpa. Both areas are shown in micrograph (j) of figure 6.8. The irradiation was performed in the Argonne HVEM with the largest spot-size (actually 6), illuminating most of the area shown in micrograph 6.8 (j). The beam was centred roughly in the centre of this micrograph.

A dose of 0.2 dpa is high in the context of the other irradiations shown in this volume. It was found that a great deal had occurred along the dislocations, leading to complex structures. As a result it did not prove possible to characterize all features seen after irradiation, which is of course desirable. There follow, therefore, some more general impressions based on some of the more striking features shown in the micrographs:

(Area 1) *Figure 6.7; 30-80° Dislocation in Fe-15%Ni-17%Cr-1%Si; 0.2 dpa at ~ 400°C.*

Observations: Pre-Irradiation Configuration;

A curved dislocation, ranging from 30° to near-edge in character, is shown before ((a)) and after ((b)-(e)) irradiation in figure 6.7. Both partials are imaged in (a), which has $|\tilde{\mathbf{g}} \cdot \tilde{\mathbf{b}}_T| = 2$ for dislocation $\tilde{\mathbf{b}}_T = \vec{\mathbf{AB}}$, from which the presence of a large number of constrictions, such as 'A', 'B' and 'C', can be seen. In the 6 $\langle 2\bar{2}0 \rangle$ -type reflections taken before irradiation, features 'A' and 'B' appear to be super-jogs²⁷, at which a short (~ 5 nm.) section of dislocation $\vec{\mathbf{AB}}$ apparently lies along, or close to, the projection of [101] (γ). If this is the case then section 'BC' will lie above 'AB', which will, in turn, lie above the 30° dislocation to the left of 'A'. Both halves of the double-jog at 'A' are apparently widely extended (~ 60 Å); the lower part of double-jog at 'B' may be narrowly dissociated (~ 30 Å) but no evidence is seen for dissociation of the upper part (to the right). No further information as to the configurations either of constriction 'C', or of the many other constrictions, was determined from the pre-irradiation micrographs.

Post-Irradiation:

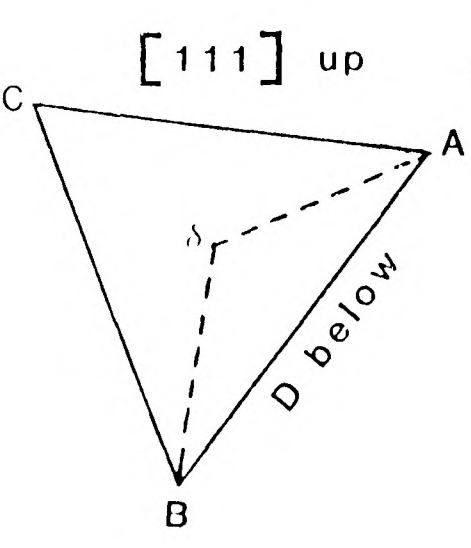
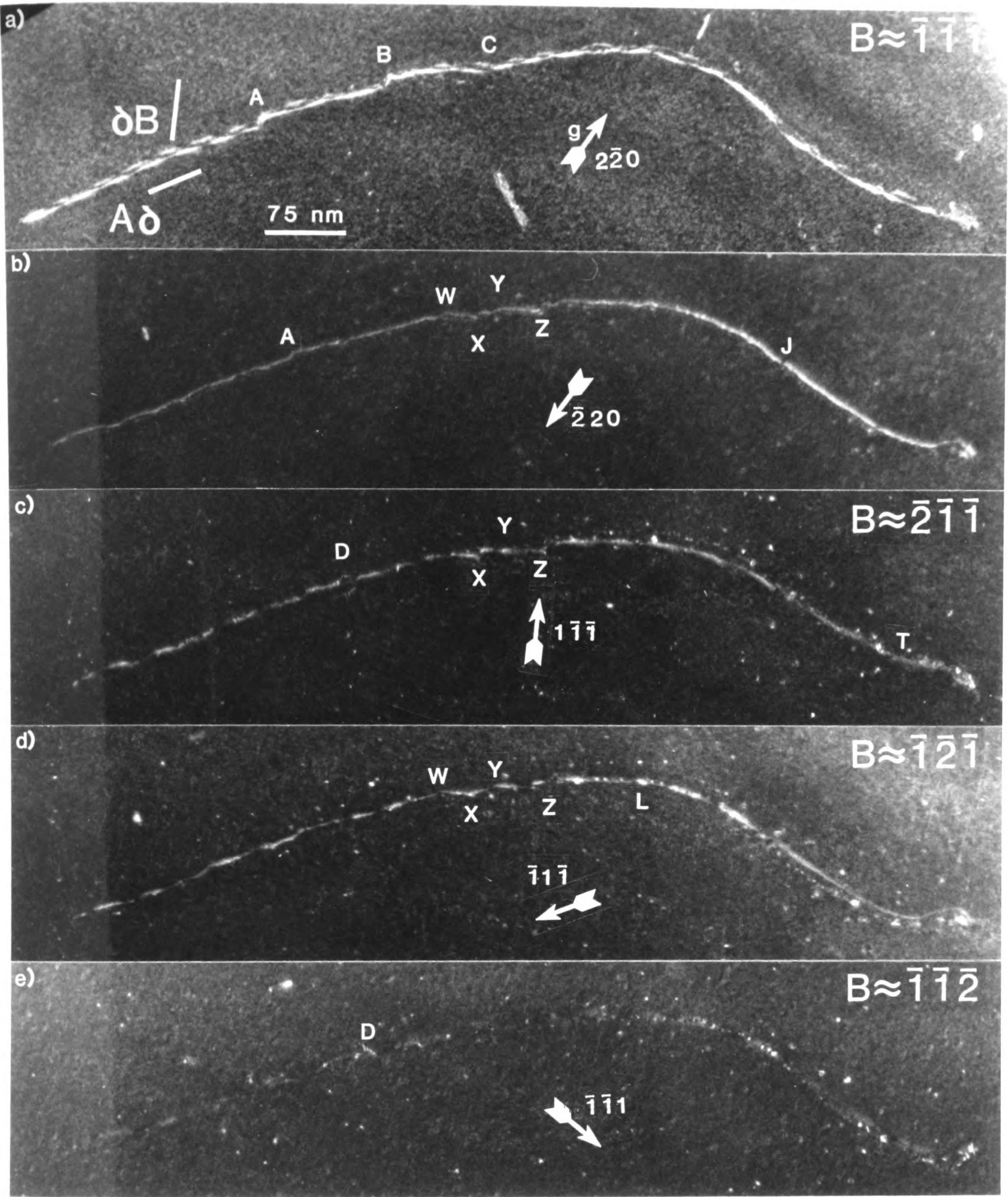
After irradiation, the separation between the partials has apparently decreased and the right-hand portion of dislocation $\vec{\mathbf{AB}}$ has straightened slightly from its pre-irradiation position, as indicated by a discrete line of clusters. The straightening of the dislocation has probably occurred by glide and some constrictions appear to have been annihilated. Conversely, many new constrictions are evident (see the $|\tilde{\mathbf{g}} \cdot \tilde{\mathbf{b}}_T| = 2$ image (b)) towards the middle of the dislocation and along the 30° section to the left of 'A'. Jog 'A' itself is apparently still extended and appears substantially unaffected by the irradiation, although it may have moved slightly to the right.

Several new cusps ('W'; 'X'; 'Y'; 'Z') have formed near the pre-existing constriction 'C'. On tilting to $\tilde{\mathbf{B}} \approx [\bar{2}\bar{1}\bar{1}]$ ((c)), the length of cusps 'X' and 'Z' is increased suggesting that they are jogs similar to those seen in figures 6.5 and 6.6. On tilting to bring planes α vertical ((d)) the shape of these cusps is considerably altered, apparently forming a faint zig-zag structure 'W-X-Y-Z'. This is reminiscent of screw dislocations and may suggest that sections of the line lie on different inclined planes. The complexity of the configuration prohibits any determination of their line directions, however. This complexity is exemplified by jog 'Z' as seen in micrograph (d).

Jog 'Z' approximately marks the extent of a discrete line of clusters. Many of these clusters are recognizable as SFT, particularly near 'T' towards the intersection of the dislocation with a foil surface.

²⁷In principle, these could also be unit jogs; large angle tilt experiments were seldom conducted prior to irradiation.

Figure 6.7



Positive line direction

$T_{be}^{rel.}$

30-80° Dislocation in
Fe-15%Ni-17%Cr-1%Si (at.%)
0.2 dpa at ~400°C

Several new clusters, such as 'L', seem to be forming close to the new dislocation position in this comparatively high dose irradiation. These clusters appear likely to be vacancy in nature but this could not be identified directly. The small background clusters could also not be characterized using standard techniques.

Finally, feature 'D', which has formed along a section of dislocation close to 30° orientation, exhibits confusing contrast. It is out-of-contrast, or too faint to be resolved, in all $\tilde{g} = \langle 2\bar{2}0 \rangle$ reflections which are taken in the (111) pole. It is also out-of-contrast in $\tilde{g} = \pm [1\bar{1}1]$ but is visible in $\tilde{g} = \pm [\bar{1}11]$ and $\pm [\bar{1}\bar{1}1]$ reflections. Strong/weak contrast could not be distinguished. In $\tilde{g} = [\bar{1}\bar{1}3]$, taken with beam direction $\tilde{B} \approx [\bar{2}\bar{1}\bar{1}]$, feature 'D' is visible as a very bright, continuous loop. On tilting from $\tilde{B} \approx [\bar{2}\bar{1}\bar{1}]$ to $\tilde{B} \approx [\bar{1}\bar{1}\bar{2}]$ it is displaced, with respect to the line, in the opposite sense to vacancy SFT suggesting that it lies within the *dilatational* strain field of the dislocation. One possible explanation for this contrast is that feature 'D' is principally an interstitial, pure-edge, prismatic loop with Burgers vector $\tilde{b}_L = \vec{BC}$. Such a loop would have been vertical in the foil when the $\langle 2\bar{2}0 \rangle$ -type reflections were taken; displacements from its opposite sides may then cancel through a column of crystal. In $\tilde{g} = [\bar{1}\bar{1}1]$ ((e)) a small region of feature 'D' shows contrast typical of a vacancy SFT. This is akin to behaviour exhibited by 'B' of figure 4.6 and it is possible that 'D' may similarly consist of both defect species. In the opposite reflection ($\tilde{g} = [11\bar{1}]$; not shown) the continued dissociation of the dislocation is confirmed by SFC seen from all regions apart from jog 'Z', segment 'XY' and a short section (~ 20 nm. long) extending to the left of point 'W'.

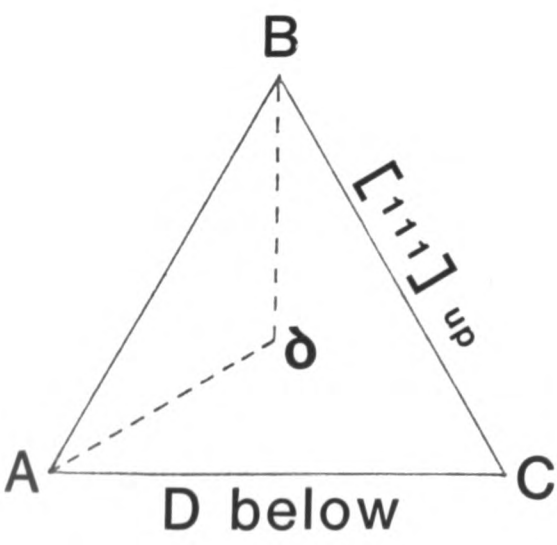
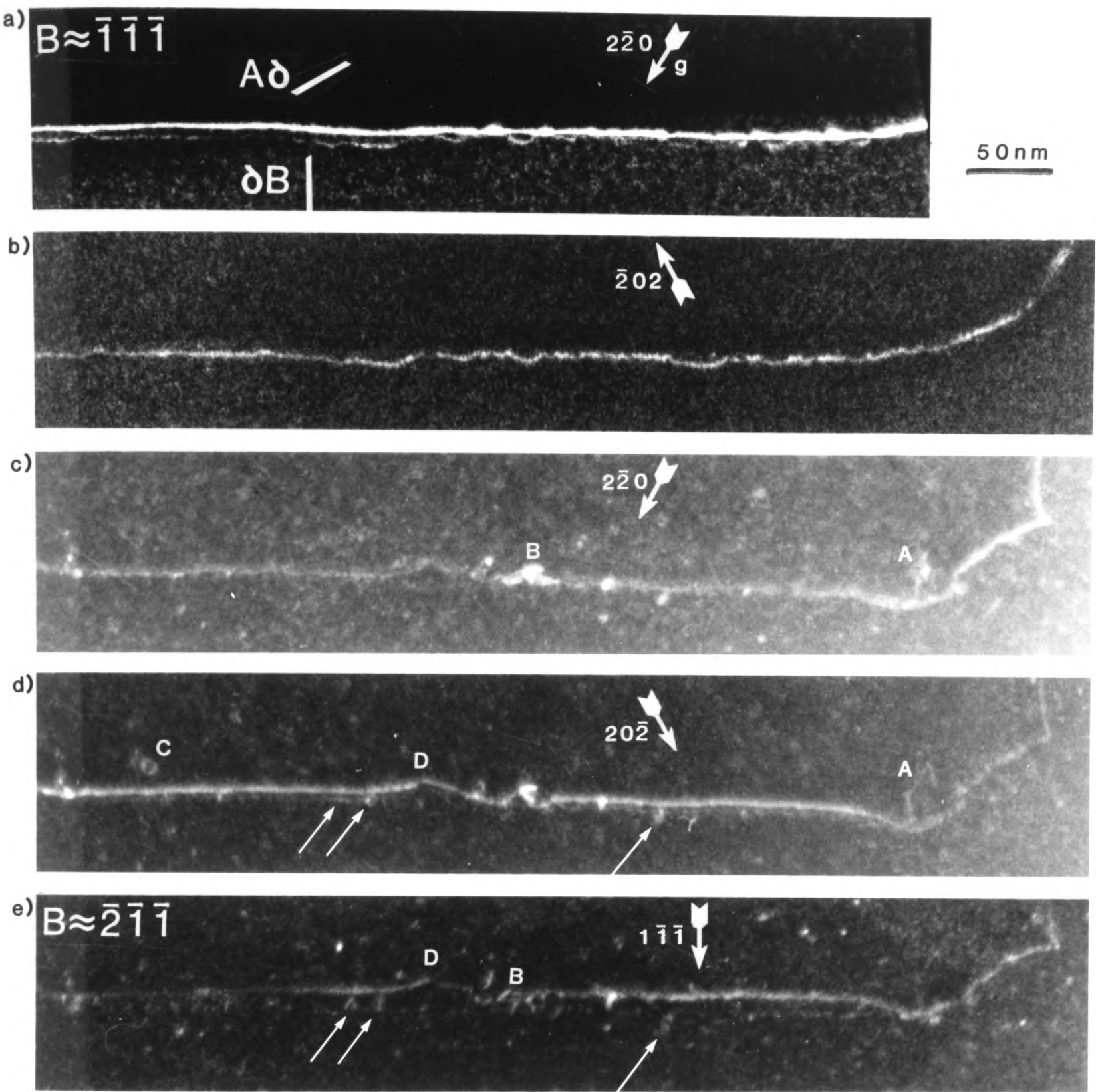
Figure 6.8; 60° Dislocation in Fe-15%Ni-17%Cr-1%Si; 0.2 dpa at $\sim 400^\circ\text{C}$.

Observations:

Micrographs (a), (b) and (j) are taken before, and all others after irradiation. Before irradiation this dislocation consists of a long, pure 60° section which curves towards screw orientation to the right-hand side. Micrograph (a) is a $|\tilde{g} \cdot \tilde{b}_T| = 2$ image, showing both partials of dislocation $\vec{AB}$, whilst (b) has $|\tilde{g} \cdot \tilde{b}_T| = 1$ and only shows the lower (edge) partial $\vec{\delta B}$. Both images show evidence for a large number of pre-existing constrictions.

In micrograph (d), partial $\vec{\delta B}$ is visible. This partial, which is seen to be heavily cusped before irradiation ((b)), is now smooth in appearance; constrictions, which are likely to represent jogs, apparently having been annihilated. Cluster damage, some of which may be recognized as SFT, is also seen to have formed in a line parallel to the dislocation. This cluster damage is shown particularly clearly in micrograph (g) and is displaced from the line in micrograph (e) in a sense consistent with it lying to the compressional strain field side of the dislocation.

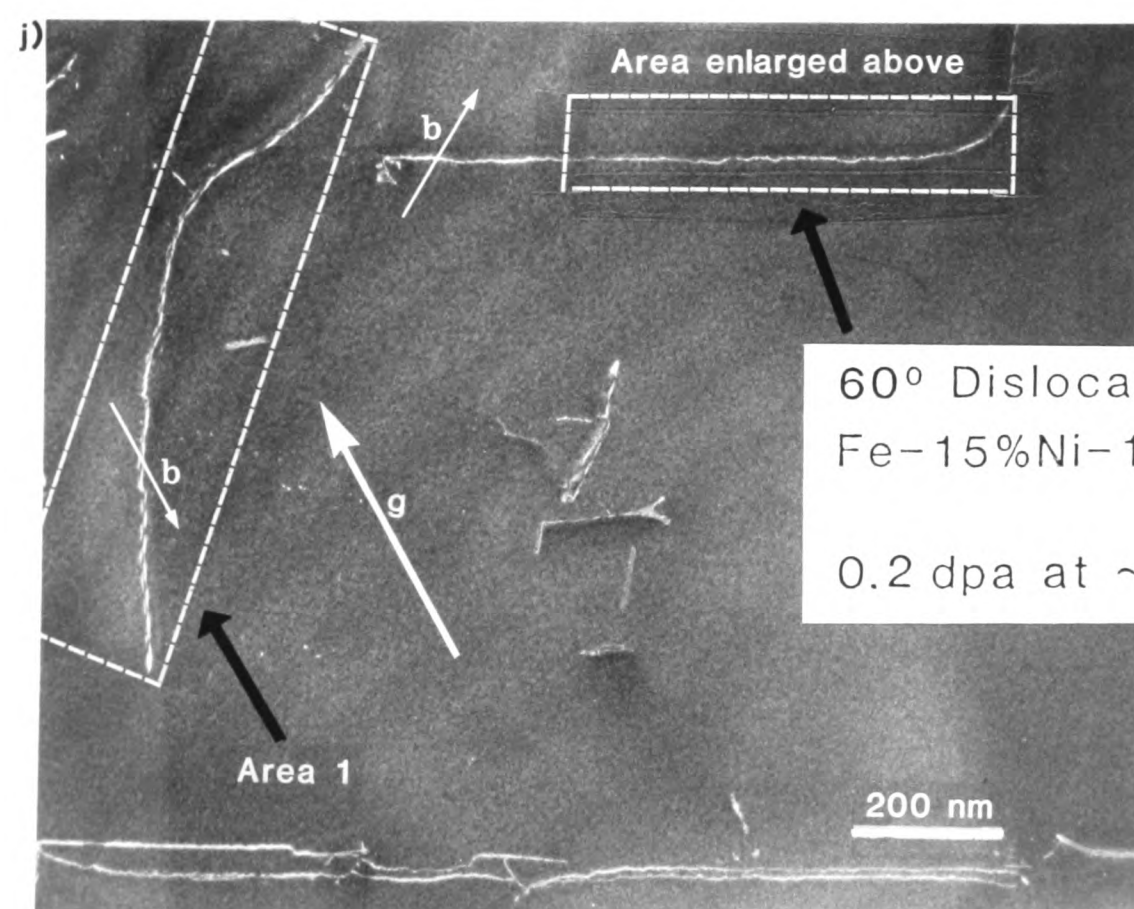
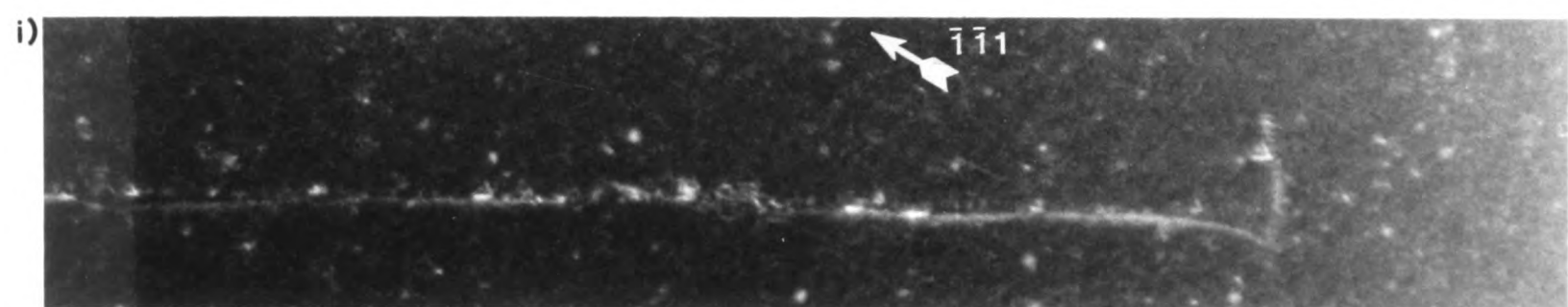
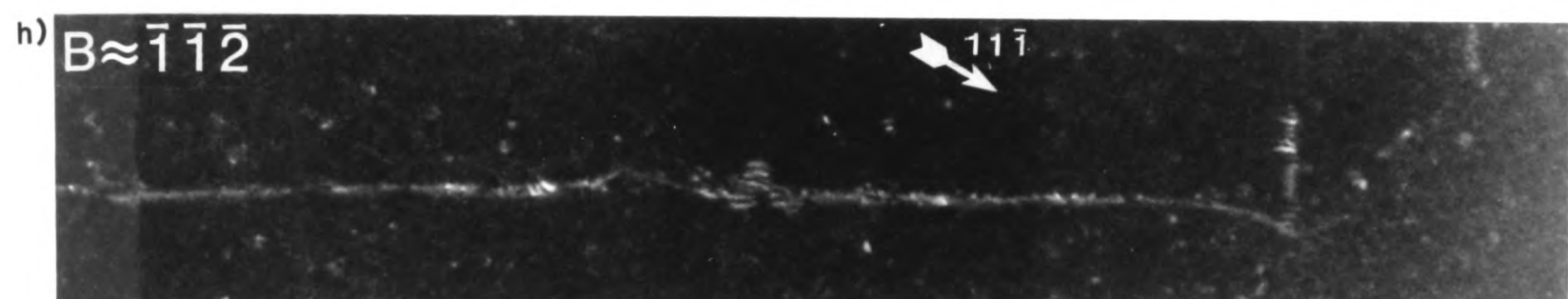
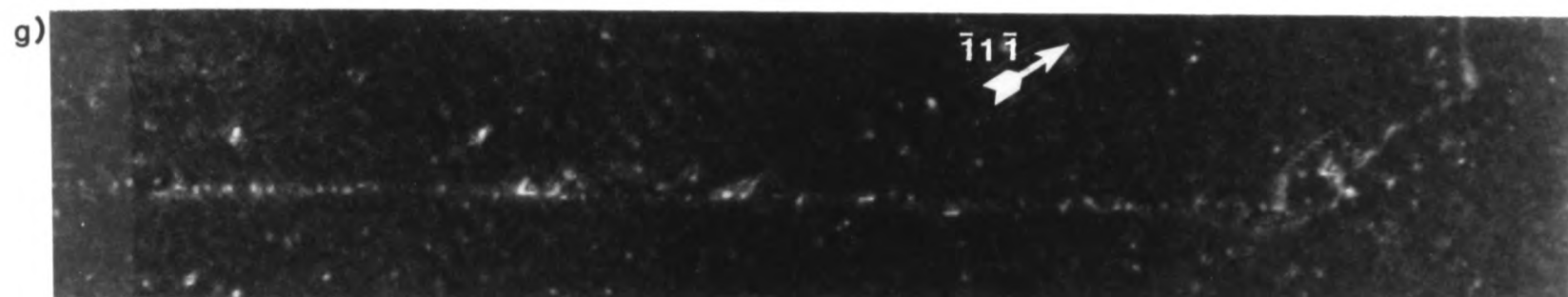
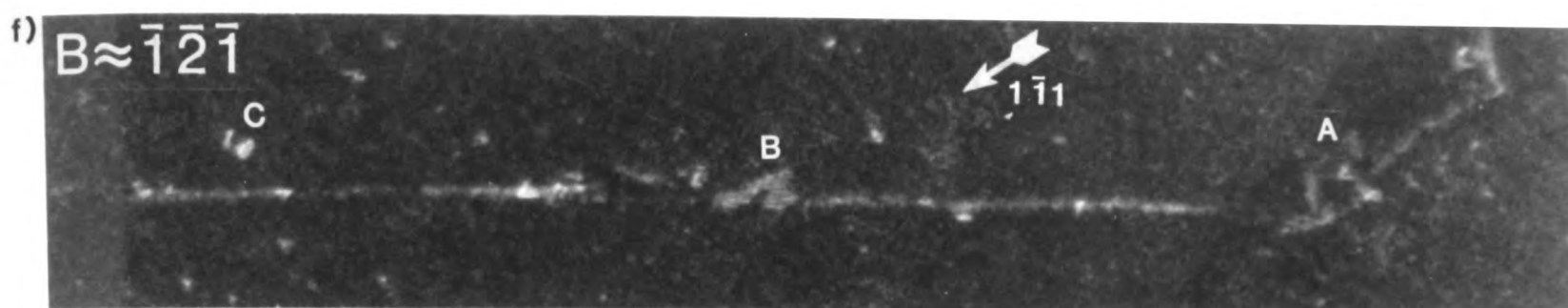
Figure 6.8
(2 pages)



Positive line direction

$$T \rightarrow \tilde{b}_e^{\text{rel.}}$$

60° Dislocation in
Fe-15%Ni-17%Cr-1%Si (at.%)
0.2 dpa at ~400°C



60° Dislocation in
Fe-15%Ni-17%Cr-1%Si (at.%)
0.2 dpa at ~400°C

A large cusp ('D') and a bright feature ('B') have formed towards the middle of the line. Feature 'B' is also apparently associated with a smaller cusp in the dislocation. On tilting the foil to $\tilde{B} \approx [\bar{2}1\bar{1}]$ ((e)) this part of the line now appears essentially straight, suggesting that 'B' is principally confined to a plane β . Analysis of contrast arising from most of 'B' (specifically the bright 'V-shape' visible in (d)) is consistent with an interstitial Frank loop of Burgers vector $\tilde{b}_L = \vec{\beta B}$: It is not visible in $\tilde{g} = \pm [02\bar{2}]$ (not shown), shows strong contrast in $\tilde{g} = [2\bar{2}0]$ ((c)) and $\tilde{g} = [20\bar{2}]$ ((d)), and weak contrast in $-\tilde{g}$ of these reflections. The stacking fault is also brightly imaged in $\tilde{g} = [1\bar{1}1]$ ((f)) and $\tilde{g} = [11\bar{1}]$ ((h)). Contrast seen in other reflections ($\langle 11\bar{3} \rangle$ type; not shown) of the contrast series confirms this characterization. The remaining section of feature 'B' is apparently visible in all $\langle 2\bar{2}0 \rangle$ -type reflections and in $\tilde{g} = [1\bar{1}\bar{1}]$ ((e)) as a faint parallelogram shape. Its possible Burgers vectors are therefore $\pm \vec{AB}$ or $\vec{BC}$. Inside/outside contrast could not be distinguished.

On tilting the foil away from $\tilde{B} \approx [\bar{1}\bar{1}\bar{1}]$, feature 'B' appears to be attached to the dislocation directly. It also appears to extend into the *compressional* side of the dislocation strain field. In micrographs (f) and (h), a 'V-shape' part of the loop appears to be absent. This is a similar shape to the cusp seen along the line in (c) or (d). Feature 'B' is plainly extremely complex!

It is thought that 'D' represents a later stage in the evolution of feature 'B'. Cusp(s) 'D' remain visible on tilting the foil to bring any one of planes α , β or γ close to the vertical. They are more prominent when the foil is tilted away from $\tilde{B} \approx [\bar{1}\bar{1}\bar{1}]$ to $\tilde{B} \approx [\bar{1}\bar{2}\bar{1}]$ ((f) for example) and thus do not appear to lie in the plane of the foil. Tilting about an axis parallel to the dislocation also appears to affect the images of cusps 'D' in a manner suggesting that they do not lie in the plane of the foil. These cusps may be similar to climbed sections of dislocations seen in many of the preceding figures of this chapter.

In common with 'B', contrast exhibited by loop 'C' (the largest background feature; shown exhibiting "outside" contrast in (d)) is also most consistent with an interstitial Frank loop, with Burgers vector $\tilde{b}_L = \vec{\gamma C}$ in this case. A few other background loops occasionally exhibited contrast suggesting an interstitial nature; their sizes (down to 10 Å) prevented a more conclusive determination, however.

Feature 'A' is particularly interesting: It is visible either as a line, or as a parallelogram-shaped loop in all $\tilde{g} = \langle 2\bar{2}0 \rangle$ type reflections (only 2 shown), suggesting that its Burgers vector lies in a plane δ . It is also visible in $\tilde{g} = \pm [1\bar{1}1]$ and $[11\bar{1}]$ but is, however, not visible in $\tilde{g} = \pm [1\bar{1}\bar{1}]$ ((e)). This is consistent with a Burgers vector $\tilde{b}_L = \pm \vec{AC}$. When planes γ are nearly vertical ((h) and (i)) this feature appears as a thin line lying parallel to $[\bar{2}11]$. Micrographs (f) and (g) show an SFT to have formed

inside this parallelogram-shaped loop. This SFT lies just to the left of the line of contrast from feature 'A' in micrographs (h) and (i). Notice that this feature has formed at a point where the dislocation has curved away from 60° orientation. This observation is frequently duplicated in the following chapter (chapter 7).

Finally, to the right hand side of each micrograph, an initially near-screw section of the dislocation is now zig-zagged either side of its original position. This behaviour is consistent with previous observations (e.g. figures 3.4 and 6.1).

Interpretation:

Two observations suggest that loop 'A' is interstitial in nature: The first, and most direct, is the formation of an SFT inside this loop²⁸. The second is derived by noting that this loop apparently shows inside contrast in $\bar{g}=[1\bar{1}1]$ ((f)) and outside contrast in the opposite reflection. This suggests a specific Burgers vector of $\vec{A}\vec{C}$. In micrograph (d), loop 'A' is plainly not completely in edge orientation. It therefore did not condense as a perfect prismatic loop but may instead have formed from the unfauling of either $\tilde{b}_L = \vec{A}\vec{\alpha}$ (vacancy) or $\tilde{b}_L = \vec{\gamma}\vec{C}$ (interstitial) Frank loops. Notice, however, that this loop is only visible as a thin line when planes γ are nearly vertical but is readily recognizable when planes α are brought to the vertical. Notice also that the entire feature is more closely aligned with planes γ than with planes α throughout the figure. In fact, this loop has partially glided towards edge orientation, such that two edges of the loop now lie along $[\bar{2}11]$. It is not understood why only one of the other two edges is visible, although it may indicate that this feature intersects a foil surface. The opposite, visible edge appears to remain on a plane γ . Loop 'A' is therefore interstitial in nature.

The 'cusp-shaped' gap in the stacking fault of feature 'B' may correspond to an unfaulted region of the Frank loop. It is possible that this region is visible as a faint parallelogram shape in micrograph (e). It would appear likely that the unfauling of this region is related in some way to the formation of the cusp on the 60° dislocation, and hence related to motion of the dislocation.

Features 'B' and 'D'

In common with the 'missing side' of loop 'A', the detailed configurations of these features are also not understood. The important observations to be noted, however, are that interstitials are attaching to, or being precipitated by, the dislocation and that these interstitials appear to have prompted motion of the dislocation out of the plane of the foil.

²⁸*Ironically this is also an observation employed to infer the nature of SFT [Kirk et al, 1986, unpublished research; referenced in Jenkins et al, 1987].*

This motion of the dislocation appears to result in cusps being visible along the line. Since these cusps apparently do not lie in δ , and continue to be visible on tilting to bring any of planes α , β or γ vertical the cusps represent climbed sections of the dislocation. This climb may occur on more than one plane and would appear likely to be interstitial. It is possible that the habit plane of the precipitated Frank loop, or the habit plane(s) of a product(s) from the unfauling of such a loop, may dictate the direction of climb. One can only speculate as to whether the unfauling of this section has actually been prompted by motion of the dislocation, or whether unfauling must occur prior to climb of the line.

Finally, this dislocation shows anomalous contrast after irradiation. The lack of a double image in the post-irradiation $|\tilde{\mathbf{g}} \cdot \tilde{\mathbf{b}}_T| = 2$ images is not a unique phenomenon; the narrowing of the partial separations, in tandem with unavoidable contamination from the HVEM, easily explains the inability to resolve both partials separately. The line of cluster damage, plainly visible in micrograph (e), is probably the origin of the “double image” in (d); certain features (examples arrowed) along the “second” weak image of micrograph (d) map to spots of contrast, or SFT, along this line of cluster damage on tilting to $\tilde{\mathbf{B}} \approx [\bar{2}\bar{1}\bar{1}]$.

Points of Discussion

- *The Sense of Dislocation Climb* :– Figure 6.9;
- *Vacancy Precipitation*;
- *The Mechanism of Climb*;

The Sense of Dislocation Climb

In the case of the left-handed screw dislocation of figure 6.1, the observation of the probable onset of right-handed helix formation suggests that climb in an interstitial sense is occurring, as observed in chapters 3 and 4. It would seem likely that the cusps/zig-zags formed along the near-screw dislocation of figure 6.2 have a similar origin. These zig-zags were found to be confined to the plane of the foil preventing any direct determination of the nature of jog climb.

For the dislocations of figure 6.3, as shown in figures 6.4 to 6.6, initially planar sections of two of the dislocations have apparently segmented; parts now lie on different planes δ . It has proved possible, by tilting the specimen through large angles, to determine the relative heights of adjacent sections. Problems arise, however, in determining the nature of the climb since it is not known which of these adjacent sections lies closer to, or on, its pre-irradiation habit plane, i.e., whether the higher

section has climbed up (interstitial climb) or the lower section has climbed down (vacancy climb).

This question may be resolved by viewing the entire irradiated region, most of which is shown in figure 6.9. Information from all three figures which show the individual dislocations (here labelled '1', '2' and '3') of this region is compiled to build up a picture of which segments are more likely to lie on their original plane(s).

Figure 6.9; Dislocations and Node in Fe-15%Ni-17%Cr-1%Si; 0.13 dpa at $\sim 400^{\circ}\text{C}$.

From the first page of the figure it can straightaway be seen that dislocation '1' (shown in figure 6.4) is principally confined to a single plane δ . Two slight cusps are visible, which may correspond to positions 'T1' and 'T2'. Notice that the node is on a lower plane δ than is the bulk of dislocation '1' to the right-hand side. Dislocation '1' therefore appears to lie on, or very close to, its original plane; the only climb being that of the super-jog, which climb is in an interstitial sense.

In micrograph (b) (page 2) the foil is tilted such that components of dislocation '2' (figure 6.5) on different planes δ are separated. Segments of this dislocation which lie on a plane δ higher than the node are marked. Since the node appears likely to lie on its original plane interstitial climb is indicated.

On the third page of the figure (micrograph (c)) large parts of dislocation '3' can be seen to lie on a higher plane, particularly nearer the node. At first this might suggest that the higher plane is actually the original pre-irradiation plane and vacancy climb has occurred: Notice, however, that the dislocation jogs back to the lower plane at the node (the jog here appears very complex and may be extended). Notice also that as the distance from the node increases, less and less of the dislocation lies on the higher plane(s) 'H'. This would appear to be strong evidence for interstitial climb, since as the distance from the node increases so does the distance from the HVEM beam centre. As was described above, the furthest extent of the HVEM beam lay close to the left-hand side of the micrographs of figure 6.5. This also corresponds closely with the bottom of this micrograph. In effect, therefore, the long section of dislocation '3' at the bottom of the figure, which lies on a lower plane δ , will have received a lower dose. This lower plane is therefore likely to represent the original plane of both dislocations '2' and '3', and the node, suggesting that the sense of climb is interstitial throughout the figure.

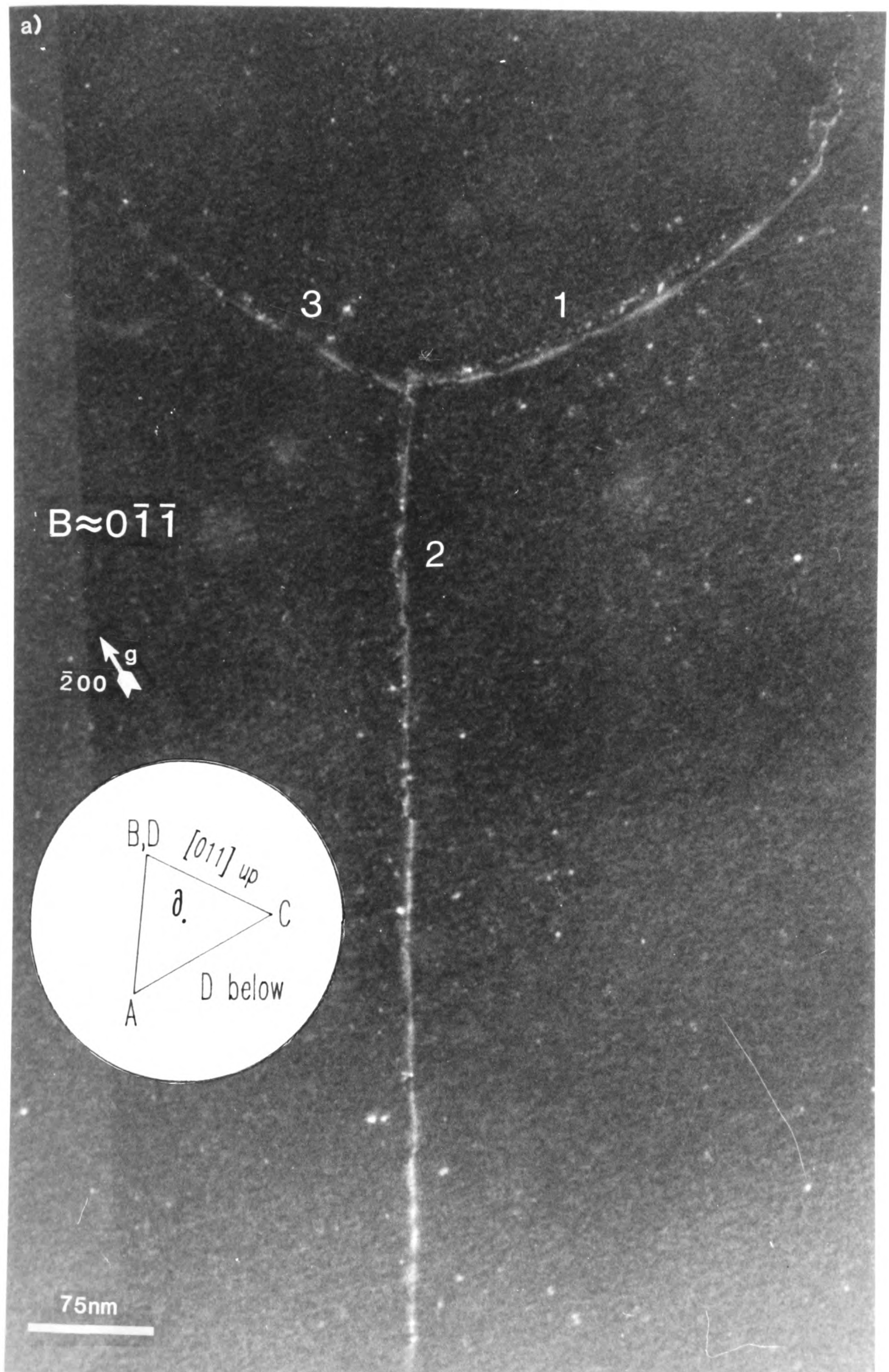
The occurrence of a small amount of vacancy climb at the pre-existing super-jog 'A' (which appears to have moved to the right), and perhaps also at 'B', along the dislocation of figure 6.7 cannot be excluded. It did not prove possible, however, to determine either the pre- or post-irradiation configurations exactly, so this could not be confirmed.

Figure 6.9
(3 pages)

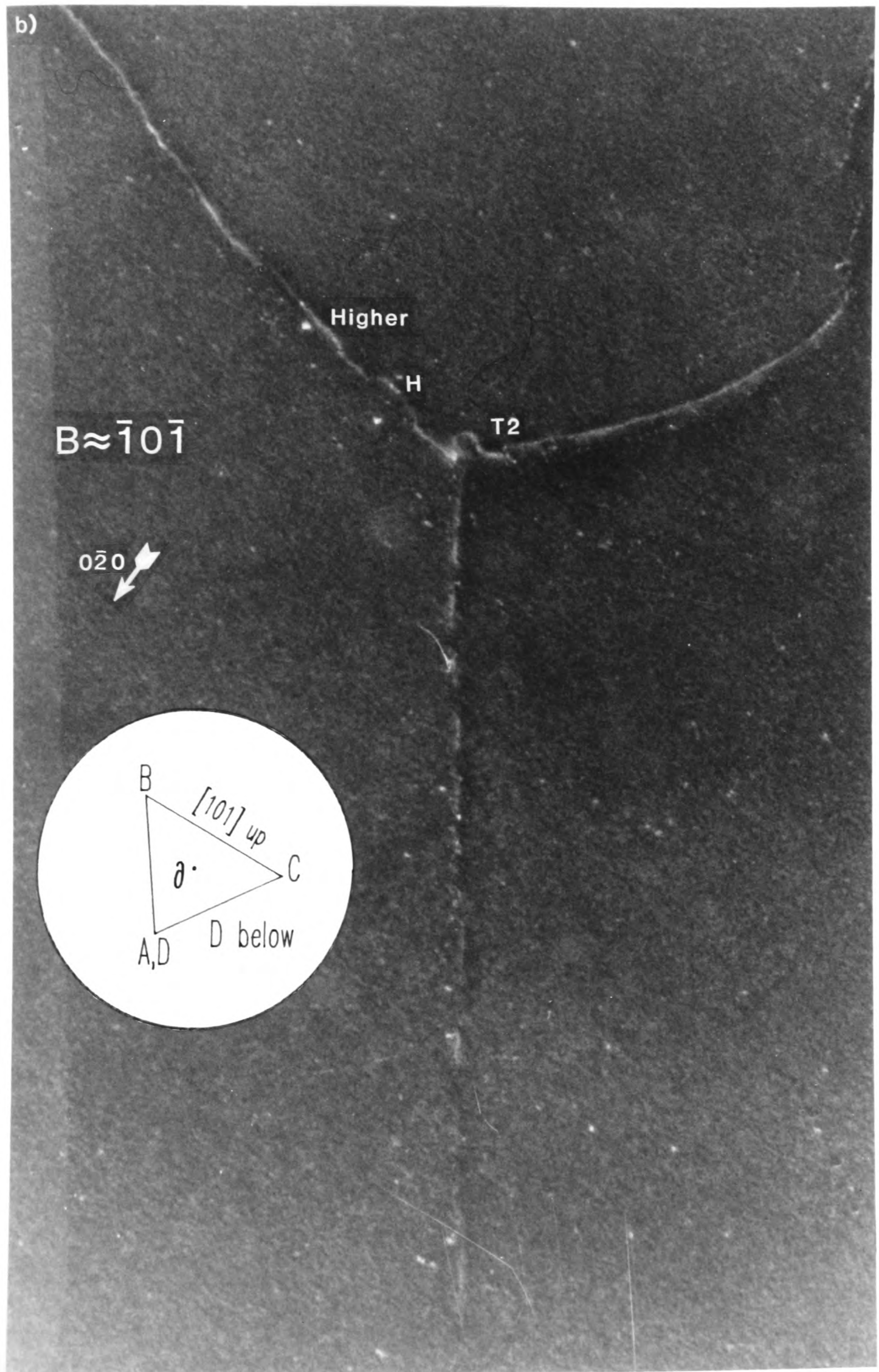
Dislocations meeting at a Node
After Irradiation

Dislocations and Node in Fe-15%Ni-17%Cr-1%Si (at.%)

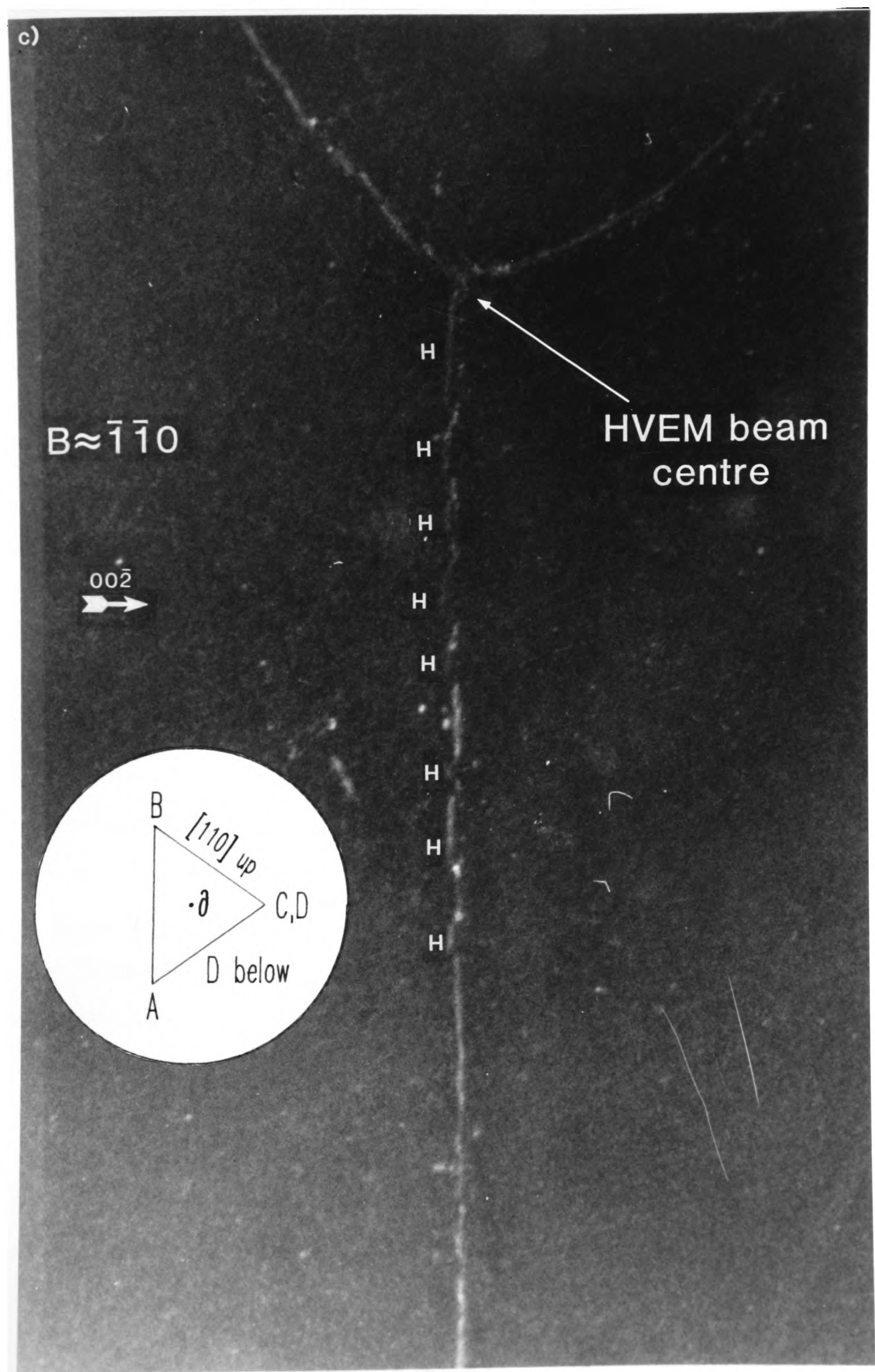
0.13 dpa at $\sim 400^{\circ}\text{C}$



Dislocations and Node in Fe-15%Ni-17%Cr-1%Si (at.%)
 0.13 dpa at ~400°C



Dislocations and Node in Fe-15%Ni-17%Cr-1%Si (at.%)
0.13 dpa at ~400°C



Lastly, the observations that the dipoles of figure 6.2 are annihilated by irradiation (see **Carter**, 1980b), and of interstitials precipitating at the line in figure 6.8, apparently reinforce the determination of climb exhibited by dislocations in this alloy as being in an interstitial sense. Arguments put forward in the preceding chapters (based on consideration of the likely point-defect super-saturations surrounding dislocations (e.g. figure 3.4)), to suggest that observations of climb are likely to result from interstitial absorption, should be equally applicable here.

Vacancy Precipitation

Figure 6.9 (particularly micrograph (a)) highlights the markedly different number densities of vacancy clusters formed near the three dislocations shown. Dislocation '1' appears evenly dissociated after irradiation. It has not obviously climbed and a large number of vacancy clusters have formed in its vicinity, including a particularly large cluster where the line passes through 60° orientation. As alluded to in the text relating to figure 6.4, it is thought that this dislocation is behaving in a way consistent with those of the preceding three chapters; interstitials being pipe diffused along the dislocation, after annihilation of pre-existing constrictions (jogs), whilst vacancies precipitate nearby. The determination of the large cluster at the point where this dislocation is precisely in 60° orientation as solely vacancy in nature may suggest that the pipe diffusion of vacancies is also important, as proposed in chapters 4 and 5 (figures 4.6 and 5.3).

