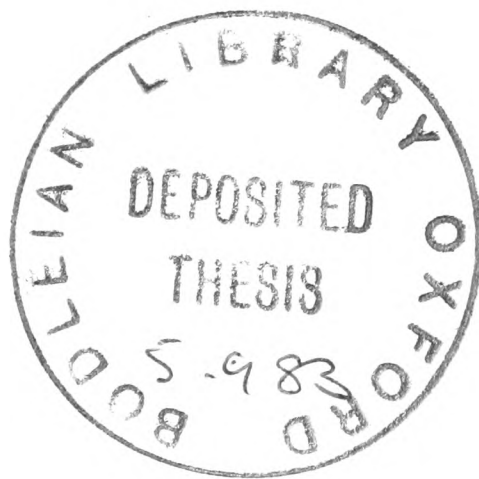


The deformation of bcc alloys

**A thesis submitted in supplication
for the degree of Doctor of Philosophy**



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A detailed study has been made of thermally activated glide between 373 K and 20 K for UHV annealed single crystals of two Nb based substitutional alloy systems containing 1 - 16 at.%Mo or 4 - 60 at.%Ta, in conjunction with a study of the deformation of UHV annealed single crystals of Nb between 4.2 K and 77 K. Whilst the addition of Ta had only a small effect on the properties of Nb as measured by activation volume and enthalpy and the temperature dependence of the flow stress, it produced a large increase in the low temperature yield stress and displaced the appearance of anomalous slip to lower temperatures, e.g. 77 K for the 10 at.%Ta alloy. Addition of Mo produced more rapid changes. Whilst the 1 at.%Mo alloy behaved like the Nb-Ta alloys, the appearance of anomalous slip was depressed to 113 K. Further additions appeared to suppress anomalous slip completely and radically alter the behaviour of the alloys. The thermodynamic analysis suggested that the more concentrated Nb-Mo alloys show a change in the rate limiting step at low temperatures, cf. a peak in the activation volume - effective stress curve. No solution softening was observed in the alloys. Complex transients were found for all the alloys at and below 77 K after changes in strain rate. The yield and thermal stresses for Nb deforming by anomalous slip were independent of temperature between 77 K and 50 K, only regaining a temperature sensitivity below 50 K. The importance of this for models of anomalous slip was discussed. Complex overshoots were observed after changes in the strain rate. Those observed at and below 20 K have been explained by reference to the change in specimen temperature produced by heat generation during dislocation glide.

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Preface.

The bcc metals and alloys have been the subject of intensive study over the last few decades and in consequence there now exists a large body of published material. Whilst the general behaviour was established fairly early, e.g. the marked sensitivity of the yield and flow stresses on temperature and strain rate, there has been considerable controversy over the interpretation of many of the experimental results. One of the major sources of disagreement has been the purity of the samples used, in particular the level of interstitial solutes, O₂, N₂, H₂ and C, for the deformation behaviour is very sensitively dependent upon their concentration.

The introduction of electron beam zone refining, whereby single crystals could be grown in a vacuum of 10^{-5} torr, resulted in a considerable improvement in the purity of the available materials and produced large changes in the observed properties, e.g. the appearance of a three stage work hardening curve and the lowering of the yield stress. Further reductions in the level of interstitial impurities were achieved by taking crystals prepared by the above method and annealing them just below their melting point in a vacuum of 10^{-10} torr. These more pure crystals, now containing 1-10 at.ppm interstitials, showed different properties to those crystals which had merely been zone refined, e.g. a yet lower yield stress and the appearance of anomalous slip.

This approach has been used successfully with several bcc metals, notably Nb and Ta, allowing one to study the behaviour of the 'pure' metal and thus to establish a reliable base from which to determine the effects of alloying, etc. Because the properties of the alloys are also sensitive to the presence of impurities, any study wishing to determine the effect of solute must study specimens of a

similar purity to those of the base metal. Whilst there have been a number of studies on such alloys, those which have looked at substitutional alloying have tended to examine particular features (1-6,12,16), e.g. solution softening, rather than establish data over a wide range of temperature and composition (7). The work on interstitial alloys has been more systematic and extensive (8-11,13-30,71). There is therefore a need for a study encompassing a variety of substitutional solutes and a wide temperature range, preferably including temperatures below 77 K for little is known about deformation behaviour in this region.

It is to this end that the present work has been directed. Single crystals of Nb-Ta and Nb-Mo have been prepared containing amounts of interstitial solute similar to the most pure base metal, Nb. Two solutes were chosen which would, it was expected, affect the deformation behaviour differently because of their dissimilar characteristics, e.g. chemical group, atomic size and elastic constants (table 10). Thus Ta, group VA, has a negligible size misfit parameter and a moderately large modulus mismatch, whereas Mo, group VIA, has both a large size and modulus mismatch. The effect of alloying was followed by determining the mechanical properties and thermal activation parameters of the alloys between approximately 20 K and 373 K and, where necessary, those of Nb (between 4.2 K and 77 K). In addition, the changes brought about in the slip morphology by the type of solute and its concentration, the temperature and the strain, were also monitored.

Some of the test results obtained in earlier work, for the Part II examination for the Honour School of Natural Science (322) have been included, with corrections, in the data presented in this thesis. The character of this data is discussed in section 1 of the Results, and in table 4.

Literature Review.

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Literature Review.

1. Introduction.

In the preface reference was made to one of the major sources of disagreement in understanding the behaviour of bcc metals and alloys, being the variation in the purity of the materials used. A second source of confusion has been the differences between the bcc metals themselves. Whilst the general patterns of behaviour are the same, e.g. the temperature dependence of the yield stress, there is a strong tendency for some specific properties to be restricted to a particular group of bcc metals. Thus, for example, all the group $\overline{\text{VA}}$ metals, V, Nb and Ta exhibit anomalous slip, although none is observed in any of the group $\overline{\text{VIA}}$ metals, Cr, Mo and W. As a consequence of this, the following literature survey is biased towards the properties of the group $\overline{\text{VA}}$ metals and their alloys. Where it is relevant, the behaviour of other bcc metals and alloys has been introduced, e.g. in the section on solution softening.

Despite the presence of some differences between the various groups of bcc metals, the general similarities in behaviour all point to a common factor peculiar to the bcc lattice. The idea that has been most successful in supplying this unifying factor is that the dislocations in the bcc lattice have a special characteristic which sets them apart from those in fcc structures. Since this special characteristic lies at the heart of any understanding of the behaviour of bcc metals and alloys, it is rational to start any survey with a consideration of this point.

2. Dislocations in the bcc structure.

The idea that the low temperature properties of bcc metals, e.g. the large rise in the yield stress below approximately $0.2T_{\text{melting}}$, were intrinsic to the metals, has produced many studies of the nature of the dislocation in the bcc structure.

Early models followed the work of Peierls (31) whose calculations indicated that a dislocation's energy could vary with position in the lattice. In these models a screw dislocation has to surmount a periodic energy barrier, the shape and size of which was assumed. Using a line energy model, the double kink nucleation energy may be calculated as a function of such parameters as stress, temperature and barrier profile (32,33). It can be shown that the Peierls stress is less for edge than for screw dislocations (34,35).

Hirsch (36) was the first to propose that the $\frac{1}{2} \langle 111 \rangle$ screw dislocation is dissociated into three non coplanar partials and that motion requires a sessile-glissile transformation. Edge dislocations cannot dissociate in this way and their cores are planar. Various possible dissociated configurations have been proposed (see 37 for a review), but in all of them recombination of the partials under the action of the applied stress and thermal fluctuations is necessary before the screw dislocation can move. After moving forward a short distance the dislocation may dissociate again into partials. This type of model has been treated theoretically using linear elasticity theory (38 - 41). The mean velocity of the dislocation, and also its effective slip plane, can be obtained from the vector sum of these small steps using rate theory and again reasonable agreement with experimental results can be obtained (38,40,41). However, the model has several unsatisfactory features such as the use of linear elasticity at small distances from the dislocation core. Also, the stacking fault energy loses its physical significance because of the large values it has to take when used as a free parameter in fitting the theoretical predictions to experimental results.

To avoid these difficulties, direct space 'atomistic' models were developed (e.g. 37,42,43). Using a computer, 'atoms' are arranged

in a model crystal, approximately $45b \times 45b \times 1b$, so that a dislocation is present. Generally, empirical interatomic potentials are used and although these are matched to some physical properties, they are not expected to represent any particular metal. The model crystal is then allowed to relax in the computer so that the 'atoms' occupy their equilibrium positions.

Although the results of these studies are complex, they show that the general idea of Hirsch (36) is correct in that the $\frac{1}{2} \langle 111 \rangle$ screw dislocation has a non coplanar configuration but it cannot be described by partials and stacking faults (e.g. see 56). Rather it should be considered as a three-fold non coplanar distribution of the Burger's vector with a generalised stacking fault (high energy). However, for non screw dislocations, one finds planar structures which may be approximated to by linear elastic models (44-46).

For any bcc metal, it seems that slip is easier when the dislocation motion produces shear on the $\{112\}$ planes in a twinning rather than an anti-twinning sense. This property could be introduced into the elastic models by an asymmetric splitting of the dislocation on the $\{112\}$ planes. In the atomistic models, the effect is seen directly in that the displacements in the dislocation core are larger and wider in extent when they are in a twinning rather than an anti-twinning sense.

The motion of the dislocation can be followed in the computer by 'stressing' the crystal. In the case of a screw dislocation there are significant alterations to the core structure and the movement is complex (37,48-50). For edge and mixed dislocations, with their planar cores, motion is simpler since core structure changes are relatively small (45,47). The studies indicate that some features of screw dislocation motion may be expected to be common to all bcc metals. These include the twinning-anti-twinning asymmetry, and the low mobility of screw relative to edge

dislocations. However, many features are sensitively dependent upon the interatomic forces, and may be expected to vary from metal to metal. These include the orientation dependence of the slip geometry and flow stress, the influence of other components of the stress tensor and the tendency for twinning (48).

The above atomistic models only apply at 0 K, for at finite temperatures the screw dislocations are thought to move by thermally activated double kink nucleation (41). Atomistic modelling of this process (51) can provide a consistent description of why, in tests on crystals, the asymmetry decreases as the temperature rises, until at high temperature it is absent (e.g. 18, 20, 52). Such models do contain arbitrary, but at present necessary, assumptions, e.g. choosing the transformation path. There have also been attempts to improve the theoretical models by removing some of the restrictive boundary conditions used in earlier models, but the quantitative results should still be viewed with caution (53-55).

3. Stress Strain Curves.

The effect of temperature on the deformation of bcc metals is seen clearly in the case of the stress strain curves, for below a temperature (T_c) characteristic of each metal the yield and flow stresses increase rapidly and the shape of the work hardening curve alters significantly (8, 22, 27, 57-69, 74). From in-situ deformation experiments (see 'in-situ studies' section 14) a pronounced change in dislocation behaviour is also observed in the vicinity of T_c , e.g. ≈ 240 K for Nb and Fe, ≈ 350 K for Mo (81). Some authors put the transition temperature at a much higher value, e.g. 550 K for Mo, using the criterion of when the yield stress becomes independent of temperature (78).

At temperatures above T_c , three-stage stress-strain curves are found for highly pure metals (e.g. 8, 22, 82). However, some authors

(e.g.66,78) consider there to be only two stages, with stage II being vanishingly short (63,66,82). As the temperature decreases towards T_c , the length of stage I increases, θ_I increases, whilst θ_{II} decreases. In the region of T_c the distinction between stages I and II disappears, and a quasi-parabolic curve is observed (63,66,82). There is, in addition, an orientation effect on the work hardening and yield behaviour. At high temperatures this effect is absent, but it becomes pronounced as the temperature is reduced towards T_c , and marked below T_c (e.g.8,22,67).

Apart from the effect of substitutional solute on the magnitude of the yield stress (which is generally to raise it) there is a modification of the work hardening behaviour (2,4,72-77). In the temperature region where the pure metal shows three stage hardening, small additions of solute increase θ_I and the length of stage I, and decrease θ_{II} , e.g. Nb-10 at.%Ta (74), Nb-2.5 at.%Mo (73), both at 290 K. Larger amounts of solute result in quasi-parabolic curves at all temperatures, but with the work hardening rate increasing with the amount of solute (e.g.74). A solute which is a strong hardener, e.g. Mo or W in Nb (4,74) will alter the rates faster than will a weak solution hardener, e.g. Ta in Nb (74). Yield drops may also appear as the amount of solute increases or as the temperature is lowered (e.g. 72,74). It has also been observed that substitutional alloying, in this case Fe-Si, can depress the temperature down to which three stage hardening can appear (72,80,83). As alloying lengthens stage I, this effect can only be observed by straining to large extensions, e.g. 100% shear strain for the Fe-Si alloys.

Interstitial solute affects the three stage curve in very much the same way as does substitutional solute (8,22,70) in that θ_I is raised, θ_{II} is decreased, and stage I lengthened, e.g. Ta-N (22).

At lower temperatures where quasi-parabolic or linear curves are observed, the general effect is to lower the work hardening rate so that at high concentrations and low temperatures it may become negative, e.g. Nb-300 at.ppm N at 90 K (8). However, the effect does depend upon the orientation of the stress axis (e.g.8,22).

4. The yield stress.

4.1 The pure metal.

The yield stress for the 'pure' bcc metal is very temperature dependent below a temperature characteristic of each metal, e.g. 300 K for Nb (8). As the temperature is lowered, the yield stress rises : e.g for Nb it rises from 600 g/mm^2 at 300 K to 35 kg/mm^2 at 4.2 K (8). The relationship between stress and temperature is usually a smooth one, although there may be a plateau in the yield stress at low temperature if anomalous slip occurs (8,101).

4.2 Substitutional alloys.

Substitutional alloying affects both the high temperature 'plateau' as well as the temperature dependent part of the yield stress. The effects are usually considered separately.

Many attempts have been made to correlate the high temperature plateau stress with solution hardening theories (discussed below in section 5), although the temperatures of testing differ considerably between different authors : e.g. 300 K to 1400 K (74,89). It should be noted that the theories were formulated for dilute alloys under conditions of no thermal activation. Also, many of the results come from alloys of up to 10 at.%, when the solutions are not dilute. As can be seen from the tables which follow, there is little agreement either as to the concentration dependence or the dominant factor producing the hardening (see also 80).

	Nb	Ta	Reference
$c^{1/2}$	Ta* V*	Re* Nb* Mo* W*	87;77,92,94,96
$c^{2/3}$	W Re Mo Ta	Re Nb Mo Hf W Ru	89; 89-91,96
C1	Hf† Ta+ V Zr W+ Mo+ Pd+	Nb Mo† W† Hf† Zr† Nb† Re†	2-4,74,84-86, 95; 93,183

+ UHV annealed material

* work supporting a $c^{1/2}$ relationship before Labusch's $c^{2/3}$ paper published

† data from indentation tests

	Nb	Ta	Reference
ϵ_a	Ta V Zr W Mo Hf	Mo W Nb Hf Zr Re	2,74,84,85 : 77, 94
ϵ_μ	Re+ Ta+ Mo+ W+	Mo+ W+ Nb+ Hf+ Ru+ Re+	89 : 92,93,96
ϵ_a and ϵ_μ	Ta W V	Re W Mo Hf Re Nb	88 : 90

+ uses K_{33} , not μ .

The functional dependence is usually arrived at by doing a best fit between the theory and the data, as the theory has adjustable parameters. Through using different combinations of the elastic moduli, one can alter the value of ' ϵ_μ ' considerably: e.g. Mordike et al (89,91,92, 96) use K_{33} after Head (98), a composite modulus used in the description of the stress field for a $1/2 \langle 111 \rangle$ screw dislocation in an anisotropic medium. However, this has been criticised (108,183). Because of

the flexibility of the method, and because of difficulty in fitting the results, several authors have concluded that the theory is inadequate to explain the results (e.g. 86,88,94 or for Fe based alloys 97).

Whilst solute additions always raise the athermal plateau (55) the same is not necessarily true at lower temperatures. The topic of solution softening is discussed in section 17. However, the presence of solution softening does make it difficult to ascertain what effect substitutional solute does have on the yield stress (e.g. 90). Studies on alloys which do not exhibit it have generally shown that the yield stress increment is proportional to "c" for temperatures above 77 K (e.g. Nb-Mo 1,74 ; Ta-Re 14). The effect of alloying on the low temperature yield stress has usually been discussed in conjunction with a thermodynamic analysis (74,99-101,218). Solute is seen as modifying the Peierls stress or the splitting of the dislocation, and thus the transformation paths for double kink nucleation. Suzuki's model for hardening (160), based on the solute impeding kink motion, has been criticised (86,100,218).

4.3 Interstitial Alloys.

As interstitials have a strong effect on the yield stress of bcc metals, the failure to work with high purity materials, either in alloys or nominally 'pure' metals, has led to contradictory results and confusion. However, there now exists a large amount of data for high purity binary metal - interstitial alloys (Nb 8-11,71,102,164,165; Ta 10,18-20,22,70,103 ; V 24,102,166-8). For a discussion of ternary solid solutions, or Fe based binary solutions, see "solution softening", section 17.

The behaviour of Nb, Ta or V polycrystals or single crystals orientated in the centre of the unit triangle doped with O, N or C is very similar. At high temperature the yield stress depends linearly

on the concentration e.g. Ta-N at 373 K (19). As the temperature is lowered, initial solute additions have a greater effect on the yield stress, but as the concentration increases, the previous linear dependence reappears. The lower the temperature, the greater the amount of solute that must be added before the linear hardening appears, e.g. 300 at.ppm is necessary in Nb deformed at 90 K (8). A slightly different approach has been to measure the temperature dependence of the flow stress in Nb-N by temperature change experiments (8). Unlike the yield stress, the concentration dependence of flow stress is very small, especially at low concentrations, e.g. 30 at.ppm N.

A variety of hardening mechanisms have been suggested. The linear hardening has been ascribed to the Snoek effect (22,103,164,167) or Mott-type hardening (166), with the non-linear region being caused by a Fleischer-type mechanism (22,103,164,166). In V-O, the data has been fitted to a $c^{2/3}$ (Labusch 126) dependence for all concentrations and temperatures (168). The model of Ono and Sommers (147) has been suggested as a general explanation (166,168), whilst observations on the concentration dependence of the flow stress of Nb-N have been taken to support the model of Kubin and Louchet (148). It should also be remembered that alloying may alter the operative slip system (e.g. 8,22,71).

There is also an orientation effect on the rate of hardening (8,18,22,71). For $\chi \approx + 30^\circ$ (for definition of χ see "crystallography" section 8) the same general dependence on temperature and concentration is observed for Ta or Nb-N (8,18,19,22), but the hardening rates at low temperature are smaller than for $\chi = 0^\circ$. The same is true to a lesser extent for $\chi = - 30^\circ$ (18,19,22). The effect of alloying is to promote maximum resolved shear stress (m.r.s.s.) slip and remove or significantly reduce the asymmetry of the yield stress present in the pure material (8,18,19,22). However, recent work on Nb-O (71) has suggested that for $\chi = + 30^\circ$, alloying promotes softening (see "solution softening" section 17). It should be noted that the yield stress for their

Nb of this orientation is 50% greater than that found in other work on high purity Nb (e.g. 59).

4.4 Effect of orientation.

For pure metals at high temperature the shear yield stress shows no dependence on orientation (χ) or testing mode, tension or compression, e.g. Ta at 354 K (20). As the temperature is lowered, an asymmetry appears which does depend upon the testing mode. In compression it is found that $\tau_y (\chi = -30^\circ) > \tau_y (\chi = +30^\circ)$. In tension the inequality is reversed (Nb 58,172 ; Ta 18,22,173,175 ; Fe 174,176,177 but see also 67 ; Cr 170 ; W 169 ; Mo 171). For $\chi = 0^\circ$, the yield stress lies either between that for $\chi = \pm 30^\circ$, or at a lower value. In Fe, this asymmetry exists down to 4.2 K (174). With Nb which, unlike Fe, shows anomalous slip, the asymmetry in the yield stress seems to disappear at 77 K when anomalous slip becomes dominant (59). However, cyclic testing (tension - compression) reveals that there is a dependence on the sense of deformation even for anomalous slip in Nb (354). It should also be noted that for a given orientation the same slip system may not operate in both tension and compression (e.g. 22,173). This twinning-anti-twinning asymmetry has been explained by reference to the asymmetrical displacements in the core of the screw dislocation (see "Dislocations in bcc structure" section 2).

There have been no systematic studies on the effect of substitutional alloying on the yield stress asymmetry, but the data which does exist indicates that it is not significantly altered, even though the operative slip plane may be changed (Nb-W 179 ; Fe-Si 180-182 ; Ta-Re, Nb 173). However, the effect of interstitial alloying appears to be to reduce the asymmetry (see preceding section).

The effect that the magnitude and sense of the stress normal to the slip plane could have on the deformation was initially inferred

from low temperature $\psi(\chi)$ curves (138 - see also "crystallography" section 8). Work on Fe (185), Ta (18,52,64) and Fe-Si (184) have correlated a rise in σ_n with a rise in the yield stress, and changes in the stress-strain curve. The rise in the yield stress is linear with σ_n , but again it is dependent upon orientation (e.g. 184). It has also been observed that a large σ_n inhibits twinning (e.g.64).

5. Solution hardening theories: no thermal activation.

Any solution hardening theory has to consider two basic problems. These are the nature and calculation of the interaction energy between the dislocation and the solute, and the statistics of the dislocation motion through the array of barriers created.

Most theories treat the interaction as being an elastic one, with a resulting force between dislocation and substitutional solute atom depending upon the relative sizes (ϵa) and elastic constants ($\epsilon\mu$) of the matrix and solute atoms. In the case of interstitial atoms, the above parameters are not used but rather the magnitude of the tetragonal strain produced by the interstitial (see 80 for a review). The interactions are usually evaluated within the framework of isotropic linear elasticity theory for the case of a single solute atom - undissociated dislocation (104,106-108,152) although more sophisticated calculations do exist (109,110,113-115). The balance between the two parameters ($\epsilon = \epsilon\mu + \alpha \epsilon a$) is usually determined by matching theoretical and experimental results, although in the theory of Labusch (112) the balance is specified. Other types of interactions have been proposed, e.g. electrical or chemical (105,111) but they are generally considered to be small in this context.

The statistical problem of how the dislocation moves through an array in the absence of thermal activation has attracted much attention. At its heart is the problem that in a random array the number of obstacles

met by a dislocation bowing out between them depends upon, amongst other factors, the strength of the obstacles and the stress. Early models (123, 124) using simple average methods for weakly repulsive point obstacles arrived at a $c^{1/2} f_m^{3/2}$ dependence (f_m = maximum force between solute and dislocation). A dislocation segment pinned at both ends was seen as breaking free from an obstacle between the pinned ends, moving forward, and being stopped by another obstacle. This was supported by the first computer simulations of the process (116,117), even for the case of strongly repulsive obstacles. Later work with this type of model has refined the statistics and allowed a far greater amount of useful data to be extracted from the simulations (118-122,125,162,163).

However, if one treats the obstacle as having a finite interaction range then the dislocation segment which moves forward lies in the potential wells of a large number of obstacles, and forward motion alters the position of the dislocation with respect to many of these. When the theory is evaluated one obtains $\tau_y \propto c^{2/3} f_m^{4/3}$. If the model is taken to the limit of point obstacles, it reduces to $c^{1/2} f_m^{3/2}$ (but also see 127-129). An array of this type of obstacle has also been studied in a computer simulation (122,130).

These models only strictly apply to dilute solutions. In solid solutions of > 0.1 at.% the stress fields of adjacent solute atoms overlap (158). Some theoretical approaches have taken the dislocation to move as a straight unit (131,132,156,157). Given the length of dislocation that moves (L), it has been possible to obtain satisfactory agreement for fcc substitutional alloys at 0 K from a relationship of the type $\tau_y \propto L^{-1/2} (c(1-c))^{1/2}$ (131,132). However, an alternative treatment allows the dislocation to bend, and then calculates the barrier to kink motion over the obstacles (133). A $\tau_y \propto c^{2/3}$ dependence is

found, and also good agreement is claimed for fcc substitutional alloys at 0 K. Both these approaches ascribe the hardening to size effect interactions with edge dislocations.

6. Solution hardening theory : with thermal activation.

Both the point and extended obstacle models have been modified to include thermal activation. The former model has been used extensively in computer simulations, and work has been done studying the effect of temperature, stress and a mixture of barrier strengths on the dislocation behaviour (134-136,139-142). Overall, the effect on the yield stress is that a rise in temperature is equivalent to a softening of the obstacles (149). The latter model, due to Labusch, indicates that as the temperature is raised from 0 K the effective obstacles become clusters of solute atoms, but that the concentration dependence of the yield stress remains as $c^{2/3}$. However, the plateau stress becomes dependent upon the velocity of the dislocations. The model has been applied to fcc materials where the temperature dependence of the yield stress is small (143).

With bcc metals there is already a temperature dependent periodic barrier to dislocation motion before any solute is added. How the solute affects the dislocation motion in this potential (i.e. at low temperatures) is a moot point (see 80 for a review). Early models attributed the whole of the low temperature strength to interstitial - dislocation interactions (144,145). Later models have had varying approaches as to the effect of adding interstitials to the metal. Some have considered the interstitials to be just barriers which reduce the free length of screw dislocations and impede the sideways motion of kinks, thus producing hardening (147,148 - see also "solution softening" section 17). Kubin and Louchet based their model on inferences made from in-situ deformation experiments (148). Even if the obstacles are

considered as having positive and negative interactions with the dislocations, a continuum treatment, coupled with a computer simulation, indicates hardening at all concentrations and temperatures (146). Other authors consider the torque around an interstitial can aid double kink nucleation (150 after 151). This leads to solution softening at low temperature (see "solution softening" section 17 for a fuller description of the model). However, this has been criticised on the grounds that the torque around an interstitial is of no consequence (152). Another model, due to Wuthrich and Frank, suggests that the effect of interstitials will depend upon the temperature, with hardening at high and very low temperatures, and softening at intermediate temperatures (153 : see "solution softening" section 17, for fuller description).

Substitutional alloys have received little theoretical treatment. It has been suggested that the observations can be correlated with the change in elastic modulus (154), or, more generally, the interatomic potential (155), but, where quantifiable, this does not seem to be a satisfactory solution (96,154,159). Some models have taken thermally activated kink motion over solute atoms as the rate controlling step (e.g.56,160,183), and satisfactory agreement between theory and some experimental results has been obtained. Some other studies have considered this process as a function of the hardening parameter ϵ , and it is seen that hardening always occurs. However, if double kink nucleation is rate controlling, then one may get softening (161).

7. Electron Drag.

Materials being deformed whilst in a superconducting state show a rise in the flow stress when the superconductivity is suppressed by a sufficiently large magnetic field. Although the effect was first observed in fcc materials, some work has been done on bcc materials

(Ta 186 ; Nb 5,187,188 ; Nb-Mo 5). The effect is attributed to the change in the viscous drag of the electrons on the dislocations as the material's state alters. In fcc materials this can be explained by inertial effects (189) in underdamped dislocation motion (190).

However, this model supposes that the obstacles to dislocation motion which have to be overcome by thermal activation are small. For bcc metals, where this is not so, it has been suggested that the effect is caused by a change in the double kink nucleation energy (5,186).

8. Crystallography.

It is generally accepted that the deformation in bcc crystals is caused almost exclusively by the motion of dislocations with $\frac{1}{2} \langle 111 \rangle$ Burger's vector (78,198). However, the slip plane shows a dependence on many factors, e.g. orientation, temperature, sense of deformation and composition. The plane will be in the $\langle 111 \rangle$ zone, but it is not necessarily crystallographic. This is in accord with the observations made from atomistic models of screw dislocation motion which indicate that movements larger than one atomic step may be composed of short segments on different slip planes (e.g.37).

In high purity metals (e.g. Nb or Ta) the slip is essentially of $\{110\}$ or $\{112\}$ at low temperatures ($T < 0.15 T_m$) (22,60,172,173,178). On the basis of the Schmid factor one can therefore expect a change between these slip planes on moving the axial orientation of the crystal across the unit triangle (193). It is observed that at room temperature these slip boundaries are displaced in the sense that indicates a twinning-anti-twinning asymmetry in the slip, i.e. slip on a $\{112\}$ plane in the anti-twinning sense requires a larger stress than slip in the twinning sense (e.g. 60,172,178 - see also "yield stress - effect of

orientation" section 4.4). As the temperature is lowered the displacements of the boundaries from the positions predicted by Schmid's Law tend to increase (172,173,178). This can be rationalised by considering the sense of the displacements in the core of the screw dislocation (e.g.37). However, the picture is complicated by the appearance of anomalous slip (e.g. 178,195,196 - see "anomalous slip" section 16) and other observations, e.g. for Ta no $(\bar{1}\bar{1}2)$ slip was observed at 197 K for $\psi = -30^\circ$ in either tension or compression (22), rather slip occurred on the $(\bar{1}01)$ primary plane. In group VIA metals, e.g. Mo, studies are complicated by the large number of slip systems that operate at low strains (199).