Dislocations '2' and '3' have climbed. Far fewer vacancy clusters are seen to be associated with these dislocations than with the unclimbed, evenly dissociated dislocation '1'. Those vacancy clusters which are in evidence are often in the proximity of climbed sections, particularly near the super-jogs at either end. This is exemplified in figure 6.6 and by the SFT at 'T1' and 'T2'.

The Mechanism of Climb

The low number density of vacancy clusters near climbing, dissociated dislocations may indicate that climb of these dislocations is a nucleation rate controlled process.

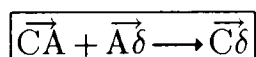
In figure 6.4, vacancy clusters are fewer near the node. This section also shows evidence for interstitial precipitation, namely climb of the super-jog and the formation of cluster 'C'. A possible mechanism for the formation of this $\vec{C}\delta/\delta\vec{C}$ dipole, based entirely on the work of **Cherns et al** (1980) in Cu-13%Al, is presented in diagram 6.2. The operation of this mechanism might be expected to require a high point-defect super-saturation prior to nucleation of the primary loop (stage (b)) (see **Cherns**, 1984).

In several of the figures extremely complex configurations are seen, involving the nucleation of

Diagram 6.2

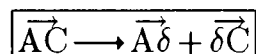
This section of dislocation, near the node, is close to edge orientation. A number of extended jogs may be present, but this cannot be determined unequivocally from the micrographs. Prior to irradiation ((a)) the right-hand partial $\vec{A}\delta$ appears to be slightly curved towards edge orientation.

In (b), dislocation $\vec{A}\vec{B}$ has constricted at one point. Formation of this constriction, evident in the micrographs, may be aided by climb of the nearby super-jog. In addition it may be prompted by, or prompt, nucleation of the perfect prismatic interstitial loop $\vec{b}_L = \vec{C}\vec{A}$ onto the near-edge partial $\vec{A}\delta$. Loop $\vec{C}\vec{A}$ then reacts with partial $\vec{A}\delta$ via:



The prismatic loop $\vec{C}\vec{A}$ may have nucleated initially as a $\vec{b}_L = \alpha\vec{A}$ Frank loop which subsequently unfaulted. Climb forces then act on loop $\vec{C}\vec{A}$, twisting it towards edge orientation, possibly promoting both the constriction of dislocation $\vec{A}\vec{B}$ and the bowing-out of partial $\vec{A}\delta$. Constriction of the node itself may prompt further bowing-out of partial $\vec{A}\delta$.

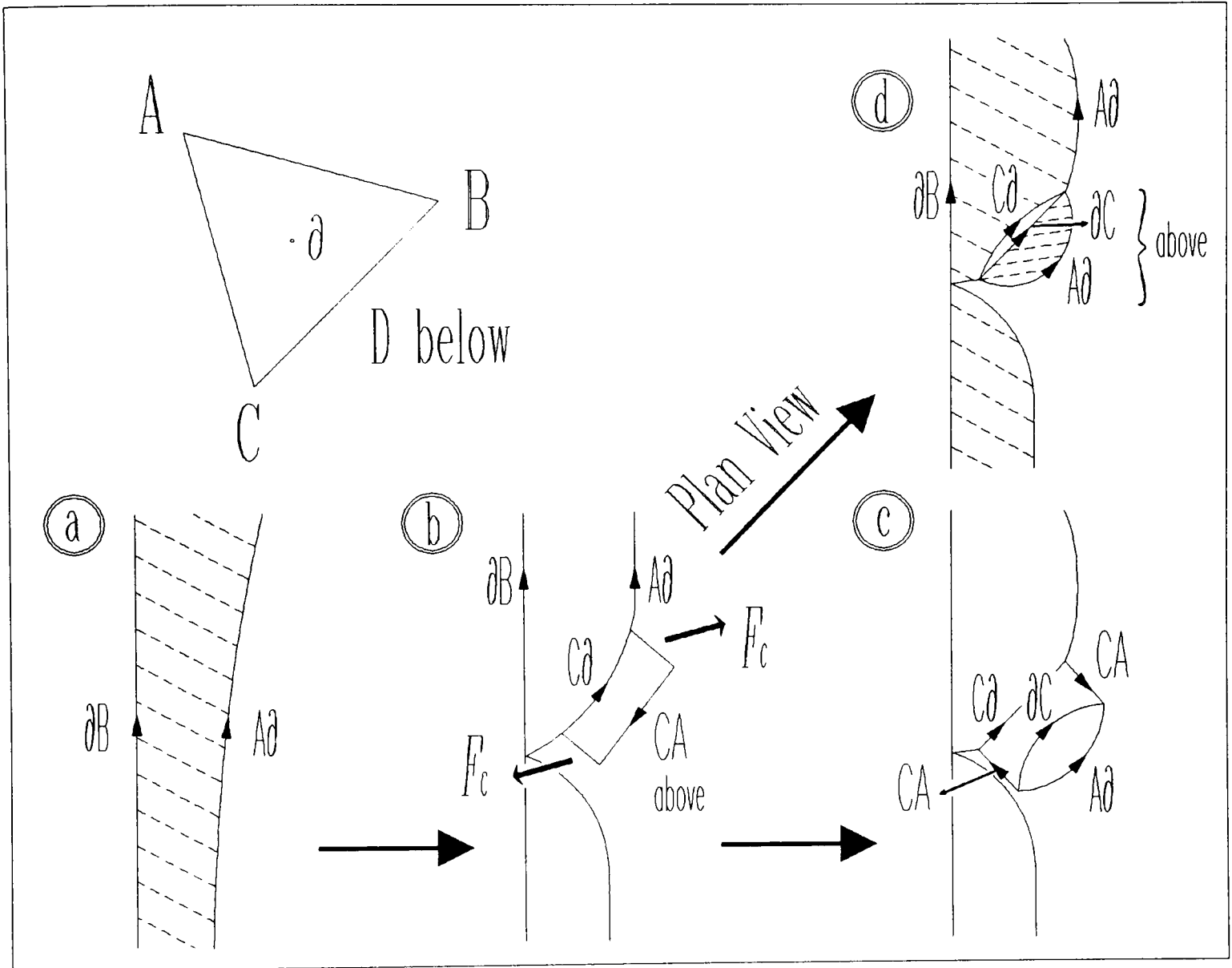
In (c), loop $\vec{C}\vec{A}$ has now dissociated on δ , via:



to form a Shockley dipole $\vec{C}\delta/\delta\vec{C}$. In ‘plan view’ ((d)), the region encompassed by this dipole is not faulted; shaded regions represent stacking faults on δ . In effect partial $\vec{A}\delta$ has now climbed.

In essence this mechanism is identical to that presumed to be operating in Cu-13%Al under conditions of both interstitial (HVEM irradiation; **Cherns et al**, 1980) and vacancy super-saturations (quenching; **Décamps et al**, 1983).

Diagram 6.2



interstitial Frank loops onto the line, which is thought to be the primary event of the mechanism of Cherns et al (see **Cherns**, 1984), and the formation of an indeterminate number of kinks and/or jogs (see, for example, features 'D', 'B' and 'Z' of figures 6.7 and 6.8). Intuitively, one would expect an activation energy requirement for these configurations, necessitating the prior generation of a local interstitial super-saturation. Consider also the observations of figures 6.5 and 6.6, in which climb of the dissociated dislocations apparently requires the nucleation of new jogs, as is also seen along the screw and near-screw dislocations of figures 6.1 and 6.2.

Observations, both direct (figure 6.8; 'B') and indirect (figure 6.4; 'C') of loop nucleation at the dislocation may suggest that the climb of these dissociated dislocations proceeds via a nucleation based mechanism. The Burgers vectors of these dislocations is unchanged by irradiation and the absence of any features with a Burgers vector different from the dislocation indicates that, if nucleation occurs, it involves loops with the same Burgers vector as the line. In essence this is the situation envisaged by **Thomson & Balluffi** (1962). Calculations performed by **Grilhé et al** (1977) indicate that once loops, with dimensions of the order of the dislocation dissociation width, have nucleated, the operation of the Thomson & Balluffi mechanism may be energetically favourable. The essential result is unaffected, however, namely that the interstitial super-saturation must exceed a certain threshold value before nucleation, and subsequent climb, will proceed.

If the dislocation bias term²⁹ is correct, and observations of climb consistent with interstitial absorption suggest that it is, the low densities of vacancy clusters close to climbing dislocations may be accounted for by assuming that dislocations act as recombination centres. The few surviving vacancy clusters, which indicate that these recombination centres are not 100% effective, may result from a tendency for dislocations to separate interstitial and vacancy fluxes (see **Hirth & Lothe**, pp.504-508). Evenly dissociated dislocations, along which interstitials may core diffuse, are not apparently acting as recombination centres, allowing the formation of dense vacancy cluster damage in their vicinities.

Such a mechanism may also explain the qualitative observation that vacancy clusters are often to be found close to jogs: As is generally assumed (**Balluffi**, 1975; **Carter**, 1980b), and apparently reinforced by observations contained in this thesis, jogs may act as efficient sinks for irradiation produced defects. In both of these examples, these defects may often be identified as interstitial in nature. As is argued by **Kiritani** (1975) and **Jenkins et al** (1987), the presence of efficient sinks for interstitials may promote the formation of vacancy clusters in their vicinity. This may explain, for example, the observations that the number density of vacancy clusters is considerably higher near zig-

²⁹*Namely that dislocations are attracting a greater number of interstitials than vacancies.*

zagged regions than elsewhere along the screw and near-screw dislocations of figures 6.1 and 6.2.

Finally, it is plausible that dissociated dislocation sections are climbing via the Thomson & Balluffi (1962) mechanism: Climbed sections of dislocations may often be seen to be extended and Burgers vectors of all dislocations are unaffected by the irradiation. Extension of the neighbouring super-jogs has also been seen. In figure 6.6, climbed sections of the dislocation occasionally exhibit inside/outside contrast suggesting nucleation of loops with the same Burgers vector as the line (see micrographs (h) and (i)), although this contrast is not apparent in other $\pm \tilde{g}$ reflection pairs.



Summary

Observations:

The behaviour of irradiated dislocations in this material may be summarized as follows:

• (Super-) Jog nucleation;	... 1 ...
• Zig-zag configurations;	... 2 ...
• Climb;	... 3 ...
• Unpinning;	... 4 ...
• Dipole annihilation;	... 5 ...
• Dipole formation;	... 6 ...
• Vacancy clusters/SFT near jogs;	... 7 ...
• Large vacancy cluster at 60°;	... 8 ...
• Large interstitial clusters at 60°;	... 9 ...
• Annihilation of constrictions and formation of line of vacancy defects;	... 10...
• Extensive glide;	... 11...
• SFT in bulk/away from dislocation;	... 12...
• Interstitial loop in bulk;	... 13...
• Narrowing of partial separations;	... 14...

Individual figures exhibiting these phenomena are listed in table 6.1.



Whilst several very small clusters were seen in the bulk after the lower dose irradiations, some of which could be identified as SFT, only one interstitial background loop was identified, after a dose of ~ 0.2 dpa. This is opposite to observations in chapters 3, 4 and 5 and may suggest a high interstitial mobility here. In the bulk, vacancies are expected to be less mobile than interstitials and thus are more

likely to be retained in the foil. Vacancy clusters formed in the bulk may thus be able to survive if interstitials diffuse quickly to the surfaces or to dislocations. It is expected that bulk interstitial defects will also form, and may account for many of the background clusters which were too small to identify using the weak-beam technique, but their rate of growth will be slow.

Dislocations in this material do not show a marked preference to lie along any particular direction(s), as is seen in chapters 3 and 4. This may have a significant bearing on the precise post-irradiation configurations but is not expected to fundamentally affect the mechanisms of dislocation climb. The lack of the ‘regimenting’ effect of energetically favourable directions along which dislocations prefer to lie may help explain the frequently complex and irregular appearance of dislocations after irradiation. It is thought that the addition of silicon to the matrix is responsible for this effect. These points are considered briefly within chapter 8.

Questions as to the rôle of silicon in the formation of new jogs along dissociated mixed dislocations, an effect not seen in preceding chapters, and the relative mobilities of irradiation generated defects in the materials studied are also addressed within chapter 8.

Table 6.1

Figure	Character	Dose (dpa)	Temperature (°C ± 50°)	Observations (see above)
6.1	0°	0.09	400	1, 3 - onset of helix, 7;
	25°	0.09	400	1, 2, (3), 7, 14;
6.2	25°	0.09	400	1, 2, (3), 5, 7, 12, 14;
6.4	50°-80°	0.13	400	3 - at jog ‘A’, 6, 7, 8, 10, 14;
6.5	60°	0.13	400	1, 3, 4, 7, 11, 14;
6.6	70°	0.13	400	1, 3, 7, 14;
6.7	30°-80°	0.2	400	(1, 2, 3,) 5, 10;
6.8	60°	0.2	400	1, 3, 9, 10, 13;
6.9	60° & 70°	0.13	400	3;

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<i>Observations</i>	100
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Chapter 7

Irradiation of Fe-15%Ni-17%Cr-2%Mo alloy

As was found to be the case for the quaternary alloy containing silicon, the addition of molybdenum to the 15%Ni pure ternary alloy apparently has an effect on the behaviour of dislocations under irradiation. Many similarities are found with the pure ternaries in particular and similar mechanisms are thought to be operating. The pre- and post-irradiation configurations are often rather complex.

Figure 7.1; Near-edge Dislocation in Fe-15%Ni-17%Cr-2%Mo; 0.03 dpa at $\sim 420^\circ\text{C}$.

Observations:

Micrograph (a) shows both partials of dislocation $\tilde{b}_T = \vec{A}\vec{B}$ prior to irradiation. The upper partial ($\vec{\delta}\vec{B}$) is the brighter and can be seen to be heavily cusped, pointing to the presence of a large number of constrictions, which may be jogs, along the line.

In the same reflection((b)), after the lowest dose irradiation performed in this study, the dislocation line has straightened a little and most constrictions have disappeared. In the second $|\tilde{g} \cdot \tilde{b}_T| = 2$ image (c) the dislocation can be seen to be evenly dissociated. The post-irradiation partial image separation at 'S'(80°) is $\sim (35 \pm 5) \text{ \AA}$. The pre-irradiation partial separation is very variable.

Very little damage is seen within the irradiated area, the two SFT's (ringed in (d)) being the only features identified. The SFT at right was confirmed to lie to the compressional side of the dislocation strain field by large angle tilt experiment. The SFT at left lies close to an inclined dislocation which may be seen prior to irradiation in micrograph (a).

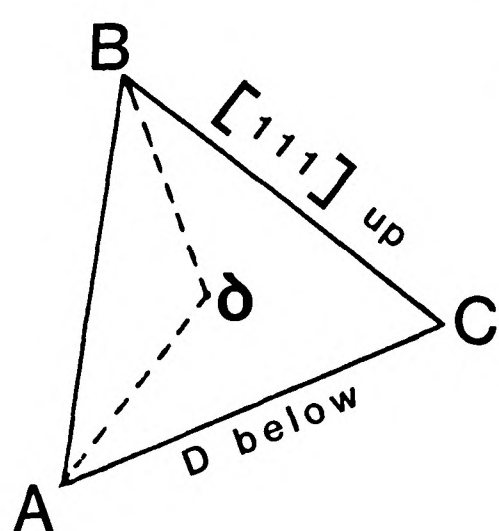
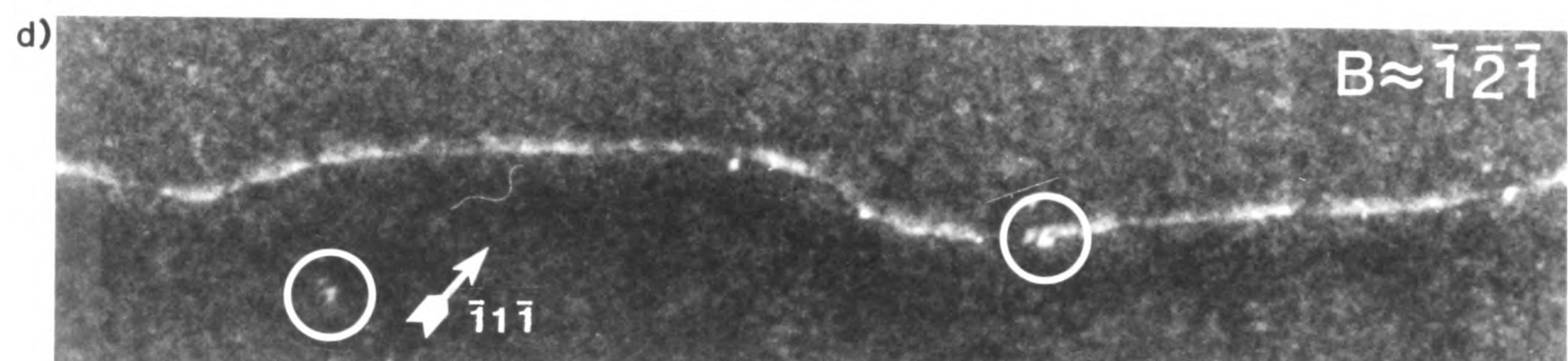
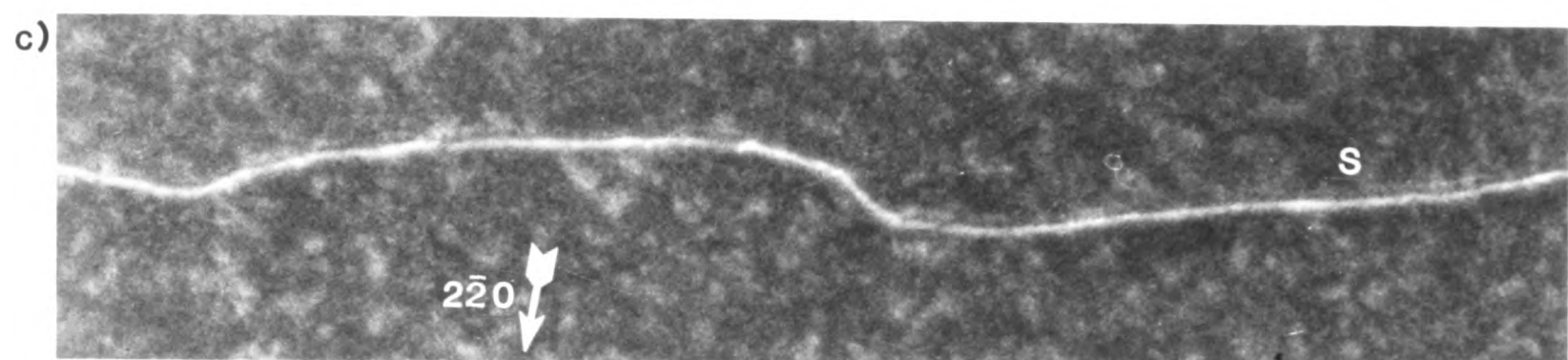
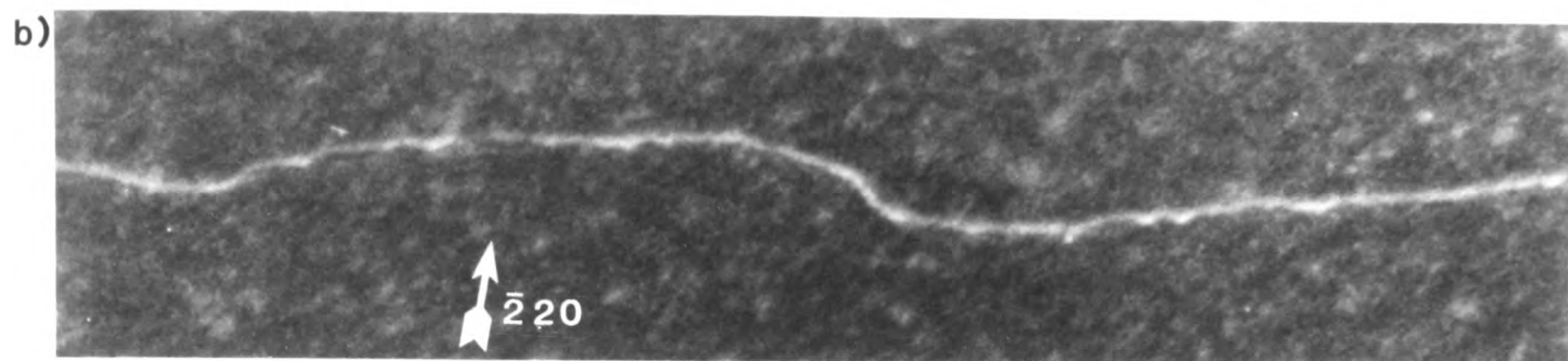
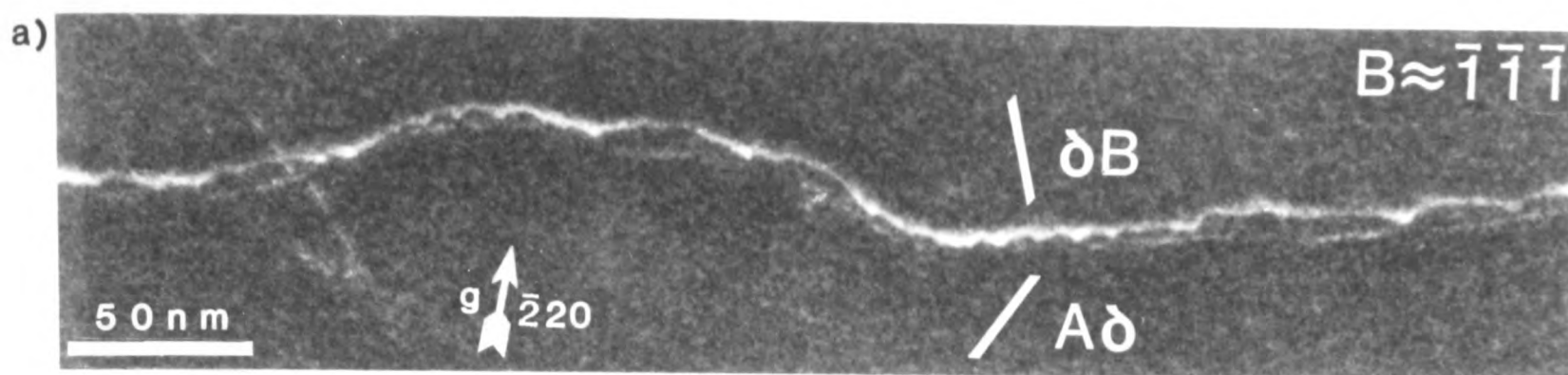
Figure 7.2; 60° Dislocation in Fe-15%Ni-17%Cr-2%Mo; 0.06 dpa at $\sim 420^\circ\text{C}$.

Observations:

Micrographs (a) and (b) are taken before, and micrographs (c) to (g) after, irradiation. The edge partial $\vec{A}\vec{\delta}$ is imaged in $\tilde{g} = [0\bar{2}2]$ ((a)) and is the brighter of the two partials visible in the $|\tilde{g} \cdot \tilde{b}_T| = 2$ image ((b)). It can be seen that the dislocation contains a large number of constrictions before irradiation, as evidenced by the cusps along $\vec{A}\vec{\delta}$. A particularly sharp cusp at 'A' may indicate pinning of the line.

After irradiation, the dislocation exhibits behaviour similar to that seen in the previous figure (figure 7.1). The upper (edge) partial is the brighter in (c), from which micrograph it can be seen that almost all constrictions along the length of the dislocation have disappeared. The dislocation has also straightened at 'A'. The lower partial is very weakly imaged in micrograph (c), however, both partials

Figure 7.1



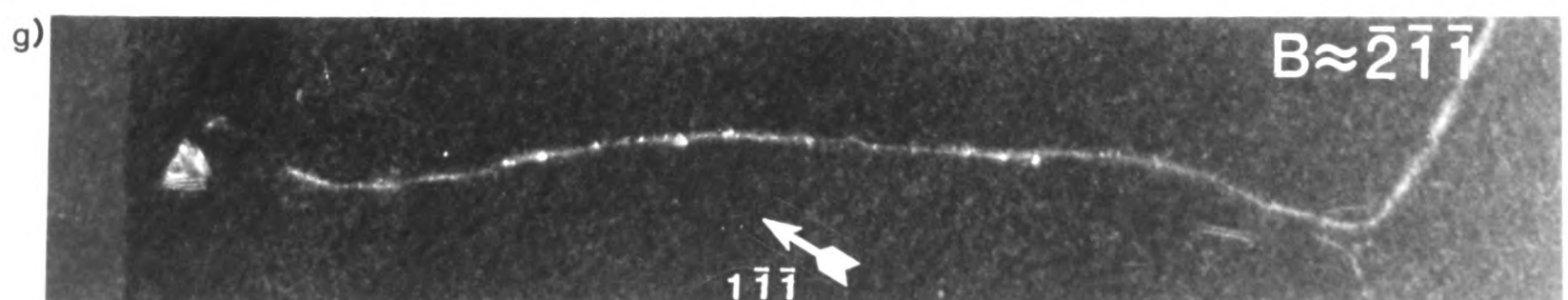
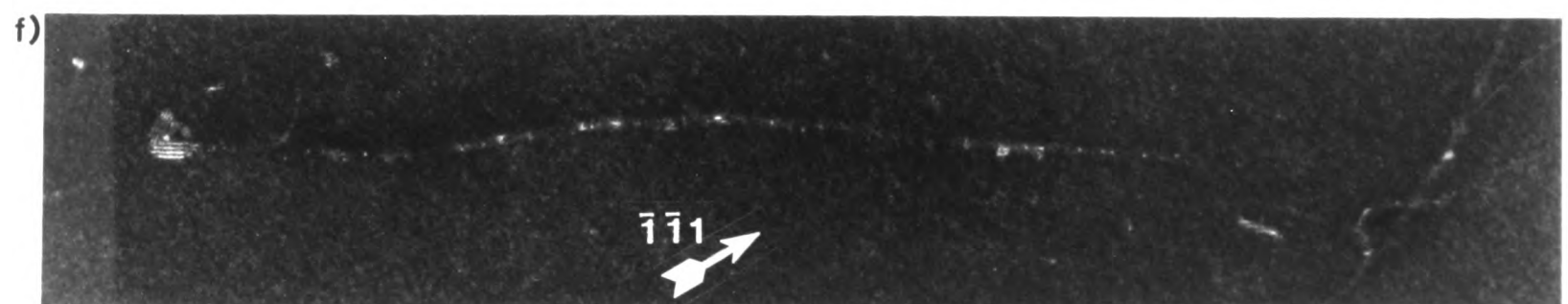
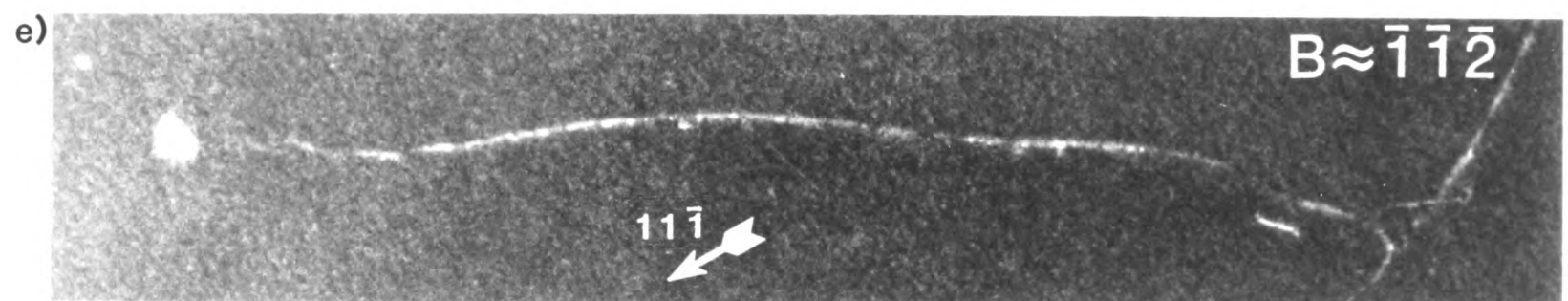
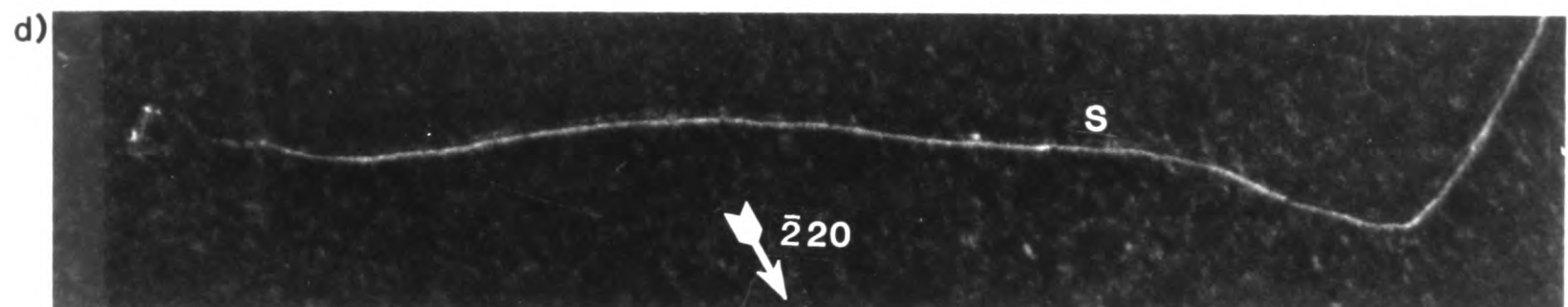
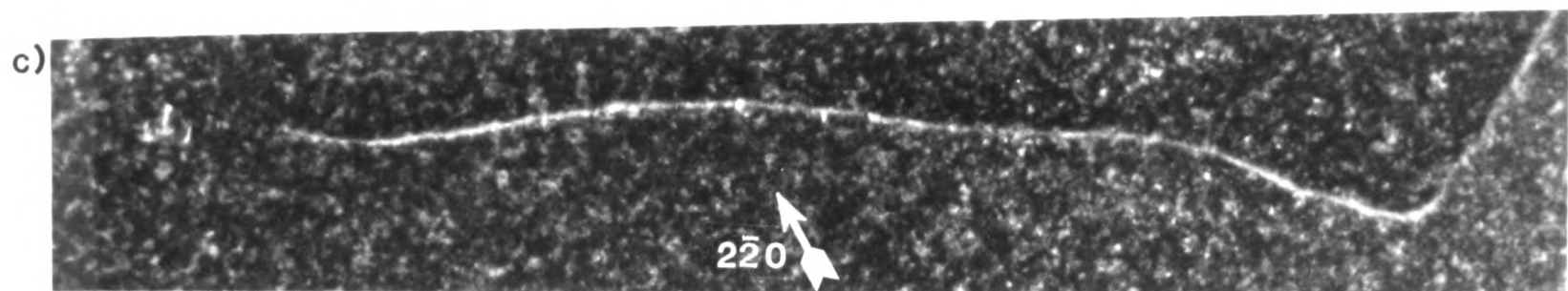
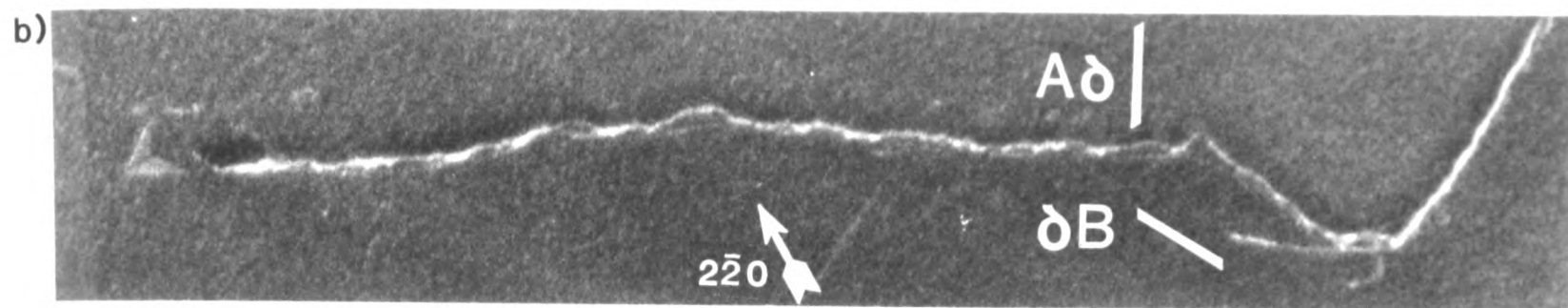
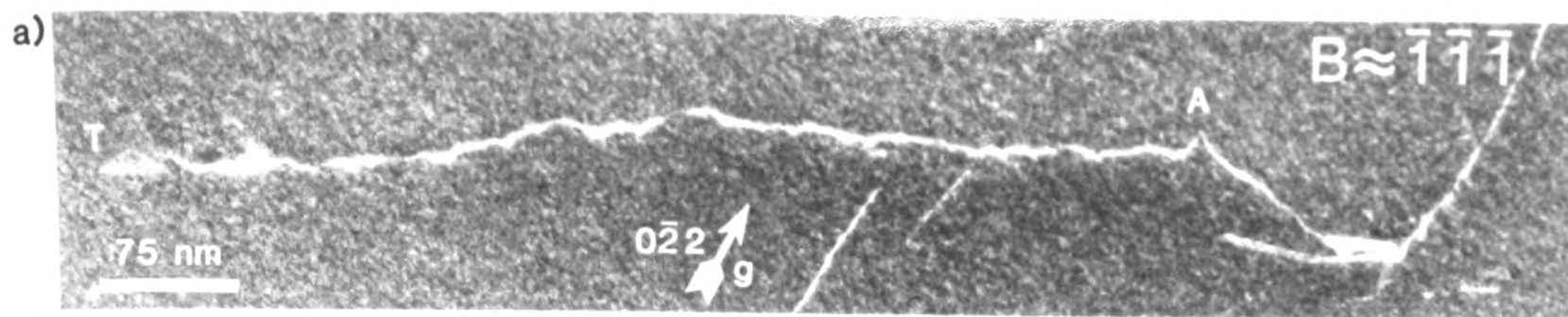
Positive line direction



Near-edge Dislocation in
Fe-15%Ni-17%Cr-2%Mo (at.%)

0.03 dpa at $\sim 420^\circ\text{C}$

Figure 7.2

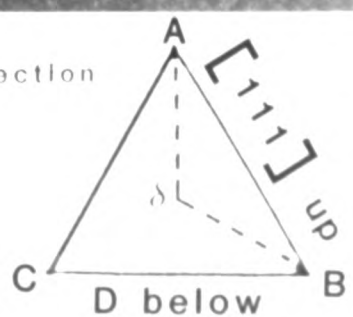


60° Dislocation in
Fe-15%Ni-17%Cr-2%Mo (at.%)

0.06 dpa at ~420°C



Positive line direction



may be seen in the opposite $|\tilde{g} \cdot \tilde{b}_T| = 2$ reflection ((d)) from which it can be seen that the dislocation is now evenly dissociated. Further evidence for the continuing dissociation of the dislocation is provided by the $\tilde{g} \cdot \tilde{b}_T = 0$ image (e), in which the stacking fault is clearly resolved. The partial image separation at 'S' (60°) is approximately 50 Å.

In the opposite reflection ((f); $\tilde{g} = [\bar{1}\bar{1}1]$) the stacking fault gives only weak contrast, allowing cluster damage along the dislocation to be more clearly seen. Many of these clusters are visible as small arrow-heads suggesting that they are vacancy SFT. These SFT were confirmed to lie ~ 15 -20 nm. above the dislocation (i.e., to its compressional strain field side) by a large angle tilt experiment (not shown).

Another feature shown both before and after irradiation is the large, fully formed, pre-existing SFT at left ('T'). Each side of this SFT is approximately 35 nm. long. After irradiation this SFT is apparently partially eroded, its sides now being only ~ 25 nm. long. This erosion appears to have occurred from all apices of the tetrahedron, but is more marked at the vertically downward pointing apex. This is best shown by the $\tilde{g} = \langle 1\bar{1}\bar{1} \rangle$ reflections (f) and (g).

Figures 7.1 and 7.2

Interpretation:

Similar behaviour is exhibited by both dislocations in these figures. The 0.03 dpa irradiation (figure 7.1) was the lowest dose irradiation performed in this study and little damage has resulted, whilst the higher density of SFT close to the dislocation of figure 7.2 is consistent with the higher dose. The disappearance of constrictions apparently occurs at an early stage in the irradiation, which is in agreement with observations first made in chapter 3. The erosion of the large SFT ('T'; figure 7.2), likely to result from interstitial absorption, may suggest that interstitials are also arriving at both dislocations. These interstitials may then core diffuse along the dislocations, particularly after the disappearance of pre-existing constrictions. Behaviour exhibited by these dislocations therefore appears consistent with mechanisms previously described (see chapter 3).

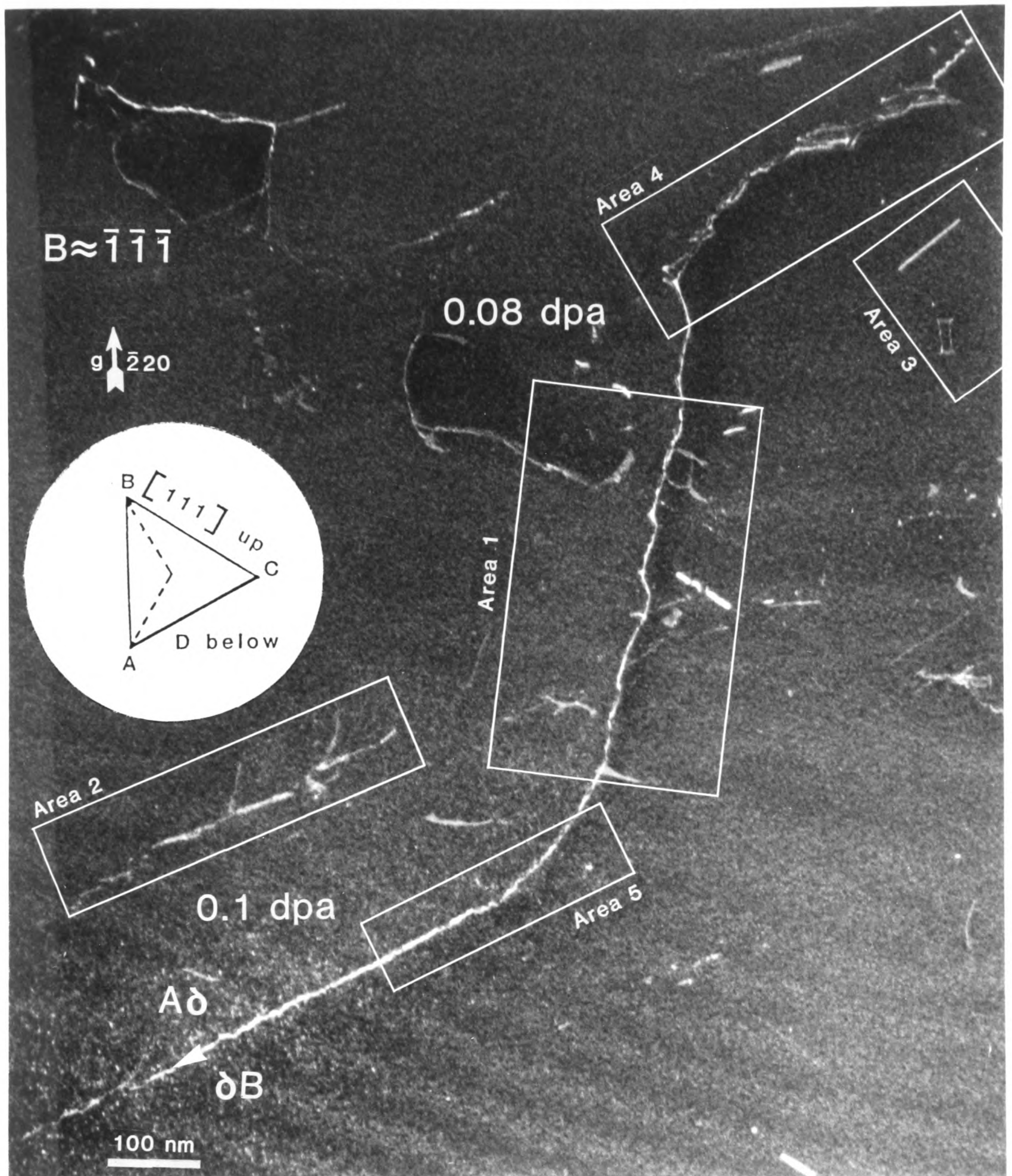
Figure 7.3; 60° - 0° - 60° Dislocation in Fe-15%Ni-17%Cr-2%Mo; Before Irradiation.

Figure 7.3 shows a long dislocation, and the region surrounding it, prior to irradiation. Five areas have been selected, which show certain interesting features after irradiation, and are enlarged in the following five figures (figures 7.4 - 7.8). Areas 1, 3 and 4 were irradiated to ~ 0.08 dpa whilst areas 2 and 5 received ~ 0.1 dpa. Both irradiations were performed at approximately 420°C. The long dislocation has total Burgers vector $\tilde{b}_T = \overrightarrow{AB}$, with the line direction and senses of individual partials as

Figure 7.3

Before Irradiation

60°-0°-60° Dislocation in Fe-15%Ni-17%Cr-2%Mo (at.%)



Before irradiation at $\sim 420^\circ\text{C}$ to doses shown

shown at lower left in the figure. The extra half-plane associated with mixed sections lies below this dislocation.

‘Area 1’: *Figure 7.4; Near-Screw Dislocation in Fe-15%Ni-17%Cr-2%Mo; 0.08 dpa at $\sim 420^\circ\text{C}$.*

Observations:

Micrographs (a) and (b) are taken before irradiation. The left-handed near-screw dislocation $\tilde{b}_T = \vec{AB}$ has $|\tilde{g} \cdot \tilde{b}_T| = 2$ in (a) whilst a 0° - 60° dislocation $\tilde{b}_T = \vec{AC}$ has $|\tilde{g} \cdot \tilde{b}_T| = 2$ in micrograph (b). A large number of other dislocations is also evident in these reflections, some of which exhibit dipole-like contrast (e.g. ‘M’; see figure 1.3) in the full series of reflections taken prior to irradiation.

After irradiation ((c) to (h)), many cusps evident along both dislocations $\vec{AB}$ and $\vec{AC}$ before irradiation have been removed, whilst three large new cusps (‘K’, ‘L’, ‘M’) have formed. The position of ‘M’ apparently corresponds to that of a pre-existing cusp, which may be a jog, where dislocation $\vec{AC}$ passes from 60° to the screw orientation. This kink is apparently confined to a plane δ and has promoted the formation of a large amount of cluster damage nearby. These clusters lie close to the pre-irradiation dislocation position and many of them can be recognized as SFT (‘T’; see micrograph (g)). They are larger and more frequent near kink ‘M’ than elsewhere along this dislocation.

In micrograph (c), there is some evidence for dislocation $\vec{AB}$ to remain dissociated near ‘K’; this is not confirmed by other reflections, however. Segment ‘X’ appears to show SFC in micrograph (e) suggesting that it is inclined in the foil. This segment is also rotated anti-clockwise on tilting from $\tilde{B} \approx [\bar{1}\bar{1}\bar{1}]$ to (121) or (211) poles. This is consistent with it being fairly steeply inclined downwards in the foil, suggesting the onset of the formation of a right-handed helix. Segment ‘Y’ also shows SFC in both micrographs (e) and (f), suggesting that it is faulted on either of planes γ or δ . Since SFC is not seen from this segment in $\tilde{g} = \pm [11\bar{1}]$, and the change in its shape on tilting suggests that it is inclined in the foil, in common with segment ‘X’, it is thought that both segments ‘X’ and ‘Y’ lie on a plane γ . The behaviour of this near-screw dislocation therefore appears to be consistent with similar observations in other materials (see figure 3.4, for example), indicating the absorption of interstitials at jogs, leading eventually to the formation of a helical structure.