In alloys, where the slip plane may change continuously with orientation, the usual method of description is the $\psi(\chi)$ curve (e.g. 191,192,198). ψ is the angle between $(\bar{1}01)$ and the pole of the observed slip plane and χ is the angle between $(\bar{1}01)$ and the plane of maximum resolved shear stress (m.r.s.s.) in the $[111]$ zone.

The general effect of substitutional or interstitial alloying is to promote m.r.s.s. slip, especially as the concentration is increased (Nb-Mo 73 ; Ta-N 22 ; Ta-Re,Nb 173 ; Fe-Si 138). As the temperature is lowered, however, the deviation from m.r.s.s. towards $(\bar{1}01)$ slip increases, especially for $\chi < 0^\circ$ in compression (e.g.73,138, 173). It is observed also that as the temperature is decreased, the deviation from the relationship $-\psi(-\chi)_{\text{ten.}} = \psi(\chi)_{\text{comp.}}$ increases (very clear in 138). The failure of this relationship, which should hold even if the twinning-anti-twinning asymmetry is present, has been ascribed to the effects of the stress normal to the slip plane (e.g.138) (see also "yield stress - effect of orientation" section 4.4.).

As with the highly pure metals, the general trends are broken by the appearance of anomalous slip in Mo-Re, Nb deformed at 77 K (194).

9. Morphology.

Work on the morphology of the slip line structure has shown that it is sensitively dependent upon many factors, but primarily upon strain, temperature and alloy composition (for reviews see 56,78,80). In the case of pure metals, the most systematic work has been done on Nb and Fe at $T > T_c$ (58,60,66,83) where T_c is approximately the temperature below which the yield stress becomes significantly temperature dependent and three stage stress-strain curves are replaced by quasi-parabolic curves (see "in-situ" section 14).

In stage I, the traces on the side surface are very uniform and straight (the top and side faces are the faces of the crystal where the primary edge and screw dislocations respectively are parallel to the surface). The top face shows a tightly packed network of wavy slip. In the transition between stages I and II conjugate slip appears on the side face in bands parallel to, and between, the primary slip traces. The number of these bands saturates after only a small strain increment (66). However, there is often considerable overshoot ($10^\circ - 15^\circ$) of the double glide boundary before the secondary system operates, and the observed system may not be the expected one (58,209). On the top face 'hills' and 'valleys' appear in the wavy slip, corresponding to positions where conjugate slip is absent or present on the side face. A little later in the transition, coarse lines of conjugate slip appear. Late in stage II, the conjugate bands break through on the side face, giving an interlocking array. In stage III, the same is seen on the top face (58,60,66,78,178). Incremental strain and polish methods have shown that in stage II the strain is carried in discrete bands on the primary slip plane, and the strain concentrated in each band simply increases with deformation (210). The picture is altered little at higher temperatures or different orientations (58,66,205,206).

Less systematic work has been done for $T < T_c$. A general observation is that the slip becomes much finer and less wavy, until it is virtually invisible even with Nomarski contrast (27,58,64,202,205-208). An exception to this is the appearance of anomalous slip in V,Nb and Ta, where the slip is crystallographic and may be coarse (see "anomalous slip" section 16).

Very little work has been done on interstitial alloys (14,70,200,201). Only the general comment can be made that as the concentration increases or the temperature decreases, the slip becomes coarser. There is also a tendency towards slip on the m.r.s.s. plane (see "crystallography" section 8).

Although substitutional alloys have received rather more attention than interstitial alloys (14,64,72,73,173,202-204) the effects of alloying are often only described as coarsening the slip. However, more systematic work on Nb-Mo (73) and Fe-Si (72,202-204) has been reported. On yielding, both Nb-Mo and Fe-Si show a stress drop. During this, slip bands are seen to form (e.g.73,203). A decrease in temperature, or an increase in concentration, increases the strain concentrated in each slip band (73,203,204). As the strain increases the surface becomes filled with slip bands, either by more forming (73) or by the original bands broadening (202,203). The slip within the bands is fairly fine (202). Testing in tension or compression may alter the exact morphology of the bands, depending on the alloy system in question (173). How long this process takes depends upon the temperature and composition. Lower temperatures or higher concentrations increase the strain necessary to fill the surface with slip bands : e.g. in Fe - 3 wt.%Si at 300 K the surface is covered after $\approx$ 1% strain, but at 200 K it is not filled until 20% shear strain (203). Polish and reformation tests show that the slip is now uniform and fine over

the whole crystal and it remains so with further straining (202). However, if the solute concentration is not too large, then at large strains the slip structure on the top face can alter to branched and wavy bands : e.g. Fe-2 wt.%Si deformed at 295 K to $\gamma = 127\%$. Unlike the bands at lower strains which have a fixed strain per band, these bands, which do not broaden or multiply, carry all further strain (202).

The effect of solute in promoting the appearance of slip bands has been attributed to the effect of solute on the dynamics of yielding and the ease of cross-slip (73). This is coupled with the increased stability of screw dislocation arrays in materials with a large τ_{μ} because of the smaller 'capture' distances (proportional to τ_{μ}^{-1}) for the screw dislocations (80,202). The effect of strain on the slip structure is dealt with in "work hardening" section 12.

10. Thermodynamic Analysis.

Thermal activation parameters have been determined using conventional analysis (221-223) for several bcc metals and alloys. (Nb based 8, 11, 74, 84, 100, 101, 211, 215, 216, 218 : Ta based 63, 94, 99, 154, 186, 207, 219 : Fe based 27, 28, 67, 159, 180, 200, 212-215 : V based 220 : see also 224). These are usually expressed as functions of the thermal stress (τ^*), where $\tau^* = \tau_y - \tau_{\mu}$. The long range internal stress (τ_{μ}) is temperature independent, except via the temperature dependence of the elastic constants. It is often taken as being equal to the yield stress at the athermal plateau (e.g. 225, 226). For bcc metals, τ^* is independent of strain. This is shown by the lack of any strain dependence in the strain rate sensitivity (e.g. 8), or by direct evaluation of τ_{μ} and τ^* : e.g. on Fe or Fe-Si (180, 225). Most authors use $\tau^*(T) = \tau_y(T) - \tau_{\mu}$. But it has been shown that the temperature dependence of the flow stress, from temperature change tests, is not at all similar to the temperature dependence of the yield

stress in Nb and Nb-N alloys (8). For substitutional alloys, however, the two dependencies are the same (7,94).

In the pure metals, both the activation volume (v^*) and the activation enthalpy (ΔH) are found to depend strongly on the thermal stress, decreasing as the stress increases (8,99,207,220). They also show a small orientation dependence (8,214, but see also 67). The dependence on τ^* , and the magnitude of v^* , approximately $10 - 20 b^3$ at large stresses, have been taken as strong support for the Peierls-Nabarro or dissociated dislocation models (see "Dislocations in bcc structure" section 2). Further support for Duesbery's dislocation model (41) has come from work on Nb and Fe which show a small peak in the $v^* - \tau^*$ relationship at intermediate stresses (27,28,213). However, other work on Fe has not shown this (67,200,214,215 : see also "Solution Softening" section 17). At very low temperatures, below 20 K, a deviation from Arrhenius behaviour has been seen for Fe and Ta (186,207,212). This has been attributed to a quantum effect on the vibration mode of the dislocation overcoming the Peierls potential.

In general, substitutional alloying increases ΔH and v^* over the values for the pure metal at all stress levels (11,63,74, 84,99-101,218-220). The greater the effect of the solute on the yield stress, the larger the effect on the above parameters. If solution softening occurs, then a reduction of ΔH and v^* may occur (e.g. 159,219). However, in alloys of Nb with Mo, Re or W, the strain rate sensitivity - temperature relationship is split into two peaks, and a very large peak is observed in the $v^* - \tau^*$ curve (100,101,218). The solute is here thought of as affecting the saddle point of the activated dislocation, and this is coupled with a mechanism change at low temperature, as in the pure metal (101).

In Fe-Si though, the results are not ascribed to the motion of individual dislocations, but rather to groups (180).

Interstitial alloying, although it affects the yield stress quite strongly, has little effect on the activation parameters except at large concentrations (8,11,27,28,165,200,215-217). Then $\Delta H - \tau^*$ is raised, and the peak in $v^* - \tau^*$ is eliminated (e.g. 8,11,27,165). As with substitutional alloys, solution softening is reflected in a lowering of the $\Delta H - \tau^*$ curve (e.g. 215-217).

11. Dislocation structures.

The dislocation structures characteristic of the three stages of the stress strain curve for the pure metals above T_c have been investigated extensively. In many ways it is very similar to that for fcc metals (56,80,232). In stage I, very few screw or secondary dislocations are observed. Rather there are local accumulations of edge dislocations and edge dipoles (75,172,178,209,229). In the stage I - II transition the density of secondary dislocations increases (229) and tangles and multipoles begin to appear (178). As the strain increases, complex tangles and sheets of dislocations, parallel to the primary slip plane form with many secondary dislocations in them (210,219). Associated with these sheets are lattice rotations (209). Ferromagnetic measurements indicate that considerable internal stresses occur in this stage, unlike stage I (234,235).

For $T < T_c$, the three-stage stress-strain curve disappears, and significant changes in the dislocation structure are observed. The structure is dominated by jogged primary screws or near screw orientation dislocations. There are tangles and prismatic loops homogeneously distributed, with some $\langle 100 \rangle$ dislocation reaction products (14,17,75, 209, 229,231). As the strain increases, the density increases, as does the number of tangles (229,230,233), but the structure remains uniform.

In Fe secondary slip may occur from the start of deformation (229,230). The development of the structures has been discussed within the context of the temperature dependence of screw dislocation motion and the ease of cross-slip and annihilation (e.g.80,209).

Systematic work on substitutional and interstitial alloys as functions of temperature, composition and strain is lacking. The most detailed is that of Foxall and Statham (137), who studied the variation of dislocation structure as a function of density in various Nb-Mo or Re alloys at different temperatures at a shear strain of $\approx 7\%$. As with all the alloys studied, the dislocation distribution is very inhomogeneous, and they approximate an increase of dislocation density to a local increase in strain. For Mo concentrations of less than 8.5 at.%, the low density structure consisted of glide loops elongated along [111]. The ends of these elongated loops were of mixed dislocation character. As the temperature was lowered, the length of the initial loops parallel to [111] increased. There was a gradual transition towards the high density regions (bands) where clusters of edge dipoles and small elongated loops, with prismatic loop debris but very few secondary dislocations, were observed. This transformation to a dipole structure occurred at higher densities as τ^* increased, irrespective of whether this was caused by an increase in solute concentration or by a decrease in the temperature. For Nb-16 at.%Mo or 9 at.%Re, deformed at 295 K, the low density region had long cusped screw dislocations and debris loops, but the squared ends of the loops contributed large lengths of near edge dislocations. The higher density regions were dominated by short edge dipoles and elliptical loops. Again, there were very few secondary dislocations (see also 76,228). This observation, that alloying promotes long screw dislocation between high density regions which have few secondary dislocations, is seen also in Ta-Re deformed

at 77 K to 2% shear strain (14). Here alloying increased the density of screws, and reduced the secondary dislocation density and the number of debris loops. Similar observations have been made on a Ta-N alloy (14,17) and a Ta-Re-N alloy which showed solution softening (14).

The low temperature structures were rationalised by considering the effect of increasing the friction stress, either by alloying or by lowering the temperature, on the ease of cross-slip and annihilation of the screw dislocations (14,137). The edge - screw dislocation velocity ratio was considered also to be reduced by alloying (14,137). Similar reasoning was applied to the production of the dipole arrays in the high density regions (14 : see also 75,228). Qualitative differences between substitutional and interstitial alloying were attributed to the different magnitudes of the solute - dislocation interaction for edge and screw dislocations (14,17).

12. Work Hardening.

Although bcc metals can show a three-stage stress-strain curve if deformed above a certain temperature (approximately T_c), there are differences which do not allow a simple transfer of fcc hardening theories (see 79,232 for recent reviews of the subject). However, it has been suggested that stage II - III is similar to stage III in fcc metals (232,242). It should be noted that some authors do not distinguish separate stages II and III, and designate them as stage II/III (66,79,83).

In stage I, the homogeneous slip lines and the similarity of the dislocation structure to that of fcc metals, together with the shortening of stage I as the temperature is raised, have been taken to indicate that the low rate of work hardening is determined by elastic interactions between dislocations (79). However θ_I is significantly temperature dependent which is not the case for fcc metals. Stage I

is terminated by secondary slip (60,229), although in Nb the double glide boundary is overshoot by up to 15 ' (e.g. 58). This has been ascribed to latent hardening of the secondary system. A model based on the stresses from a pile-up has been used to explain the observed secondary system (58).

In stage II and III (or II/III), the slip becomes very much more inhomogeneous. Surface studies have shown that during these stages secondary slip is concentrated into bands which are 'fixed' in position in the crystal (66). Internally, dislocation sheets form which are approximately parallel to the slip plane. Throughout these stages the long range internal stress rises, e.g. as indicated by strain rate cycling and ferromagnetic measurements (225,234) (but see 79 for a full review). These observations have been interpreted as suggesting that glide takes place by dislocation motion between the dislocation sheets which are the source of the internal stress (66,79, 202,210). This model is different from other work hardening models, where either the glide dislocations coincide with the dislocation sheets (e.g. 239,240), or the sheets are taken to form continuously throughout stage II/III (241). However, what contribution to the work hardening is from elastic interactions between dislocations in between the sheets is not known (66).

At low temperature one has a quasi-parabolic stress-strain curve. The homogeneity of the dislocation structure and the slip lines, coupled with the observation that only τ_p increases during the deformation (e.g. 225), has led to the conclusion that elastic interactions between screw dislocations are mainly responsible for the work hardening (79,229,230,243). Although various theories have been proposed (237,238) and satisfactory agreement between theory and experimental results obtained (e.g.229,230), the theories have been criticised on

the grounds that the assumptions made are not physically justified by the evidence to hand (79). The observed agreement between theory and experiment can be obtained through the free parameters in the equations (79).

When alloys exhibit three-stage hardening, the work hardening rate in stage I is greater than in the base metal, whilst in stage II it is lower (e.g.73). In stage I the effect has been attributed to the increased density of dislocations, especially screw dislocations, on the primary slip plane (e.g.72,73,83). The greater density is made possible because of the larger value of τ_p in the alloys, which favours the formation of screw dislocation arrays of higher density than in the pure metal (79,83,204). The larger the solute concentration, the greater the work hardening rate, as is observed (73). The large primary dislocation density causes considerable latent hardening of the secondary slip systems, so that stage I is lengthened (72,83). In higher concentration alloys the effect is so great that sometimes stage II is never initiated (72).

Measurements of the work hardening rates in Fe-Si between 113 K and 473 K have shown that θ_I is temperature dependent (72). It has been proposed that, at strains where the slip is occurring in discrete bands, the work hardening is caused by the long-range stress between the bands affecting their growth (72,236). As the band separation increases as the temperature falls (204) the work hardening rate decreases. Since with increasing strain the bands broaden to fill the crystal, this work hardening mechanism is replaced eventually by one of elastic interactions between individual dislocations (72).

13. Etch pit studies.

There are few reported studies on dislocation velocities measured by etch pit methods (Nb 250 : Fe 246 : Fe-Si 244,245,248,249,251).

These show that at room temperature the velocity of a screw dislocation in Fe-Si is less than that for an edge dislocation (245,249). At 196 K the velocity of both types of dislocation, considered as a function of stress, is reduced (249,250). No asymmetry in the motion of the screw dislocation has been observed (249,250).

However, the above work has been criticised for taking the velocity of dislocations at the head of a glide band to be equivalent to the velocity of an isolated dislocation (247). By following the growth of slip bands in Fe-Si it was found that the velocity of a slip band was strongly affected by the proximity of other slip bands. It was concluded that the velocity as measured above was not related in any simple way to the velocity of an isolated dislocation.

14. In-situ studies.

Although the straining of specimens inside a HVEM is a comparatively recent innovation, much work has been done in applying it to bcc materials (Fe 81,252-254 : Nb 81,255-257 : Mo 81 * Fe-Si 258,259, 261 : Mo-Nb 260 : Fe-C 262,270 : Nb-Ta,Mo 263). This is partially because of their relatively low dislocations velocities, vis à vis fcc metals, which greatly facilitates observation (267). The limitations of the method, e.g. surface effects and radiation damage, have been analysed (266,267,271), and the results obtained from the deformation studies recently reviewed (81,148,265,267).

Below a critical temperature (T_c), the dynamic dislocation behaviour alters drastically, reflecting the change in properties observed in macroscopic tests (81). This temperature is between 200 K and 240 K for Nb and Fe, and about 350 K for Mo as observed from the in-situ tests (81,267). In the low temperature region at stresses below the yield stress little screw dislocation motion is observed, whilst long $\langle 111 \rangle$ screw dislocation dipoles are formed by fast movement of edge-mixed dislocations (252,253,257). The screw

dislocation density continues to rise until the macroscopic yield, when large scale screw dislocation motion takes place. Although screw dislocation networks have been built up on several planes, usually only one of them operates to any great extent (81). The screw dislocations move at a constant speed, but more slowly than the mixed dislocations (253,262). Unlike the curved mixed dislocations, they always remain straight. A dynamic balance between annihilation and multiplication is set up (81). How the screw dislocations interact with obstacles depends upon the material in question and the type of obstacle (267). If the obstacle is strong, e.g. an attractive junction or super jog, then in the case of the latter one can get a wandering source or a prismatic debris loop, depending on the cross slip distance. They are called wandering sources as the super jog can move along the dislocation line between operations of the source whereas the junction dislocation gives rise to a single ended fixed source (256,261,265,268). Wandering sources have been observed in Nb and Fe but not Mo, whereas fixed sources have been seen in Fe and Mo, but not Nb (81,252,269). For weak obstacles, e.g. dislocation cutting, the screw dislocation remains straight, bending only locally before breaking through (148,267). In Nb at least, such elementary jogs have little effect on the screw dislocation velocity (257). At larger strains, in Nb, the absence of cell formation can be explained by considering the temperature dependence of tangle initiation at prismatic loops (255). The calculated transition temperature (≈ 180 K) agrees well with the observed value (200 K).

As the temperature is raised towards T_c , the screw dislocations become more mobile and the edge - screw dislocation velocity ratio (v_e/v_s) falls (265). When by-passing obstacles, the screw dislocation becomes closer to the equilibrium curved shape, which it assumes fully above T_c (81). Having passed T_c the dislocations, which are now of mixed

character, move rapidly and discontinuously, multiplying by Frank-Reed sources (81), although wandering and fixed sources have been seen in Fe (268). The behaviour is similar to that for fcc materials (81,267).

These observations give direct support to the view that screw dislocations have a large, temperature dependent, lattice friction stress, and that their motion is governed by a double kink mechanism (81,265,267).

Such work as has been done on substitutional alloys (258-261, 263) has shown that, although there are strong similarities to the pure metals, there are some significant differences for strong hardeners, e.g. Si in Fe or Mo in Nb. The velocity of both edge and screw dislocations is reduced, but the effect is greater for edge - mixed dislocations. The critical temperature appears to be raised: e.g. Nb-5 at.%Mo has straight screw dislocations at 300 K (263). The screw dislocation jog density is also raised. In the case of Nb alloyed with Ta there is little change, reflecting the small effect the alloying has on the mechanical properties (263).

Interstitial alloying has been little studied, the only work being on Fe-C which shows solution softening (262,270). This seems to be characterised by discontinuous screw dislocation motion at low temperatures (see "solution softening" section 17).

15. Microplasticity.

In bcc metals one can have significant plasticity generated at stresses well below the macroscopic yield stress. Whether this is caused by the same mechanism which dominates after the macro yield was a controversial subject (278-280), but now it is generally accepted that the two regions are controlled by different mechanisms (272-274,277).

This view has been supported by 'in-situ' deformation experiments which have shown directly that edge - mixed dislocation segments move well below the macroscopic yield stress, which is associated with

large scale screw dislocation motion.

Two basic methods have been used, both depending upon high strain sensitivities, e.g. $2 \times 10^{-7} \epsilon_p$ (274). In one, stress relaxation (277,282) or strain rate changes (273) allow a thermodynamic analysis. In the other, stress cycling (95,264,272,274,276,281) can be used to establish the onset of reversible dislocation motion (σ_e) and then plasticity (σ_a). However, σ_e depends upon the sensitivity of the apparatus and σ_a has a rather arbitrary definition. Sometimes these are coupled with a 'flow' stress at a particular strain (e.g.95,276) or strain rate (373).

From such work it can be seen that the microyield stress has a much lower temperature dependence than the macroyield, this temperature dependence being all the smaller at decreasing plastic strains or lower strain rates (95,264,273,274,276). The strain generated before the macroyield (free strain) decreases as the temperature falls. Alloying also reduces this free strain, and the transition between microflow and macroyielding is more precisely defined (e.g.273). Its effect on the microflow stresses is that the lower the strain rate or strain considered, the greater the amount of solute that has to be added before the temperature dependence of the associated flow stress is raised (95,273,274) : e.g. in V-N the stress at $\dot{\epsilon} = 3.2 \times 10^{-7} \text{ s}^{-1}$ does not significantly alter its temperature dependence until ≈ 1600 at.ppm 0 + N, whereas that at $\dot{\epsilon} = 3.2 \times 10^{-6} \text{ s}^{-1}$ is significantly altered by 800 at.ppm (273). It has also been observed that σ_e in Ta and Fe show little or no orientation dependence, whereas the macroyield does (176,277). From stress cycling work it has been found that the strain generated per cycle decreases rapidly until only reversible dislocation motion is observed (exhaustion cycling). To generate more strain either the stress or the temperature must be

raised (272). It has been observed in stress relaxation experiments that v^* in the microstrain region is much larger than that after the macroyield (273-275,285).

These results indicate that the whole of the microflow region is determined by the thermally activated motion of edge and mixed dislocations (264,272-274,277,286). These interact strongly with solute atoms, and the effect of the solute and stress cycling can be rationalised by considering how much strain can be generated by the edge - mixed dislocations before they are pinned in front of an obstacle which is too strong for them to overcome (272). As the deformation proceeds, the mobile density of edge - mixed dislocations decreases and the transition to screw dislocation motion takes place at the macroyield (272-274).

The above refers to work on Nb, Ta, V and Fe. Mo shows the same general type of behaviour, but it is rather more complex (283,284). The microstrain region is rather larger, up to 3% axial strain, and it is thought that more than one dislocation mechanism is operative in this region : e.g. surface sources (283).

16. Anomalous slip.

As the deformation temperature is lowered for high purity group $\bar{V}A$ metals (V, Nb, Ta), the primary slip system $(\bar{1}01) [111]$ is replaced by the anomalous systems $(0\bar{1}1) [111]$ and $(0\bar{1}1)[\bar{1}11]$, the fifth and sixth most highly stressed systems, for a wide range of orientations (V 23,24,196 : Nb 8,58,59,178,288,289 : Ta 18,173,195,291). Anomalous slip starts to occur at different temperatures for the three metals; 175 K for V (24) and Nb (178), and 77 K for Ta (18,195), but its initiation is dependent upon purity and orientation. Whilst anomalous slip in Nb occurs down to 4.2 K (61), there is some evidence that no anomalous slip occurs in Ta deformed below 40 K (64,207). Unlike the fine wavy

primary slip, the anomalous slip consists of straight crystallographic slip lines, accurately parallel to $(0\bar{1}1)$ and usually continuous around the crystal (24,178,195,196). When viewed under Nomarski contrast, no dead band is observed (24,196) but rather a region of reduced contrast where $[011]$ is parallel to the crystal surface (195,288,289). This is consistent with shape change experiments on Nb (288) which showed that $(0\bar{1}1)$ $[\bar{1}11]$ and $(0\bar{1}1)[111]$ carried respectively 46% and 26% of the total strain, thus giving an 'effective' Burger's vector near $[011]$. This observation, that the two Burger's vectors $[111]$ and $[\bar{1}11]$ are operating together, is further strengthened by electron microscopy on Nb deformed at 77 K. Cross-grids of $[111]$ and $[\bar{1}11]$ screw dislocations are seen built into the $(0\bar{1}1)$ plane (289 : see also 24,292).

The stress strain curve is dependent upon temperature, purity and orientation. In high purity Nb orientated in the centre of the unit triangle and deformed at 77 K, a serrated yield is observed accompanied by audible clicks, the size of the load drops being between 1 - 4 kg/mm² (axial). At larger strains or higher temperatures they did not occur (59). Load drops are still present at this temperature, even when there is no anomalous slip, if the crystal has a large χ value (59 but see 291,293). Doping with oxygen reduces the load drops in size and frequency, as well as slowly eliminating the anomalous slip (71). In Nb the load drops may be suppressed by prestraining 0.7% (axial) at room temperature, or by abrading the surface ; slip still takes place on the anomalous plane but with a finer morphology. Load drops were not reported for the early work on anomalous slip in Nb and Ta (18,58,178, but see also 291), nor have any been observed with V (24,196).

Anomalous slip has been observed only in high purity V, Ta and Nb crystals and those doped with small quantities of O or N : e.g. 30 at.ppm N or 400 at.ppm. O will suppress anomalous slip in Nb at

77 K (8,23,71). Lower concentrations raise the yield stress and coarsen the slip line structure. No group $\bar{V}A$ substitutional alloys have been reported to show anomalous slip. In the case of zone refined Ta, anomalous slip occurs for orientations $\chi < -15^\circ$. This is extended to $\chi < +10^\circ$ for UHV annealed Ta (18,173,195). This also underlines the point that the appearance of anomalous slip depends upon temperature, purity and orientation, and that they are all interconnected factors (18,24,173,195). As one moves towards the (011) - ($\bar{1}11$) boundary, the Schmid factor goes to zero for the anomalous plane, so eventually there will be a change in the slip system (e.g. 195,289). The morphology of the slip system and of any other slip system operating, is also orientation dependent (24,195,289): e.g. Nb crystals near [001] deformed at 77 K, show coarse ($0\bar{1}1$) slip lines with deformation on the other three {110} planes (289), whilst for centre-triangle orientations (71,348) just fine ($0\bar{1}1$) slip is observed.

In Fe no anomalous slip is observed at any temperature down to 4.2 K (e.g. 61). In the case of Mo it is a debated point whether one has 'true' anomalous slip or just slip on the ($0\bar{1}1$) plane (199, 294,295). The early stages of deformation in Mo are complex because of the multiplicity of slip systems which operate before the primary system dominates (e.g. 296). It has been proposed that their operation is governed by surface sources, and that each system will only appear over a certain region of the crystal's surface. Although some work shows slip on ($0\bar{1}1$) encircling the crystal at low strain, considered to be anomalous slip, it is replaced by the primary system as the strain increases (295). This interpretation was not supported by Jeffcoat et al (199) who claim that the presence of ($0\bar{1}1$) lines at low strains can be explained by a surface source theory (296).

However, anomalous slip has been observed in Mo - 5 at.%Re for $\chi < -11^\circ$ and Mo - 5 at.%Nb for $\chi < 0^\circ$ deformed at 77 K to 3% axial strain (194). (This is contrary to the effect of substitutional alloying in group $\overline{\text{VA}}$ metals : e.g. 1.5 at.%Mo suppressed anomalous slip in Nb deformed at 77 K (73)). Coarse slip lines were seen which were continuous around the crystal without a well defined dead band but rather a minimum step height near where [011] was parallel to the surface. This apparent Burger's vector varied systematically with the crystals' orientation.

Three basic types of models have been put forward to explain anomalous slip, viz: atomistic, surface effects and variations on coplanar double slip. Using a computer model it is possible to find an interatomic potential which will give slip on the $(0\bar{1}1)$ plane (297). However, as it is a one Burger's vector model, and predicts a change in the anomalous plane between tension and compression (302) which is not observed (289), it has been discarded. The surface effect theory is based on the idea that image forces at the surface of a crystal can produce kinks on screw dislocations, which then propagate away from the surface on the anomalous plane (298,299). This model has been criticised because it predicts the wrong orientation dependence of the slip plane (e.g.24,294) and because it suggests that with round specimens the operative plane should change around the specimen's surface (294). These objections have been accepted with respect to anomalous slip in group $\overline{\text{VA}}$ metals (295,301) but the model is used sometimes to explain unexpected slip systems in Mo (295,303,304).

The variations on double coplanar slip models are all based on electron microscopy studies. It was observed from in-situ straining experiments on Nb that unlike attractive junctions the behaviour of repulsive junctions was temperature dependent. At high temperatures

primary screw dislocations can cut through secondaries but at lower temperatures they were unable to do so, and each dislocation was deflected on to the slip plane with the fewest forest dislocations, i.e. the $(0\bar{1}1)$ plane. The model is able to give the same plane in tension and compression and the correct orientation dependence (292). The second variation is that two crossed screw dislocations exert a torque upon each other, and in a network of crossed screw dislocations with junction dislocations this torque can aid production of kinks on the junction dislocations, allowing the network to move at lower stresses than an isolated screw dislocation (295,301,302,304,305,348). Such networks have been observed to move in Mo (295,304,305). Junction dislocations have been seen also in Nb (289,290) and V (24) although not in V - 800 at.ppm O when there is no anomalous slip. The last theory is similar to the previous one, except that no junction dislocation is formed (289). The torque bends the dislocations from screw towards being one of mixed character. As these have a lower Peierls stress, the network can again move more easily than a single dislocation. Any factor which affects the ratio between the velocity of screw and edge - mixed dislocations will affect the initiation of the anomalous slip.