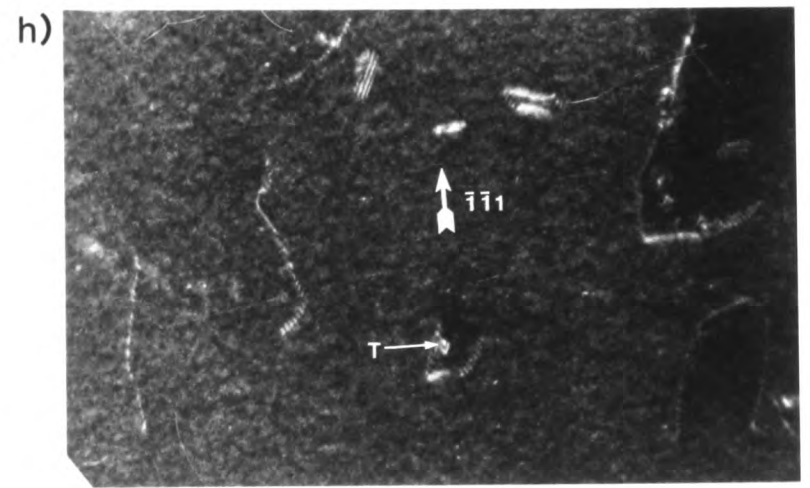
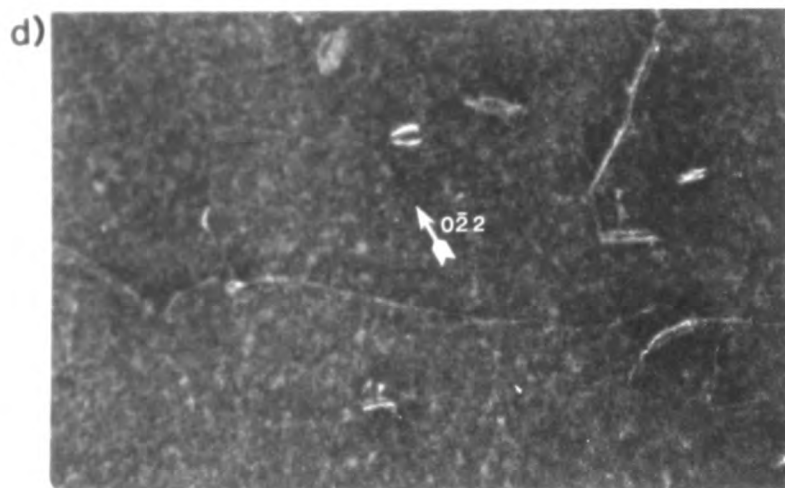
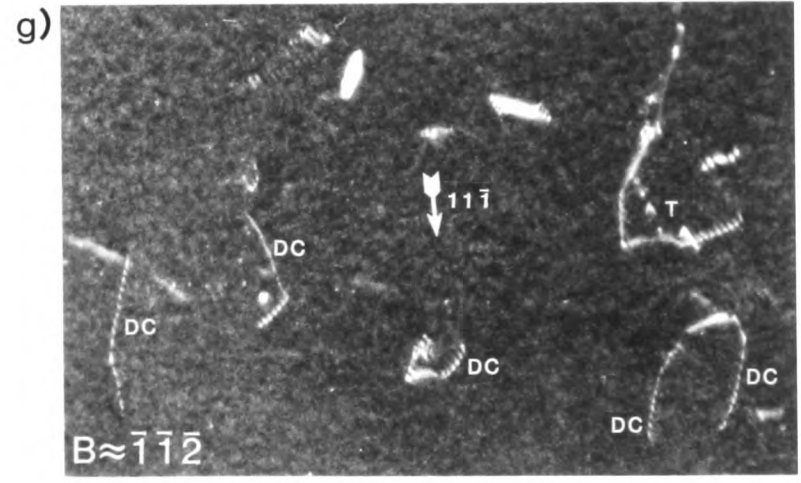
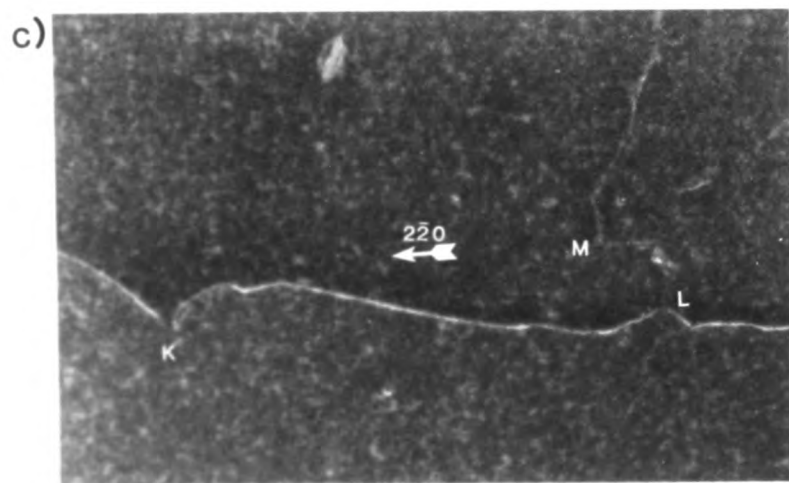
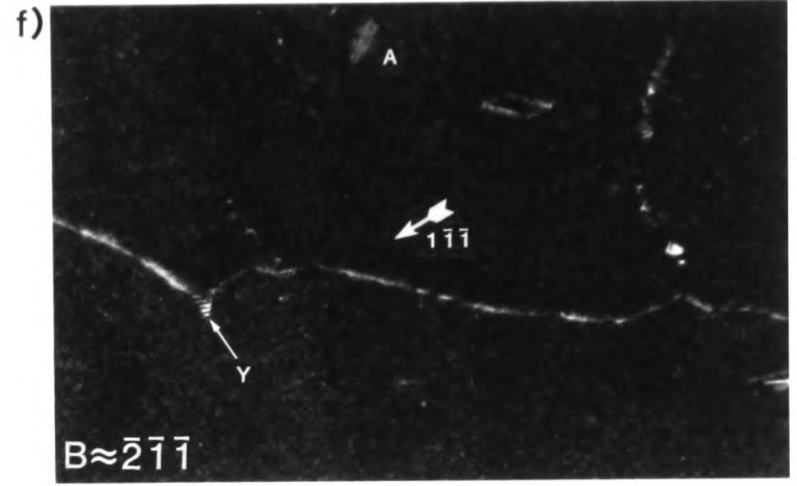
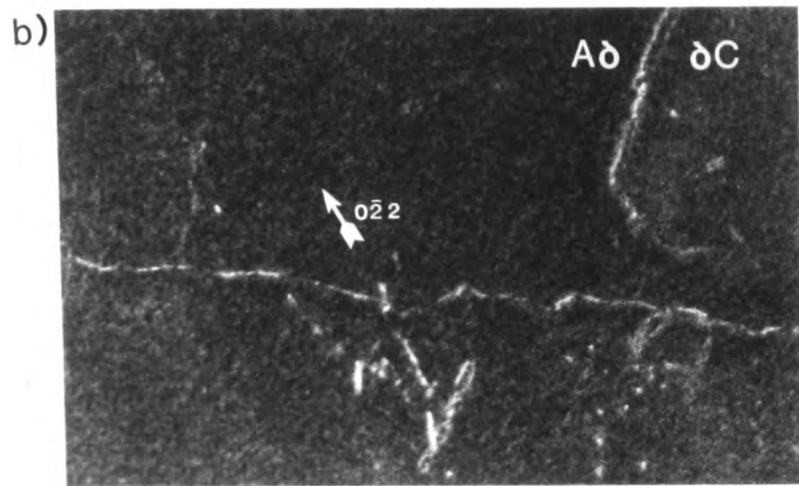
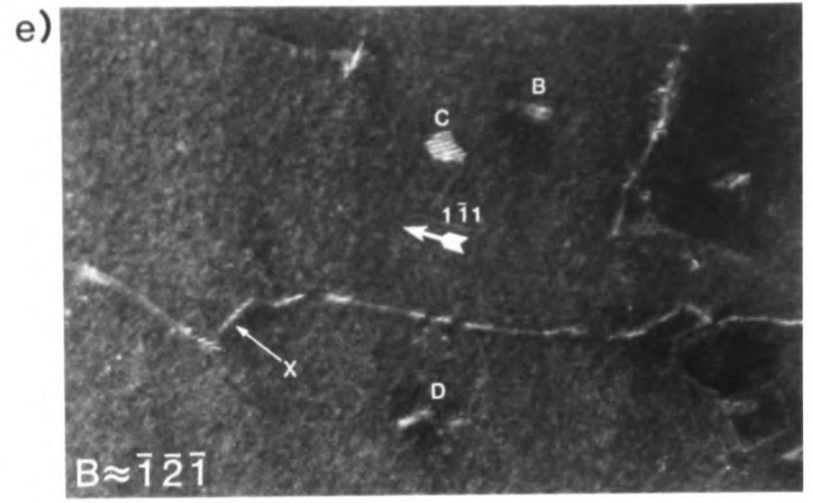
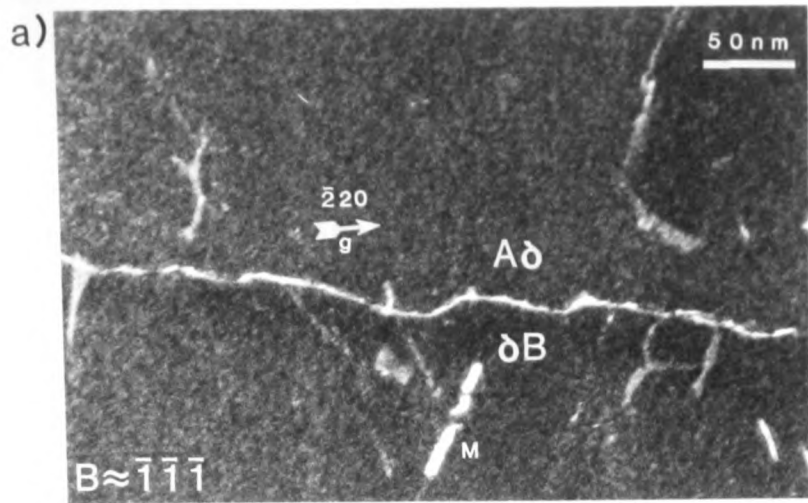
Several features can be seen in the background, removed from the dislocation lines; those labelled could be characterized as follows:

‘A’, is an interstitial Frank loop with Burgers vector $\vec{\alpha A}$;

‘C’, is an interstitial Frank loop with Burgers vector $\vec{\gamma C}$;

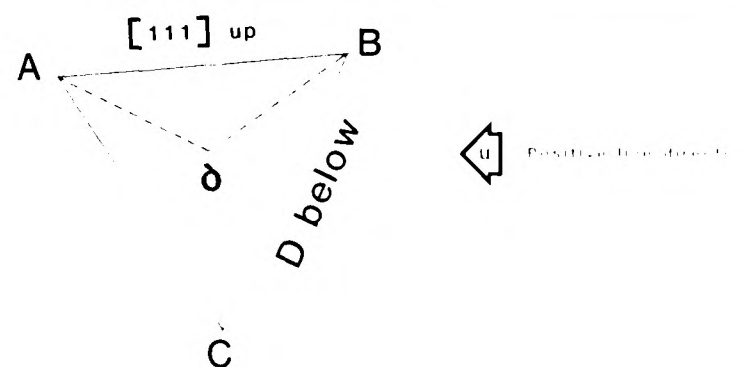
Feature ‘B’ is apparently more complex and may represent an interstitial perfect prismatic loop with Burgers vector $\tilde{b}_L = \vec{DB}$, formed via the unfauling of a Frank loop $\tilde{b}_L = \vec{\beta B}$, in the process of

Figure 7.4
‘Area 1’



Near-screw Dislocation in
Fe-15%Ni-17%Cr-2%Mo (at.%)

0.08 dpa at ~420°C



gliding towards edge orientation. Contrast seen in $\tilde{g} = \pm [11\bar{1}]$ is believed to result from dissociation of the loop edges onto inclined planes (β in this case). The contrast exhibited by parallelogram shaped loops in this material is examined in figure 7.6.

In common with many features marked in micrograph (g), feature 'D' apparently has Burgers vector $\tilde{b}_T = \pm \vec{DC}$. In micrograph (h), however, it is particularly noticeable that a small SFT has formed within feature 'D'. This suggests that 'D' is interstitial in nature and is therefore likely to have Burgers vector $\tilde{b}_L = \vec{DC}$. The formation of this SFT may be related to the apparent absence of one side of this feature, which may indicate intersection with a foil surface. Many of the other dislocations 'DC' may have been pre-existing.

Finally, notice that dipole 'M' has disappeared after irradiation.

Interpretation:

As mentioned above, the zig-zagging of dislocations in this figure is apparently consistent with previous observations. This is probably a result of interstitial climb, as may be the disappearance of dipole 'M'. Interestingly most of the pre-existing cusps, particularly towards the middle of dislocation $\vec{AB}$, do not appear to have given rise to a zig-zagged section of the line. These pre-existing cusps are close to the dipole 'M', and numerous other possible defect sinks, and may not have received a large interstitial flux. An alternative explanation, discussed in chapter 8, is that some pre-existing cusps may originate from solute pinning. It is possible, therefore, that only those cusps which represent jogs (see **Carter & Ray**, 1974) lead to zig-zagging of the line.

'Area 2': *Figure 7.5; Dislocations in Fe-15%Ni-17%Cr-2%Mo; 0.1 dpa at $\sim 420^\circ\text{C}$.*

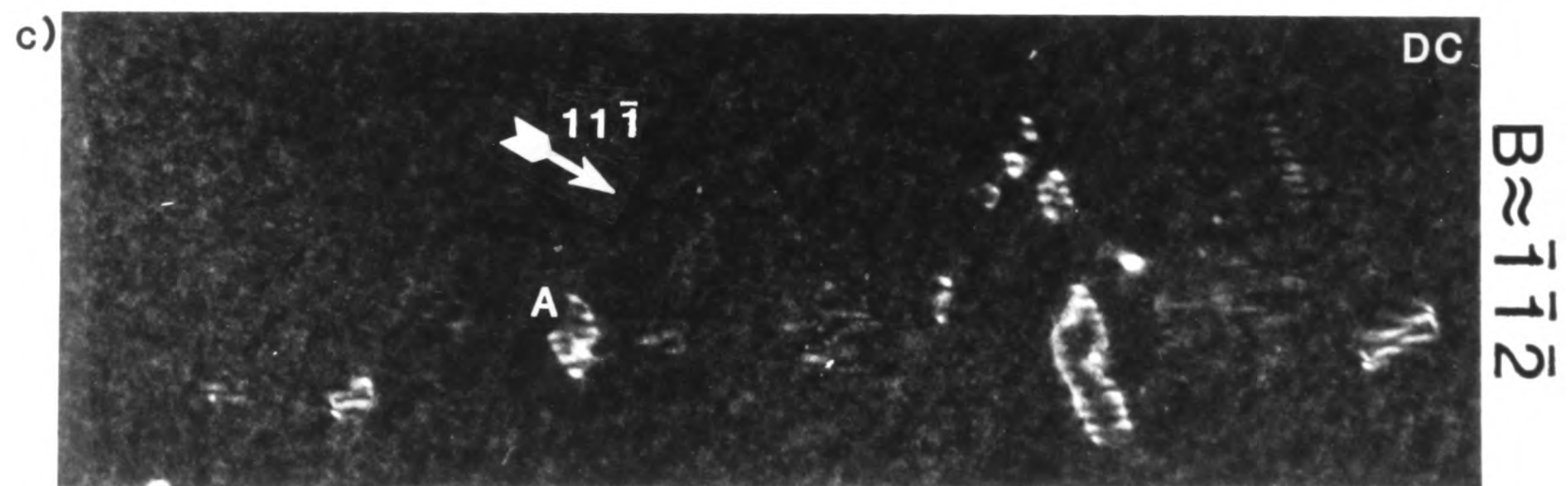
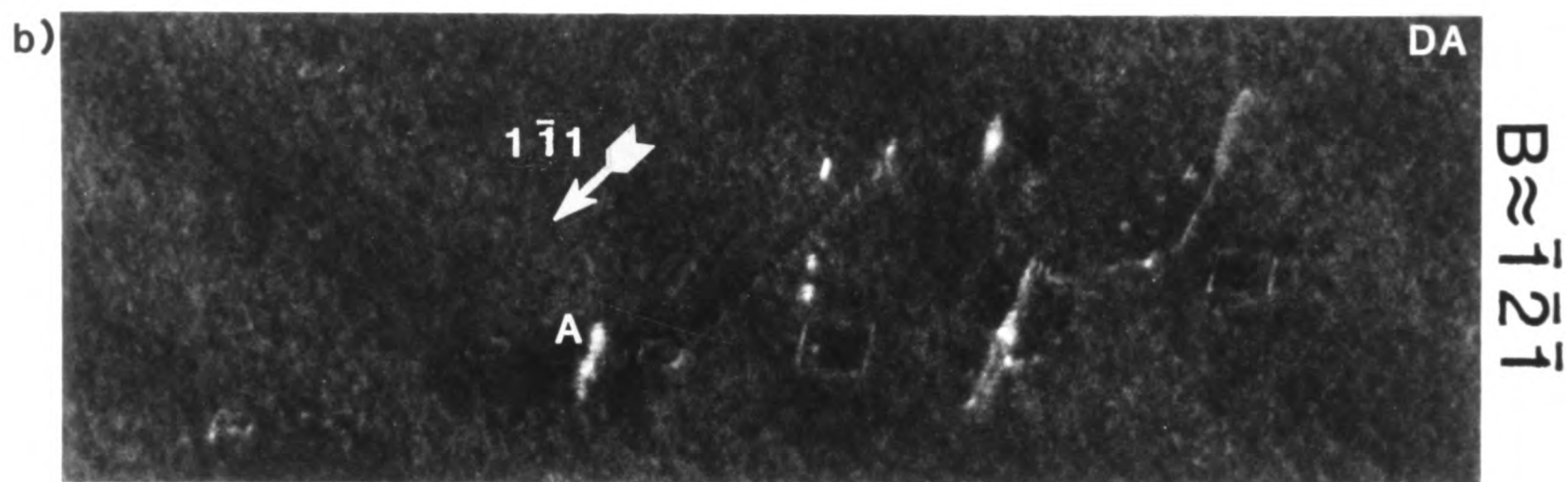
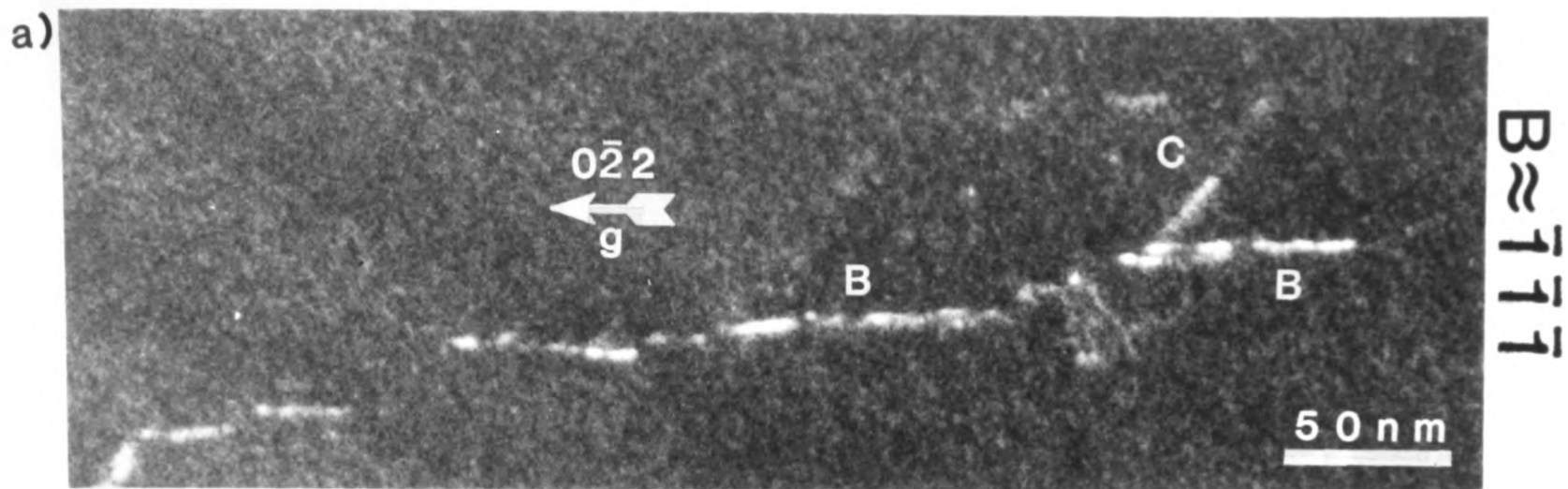
Observations:

In this three micrograph figure large interstitial clusters are seen to have formed preferentially close to dislocations.

The dislocations shown in micrograph (a), which was taken prior to irradiation, were all confirmed to have Burgers vector $\tilde{b}_T = \pm \vec{AD}$ via a contrast analysis. Dislocations marked 'B' are believed to be glissile on planes β , and that marked 'C' to lie on a plane γ . It is probable that these dislocations are steeply inclined in the foil, running from surface to surface.

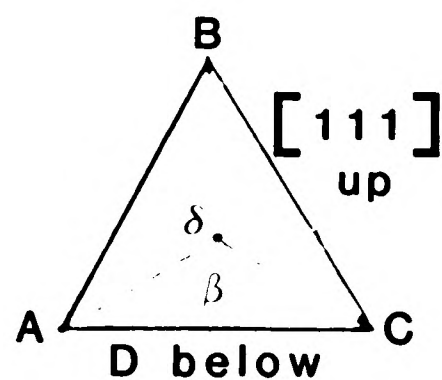
Micrographs (b) and (c) are taken after irradiation. In these, and all other, reflections of the full contrast series dislocations 'B' are no longer seen. Dislocation 'C' has a dramatically altered shape, having moved away from a small amount of vacancy cluster damage close to its pre-irradiation position. A number of large parallelogram-shaped loops are seen. These loops all have Burgers vector

Figure 7.5
‘Area 2’



Dislocations in
Fe-15%Ni-17%Cr-2%Mo (at.%)

0.1 dpa at $\sim 420^\circ\text{C}$



$\tilde{b}_L = \vec{D}\vec{A}$, shown here in ‘outside’ contrast. The top edges of these loops (as the figure is viewed) apparently lie very close to the former positions of dislocations ‘B’. Feature ‘A’ shows as a thin line aligned with the trace of plane γ in this micrograph. On tilting to the (112) pole, however, it can be seen that ‘A’ does not lie on a plane γ , but shows very similar contrast to a number of other loops, which were confirmed to be of interstitial type, with Burgers vector $\vec{D}\vec{C}$, by a full contrast analysis. Notice, however, that the edges of these loops show ‘strong’ contrast in $\tilde{g} = [11\bar{1}]$, as does feature ‘D’ of the preceding figure (figure 7.4). Contrast from these loops, which appears confusing at first, is examined employing similar features in the following figure.

‘Area 3’: Figure 7.6; Dislocations in Fe-15%Ni-17%Cr-2%Mo.

Pages 1 & 2: Before Irradiation and (after) 0.08 dpa at $\sim 420^\circ\text{C}$; Page 3: Schematic Diagram.

Observations:

The growth and morphology of, and contrast from, interstitial loops nucleated at pre-existing features is examined in this figure. Micrographs (a) to (c) show three independent $\tilde{g} = \langle 2\bar{2}0 \rangle$ type reflections prior to irradiation. Both features ‘A’ and ‘B’ are in contrast in (a) and (b). In micrograph (c), ‘B’ is out-of-contrast whilst ‘A’ shows very weak contrast in both this reflection and its opposite (not shown). In $\tilde{g} = \pm [2\bar{2}0]$ feature ‘A’ showed strong/weak contrast typical of a dipole. This is consistent with these features having Burgers vector $\tilde{b} = \vec{D}\vec{A}/\vec{A}\vec{D}$ (‘A’) or $\vec{D}\vec{A}$ (‘B’). It should be noted that dislocation ‘A’ is aligned closely with $[0\bar{1}1]$, whilst loop ‘B’ has its longer sides aligned with $[\bar{1}10]$.

After irradiation large rectangular loops now occupy the former positions of ‘A’ and ‘B’. Some part, or all, of both of these loops is apparently visible in all reflections taken after irradiation. It can be seen that all parts of loops at ‘A’ are in contrast in $\tilde{g} = \pm [0\bar{2}2]$, $[20\bar{2}]$ and $[11\bar{1}]$ whilst only sections are visible in other reflections. These loops apparently show ‘inside’ contrast in $\tilde{g} = [0\bar{2}2]$ and $[\bar{1}\bar{1}1]$. Similarly, the now very large loop at ‘B’ is only wholly visible in $\tilde{g} = \pm [0\bar{2}2]$, $[\bar{2}20]$ and $[11\bar{1}]$. It apparently shows ‘inside’ contrast in $\tilde{g} = [0\bar{2}2]$ and $[\bar{1}\bar{1}1]$.

The visibility of isolated loop segments in several reflections should not be confused with residual $\tilde{g} \cdot \tilde{b} \wedge \tilde{u}$ contrast (see, for example, Hirsch et al, 1965, p.261 and their figures 7.20 & 11.14), since the side perpendicular to $\tilde{g}$ is out of contrast here. It is believed, however, that these reflections still represent $|\tilde{g} \cdot \tilde{b}_T^{\text{loop}}| = 0$ as explained below. This allows a preliminary characterization of loops at ‘A’ and ‘B’ as perfect prismatic loops, with Burgers vectors $\vec{D}\vec{C}$ and $\vec{D}\vec{A}$ respectively. These Burgers vectors represent interstitial defects.

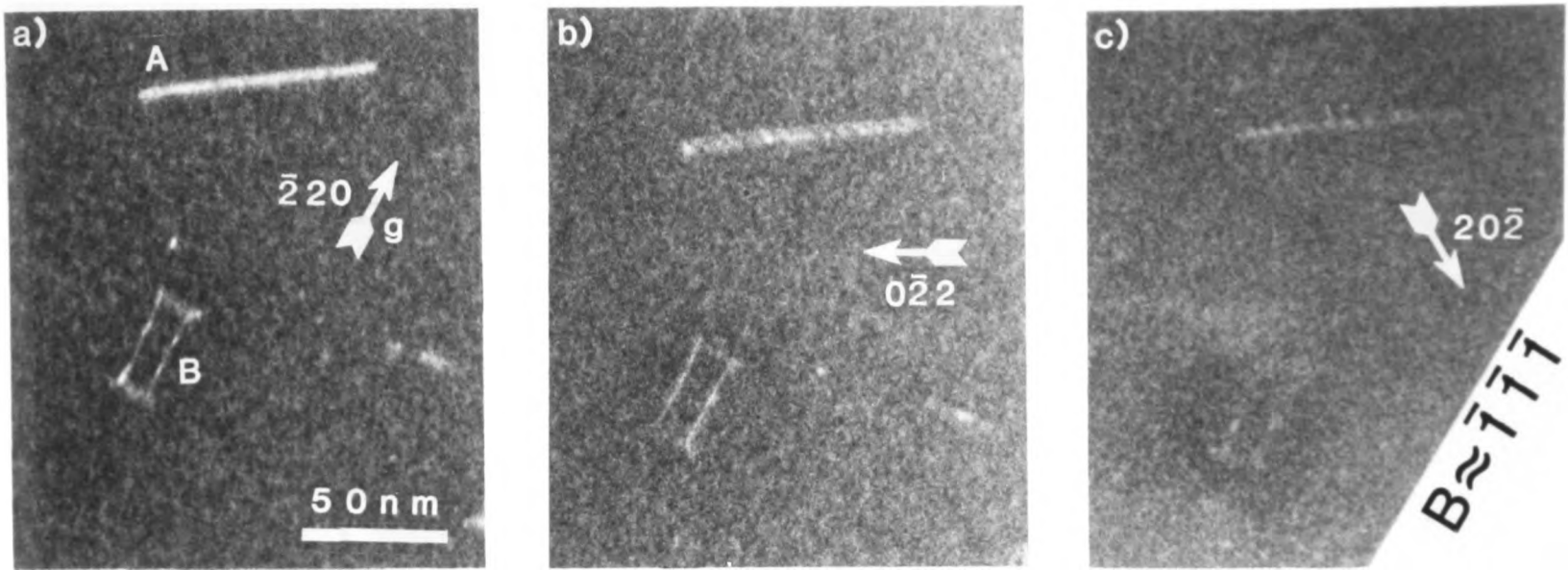
The characterization of these loops as being of interstitial type is reinforced by the frequent observations of vacancy damage close to their inner peripheries. These SFT are at various stages in

Figure 7.6
(3 pages)

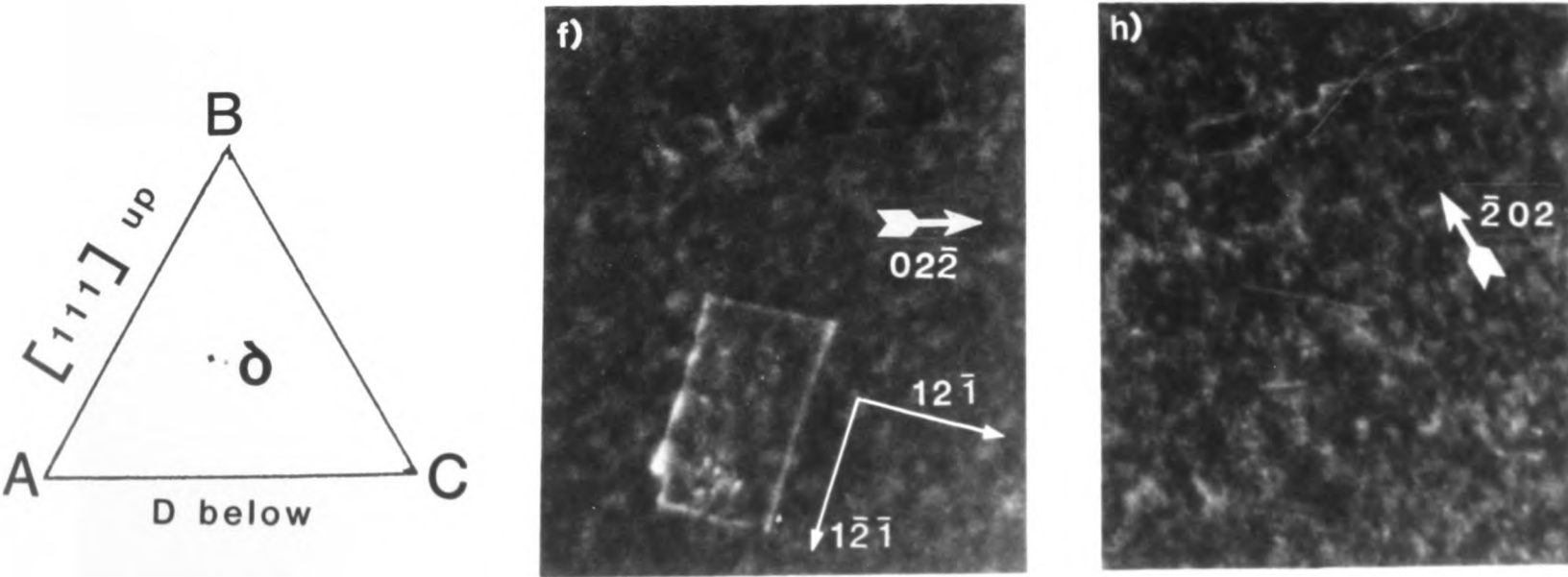
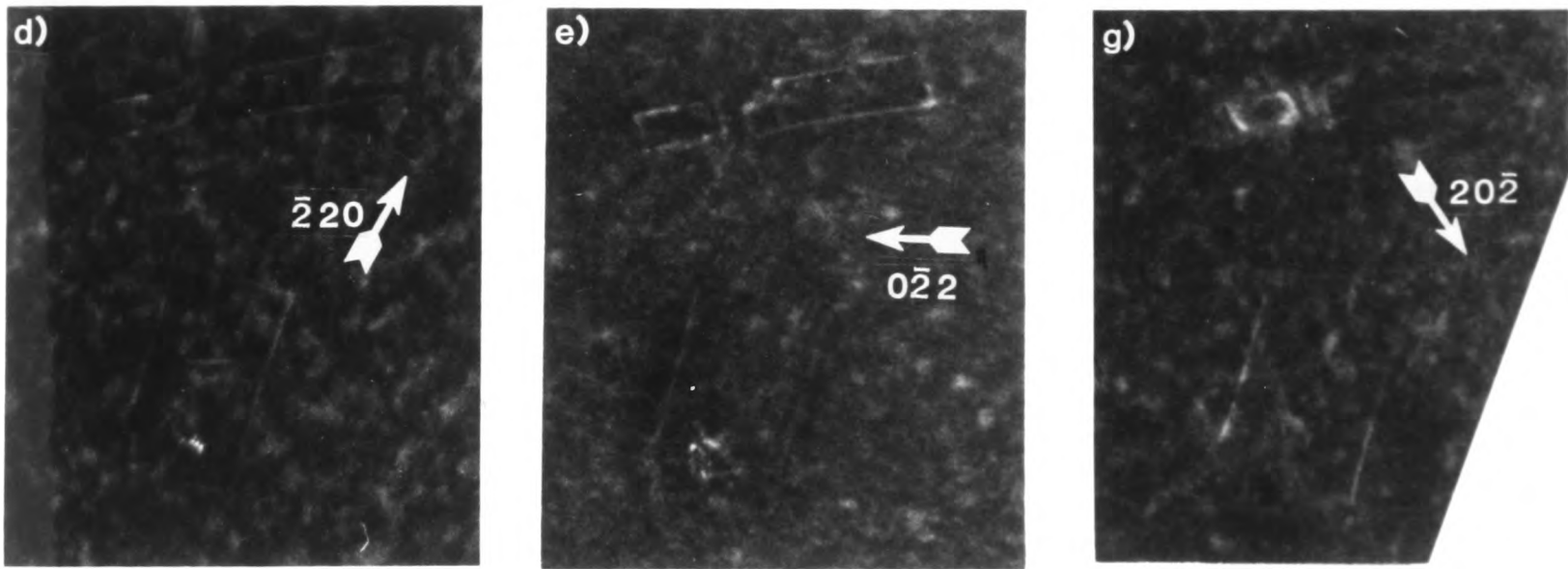
‘Area 3’

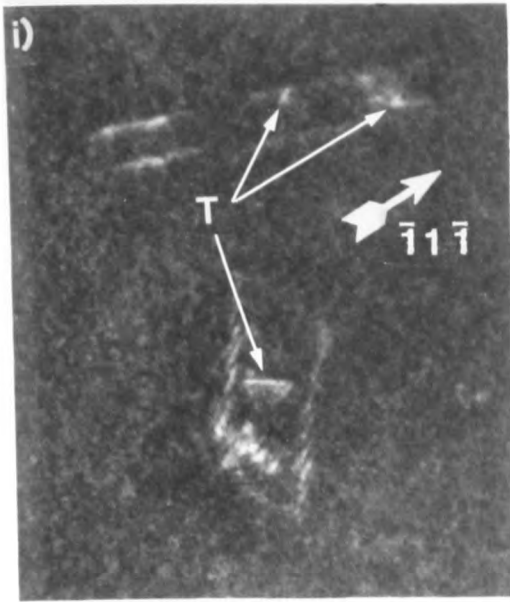
Dislocations in Fe-15%Ni-17%Cr-2%Mo (at.%)

Before Irradiation

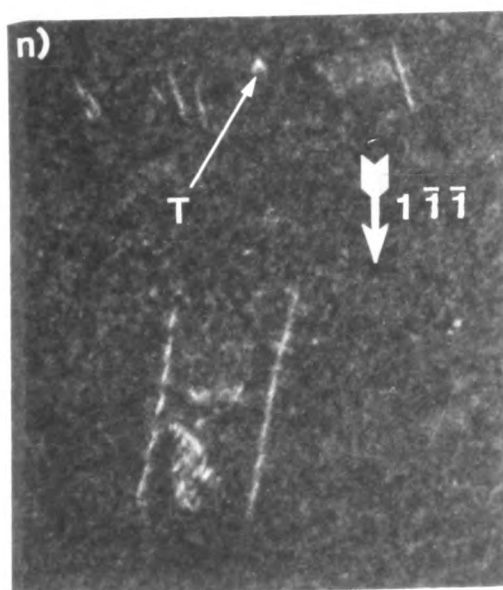
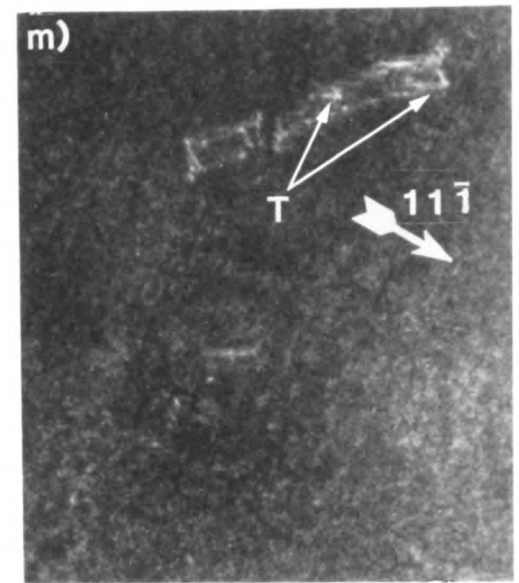
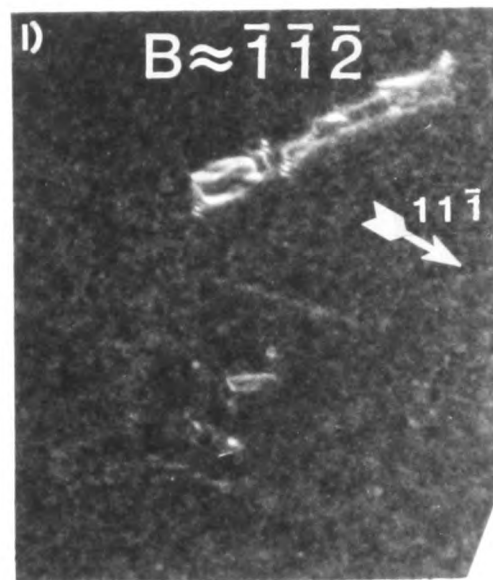
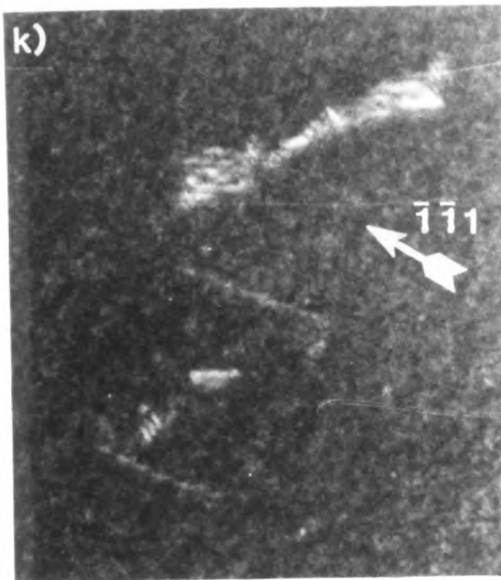
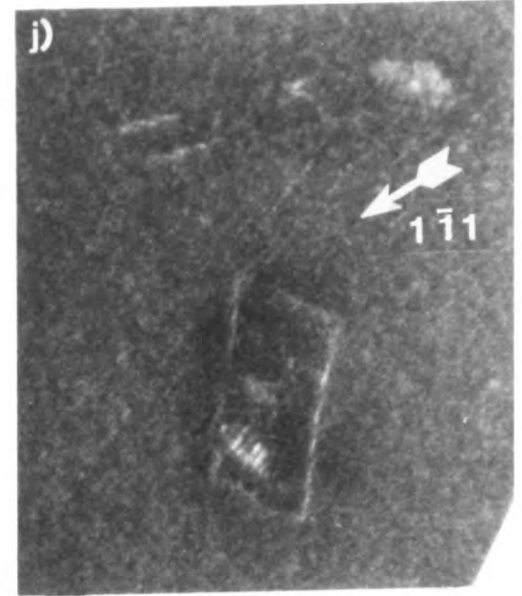


0.08 dpa at $\sim 420^\circ\text{C}$

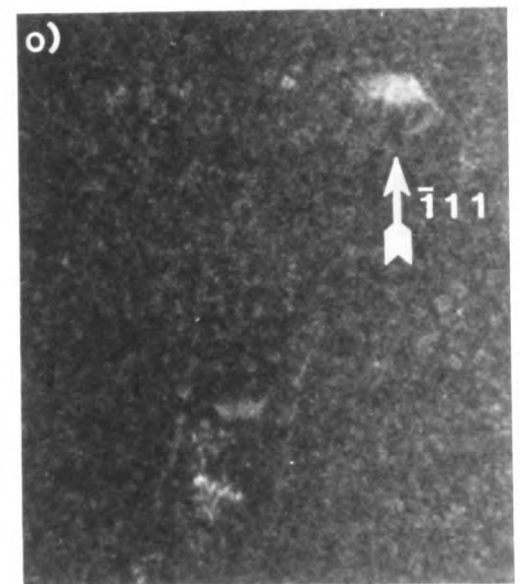




$$B \approx \frac{1}{2}[111]$$

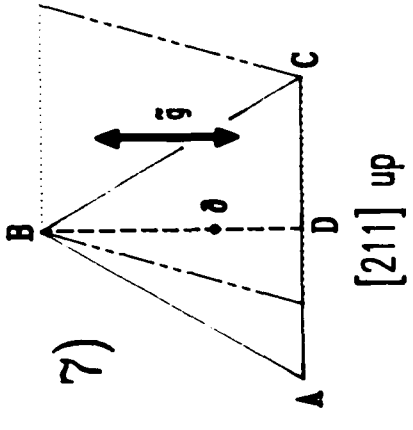
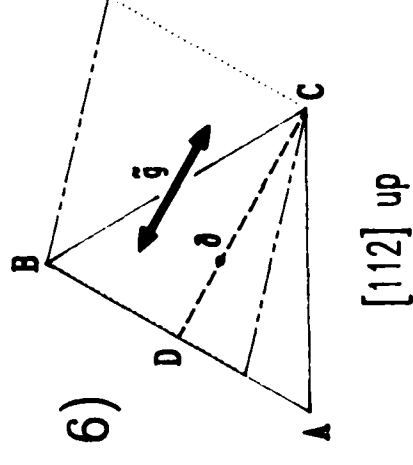
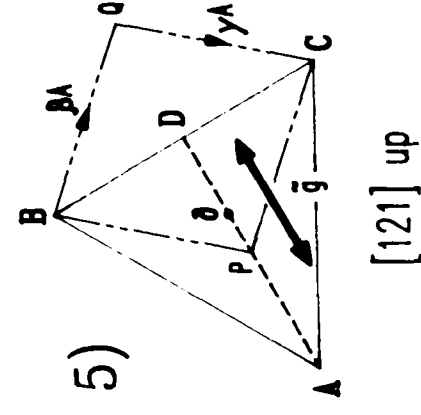
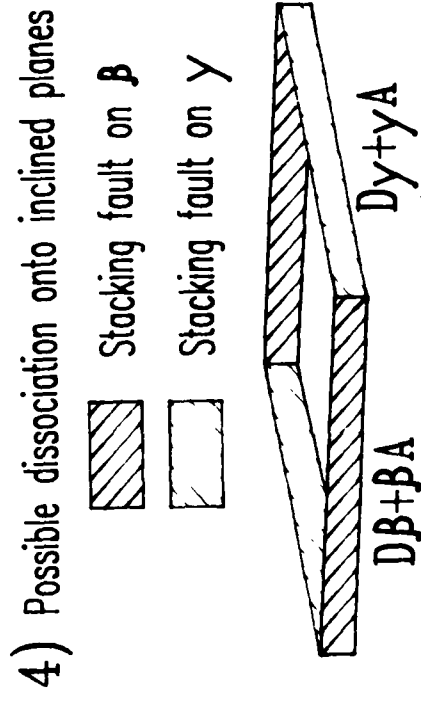
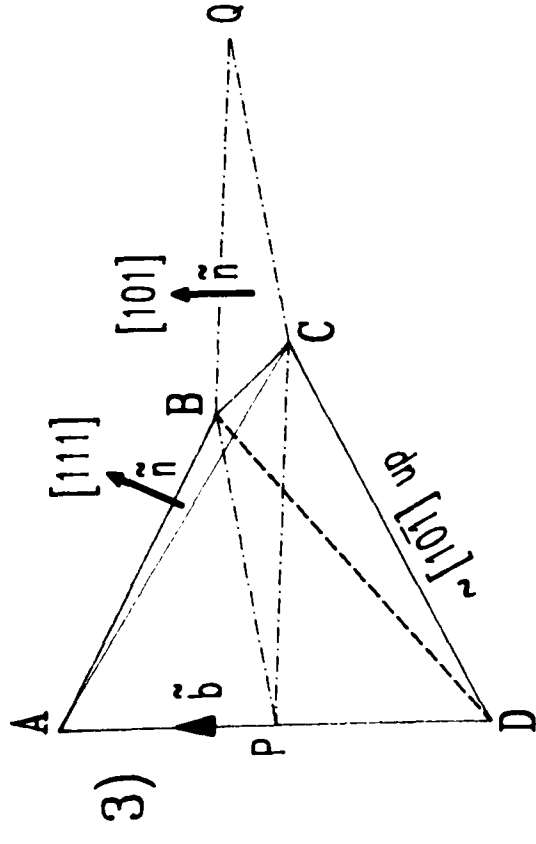
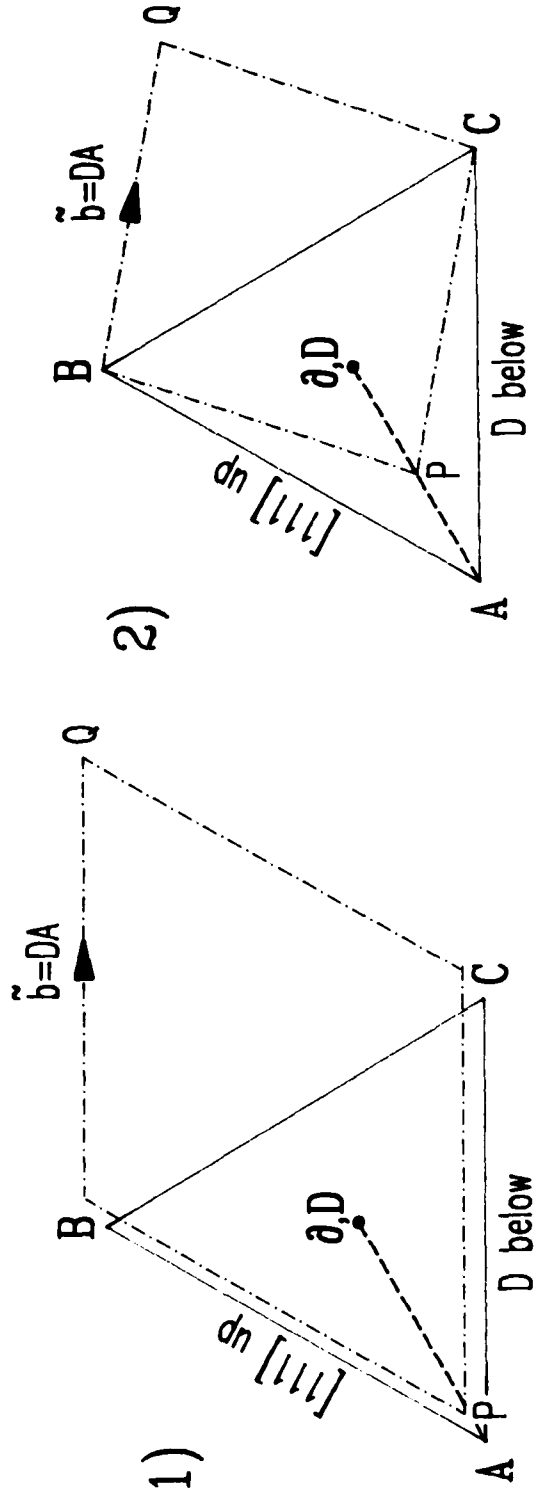


$$B \approx \frac{1}{2}[111]$$



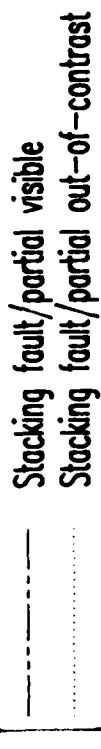
Dislocations in Fe-15%Ni-17%Cr-2%Mo (at.%)

0.08 dpa at ~420°C



Possible Explanation of Contrast from Loop 'B'

- 1) Formation of perfect prismatic loop on α .
- 2) Sections CP and BQ may glide on β . Sections PB and QC may glide on γ . $[111]$ view. Loop DA now in pure edge orientation.
- 3) Near $[101]$ view. Loop DA on (101) plane.
- 4) DA may be dissociated on β and γ .
- 5) - 7) Schematic diagrams of expected contrast and loop shape on tilting to $\{112\}$ poles.



their formation³⁰.

As in shown in micrograph (f), the edges of loop 'B' align very closely with the projections of directions $[12\bar{1}]$ and $[1\bar{2}\bar{1}]$. This result is confirmed by tilting to (121), (112) and (211) poles. After irradiation this loop therefore lies at, or very close to, pure edge orientation on a (101) plane. The 'segment' contrast in $\tilde{g} \cdot \tilde{b}_T = 0$ images may be explained by assuming that edges of the loop have dissociated on the inclined planes β and γ . This is sketched schematically in the third page of this figure, which also depicts the glide of loop $\tilde{b}_L = \vec{D}\vec{A}$, formed via the unfaulting of a $\tilde{b}_L = \vec{D}\delta$ Frank loop lying in the plane of the foil, towards edge orientation. It is thought that the pre-irradiation position of loop 'B', in which two edges still lie along $[1\bar{1}0]$ in the plane of the foil, represents an intermediate configuration in this glide process.

Contrast exhibited by the loops at 'A' (Burgers vector $\tilde{b}_L = \vec{D}\vec{C}$), which lie on, or very close to, a (110) plane can be shown, via a similar treatment, to be consistent with adjacent edges of these loops being dissociated on planes α and β . These loops may similarly have condensed originally as $\tilde{b}_L = \vec{D}\delta$ Frank loops.

Finally, notice that in all three figures 7.4, 7.5 and 7.6 loops which have Burgers vectors $\vec{D}\vec{C}$ have all formed close to pre-existing features which were aligned along, or close to, $[1\bar{1}0]$.

Figures 7.5 and 7.6

Interpretation:

The frequency of large interstitial loops in the bulk (see figure 7.4) is considerably less than close to pre-existing dislocations. It appears, therefore, as if interstitials have migrated to pre-existing dislocations, which have then acted as preferential loop nucleation sites. The growth of feature 'B' of figure 7.6, which has the same Burgers vector before and after irradiation, simply suggests interstitial climb. In both figures, however, pre-existing dislocations with Burgers vectors $\vec{D}\vec{A}$ have also given rise to loops of Burgers vector $\vec{D}\vec{C}$. This may suggest that the species of either the Frank loops which initially nucleate, or the prismatic loops subsequently formed, may be affected not only by the Burgers vector of the parent dislocation but also by its line direction.

The dissociation of loop edges onto inclined planes may also provide an explanation for the confusing contrast exhibited by loops $\vec{D}\vec{C}$ of figures 7.4 and 7.5 if one assumes that the strong contrast in $\tilde{g} = [11\bar{1}]$ arises not from the loop itself but instead from intrinsically faulted regions on planes α and

³⁰A fully formed SFT is arrowed in (n); a nearly fully formed SFT is also arrowed in (m); a SFT at an early stage in its formation (see figure 6.4) is the third feature arrowed in (i). See **Jenkins (1973)** for further details.

β . These micrographs were not taken with $s=0$ at $2\tilde{g}$, however, as required for the accurate determination of the unique sense of a fault vector ($\tilde{R}$) employing solely dark-field imaging (see, for example, **Thomas & Goringe**, 1979, p.181).

There have been very many similar observations, employing conventional strong-beam imaging, of unfaulted Frank loops on $\{110\}$ planes (e.g. **Kuhlman-Wilsdorf & Wilsdorf**, 1960; **Condat & Fayard**, 1972; **Imeson**, 1978). The paper of Condat & Fayard, in which vacancies have aggregated to form large loops after quenching in AuCu_3 , is particularly interesting in that loops are also observed in $\{013\}$, but not on $\{012\}$ (see figure 3.4), planes.