17. Solution softening.

Early experiments on alloying nominally pure metals suggested that at low temperatures, approximately 80 K - 120 K, there was a reduction of the yield stress below that of the base metal for low concentrations of substitutional solute: e.g. 1 - 3 at.% (77,154,216). At the same time, the high temperature strength increased. Considerable controversy ensued as to whether the softening was intrinsic, characteristic of a binary bcc alloy, or extrinsic, owing to other factors, such as impurities (e.g. 1,216,224). Later work with purer alloys and base

metals has produced a more coherent picture, but many difficulties still exist (see 224 for a recent review of the subject).

For group $\bar{V}A$ metals (V, Nb, Ta) the evidence seems to favour an extrinsic mechanism for softening (7-16, 19, 20, 22, 273 but see also 3, 308, 348). If UHV annealed alloys are used, e.g. Nb-Mo, Ta-Re (1, 7, 13, 14) and the yield stress referred to the UHV annealed base metal, then only hardening is observed. The same is seen with interstitial alloys : e.g. Nb- O, N, C, H and Ta - O, N (8, 10, 11, 13, 19, 20, 22). However, ternary alloys made from UHV annealed base materials, e. g. Ta-Re-N or Nb-N-O (11, 14) show softening initially but hardening appears as the concentration of one of the solutes is raised further.

The maximum softening occurs at about 50 - 200 at.ppm. The results of early experiments are seen to be misleading because the purity of the base metals and the alloys was not high enough. The softening is usually explained by a scavenging model. Solute complexes or clusters are considered to form, reducing the barrier to dislocation motion. As the concentration is raised hardening mechanisms dominate (see also 308). Further support comes from experiments which show that the softening of Ta-Re-N alloys depends upon the rate it is cooled after annealing (13). Slow cooling gives a large softening, whilst a fast cool results in hardening. Internal friction experiments indicate that Re-N complexes form (15, 16). However, this does not seem to be the case for V-Ti where internal friction work has not found any such associations (287). Pink and Arsenault (224) pointed out that although scavenging undoubtedly does occur, it may not affect the stress for deformation. It is possible that scavenging is not the softening mechanism as such, but merely creates the conditions for it.

Some recent work on UHV annealed Nb doped with up to 400 at.ppm oxygen orientated for glide on either the anomalous system or $(\bar{2}11)$ has

cast some doubt on the extrinsic interpretation (61,71). The specimen showing anomalous slip hardened as the oxygen concentration rose with the anomalous slip slowly being suppressed, whilst the one orientated for $(\bar{2}11)$ slip showed only softening. Meshii et al explain their results by the different action of interstitials on the two different deformation mechanisms operating, viz: co-operative double glide for $(0\bar{1}1)$, single glide for $(\bar{2}11)$. Thus it is an intrinsic property of the material. Whilst their observation on the effect of alloying on anomalous slip is in agreement with other work (e.g. 8,18,287) that for the $(\bar{2}11)$ slip orientation is not (but see also 307). Work on UHV annealed and doped Nb-N and Ta-N deforming on the same system $(\bar{2}11)[111]$ shows only hardening (8,18,20,24). It should also be noted that the yield stress for this orientation obtained by Meshii et al (61,71) is 50% larger than that obtained by Garrett-Reed and Taylor (59) and others, whilst the yield stress for the anomalous slip is in good agreement. Similar observations have also been made on electron irradiated Nb (348).

The experimental results for Fe, especially the most recent, tend to indicate an intrinsic mechanism of softening (25-29,200,213, 224,262,300,309,310-312 - most of these are for Fe-N,C). However, unlike the work on Nb and Ta, a wide range of preparation techniques has been used (e.g. 27-29,200, but see also 313). Certain questions as to the H_2 levels in the specimens have been discussed (e.g. 27,28, 213) as well as an ageing effect (312,314). Although the exact form of the yield stress-temperature curve depends upon the experiment in question (e.g. 26,27,349 or 29,312), the general conclusion is that softening occurs over the temperature range 100 K - 200 K, with hardening at higher and lower temperatures. At 150 K, where the maximum softening occurs, hardening does not appear until about 1200 at.ppm N is present. There is also an orientation effect,

the softening being most pronounced for slip on the {211} planes (312).

What little electron microscopy has been done on softened materials has shown that the dislocation structure is very little altered (e.g. Ta-Re-N 14). However, if in-situ straining experiments are done, a significant difference is seen between Fe and softened Fe-C alloys (262,270). In the case of Fe, the motion of screw dislocations is smooth and continuous. For Fe-C alloys, it is jerky and discontinuous. A detailed analysis concluded that the jerky motion was due to a preferential nucleation of double kinks in the vicinity of carbon atoms, this process occurring in parallel with 'ordinary' double kink nucleation (215,270,310). Thus these authors concluded that softening in Fe-C is intrinsic in nature. However, with Fe-Ni, Ti alloys which also show softening in τ^* , the screw dislocations remain straight whilst moving, and their motion is continuous (300). For these alloys it was concluded that the effect of substitutional solute is non-localised, reducing activation enthalpy by a change in interatomic potential.

Many theories have been put forward to explain 'intrinsic' softening, ranging from impurity induced cross-slip (318) to electron concentration (317) or an increase in mobile density (3 - see 80,224 for a recent review and comments). The most commonly cited models at present approach the problem from different view points (147,150, 153). Using the model of a spherical defect exerting a torque on a screw dislocation (151,315) Sato and Meshii extended the calculation to an interstitial atom (150). The torque should locally enhance the double kink nucleation rate, but the interstitial atom acts also as an obstacle to sideways kink motion. The overall effect depends on the temperature and concentration. When evaluated, this model gives low temperature softening, and high temperature strengthening.

However, it has been criticised on the ground that the torque around an interstitial, which is not a spherical defect, is of no consequence (152). Also it is difficult to explain, using this model, why hardening should reappear at lower temperatures, e.g. in Fe (224).

In the 'unified model' the various rate determining steps and activation enthalpies associated with a dissociated dislocation passing an obstacle are considered (153, 316). These depend on whether the length of dislocation involved in forming a double kink is larger, smaller, or about the same as the separation of obstacles along the dislocation. As this is a function of stress, temperature and obstacle concentrations, it is possible for the model to indicate three different regions, with hardening at low and high temperatures, and softening in the intermediate temperature range. The weaknesses of this model have been criticised by several authors (154, 224, 306).

The third model commonly cited considers the interstitial atom to be impenetrable, double kink nucleation then takes place on screw dislocation segments pinned at both ends (147). The model indicates that this will effectively create a concentration dependent long range back stress, which is not additive to the thermal stress required for double kink nucleation. Whichever mechanism requires the larger stress will control the yield stress. At high temperature there is a concentration dependent hardening, but at low temperature the interstitials have no effect on the yield stress. If, however, the thermal stress is considered to be equal to the difference between the yield stress and the athermal plateau, then pseudo alloy softening appears when experimental data is analysed in this way. This has been observed in some experiments, where alloy softening was seen in the 'thermal' stress but not in the yield stress (e.g. 200, but see also 224).

Experimental Method.

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Experimental Method.

1. Crystal Preparation.

The production of high purity single crystals was a four stage process. The first stage was to tie a suitable quantity of alloying element, in the form of rod or sheet, to a niobium rod of approximately 4 mm diameter, using very thin niobium wire (see table 3). All the components had been suitably cleaned prior to this (table 1). After being tied together, the 'rod' was washed in methanol and dried so as to minimise contamination from handling.

Single crystals of random orientation were grown from these 'rods' in an electron beam zone melting furnace (319). To ensure homogeneity, three zone passes at a zoning speed of 4 mm/min were given, the direction of zoning being reversed for each pass. Previous work with this machine (320) had shown that the composition fluctuations were ± 0.2 at.% in a Nb-5 at.%Mo alloy. The pressure was usually between $3 - 8 \times 10^{-6}$ torr, although for the first four crystals it tended to be between 5×10^{-6} and 1.5×10^{-5} torr. A liquid nitrogen trap was always used, both for this stage, and the final seeding pass. A short length was cut from each end of the crystal, and the rest, typically 20 - 25 cm, was cold swaged to 3.6 mm diameter. Care was required when swaging the 11 at.%Mo alloys because of brittleness, and so for the 17 at.%Mo no swaging was attempted, and instead it was centreless ground to 3.6 mm diameter.

One of the end pieces was then reorientated to a centre triangle orientation in a two circle goniometer, and used as the seed for growing a short length of crystal from the other end piece. The orientation of this was then checked, and if satisfactory, it was used to seed the complete swaged rod. Care was taken that this pass was in the correct sense with respect to the preceding three zone passes.

This procedure of growing from a vertical, rather than an angled, seed had been found to give fewer failures. The vertical seed was kept and used to seed subsequent crystals of the same composition. It was found necessary to reduce the growth speed in this seeding pass from 4 mm/min used for Nb and Nb-Mo alloys, to 1 mm/min for the Nb-Ta alloys. At the higher speed the Nb-Ta alloys reseeded themselves near [001]. Even the lower speed was not entirely successful, especially with the 60 at.%Ta alloy. The orientation of the final crystal was checked at two points along its length, one near the start and one near the end. The orientations of the crystals is shown in figure 1.

The single crystals were then centreless ground to 2.9 mm diameter, and chemically polished in 60 : 40 conc.HNO₃ : 48% HF to approximately 2.8 mm diameter. The polishing was done in a flat bottomed rectangular polythene dish, and the crystals were rolled around so as to get a uniform reduction along their whole length. Each crystal was then vacuum annealed on its own (see comment at end of this section) at a temperature just below its solidus by passing a large current through it, up to 350 A in some cases, whilst maintaining a pressure of approximately 10^{-10} torr. To reach this state, the current, and hence the temperature, was raised slowly, allowing the pressure to decrease sufficiently before again raising the current. As the specimen's temperature cannot be measured directly in this furnace, the ending of the anneal is controlled by connecting a length of Wb or alloy of smaller cross-section than the crystal in series with it. Being of smaller cross-section, it will be hotter than the crystal. The diameter of this 'fuse' was calculated by assuming a balance between heat input from the current and heat loss by radiation. It was adjusted to a value that would allow this 'fuse' to melt when the crystal being annealed was approximately 100 K below its solidus.

Its melting broke the electrical circuit, and the crystal then cooled by radiation. The crystals usually spent at least 30 hours at a temperature above that at which they evaporated and usually 6 hours at a temperature which was only approximately 70 K below their maximum annealing temperature (but see discussion, section 1). The final pressure was usually between $5 - 9 \times 10^{-11}$ torr, with the crystals having typically spent at least 30 hours below 5×10^{-10} torr. However, the first four crystals (287,292,295,296) only reached approximately 10^{-9} torr. This was traced to a faulty gold 'O' ring which was replaced before any further anneals were done.

The UHV furnace that was used for the anneals was modified several years before the start of this work so as to hold only one crystal at a time rather than two crystals which were parallel to each other. Work by Garrett-Reed (326) had shown that such an arrangement resulted in the two crystals bowing away from each other during the anneal because the magnetic force resulting from the large currents flowing through them was sufficient to cause significant creep at such elevated temperatures.

2. Mechanical testing.

After being removed from the UHV furnace, the single crystal rods were glued with Durafix to a wooden block. Test specimens were cut from the crystal using a SiC slitting wheel and jig built by Taylor. The first and last 10 mm of the crystal were discarded, as they did not reach as high an annealing temperature as the rest of the rod. This was because they were adjacent to the physically larger, hence cooler, Nb grips which hold the ends of the crystal in the UHV furnace. From the remaining crystal, approximately twenty 9 mm compression or four 4 cm tension specimens could be cut.

Compression specimens were ground in a jig on SiC paper to produce right cylinders and then polished to remove surface damage and to give a good surface for slip trace analysis. All specimens, except the 60 at.%Ta alloy, were polished in 60 : 40 HNO₃:HF (see table 1). The 60 at.%Ta alloy had a matt surface after the anneal because of evaporation at the very high annealing temperature. A polishing solution for this alloy which enabled one to see the slip traces was 5:2:2 conc.H₂SO₄ : conc.HNO₃ : 48% HF. This solution had to be freshly made. However, the surface ~~was~~ still very inferior to that obtained with the other alloys or Nb. In all cases, immediately after being taken out of the chemical polish, they were washed in water, then analar methanol, and then dried in a hot-air blower. The specimens' dimensions were measured with a micrometer.

For the tension specimens, mild steel grips designed by Taylor (328) were used. Because of the design of the tension jig, the distance between the transverse pin-holes had to be 54 ± 2 mm. Therefore the specimens were all cut to 4 cm, and their 'hole' in the grips was drilled to 7.5 mm deep. This gave the specimens a gauge length of approximately 2.5 cm. Holes 5 mm deep were tried, but some specimens pulled out of the grips before yielding. Specimens were polished as above until they fitted tightly into the hole, and then the grips were squeezed on by passing the grip and specimen through a die. The specimen's diameter was measured with a micrometer and the gauge length with a ruler.

This technique was satisfactory for all the Nb specimens, and for 10 at.%Ta alloy tested at 77 K and 60 K, but not for any other of the alloys. At, or below, 77 K, the 1 at.%Mo alloy twinned, and the other alloys all necked near the grips immediately upon 'yielding'. To try to get around this, some specimens of each composition were thinned along a section of their gauge length, with a rounded shoulder

up to the grip. Reductions in cross-sectional area of up to 3:1 between the gauge length and the unthinned section were tried, as were several shoulder profiles, but the specimens yielded and necked by the shoulder. However, using unthinned specimens, a small prestrain at room temperature was found to give reasonable stability for 1 and 3 at.%Mo, and 10 and 60 at.%Ta alloys at the lower temperatures. This stability was limited for the 5 at.%Mo, and almost non-existent for the 11 at.%Mo alloy, which still necked very soon after yield. The 1 at.%Mo, 10 and 60 at.%Ta alloys were given a room temperature prestrain of approximately 0.5 axial %, whilst the 3, 5 and 11 at.%Mo alloys approximately 1 - 1.5 axial %. Although the size and presence of this prestrain affected the low temperature properties, it was used in all the tension tests, except as stated above.

Two machines were used for the mechanical tests. One was only for compression tests between 373 K and 77 K, whilst the second was for tension or compression tests at room temperature, and between 77 K and 4.2 K.

The compression machine was of a Polanyi type, described in (321) and modified by (322). It was further modified (see below), but first consider an outline of its operation. The testing procedure for sub-ambient work was to put a copper beaker of isopentane around the jig and specimen (which was under a 5 kg. load to keep it properly seated) and then a constant temperature bath around that. For tests at liquid nitrogen temperature, the beaker of isopentane was not used. The temperature was checked with a chromel-alumel thermocouple (but also see below). Boron nitride was used as a lubricant to reduce end effects in all tests. The output of the load cell was fed to the recorder via a control box (30) which allowed suppression of the load to give 100 kg. full scale deflection irrespective of the actual load.

This was used for all tests. The specimen was deformed initially at a constant cross-head speed, and x 10 strain rate changes were done, about ten in all. However, for the Nb-Mo alloys zonal glide often occurred before it was possible to make all ten changes. For temperature change tests, the specimen was strained a few axial %, then unloaded to approximately 10 kg., and the temperature bath changed. When the new temperature had stabilised, the specimen was reyielded. The changes took place only between 'adjacent' temperatures, e.g. 225 K - 175 K and not 225 K - 158 K [table 2]. On average, between three and five temperature changes were carried out on a specimen. Because of the isopentane bath, there was no danger of the jig freezing as the temperature was lowered, or of the specimen and jig having to be warmed up, dried and recooled. Only for the temperature change 113 K - 77 K was this necessary. Usually only changes to a lower temperature were done, but a few were done for an increase in temperature.

Four modifications were made after the work of (322).

- 1) The Telsec 2000 load-time recorder was replaced with a Hewlett-Packard 7045A x-y recorder and a Sandborn 7DCDT 500 differential transducer.
- 2) The compression jig used for the tests in (322), and some of the early tests after that was replaced by a new one designed by Taylor. This had a much lower thermal mass and was physically much smaller ; it allowed the use of a much smaller copper beaker, approximately 4 cm in diameter rather than 9 cm. The new jig was also slightly harder, 1.36 $\mu\text{m}/\text{kg}$, rather than 1.53 $\mu\text{m}/\text{kg}$.
- 3) A chromel-alumel thermocouple was added to the jig. It was calibrated by direct immersion in the solid- liquid baths (table 2) and in liquid nitrogen. It was observed that the temperature inside the isopentane bath was slightly higher than the constant temperature

bath around it. The difference depended on which jig was in use, and where the thermocouple was placed on the jig, e.g. whether the tip was taped to the top anvil, left hanging free in the gap between the anvils, or glued inside a mock specimen made of brass placed in the jig as if it were a specimen. The largest difference was when a solid - liquid isopentane temperature bath was used with the older, larger anvils. The specimen's temperature was 123 K, whilst the surrounding temperature bath was at 113 K. With the small jig, the difference was only 1 - 2 K at this temperature. In both cases, the difference between the inside and outside temperatures decreased as the temperature increased.

4) A stepping motor replaced the induction motor and gearbox. This increased the cross-head speed to 1 or 10 $\mu\text{m}/\text{sec}$ ($\dot{\epsilon} \approx 1.2 \times 10^{-4}$ or $1.2 \times 10^{-3} \text{ s}^{-1}$) as opposed to 0.333 or 3.33 $\mu\text{m}/\text{sec}$ ($\dot{\epsilon} \approx 4 \times 10^{-5}$ or $4 \times 10^{-4} \text{ s}^{-1}$). With the induction motor, the initial CHS speed was always 3.33 $\mu\text{m}/\text{sec}$, whilst 1 $\mu\text{m}/\text{s}$ was used with the stepping motor. Of the tests done since the work of (322), those on crystals 297, 300 and 302, unless otherwise stated, were at 3.33 $\mu\text{m}/\text{sec}$. All other tests were at 1 or 10 $\mu\text{m}/\text{sec}$. The stepping motor system also eliminated the vibration of the whole compression machine caused by the induction motor's eccentric gearbox.

The second machine was built for Taylor, and is described in (8). Jigs for tension or compression could be fitted and up to six tension or ten compression specimens could be loaded as one batch and sealed into a chamber filled with helium gas. This chamber was then cooled in one of two ways. Early work used the boiling and triple points of suitable cryogenic liquids (table 2). However, this leaves inaccessible a large temperature range between 20 K and 60 K. Later,

by fitting a controllable needle valve to the bottom of a transfer tube in a dewar of liquid helium, it was possible to control the gas flow rate around the container. By suitably altering this one could cool the specimens and then stabilise the temperature at any chosen value. The output of the thermocouple would drift typically by less than 5 μV during a test. The sensitivity of the Au - 0.056 at.%Fe v. chromel thermocouple was approximately 20 $\mu\text{V/K}$. Thus stable temperatures between 77 K and 5 K were accessible.

The early tests on this machine, which were in compression, used an induction motor, giving a CHS of 1 or 10 $\mu\text{m/sec}$. Before the tension tests, it was replaced by a dual angle stepping motor, the speed of which could be set from an external control box. The CHS for the tension tests was chosen so that the strain rate in tension and compression would be similar. The strain rate could be altered 'instantaneously' by $\times 10$ ($1.2 \times 10^{-4} - 1.2 \times 10^{-3} \text{ s}^{-1}$) using a set of magnetic clutches and gearbox as with the Polanyi machine. However, for some of the early tests when the gauge length was 3 cm, as opposed to 2.5 cm for the majority of the tests, the motor would stall when the CHS was increased to 30 $\mu\text{m/sec}$ ($\dot{\epsilon} \approx 1.2 \times 10^{-3} \text{ s}^{-1}$). In such cases, a CHS of 3 $\mu\text{m/sec}$ was used to determine the yield stress, but the strain rate change tests were carried out using 1 and 10 $\mu\text{m/sec}$ ($3 \times 10^{-5} - 3 \times 10^{-4} \text{ s}^{-1}$). Also, after some observations made on Nb below 20 K, some $\times 2.5$ changes ($1.2 \times 10^{-4} - 4.8 \times 10^{-5} \text{ s}^{-1}$) were done by altering the motor speed. The control unit had two motor speed controllers, and the strain rate change was made by switching between them. It was not possible to do a $10^{-4} - 10^{-3} \text{ s}^{-1}$ change in this way, because of the limits on the speed ranges of the two controllers.

The temperature change testing procedure was similar to that used on the Polanyi machine : i.e. the specimen was deformed at an

initial strain rate of $1.2 \times 10^{-4} \text{ s}^{-1}$, and after some strain rate changes it was unloaded and re-deformed at a lower temperature. However, because of the smaller number of specimens of a given composition, and the problem of stability of deformation, there were some differences. Few strain rate changes were done to keep the total strain small and to try to avoid necking.

Strain rate changes at some temperatures, especially at or below 20 K could only be done on a specimen which had already been deformed at a higher temperature (other than room temperature). The temperature changes were small, 10 K or 20 K, and when the temperature was below 20 K, only 5 K. Only changes to a lower temperature were done. If, for any reason, a specimen was warmed up to room temperature after being deformed at low temperature, it was not retested, as it was observed that its behaviour was usually different, e.g. the flow stress may be up to 20% higher.

3. Surface examination.

The specimens were kept as clean as possible after testing. Those tested in helium gas were allowed to warm up to room temperature in the gas before being removed from the jig. Specimens which had been in the isopentane bath or in liquid nitrogen were removed at the end of the test and immediately put into a beaker of warm methanol, then washed in ether in an ultrasonic bath, and dried with a hot-air blower. Optical examination was made in a Zeiss Ultraphot with Nomarski interference contrast at a magnification of x 500. For slip line determination, the usual x-ray and multiface analysis was used. This involved measuring the angle the slip line makes with the specimen axis at 10° intervals around the specimen. Usually only half the surface was measured, but sometimes it was necessary to examine the

whole surface. Occasionally the observations were made at 5° intervals. The protractor eye piece was not of the same optical quality as the Zeiss eye pieces and very occasionally the slip line angles were estimated by eye using the ordinary eye pieces, as they could not be resolved with the protractor eye piece.

4. Composition.

The compositions of the alloys 'as made up' are given in table 3, together with the compositions of the UHV annealed crystals determined from density measurements or x-ray microprobe data. Although there were variations between crystals of nominally the same composition, for convenience, they will be referred to by their nominal compositional group.

The density (d_s) was evaluated by weighing a deformed specimen in air (W_s), then in xylene (W_x), and substituting the values into:-

$$d_s = W_s \left[\frac{P_x}{W_s - W_x} \right] \quad (1)$$

where P_x is the density of xylene. For the Nb-Ta alloys the lattice parameter is effectively independent of composition (it varies linearly from 3.3007×10^{-10} m to 3.3026×10^{-10} m) so that the Ta concentration (A, in atomic %) is given by :-

$$A = 100 \left[\frac{d_s - d_n}{d_t - d_n} \right] \quad (2)$$

where d_n and d_t are the densities of Nb and Ta respectively (8.57 and 16.6 g/cm³). The sensitivity of the method for Nb-Ta alloys (substitute for d_s in equation (2) from equation (1), differentiate, and substitute in typical values) with the apparatus used (a last figure sensitivity of 0.1 mg) was 0.5 at.%. For crystals 295 - 297, all Nb-Ta alloys, the density was measured as a function of

position along the crystal. No Nb-Mo alloys were analysed by this method, for not only is the difference in density between the constituents small (1.65 g/cm^3 as opposed to 8.03 g/cm^3 for Nb and Ta), thus lowering the sensitivity of the method, but the lattice parameter of the Nb-Mo alloys is not a simple function of composition (327).

A Cameca x-ray microprobe was used to determine the composition of some of the specimens. The data were corrected for atomic number factor, absorption and fluorescence using the equations and data from references 197, 323-325. Usually only one specimen from each crystal was analysed.

Experimental Results.

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Experimental Results.

1. Alloy characterisation and use.

The composition of some of the crystals used in this study was determined by electron probe microanalysis or density measurement (see table 3). As may be seen, there is good agreement between the 'as made up' composition and the measured values, with the exception of the 60 at.%Ta crystals where the final composition is approximately 10 at.% higher than as made up.

The electron microprobe results include an estimate of the composition range, $\pm 2\sigma_m$, for which there is a 95% probability that the true composition lies within the limits stated. This is merely a statistical factor, calculated from data obtained on only one specimen per crystal, and as such it gives no information as to compositional variations which may exist over the length of the crystal in question.

On the other hand, the density measurements, done on only the Nb-Ta alloys, were made as a function of position along the crystal ; 90% of the compression specimens cut from crystals 295 and 297 were analysed, as were 25% of those from crystal 296. The composition values given in table 3 are mean values with a compositional range indicated so as to cover the range found between specimens (the theoretical sensitivity of the method with Nb-Ta alloys with the apparatus used was 0.5 at.%). There was a tendency with these Nb-Ta alloys, especially the 4 at.%Ta alloy (295), for the one or two specimens at the ends to have a higher solute content than the bulk : e.g. 6.3 at.%Ta rather than 3.7 at.%Ta for crystal 295. These 'end' specimens have not been included in the average composition referred to above or its range since the specimens gave anomalous results when tested mechanically. Data from them has also been excluded from the rest of the results section.

The compositions were not determined for all the alloy crystals but one may take the 'as made up' values as a reasonable estimate for, as has been indicated above, a good correlation exists between the 'as made up' and the 'as analysed' values. As another guide, the room temperature yield stresses have been included in table 3 for they reflect the differences in the 'as made up' compositions, e.g. in the case of the 11 at.%Mo crystals.

It can be seen from table 3 that the alloy crystals in any of the nominal composition groups have a spread in their actual composition. Since the mechanical properties of the crystals are sensitively dependent on the solute content, amongst other factors, it is important to note the distribution of results amongst the crystals in each group (see table 4). As may be seen, the great majority of the compression data was obtained from just one crystal per group, whereas in the case of tensile tests, two crystals provided most of the results, despite the appearance in table 4 that the data was collected from three to four crystals per alloy.

The dimensional measurements made on crystals after removal from the UHV furnace show that over the 3 - 4 cm adjacent to the grips there was a decrease of approximately 2 - 3% in the diameter (about 1 - 1.5 cm of this part of the crystal was always discarded and was not used for making specimens). Along the rest of the crystal the diameter typically varied by only $\pm 0.25\%$. However, the 'fuse' (see Experimental Method section 1 for description) was always found to have a significantly greater reduction in its centre where it had melted, typically 5 - 10%.

The orientations of the alloy single crystals are shown in fig.1. With a few exceptions, viz. 296, 300 and 326, they were generally close to the centre of the stereographic triangle, with

$\chi = 0 \pm 5^\circ$ and $\lambda = 50^\circ \pm 2^\circ$ (where χ is the angle between the m.r.s.s. plane and $(\bar{1}01)$ and λ is the angle between $[111]$ and the axis of the crystal). The orientation of each crystal was determined at both ends as a check on any changes over that length, but the difference between the two values was always within the accuracy of the Laue method, viz. $0.5^\circ - 1^\circ$.

As noted in the preface, some data from specimens tested during work for the part II examination for the Honour School of Natural Science (322) have been included in this thesis, eg. yield stresses, strain rate sensitivities and the temperature change test results. As can be seen in table 4, this earlier data has been supplemented by between 50 - 100% more tests on each of the alloys concerned, thus establishing the results on a sounder and broader footing, e.g. no tests were done at 145 K or below 77 K in the earlier work. The earlier data has also been corrected for the error which became apparent during the present work, namely the difference between the nominal and actual test temperatures (see "Experimental Method" section 2). Thus the curves for $\Delta H - \tau^*$ and $\Delta H_0 - \tau^*$ are the result of fresh calculations on the corrected data. The slip morphology work on these alloy compositions was all done during the present work.

2. Stress-strain behaviour.

2.1 Introduction.

Before considering the calculation and description of the stress-strain curves, several general points should be borne in mind.

(I) In virtually all the tests strain rate changes were performed and so in order to produce a curve for deformation at a constant strain rate (strictly, though, a constant cross-head speed) the strain rate change sections were effectively 'drawn out'. This procedure is

only justified if the work hardening rate is independent of the strain rate. Although this was generally true for the Nb-Mo alloys, it was not so for the Nb-Ta alloys and accordingly, although stress-strain curves are given for the Nb-Ta alloys, they are qualified ones.