‘Area 4’: *Figure 7.7; 0° - 60° Dislocation in Fe-15%Ni-17%Cr-2%Mo; 0.08 dpa at $\sim 420^\circ\text{C}$.*

Observations:

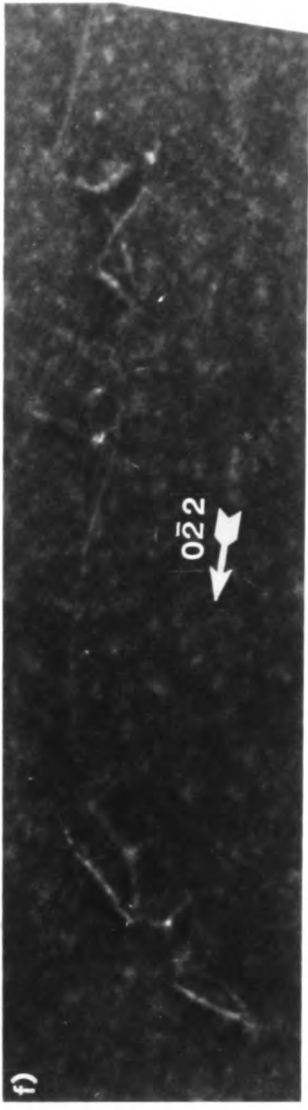
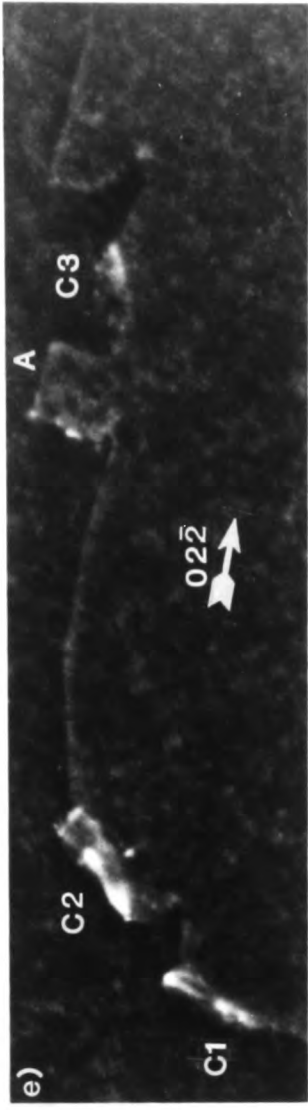
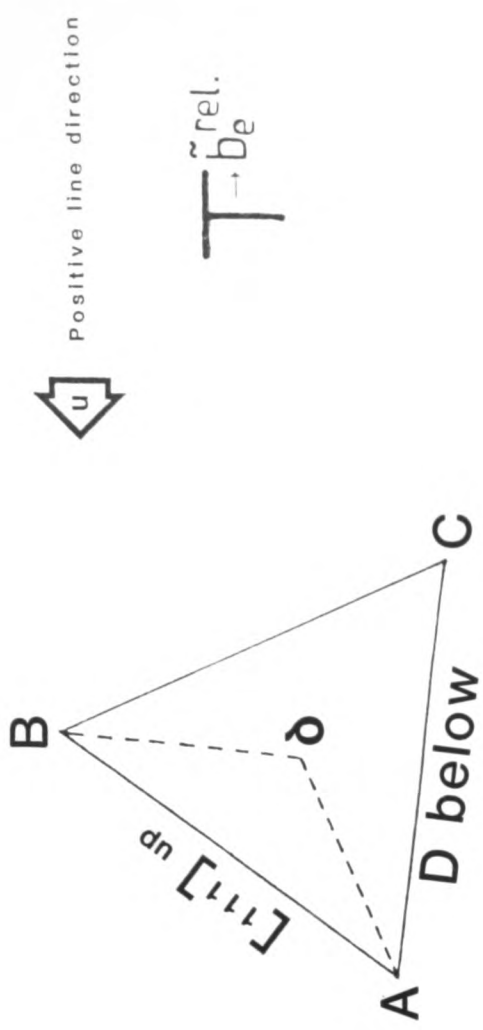
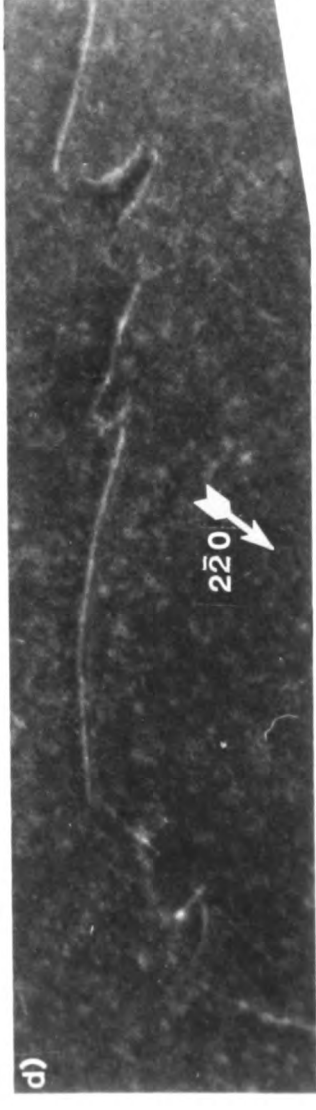
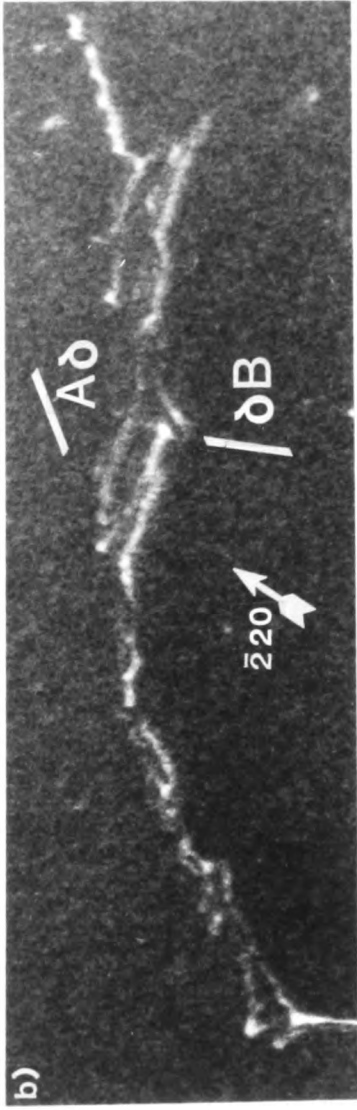
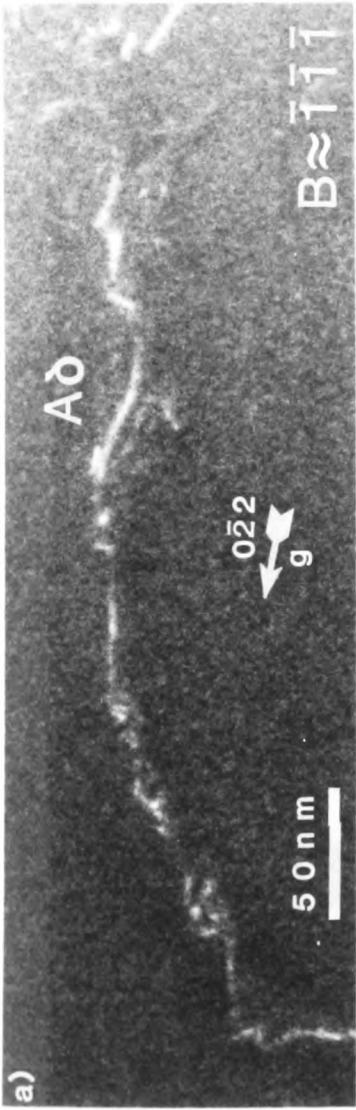
The dislocation shown within this area is particularly complex both before (micrographs (a) and (b)) and after (micrographs (c) to (n)) irradiation. Before irradiation the total Burgers vector of the dislocation(s) is apparently $\vec{AB}$; the dislocation therefore varies from screw to 60° orientation from left to right. In micrograph (a) only partial $\vec{A}\delta$ is imaged; this image seems to correspond to the uppermost line of contrast visible in (b), for which $|\tilde{g}\cdot\tilde{b}_T|=2$ for dislocation $\tilde{b}_T=\vec{AB}$. The lowest line of contrast in (b) is partial $\vec{\delta B}$. These partials are obviously far from equilibrium, being separated by as much as 25 nm. in places. There also appear to be many other features, which could not be individually characterized, between these two partials. Some of these features showed dipole-like contrast.

After irradiation the dislocation now has a substantially altered form. The tangle of features visible before irradiation between the two partials has disappeared, leaving a relatively evenly dissociated section seen towards the centre of the $|\tilde{g}\cdot\tilde{b}_T|=2$ micrographs (c) and (d). The stacking fault separating these partials can be seen in (n), as can a sparsely populated line of clusters lying parallel to it. These clusters (two are ringed) are seen to be displaced with respect to the dislocation on tilting from $\tilde{B} \approx [\bar{2}\bar{1}\bar{1}]$ to $\tilde{B} \approx [\bar{1}\bar{1}\bar{2}]$ ((l) to (n)) in a sense consistent with their lying to the compressional strain field side (below) the dislocation. The large amount of this motion (~ 5 -6 mms. at 300kx) suggests that the clusters may be substantially vertically removed (up to 40 nm.) from the line.

Four large loop-like features (‘A’, ‘C1’, ‘C2’ and ‘C3’) are seen after irradiation. All of these features apparently lie very close to the dislocation: Loop ‘A’ appears very similar to interstitial parallelogram-shaped loops shown in preceding figures (e.g., figures 7.5 and 7.6) and exhibits contrast consistent with a Burgers vector $\tilde{b}_T=\vec{DA}$. Edges of this loop appear to be dissociated on the inclined planes β and γ . Other evidence pointing towards loop ‘A’ being an interstitial type defect is provided by both topological considerations (see below) and the observation of a small SFT (‘T’; arrowed in (k)

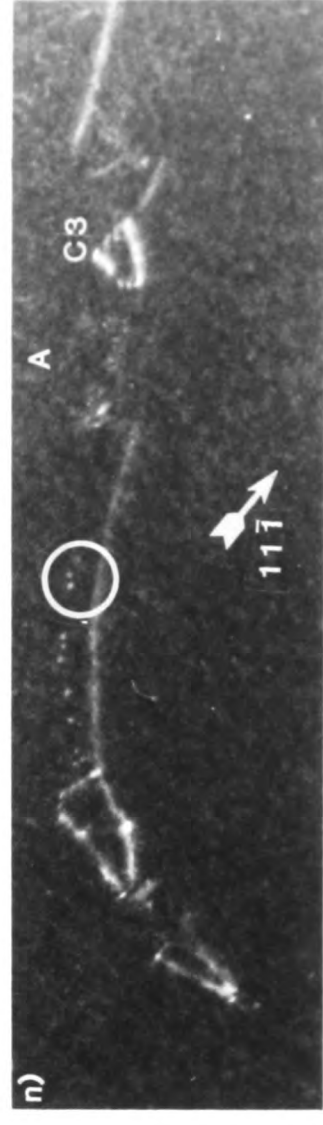
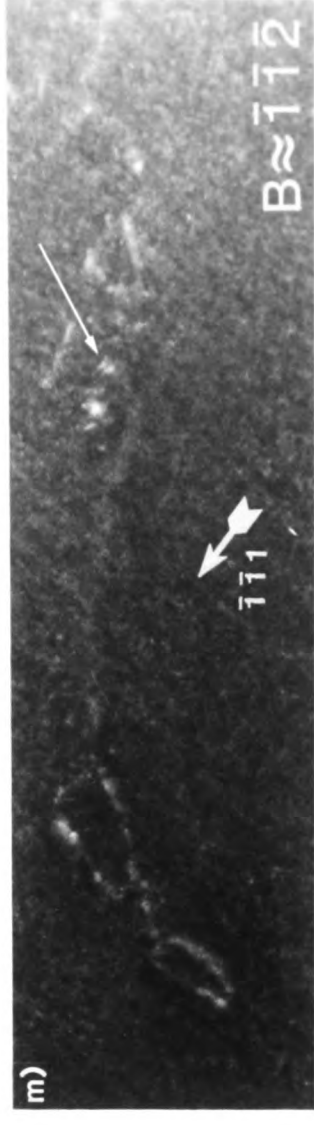
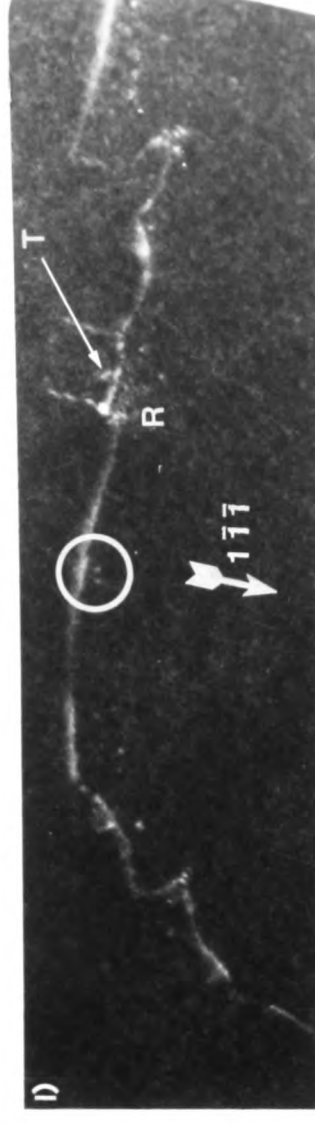
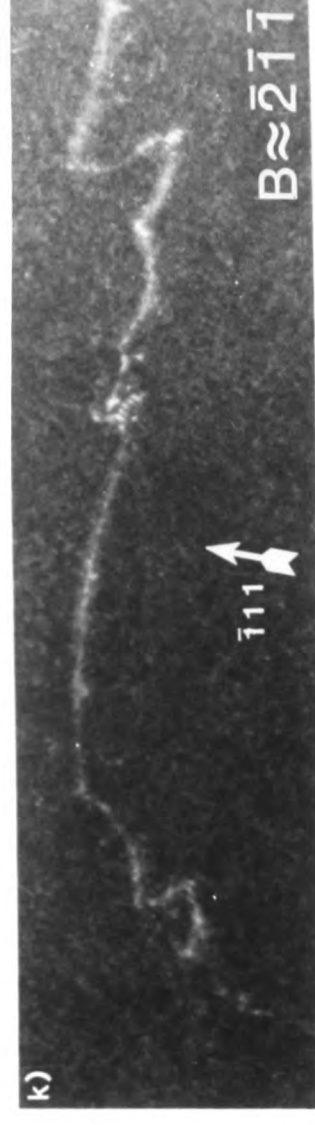
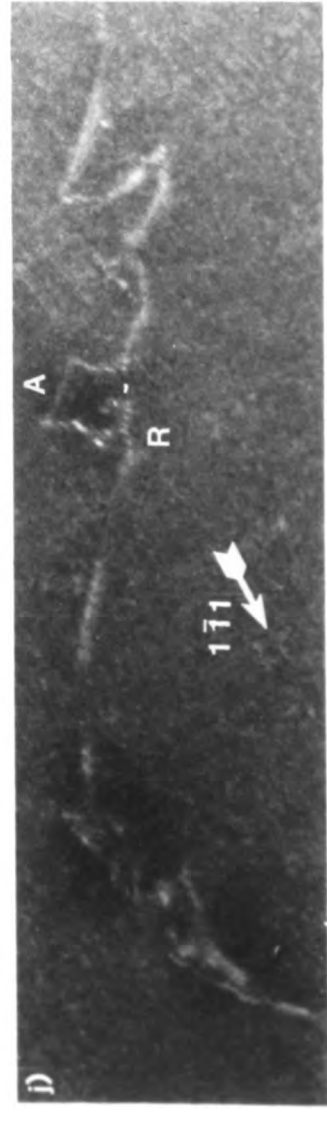
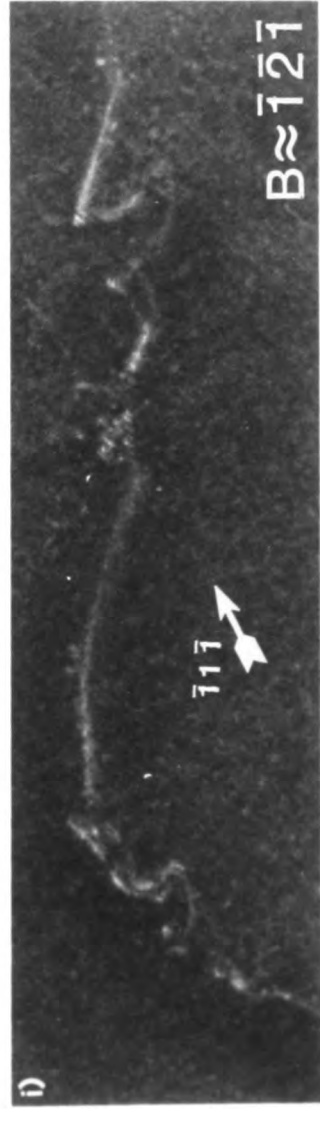
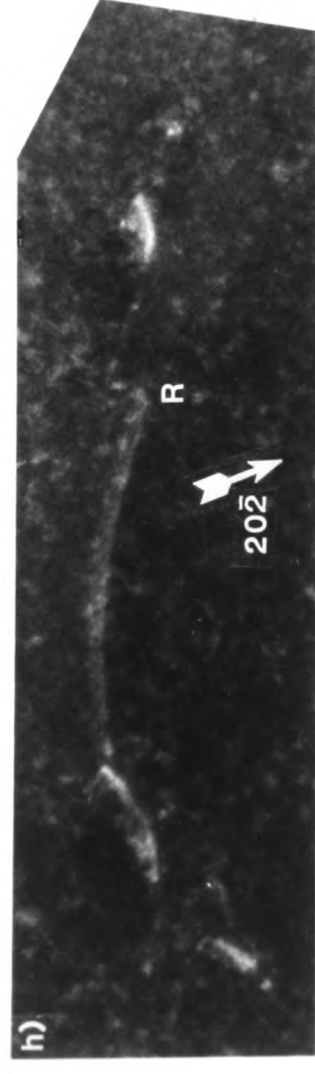
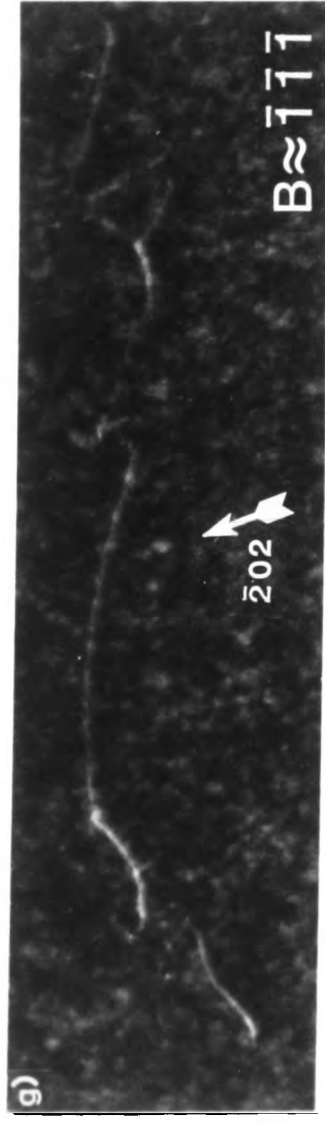
Figure 7.7
(2 pages)

‘Area 4’



0° – 60° Dislocation in Fe–15%Ni–17%Cr–2%Mo (at.%)

0.08 dpa at ~420°C



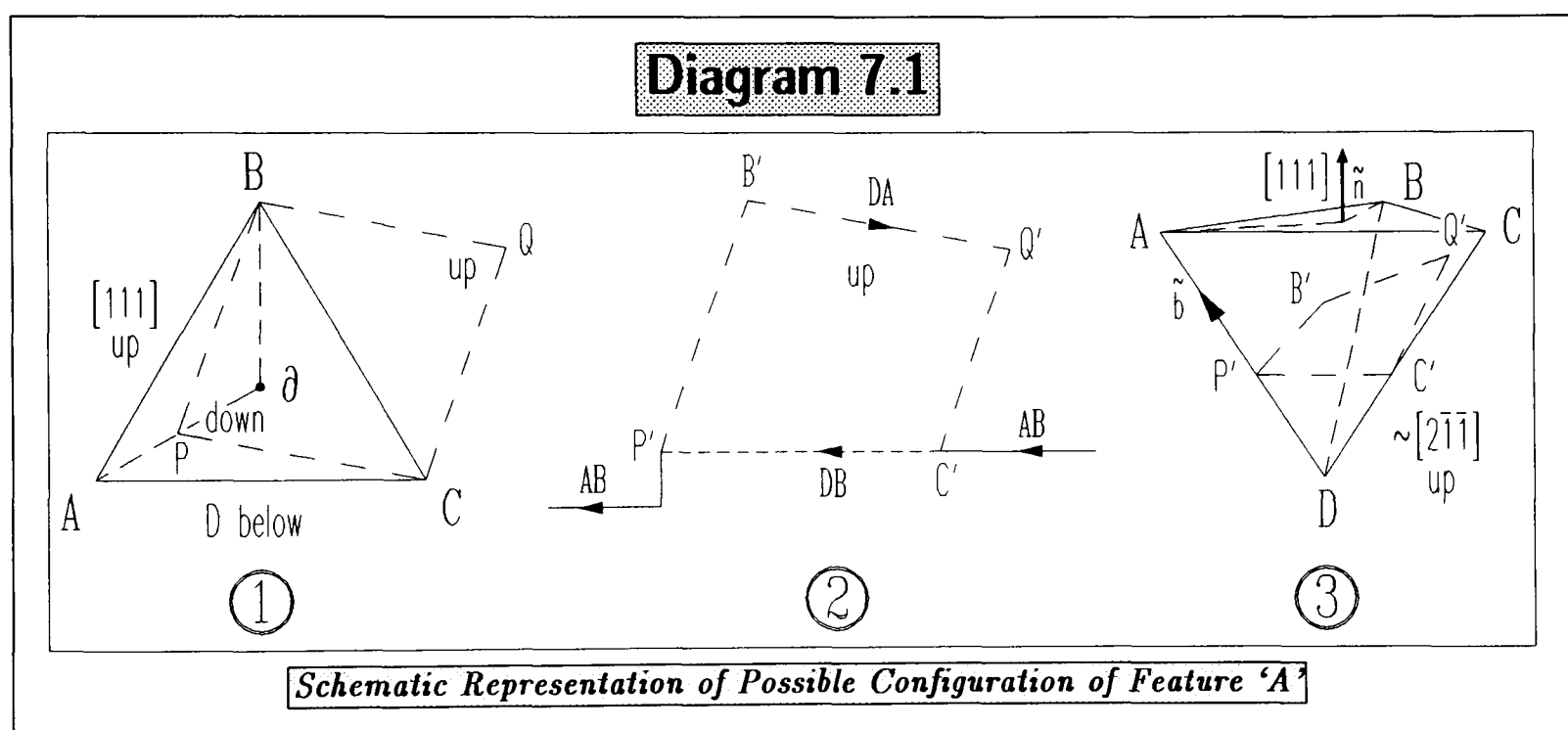
0° - 60° Dislocation in Fe-15%Ni-17%Cr-2%Mo (at.%)

0.08 dpa at ~420°C

and (l)) near its inner periphery.

In contrast with features seen in preceding figures, however, loop 'A' appears to have reacted with the nearby dislocation $\vec{AB}$. The section of dislocation $\vec{AB}$ adjacent to 'A' is either out-of-contrast, or shows only very weak contrast, in $\tilde{g} = \pm [02\bar{2}]$, $[1\bar{1}1]$ and $[11\bar{1}]$. Furthermore this section is very bright in $\tilde{g} = \pm [1\bar{1}\bar{1}]$, whilst the opposite loop edge is out-of-contrast as expected (see figure 7.6). This is consistent with Burgers vectors $\pm \vec{\delta B}$ or $\vec{DB}$. Topological considerations dictate that this section should have Burgers vector $\vec{DB}$. It is therefore in pure edge orientation. A large zig-zag is visible in dislocation $\vec{AB}$ at 'R', just to the left of feature 'A'. On tilting to beam direction $\tilde{B} \approx [\bar{1}2\bar{1}]$ this zig-zag is no longer visible and thus it may lie on a plane α . On tilting to $\tilde{B} \approx [\bar{2}1\bar{1}]$ (see (l)), zig-zag 'R' appears shorter than in micrographs (g) or (h) suggesting that dislocation $\vec{AB}$ to the left of it (as the figure is viewed) lies above the complex section to the right.

The 'local' configuration at loop 'A' is sketched below (Diagram 7.1). Dislocations are shown constricted for simplicity.



With tetrahedron ① orientated as shown, sides of a pure edge loop $\vec{DA}$ lie parallel to directions 'PB' and 'BQ'. 'P' lies below, and 'Q' above the plane of the paper (δ). Loop $[P'-B'-Q'-C']$ therefore extends upwards from the parent dislocation when drawn as in ②. Since the extra half-plane lies below dislocation $\vec{AB}$ such a loop is expected to be of interstitial type. The sessile reaction product, $\vec{DB}$, may constrain the glide of the loop, such that it does not lie entirely in pure-edge orientation. Its actual configuration is probably closer to that super-imposed on tetrahedron ③.

Loops 'C1', 'C2' and 'C3' appear similar to one another. All edges of these features are visible in $\tilde{g} = \pm [02\bar{2}]$, $[20\bar{2}]$ and $[11\bar{1}]$. They apparently show strong contrast in $\tilde{g} = [02\bar{2}]$ and $[11\bar{1}]$ which is consistent with a Burgers vector of $\tilde{b}_T = \vec{CD}$; indicative of a vacancy cluster. There are potential

problems with this characterization, however (see ‘*Interpretation*’ section below, in which an alternative characterization is outlined). On tilting to $\tilde{B} \approx [\bar{1}\bar{1}\bar{2}]$ ((m) and (n)), loops ‘*C1*’ and ‘*C2*’ appear slightly larger than in micrographs taken with beam direction $\tilde{B} \approx [\bar{1}\bar{1}\bar{1}]$ (see (e) and (f)) and thus may be shallowly inclined in the foil, such that they extend slightly *below* the dislocation; again indicative of a vacancy cluster. The change in size of loop ‘*C3*’ is in the same sense and is greater than for loops ‘*C1*’ and ‘*C2*’. This is consistent with what may be depth oscillations exhibited by this loop (‘*C3*’) in micrograph (n) .

Finally, notice that the line of vacancy clusters apparently projects onto the lower jog of zig-zag ‘*R*’ (see (n)).

Interpretation:

Loops ‘C’

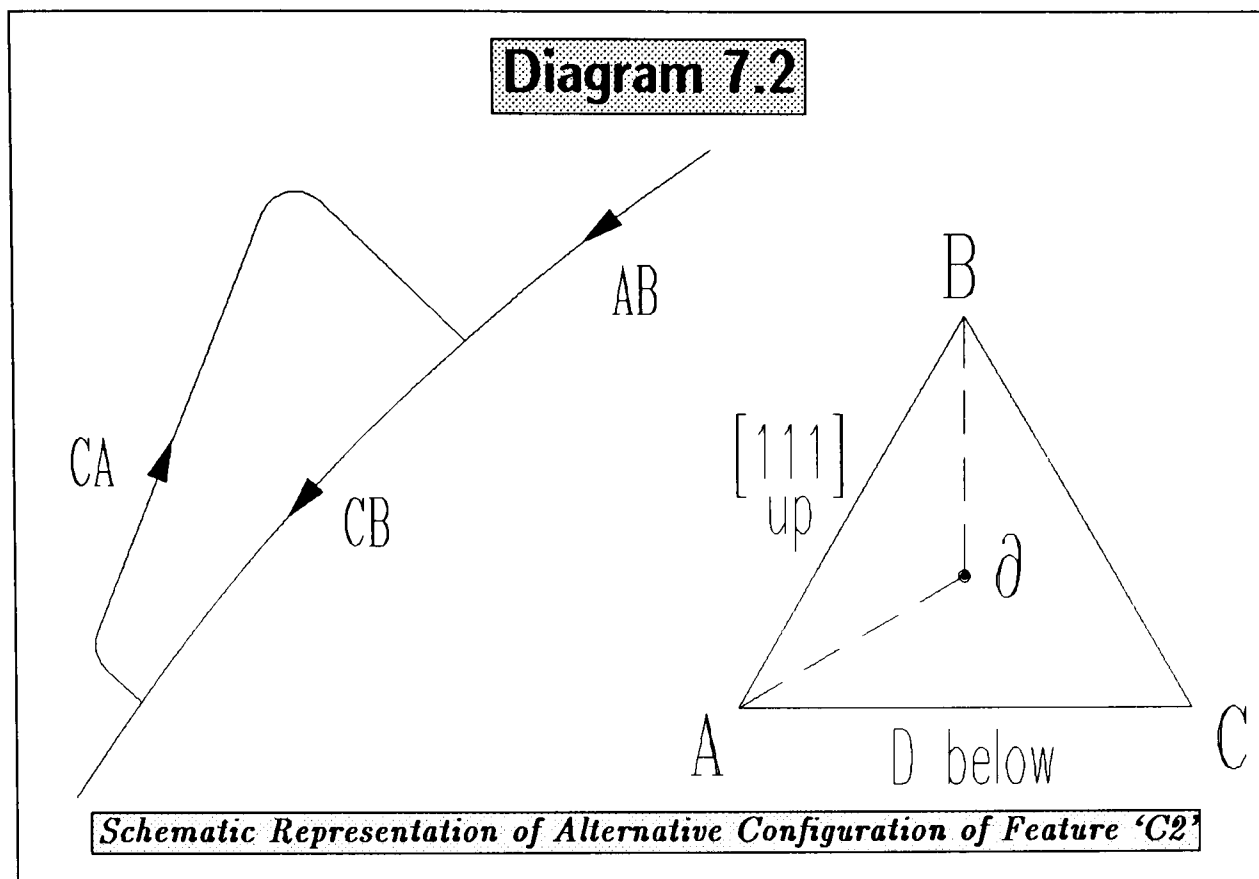
Owing to the complexity of loops ‘*C*’ (see (i) and (j) for example), their detailed configurations have, so far, eluded discovery by the author. As mentioned above contrast exhibited by these features may be explained by assuming that they have Burgers vector $\vec{CD}$ and, since these loops do not appear to lie in the plane of the foil, vacancy type defects are indicated. Whilst further experimentation may indeed prove this to be the case, it should be noted that the formation of loops $\vec{CD}$, such that they extend downwards from the post-irradiation position of dislocation $\vec{AB}$, seemingly requires that they glide *away* from edge orientation from either $\tilde{b}_L = \vec{C}\gamma$ or $\delta\vec{D}$ Frank loops.

With this in mind, an alternative, and speculative explanation of contrast seen from these features is also presented (see diagram 7.2). Loop ‘*C2*’ is considered in this case. This loop is supposed to consist of dislocations with Burgers vectors in the plane of the foil (δ) and to have reacted with the parent dislocation, as is suggested by several of the micrographs (see (g) and (h) for example). An explanation of the contrast shown by the micrographs, with respect to this alternative configuration, is given in the caption.

‘Area 5’: *Figure 7.8; 30°-60° Dislocation in Fe-15%Ni-17%Cr-2%Mo; 0.1 dpa at ~ 420°C.*

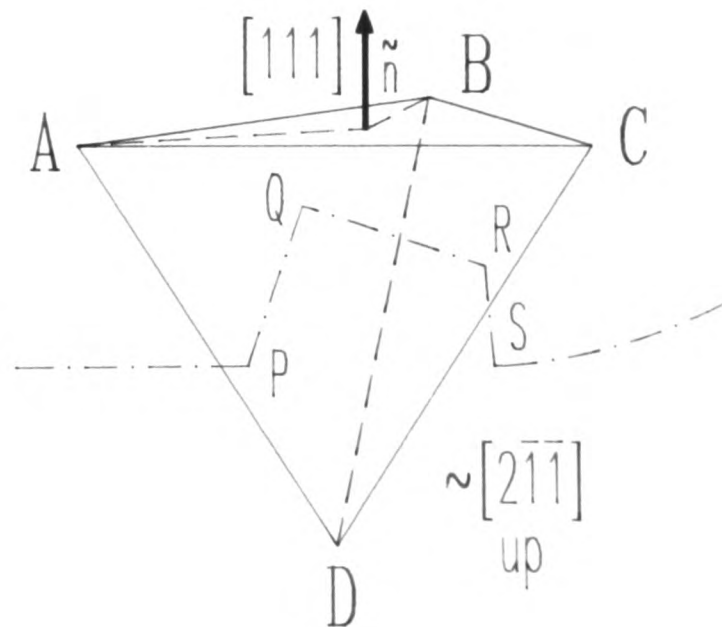
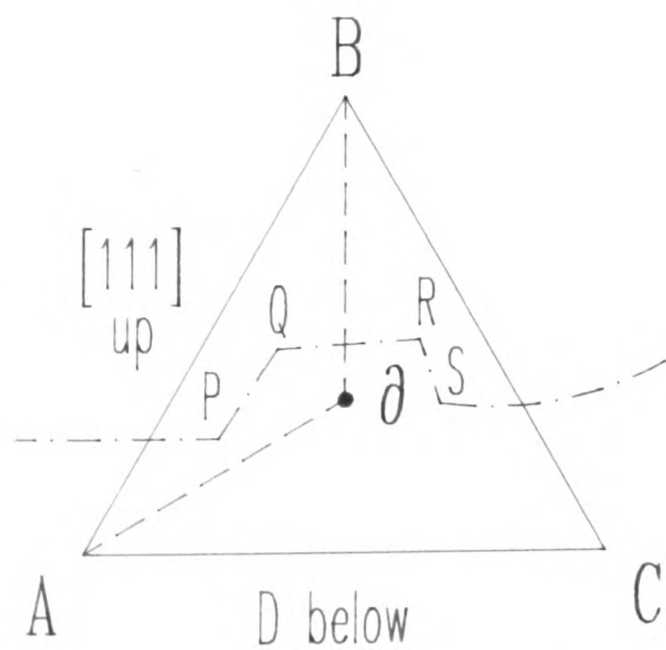
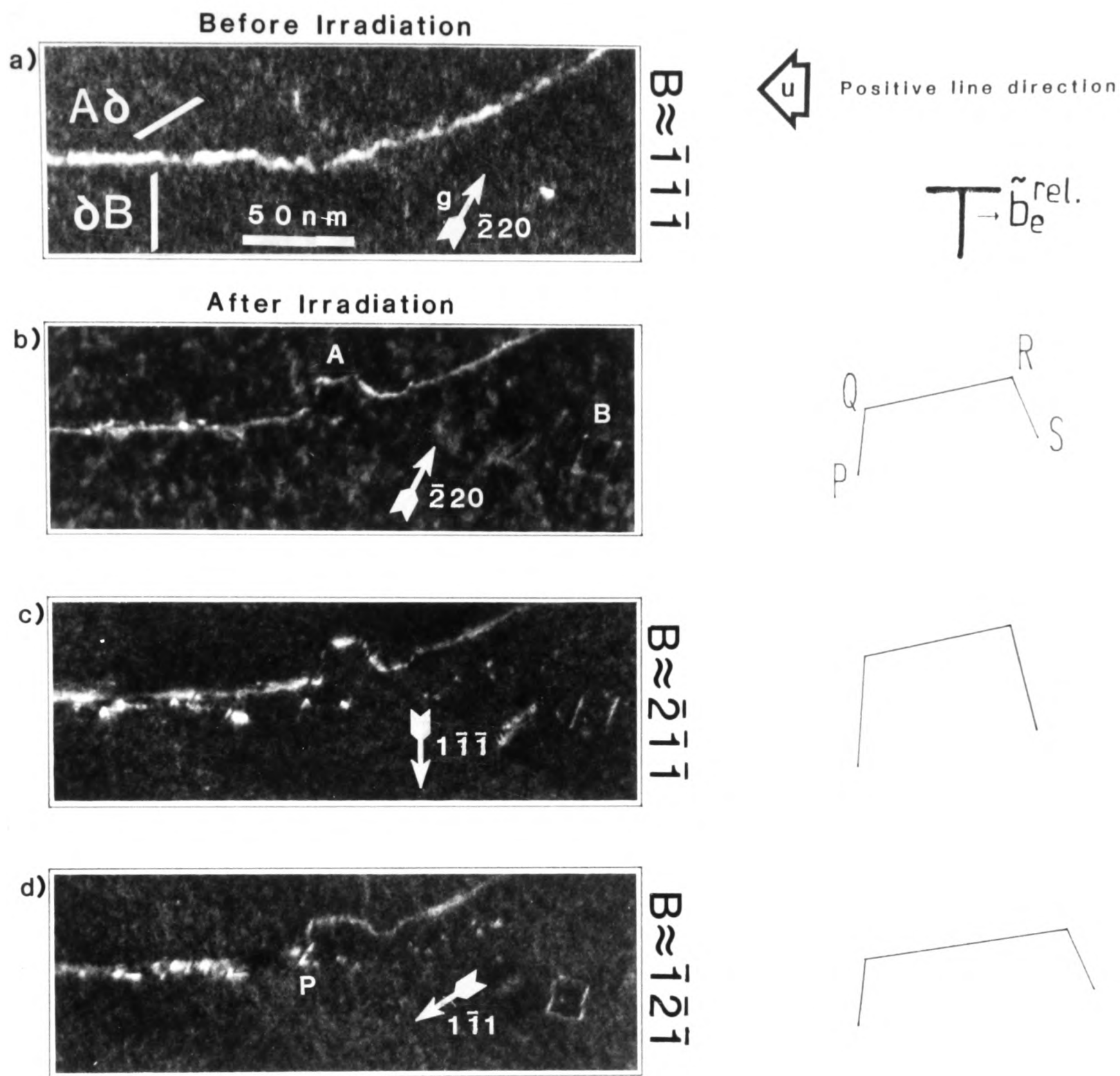
Observations:

In the final area enlarged from figure 7.3, climb of a short (~ 25 nm.) section (‘*A*’) of the line is seen. Micrographs (a) and (b) show $|\tilde{g} \cdot \tilde{b}_T| = 2$ images before and after irradiation respectively, in which an initially very heavily cusped dislocation has now become fairly evenly dissociated. The irradiation has also prompted the formation of cluster damage within the compressional strain field of the line (see (b) , (c) and (d)). To the left of ‘*A*’, several of these clusters are resolvable as SFT. Notice also that to the left of ‘*A*’ the cluster damage is fairly dense, ceasing with a SFT near ‘*P*’. To the right



The upper section (as figure 7.7 is viewed) of each loop is visible in $\tilde{g} = \pm [1\bar{1}1]$ and $[11\bar{1}]$, very bright in $\tilde{g} = [02\bar{2}]$ and out-of-contrast in $\tilde{g} = \pm [1\bar{1}1]$. This suggests a Burgers vector of $\tilde{b}_T = \pm \vec{AC}$. The lower section of the loop, where it meets the parent dislocation, is visible in $\tilde{g} = \pm [11\bar{1}]$ and $[\bar{1}11]$, bright in $\tilde{g} = \pm [202]$ and out-of-contrast, or very weak in $\tilde{g} = \pm [1\bar{1}1]$. This is consistent with a Burgers vector $\tilde{b}_T = \pm \vec{BC}$. The senses of these Burgers vectors is determined by considering the topology of the configuration. The apparent strong/weak contrast behaviour shown by these features is consistent with the sense of the $\left(\frac{3}{4}/\frac{1}{4}\right)$ loop $\left(\vec{CA}/\vec{CB}\right)$ shown. If these loops lie in the plane of the foil, point defects are not accommodated and dislocation $\vec{AB}$ may form by the glide and reaction of $\vec{AC}$ and $\vec{CB}$. For dislocations $\vec{AC}$ and $\vec{CB}$ lying on different planes δ , their dissociation may form a $\vec{C}\delta/\delta\vec{C}$ dipole (Cherns et al, 1980), the presence of which may be suggested by micrographs (e) and (f). This configuration does not easily explain the low visibility of dislocations $\vec{AC}$ and $\vec{CB}$ in $\langle 2\bar{2}0 \rangle$ -type reflections for which $|\tilde{g} \cdot \tilde{b}_T| \neq 2$ and the real configurations of features 'C' are likely to be considerably more complex than that depicted here.

Figure 7.8
‘Area 5’



30°–60° Dislocation in Fe–15%Ni–17%Cr–2%Mo (at.%)

0.1 dpa at ~420°C

of 'P', i.e the region of the line below feature 'A' and the 30° section, the line of cluster damage becomes sparsely populated. This 30° section of the dislocation apparently lies some distance from the clusters: Notice that the entire line of cluster damage is displaced from the line by the same amount, in the same sense on tilting from (211) to (121) poles; these clusters therefore all lie, at least approximately, the same distance away from the dislocation, suggesting that glide, and not climb, is the origin of this displacement.

Shown to the right of micrographs (b), (c) and (d) are schematic representations of the projected images of feature 'A'. An analysis of the senses of inclination, and approximate line directions, of segments 'PQ', 'QR' and 'RS', which may be confirmed by inspection of the micrographs shown, yields the configuration of feature 'A' as drawn super-imposed upon the tetrahedra below the micrographs: 'Q' is above 'R', which, in turn, is above 'P' and 'S'. The change on tilting in the projected length of segment 'PQ' suggests that points 'P' to 'Q' are vertically separated by $\sim 18\text{nm}$. Points 'P' and 'S' may lie on the same plane δ . None of segments 'PQ', 'QR' or 'RS' lies in a plane δ and SFC is not evident from any part of feature 'A' in images which have $\tilde{\mathbf{g}} \cdot \tilde{\mathbf{b}}_T = 0$ (not shown; feature 'A' appears completely out-of-contrast) for dislocation $\tilde{\mathbf{b}}_T = \vec{\mathbf{AB}}$.

Feature 'B' appears similar to 'B' of figure 7.6 in all reflections of the full contrast series. This is consistent with a perfect prismatic, interstitial, loop of Burgers vector $\tilde{\mathbf{b}}_L = \vec{\mathbf{DA}}$. The change in its projected shape on tilting suggests that it lies on, or very close to, a (101) plane. The entire loop is visible in $\tilde{\mathbf{g}} = [1\bar{1}1]$ ((d)) whilst the sides perpendicular to $\tilde{\mathbf{g}}$ are out of contrast in $\tilde{\mathbf{g}} = [1\bar{1}\bar{1}]$ ((c)), suggesting that its individual sides are dissociated on planes β and γ .

Finally it should be noted that, in common with 'C1' and 'C2' of figure 7.7, feature 'A' has formed at a point in the dislocation line where it tends away from 60° towards the screw orientation.

Interpretation:

No line dislocation, apart from that shown by these micrographs, is imaged in any reflection of the full contrast set. All possible perfect and Frank dislocations would be visible in more than one reflection of the series and, thus, the entire length of the dislocation shown is likely to have the same Burgers vector ($= \vec{\mathbf{AB}}$).

The configuration represented within the two tetrahedra, which depict top and side views, is sessile. This feature must therefore have accommodated point-defects since its formation cannot solely be a result of glide. It can also be seen that this feature extends into the dilatational strain field of the rest of dislocation $\vec{\mathbf{AB}}$, suggesting that interstitials are the defects accommodated. Hence, a short

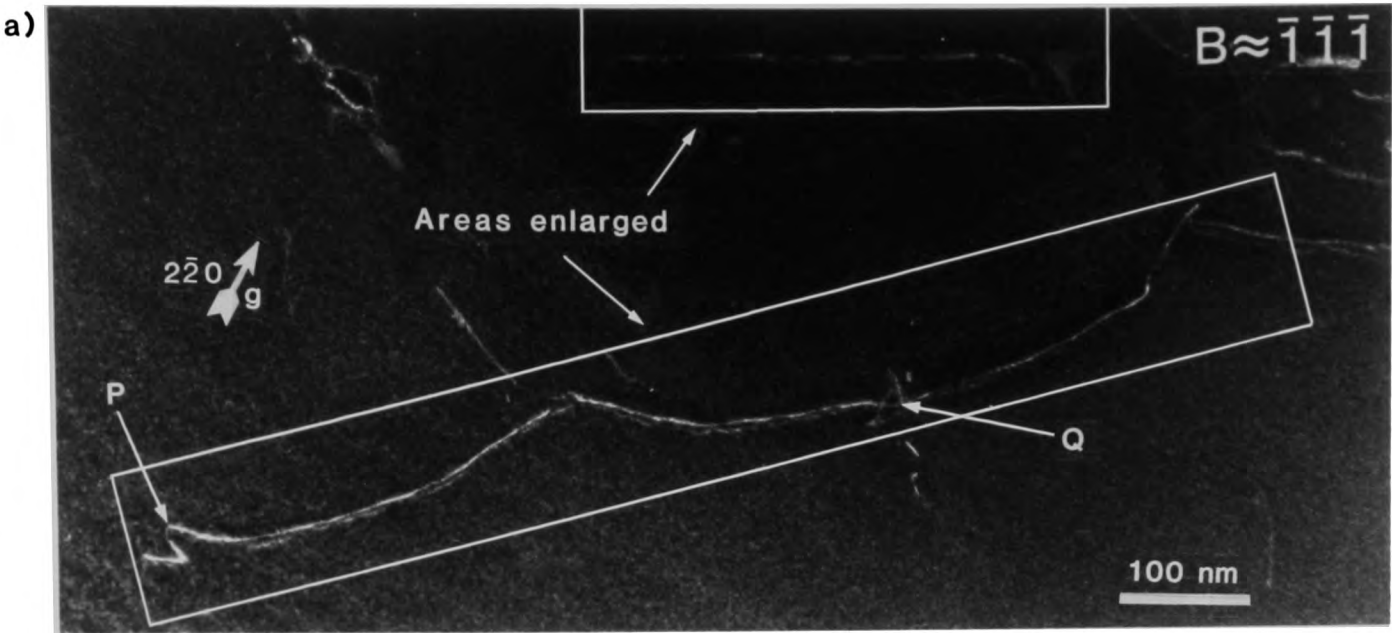
section of dislocation $\overrightarrow{AB}$ appears to have climbed. This climb has resulted in, or resulted from, double-jog nucleation and is likely to indicate the absorption of interstitials.

The observation in figure 7.8 of climb consistent with interstitial absorption at the point where dislocation $\overrightarrow{AB}$ curves away from 60° orientation, may suggest that features 'C1' and 'C2' of figure 7.7, which have formed at a similar point, have a similar origin. The extension of loops 'C1' and 'C2' *below* the dislocation, an observation apparently incompatible with their being interstitial in nature, may, perhaps, be explained if adjacent line sections have moved, by both climb and glide, possibly via the formation of intermediate configurations similar to 'A'. The mechanism of formation is uncertain here, however, since features in both figures, if formed from Frank loops, apparently require rotation *away* from edge orientation. This may suggest that Frank loops are not, in fact, the origin of these observations. The line of clusters in figure 7.7, which apparently lies a considerable distance (~ 40 nm.) below the dislocation, but which clusters are commonly seen to lie much closer (~ 15 - 20 nm.) to the dislocation position (e.g. figure 7.2), may indicate that the evenly dissociated (after irradiation) central section of this dislocation has undergone such climb. The complexity of the dislocation prior to irradiation may have aided this process in some way, perhaps via the climb of many, large, pre-existing jogs. It is particularly noticeable, however, that the vertical height of feature 'A' (figure 7.8), at ~ 18 nm., would account for the extra displacement of clusters from the dislocation of figure 7.7 rather neatly. The complexity of the dislocation of figure 7.7 makes direct correlation of the cluster and the pre-irradiation dislocation positions very difficult, however. Hence, the assumption that these clusters are substantially vertically removed from the post-irradiation dislocation position because the dislocation has climbed must therefore be treated with considerable caution.

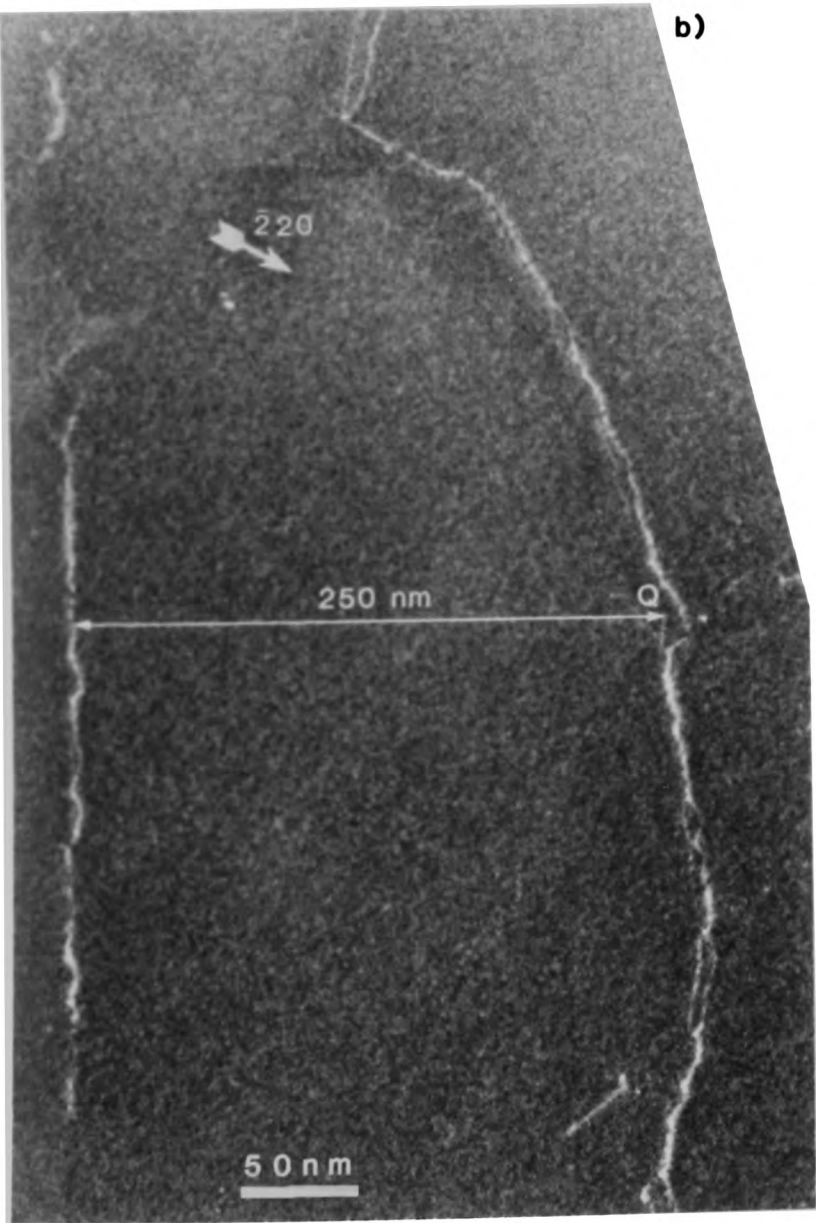
Figure 7.9; Dislocations in Fe-15%Ni-17%Cr-2%Mo; Before Irradiation and 0.14 dpa at $\sim 420^\circ\text{C}$.

A region of foil is shown before irradiation in the low magnification micrograph (a). The two highlighted areas and shown in detail in figures 7.10 and 7.11. After irradiation, segments of the long dislocation in the lower of these areas have glided and straightened (see figure 7.10), leading to a significantly altered form. However, careful comparison of both low and high magnification prints, taken before and after irradiation, with the same diffraction vector and foil orientation, show at least two points ('P' and 'Q') which have apparently remained stationary under irradiation. Point 'Q' is particularly important in that its existence enables the measurements shown in micrographs (b) and (c) of this figure to be made. These measurements are examined in the text relating to figure 7.11.

Dislocations in Fe-15%Ni-17%Cr-2%Mo (at.%)



Before Irradiation



0.14 dpa at $\sim 420^\circ\text{C}$

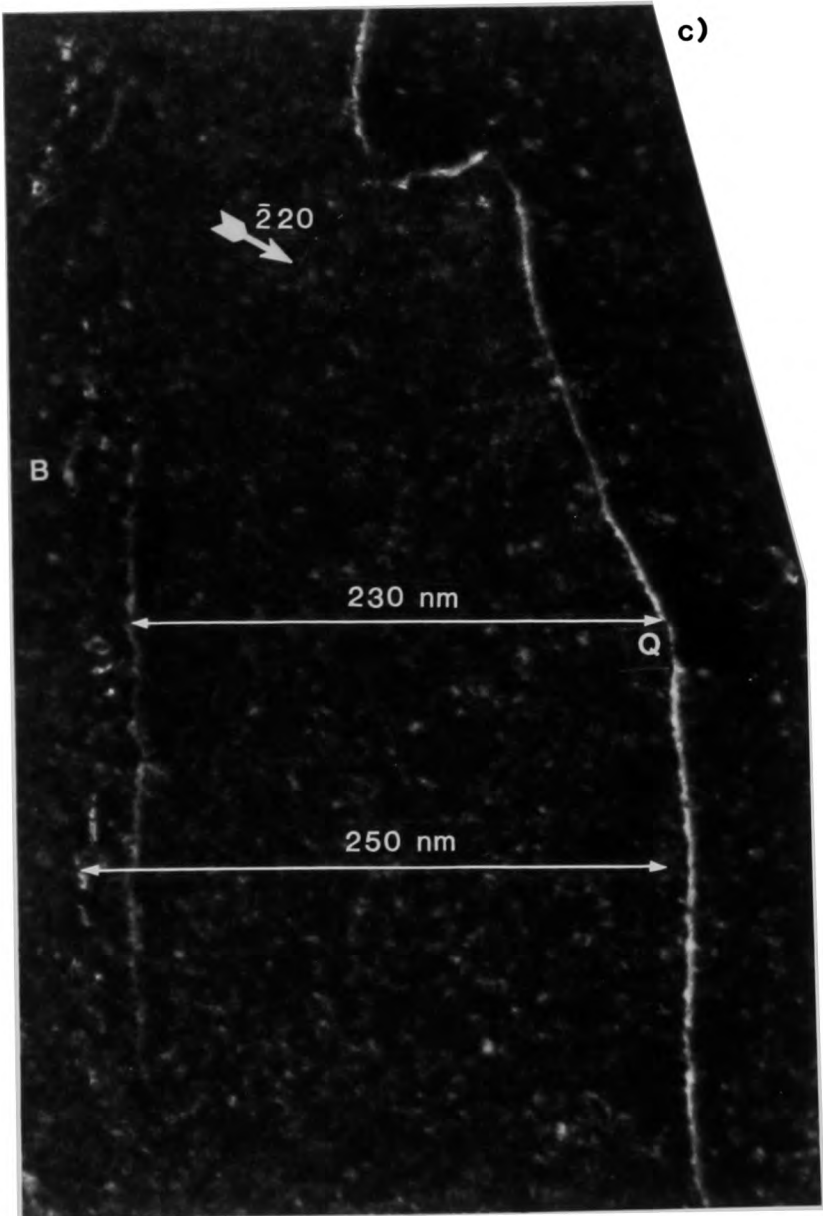


Figure 7.10; 0° - 60° Dislocation in Fe-15%Ni-17%Cr-2%Mo; 0.14 dpa at $\sim 420^\circ\text{C}$.

Observations:

Micrograph (a) is taken prior to irradiation. The bulk of the dislocation lies within the 40° - 60° orientation range, but also approaches screw and near edge orientations at various points along its length. Before irradiation this dislocation contains very many constrictions, as well as points, where one partial is apparently pinned, at which the partial separation is very large (e.g. 'A' (near 'Q') and 'B'). The line also seems to have been pinned at other points, and to have bowed-out between them.

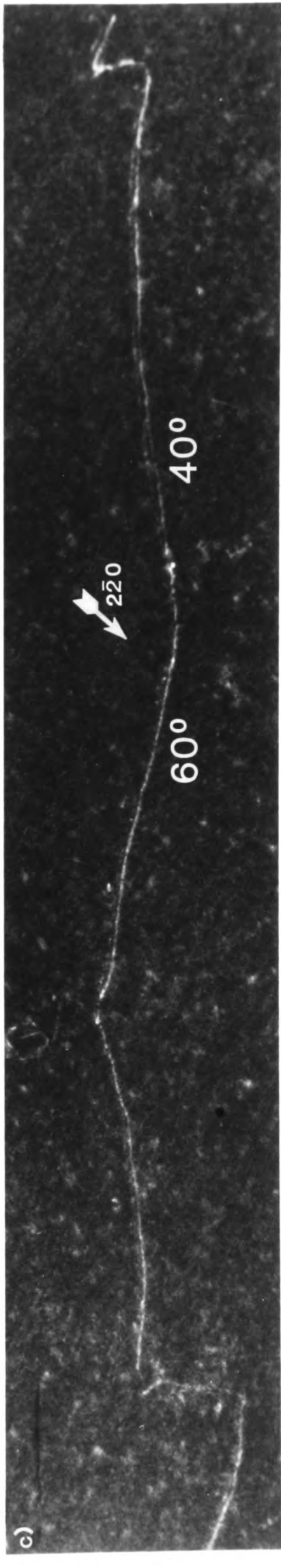
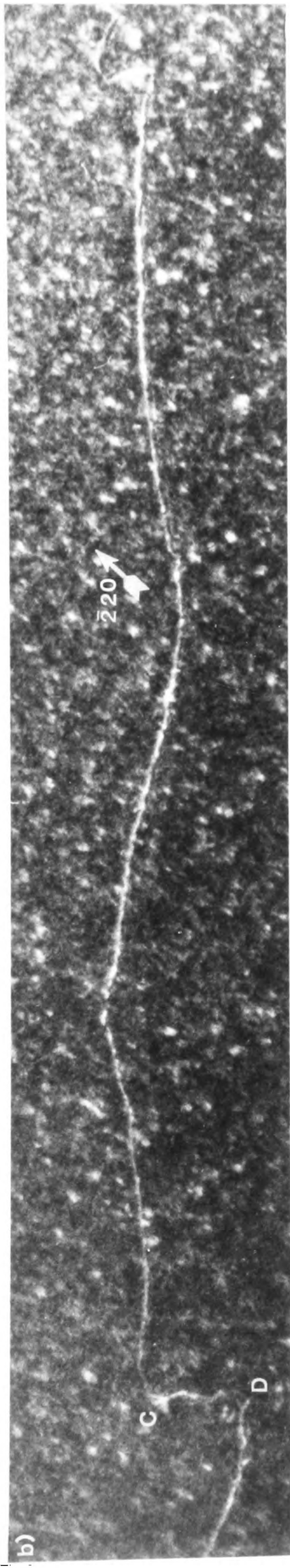
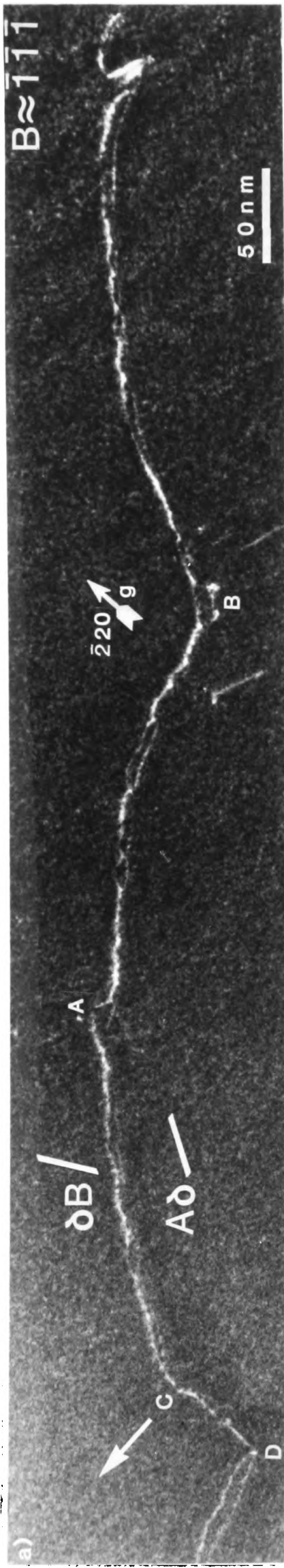
After irradiation (micrographs (b) to (f)), the upper, near-edge, partial ($\vec{\delta\vec{B}}$) has a dramatically smoother appearance ((b)), constrictions having disappeared. The dislocation has also glided and straightened after irradiation, particularly around the previously bowed out section near 'B'. Long sections of the line now lies near 60° ($[01\bar{1}]$) and 40° (very near/along $[\bar{1}3\bar{2}]$) orientations. The upper and lower partials are the brighter in the $|\vec{g}\cdot\vec{b}_T|=2$ images (b) and (c) respectively. In both of these micrographs, although better imaged in (c), the dislocation can be seen to be relatively evenly dissociated. Confusingly, the partial image separation appears to be greater along the 40° section ($\simeq 40\text{ \AA}$) than along the 60° section ($\simeq 33\text{ \AA}$). Along the entire length of the dislocation, except near pre-existing constrictions, the partial separation has apparently narrowed after irradiation, however.

The section of dislocation between 'C' and 'D', which lay initially close to screw orientation, has rotated towards edge orientation. A detailed comparison of pre- and post-irradiation micrographs show that this has apparently occurred via the motion of points 'C' and 'D' in directions roughly perpendicular to $[1\bar{1}0]$, as indicated by the large arrow in micrograph (a). This determination is complicated, however, by the possibility that some very short segments of the line, particularly near 'D', do not lie in the plane of the foil (see micrograph (e), for example).

In the second page of this figure (micrographs (d) to (f)) several features are highlighted. Features 'E', 'F', 'G' and 'L' all form part of a line of cluster damage lying close to the pre-irradiation dislocation position. These clusters become smaller as the thickness of the foil decreases (left to right). Features 'E' and 'G' often show arrow-head contrast (particularly in (e)) typical of SFT. Features 'F' and 'L' could not be characterized employing standard techniques, but the implication that they are vacancy-type defects is reinforced by the observation that all features 'E', 'F', 'G' and 'L' are displaced from the dislocation line image by the same amount on tilting to the (211) pole ((f)). The sense of this motion is consistent with their lying towards the compressional side of the dislocation strain field (above). SFT 'G' apparently lies very close to the pre-irradiation position of cusp 'D'.

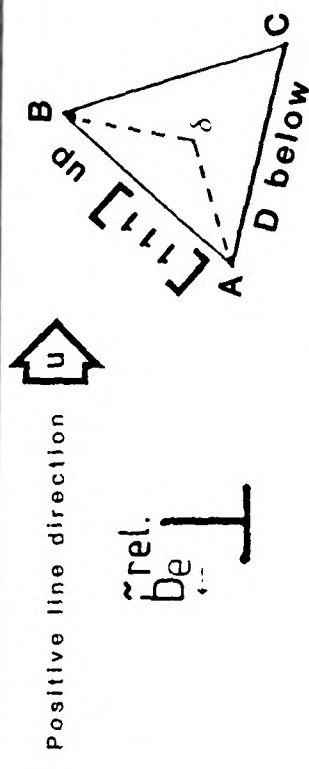
Interstitial clusters, such as 'H', are also seen in this irradiation. This feature shows 'inside'

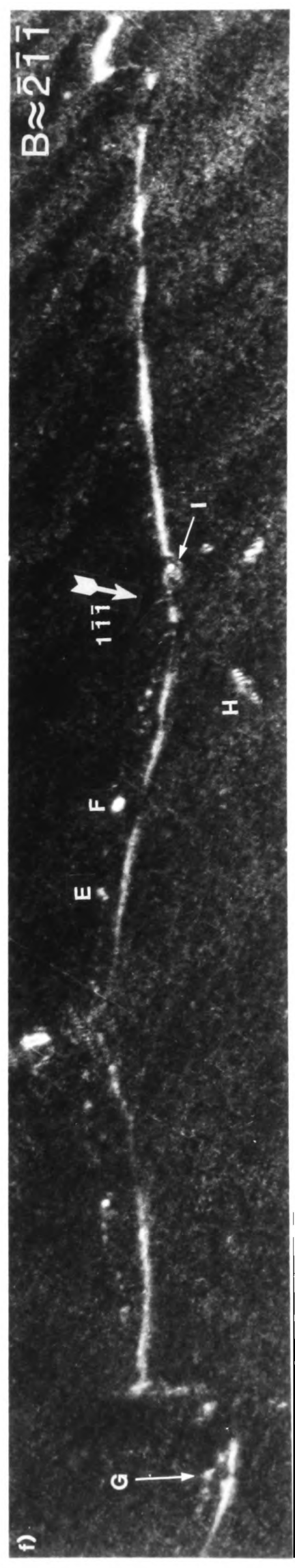
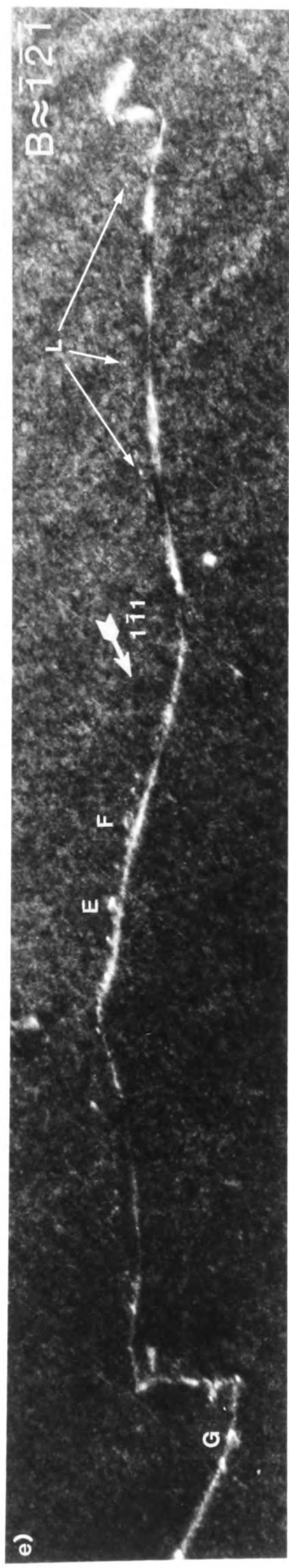
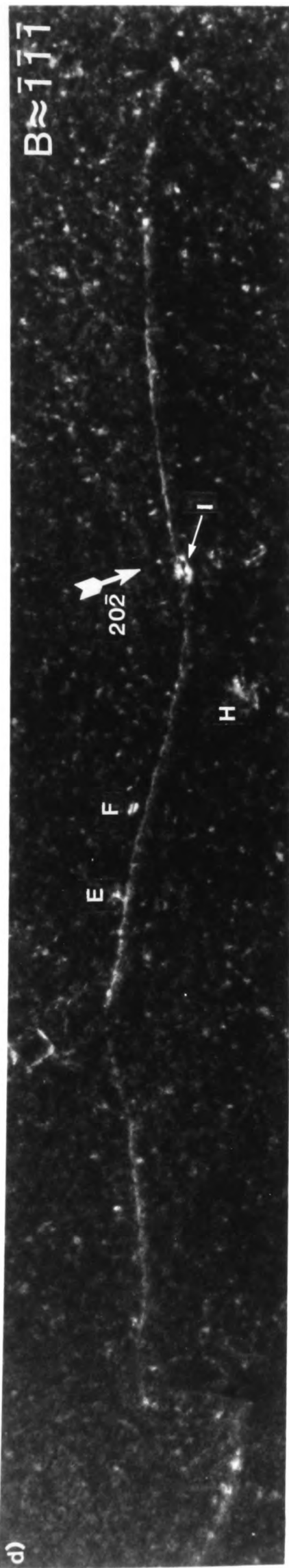
Figure 7.10
(2 pages)



0° – 60° Dislocation in Fe-15%Ni-17%Cr-2%Mo (at.%)

0.14 dpa at ~420°C





0° - 60° Dislocation in Fe-15%Ni-17%Cr-2%Mo (at.%)

0.14 dpa at ~420°C

contrast in $\tilde{g}=[0\bar{2}2]$ (not shown) and ‘outside’ contrast in $\tilde{g}=[20\bar{2}]$ ((d)). It is out-of-contrast in $\tilde{g}=\pm[2\bar{2}0]$ ((b), (c)) but is visible in reflections taken with beam direction $\tilde{B}\approx[\bar{1}\bar{1}\bar{2}]$ (not shown). Notice, however, that this feature is roughly aligned with the trace of plane γ . This is consistent with a perfect loop, of Burgers vector $\tilde{b}_L=\vec{D}\vec{C}$, which has formed via the unfaulting of a $\tilde{b}_L=\vec{\gamma}\vec{C}$ interstitial Frank loop and which is gliding towards, but has not yet reached, pure-edge orientation. The bright fringes visible in $\tilde{g}=[1\bar{1}\bar{1}]$ ((f)) may arise from the dissociation of edges of this loop onto a plane α , as detailed in figure 7.6.

Feature ‘I’ is interesting in that it seems to lie very close to the dislocation itself, at one end of the 60° dislocation segment. On tilting from the (111) to the (211) pole (micrographs (d) and (f); partial $\delta\vec{B}$ is imaged in both) the image of this loop is extended in a sense consistent with its lying to the dilatational side of the dislocation strain field, although it is still apparently in contact with the dislocation. This loop is visible in $\tilde{g}=\pm[20\bar{2}]$ and $[0\bar{2}2]$ (not shown), and out-of-contrast, or very faint, in $\tilde{g}=\pm[2\bar{2}0]$. Its contrast is generally very similar to that exhibited by ‘H’ (see, for example, micrograph (f)). However, feature ‘I’ is not apparently visible in reflections taken in the (112) pole, suggesting that it may be confined to a single plane γ . It shows ambiguous inside/outside contrast in the full set of reflections taken, unfortunately prohibiting the reliable use of this technique. This may be a result of its proximity to the dislocation.

Interpretation:

Even after the relatively high dose of 0.14 dpa this dislocation may still be seen to be dissociated. However, after the low dose irradiations of figures 7.1 and 7.2 the partial separations have already decreased. A very large scatter in the measurements (see chapter 8) of pre- and post-irradiation separations prevents a detailed, quantitative, analysis. However, the above observations may suggest that the magnitude of this effect is relatively dose insensitive.

The behaviour of points ‘C’ and ‘D’, along a screw section of the dislocation of figure 7.10, is apparently consistent with the jog climb model originally outlined in figure 3.7. Subsequent climb or glide of short sections of the dislocation out of the plane of the foil may indicate the onset of helix, or ‘flat-helix’, formation. For non-screw sections, large scale climb of the dislocation, similar to that shown in figure 6.9, is not indicated here, although some reflections, taken at a large angle of tilt away from $\tilde{B}\approx[\bar{1}\bar{1}\bar{1}]$, may suggest some motion in a sense consistent with interstitial climb of a very short section of the line at ‘I’. Cluster ‘I’, which lies where the dislocation tends away from 60° , is referenced again within the ‘Points of Discussion’ section towards the end of this chapter. This cluster may be

similar to features seen at similar locations in other figures.

Figure 7.11; Sessile Dislocation in Fe-15%Ni-17%Cr-2%Mo; 0.14 dpa at $\sim 420^\circ\text{C}$.

The second highlighted area shown in figure 7.9 (a) is examined in this figure. This area contains a sessile dislocation which has apparently moved away from a dense cloud of clusters formed very close to its pre-irradiation position. Micrographs (a) and (b) are taken before, and all others after, irradiation. As with all dislocations shown in this thesis, with the exception of those of chapter 5, the specimen containing it was sectioned as close to the [111] orientation as possible (normally within 2° ; always within 5°).

Observations:

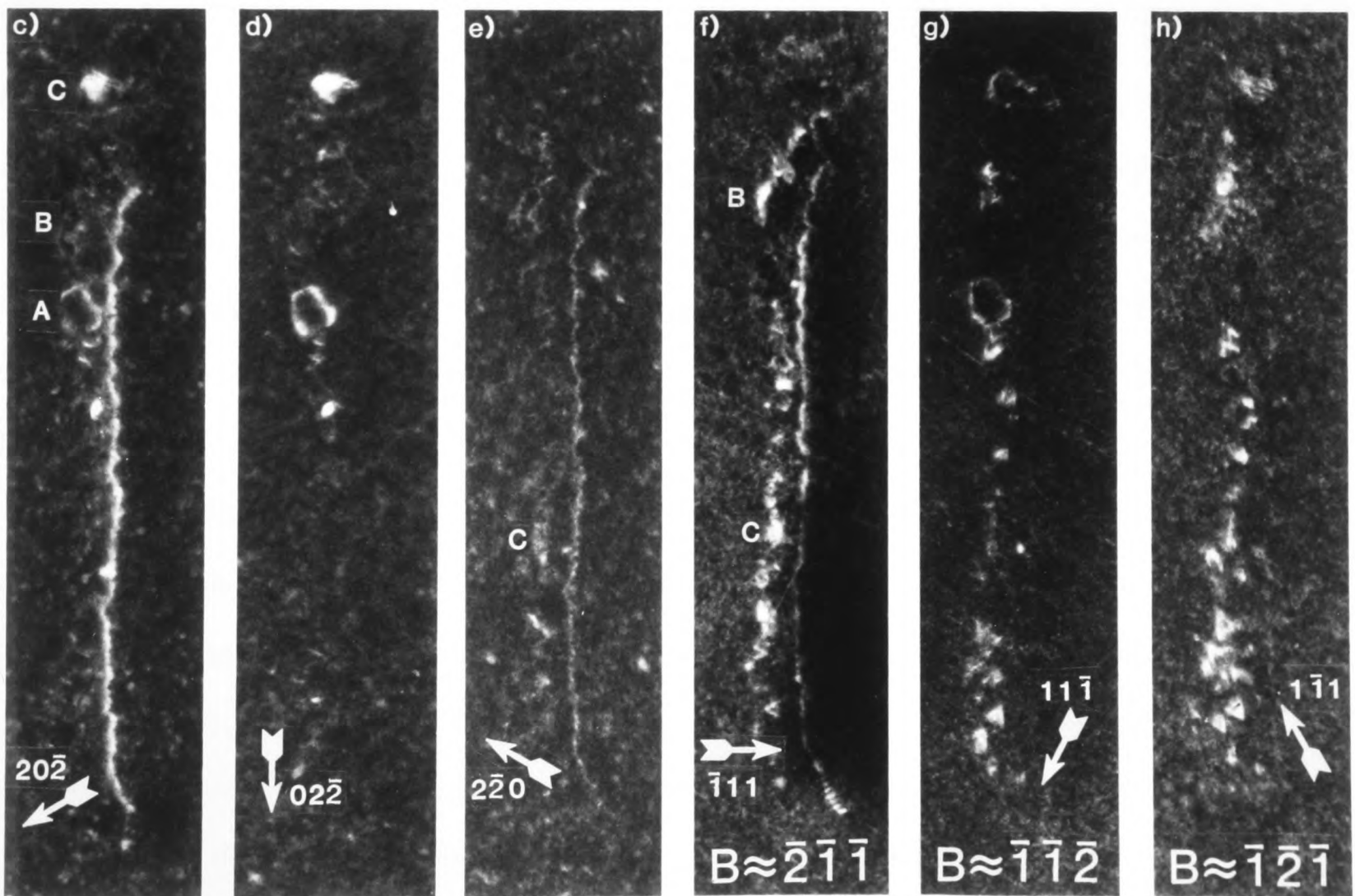
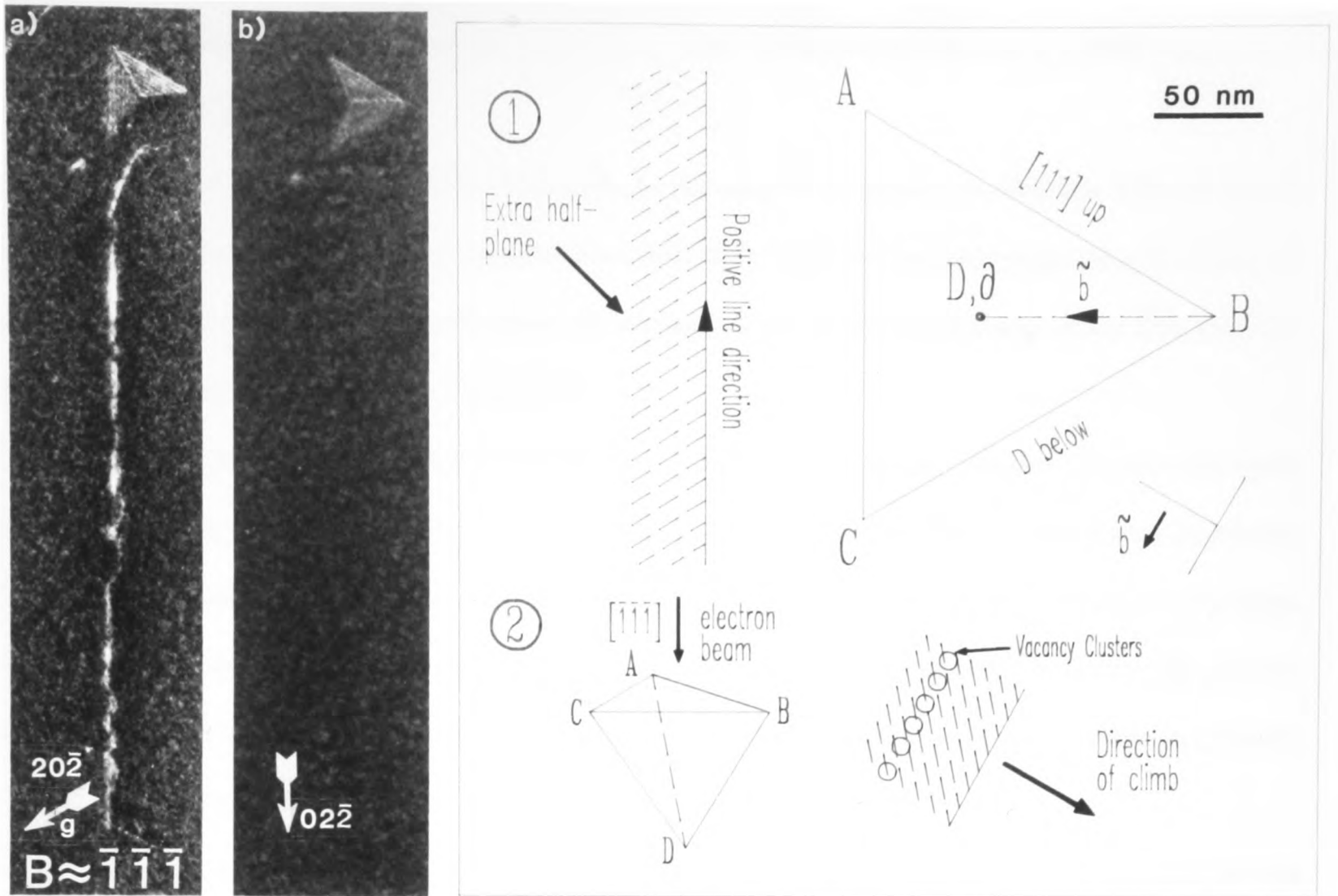
This dislocation is visible in $\tilde{g} = \pm [20\bar{2}]$ ((a)) and $[\bar{2}20]$ (figure 7.9 (b)) but is out-of-contrast in $\tilde{g} = \pm [02\bar{2}]$ ((b)). This is consistent with a Burgers vector $\tilde{b}_T = \pm \vec{BD}$. With a projected length of $\sim 250\text{ nm}$, it may not be inclined at an angle of greater than 21° to the plane of this 100 nm. thick foil and thus is not glissile on either of planes α or γ ³¹. It lies accurately along the projection of $[0\bar{1}1]$ and may therefore be in pure edge orientation. This dislocation may possibly have been formed by a Lomer reaction, although surviving segments of the reactants are not seen. This prevents the assignation of a unique Burgers vector.

After irradiation the line now shows fine scale cusping; very similar behaviour to that shown by the Lomer dislocations of figures 3.6 and 4.5. The period of these cusps, which varies from ~ 3 to $\sim 10\text{ nm}$., is also similar.

A high density of cluster damage has formed close to the dislocation. Many of these clusters can easily be identified as SFT (see (h), for example) suggesting that this damage is vacancy in nature. The cluster density here is very much greater than along the, simultaneously irradiated, glissile dislocation shown in figure 7.10. Notice also that these clusters appear to have formed more as a cloud than those seen to form ‘discrete’ lines after irradiations of glissile dislocations (e.g. figures 4.2, 6.7, 7.8). Further evidence suggesting that this line of cluster damage is vacancy in nature is provided by the large loop at ‘A’: This loop is visible in $\tilde{g} = \pm [20\bar{2}]$, $[02\bar{2}]$ and $[11\bar{1}]$, but is out-of-contrast in $\tilde{g} = \pm [2\bar{2}0]$, $[\bar{1}11]$ and $[1\bar{1}1]$, suggesting a Burgers vector of $\pm \vec{CD}$. This loop also displays strong/weak contrast in $\pm \tilde{g}$ reflection pairs allowing its characterization as a perfect prismatic vacancy loop with Burgers vector $\tilde{b}_L = \vec{CD}$. It only shows strong ‘inside’ contrast in the micrographs shown in this figure.

³¹For this dislocation to be glissile before irradiation on either of planes α or γ , its line direction must be $[2\bar{3}1]$ (α) or $[213]$ (γ). These directions make an angle of $\sim 68^\circ$ with the (111) plane.

Figure 7.11



Sessile Dislocation in Fe-15%Ni-17%Cr-2%Mo (at.%)

0.14 dpa at $\sim 420^\circ\text{C}$

It is noticeable that loop 'A' lies at the bottom end of a gap in the line of cluster damage. Above this gap features at 'B' are also apparently vacancy in nature, showing 'outside' contrast in $\tilde{g}=[20\bar{2}]$ and $[2\bar{2}0]$ and 'inside' contrast in $\tilde{g}=[\bar{1}11]$. They are out-of-contrast in $\tilde{g}\pm[02\bar{2}]$ suggesting a Burgers vector of $\vec{BD}$.

Since this line of cluster damage appears to be vacancy in nature, in common with all similar observations in other irradiations, it would seem likely that it lies to the compressional side of the dislocation strain field. This supposition allows the assignation of the unique Burgers vector $\vec{BD}$, with line direction as indicated in ①, to the dislocation.

As is shown by micrographs (b) and (c) of figure 7.9, careful reference to pre- and post-irradiation prints at both low and high magnifications, containing feature(s) with known position(s) (such as 'Q'), yields the observation that this dislocation has apparently moved ~ 20 nm. to the right. This motion is consistent with either interstitial climb, as shown schematically in sketch ② (motion along $[\bar{1}00]$), or $\{100\}$ glide (motion along $[011]$). The furthest extent of the cluster damage apparently lies at the pre-irradiation position of the dislocation.

Finally, in common with figure 7.2, the erosion of a large pre-existing SFT is also seen in this figure. A smaller remnant ('C') survives this irradiation as might be expected given the higher dose.

Interpretation:

As outlined within the 'Interpretation' section of figure 7.2, the erosion of the large pre-existing SFT is likely to result from interstitial absorption. It would appear likely, therefore, given the formation of copious vacancy clusters, that interstitials are also migrating to the sessile dislocation and, as set out within the 'Points of Discussion' section in chapter 3, that both the fine-scale cusping and the motion of this 'sessile' dislocation is therefore a result of interstitial climb.

Unfortunately micrographs were not taken in the (100) pole, from which it could easily have been seen whether climb or glide is responsible for the motion of the dislocation. However, whilst the sessile dislocations of figures 3.6, 4.5 and 4.7 were only slightly displaced from their original positions, the magnitude of the motion here is apparently sufficient for climb to be distinguished from glide, in spite of the smaller range of specimen tilt available than might otherwise have been desired. This determination is contained within the 'Points of Discussion' section which follows.

Finally, the gap in the line of cluster damage between 'A' and 'B' may result from vacancies migrating to these large vacancy loops rather than forming into small SFT.

Points of Discussion

- *Sessile Dislocations: Climb or Glide?*
- *The Distribution of Vacancy Damage.*
- *When a Dislocation Tends Away from 60° Orientation.*

Sessile Dislocations: Climb or Glide?

As mentioned above (figure 7.11) the sense of motion of the sessile dislocation $\tilde{b}_T = \vec{B}\vec{D}$ (δ/β) is consistent either with glide along $[011]$ or interstitial climb along $[\bar{1}00]$. These may be distinguished by making careful measurement of the distances of vacancy clusters from the post-irradiation dislocation position at $\tilde{B} \approx [\bar{1}\bar{1}\bar{1}]$ and $\tilde{B} \approx [\bar{2}\bar{1}\bar{1}]$ foil orientations. The above measurement may be made employing any cluster which may be recognized in any reflection taken at both specimen orientations. Two such clusters are 'B' and 'C' (Burgers vectors $\vec{B}\vec{D}$). Cluster 'B' is seen to lie furthest from the dislocation in the post-irradiation micrograph 7.9(c) and is therefore most likely to correspond to the pre-irradiation dislocation position, rather than any intermediate position. The actual measurements made on cluster 'B', at a total magnification of 2100kx and employing $\tilde{g} = [\bar{2}20]$ and $\tilde{g} = [\bar{1}11]$ reflections respectively (cluster 'B' exhibits 'inside' contrast in both), are 27.3 nm. ($= D_{(111)}$) at the (111) pole and 21.7 nm. at the (211) pole ($= D_{(211)}$). The measurement error is ± 0.4 nm..

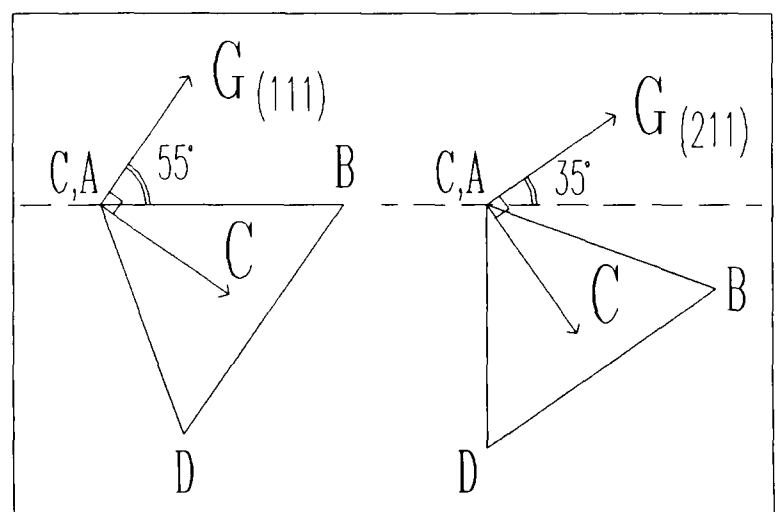
The results of this foil tilt on the projections of both unit glide (G) and climb (C) motion is represented schematically in diagram 7.3. In effect motion due to glide along $[011]$ will be more in evidence on tilting to $\tilde{B} \approx [\bar{2}\bar{1}\bar{1}]$ from $\tilde{B} \approx [\bar{1}\bar{1}\bar{1}]$, whilst the projected distance of interstitial climb motion along $[\bar{1}00]$ is decreased for the same tilt. For glide motion, the ratio of the two projected distances (R) is given by:

$$R = \frac{G_{(111)}}{G_{(211)}} = \frac{\cos 55^\circ}{\cos 35^\circ} \simeq 0.7$$

Whilst for climb motion the same ratio is given by:

$$R = \frac{C_{(111)}}{C_{(211)}} = \frac{\cos 35^\circ}{\cos 55^\circ} \simeq 1.4$$

Diagram 7.3



The above measurements yield a value for the ratio R ($= D_{(111)}/D_{(211)}$) of approximately 1.25. Similar measurements performed on other features within the line of cluster damage also yield ratios $1.2 < R < 1.3$. The exact ratio of 1.4, as expected for pure climb motion of the line, was not

found. This may indicate either that motion is a combination of both climb and glide, or the presence of a systematic error resulting from different positions of the weak-beam image peaks relative to the dislocation core for the two reflections. For austenitic stainless steels, weak-beam image peaks lie up to 1.4 nm. from the dislocation cores (Bampton et al, 1978).

Even for the worst possible case, where the shift in image peaks is at a maximum and is additive for the two reflections, correction for the ensuing over-estimation of the change in the measured separations of clusters and line on tilting from (111) to (211) poles still yields $R > 1$ (actually 1.1). The motion of the sessile dislocation of figure 7.11 thus appears to result from interstitial climb. This is also apparently confirmed by the qualitative observation that the cusps along the dislocation appears 'smoother' when viewed with beam direction $\tilde{B} \approx [\bar{2}\bar{1}\bar{1}]$ ((f)) than when viewed with beam direction $\tilde{B} \approx [\bar{1}\bar{1}\bar{1}]$ (e.g. (e)).

The distribution of vacancy clusters may also indicate climb. As outlined in chapter 4 (figure 4.5), the formation of a 'cloud' of vacancy clusters may be indicative of gradual climb motion rather than discrete glide motion (see also diagram 4.3). Finally, the heavily cusped appearance of the dislocation apparently reinforces the supposition that core diffusion is not an efficient process along these sessile dislocations (see '*Points of Discussion*' in chapter 3), resulting in locally non-uniform climb rates. Alternatively, the cusps may also suggest that the line is climbing via the absorption of interstitial clusters, which must have the same Burgers vector as the dislocation, rather than single interstitials.

The Distribution of Vacancy Damage

In general the relative frequencies of vacancy clusters associated with different dislocation configurations appears to be consistent with observations made in the quaternary alloy containing silicon. Vacancy clusters are seen lying parallel to the evenly dissociated dislocations of figures 7.1 (0.03 dpa), 7.2 (0.06 dpa) and 7.7 (0.08 dpa). The density of vacancy clusters is substantially greater along the 60° section of the dislocation of figure 7.8 (0.1 dpa), culminating in a large SFT ('P') near the large double-jog ('PQ') where the line has climbed. Vacancy clusters are sparse along the climbed section. This is consistent with this climbed section having acted as a recombination centre, as outlined within the '*Points of Discussion*' section of chapter 6, whilst the jog itself has acted as an efficient sink for interstitials. The absence of vacancy clusters close to features 'C'³² of figure 7.7 (0.08 dpa) may further indicate the action of complex configurations, the formation of which may require the initial

³² Unless, of course, loops 'C' are vacancy clusters.

generation of an atmosphere of defects around them, as recombination centres.

The action of jogs as efficient sinks for interstitials may also explain the presence in figure 7.10 (0.14 dpa) of SFT 'G' close to the initial position of cusp 'D', which cusp appears subsequently to have climbed suggesting the presence of a jog.

The situation in figure 7.4 (0.08 dpa) may be slightly more complex: Vacancy clusters are only seen along sections of the dislocation presumed to contain jogs which have climbed; consistent with these jogs acting as efficient sinks. This may not be an effect of a nearby efficient interstitial sink, however, but rather of the now greater hydrostatic strain field associated with these sections as they are twisted away from screw orientation. The clusters at 'T' do appear, however, to lie close to the pre-irradiation dislocation position, which seemingly reinforces the former explanation. The low density of vacancy clusters elsewhere is consistent with the lower hydrostatic strain field of near-screw dislocations (see the '*Interpretation*' section of figure 3.4).

The very high density of vacancy clusters associated with the sessile dislocation of figure 7.11 (0.14 dpa), a substantially higher density than seen along the glissile, dissociated dislocation of figure 7.10 (also 0.14 dpa), suggests that climb is a particularly easy process here. This apparently points towards climb proceeding simply via the assimilation of single interstitials, or possibly of very small interstitial clusters. It can be seen, particularly from micrographs (f) and (h) of the figure, that the area contained within vacancy clusters is of the order of the area over which the dislocation has climbed. This sessile dislocation therefore appears to be acting as a very efficient sink for both interstitials and vacancies.

When a Dislocation Tends Away from 60° Orientation...

Figures 7.4, 7.7, 7.8 and 7.10 all show evidence for the formation of new features at the point where the dislocation tends away from, or towards, 60° orientation. In the case of figure 7.4, the formation of kink 'M', if attributable to jog climb, is likely to result from the absorption of interstitials. In the case of figure 7.8, climb of the line itself, in a sense consistent with interstitial absorption, is seen. Feature 'I', formed where the dislocation of figure 7.10 tends from 60° to 40°, apparently lies to the dilatational strain field of the dislocation. All of these features may indicate core diffusion, as depicted in diagram 4.4. The core diffusion of interstitials is thought to account for a lack of visible interstitial clusters along the dislocations of figures 7.1 and 7.2.

The initial characterization of features 'C' of figure 7.7, two of which lie where the same dislocation as is shown in figure 7.8 tends from 60° to 0° orientations, as vacancy in nature therefore

seems incongruous at this time. The configurations of these features remain unsolved, but are discussed further in chapter 8.

Summary

Observations:

In this material, the following observations are made:

- Disappearance of constrictions; ... 1 ...
- Narrowing of partial separations; ... 2 ...
- Erosion of pre-existing SFT; ... 3 ...
- SFT/vacancy cluster formation; ... 4 ...
 - as a discrete line parallel to evenly dissociated dislocation; ... a ...
 - in a cloud near a sessile dislocation; ... b ...
 - near a jog; ... c ...
- Preferential interstitial loop formation near a dislocation; ... 5 ...
- Zig-zag formation; ... 6 ...
- Climb; ... 7 ...
- Dipole disappearance; ... 8 ...
- Loops formed along, and reacted with, dislocation; ... 9 ...
- Interstitial cluster along 60° dislocation; ...10 ...
- Fine-scale cusping; ...11 ...
- Unpinning. ...12 ...

Individual figures exhibiting these phenomena are listed in table 7.1.

Pre-existing constrictions are often seen to have disappeared in this material, in common with the pure ternaries. This may then allow the core diffusion of point-defects attracted to the dislocations as outlined within the ‘Points of Discussion’ section in chapter 3. In this material interstitials appear to be the principal defect species undergoing core diffusion.

With the exception of figure 7.8, and possibly also figure 7.7, dissociated dislocations of mixed or edge character do not generally appear to have climbed, as was seen in chapter 6. The climbed section of the dislocation in figure 7.8 is very short and lies where the dislocation tends away from 60° orientation.

The observation that pre-existing constrictions along the near-screw dislocation of figure 7.4 have not always given rise to a zig-zag configuration may indicate solute de-segregation. This is

discussed within chapter 8.

Finally, as was seen in chapters 3, 4 and 5, but not in chapter 6, interstitial loops have formed in the bulk, after all apart from the lowest dose (0.03 dpa) irradiation. The presence of many other loops close to dislocations is not reminiscent of previous chapters, however.



Table 7.1

Figure	Character	Dose (dpa)	Temperature (°C ± 50°)	Observations (see above)
7.1	80°	0.03	420	1, 2, 4a, ;
7.2	60°	0.06	420	1, 2, 3, 4a, 12;
7.4	~ 0°	0.08	420	1, 4a, 4c, 5, 6, 7- onset of helix, 8;
7.5	glissile?	0.1	420	4a, 5;
7.6	sessile	0.08	420	5, 7- of prismatic loop;
7.7	0°- 60°	0.08	420	1, (2), 4a, 5, 8, 9;
7.8	30°- 60°	0.1	420	4a, 4c, 7-double jog formation;
7.10	0°- 60°	0.14	420	1, 2, 4a, 4c, 6, (7- helix?), 10, 12;
7.11	sessile	0.14	420	3, 4b, 7- of sessile dislocation, 11

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Chapter 8

DISCUSSION

The Mechanisms of Interaction of HVEM Generated Point-Defects with Line Dislocations

8.1 : Fundamental Mechanisms:

The fundamental mechanisms by which point defects are interacting with dislocations in all the alloys studied appear to stem from the trapping of defects at, or close to, the dislocation core. The trapping of defects arises from a reduction in defect formation energy within the dislocation strain field. These trapped defects may then undergo one, or more, of the following process; leading to one, or more, of the following observations:

<u>Process</u>	<u>Observation</u>
• Defects attach to the line directly;	Climb
• Defects remain near the line;	Small cluster precipitation
• Defects pipe diffuse until they encounter (for example):	
Surfaces	Lack of evidence for defect precipitation near line
Jogs	Jog climb

Pipe diffusion is also thought to be involved in the observations of defects precipitating at sections of dislocation of exactly 60° character, and/or of the possible build-up of a very complex dislocation network at a non-planar node (figure 4.7). Pipe diffusion may also be implicated in the formation of other, frequently complex, configurations in the 15%Ni+2%Mo alloy where a dislocation tends away from, or towards, 60° character.

It is shown below that most, or all, of these observations may be understood in terms of the same model, which attempts to balance these competing processes. Initially we consider principally the three ternary alloys. Observations of irradiated dislocations in the quaternary alloys, which are discussed slightly later in this chapter (section 8.3), may also be understood in terms of this model, which is modified slightly to take into account the expected influences of the minor element additions.

Within this section (§8.1) we summarize, first, certain key observations, and associated ‘explanations’ put forward within individual ‘*Points of Discussion*’ sections. In §8.2 we attempt to isolate materials dependent trends in the results.

8.1.1 Small Cluster Precipitation:

Small clusters are commonly found to be associated with irradiated dislocations. For the 25%Ni and 40%Ni pure ternaries, and the quaternary alloys, this cluster damage is frequently

interspersed with SFT. In all of these cases³³, both small clusters and SFT were always found to lie to the compressional strain field side of the dislocation. In other cases, where SFT are not seen amongst small clusters, which exhibit contrast inconsistent with any perfect or Frank loop variety, the clusters are also found to lie to the compressional strain field side of the dislocation³⁴. It is thought, therefore, that all of these small clusters which lie close to a pre- or post-irradiation position of a dislocation are vacancy in nature. Further evidence cited (see '*Interpretation*' section of figure 3.4), leading to the same conclusion, was the apparent absence, or very low density, of these clusters close to screw dislocations; this is believed to result from the low first-order size-effect interaction of a vacancy with the decreased hydrostatic strain field around near-screw dislocations (see, e.g., **Hirth & Lothe**, pp.497-506).

Hence, it would seem likely, although it cannot be directly confirmed, that the very small clusters seen to have formed almost homogeneously along dislocations of mixed character in the 15%Ni ternary alloy (figures 3.1, 3.2 and 3.3) are also vacancy in nature (whilst this characterization is originally made within the '*Interpretation*' section of figure 3.3, most of the corroborative evidence is not presented until later).

8.1.2 The Lack of Evidence for Interstitials along Evenly Dissociated, Glissile, Dislocations:-

Pipe Diffusion.

As mentioned initially in chapter 1, dislocations are expected to attract a greater number of interstitials than vacancies. Thus, the presence of vacancy clusters close to dislocations suggests the action of dislocations as efficient sinks for interstitials (see **Jenkins et al**, 1987). In figure 3.4, interstitial absorption is likely to have led to the climb of a left-handed screw dislocation to form a right-handed 'flat-helical' configuration; evidence of interstitials, albeit only of a few, is thus apparent. However, in the ternary alloys, the lack of evidence for interstitials along evenly dissociated dislocations of mixed, or edge, character (e.g., figures 3.1, 3.2, 3.3 and 3.5 for the 15%Ni ternary alloy) is particularly marked (with the possible exception of the two clusters 'A' of figures 3.1 and 3.2 mentioned below). Mixed and edge dislocations are expected to attract a greater number of interstitials than screw, or near-screw dislocations, by reason of their larger hydrostatic strain field (see, e.g., **Hirth & Lothe**, p.503; eqn. 14-42). These interstitials have not recombined with the high density of very small vacancy clusters and neither have they led to climb of the line (e.g. figures 3.1, 4.5 and 5.3).

A possible explanation for this apparent excess of vacancies migrating to dislocations is

³³See figures 3.4, 4.3, 4.4, 4.5, 4.6, 5.3, 6.1, 6.2, 6.4, 6.5, 6.6, 6.7, 6.8, 6.9, 7.1, 7.2, 7.4, 7.5, 7.6, 7.7, 7.8, 7.9, 7.10 and 7.11.

³⁴Figures 3.6, 4.2, (5.1), 5.3, 5.4 and all figures of the above footnote.

introduced in the '*Points of Discussion*' section of chapter 3; namely that interstitials are able to core, or pipe, diffuse readily along evenly dissociated, glissile, dislocations until they either encounter a barrier, such as a jog, kink or Lomer dislocation, or are lost at the surfaces. In the case of a Lomer dislocation, which is likely to be constricted (see '*Points of Discussion*', chapter 3), interstitials are apparently able to attach easily to the line, allowing the formation of vacancy clusters nearby and leading to climb along the entire length of the dislocation.

In the case of mixed and edge dislocations, it is shown in figure 3.7 that the sense of jog climb will be such as to lead to their mutual annihilation (the origins of these jogs are probably thermal and they are thus likely to be of equal height; see **Hirth & Lothe**, p.495). Presumably, pipe diffusion may then proceed with relative ease. In the case of screw, and near-screw, dislocations, however, the climb of individual jogs may lead to the formation of a zig-zag configuration. This zig-zag configuration may further inhibit pipe diffusion such that climb of intervening segments is able to occur. The climb of dissociated dislocations is discussed in section 8.1.4.

8.1.3 The Formation of Large Clusters on 60° Dislocations:

The concept of pipe diffusion is further employed in chapter 4 to account for both the presence of a large cluster *very* close to a dislocation in exactly the 60° orientation (see diagram 4.4), and for the seemingly very complex network built up at the node of figure 4.7, probably by the intersection of lengths of dislocation which were not pre-existing.