(II) Because of the alterations made to the Polanyi compression machine part way through this work, the basic strain rate to which the stress-strain curves have been reduced differs between the alloys. For the 1, 2, 5 and 11 at.%Mo and 4, 10 and 60 at.%Ta alloy specimens from crystals 302, 300, 287, 292, 295, 297 and 296 respectively, the stress-strain curves are shown for a strain rate of $4 \times 10^{-4} \text{ s}^{-1}$ unless otherwise stated, e.g. many of the 1 at.%Mo and 10 at.%Ta alloy specimens prestrained at room temperature and reformed at 77 K were strained at the lower strain rate, $1.2 \times 10^{-4} \text{ s}^{-1}$ (see tables 5 and 6). All other compression curves, and all those determined in tension, are for a strain rate of $1.2 \times 10^{-4} \text{ s}^{-1}$. These comments apply equally to the yield and thermal stresses presented in the next section.

(III) In the compression tests, apart from the effects of end constraint on the work hardening rate (e.g. see ref: 101), there was also a tendency for zonal glide to occur in the Nb-Mo alloys, this being more pronounced at higher Mo levels and lower temperatures. The Nb-Ta alloys showed little or no tendency to deform in this fashion. Although tests were stopped as soon as it was obvious that zonal glide had started, usually when the load began to fall, the procedure does not guarantee that zonal glide had not started prior to that time. Thus, as the Nb-Mo alloy tests were frequently terminated by the appearance of zonal glide, the apparent work hardening rate towards the end of these tests may be deceptively low.

(IV) In tension, no results are given once the specimen has started to neck. This means that very little data is available for the Nb-5 and 11 at.%Mo alloys, for, as was mentioned in the "Experimental Method" section 1.2, necking occurred very early on in their deformation, even to the extent of doing so as the specimen yielded. Likewise, specimens which have twinned are not shown here.

In calculating the stress-strain curves, due care must be taken to present the results in such a way that meaningful comparisons may be made (i.e. to compare like with like). As will be seen later, the change from compression tests, mainly used at or above 77 K, to tension tests, used at or below 77 K, happens almost to coincide with the temperature at which several of the materials used here change their main slip system from primary glide, $(\bar{1}01)[111]$, to anomalous slip. As is set out below, this has resulted in different representations of stress and strain for the two types of testing.

Surface analysis of compression specimens has shown that, with few exceptions, the specimens had deformed by glide on $(\bar{1}01)[111]$ as the main slip system (or, in the case of the 2 at.%Mo, crystal 300, since its orientation is such that $\chi = + 15^\circ$, the main slip plane was the m.r.s.s. plane). The exceptions show anomalous slip to a greater or lesser extent, viz. Nb and 1 at.%Mo at 113 K and 77 K, 4 at.%Ta at 113 K, and possibly 4 at.%, and 10 at.%Ta at 77 K. In these alloys the anomalous slip was secondary to the main slip system, hence all the load-displacement data from the tests (or load-time in the early work) were converted to shear stress-shear strain curves using the standard single glide equations for the primary slip system (321,330). Although surface analysis of the deformed specimens had shown that in some cases secondary slip had occurred well before the stress axis

had reached the conjugate boundary, double glide has not been taken into account in the calculations. It is thought that its contribution to the total shear strain was small, and to include it would not really be justified, especially given comments I and III above. The sole exception to this was crystal 296, 60 at.%Ta, where determination of final orientations after deformation indicated that extensive conjugate slip had occurred (322). This is consistent for its initial orientation was very close to the conjugate boundary (see fig.1). In this case the double glide equations (321, 329) have been used, assuming that double glide started when the stress axis reached the conjugate boundary.

The presentation of the tension results is complicated by the presence of anomalous slip as the probable main slip system for Nb, 1 at.%Mo and 10 at.%Ta, because there is no accepted way of describing anomalous slip by shear stresses and shear strains. Although the other alloys showed primary glide, the results have all been calculated in terms of elongation and true axial stress so that they can all be presented on the same basis.

2.2 Compression.

2.2.1 Niobium (fig. 2).

As can be seen in fig.2 tests were done at only a few temperatures. In all cases the curves were parabolic in character. However, at 77 K the yield and initial flow was distinguished from that at higher temperatures by the presence of a small number of load drops. No clicks were heard from the equipment when they occurred. The load drops were usually approximately 0.05 kg/mm^2 (shear) in size, ranging up to 0.2 kg/mm^2 (shear). After 5% shear strain they ceased.

As shall be seen later, anomalous slip was dominant at 113 K and 77 K, whilst only primary glide was seen at 145 K (see "Morphology" section 5.2.1).

2.2.2 Nb - Ta alloys (fig. 3 - 5).

The addition of 4 at.%Ta has very little effect on the general shape of the Nb stress-strain curves (see 8 for Nb curves), i.e. three stage hardening at room temperature, with a transition to 'parabolic' at lower temperatures, e.g. 158 K, 113 K. No curve is included for deformation at 175 K. The two specimens tested at this temperature were both from the end of the crystal and they gave very anomalous results. At 113 K the 4 at.%Ta alloy showed a small amount of anomalous slip, cf. Nb (above) where it is dominant at this temperature. Only at 77 K did significant differences appear for the alloy twins whilst Nb deforms by anomalous slip. This twinning at 77 K can be suppressed by prestraining the specimen at room temperature, here by about 6% shear strain. Upon reyielding at 77 K there was a short region of negative then positive work hardening with slip on the primary plane, but after 15% shear strain zonal glide appeared.

On increasing the Ta content to 10 at.% one still finds very clear three stage hardening at room temperature like Nb, but as the temperature is lowered distinct differences appeared. Specimens deformed between 225 K and 113 K show a short region of low work hardening after yield, then a long region of positive, but slowly decreasing, work hardening. The hardening rate was approximately independent of temperature over the range in question. At 77 K twinning was again encountered, and as with the 4 at.% alloy room temperature prestraining was used to suppress it. However, it was found that there was a wide scatter in the reyield stresses and subsequent flow behaviour. For the seven specimens which were deformed at 77 K after room temperature prestraining, which ranged from 3.2 to 5% shear strain, the reyield stress varied between 33.4 and 26.7 kg/mm², with a tendency for large prestrains to result in the

low reyield stresses and vice versa (see table 5). Those which had the lower reyield stresses showed the highest work hardening rates after yield, whilst those with the highest reyield stresses had negative work hardening after yield. As shall be seen later (section 2.2.3) the 1 at.%Mo alloy also shows this type of behaviour at 77 K after various amounts of room temperature prestrain. In this case, however, one may positively associate the different reyield stresses and the prestrains with the presence or absence of anomalous slip, but for the 10 at.%Ta alloy the results are not so unequivocal (see "Morphology" sections 5.2.2 and 5.2.3).

For the 60 at.%Ta alloy the stress-strain curves were usually linear, but for temperatures between 225 K and 113 K there was a slow but continuous decrease in hardening rate after 20 - 25% shear strain. The hardening rate was generally higher than that found in the 10 at.%Ta alloys. The curve for deformation at 77 K was again taken from a specimen given a prestrain, some 5% shear strain, at room temperature. No specimen was tested at 77 K without this prestrain.

2.2.3 Nb - Mo alloys (fig. 6 - 11).

The effect of Mo additions to Nb was to bring about rapidly a significant change in the stress-strain behaviour of the metal. The three stage - linear - parabolic sequence with decreasing temperature found with Nb was replaced, under most conditions, by a stress-strain curve which showed a region of negative work hardening after yield, extending to 10% shear strain in some cases, followed by a section of positive hardening. This in turn may be replaced at large strains by a region of positive, but slowly decreasing, work hardening. Within this general description several trends are apparent :-

(I) The temperature at which negative work hardening after yield appears increased as the Mo content increased, e.g. in 1 at.%Mo alloy it appeared at 158 K, in 5 at.%Mo alloy at 290 K and in 11 at.%Mo alloy at 373 K. The extent of this region generally increased for a particular alloy as the temperature was lowered, although this was less true for the higher concentration alloys, 11 and 16 at.%Mo. However, there does not appear to be any systematic relationship between the duration of the negative work hardening and the alloy concentration.

(II) At temperatures greater than those mentioned in (I) above, the stress-strain curves were generally linear for the small strains considered here, usually less than 15% shear strain. For the 1 at.%Mo alloy at room temperature, deformed to 22% shear strain, there was a distinct three stage curve. The similarity in this case to Nb is very clear when one looks at the slip morphology (see "Morphology" section 5.2.3).

(III) Although care must be exercised when using these compression curves to make observations about the work hardening rates, one may note that for a particular alloy the linear hardening rates were generally rather temperature insensitive, e.g. for 11 at.%Mo at temperatures between 373 K and 113 K the hardening rate was $25 \pm 3 \text{ kg/mm}^2$ (shear). However, this average linear hardening rate increased as the Mo level was raised, e.g. it increased from 14 to 25 to 38 kg/mm^2 (shear) as one goes from 1 to 11 to 16 at.%Mo.

(IV) As the Mo levels were increased, the temperature at which virgin crystals twinned decreased. Pure Nb of this orientation glides when compressed at 77 K, whilst the 1 and 2 at.%Mo alloys required a room temperature prestrain before testing at 77 K, otherwise they twinned (but also see below). The 5 and 11 at.%Mo alloys deformed by slip at this temperature, whilst 16 at.%Mo alloy could be deformed by slip

at temperatures down to and including 8 K without twinning at yield. However, the 16 at.%Mo alloy did twin part way through the test at 20.4 K and the specimen deformed at 8 K did have a number of large load drops, corresponding to a stress change of up to 26 kg/mm^2 (shear) in size, although no twinning was found.

(V) As the Mo content was raised, the alloys showed an increasing tendency for zonal glide to appear, and for it to do so at smaller and smaller strains. Very few of the 1 at.%Mo specimens showed any zonal glide, whereas virtually all the 5, 11 and 16 at.%Mo specimens, given smaller strains than the 1 at.%Mo alloy, had their deformation stopped by the appearance of zonal glide. In addition to this concentration dependence there was a temperature dependence. For alloys with 11 at.%Mo or less, the general tendency over the temperature range in which they were tested, viz. 373 - 77 K, was for zonal glide to appear at smaller strains as the temperature was lowered. For 16 at.%Mo, this trend was followed down to 113 K and 145 K when zonal glide appeared after only 5% shear strain, but as the temperature was reduced further it was not initiated until much larger strains, e.g. 16% shear strain at 50 K.

The behaviour of the 1 at.%Mo alloy at 77 K is in contrast to the reasonably consistent trends observed in the alloys generally. As with the 10 at.%Ta alloy, the room temperature prestrain necessary to suppress twinning at 77 K resulted in a wide scatter in the reyield stresses and significant variations in the subsequent work hardening (see table 6). Specimens which had been given a larger prestrain (approximately 4% shear) tended to have higher reyield stresses (approximately 31 kg/mm^2), the converse being true for specimens given a small prestrain (approximately 3% shear), although striking exceptions are 302/19 and 302/20. Those with the higher reyield

stresses exhibited negative work hardening immediately after yield, accompanied by a small number of serrations in the flow, approximately 0.3 kg/mm^2 in size. Specimens with lower reyield stresses had higher work hardening rates and smooth flow (fig. 6 shows examples of both types). Coupled with these wide variations in the stress level, one also has the variation in the operative slip systems (see also "Morphology" section 5.2.3). At 113 K, 1 at.%Mo deformed by a mixture of glide on the primary and anomalous systems. At 77 K, specimens with a reyield stress greater than $30 - 31 \text{ kg/mm}^2$ (shear) tended to show primary slip, whilst those below this reyield stress level showed a mixture of primary and anomalous slip.

2.3 Tension.

Because of the small number of tension specimens available, especially for the alloys, no attempt was made to establish the full extent of the stress-strain curve, i.e. until necking appeared. Rather it was normal practice to unload the specimen after a relatively small strain and to then use it in temperature change tests. Thus the lengths of the stress-strain curves in fig. 11 - 18 should not be taken as indicating the maximum strain attainable before necking or other instabilities intervene.

2.3.1 Niobium (fig. 11 - 13).

The stress-strain curves for Nb deformed between 77 K and 20 K fall into two or three groups, viz. 77 K - 60 K, an "intermediate stage" at 50 K, and then 40 K and below. Although significant differences are apparent between the behaviour at these temperatures, two main common factors exist over the whole temperature range. In fact, these common factors may be extended down to 8 K, for although no stress-strain

curves were determined for virgin crystals below 20 K, temperature change tests gave stress-strain information at four temperatures between 20 K and 4.2 K. The first common factor is that at all these temperatures the specimens deformed by anomalous slip, the morphology of which was virtually independent of temperature (see "Morphology" section 5.3.1). Secondly, the work hardening rate over the first 5% axial strain, approximately $270 \pm 20 \text{ kg/mm}^2$ (axial) was likewise independent of temperature down to at least 10 K, although it was occasionally lower below 10 K. Although these two factors hold good for all temperatures between 77 K and 8 K, over the restricted interval of 77 K to 40 K there was no change in the yield and flow stresses. Only at 30 K and 20 K did the stress levels start to rise again.

The first group of curves, 77 K to 60 K, are distinguished by the presence of numerous load drops both before and after the onset of macroscopic plasticity. There were usually between one to three load drops, approximately 1 kg/mm^2 (axial) in size, at stresses well below yield, up to 9 kg/mm^2 (axial) lower in some cases. They appeared only at these temperatures and were never observed at any other temperature or in any of the alloys. At 'yield' there were several large load drops, $2 - 3 \text{ kg/mm}^2$ (axial), followed by several smaller ones, about 1 kg/mm^2 (axial), then an extended period of small, frequent serrations which eventually faded out, leaving a smooth work hardening curve. In the vast majority of tests no clicks were heard during the load drops. Although on first inspection the curves look identical, there are differences between them, notably in the nature of the load drops. Apart from the initial large load drops at yield ($2 - 3 \text{ kg/mm}^2$ axial), as the temperature was lowered the load drops became smaller and less frequent, e.g. at 77 K the average load drop was approximately $0.5 - 0.25 \text{ kg/mm}^2$ (axial), whilst at 62 K it was 0.1 kg/mm^2 (axial).

They also appeared to disappear, or become non-detectable, at smaller strains at lower temperatures, e.g. decreasing from 5.5% axial strain at 77 K, to 4% at 70 K, and 3% at 60 K.

At 50 K there was extremely irregular flow. These were not load drops as above but, rather, slow and irregular variations in the flow stress. After yield and a period of decreasing 'work hardening' the stress-strain curve becomes roughly linear, showing a (linear) hardening rate the same as that at any temperature below 77 K.