Large clusters are seen along lengths of 60° dislocation in all the alloys studied. These clusters are considerably larger than the small (vacancy) cluster damage referred to above (§8.1.1). In figure 5.3 (40%Ni), cluster 'G', which often appears similar to 'B' of figure 6.4 (15%Ni+ Si), is apparently solely vacancy in nature. There may be indirect evidence that vacancy pipe diffusion is relatively efficient in the 40%Ni alloy³⁵. In figure 4.6 (25%Ni), feature 'B', which has certain similarities with both features 'B' of figure 6.4 and 'I' of figure 7.10 (15%Ni+Mo), appears to be composed of both interstitial and vacancy defects. In figures 3.1 and 3.2 (15%Ni), two large clusters ('A') lie towards the middle of lengths of dislocation which are exactly in 60° orientation. Both of these clusters appear to lie very close to the dislocation and are substantially larger than any other features seen along the line. The fact that neither of these clusters could be characterized using standard techniques may be attributable to the proximity of these features and the dislocation, the presence of a heavy contamination layer formed after this specimen was maintained at temperature for 2 hours in the HVEM, and/or the possibility

³⁵Figure 5.4; if large scale atomic redistribution is occurring via the core diffusion of vacancies towards the surface.

that these clusters are three-dimensional. In the 15%Ni ternary alloy, the lack of evidence for interstitials along evenly dissociated dislocations of mixed character is particularly marked. However, climb of the screw dislocation (figure 3.4) by the absorption of interstitials indicates that interstitials are migrating to dislocations and, thus, the pipe diffusion of interstitials appears to be an efficient process here. Notice also that the very small clusters of figures 3.1, 3.2 and 3.3, which are thought to be vacancy in nature and which lie very close to the dislocation core, have not coalesced into larger features but are instead almost homogeneous along the entire length of the dislocations. This may indicate that the pipe diffusion of vacancies is not an efficient process here (see §8.2.3). It would seem possible, therefore, that clusters ‘A’, the origins of which seem likely to involve core diffusion, are actually interstitial in nature, formed from ‘plated-out’ pipe diffused interstitials via the mechanism of diagram 4.4. It would also seem possible that the nature of clusters precipitated preferentially at points where the dislocation is exactly in 60° orientation should represent the nature of the principal core diffusing defect species. This is summarized in the following chart:

<u>Material</u>	<u>Nature of Cluster at 60°</u>
40%Ni ternary	Vacancy
25%Ni ternary	Vacancy & Interstitial
15%Ni ternary	Interstitial ?

8.1.4 Dissociated Dislocation Climb:

For glissile dissociated dislocations in the ternary alloys, large scale climb is apparently only evident along screw and near-screw dislocations (figures 3.4, 3.5, 4.1 and 5.4). In all of these cases, and, in fact, for all irradiations performed, the Burgers vectors of dislocations remain unchanged after irradiation. Dipole formation is also not seen (except for one case in the 15%Ni+Si alloy) and thus the mechanism of **Cherns et al** (1980) does not seem to be operating.

In the 40%Ni alloy, climb is not seen along theunjogged screw dislocation of figure 5.2, suggesting both that the nucleation of new jogs is not occurring and that pre-existing jogs are important as sites at which zig-zagging occurs for these ternary alloys. This is apparently confirmed by the correlation between pre-existing jogs and post-irradiation zig-zag configurations exhibited particularly by the dislocations of figure 3.4 and 4.2.

In the ‘*Interpretation*’ section of figure 5.4, it is argued that the mechanisms of **Thomson & Balluffi** (1962), or of **Condat & Décamps** (1985), may be operating. The operation of either mechanism may explain the observations, for all climbed screw dislocations, that the Burgers vectors of climbed sections are unchanged and that the dislocations apparently remain dissociated. We consider first the

Grilhé et al (1977) performed a number of calculations aimed at determining the activation energies for operation of the Thomson & Balluffi mechanism. These calculations were accomplished by evaluating the energies of an assumed, and apparently plausible, intermediate configuration consisting of an evenly, and widely, dissociated parent dislocation and a partially extended prismatic loop nucleated onto their partial $\vec{B}\gamma$ (see their figure 2). In performing these calculations, via methods employed by **Jøssang & Hirth** (1966), all dislocations are assumed to be straight allowing them to approximate 91 interaction energy terms. This is the same method employed in the predictions that Lomer dislocations should be widely dissociated (see '*Points of Discussion*', chapter 3). The essential results from these calculations are that a critical loop size exists, likely to have dimensions of the order of the dislocation dissociation width, below which extended double jog formation will not occur, and that as SFE is increased a transition from extended double-jog (loop of critical size) nucleation rate-controlled, to bulk diffusion rate-controlled, climb will occur. Their calculations also hint at the expected shapes of nucleated prismatic loops (rectangular with long axes along the dislocation) and provide values for the numbers of defects required to form a loop of critical size as a function of SFE (number decreases with dissociation width). As pointed out by **Grilhé et al**, simple elasticity considerations render these trends likely but it appears that caution should be employed in the liberal, and frequent, use of absolute values arising from these, and similar, calculations; particularly when they are used in a predictive capacity.

Later in their paper, **Grilhé et al** refer to calculations aimed at determining the activation energies for double extended jog formation as a function of total dislocation character. In this case they predict that the activation energy for operation of the Thomson & Balluffi mechanism will show a shallow minimum for dislocations of approximately 45° total character. However, they also appear to state (their §3.1) that their calculations always employ *the same* basic intermediate configuration *irrespective* of the total dislocation character; namely the nucleation of the initial prismatic loop onto partial $\vec{B}\gamma$. Bearing in mind the potential importance of the character of the parent partial in CuAl, as exemplified by the work of **Cherns et al** (1980), this assumption would appear patently *unlikely*. If this is indeed the case, failure to take the expected nucleation of the prismatic loop onto the partial of greater edge character into account may have significantly altered their results, since all 91 interaction energy terms will be different. The reliability of these results must therefore be questioned. Nevertheless, we make one qualitative observation, namely that the activation energy for operation of

the Thomson & Balluffi mechanism *may* decrease as the dislocation tends towards screw character simply as a function of the decreased dissociation width. This effect will be reduced, and quite possibly negated, however, by the greater difficulty inherent in nucleating a rectangular prismatic loop along the dislocation, of the same Burgers vector as the dislocation, if such a loop (and hence the parent dislocation) is not in pure-edge orientation (consider the climb force).

In 1985 **Condat & Décamps** proposed a mechanism to explain their observations of segmented helices formed after quenching in low SFE CuSi and CuAl alloys (see ‘*Interpretation*’ section of figure 3.4 for references). These segmented helices were formed along screw dislocations, whilst mixed and edge dislocations in the same specimen assumed complex structures involving the formation of many Shockley and stair-rod dipoles along individual partials (**Décamps et al**, 1983). Shockley dipoles frequently possessed different Burgers vectors from their parent partials. **Décamps et al** (1983) interpreted their observations in terms of the nucleation based mechanism of **Cherns et al** (1980), involving the copious nucleation of vacancy type Frank loops, some of which subsequently unfault, onto individual partials.

The hypothesized nucleation of Frank loops led **Condat & Décamps** (1985) to explain their observations of segmented helices via a similar route. Their mechanism is reproduced in figure 8.1. Certain features of this mechanism which should be noted are, first, that alignment of partial $\vec{A}\delta$ such that it is in pure-edge orientation along $[10\bar{1}]$ must occur *prior* to the initial nucleation of loop $\vec{A}\alpha$ and, second, that the perfect loops, with Burgers vectors $\vec{A}\vec{B}$, are composed of glissile edges. If the calculations of **Bullough et al** (1964) and **Bacon et al** (1970), referred to in the ‘*Interpretation*’ section of figure 3.5, may be extended to other materials then it is possible that these edges may align along $\langle 123 \rangle$ directions. The diagram of **Condat & Décamps** (1985) shown in figure 8.1 appears slightly misleading at first: Nucleation of a second loop $\vec{B}\beta$ onto partial $\delta\vec{B}$ proceeds between steps (b) and (c) whilst point N' is below, and point M above, the dissociated dislocation $\vec{A}\vec{B}$. Unfortunately time does not permit the redrawing of this figure for the interstitial case but its method of operation will be similar, except that loop $\vec{\alpha}\vec{A}$ must unfault to form $\vec{B}\vec{A}$ *prior* to reaction with $\vec{A}\delta$, leading to the formation, and subsequent mutual annihilation, of a dipole $\delta\vec{B}/\vec{B}\delta$. The sense of rotation of the helix (its left or right-handedness) is also reversed. Finally, the nucleation criteria for the operation of this mechanism have not been analysed, as has been done for the **Thomson & Balluffi** (1962) mechanism (**Grilhé et al**, 1977). It seems improbable, however, that its activation energy should be zero since sufficient torque must be exerted on dislocation $\vec{A}\vec{B}$ to prompt the glide of partial $\delta\vec{B}$ towards point K .

Figure 8.1

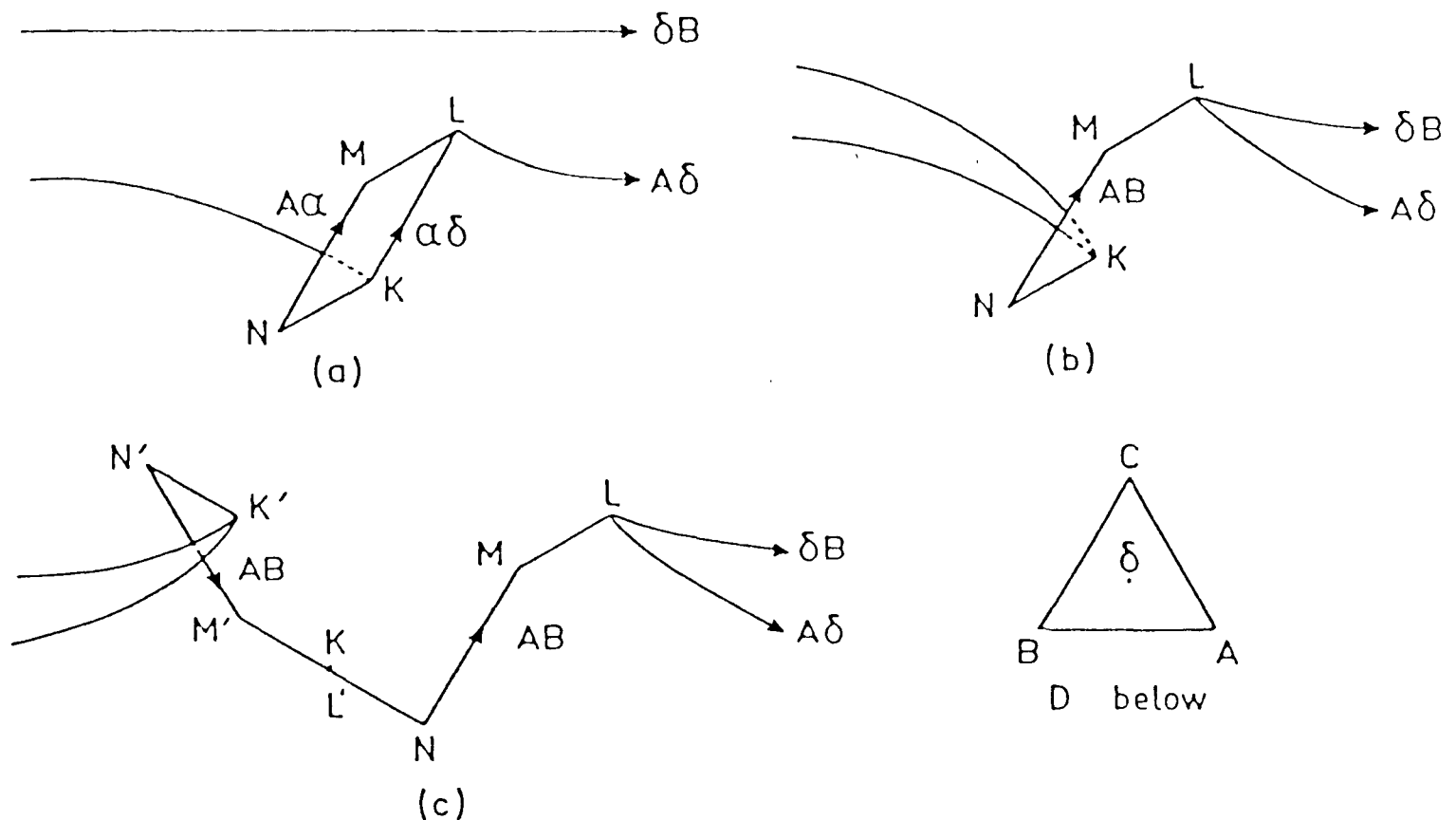


Diagram from *Condat & Décamps (1985)*

VACANCY CASE

- (a) Initial Nucleation of $\vec{b} = \vec{A\alpha}$ loop above partial $\vec{A\delta}$ of evenly dissociated dislocation,
- (b) Constriction of dislocation $\vec{AB}$ to form partial $\vec{\alpha B}$ which unfaults loop $\vec{A\alpha}$,
- (c) Secondary nucleation of loop $\vec{B\beta}$ below partial $\vec{\delta B}$ to form $\vec{AB}$ via similar route to (b).
Inclined lengths of dislocation $\vec{AB}$, $\vec{M'L'}$ and $\vec{KN}$ may glide and straighten on γ .

INTERSTITIAL CASE

- (a) Initial Nucleation of loop $\vec{b} = \vec{B\Lambda}$ below partial $\vec{A\delta}$ to form $\vec{B\delta}$,
- (b) Constriction of dislocation $\vec{AB}$; $\vec{B\delta}/\vec{\delta B}$ annihilate,
- (c) Secondary nucleation of loop $\vec{AB}$ above partial $\vec{\delta B}$. Inclined lengths straighten as for the vacancy case.

Therefore, in reference to both mechanisms, we refer below to a ‘critical size loop’, by which we mean a cluster of interstitial defects formed close to the dislocation of sufficient size such that either mechanism is able to proceed unhindered. The term ‘super-saturation threshold’ is also employed, denoting the local point-defect super-saturation level required such that these loops are formed; it seems likely that this will vary with dislocation dissociation width. In the event that these ‘critical size loops’ require fewer defects than the minimum required to nucleate a loop in the immediate vicinity of the dislocation (n_i in Grilhé et al, 1977) double-jog nucleation, or the formation of segmented helices, will be effectively a diffusion rate-controlled process (near the dislocation the activation energy for loop nucleation is likely to be small) and there will be no interstitial super-saturation at the dislocation. (see, e.g., Balluffi, 1975; Hardy, 1985). If loops nucleated in the immediate vicinity of the dislocation must grow prior to operation of either mechanism, a super-saturation will develop. Climb may then be said to be nucleation, or assimilation, rate-controlled.

We now consider observations made in these Fe-Ni-Cr alloys: The lack of evidence for climb alongunjogged dislocations, of whatever character, suggests that the local point-defect (interstitial for HVEM irradiation) super-saturation is always in the region where climb is nucleation rate-controlled: The critical loop sizes either for extended double-jog (Thomson & Balluffi) or segmented helix (Condat & Décamps) formation are not being exceeded because defects are able to pipe diffuse readily along dislocations. As mentioned above this is expected to occur after the annihilation of pre-existing jogs for mixed and edge dislocations.

For the case of jog climb, evidence, particularly in the 15%Ni+Si alloy but also in the ternaries, suggests that climb occurs readily, allowing the nucleation of copious vacancy clusters near dislocations of mixed or edge characters (e.g. figures 4.2, 5.1, 6.4). Jog climb therefore appears to proceed uninhibited and is thus likely to be bulk, and core, diffusion rate-controlled; i.e. the activation energy for its occurrence is likely to be less than that required for the climb of a dissociated dislocation. The climb of sessile dislocations similarly appears to be diffusion rate-controlled.

Hence, in order for climb to have occurred between jogs on screw dislocations, it must become more favourable than either further climb of the jogs themselves or pipe diffusion. Intuitively, the origin of this effect would appear as follows: Pre-existing jogs climb away from screw dislocations. As the zig-zags form, line tension forces will inhibit further jog climb. All sections of the line will have developed edge components. If pipe diffusion is inhibited, a super-saturation builds-up which results in climb. This is now analysed in a little more detail below with respect to experimental observations.

Evidence is presented in figures 3.5 and 4.1 which suggests that the lateral climb of jogs is the first process in the formation of helical configurations, as depicted in **Hirth & Lothe** (p.626). (This has important ramifications on the mechanism of **Condat & Décamps** as discussed below).

In order for a helical configuration to form, further climb of the jogs themselves must be inhibited (assuming that pipe diffusion along these intervening segments still occurs) such that climb occurs along intervening segments (by either mechanism); in effect, a restraining force must act on jogs such that the local interstitial super-saturation attains a sufficient level for climb to occur along intervening dissociated dislocation segments. Possible origins of this force are indicated in the text relating to diagram 4.3; namely the alignment of segments of dislocation along apparently favourable directions and the increase in line tension forces arising from jog climb. Pinning due to vacancy clusters is obviously not likely here.

In figure 5.1, jogs along the dislocation appear to have acted as effective barriers to core diffusion. For a screw, or near-screw, dislocation pre-existing jogs will not annihilate (see figure 3.7) and will continue to act as core diffusion barriers³⁶. It is possible that jogs along a dislocation line may have a similar effect on the core structure as the kink shown in diagram 3.2. The presence of lasting barriers to core diffusion may then allow the super-saturation threshold for 'critical size loop' nucleation to be exceeded. This would explain the lack of evidence for climb (**Thomson & Balluffi**), or loop nucleation (**Condat & Décamps**, via **Décamps et al**, 1983), along mixed and edge dislocations in the ternary alloys.

At no stage is a two-dimensional, zig-zagged, screw dislocation seen to be composed of segments aligned along $\langle 110 \rangle$ directions. This would appear to be a necessary pre-requisite of the mechanism of **Condat & Décamps** (1985) and may suggest that the **Thomson & Balluffi** (1962) mechanism is, in fact, operating along dissociated dislocation sections between jogs. Alternatively, if the critical loop size required for nucleation of the initial Frank loop is very small, it is possible to imagine ways in which such a loop could nucleate very close to a jog, which may be constricted, unfault and then climb directly along the dislocation³⁷; this would have the same result as the

³⁶*The action of jogs as barriers to core diffusion has been assumed, indirectly, for a number of years (e.g., **Balluffi**, 1975) and throughout this thesis. It is extremely difficult to confirm, however, in the absence of direct experimental, or theoretical, determinations of dislocation diffusion coefficients for jogged, and unjogged dislocations of the same character. It seems possible that jogs along dislocations may be a contributory factor to the large scatter found in experimental determinations of dislocation diffusion coefficients (e.g., **Balluffi**, 1970).*

³⁷***Segall & Shoaib** (1970) suppose a similar mechanism.*

nucleation of a single, large loop $\overline{AB}$ along the entire length of a dislocation segment between jogs. A 'flat-helical' configuration would result from the nucleation, unfauling and climb along the dislocation, of different inclined Frank loops on two adjacent segments. Such a process may also inhibit further jog climb. At the time of writing this problem has not been resolved but the nucleation criteria for small (or large) Frank loops along these dislocations seem worthy of further study.

In summary, therefore, the essential conclusion to be drawn from the above treatment is that the climb of screw dislocations to form helices, or 'flat-helices', is likely to be a nucleation rate-controlled process. This will require the build-up of an interstitial super-saturation at the line until the threshold for climb of short, dissociated, dislocation segments is reached. The build-up of this super-saturation, which must, of course, be lower than the bulk super-saturation, is likely to result from decreases in the efficiency of pipe diffusion (arising from the presence and survival of pre-existing jogs on the line) and dislocation core energies (arising from the dislocation twisting to lie along favourable directions), and an increase in line tension (arising from the extension of dislocation segments as the initial, principally two-dimensional, zig-zag is formed).

8.2 : The Effects of Increasing Nickel Content:

The effects of increasing nickel content on post-irradiation dislocation configurations appear slight in these experiments. In all three ternary alloys, only screw, and near-screw dislocations are observed to climb, whilst mixed, and edge, dislocations appear little affected by irradiation: The mechanisms of interaction of HVEM generated point defects therefore appear very similar, involving principally the pipe diffusion of interstitials whilst vacancies cluster nearby. Some qualitative differences in post-irradiation dislocation structures, ^{are evident} from which much may be inferred. The remarks made below are speculative, however, since it is possible to think of many other possible origins for the effects discussed.

8.2.1 The Formation of Interstitial Loop Depleted Zones (LDZ):

Both screw, and (initially) near-screw dislocations of figures 5.2 and 5.4 apparently lie at the centre of interstitial loop depleted zones (LDZ). In the case of figure 5.4, this interstitial LDZ is particularly marked; extending up to 100nm. from the near-screw dislocation. In contrast, zig-zagged dislocations in the 15%Ni ternary alloy (figures 3.4 and 3.5), show no evidence for the formation of LDZ's after irradiations which have given rise to similar densities of interstitial background loops. In figure 3.4, formation of the 'flat-helical' configuration apparently involves the assimilation of approximately 4000 (2700–8000) interstitials over an 85nm. length of dislocation. In figure 5.4 the

number of interstitials absorbed, over the entire length of the helical part of the line (~ 250 nm.), appears to be only of the order of 4000.

Whilst both these numbers are extremely approximate, it may be noted that, in absolute terms, they represent only very small fractions of the total number of defects produced near irradiated dislocations; for example, the 0.05 dpa irradiation of figure 3.4 is expected to displace ~ 4000 atoms within a cubic volume with dimensions of only 9 nm. in this material.

We therefore have apparently contrasting behaviour; in the case of the 15%Ni ternary alloy, the lack of an interstitial LDZ is consistent with the absorption of only a few atoms; in the 40%Ni alloy (figure 5.4), however, it can easily be seen that the total area ‘missing’ from background loops is considerably greater than the total area of the helical turns. We thus have a shortfall between the number of interstitials likely to have migrated *to* the dislocation, and the number of interstitials apparently absorbed *at* the dislocation.

We have argued above that jogs act as effective barriers to core diffusion. Hence, the possibility that this ‘shortfall’ should be due to the core diffusion of interstitials to a surface, as supposed to be the case for theunjogged screw dislocation of figure 5.2, would seem unlikely. We have also argued (§8.1.4) that the climb of these dislocations is not likely to be diffusion rate-controlled; requiring instead the build-up of a local interstitial super-saturation prior to the nucleation of whatever configuration is required for climb to proceed (a prismatic loop in the case of the **Thomson & Balluffi** (1962) mechanism). It would appear plausible, therefore, that screw dislocations in both alloys (and presumably also the 25%Ni alloy) are acting principally as recombination centres, absorbing only the net excess of interstitials (dislocation bias) to form a helix. The dislocation of figure 5.4, to which a great many interstitials have apparently migrated, although only a few appear to have been absorbed, would seem to be particularly efficient as a recombination centre. A possible dependence of the dislocation bias on nickel concentration is discussed later in the chapter.

8.2.2 *The Narrowing of Partial Separations:*

Measurements of apparent partial image separations before and after irradiation are shown in table 8.1 for all the materials studied. In almost all cases these separations have narrowed. Several possible origins for this effect must be considered:

In HVEM irradiated silver (**Hardy**, 1985; **Jenkins et al**, 1987) partial separations were seen to decrease at an early stage of irradiation. **Hirsch** (unpublished work) in considering the effect of small inclusions nucleated preferentially within the dislocation strain field, showed that for a dislocation

Table 8.1

Apparent Partial Image Separations (Å) and Dislocation Character

Figure	Pre-Irradiation		Post-Irradiation		Material
3.1 (1)	105	70°	}	40	15%Ni ternary ^a
3.1 (2)					
3.2	85	60°			
4.2	40-50	40°		~ 30	25%Ni ternary
4.3				~ 30 ^b	
4.6	40	60°		35	
5.3	30	60°		~ 30 ?	40%Ni ternary ^c
6.1	35 ± 10 ^d	0°		35	15%Ni+Si
	55 ± 10	25°			
6.2	45	25°		30	
6.4	78	75°		47	
6.6	70	70°			
7.1	}	~ 100	60°	35	15%Ni+Mo ^e
7.2				50	
7.10				40	
				33	

^aDifficult to measure for 15%Ni ternary alloy owing to heavy contamination. All post-irradiation partial image separations are apparently ~ 30-50Å from estimates of stacking fault width.

^bApparently not altered greatly from pre-irradiation value.

^cAs described in chapter 5, contamination and specimen orientation problems often prevented these measurements.

^dSee figure.

^eIt is not possible to determine equilibrium pre-irradiation partial image separations in this material.

greater than 30° in character the partial separation may constrict. The same mechanism also predicts a force aiding, and perhaps prompting, glide and cluster raft formation for near 60° dislocations, as suggested by **Hardy** (1985). For dislocations of less than 30° character, however, the opposite sense of the edge components of the partials might be expected to lead to a *widening* of the partial separations. This latter effect, although it is expected to be small, was not obvious in silver, whilst the opposite is true in Fe-Ni-Cr (e.g. figures 4.2, 6.1 and 6.2).

Disregarding the possible widening of partial separations for screw dislocations, the possibility that constriction of the partials is due to small clusters is consistent with observations in silver (discussed later in this chapter, see §8.5.1); small clusters were always seen to be associated with irradiated dislocations.

In these Fe-Ni-Cr alloys small clusters are also seen associated with dislocations, and the same, or similar, mechanism may be operating. In some cases, however, partial separations are seen to have decreased in the absence of visible clusters (e.g., figure 6.1, 6.2, 7.1). In addition, if narrowing is due to small clusters then dislocations which have glided away from lines of clusters (e.g. figure 4.2) would be expected to re-dissociate. The motion of dislocations away from lines of clusters was a common observation in these experiments, most, but not all of which are shown in this thesis; dislocations were *never* observed to redissociate at any stage, however. Finally, in some cases the degree of narrowing of partial separations does not appear to depend on the cluster density (e.g. compare figures 7.1 and 7.2). Figure 7.10, however, shows the degree of narrowing to be greater for the 60° dislocation section, with which many vacancy clusters are associated, than for the 40° dislocation section, with which very few clusters are associated.

The possibility that small clusters are indeed responsible cannot be discounted, therefore. The large spread in the measurements may be ascribed simply to a large experimental scatter, one origin of which may be the omission of image shift corrections (e.g., **Cockayne et al**, 1971), and that where narrowing has occurred, and visible clusters are not seen, sub-microscopic clusters are responsible.

It should be noted, however, that taking the experiments as a whole, the correlation between partial constriction and small cluster damage was not obvious. A number of other effects, particularly major element segregation as considered below, may also lead to a decrease in partial separations.

Major Element Segregation:

Radiation Induced (R.I.S.) and Thermal Segregation

The theory behind the phenomenon of R.I.S. has been extensively reviewed by **Marwick** (1978), **Perks** (1988), **Ashworth** (1989) and **English et al** (1990) and is therefore not duplicated here.

The variation of SFE (i.e. partial separation) with nickel content in Fe-Ni-Cr alloys is well documented (see table 1.2), as is the depletion of chromium, and corresponding enrichment of nickel, which have been found to be over-sized under-sized respectively in Fe-Ni-Cr alloys (e.g., **Dyson & Holmes**, 1970; **Straalsund & Bates**, 1974), at defect sinks such as surfaces, grain boundaries, voids and cavities, under a variety of heat treatment and irradiation conditions³⁸. Nickel depletion, and corresponding chromium enrichment, at the peak damage centres of proton (**Kimoto & Shiraishi**, 1987), and heavy-ion (**Perks**, 1988; **English et al**, 1990) irradiations has also been observed.

A similar effect may be expected to occur at dislocations (e.g., **Kenik**, 1986), and indeed has recently been observed, by X-ray mapping in a STEM after ion irradiation to 5 dpa (**Shepherd**, 1990). This paper is worthy of note in that segregation to dislocations lying *in the plane* of thin foils has been detected, an accomplishment until very recently thought to be beyond the capabilities of analytical techniques (e.g., **Saka**, 1984). Major element segregation, previously seen to alter partial separations under low-dose HVEM irradiation (e.g., **Saka**, 1983, 1984), may therefore have a major effect in Fe-Ni-Cr alloys.

One feature which is apparent on reference to table 8.1, even though there is a relatively large scatter in the measurements, is that the post-irradiation partial separation appears to be relatively independent of material: All post-irradiation partial separations apparently correspond to those expected for nickel concentrations of the order of 30 to 40%. The observation that partial separations have narrowed greatly for the 15%Ni alloys, but little, if any, for the 40%Ni ternary alloy is consistent with experimental evidence which shows that the degree of major element segregation decreases sharply with increasing nickel content (e.g., **Muroga et al**, 1990), exhibiting a minimum at 45%Ni (**Perks et al**, 1989).

In all of these studies, however, significant segregation is not expected to occur below a total dose of a few dpa. Before, dismissing R.I.S. to dislocations as a potential origin for the narrowing of partial separations in these HVEM irradiated Fe-Ni-Cr alloys entirely, however, we note both the paper of **Muroga et al** (1987), in which a significant degree of compositional redistribution ($\sim 5\%$ Ni depletion at the beam centre) was observed for a 100 nm. thick foil after only 1.5 dpa, and the strong dependence of the apparent SFE on temperature in Fe-Ni-Cr alloys (e.g., **Bampton et al**, 1978). **Muroga et al** (1987) attribute the magnitude of Ni depletion to a thin film effect, arising from the

³⁸For example: **Okamoto & Wiedersich**, 1974; **Marwick**, 1978; **Rothman et al**, 1980; **Sethi & Okamoto**, 1981; **Hishinuma et al**, 1981; **Lee & Mansur**, 1985; **Takahashi et al**, 1987; **Ashworth**, 1989; **Muroga et al**, 1990.

expected importance of surface diffusion (e.g., **Schüle & Kornmann**, 1980) and segregation (e.g., **Okamoto et al**, 1982) mechanisms. Whilst even this degree of segregation alone is still not sufficient to explain the large decreases in partial separations, it may be possible that dislocations, which have already narrowed on heating and which would normally partially redissociate on cooling (e.g., **Latanison & Ruff**, 1971), have been pinned by enhanced segregation.

Finally, non-equilibrium, thermal (vacancy) segregation should also be mentioned: Given the possibilities that surfaces and dislocations may be particularly important as both vacancy sources and sinks during warming, and that surface segregation and diffusion mechanisms may be dominant, the possibility of a very localized 10-15% change in nickel composition at dislocations cannot be discounted. Such an effect may be accentuated at dislocations, if segregation may proceed via pipe diffusion, by a substantially lower vacancy formation energy near the core if chromium may diffuse as a bound vacancy complex (perhaps explaining observations by **James & Shepherd**, 1989, and **Dumbill et al**, 1990, of chromium *enrichment* at grain boundaries) under these conditions. The vast majority of experimental data suggest, however, that chromium diffuses principally by vacancy exchange (the ‘inverse Kirkendall effect’, see **Marwick**, 1978) and thus nickel *depletion* would be expected if dislocations are acting as vacancy sources. A future experiment will investigate possible thermal and radiation-induced segregation effects on partial separations (see chapter 9).

The suggestion described in chapter 7 (see ‘*Interpretation*’ section of figure 7.10) that the degree of narrowing seems to be relatively, or completely, independent of dose, may suggest that its origins are, in fact, thermal; this has not yet been directly confirmed since weak-beam micrographs with sufficiently good resolution such that individual partials are resolved were not taken after warming to the irradiation temperature in the HVEM but prior to irradiation.

In summary, therefore, it does not appear to be possible to differentiate absolutely between the above possibilities. For example, the observation in figure 7.10 that the 40° dislocation section is apparently more widely dissociated than the 60° dislocation section may be explained by the lack of vacancy clusters, the lower interaction energy for the nearer screw section with radiation induced bulk super-saturations or the higher defect formation energy along this section if matter transport occurs along dislocation cores (which connect surfaces).

8.2.3 *Vacancy and Interstitial Pipe Diffusion:*

Evidence presented within the ‘*Summary*’ section of chapter 5 is consistent with pipe diffusion, in general, being particularly efficient along dislocations in the 40%Ni alloy. We examine, however, any

relative effects as a function of nickel content here; particularly with regard to vacancy pipe diffusion.

In the 15%Ni ternary alloy, the small clusters, presumed to be vacancy in nature (§8.1.1), were found to lie very close to the stacking fault; too close to permit a direct determination of whether they lie within the dilatational or compressional strain field of the dislocation. After a dose of 0.07 dpa (figure 3.3) these clusters are very great in number and extremely small ($\lesssim 1$ nm.). This is consistent with a rather low vacancy mobility along dislocations in this material.

As the nickel content is increased to 25%, however (chapter 4), vacancy clusters apparently lie at greater distances (~ 15 -20 nm.)³⁹ from the post-irradiation line positions. Whilst doses are higher (typically ~ 0.1 dpa) here, most vacancy clusters are several nanometres in size and easily recognizable as SFT (e.g. figure 4.4). The number density of clusters also appears to be less than for the 15%Ni ternary alloy.

In the 40%Ni alloy (chapter 5), the relatively few observations of irradiated dislocations does not allow a ‘typical’ value for the displacement of vacancy clusters from the line to be given; the only ‘reliable’⁴⁰ value being that of ~ 25 nm. for figure 5.3. Notice, however, that for figure 5.3, along which dislocation pipe diffusion is thought to occur, the number density of vacancy clusters is substantially less. Cluster ‘G’, at ~ 40 nm., is also by far the largest vacancy cluster seen after any irradiation. It is uncertain, however, how many vacancies are contained within this cluster since only one of its dimensions is known. Apparently, small vacancy clusters have not nucleated close to this dislocation.

One potential source of greater displacement of vacancy cluster damage from the line is if interstitial climb has occurred. This appears to be the case in figure 5.4, however, in all of the other figures, which show glissile, dissociated dislocations, such motion of the line is shown to be unlikely. It is thought, instead, that the increase in the separation of clusters from the line may indicate a change in the effective dislocation bias. This may, in turn, be linked to a change in the vacancy dislocation diffusion coefficient.

As outlined above, it would appear that the diffusion of vacancies along 60° dislocations in the 15%Ni ternary alloy is considerably less efficient than along 60° dislocations in either the 25%Ni or,

³⁹ Figures 4.2, 4.3, (4.5), 4.6 and see ‘Summary’ of chapter 4.

⁴⁰ All of these values are subject to measurement errors, and errors incurred through the likely non-identical displacements of the weak-beam images from the dislocation core under different diffraction conditions and from a possible ‘random’ element (Cockayne, 1970) as outlined in text relating to figure 4.3. These values for the apparent separation of vacancy clusters from dislocations are shown for comparison only and, therefore, should not be taken as absolute.

particularly, the 40%Ni ternary alloys (both interstitials and vacancies are highly mobile in the bulk for all materials studied; see chapter 1).

Whilst resistivity measurements have shown vacancy migration enthalpy in the bulk to be relatively insensitive to composition⁴¹ in the range 20-45%Ni (Dimitrov et al, 1987, 1988), it is possible that the same is not true of diffusion along dislocations since the narrower partial separations may have a significant effect on the core structure.

Experimental determinations of core diffusion parameters are scarce; the majority of values currently available are extrapolated from so-called 'tail' studies (e.g., Chen et al, 1989) of grain boundary diffusion (Atkinson & LeClaire, 1984), the interpretation of which has proved to be complex (LeClaire & Rabinovitch, 1984). In his review of a total of eight experiments, up to 1970, aimed at the determination of dislocation diffusion coefficients in Ag, Ni, Al and Cu, Balluffi (1970) attempts to correlate the six different experimental methods employed. He describes the data as "*sparse and imprecise*" but at one point is able to conclude:

"There is reason to believe that the diffusivity along dissociated dislocations with total Burgers vectors $\tilde{b} = \frac{a}{2} \langle 110 \rangle$ decreases as the degree of dissociation into partial dislocations increases."

Balluffi (1970) attributes this effect to decreased core distortion and 'structural loosening'⁴² as the dislocations relax by dissociation. This view has since neither been confirmed nor denied; appearing, in fact, to have been largely ignored, although Collins (1987), in studying point defect trapping at dislocations in Al, Ni, Cu and Au, also hypothesized that such an effect may exist.

If dislocation pipe diffusion is indeed becoming more efficient here, interstitial defects might also be expected to diffuse more easily. In addition, the activation enthalpy for interstitial migration, determined by bulk resistivity studies (Dimitrov and Dimitrov, 1988), decreases by nearly a factor of 2 over the range 20-75%Ni.

As outlined in chapter 4 (diagram 4.4), it is believed that defect clusters formed on 60° dislocation sections result from the preferential drift of core diffused defects towards these regions. It is thought that a super-saturation threshold is reached, above which a defect cluster may precipitate, before back-diffusion becomes significant. Interstitials appear to be migrating to dislocations in the 40%Ni ternary alloy. Apparently, these interstitials are not giving rise to visible clusters, or climb, and

⁴¹Interstitial mobility increases substantially with increasing nickel content.

⁴²Nabarro F R N, 1948, "Internal Stresses in Metals and Alloys", Institute of Metals, London, p.237.

pipe diffusion is implicated. If pipe diffusion is particularly easy, or loop nucleation particularly difficult, along dislocations in this alloy, it may be possible that the super-saturation threshold for loop nucleation near the dislocation is not being exceeded. Therefore, back-diffusion may account for the lack of an interstitial cluster near vacancy cluster 'G' (see figure 5.3). The only 'pipe' diffusion data for the same alloying system over a range of partial separations available is that noted in an appendix by Rothman et al (1980), who describe extremely approximate derivations of the grain boundary diffusion coefficient and, possibly wisely, draw no conclusions from them. It is interesting to note, however, that the values given show an order of magnitude increase in the grain boundary nickel self-diffusion coefficient from Fe-15%Cr-20%Ni to Fe-15%Cr-45%Ni; apparently consistent with the above hypothesis⁴³. In the absence of more precise data for any alloying system, however, an increase in the efficiency of pipe diffusion with decreasing partial separation must remain somewhat speculative at this time.

8.2.4 Dislocation Bias:

One possible explanation for the absence of vacancy clusters very close to the dislocation of figure 5.3 is that the vacancy super-saturation is too low for cluster nucleation. In other words, the dislocation is acting as a good sink for vacancies, which vacancies may undergo subsequent core diffusion. Whilst absolute values for interstitial and vacancy sink strengths may not be determined directly from these experiments, since the number of defects absorbed at dislocations is unknown, a low vacancy super-saturation near dislocations in the 40%Ni ternary alloy may signal an effective change in the dislocation bias. A later section (§8.4.1) will consider the possibility of a dependence of the dislocation bias on nickel content.

Another possible origin for the lack of visible vacancy clusters near most of the dislocation of figure 5.3 may involve recombination. It would seem plausible that recombination should be enhanced for the high nickel alloy if pipe diffusion of both defect species is occurring, since a reduction in the 'radius' of the 'pipe' will decrease the average separation of interstitial and vacancy fluxes. However, this does not explain changes in the separation of clusters and line as nickel content is altered.

Recombination may be implicated, however, by the formation of a small population of vacancy clusters to the left-hand side of figure 5.3, micrograph (h). These clusters are believed to result from the action of the nearby surface as a dominant sink for the more mobile interstitials. Clearly vacancy core diffusion is not so efficient that cluster formation is entirely inhibited. Hence, the lack of vacancy

⁴³*The defect formation energies must be considered here.*

clusters parallel to the sections of the dislocation which lie towards the centre of the foil, where the surfaces are not dominant sinks, may indicate that recombination is also important.

8.2.5 Favourable Crystallographic Directions for Irradiated Dislocations:

In chapter 3, it was hypothesized that particular directions may be favourable because of a reduction in dislocation core energy. As mentioned in the 'Interpretation' section of figure 3.5, calculations employing both isotropic (Bullough et al, 1964), and anisotropic (Bacon et al, 1970), elasticity have suggested that core energy terms may be of considerable relevance. Segall & Shoaib (1970) similarly believe core energy terms to be important in the evolution of segmented helices in aluminium but do not expand sufficiently upon their analysis of the micrographs to enable a particular direction(s) to be determined.

In figure 3.4, it is noted that the 'flat-helical' configuration appears to consist solely of segments aligned along $\langle 123 \rangle$ and $\langle 110 \rangle$ directions. A low dose irradiation (figure 3.5) also found dislocation segments with line directions consistent with $\langle 123 \rangle$ orientations. In the 25%Ni ternary alloy, dislocations are also seen to lie along apparently favourable directions, including $\langle 123 \rangle$. This is believed to aid in the formation of discrete lines of clusters lying parallel to the pre-irradiation position of dislocations (see diagram 4.3). As distinct from the 15%Ni ternary alloy, a dislocation segment apparently lying along $\langle 112 \rangle$ is also seen. In the 40%Ni alloy, the existence of favourable crystallographic directions is not particularly in evidence; it may be noted, however, that sections of the near-screw dislocation of figure 5.4, if projected onto a plane δ , would lie along $\langle 112 \rangle$ and $\langle 110 \rangle$ directions.

If these crystallographic directions are indeed favourable as a result of core energy terms, the absence of dislocation segments lying along $\langle 123 \rangle$ directions in the 40%Ni alloy may indicate a different core structure. The experimental data is obviously very sparse here and we draw no conclusions from this, speculating merely that information about dislocation core structures might be gleaned from these directions.

Whilst the quaternary alloys are considered below, we note here that dislocations in these alloys do ^{not} appear to show a preference to lie along particular directions. It seems likely that this is also a result of core modification, owing to the presence of segregated solutes (see Hirth & Lothe, p. 512).

8.3 : Quaternary Alloys

8.3.1 The Effects of Silicon Addition:

In chapter 6 ('Points of Discussion') it was argued that in order for new jogs to form along

dislocations in this 15%Ni+Si alloy, an observation not shared with the ternary alloys, a super-saturation of interstitials must develop. In the ternary alloys, appreciable interstitial super-saturation levels are only expected near screw, and near-screw, dislocations along which pipe diffusion has been inhibited by the presence, and survival, of pre-existing jogs. Similarly, barriers to pipe diffusion must exist for this quaternary alloy. However, the absence of (sufficient numbers of) pre-existing jogs along the screw, and near-screw, dislocations of figures 6.1 and 6.2 requires a new origin for these barriers. In addition, pre-existing jogs along mixed dislocations are expected to annihilate (figure 3.7) and are therefore unlikely to act as lasting barriers to core diffusion.

8.3.1.1 Silicon Segregation and a Possible Origin of Barriers to Core Diffusion:

Detailed reviews of observations of R.I.S. of silicon, first seen by **Okamoto & Wiedersich** (1974, 1975)⁴⁴, are provided by **Fisher et al** (1980) and **Ashworth** (1989). The important result from these studies in terms of the present work is that silicon has always been seen to migrate towards defect sinks (e.g., **Piller & Marwick**, 1977; **Nakata & Masaoka**, 1987; **Ashworth**, 1989; **Perks et al**, 1989) under a variety of irradiation conditions. Under HVEM irradiation conditions, it has been shown that significant silicon segregation may occur at low doses in NiSi alloys (**Muroga et al**, 1983; **Bartels et al**, 1988) whilst resistivity measurements indicate that a Si-interstitial complex may be as mobile as a single interstitial in Fe-16%Cr-20%Ni to 45%Ni alloys (**Dimitrov & Dimitrov**, 1984). Under heavy-ion irradiation, considerable segregation of silicon to surfaces has been found after doses as low as 0.05 dpa (**Rehn et al**, 1979). The mechanism of silicon segregation appears to be the subject of some conjecture, however, and hence a concise review of some relevant papers is given in the following section (§8.3.1.2).

As shown above, it is likely that silicon is segregating towards dislocations, which are acting as sinks for point-defects. This will relax the strain field of the dislocation⁴⁵. It should be noted that, as shown in figures 18-4 and 18-9 of **Hirth & Lothe**, little bulk segregation is necessary, since solute atoms need migrate only a short distance from one side of the dislocation to the other to cause significant relaxation of the core.

As detailed in section 8.2.3, core diffusion appears to be a less efficient process in the 15%Ni ternary alloy than in the higher nickel alloys; this is believed to arise from a greater relaxation of the dislocation strain field by reason of the large partial separation. Hence, further relaxation of the

⁴⁴Possibly also seen by **Leitnacker et al**, 1973.

⁴⁵Silicon is likely to be undersize in this 15%Ni+Si alloy (see §8.3.1.2) and, therefore, is expected to segregate towards the compressional stress field of dislocations (**Hirth & Lothe** show the case for oversize solutes in their figures 14-13, 18-4 and 18-9).

dislocation strain field by the redistribution of solute atoms, may inhibit pipe diffusion sufficiently to allow the build-up of an interstitial super-saturation and lead to double-jog nucleation. As detailed in section 8.1.4, the climb mechanism of screw, and near-screw dislocations is uncertain. Objections to the criteria of **Grilhé et al** (1977) are removed, however, for mixed and near-edge dislocations and, as outlined in chapter 6 (*'Points of Discussion'*) it seems likely that the **Thomson & Balluffi** (1962), or similar, mechanism is operating here and that dislocations are acting as effective recombination centres.

8.3.1.2 The Mechanism of Silicon Transport:

Silicon has been found to be undersized in nickel (**King**, 1966). It is also undersized in 316 (**Straalsund & Bates**, 1974), 12%Cr-15%Ni (**Mazey et al**, 1980)⁴⁶ and 16%Cr-25%Ni steels (**Dyson & Holmes**, 1970) which is consistent with the widely held belief that silicon migrates via a bound interstitial complex (e.g., **Piller & Marwick**, 1978; **Sethi & Okamoto**, 1981; **Averback et al**, 1983; **Garner & Wolfer**⁴⁷, 1984). It appears likely, therefore, that silicon is also undersize in the Fe-17%Cr-15%Ni-1%Si alloy studied and migrates via an interstitial mechanism. Many authors, however, have considered the possibility that silicon forms bound vacancy complexes⁴⁸ (e.g., **Fisher et al**, 1980 [Binding Energy 0.35eV]; **Gupta**, 1980 [B.E. 0.73eV]) and may thus migrate via a vacancy mechanism (e.g., **Macht et al**, 1987; **Nakata & Masaoka**, 1987; **Ashworth**, 1989; **Perks et al**, 1989), although **Dimitrov & Dimitrov** (1984) find a very low (≥ 0.04 eV) silicon-vacancy binding energy. As mentioned by **Macht et al** and **Ashworth**, observations that the thermal diffusivity of silicon in nickel is considerably more efficient than self-diffusion (see **Macht et al** and **Ashworth** for references) is consistent with a vacancy mechanism. This possibility is further strengthened by research in progress at Harwell which indicates that additions of silicon may sometimes *increase* the lattice parameters of Fe-19%Cr-40%Ni steels (**C M Shepherd** - private communication). **Dyson & Holmes** (1970) report that silicon is slightly oversize in an Fe-18%Cr-10%Ni steel. Reports of large deviations from ideal behaviour for Si in 316 stainless steel, Fe, Cu and Ni (**Straalsund & Bates**, 1974; **King**, 1966) suggest different bonding from the pure state for silicon in substitutional solution.

⁴⁶ The lattice parameter of alloy 3 of **Mazey et al**, 1980, should be 0.35841 nm. (**C M Shepherd** - private communication).

⁴⁷ The paper of **Garner & Wolfer** (1984) describes silicon as "smaller in Fe-Ni-Cr alloys" and supplies the following reference: **Bates J F**, 1980; "Partial Molar Volumes and Size Factor Data for the Fe-Ni-Cr-Si System"; HEDL-SA-2310.

⁴⁸ Work in progress at Kyushu University (**Watanabe et al**, 1989) indicates that phosphorous, which has been shown to migrate via a bound interstitial complex (**Perks et al**, 1989; **Murphy & Perks**, 1990), may also form bound vacancy complexes.

8.3.1.3 Core and Bulk Diffusion in the 15%Ni+Si alloy:

In all cases, except for the dislocation of figure 6.4, core diffusion appears likely to be inefficient in this material. In chapter 6 (*'Points of Discussion'*) it was argued that the presence of copious vacancy clusters along the evenly dissociated and unclimbed dislocation of figure 6.4, including a particularly large cluster ('B') where the line passes through 60° orientation, is indicative of the efficient core diffusion of interstitials. Less efficient core diffusion of vacancies is also implicated. It is not understood why this dislocation should have behaved differently from others irradiated at the same time, although it may be related to the presence of a particularly large super-jog close to the node prior to irradiation. However, the occurrence of vacancy core diffusion, and the possibility of *particularly* efficient interstitial core diffusion (see section 8.2.3), may be contrasted with the 15%Ni ternary alloy. In addition, the lack of interstitial clusters in the bulk, seen to varying degrees after irradiation in all the other materials, apparently suggests a very high interstitial mobility.

Rothman et al (1980) found a 40% increase in bulk diffusivity, although the experimental errors quoted are large, on adding silicon to their Fe-Ni-Cr ternary alloys under conditions of high vacancy super-saturations. Since no effect was seen, within (a large) experimental error, on the *activation enthalpies* for bulk diffusion they assumed that the principal origin for the increased diffusivity lay in increases in the pre-exponential coefficient (D_0 , see **Manning**, 1968, p.29). An increase in D_0 was also proposed by **Fisher et al** (1980). Similarly **Dimitrov & Dimitrov** (1982) find no effect on the activation enthalpies for interstitial migration on the addition of trace amounts of silicon or titanium to their ternary alloys (The same authors (**Dimitrov & Dimitrov**, 1984) later report a slight *decrease* in vacancy mobility for the same additions, as was found by **Fisher et al** (1980)).

As noted in chapter 2, an error of $\pm 50^\circ\text{C}$ is plausible for the Argonne heating stage. Whilst this *may* offer an explanation for increased defect mobilities, this is plainly not the case for the 40%Ni ternary alloy since copious interstitial loop nucleation is seen in the bulk whilst core diffusion is apparently very efficient (see §8.2.3). It seems more likely, therefore, that the origin of the high interstitial mobility in this alloy is, indeed, an increase in D_0 . This is consistent with observations by **Bartels et al** (1988) which indicate that only 2 to 10 Frenkel defects are required to segregate a silicon atom at 260°C.

8.3.2 The Effects of Molybdenum Addition:

At the start of chapter 7, it is noted that behaviour exhibited by dislocations in the 15%Ni+2%Mo quaternary alloy is substantively similar to that shown by dislocations in the

ternary alloys. A screw dislocation is seen to form a zig-zag configuration, parts of which may lie out of the plane of the foil (figure 7.4), whilst interstitials apparently pipe diffuse along edge and mixed dislocations, allowing the formation of vacancy cluster damage nearby (figures 7.1, 7.2, 7.3, 7.4, 7.7, 7.8 and 7.10). Also in common with previous observations, a sessile dislocation (figure 7.11) is seen to have climbed by the absorption of interstitials, which is consistent with the pipe diffusion of interstitials being inefficient along these dislocations.

8.3.2.1 *'Equilibrium' Solute Segregation:*

Dislocations in this 15%Ni+Mo alloy are extremely complex in appearance before irradiation. Glissile dislocations in the plane of the foil are seen to be very heavily cusped (e.g., figures 7.1, 7.2 and 7.8), sometimes to the point where it does not prove possible to determine the pre-irradiation configuration (figure 7.7). The possibility that many of the pre-existing cusps arise not from jogs, as commonly assumed throughout the rest of this thesis, but from solute pinning is suggested in the *'Interpretation'* section of figure 7.4. This arises from the observation that most pre-existing cusps along this screw dislocation have not given rise to zig-zags, as would be expected were they to be jogs (see figure 3.7).

As described in chapter 2, dislocations were rarely introduced by cold work, it having been found that the dislocation density in the as received specimens was ideal for this study. The single crystals supplied by Metals, Crystals & Oxides Ltd. had been grown from the melt, leading to high concentrations of vacancies, and slowly cooled. Recently **James & Shepherd** (1989) and **Dumbill et al** (1990), employing STEM microanalysis with a probe size $\sim 3\text{ nm.}$, both report incidences of Mo enrichment at grain boundaries in 316 and PE16 steels during aging. On cooling the effect is very marked. **Dumbill et al** observe a *"synergism between the segregation of boron and molybdenum"* and believe segregation on cooling to be proceeding, in this case, via a mechanism which may involve a bound boron-molybdenum-vacancy complex. They point towards atom probe data (their reference 16) which shows the existence of a variety of other bound vacancy complexes. In addition, **James & Shepherd** note that segregation to grain boundaries, *"particularly of molybdenum"*, is accentuated for a low carbon content and slow cooling.

Hence, the presence of significant quantities of molybdenum at dislocations in the 15%Ni+Mo alloy prior to irradiation would appear likely, both from the point of view of the above experiments and the observed complexity of pre-irradiation configurations in this study.

8.3.2.2 Non-Equilibrium Solute De-Segregation (R.I.S):

Dislocations are seen to be 'smoother' in appearance after all irradiations performed in this material. This is expected for mixed and edge dislocations in the ternary alloys, where most, or all, pre-existing cusps and constrictions are thought to correspond to jogs (**Carter & Ray**, 1974), however, if cusps in the 15%Ni+Mo alloy result from solute pinning then solute de-segregation must be proceeding.

Unusually, there appears to be a general consensus on the mechanism of R.I.S. of molybdenum in Fe-Ni-Cr alloys. This arises principally from the fact that Mo is substantially oversized in Fe-Ni-Cr alloys (e.g., **Straalsund & Bates**, 1974; **Dyson & Holmes**, 1970), with values for the misfit parameter (Ω_{sf}) as high as +35.9% (S & B⁴⁹). Under irradiation, molybdenum has also always⁵⁰ been observed to migrate in the opposite direction to a vacancy flux (the inverse Kirkendall effect, **Marwick** 1978), which is consistent with its high diffusivity (**Kucera & Stransky**, 1982; **Alberry & Haworth**, 1974).

Observations of vacancy cluster nucleation close to dislocations (copious in the case of figure 7.11) show that dislocations are acting as sinks for vacancies. It is probable, therefore, that any molybdenum which had segregated previously to the dislocation will now be depleted.

8.3.2.3 Pipe Diffusion in the 15%Ni+Mo alloy:

Observations of vacancy clusters forming close to the now evenly dissociated dislocations suggest that the pipe diffusion of interstitials is occurring in this alloy. Pipe diffusion of vacancies may also be occurring although there is no direct evidence of this. However, the work of **Dumbill et al** (1990), in which efficient grain boundary diffusion of molybdenum is seen in PE16 during aging, raises the possibility that molybdenum is also core diffusing in the 15%Ni+Mo alloy studied here.

The core diffusion of molybdenum may provide an explanation for some, or all, of the shallowly inclined features seen where dislocations tend away from 60° orientation (loops 'C' of figure 7.7 and feature 'A' of figure 7.8). As outlined in chapter 7 ('Points of Discussion'), these features are thought to result from core diffusion but are otherwise of uncertain origin. In diagram 7.2, loops 'C' are shown to consist of Burgers vectors lying in a plane δ ; it is pointed out that if these features lie entirely in the plane of the foil they might be expected to contract by glide, since point defects are not accommodated, and react to form one dislocation $\tilde{b}_T = \vec{AB}$. As explained in text relating to figure 7.7, the extreme complexity of loops 'C' unfortunately does not allow their precise configurations to be determined; the suggestion is made, however, that if they are inclined, they are inclined in a sense

⁴⁹This compares with the value of $\Omega_{sf} = -2.9\%$ quoted by the same authors for silicon.

⁵⁰**Nakata & Masaoka** (1987) and **Ashworth** (1989), amongst others, provide observations of, and references to, the depletion of molybdenum at vacancy sinks (first seen by **Sethi & Okamoto**, 1981).

consistent with a vacancy defect.

In the '*Points of Discussion*' section of chapter 7, the suggestion is made, however, that these defects are related to interstitial fluxes. It is possible that an explanation may be found by considering that molybdenum is expected to migrate in the opposite sense to a vacancy flux (§8.3.2.2 above) under irradiation. This may also be true of molybdenum segregation along irradiated dislocations. Notice also, first, that by the drift mechanism of figure 4.4, vacancies and interstitials are expected to migrate in the same direction along dislocations, i.e. *towards* regions in the 60° orientation, and second, that if features 'C' are inclined in a sense consistent with a vacancy defect, they extend from the dislocation *in the direction from which a bulk diffusing vacancy is likely to have come*.

Hence, we arrive at the highly speculative explanation that these defects, and possibly others which lie where a dislocation tends away from 60°, consist principally of molybdenum rich regions, which, by reason of the large lattice strains thereby introduced, yield complex contrast. These regions are perhaps in the form of platelets bounded by pinned dislocations and may arise from the operation of the inverse Kirkendall effect along both the dislocation core and in the bulk.

In the absence of greater experimental evidence⁵¹ to support this hypothesis, which does not immediately explain every feature seen (if feature 'A' of figure 7.8, which apparently extends into the compressional strain field, has a similar origin a new hypothesis must be developed) extreme caution is advised. It may be noted, however, that similar precipitation of molybdenum rich regions may explain much of the loop nucleation observed near dislocations in this alloy, the Burgers vectors of which apparently depend principally on the orientation of the parent dislocation (see '*Interpretation*', figures 7.5 & 7.6), employing criteria suggested by the work of Cahn (1957).

8.3.2.4 Recombination and Climb:

It is noted in the '*Interpretation*' section of chapter 7 that the distribution of vacancy clusters close to dislocations is consistent with the action of complex configurations as recombination centres. The situation may be analogous to that of the 15%Ni+Si alloy, in which barriers to core diffusion are thought to lead to the build-up of an interstitial super-saturation. In the 15%Ni+Si alloy, these barriers are believed to arise initially from Si rich regions, which then lead to jog nucleation, probably via the Thomson & Balluffi (1962) mechanism. It seems likely, therefore, that barriers to core diffusion in the 15%Ni+Mo alloy similarly arise from molybdenum rich regions. Hence the observation that vacancy cluster formation is significantly suppressed near complex configurations, such as features 'C' of figure

⁵¹A future experiment may shed more light on these features; see chapter 9.

7.7, may provide further evidence for the Mo segregation proposed above.

In the 15%Ni+Mo alloy, however, the action of these barriers appears rarely to have led to significant climb of the line. This suggests that the super-saturation levels reached along dislocations in the 15%Ni+Si alloy are not being attained here. Whilst this may suggest a change in the bias term, such that dislocations are less important as defect sinks in the 15%Ni+Mo alloy, other possible origins for such an effect are very great in number.

8.4 : Further Notes on Void Swelling

8.4.1 Nickel Dependent Dislocation Bias:

One factor which is not contained in the list of table 1.1, but is mentioned in section 8.2.4, is the possibility that the dislocation bias term is reduced for a higher nickel content. We review two similar suggestions, and corresponding explanations, made in the literature.

Lee & Mansur (1985) suggest a possible change in the dislocation bias term to account for their observations that the minimum critical cavity size for rapid growth increases by approximately a factor of 10, to ~ 5 nm., on raising nickel content of an ion irradiated Fe-15%Cr-xNi ternary alloy from 15% to 35%. Their suggestion arises from calculations performed by the authors, based on a theoretical treatment of parameters governing the minimum critical cavity size (reviewed by **Mansur et al**, 1986), which indicate that only the bias term may lead to an effect of this magnitude. It is uncertain from the text, however, whether the possible ramifications of increased interstitial mobility (**Dimitrov & Dimitrov**, 1988) were considered. **Lee & Mansur** (1985) hypothesize that the decrease in the bias term for higher nickel levels may be due to a fundamentally different mechanism of interaction between irradiation produced point defects in the low and intermediate Ni content alloys studied. Finally, microstructural evidence is presented which they argue is consistent with a low value for the bias term for their Fe-15%Cr-35%Ni alloy; basically this centres around a low interstitial background loop growth rate⁵². It may be noted here that this apparently contradicts observations in the 40%Ni alloy (chapter 5) in which background loops apparently grow quickly, although, as explained originally in chapter 3 (*'Points of Discussion'*), there are many difficulties in determining relative background loop growth rates from these experiments. The author is not aware of any other studies of detailed microstructural evolution as a function of dose in low and high swelling materials.

Garner & Wolfer (1984) review earlier treatments aimed at modelling the swelling behaviour of

⁵²The route to a reduction in bias must involve a supposed attraction of vacancies by an interstitial super-saturation to explain differences in the relative arrival rates of interstitial and vacancy defects.

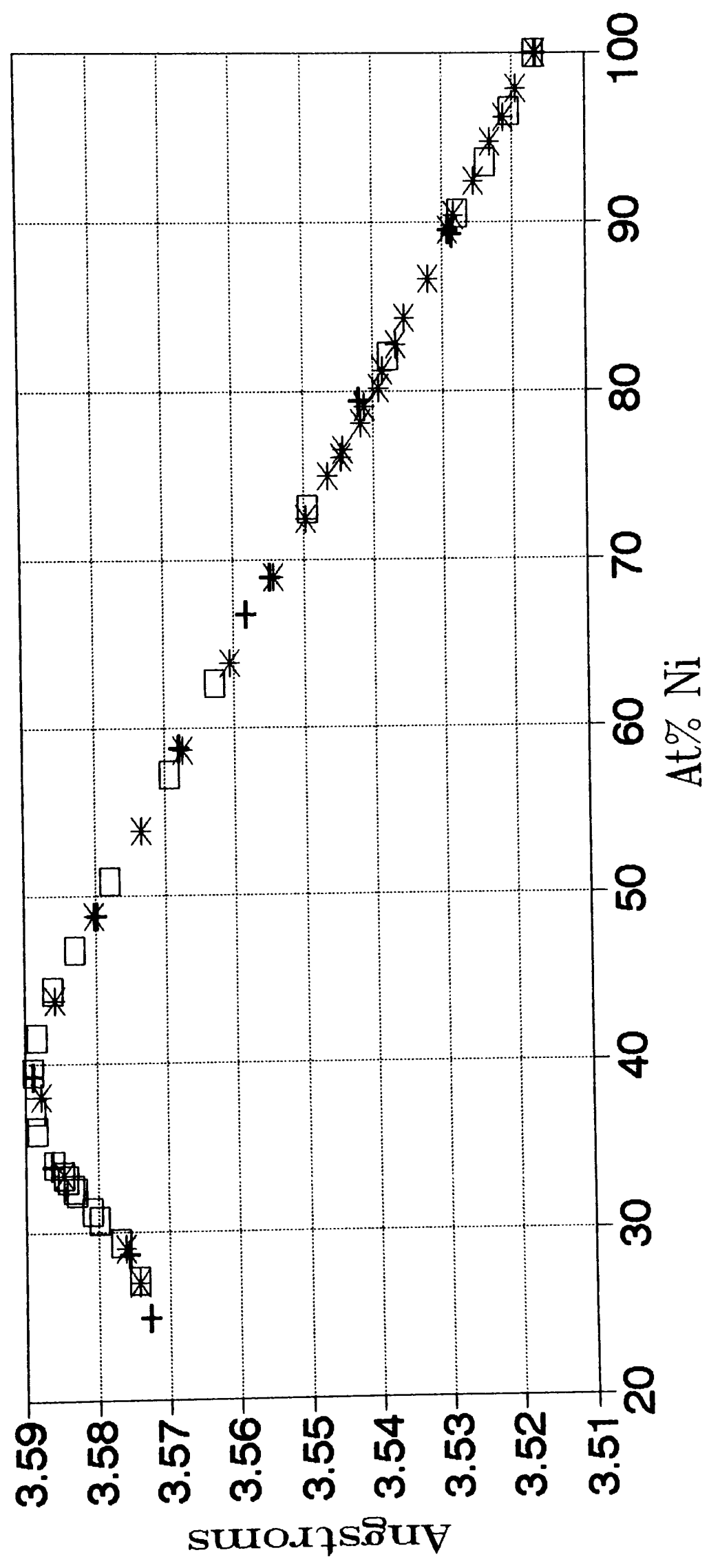
Figure 8.2

Fe-Ni Binary Alloy Lattice Parameters after Pearson (1958)

Showing maximum in lattice parameter at $\sim 35\%Ni$. Regions with this composition in the matrix seem likely to attract vacancies.

Figure 8.2

Fe-Ni Binary Alloy (FCC Phase)
Lattice Parameters



□ Owen & Sully, 1937 + Jette & Foote, 1936 * Bradley et al, 1937

Fe-Ni-Cr alloys. Initially the paper reviews the ‘coated void’⁵³ approach of **Wolfer** (1983) in which published diffusion and thermodynamic data for the *binary Fe-Ni* system was employed to predict that vacancies will experience a reversal in the sign of the Kirkendall effect at nickel levels $\sim 35\%$; below this level vacancies are expected to migrate towards regions of higher nickel concentration whilst the opposite occurs for nickel concentrations higher than $\sim 35\%$. Such an effect, seemingly consistent with elasticity considerations (see figure 8.2), would lead to a minimum in swelling *rate* at 35%Ni but appears unlikely to explain a prolonged incubation period.

Garner & Wolfer (1984) also note that an increase in vacancy diffusivity will inhibit void nucleation. Their approach centres on an imbalance in the diffusivities of different alloying elements, and a general increase in diffusivity (100% is quoted) between high (20%Ni) and low (45%Ni) swelling alloys, both of which results have been inferred from absolute values shown in table 3 of **Rothman et al** (1980). One must, however, be wary of accepting these values in view not only of the substantial experimental errors quoted by, but also of the following two statements contained within the paper of Rothman et al: “*No qualitative difference is observed between diffusion behaviour of high and low-swelling alloys*” and “*There appears to be no correlation between swelling and the diffusion behaviour of the major (alloying) components*”. As is pointed out by **Muroga et al** (1990), no explanation is offered by this theory for the minimum in swelling at intermediate nickel concentrations.

Hoyt & Garner (1990a) also employ the binary Fe-Ni system in calculations which suggest a dependence of the bias term on nickel content for pre-existing *line* dislocations. Here the initial bias of an edge dislocation as a function of nickel content has been computed. This was accomplished employing the **Larché & Cahn** (e.g., 1985) treatment of stress relaxation due to a solute atmosphere and assuming a linear dependence of the lattice parameter on nickel concentration (Vegard’s Law⁵⁴), By this assumption Ni is always oversize in the matrix (see **Pearson**, 1958); it is undersize in steels (**Dyson & Holmes**, 1970; **Straalsund & Bates**, 1974 and see figure 8.3). **Hoyt & Garner** (1990a) then relate their calculations, via a factor of 2 increase in vacancy bulk-diffusion coefficient (also attributed to **Rothman et al** (1980)), to the minimum critical size cavity concept to explain the intermediate nickel level reduction in swelling for Fe-15%Cr-xNi alloys seen by **Johnston et al** (1974).

⁵³*The enrichment of nickel, and other undersized elements which diffuse with a defect flux, at voids is a common observation (e.g., Hishinuma et al, 1981; Muroga et al, 1988a, 1990).*

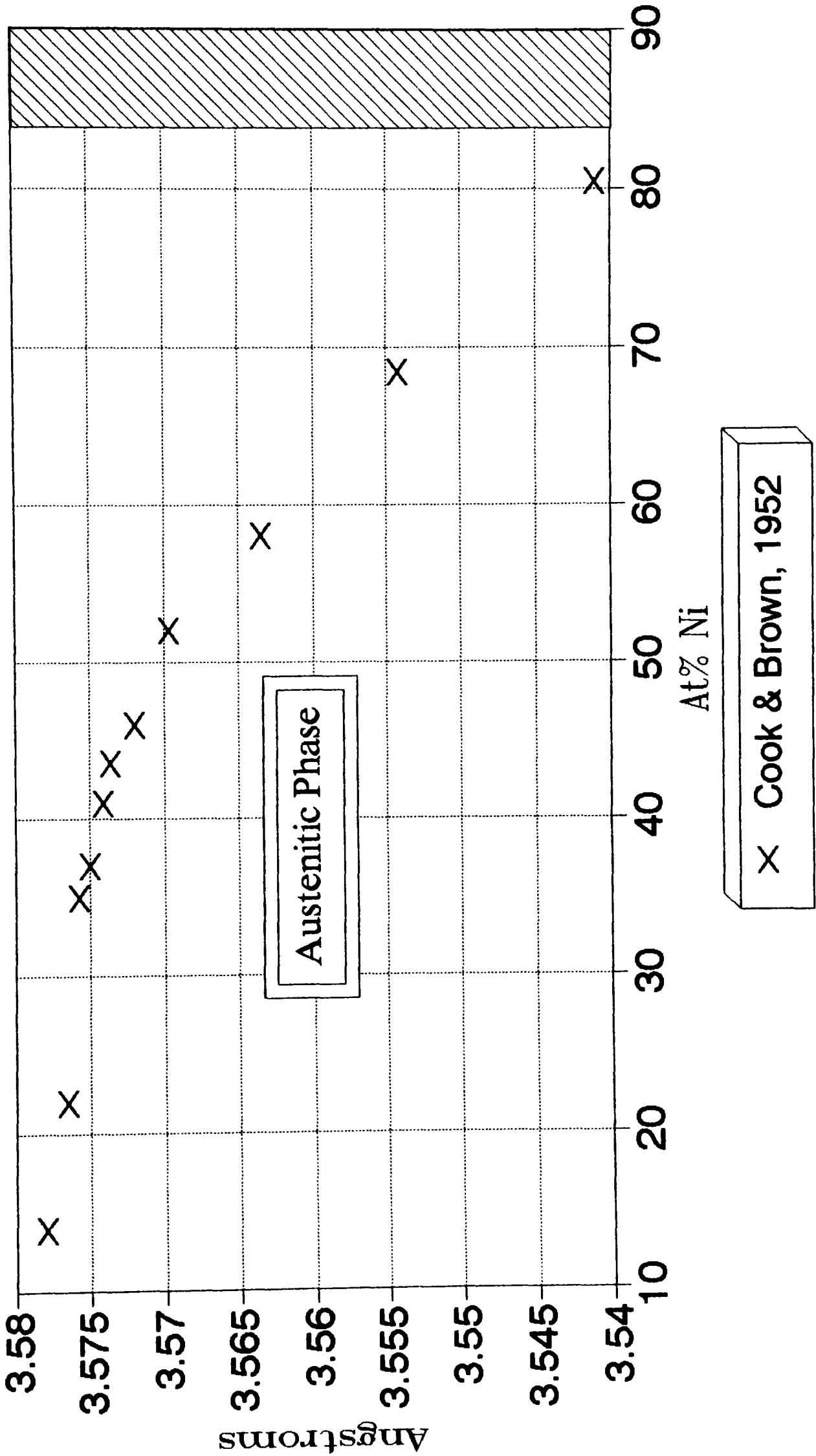
⁵⁴**Vegard** (1921, 1928). *This is a poor approximation for the binary FeNi system (see figure 8.2) although this would not substantially have altered the results of Hoyt & Garner. Hume-Rothery and co-workers (1948, 1962 - see Dyson & Holmes, 1970, for references) have shown that Vegard’s law is seldom obeyed in real systems.*

Figure 8.3

**Fe-17%Cr-xNi Ternary Alloy Lattice Parameters
after Cook & Brown (1952)**

Figure 8.3

Fe-17%Cr-xNi Ternary Alloy
Lattice Parameters



It would seem likely, from elasticity considerations, that the segregation of an oversize solute, such as nickel in the binary Fe-Ni system, to the dilatational side of a dislocation would tend to reduce the bias term. **Norris** (1975) also deals with the effects of the segregation of *oversize* solutes to dislocations and similarly predict a reduction in the dislocation bias.

In austenitic Fe-Ni-Cr alloys, however, nickel is always undersize (shown in figure 8.3 for Fe-17%Cr-xNi and see **Dimitrov & Dimitrov**, 1988) and the validity of calculations performed on the binary Fe-Ni system as models for these alloys is not immediately obvious. As hypothesized most recently by **Shepherd** (1990), the segregation of undersized nickel to dislocations (seen directly by Shepherd) might be expected to increase the dislocation bias. A paper more recently submitted for publication (**Hoyt & Garner**, 1990b), which (from the abstract) considers the Fe-Ni-Cr system directly, may shed light on this matter.

8.4.2 *Relevance of Observations Made in This Study, Summary & Further Speculation:*

In all the materials studied it is thought that significant numbers of interstitials have migrated to dislocations. Indirect evidence of this is the precipitation of vacancy cluster damage. We have argued that pipe diffusion is an important process in these materials. **Balluffi & Grenato** (1979) note that pipe diffusion is expected to proceed with an activation energy ~ 0.7 that of bulk diffusion. At all irradiation temperatures employed, bulk diffusing interstitials are extremely mobile. Hence, no significant interstitial super-saturation is expected to develop close to dislocations along which interstitials are pipe diffusing. It follows, therefore, that these dislocations are acting as perfect, or nearly perfect, sinks for bulk diffusing interstitials.

The same is not always true of vacancies, however. The precipitation of vacancy clusters near dislocations necessitates a non-zero value for the vacancy super-saturation. As is argued in section 8.2.3, vacancy pipe diffusion is thought to be difficult in the 15%Ni ternary alloy, and to become progressively more easy as the nickel content is increased to 25% and 40%. The implication of this is that evenly dissociated, glissile, dislocations in the 15%Ni ternary are not good vacancy sinks and are thus likely to have a significant bias term. These dislocations also do not appear to be acting as efficient recombination centres (survival of vacancy clusters very close to the stacking fault). As nickel content is increased, the corresponding increase in vacancy mobility along dislocations, and corresponding decrease in the frequency of vacancy clusters, is consistent with the action of dislocations as better sinks for vacancies. Since dislocations in the ternary alloys appear to be acting as near perfect sinks for interstitials, the implication is that the bias term is reduced as the nickel content is increased.

As is noted by **Lee & Mansur** (1985), **Hoyt & Garner** (1990) and many others, even a small decrease in dislocation bias on increasing nickel content ^{may} alone account for the large decrease (by a factor ~ 10 , **Bates & Johnston**, 1977) in swelling on progressing from 15%Ni to 40%Ni levels.

The addition of 1% silicon to a 15%Ni matrix has a complex effect. We have argued above that the climb of these mixed, dissociated dislocations requires an interstitial super-saturation. These dislocations are therefore not perfect sinks for interstitials. Vacancy clusters along evenly dissociated dislocations, on the other hand, appear very similar to the 25%Ni alloy (compare figures 6.4 and 4.6). The implication here is that the addition of silicon generally also reduces the dislocation bias, but, as oppose to increasing nickel content, the principle origin here is a reduction in the efficiency of interstitial core diffusion. These dislocations also act as recombination centres and the behaviour of an individual dislocation relies on the heterogeneous re-distribution of silicon atoms. On the evidence of the micrographs, namely that interstitial climb has occurred where very few vacancy clusters are seen, a significant net excess of interstitials still arrive at dislocations: One might expect, therefore, that line dislocations, on the addition of 1%Si to a Fe-17%Cr-15%Ni alloy, will reduce swelling by a lesser factor than will increasing nickel content to 40%. Experimentally this would be extremely difficult to verify.

The effect on the dislocation bias is unclear for molybdenum additions; pipe diffusion of interstitials seems to be occurring in a similar fashion to the 15%Ni ternary alloy. Vacancy clusters appear larger and further removed from the line, for a comparable dose, which might suggest that these dislocations are more efficient as vacancy sinks than are dislocations in the 15%Ni ternary alloy. Recombination seems to be occurring near large features, which may be Mo rich regions and it is plausible, therefore, that the major effect of Mo may involve defect trapping. In real materials, grain boundaries are likely to be particularly important here (**James & Shepherd**, 1989; **Dumbill et al**, 1990). Whilst the current view appears to be that molybdenum additions reduce swelling (see **Ashworth**, 1989), recent experiments at Harwell find complicated results (**T W Williams & C M Shepherd** - private communication). **Ashworth** (1989) also found a confusing variety of effects on adding molybdenum and remarks that "*additions of ... Mo (and Si) ... have a significant effect on the microstructural development during irradiation*", but was unable to characterise the microstructure after his 0.6 and 3.6dpa irradiations. In view of the experiments shown in chapter 7 (see also figure 8.4), typically to doses $\simeq 0.1$ dpa and in which microstructural development is often already too complex to analyse fully, the difficulties encountered by **Ashworth** are hardly surprising. **Ashworth** (1989) also hypothesizes that the swelling response of Mo doped steels is principally a result of defect trapping.

It seems likely that screw dislocations in all the materials are acting as recombination centres. The effect is most marked for the 40%Ni alloy. It would also seem likely that the *bias* of screw dislocations should be similar to that of dissociated mixed, and edge dislocations and no evidence is found in the experiments to the contrary.

The behaviour of sessile dislocations is interesting. These dislocations will be introduced by cold work. Experiments in the 15%Ni, 25%Ni ternary, and 15%Ni+Mo quaternary, alloys suggest both that these dislocations may climb as undissociated dislocations and, that their climb does not require the build-up of a significant interstitial super-saturation. These dislocations may therefore be thought of as good sinks for interstitials. On the other hand, copious vacancy cluster nucleation is seen in their vicinity. As argued above, this will require a non-zero super-saturation and hence these dislocations will not be as good a sink for vacancies as for interstitials. As far as dislocations are concerned, this effect, in FCC materials, should be independent of materials composition and may partially explain the initially *increased* swelling exhibited by material cold-worked to $\lesssim 50\%$ (see **Leffers et al**, 1983).

Lastly, recent molecular dynamics simulations in FCC materials (**Huang et al**, 1989) find that the migration energies for core diffusion are *higher* than for bulk diffusion⁵⁵. A similar result has also been obtained in BCC materials (**Miller et al**, 1981). Both papers question conventional theories of pipe diffusion (e.g., **Atkinson & LeClaire**, 1984). It may be noted that, along dislocations, an *increased* activation energy for diffusion, particularly of vacancies, is implicit in results from this thesis.

8.4.3 High Dose:

Microstructural evolution at higher doses has not been considered in this study. However, we mention it briefly with reference to figure 8.4, which may show the onset of network formation. **Lee & Mansur** (1985), in (Ni + He) dual-ion and sequenced He injection-anneal-Ni ion irradiations, highlight the correlation between the formation of a dislocation network and the onset of significant swelling. Two possible explanations for this are suggested by work contained in this thesis: First, the reduction in bias for high nickel levels is expected to be greatest for line dislocations along which both defect species may pipe diffuse to an external sink (such as a surface or grain boundary) and, second, the onset of increased swelling rate may be related to the calculations of **Zinkle et al** (1987), which suggest that in the presence of gas atoms, such as helium, vacancy clusters may be more stable as voids rather than SFT. SFT are commonly seen near irradiated dislocations (this study), whilst voids are also often found near dislocations when gas atoms are present (e.g., **Norris**, 1971; **Rothaut et al**, 1983; **Singh et al**,

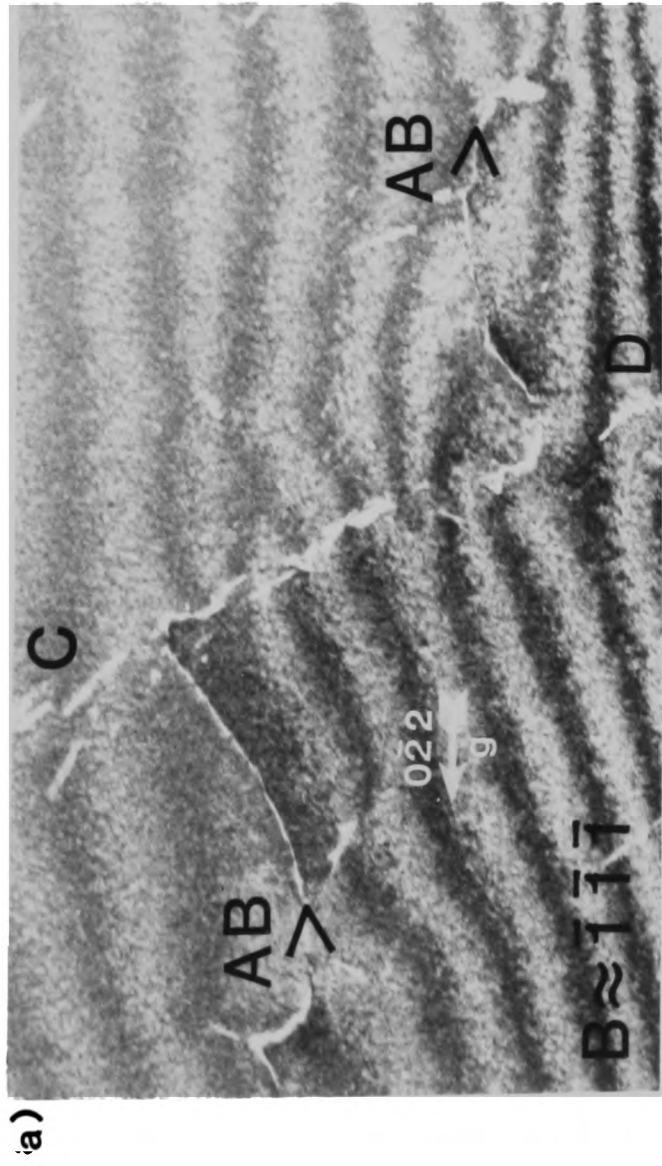
⁵⁵This says nothing about either the defect diffusivities, which are the quantities generally measured by experiment, or about the total activation energy for pipe diffusion (Q_d).

Figure 8.4

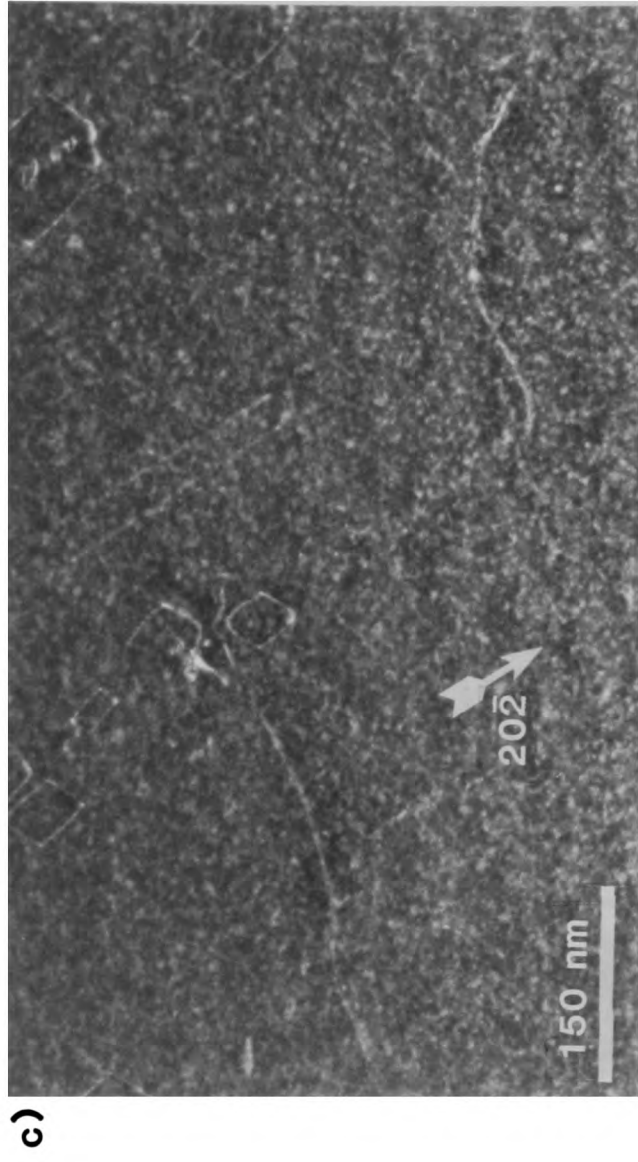
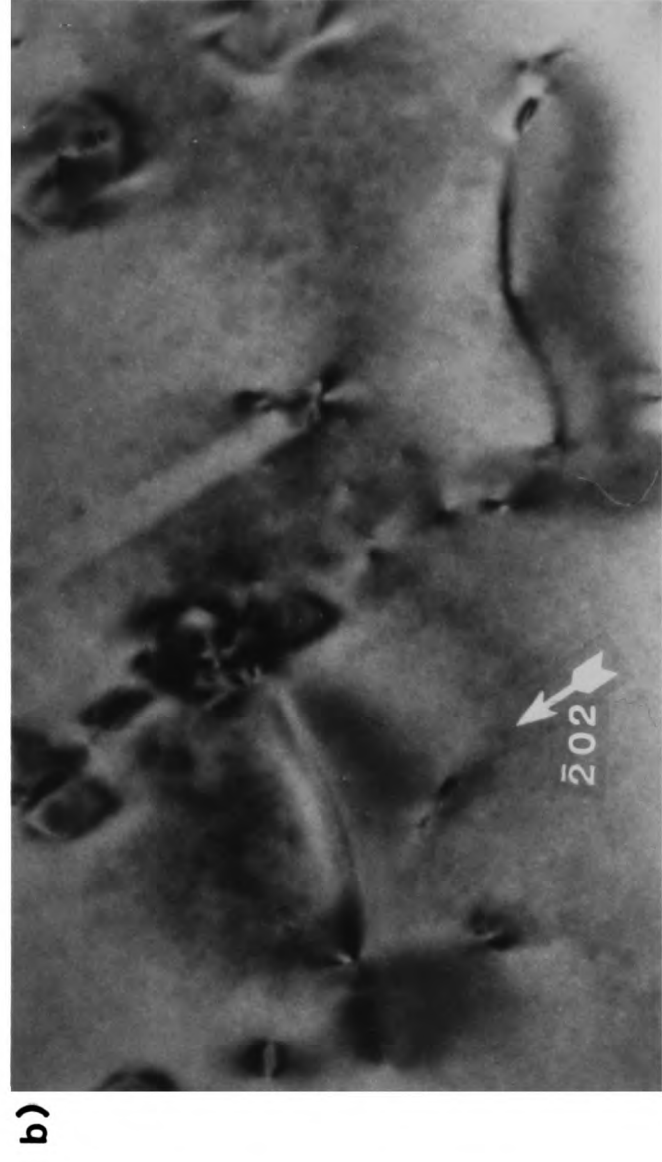
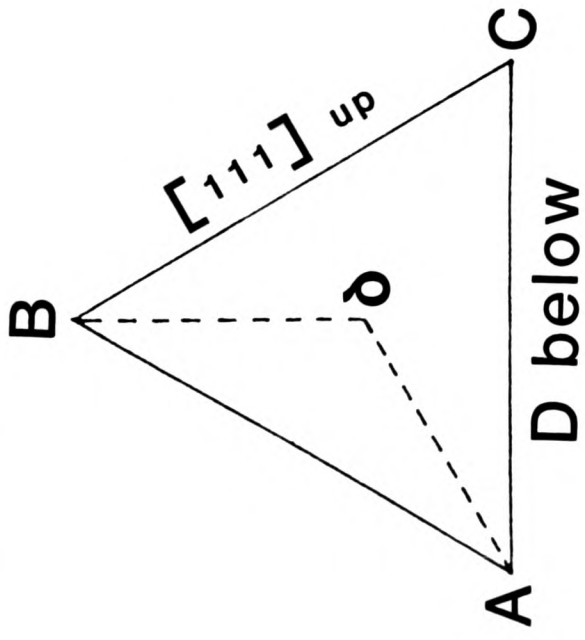
Micrograph (a) forms part of a contrast series taken around the (111) pole before irradiation. Dislocations $\tilde{\mathbf{b}}_{\text{T}} = \overrightarrow{\text{AB}}$ at right and left of the micrograph are glissile on δ , with line directions as indicated. The series of short dislocations between points 'C' and 'D' appear to have Burgers vector $\tilde{\mathbf{b}}_{\text{T}} = \overrightarrow{\text{CD}}$.

Micrograph (b) is a bright-field image taken after irradiation, the principal excited reflection being $[\bar{2}02]$ as indicated although other reflections were also active. The presence of several large new parallelogram-shaped loops as well as the disappearance of the dislocations between 'C' and 'D' is evident. The loops are now as large as 100 nm. across and their positions correspond to those of pre-existing features.

Dislocations $\overrightarrow{\text{AB}}$ are also of interest: They were observed to straighten, removing cusps, at an early stage in the irradiation. Small clusters were also seen to form nearby. Some of these clusters are faintly visible in micrograph (c). The large parallelogram-shaped loops, characterized elsewhere as interstitial, are not seen along these dislocations.



Before Irradiation



0.4 dpa at $\sim 420^{\circ}\text{C}$

Dislocations in Fe-15%Ni-17%Cr-2%Mo (at.%)

1984; Kimoto et al, 1986; Lee & Mansur, 1990b). It has been found that helium may pipe diffuse along dislocations (Buters et al, 1987; Subbotin, 1983). Hence, another possible explanation for the onset of increased swelling is that, on formation of a network, helium migrates throughout the irradiated area to small vacancy clusters nucleated near dislocations. Singh et al (1984) also propose that the core diffusion of He is responsible for their observations in proton irradiated Al. This latter explanation would obviate the need for an alteration in the dislocation bias partway through the irradiation (Lee & Mansur, 1985).

8.5: Other Materials

8.5.1 Silver:

The general observations made, and explanations given, of the behaviour of HVEM irradiated dislocations in silver (Hardy, 1985; Jenkins et al, 1987) are summarized below.

Fundamentally, there are three results from this study (see Jenkins et al, 1987):

- Dislocation partial separations decrease at an early stage in the irradiation;
- Dense vacancy cluster damage (often SFT) is seen to form close to dislocations;
- No evidence is seen for (major) climb motion of the line.

Jenkins et al (1987) conclude that dislocations are acting as good sinks for interstitials, allowing the precipitation of dense vacancy cluster damage in their vicinity. They are undecided, however, on the detailed mechanisms of interaction of HVEM generated point defects and dislocations. As noted by Hardy (1985), the bias term (Heald, 1975) renders it likely that a greater flux of interstitials will migrate to dislocations. Further experimentation by Hardy⁵⁶ led him to conclude, incorrectly (Jenkins et al, 1987; Sigle et al, 1988; Kojima et al, 1989), that SFT nucleated near the line were interstitial in nature.

Bearing in mind similar observations in these Fe-Ni-Cr alloys, of occasionally dense SFT formation close to dislocations, which dislocations are not apparently climbing, it would seem likely that fundamentally similar mechanisms are operating; i.e., that observations in silver may be explained by the pipe diffusion of interstitials whilst vacancies cluster within the compressional strain field of the dislocation. The distribution of clusters with respect to dislocations cannot be determined from micrographs shown by Hardy (1985), however, and it is uncertain whether large angle tilt experiments were undertaken. Pipe diffusion has been found to be particularly easy in silver (Gjostein, 1973, gives values for Q_d/Q_l of 0.3 and 0.4 for Ag¹¹⁰ tracer diffusion along grain boundaries in Ag). This pipe

⁵⁶The “ $2\frac{1}{2}$ D” technique (Mitchell & Bell, 1976) was employed. The reliability of this technique for the determination of the nature of small SFT has since been called into question (Piskunov, 1985).

diffusion may be significantly enhanced by a narrowing of the partial separations (see below and §8.2.3), which may, in turn, explain these low activation energies.

The final point concerns the origin of decreases in the partial separations: In Fe-Ni-Cr, a number of possibilities are suggested (§8.2.2). These different possibilities arise because irradiations were carried out at elevated temperatures, and because compositional redistribution is a factor in Fe-Ni-Cr alloys. In the paper of **Jenkins et al** (1987), in which the short section of dislocation shown has narrowed, the specimen was thinned *immediately* prior to insertion into the Argonne HVEM. The material employed was 3N purity Ag and, hence, it seems unlikely that segregation is a factor here. In addition, the irradiation was performed at room temperature and, therefore, any dependence of the SFE on temperature may be excluded. Hence, relaxation of the dislocation stress field by vacancy inclusions (see §8.2.2) seems to be the likely origin of this phenomenon in silver. In Fe-Ni-Cr, however, all of the variables mentioned in §8.2.2 may have a rôle.

8.5.2 Cu-13%Al:

The formation of interstitial (**Cherns et al**, 1980) and vacancy (**Décamps et al**, 1983) type dipoles, consistent with loop nucleation onto widely extended dislocations in this low SFE alloy, will require the build-up of defect super-saturations. By the above model, the development of this super-saturation is only expected to occur if pipe diffusion is inefficient. As is outlined in section 8.2.3, very little data exists relating the efficiency of pipe diffusion to partial separation, however, it seems plausible that the efficiency of pipe diffusion should be poor for Cu-13%Al (where the separation between partials can vary up to $\sim 800 \text{ \AA}$ for 60° dislocations (see Cherns et al, their figure 2(a)); an order of magnitude greater than for the Fe-Ni-Cr alloys of the present study).

One feature which is notably absent from the post-irradiation micrographs shown by both Cherns et al and **Hardy** (1985; Cu-10%Al) is vacancy cluster damage. At room temperature and above, however, both interstitial and vacancy defects are expected to be mobile (**Cherns et al**, 1980; **Hardy**, p.34). **Décamps et al** (1983) show that dislocations act as sinks for vacancies and hence it appears that significant recombination is occurring. This is entirely consistent with observations made in Fe-Ni-Cr, particularly in the 15%Ni+Si alloy of chapter 6. The hypothesis of **Cherns et al** (1980) that a decrease in partial separation may result in operation of the **Thomson & Balluffi** (1962) mechanism is also consistent with observations in chapter 6.

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Appendix



Chapter 9

Conclusions and Suggestions for Future Work

Evidence presented in this thesis is consistent with the following conclusions:

9.1 The Mechanisms of Interaction of HVEM Generated Point-Defects with Dislocations:

Similar interactions appear to be occurring in all three ternary alloys:

Interstitials:

- Interstitials pipe diffuse along all dislocations either until they encounter a surface or a favourable site at which they may precipitate, such as a jog. The pipe diffusion of interstitials seems likely to be very efficient.
- In the case of glissile, dissociated dislocations of greater than about $20-30^\circ$ in character, jogs climb together and annihilate. These jogs appear constricted and their climb, by the absorption of a net excess of interstitials, appears to be a diffusion rate-controlled process. After the annihilation of the jogs, interstitials pipe diffuse along the dislocation. The climb of sessile dislocations and interstitial loops also appears to be principally diffusion rate-controlled.
- For screw, and near-screw, dislocations jogs climb away from the dislocation until the climb force on the jogs is balanced by increases in line tension of the adjoining segments. This may be aided by alignment of these sections along particularly favourable crystallographic directions. Jogs along screw dislocations continue to act as pipe diffusion barriers, allowing the build-up of an interstitial super-saturation which eventually prompts the climb of adjoining segments.
- The generation of an interstitial super-saturation apparently prompts greater recombination.

Vacancies:

- The generation of vacancy clusters is seen close to all dislocations with appreciable edge components. The sizes of these clusters, and their separation from irradiated dislocations increases, whilst the frequency of vacancy cluster formation decreases, as nickel is added.
- Vacancies appear to undergo very little, or no, pipe diffusion in the 15%Ni ternary alloy. As nickel content is increased, however, the pipe diffusion of vacancies appears to become substantially more efficient.

The behaviour of the Fe-17%Cr-15%Ni alloy appears to be modified as follows by the addition of 1% silicon:

- Heterogeneous silicon segregation to, along, and/or around dislocations is thought to be the origin of barriers to pipe diffusion.
- The creation of these barriers leads to the build-up of a net interstitial super-saturation.
- The build-up of this saturation apparently leads to greater recombination, whilst the dislocation bias for interstitials eventually results in climb. This leads to the creation of jogs along erstwhile evenly dissociated dislocations.
- The mobility of interstitials in the bulk may be increased, as may the mobilities of both defect species along 'barrier-free' dislocation cores.

The addition of 2%Mo to the Fe-17%Cr-15%Ni alloy apparently leads to:

- Very complex dislocation structures prior to irradiation. This is thought to arise from the thermal segregation of Mo to dislocations.
- The generation of large loops close to irradiated dislocations.
- The generation of large loop-like features attached to dislocations, the formation of which may involve the pipe diffusion of Mo.
- No observed effect on interstitial, but a possible, slight, enhancement of vacancy pipe diffusion.

The following conclusions are more speculative in nature:

- Two possible origins of reduced swelling exhibited by intermediate Ni content alloys are: A reduction in the bias term for line dislocations and greater defect recombination owing to a reduction in the width of dislocation cores along which defects are diffusing.
- This reduction in the bias term is thought to arise from an increase in the efficiency of vacancy pipe diffusion, resulting in dislocations acting as better sinks for vacancies. Dislocations along which significant pipe diffusion is occurring act as perfect, or nearly perfect, sinks for interstitials.
- Reduced swelling exhibited by Si-doped Fe-Ni-Cr alloys also may be attributable in part to a reduction in the dislocation bias. This reduction arises because dislocations along which pipe diffusion has been inhibited are acting as less efficient sinks for interstitials. Greater defect recombination at dislocations may also aid in reducing swelling.
- The effects of Mo additions on swelling are not clear from these experiments. Greater defect recombination at large loops formed along dislocations is implicated, however.

The final points concern pipe diffusion:

- The activation energy for the pipe diffusion of vacancies, particularly in the 15%Ni ternary alloy, is *greater* than that of bulk diffusion.
- The widely held view that interstitial pipe diffusion is inefficient is contradicted by these experiments.
- Observations in CuAl and Fe-Ni-Cr and Ag are consistent with an increase in the efficiency of pipe diffusion as partial separation is reduced.

9.2 Some Suggestions for Further Research:

One of the basic assumptions underlying the conclusions from this thesis is that interstitials migrating to a dislocation may pipe diffuse along it. Recent computer simulations have highlighted the possibility and further such simulations, on a wide variety of dislocation core structures are required. Ideally the core structures resulting from the potentials employed should be compared with ‘known’ core structures from HREM studies, since the predictions of anisotropic elasticity theory may not always be reliable (c.f., Lomer dislocations; favourable crystallographic directions).

The dislocation diffusion coefficients should be measured for the Fe-Ni-Cr system under conditions of interstitial and vacancy (radio-active tracer diffusion during low-temperature irradiation & recovery), and vacancy (thermal) super-saturations and compared with computer simulations. This may shed light on the diffusing defect species but is not an experiment to be treated lightly.

Any thermal effects leading to narrowing of the partial separations in the specific alloys studied here will be evident in a thermal cycling experiment. The recent work at Harwell suggests the possibility that R.I.S. to irradiated dislocations may be detected; any quantitative results are unlikely to be reliable at this stage, however, it would be interesting to study the dislocations irradiated by the author specifically since microstructural information is already available. This may be particularly relevant to the features decorating dislocations in the 15%Ni+Mo alloy. Microstructurally significant Si segregation may be difficult to detect.

The suggested origins for a decrease in dislocation bias as nickel content is increased hinge on several observations. Two of these in particular need to be verified:

1) A 60° dislocation in the 15%Ni ternary alloy should be successively irradiated to a higher dose in the Argonne HVEM. Micrographs in $\bar{g} = \langle 11\bar{1} \rangle$ reflections should be taken at intermediate dose points in a low-keV TEM with better resolution since the HVEM is incapable of resolving the tiny ($< 10\text{\AA}$) clusters. Hopefully the nature of these tiny clusters will then be definitely established. In essence this experiment is very similar to that performed by **Jenkins et al** (1987).

2) Further observations and characterization of vacancy cluster damage near dislocations in the 40%Ni alloy is required. Experiments should be performed on (111) foils under vacuum conditions similar to those employed here (see chapter 5) in the Argonne HVEM.

Finally, the scope for extending these experiments (e.g., higher nickel (70% ?), temperature dependence (see chapter 2 before this is attempted), heavy-ion irradiation) is limited principally by the availability of the necessary finance and equipment (one is likely related to the other). Sources of 1MeV electrons are now scarce, and those that survive are unlikely to do so for long. Different electron irradiation sources, such as 400kV microscopes, may provide a solution.



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Appendix

A Possible Mechanism for the Formation of the Node of Figure 4.7

Diagrams A.1 to A.8:

Due to the experimental difficulty encountered in ascertaining precise pre- and post-irradiation configurations of the node region, a mechanism for its formation, and subsequent behaviour under irradiation of the three dimensional structure so formed, is proposed below. For the mechanism to operate a particular initial configuration is assumed, whilst its driving forces are an external stress and various energetically favourable dislocation reactions. For the sake of simplicity all glide is resolved onto $\{111\}$ planes.

As can be seen from the micrographs, the node of figure 4.7 includes dislocations $\vec{AB}(\delta)$, $\vec{AC}(\delta)$ and $\vec{CD}(\alpha)$. Dislocations $\vec{AB}$ and $\vec{AC}$ are both in 60° orientation and are lying on, say, planes δ_1 and δ_2 respectively. It would seem unlikely that $\delta_1 = \delta_2$ and it is assumed that these dislocations are not, in fact, co-planar. In order, however, for these two dislocations to have interacted it is probable that they lay initially close enough to feel each other's strain field. The third dislocation intersecting at the node (dislocation $\vec{CD}$) appears to lie along the projection of direction $\vec{CD}$ on δ . Again it would seem unlikely that this dislocation was formed at exactly this location. It should be noted that were dislocation $\vec{CD}$ to be in pure screw orientation its glide cylinder would include both planes α and β . Dislocations $\vec{AB}$ and $\vec{AC}$, being in 60° orientation can only glide easily on δ .

One now has to consider the history of this specimen: Prior to thinning this foil was subjected to a moderate amount of cold work by bending. This will cause stresses within the foil greater than the Peierls stress and may be the cause of the α slip trace seen in figure 4.7 (j). These stresses will also lead to forces on dislocations $\vec{AB}$, $\vec{AC}$ and $\vec{CD}$.

It is thought that dislocation $\vec{CD}$ has glided through the foil to intersect with $\vec{AC}$, and thence to be channelled towards the point of closest approach of dislocations $\vec{AB}$ and $\vec{AC}$, as shown by the following mechanism. With no idea as to the formation points of the three dislocations a large number of initial configurations were considered; the precise starting points of the three dislocations is unimportant, however, in the context of this mechanism. It was found empirically that a pre-irradiation configuration close to that shown in drawing 4.7 (h) could best be achieved by assuming that, at some point, dislocation $\vec{AC}$ was above $\vec{AB}$; that dislocation $\vec{CD}$ should run from top to bottom

of the foil, as shown in stage 1.I, and that it should first intersect with dislocation $\overrightarrow{AC}$. Every possibility was, however, not considered and other initial conditions may exist which also lead, via similar treatments to that below, to pre-irradiation configurations which satisfy experimental observation.

With the configuration shown in stage A.1.I components of forces on the dislocations resulting from this external stress are supposed to act such that dislocation $\overrightarrow{CD}$ is gliding to the left on β whilst dislocations $\overrightarrow{AB}$ and $\overrightarrow{AC}$ feel upward and downward climb forces respectively. This is represented by stage A.1.II, in which the sense of the half-planes of dislocations $\overrightarrow{AB}$ and $\overrightarrow{AC}$ is also shown.

The climb forces on dislocations $\overrightarrow{AB}$ and $\overrightarrow{AC}$ will prompt them to accommodate interstitials and vacancies respectively, via the formation of double jogs on planes α and β respectively, as shown in stage A.1.III. Whilst these jogs may form at any point along the line, the activation energy for their formation at the points shown, i.e. such that each super-jog interacts with the neighbouring dislocation, is significantly less than at other points along the line: Firstly; individual stair-rod dislocations so formed can react with the activating partial of the second dislocation and secondly; the formation of these super-jogs requires the transport of atoms, as shown in stage A.1.IV, although vacancies are not mobile at room temperature over long ranges in Fe-Ni-Cr (see chapter 1). At the point of closest approach of the two dislocations, the distance across which matter must be transported is minimized; hence, climb forces felt by the dislocations may be sufficient here for the jog nucleation proposed. Intuitively one might expect the length of the transported section to be a function of the vertical separation between dislocations $\overrightarrow{AB}(\delta_1)$ and $\overrightarrow{AC}(\delta_2)$, as well as of the magnitude of the climb force, although calculations have not been performed to verify this.

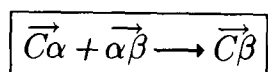
In stage A.1.III, dislocation $\overrightarrow{CD}$, which was gliding initially on β , is now intersecting dislocation $\overrightarrow{AC}$. This has caused $\overrightarrow{CD}$ to cross-slip onto α and glide along dislocation $\overrightarrow{AC}$ (see diagram A.10); particularly easy if dislocation $\overrightarrow{CD}$ is in pure screw orientation. The possibility that local constriction may result from this intersection by the formation of a short length of Lomer dislocation is also addressed in diagram A.10.

In diagram A.2 (stage 2) the configuration shown in stage A.1.III is enlarged. Dislocation $\overrightarrow{CD}$ has now separated into two sections (via the mechanism of diagram A.9). The lower acute super jog on $\overrightarrow{AC}$ is also likely to have been partly, or totally, constricted by the process and, for simplicity, is shown constricted here. It appears constricted in the micrographs. The upper section of $\overrightarrow{CD}$ is now being channelled along $\overrightarrow{AC}$ towards the jog complex at centre via the formation, under external stress, of an

alternative Lomer-Cottrell style configuration⁵⁷ similar to that shown in diagram A.10.3. This configuration is effectively ‘zipping up’ the dissociated dislocation $\overrightarrow{AC}(\delta_1)$ (see footnote). Since the lower section of $\overrightarrow{CD}$ is now pinned at the lower acute super-jog a long length of total Burgers vector $\overrightarrow{AD}$ is formed.

In diagrams A.3 to A.6, only the central jog complex of diagram A.2 is shown. Stages 3 and 4 are presented as occurring in order, although in reality they are likely to be effectively simultaneous. In A.3, dislocation $\overrightarrow{CD}$ has now glided along $\overrightarrow{AC}$, reacting to form a length of constricted dislocation $\overrightarrow{AD}$, to intersect with the double extended jog configuration. A surviving portion of the ‘zipping’ Lomer-Cottrell lock is represented as Burgers vector $\overrightarrow{\delta\alpha}$ here.

The leading partial, $\overrightarrow{C\alpha}$, may now react with the stair-rod dislocation $\overrightarrow{\alpha\beta}$ via:



This reaction produces a continuous length of dislocation $\overrightarrow{\beta C}$ on plane β , pinned only where it intersects with stair-rod $\overrightarrow{AC}/\delta\beta$. Line tension forces will now tend to straighten dislocation $\overrightarrow{\beta C}$ leading to the configuration of diagram A.4. The initial ‘zipping’ Lomer-Cottrell lock ($\overrightarrow{AC}(\delta_1)$ and $\overrightarrow{CD}(\alpha)$) has

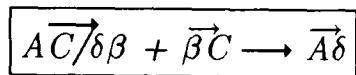
⁵⁷The possibility that the entire section between the central jog complex and the lower acute super-jog should exist as a ‘type 1’ Lomer-Cottrell lock is unlikely: Firstly, line tension forces (see **Whelan**, 1959) will tend to straighten partials $\overrightarrow{C\alpha}$ and $\overrightarrow{\alpha D}$, which intersect $\overrightarrow{AC}$ at an angle of 60° , and secondly; ‘type 1’ extended dislocation barriers have a very low resistance to collapse under external stress (**Hirth & Lothe**, p.803). The latter consideration may result in this stretch of dislocation (between ‘A’ and ‘C’ in figure 4.7) consisting of the Frank and Shockley partials $\overrightarrow{A\alpha}$ and $\overrightarrow{\alpha D}$; this, however, requires extensive bow-out of the partial $\overrightarrow{\alpha D}$ and widening of the stacking fault on α . Alternatively $\overrightarrow{A\alpha}$ and $\overrightarrow{\alpha D}$ may constrict to form the Lomer dislocation $\overrightarrow{AD}$. This is an energetically neutral reaction and resistance to bow-out of $\overrightarrow{\alpha D}$ may provide any driving force required. $\overrightarrow{AD}$ may then redissociate to $\overrightarrow{A\alpha} + \overrightarrow{\alpha D}$ or to $\overrightarrow{A\delta} + \overrightarrow{\delta D}$. In stage 2 yet another possibility is represented, involving the moderately energetically *unfavourable* reaction $\boxed{\overrightarrow{\delta\alpha} + \overrightarrow{\alpha D} \longrightarrow \overrightarrow{\delta D}}$, the driving forces for which may be the resistance to bowing-out of $\overrightarrow{\alpha D}$ and the reluctance of $\overrightarrow{A\delta}$ to turn towards screw orientation, i.e. increases in line tension.

Bearing in mind experimental evidence that Lomer dislocations are mostly undissociated here (e.g. figures 4.5 and 7.11) it is most likely that the bulk of this section will quickly form into the constricted perfect dislocation $\overrightarrow{AD}$. Hence, the representation of this section as being composed of sections with Burgers vectors $\overrightarrow{\delta C}$, $\overrightarrow{\delta\alpha}$, $\overrightarrow{\delta D}$ and $\overrightarrow{AD}$ is a compromise between some of the above possibilities.

Thus, the answers to questions as to *whether* the edge partial intersects the line of partial δB , to form a constriction, and *how long* the constricted region is, seem fairly plain. The exact configuration hinges simply on *where* this constriction occurs. This final point is of fairly academic interest, however, since it can easily be shown that the mechanism continues to develop in a similar fashion given any of the above eventualities.

now effectively been annihilated. Any continued force aiding glide along α of dislocation $\overrightarrow{CD}$, coupled with the mutual attraction of dislocations $\overrightarrow{C\alpha}$ and $\overrightarrow{\alpha C}$, will also tend to cause these two dislocations to approach one another as shown.

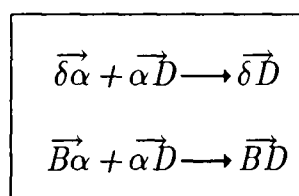
In A.5, dislocations $\overrightarrow{C\alpha}$ and $\overrightarrow{\alpha C}$ have annihilated, forming another Lomer-Cottrell style, 'type 1' configuration ($\overrightarrow{AB}(\delta_1)$ and $\overrightarrow{BD}(\alpha)$). Line tension forces have now straightened dislocation $\overrightarrow{\beta C}$, allowing occurrence of the reaction:



Line tension forces will now act in the direction of the large arrows shown in A.5, tending to straighten the now continuous length of dislocation $\overrightarrow{A\delta}$. This force, coupled again with any continued glide of $\overrightarrow{CD}$ on α , may lead to the configuration of diagram A.6. Dislocation $\overrightarrow{CD}$ is shown to be constricted along its entire length for simplicity, although it is probably still dissociated above its point of intersection with $\overrightarrow{AC}$ and $\overrightarrow{AB}$.

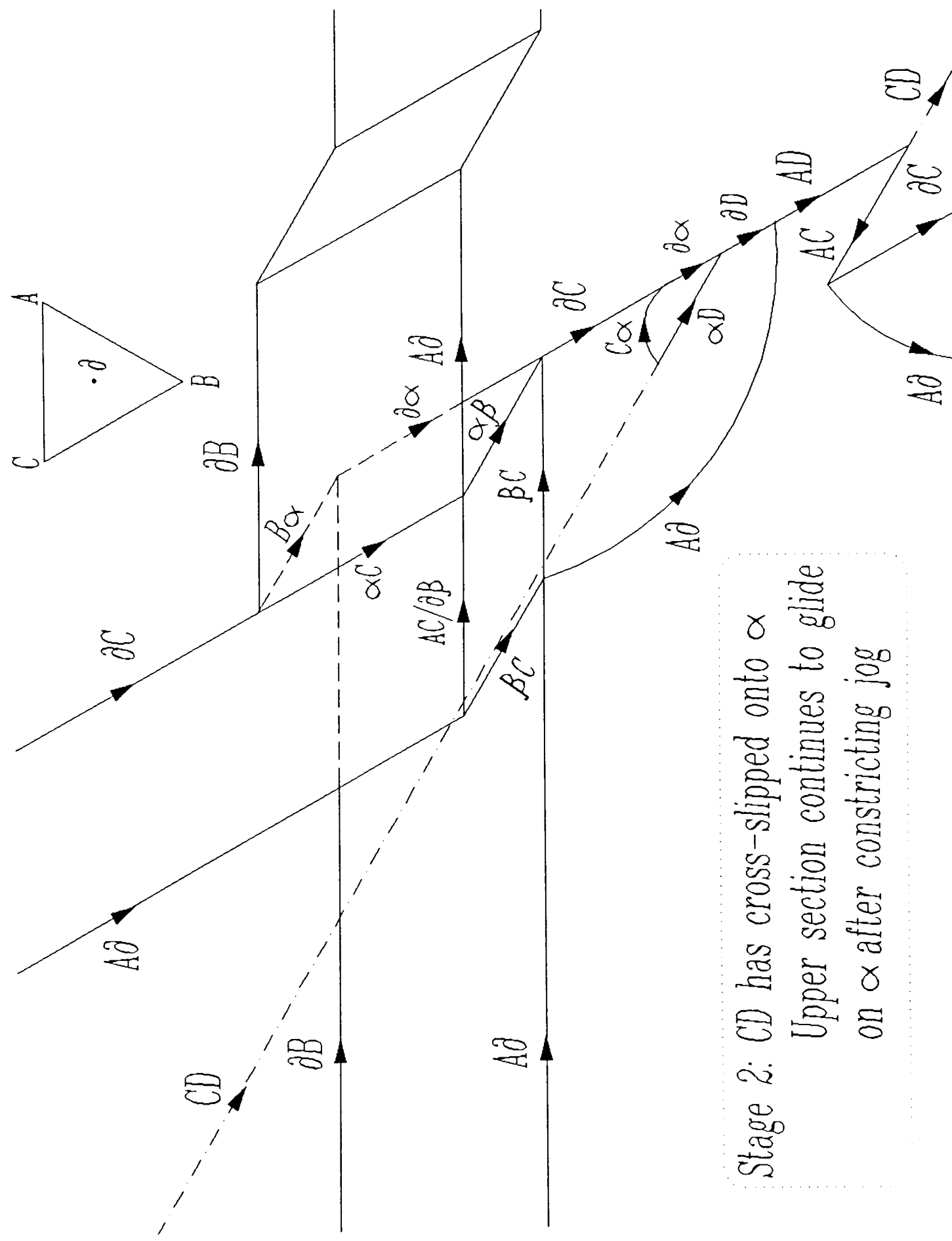
The wide dissociation of dislocation $\overrightarrow{AB}(\delta_1)$, seen to the left in the micrograph of figure 4.7 might be explained by the continued presence of the Lomer-Cottrell lock shown towards the centre of stage 6. The configuration shown here may be particularly stable against both collapse and further glide, since both dislocations $\overrightarrow{AB}(\delta_1)$ and $\overrightarrow{BD}(\alpha)$ of the lock extend at a large angle (60°) to the locking stair-rod. It is possible, therefore, that this stage may actually represent the initial configuration shown by the pre-irradiation micrographs of figure 4.7.

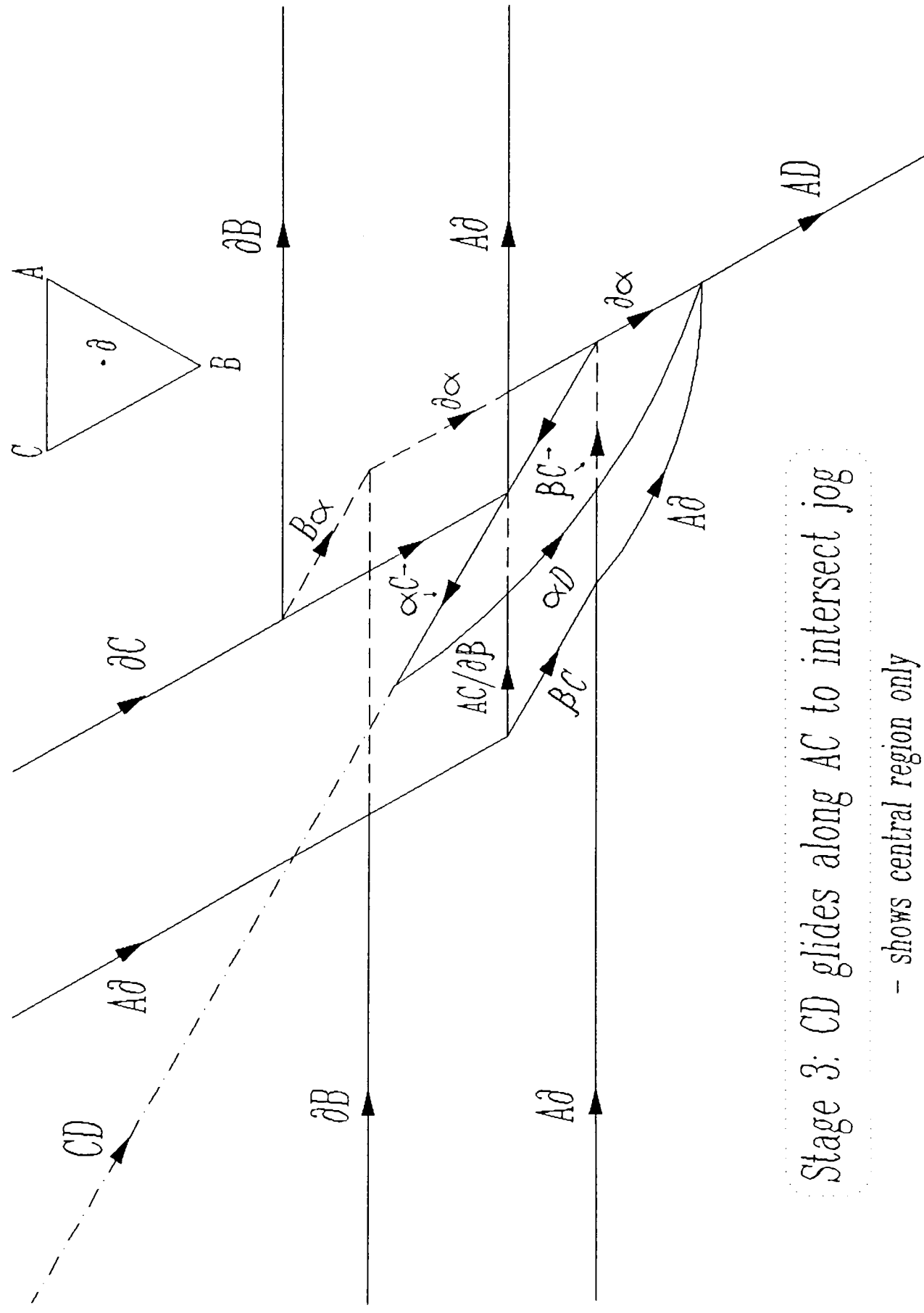
The entire intersection region is shown in the next diagram (A.7). This diagram corresponds topologically to the pre-irradiation configuration shown in figure 4.7(h) via the energetically unfavourable constrictions:



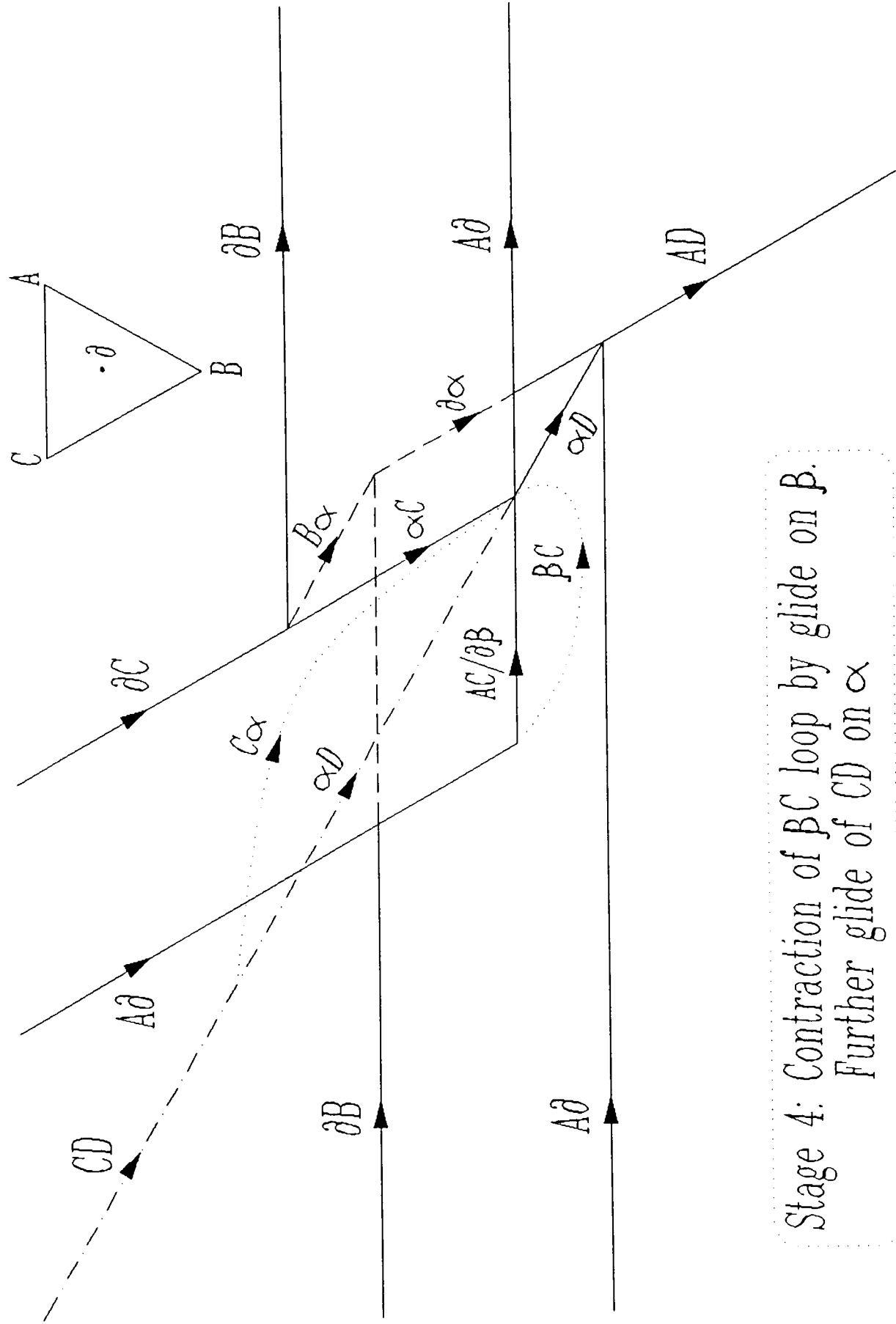
As mentioned above, it seems likely that these reactions have not, in fact, occurred prior to irradiation, although the possibility might be entertained given continued external stress prompting further glide of $\overrightarrow{CD}$ on α .

The glide of partial $\overrightarrow{A\delta}$ on δ_2 (upward as shown in these diagrams) might lead to constriction of the obtuse super-jog on the right hand section of $\overrightarrow{AB}$ as shown. It is difficult, however, to discern the configuration of this super-jog from the micrographs so this constriction is merely suggested here.

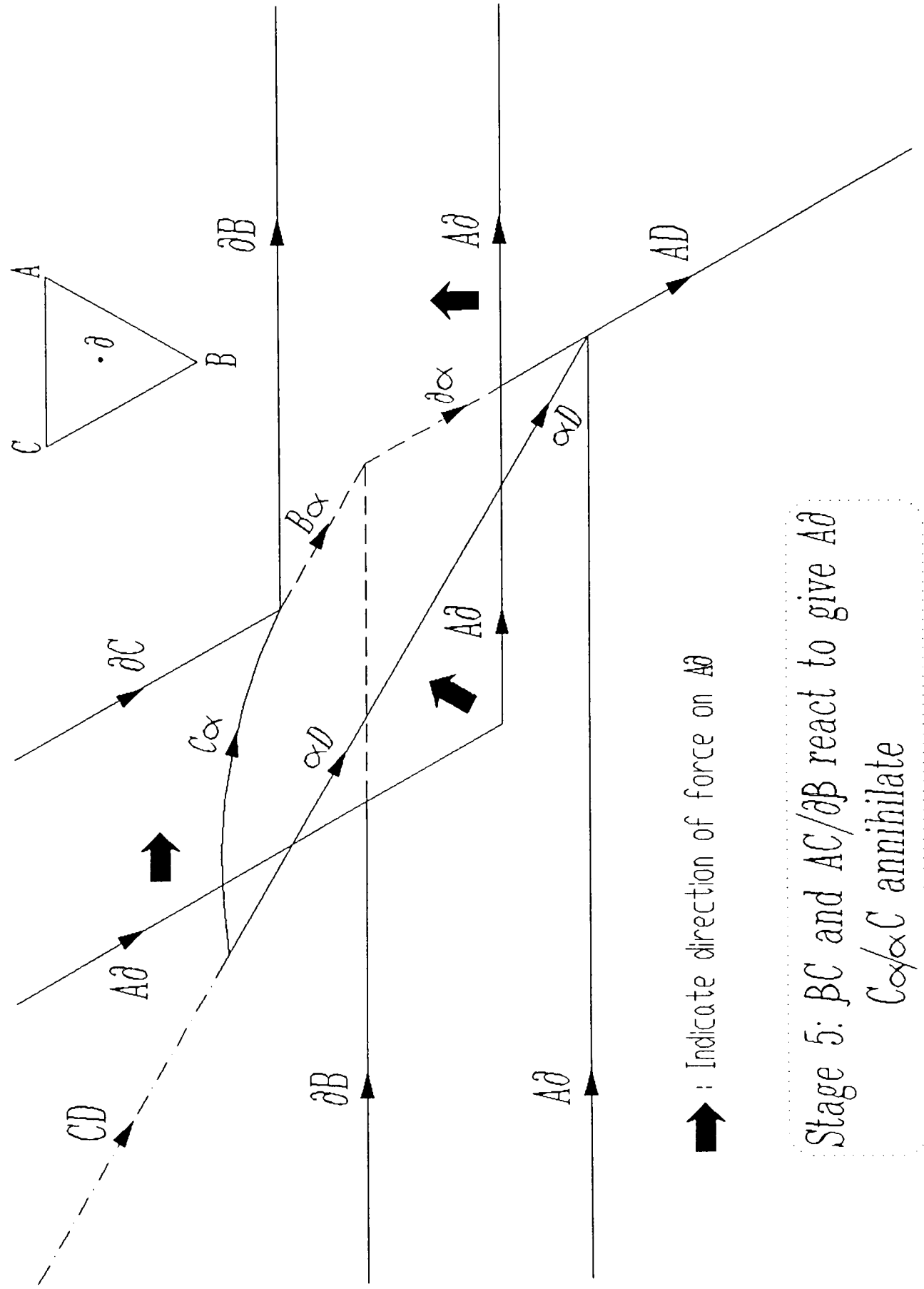


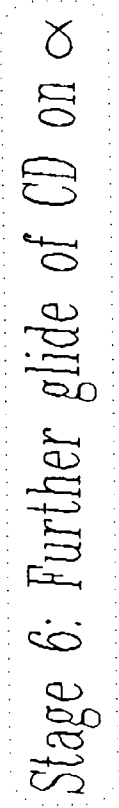


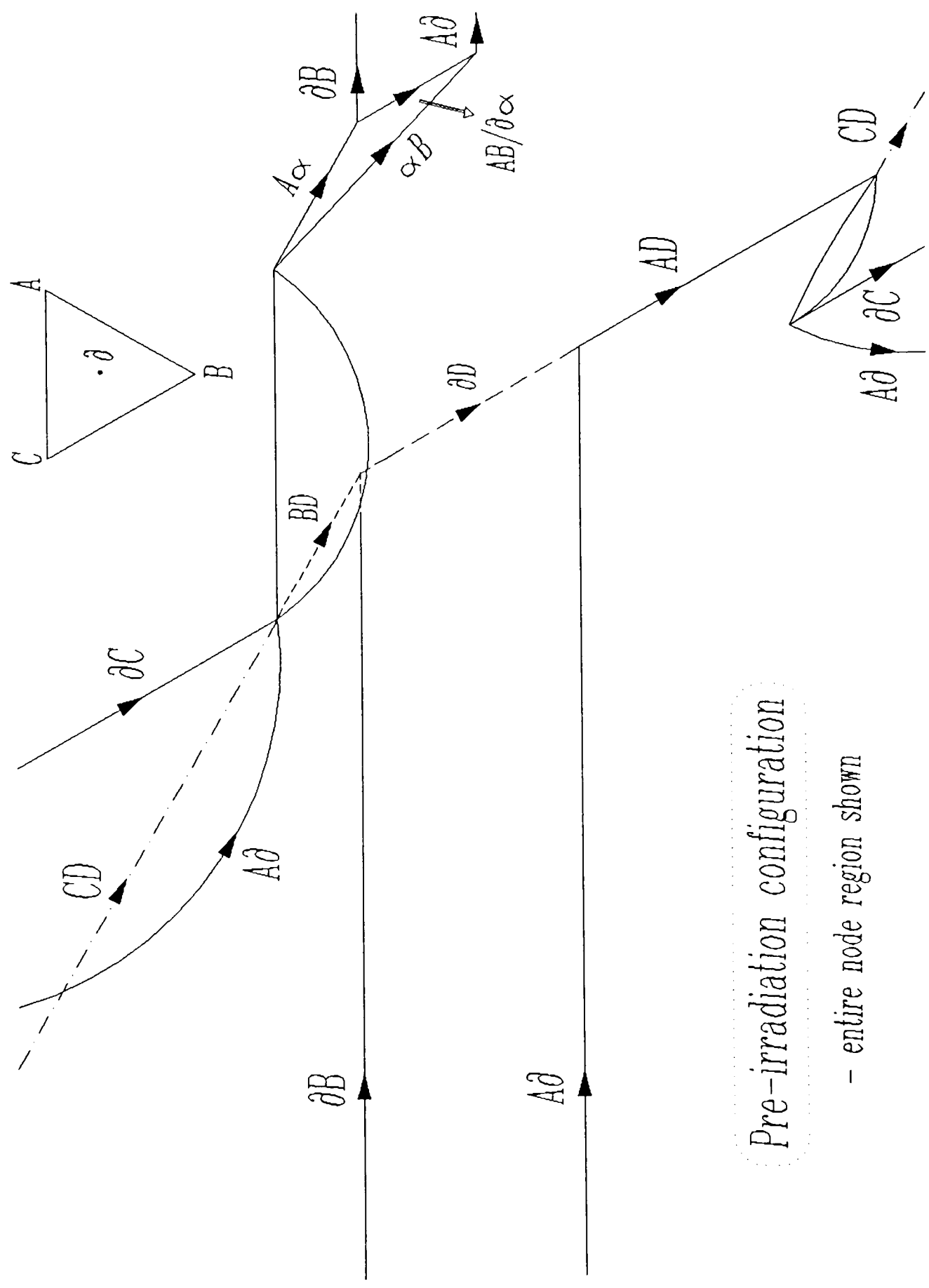
-- shows central region only



Stage 4: Contraction of βC loop by glide on β .
Further glide of CD on α







Pre-irradiation configuration

- entire node region shown

Diagram A.8: "Irradiation";

Employing the criterion that a parent dislocation may not be rotated to accommodate precipitated Frank loops, a very great number of possible sites for these loops are found.

Some of these sites are indicated in diagram A.8, perhaps the most obvious of which is that labelled '1', at which a loop with $\tilde{b}_L = \vec{D}\delta$ may be precipitated. . Formation of this loop may annihilate the stacking fault between partials $\vec{\delta B}$ and $\vec{A}\delta$, possibly explaining the observed narrowing of the left-hand section of dislocation $\vec{AB}(\delta_1)$ (see figure 4.7).

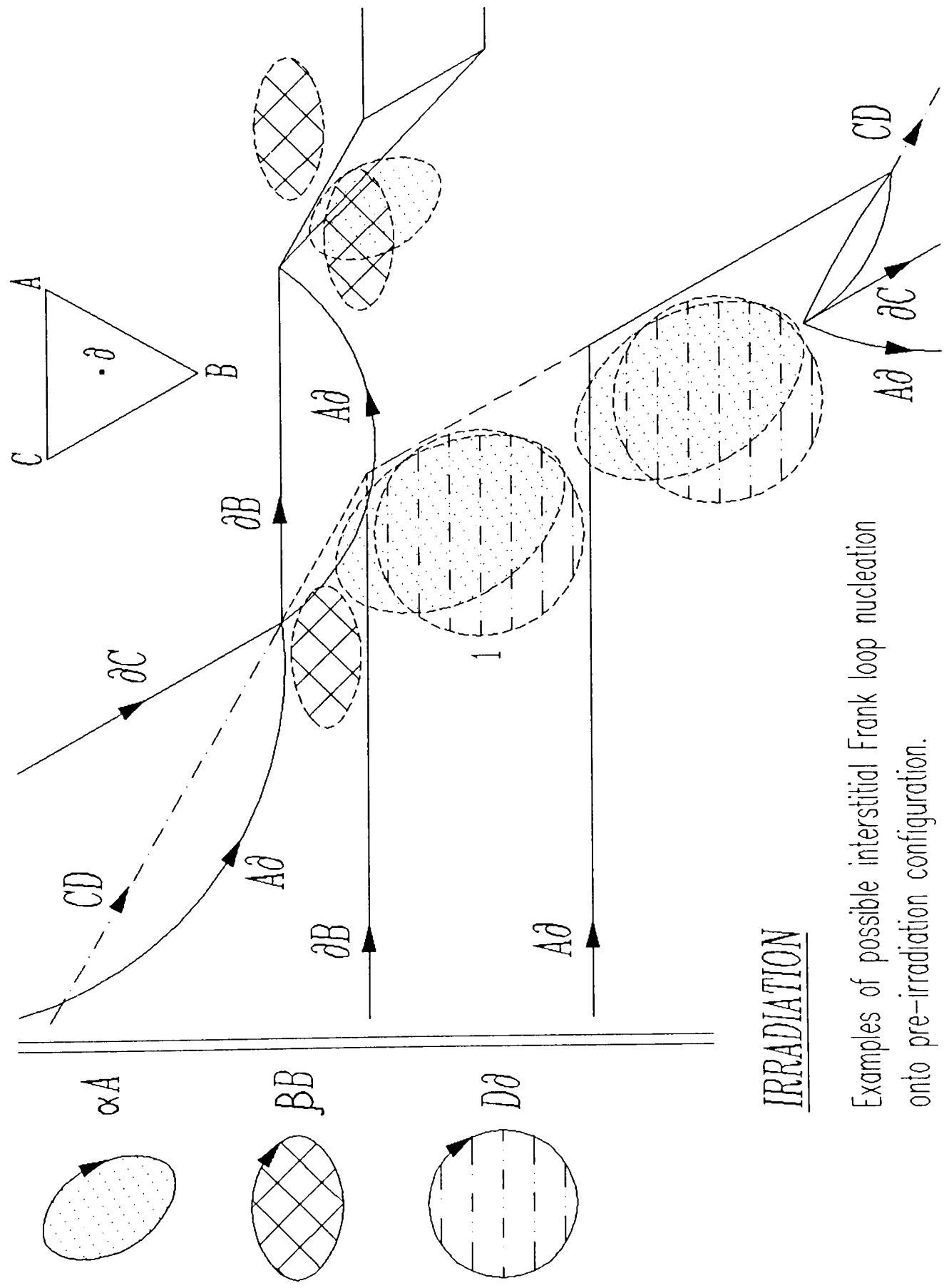
An alternative to the above is provided by the observation that the Lomer-Cottrell configuration of stage 6 (diagram A.6) contains an inherent vacancy loop, $(\vec{D}\alpha : \vec{B}\alpha : \vec{\delta}\alpha)$. Interstitials might precipitate within this region, essentially as a $\tilde{b}_L = \vec{\alpha A}$ Frank loop, to form the sessile dislocations $\vec{AD}(\alpha)$ and $\vec{BA}(\beta)$.

It is uncertain whether dislocations must constrict prior to the precipitation of interstitials as Frank loops; however, with, or without primary constriction of the super-jogs the partial separations here will be very small, particularly under irradiation when partials separations are often seen to narrow. In any event there are a large number of short segments, which probably trap pipe diffused defects, leading to a huge array of possible sites for interstitial precipitation.

The effectively limitless number of ensuing intersections, via climb, glide and/or rotation of precipitated loops, theoretically allows the generation of any Burgers vector. This may explain the extreme difficulty encountered in attempts to discern the post-irradiation configuration experimentally. Additionally, loops $\vec{\alpha A}$, $\vec{\beta B}$ and $\vec{D}\delta$, for which the pre-irradiation configuration proposed here already provides initial sites, may themselves intersect to provide further favourable sites for precipitation, before, or after, unfauling.

Finally, the configuration shown in this series of diagrams represents the simplest case giving rise to contrast consistent with the pre-irradiation micrographs; the application of this method to more complex possible starting configurations (e.g. the nucleation of quadruple extended super-jogs directly *co-planar* onto the intersection of $\vec{AB}$ and $\vec{AC}$) yields a far greater number of favourable sites for loop nucleation, including the $\vec{\gamma C}$ species.

In conclusion, therefore, irradiation of this dislocation node is expected to prompt the generation of an immensely complex, and very compact network of dislocations. These dislocations may have any conceivable Burgers vector, although perfect prismatic and Lomer dislocations, formed by the intersection of unfaulted Frank loops, may predominate.



IRRADIATION

Examples of possible interstitial Frank loop nucleation onto pre-irradiation configuration.

Diagram A.9: “Splitting of $\vec{CD}$ at acute super-jog on $\vec{AC}$ ”;

Diagram 9 of this series expands on the proposed separation of dislocation $\vec{CD}$ into two sections when intersecting the lower acute super-jog on $\vec{AC}$. This is shown as occurring between stages A.1.III and A.2 above. In the process the super-jog may also be constricted: (1) shows the initial configuration, in which dislocation $\vec{CD}$ is still gliding on β towards the evenly dissociated dislocation $\vec{AC}$. In (2), dislocation $\vec{CD}$ has now intersected with dislocation $\vec{AC}$, causing a local constriction (via collapse of a ‘type 1’ Lomer-Cottrell lock and possibly also the formation of a short length of Lomer dislocation - see diagram A.10) and cross-slip of $\vec{CD}$ onto α .

In (3) dislocation $\vec{CD}$, and the local constriction, have reached the top of the acute super-jog, via the ‘zipping’ and ‘unzipping’ mechanism portrayed in diagram A.10.4. The lower section of $\vec{CD}$ is now parallel to the partial $\vec{C}\beta$ of the jog; since the reaction $\boxed{\vec{CD} + \vec{C}\beta \rightarrow \frac{1}{6}[\bar{4}\bar{5}1]}$ is not energetically favourable, an atomistically highly distorted region will be formed. This region may now act as a barrier to further glide of the lower section of dislocation $\vec{CD}$ on α , effectively pinning it along the length of the super-jog.

The upper section of $\vec{CD}$ is, however, only pinned where it intersects the top of the super-jog. Therefore, under the action of the external stress it may bow out on α as shown in (4). In bowing out, dislocation $\vec{CD}$ may be brought close enough to partial $\vec{\delta C}$ on δ_1 to prompt their reaction, which reaction proceeds as before and may produce another local constriction (5). This produces a closely spaced dislocation dipole $\vec{CD}/\vec{DC}$. In (6) the dipole is annihilated and the upper section of $\vec{CD}$ continues along $AC(\delta_1)$ to form $\vec{AD}$ as shown in diagram A.2. Dislocation $\vec{CD}$ is now in two sections, and the entire super-jog may be constricted.

The remaining upper section of dislocation $\vec{CD}$ continues to glide on α , forming $\vec{AD}$, towards the central jog complex shown in preceding diagrams (7).

Splitting of CD at acute super-jog on AC

1. CD gliding on β towards AC.
2. CD intersects AC and cross-slips onto α .
Causes local constriction.
3. CD intersects, and constricts top part of jog.
4. Lower section of CD pinned at jog. Upper section bows out under stress.
5. Upper section of CD reacts with ∂C .
6. CD/DC annihilate. Lower part of jog constricts.
7. CD split. Upper section glides towards node core.

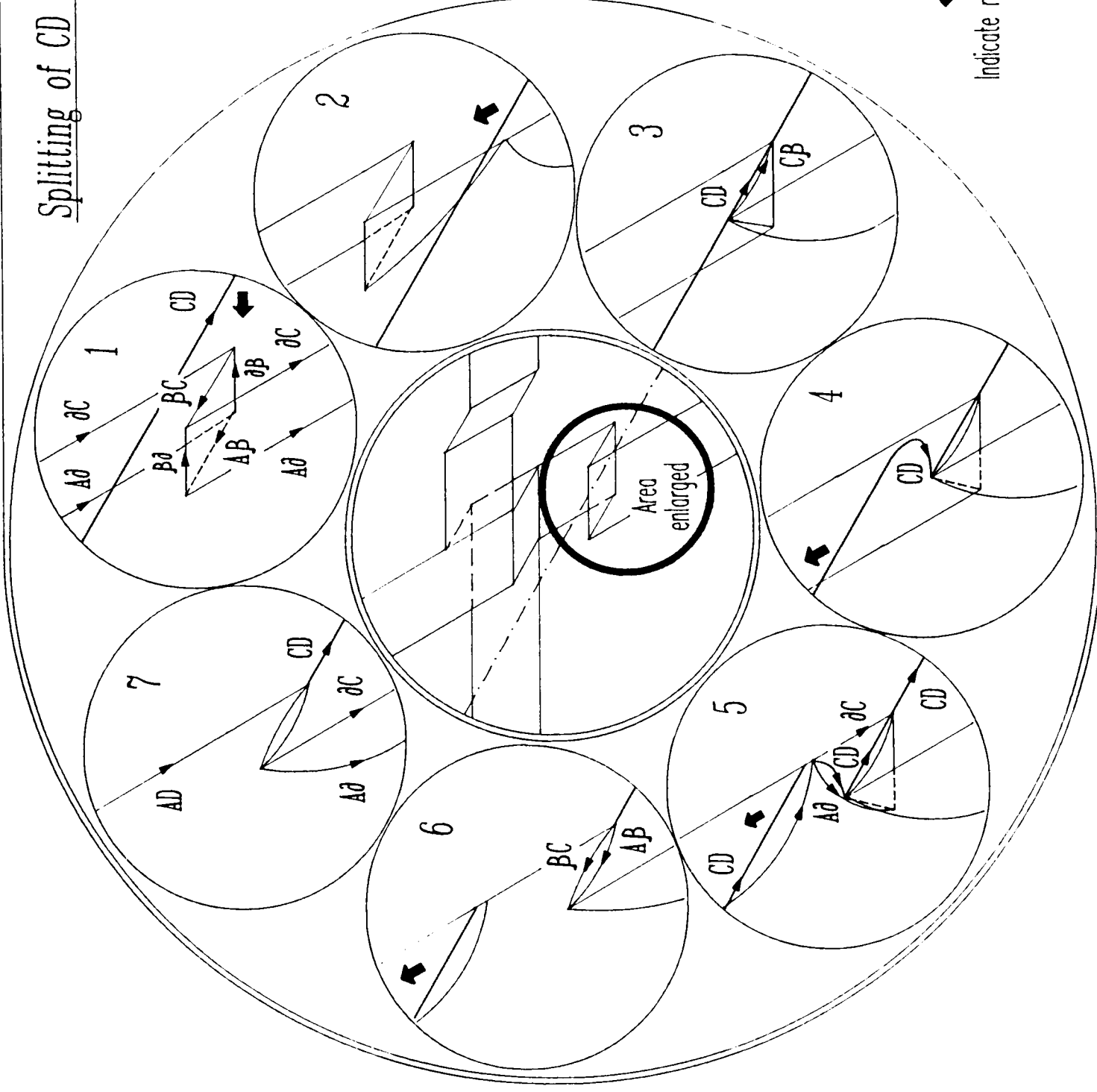
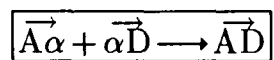


Diagram A.10: “Formation and glide of local constriction on $\vec{AC}$ by $\vec{CD}$ ”;

In diagram A.10 the possibility that a constricted section of line may be formed by glide, under stress, of a Lomer-Cottrell lock is suggested. Initially (1), $\vec{CD}$ is gliding on β towards $\vec{AC}(\delta)$. On intersection (2) a short length of ‘type 1’ (Lomer-Cottrell) barrier is formed, prompting cross-slip of $\vec{CD}$ onto α . A point like constriction is formed⁵⁸. ‘Type 1’ barriers have a very low resistance to collapse under external stress (Hirth & Lothe, p.803), and hence a temporary intermediate configuration similar to that shown in (3) may result. Prior to the complete annihilation of the barrier, however, the action of any external stress may lead to slight bowing-out of the trailing partial $\vec{\alpha D}$. In turn, this may prompt operation of the energetically neutral reaction:



i.e. the eventual formation of a Lomer dislocation. (The configuration shown in (3) involves the moderately energetically unfavourable reaction $\vec{\delta\alpha} + \vec{A\delta} \longrightarrow \vec{A\alpha}$; obviously this may be avoided by requiring constriction of the top section of the Lomer-Cottrell lock to form $\vec{AD}$ directly.)

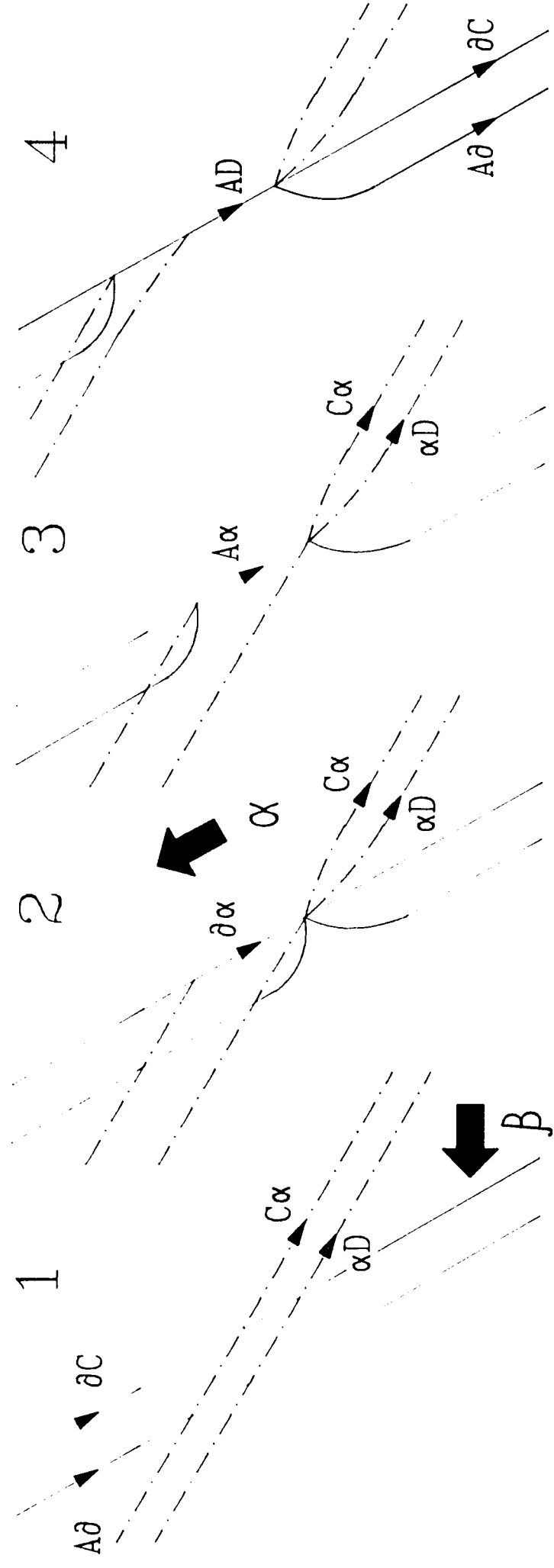
‘Type 1’ Lomer-Cottrell barriers are particularly stable against redissociation (‘unzipping’ - see Hirth & Lothe, p.804) as is occurring at the lower section of $\vec{CD}$. Additionally, Lomer dislocations commonly seem constricted in these materials, and therefore may be particularly stable. Hence a length of dislocation $\vec{AD}$ might precede, and may actually be *required* to precede, the ‘unzipping’ lower section of dislocation $\vec{CD}$ as the entire configuration progresses along α (4). It is possible that the length of the intervening section $\vec{AD}$ would be related to the level of external stress, or, in other words, to the speed of the zip.

References:

Karnthaler H P, Wintner E, 1975; Acta Met. 23, 1501

Whelan M J, 1959; Proc.Roy.Soc A249, p.114

⁵⁸This diagram (2) is exactly analogous to that shown in figure 22-14(c) of Hirth & Lothe (p.802), which corresponds to the configuration predicted by Whelan, 1959 and seen by Karnthaler and Wintner (1975; their figure 2).



← Motion of CD

Formation and glide of local constriction on AC by CD