Between 40 K and 20 K the flow was perfectly smooth and, after a short, approximately 0.5% axial strain, region of low work hardening after the fairly abrupt yield, one has a linear stress-strain curve. Below 20 K, as mentioned above, no virgin crystals were deformed, but from temperature change tests one may note that this smooth flow occurred at all temperatures down to and including 8 K, with no tendency to neck or twin at all for the strains in question, up to 10% total axial strain. Even at 8 K there was significant stable plasticity, e.g. at least 3% axial strain. However, when deformation was attempted at 5 K or 4.2 K after deformation at 8 K, and other higher temperatures, it was not possible to get stable flow. Rather there were large, and very audible, load drops, up to 20 kg/mm^2 (axial) in size. However, in all cases, when the specimens were examined afterwards no trace whatsoever of twinning, grip slip, necking, shear zones or any coarsening of the fine anomalous slip was found even after six load drops of this size (see also "Morphology" section 5.3.1).

The changes in the flow behaviour of virgin crystals as the temperature was lowered, viz. serrated to irregular to smooth, were found also when temperature change tests were done : i.e. the flow behaviour of a specimen was directly related to the temperature

at which it was currently being deformed, and was not affected by previous deformation, except in that serrations were only present if the strain was below a certain level as in the case of virgin crystals.

2.3.2 Nb - Ta alloys (fig. 14 and 15).

There were only six tension specimens for the 10 at.%Ta alloy so the results obtained are rather sparse. Those tested at 77 K and 60 K deformed by glide, but at 50 K the specimen twinned. For deformation at 40 K and 29 K the specimens were given a room temperature prestrain, some 0.5% axial strain, but almost immediately after the 'reyield' at the lower temperature they too twinned. (For these prestrained specimens the 0.15% offset yield criterion was used because there was no clear transition between elastic and plastic behaviour). Therefore only stress-strain curves at 77 K and 60 K are given in fig.14.

At both these temperatures the yield and initial flow in virgin crystals was distinguished by load drops and a high, but constantly decreasing, work hardening rate.

The load drops were of a reasonably constant size, over the first 2% axial strain, $0.1 - 0.2 \text{ kg/mm}^2$ (axial) at 77 K, $0.3 - 0.5 \text{ kg/mm}^2$ (axial) at 60 K, although those at 60 K occurred less frequently. No clicks were audible during the load drops. At 77 K the load drops became less numerous as the strain increased, finally disappearing at about 2% axial strain, whilst at 60 K, at about the same strain, the load drops increased to 2 kg/mm^2 (axial) and the overall hardening rate became negative. Examination showed that this specimen was starting to neck. Both these specimens showed extensive anomalous slip (see "Morphology" section 5.3.2). Only one prestrained specimen was deformed without twinning (362/2 - see fig. 14). It was given an 0.6% axial strain at room temperature and then deformed at 77 K, when it showed the same high work hardening rate after yield as the virgin

crystal, but it did so at a lower overall stress level. However, there were no load drops and surface analysis showed that the only operative slip system was the primary.

Although the 60 at.%Ta alloy deformed by glide at 77 K without requiring a prestrain, the specimens necked immediately upon yielding. Thus all subsequent tests were done on specimens prestrained 0.6% (axial) at room temperature for it gave the specimens a certain degree of stability against this premature necking, although it also lowered the stress necessary for deformation (see "yield stress" section 3.3.2, and table 7). The stress-strain curves for these prestrained specimens deformed between 77 K and 30 K were all very similar, showing a very high initial rate of work hardening which slowly decreased with increasing strain.

In the temperature change tests, all the specimens had final strains in excess of 11% (axial) whilst showing no signs of necking. Even at 13 K, the lowest temperature at which this alloy was deformed, there was stable flow.

2.3.3 Nb - Mo alloys (fig. 16 - 18).

As mentioned in the introduction, considerable problems were found when the Nb - Mo alloys were deformed in tension. With the exception of 1 at.%Mo alloy, which twinned, all the other alloys necked immediately upon yielding. As described in the "Experimental Method" section 1.2, various approaches were tried to circumvent this problem. The only one which seemed to offer any prospect of stable deformation at low temperatures was first to prestrain the specimen at room temperature even though it altered the low temperature behaviour. The 11 at.%Mo alloy still had virtually no stability or, at best, showed about 0.5% axial strain before necking, so no stress-strain curves are reproduced here.

For the 1 at.%Mo alloy the effect of a room temperature prestrain on the subsequent deformation was very clear. At 77 K virgin crystals twinned, with one exception (326/6) which twinned after 0.5% axial strain. In this case, prior to the twinning, the curve showed numerous load drops of approximately 0.5 kg/mm^2 (axial) in size. Surface analysis afterwards showed that anomalous slip was present along with slip on other systems (see "Morphology" section 5.3.3). A prestrain of 0.54% (axial), specimen 370/3, resulted in a parabolic stress-strain curve with a yield stress some 10 kg/mm^2 (axial) lower than for the virgin crystal. Over the first 0.5% axial strain there was a series of small load drops, less than 0.1 kg/mm^2 (axial), each of which was accompanied by a sharp and very audible click. Slip was a mixture of anomalous and primary. A bigger prestrain, 1.2% (axial), specimen 332/1, produced a very similar stress-strain curve at a higher stress level, but below that for the virgin crystal. However, surface analysis showed that there was only primary slip present. This effect, viz. the size of the prestrain affecting the stress level and operative slip system on reyield, was also observed at 60 K. For the remaining tests the prestrain was kept to approximately 0.5 - 0.6% (axial) and slip was always found to be a mixture of anomalous and primary (but see "Morphology" section 5.3.3). At temperatures below 77 K the stress-strain curves were all parabolic with high work hardening rates after yield. In some cases the transition between elastic and plastic behaviour was very gradual, e.g. 370/1 at 60 K or 375/2 at 50 K. Total strains of 6 - 8% (axial) were commonly obtained, without any necking, from specimens used in temperature change tests. It was also possible to get stable flow in these tests at temperatures down to at least 8 K, where the specimen was deformed by 0.5% axial strain.

The stress-strain curves for the 3 at.%Mo and 5 at.%Mo alloys were fairly similar in character. Both alloys had parabolic type hardening curves, although at a lower work hardening rate than found with the 1 at.%Mo alloy. It was difficult in the case of either alloy to obtain much strain before necking appeared. In both single temperature and temperature change tests, maximum total plastic strains obtained during the low temperature tests were limited to approximately 5% and 3% (axial) for the 3 at.%Mo and 5 at.%Mo alloys respectively. If the strain was kept small, e.g. below 2% (axial), it was possible with the 3 at.%Mo alloy to get stable flow down to 8 K. The lowest temperature with stable flow reached for the 5 at.%Mo alloy was 20 K. Neither alloy showed any anomalous slip in any of the tests.

3. Yield and Thermal stresses.

3.1 Introduction - yield stress.

The vast majority of specimens used in this study showed a clear transition from elastic to plastic behaviour. In these cases one of three methods was used to determine the yield or reyield stress. When positive work hardening occurred after yielding, the yield stress was calculated from the intersection of the extrapolated elastic-plastic parts of the deformation curve, e.g. for the 4 at.%Ta - fig. 3. If there was negative work hardening after the yield then the yield stress was taken as the maximum stress before it began to fall, e.g. for the 5 at.%Mo - fig. 8. The third method was used with specimens which showed serrated yielding, e.g. Nb or 10 at.%Ta alloy deformed in tension at 77 K, fig. 11 - 13. Here, the yield stress was taken as the stress immediately preceding the first large load drop. The values obtained in this way were identical, for all practical purposes, to those

determined by the extrapolation method. In the few cases where these methods were not suitable, a 0.15% (axial) offset yield stress was used, e.g. some of the 10 and 60 at.%Ta alloys deformed in tension after a room temperature prestrain.

Because of the relatively small number of yield stress determinations at any one temperature, 'statistical' error bars have not been determined or used in fig. 19 and 20. Instead, where several values exist for the yield stress at a particular temperature, the range of values is shown in the figure by a vertical bar. Exceptions are the reyield stresses of the 1 at.%Mo and 10 at.%Ta alloys at 77 K after a room temperature prestrain (see tables 5 and 6). The vast majority of multiple tests had a scatter of $\pm 3\%$ around the mean value. General comments about the strain rate at yielding for the various specimens and the slip systems onto which the stress has been resolved have already been given in section 2.1.

3.2 Compressive yield stress.

3.2.1 Niobium (fig. 19).

Yield stresses were determined at only three temperatures, viz. 145, 113 and 77 K. The curve for the temperature dependence of the yield stress shown in fig. 19 and 20 is reproduced from the data of Bowen and Taylor (8) for Nb containing an estimated 0.3 at.ppm N_2 . Good agreement was found with their data at 145 K and 113 K. At 77 K, however, the 0.15% (axial) offset yield stress, which coincided with the first large load drop, was larger than the value found by Bowen and Taylor (8), 22 as opposed to 15 kg/mm^2 (shear). Although some small load drops had occurred below this 'yield' stress, the first of these was at 19.4 kg/mm^2 (shear), still well above Bowen and Taylor's yield stress.

3.2.2 Nb - Ta alloys (fig. 19).

The effect of Ta additions was to raise the yield stress at all temperatures above 77 K. For a given composition, the size of the stress increment increased as the test temperature decreased. At a fixed temperature the size of the increment increased as the amount of solute rose. The effect of solute concentration on the yield stress was, however, subtly different at the two temperature extremes. At low temperatures small additions of Ta caused a rapid rise in the yield stress, e.g. 4 at.%Ta raised the 113 K yield stress by 3.6 kg/mm^2 (shear), and 10 at.%Ta raised the same yield stress by a further 4.5 kg/mm^2 (shear). Further additions produced far smaller changes, e.g. raising the Ta level to 60 at.% only increased the 113 K yield stress by an additional 2.7 kg/mm^2 . At high temperatures the opposite was found. Small Ta additions resulted in small increases in yield stress, larger additions causing proportionally larger increases.

At 77 K the specimens twinned and a room temperature prestrain was used to suppress this. However, as has already been discussed (e.g. "stress-strain" section 2.2.2 and table 5), the reyield stress and the operative slip systems were found to be rather variable, possibly because of the presence of anomalous slip. The 77 K yield stress used in fig. 19 was that found for specimens deforming on the primary system but because of the dependence on the size of the room temperature prestrain the yield stress curve between 113 K and 77 K has been marked with a dotted line only. It should be noted that anomalous slip in Nb starts to appear at about 145 K, and anomalous slip has been observed also in the 4 at.%Ta alloy deformed at 113 K (see "Morphology" section 5.2.1 and 5.2.2.).

3.2.3 Nb - Mo alloys (fig. 20).

Before considering the general effects of alloying with Mo, three points should be borne in mind.

(a) The orientation of the 2 at.%Mo alloy, crystal 300, was different from that of the other alloys, having $\chi = + 15^\circ$ rather than approximately $+ 2^\circ$. This may account for the yield stress being lower than that for the 1 at.%Mo alloy below 150 K (see discussion section for more details).

(b) The 1, 2 and 3 at.%Mo alloys were prestrained to suppress twinning at 77 K, hence the use of dotted lines in fig.20 below 113 K, but for at least the 1 at.%Mo alloy the reyield stress and the operative slip system were very dependent on the size of the prestrain (see "stress-strain" section 2.2.3 and table 6). It was also found that prestraining the 5 and 11 at.%Mo alloys before reyielding at 77 K lowered their 'yield' stresses (table 7).

(c) With the exception of the 1 at.%Mo alloy deformed at 113 K and 77 K, only the primary and conjugate slip systems were found over the whole range of compositions and temperatures studied. The 1 at.%Mo at 113 K deformed with a mixture of primary and anomalous slip (see "Morphology" section 5.2.3), and the same was seen at 77 K if the prestrain was not too large (see table 6). The yield stress given for the 1 at.%Mo alloy in fig.20 was that found when the prestrain was large enough to promote only primary slip. When there was a mixture of primary and anomalous slip the yield stress could be as much as 12 kg/mm^2 (shear) lower.

The general effect of alloying with Mo was to raise the yield stress at all temperatures and, with two exceptions, increasingly large stress increments were found as the quantity of solute increased. However,

as was found with the Nb-Ta alloys, the effect of small solute additions on the yield stress was much more marked at low temperatures. Both the exceptions related to the 1 at.%Mo alloy, which had a larger yield stress than the 2 at.%Mo alloy below 150 K and equalled the yield stress of the 3 at.%Mo at 113 K and possibly exceeded it at 77 K. Within these general observations a slight difference was apparent between the alloys with 5 at.%Mo or less and those with a higher Mo content. In respect of the former group of alloys there was an ever rising rate of hardening as the temperature decreased which led to a considerable divergence between the yield stress curves for the alloys and Nb below approximately 120 K - 150 K. For the 11 and 16 at.%Mo alloys, on the other hand, the yield stress increased with decreasing temperature at an approximately constant rate at all temperatures.

The yield stress increment over Nb also rose constantly as the temperature fell, showing only a small region of constant hardening between 120 K and 170 K.

3.3. Tensile yield stress.

3.3.1 Niobium (fig. 21).

The yield stress for Nb showed a very clear athermal region between 40 K and 77 K. The strong temperature dependence usually associated with the yield stress reappeared only when tests were done at or below 30 K. The values shown in fig.21 are the average of at least three tests for temperatures between 40 K and 77 K. Only single tests were done at 30 K and 20 K.

The variation in the yield behaviour with temperature, e.g. pre- and post-yield load drops and the transition to smooth flow and yielding, have been discussed already in the "stress-strain" section 2.3.1, and reference should be made there for a detailed description of these changes.

3.3.2 Nb - Ta alloys (fig. 21).

The yield stress curve for the 10 at.%Ta alloy was made up with data from virgin and room temperature prestrained specimens. The virgin specimens, deformed at 77 K and 60 K, exhibited serrated yields and plentiful anomalous slip. In addition, the specimen deformed at 60 K showed a few small load drops some 4 kg/mm^2 (axial) below the stress for general plasticity. The tests at 30 K and 40 K used prestrained specimens in an unsuccessful attempt to avoid twinning. However, the specimens had yielded sufficiently to allow determination of the 0.15% axial offset yield stress before they twinned. The interpretation of these results is complicated by the observation that a room temperature prestrain can alter the reyield stress. For example, one specimen was strained at 0.6% (axial) at room temperature and then deformed at 77 K. The 'yield' stress was 7.7 kg/mm^2 (axial) lower than that for the virgin crystal and the slip system was altered from the anomalous to the primary. Thus, although the points lie on a single curve on the graph, the yield stresses from the two types of specimens have been distinguished by using a dotted line in fig. 21 to join the points from prestrained specimens.

The effect of prestrain is further seen with the 60 at.%Ta alloys. A virgin specimen deformed at 77 K 'yielded' at 62 kg/mm^2 (axial) but then necked down very rapidly. Prestraining suppressed this premature necking but the yield stress decreased to 30.5 kg/mm^2 (axial). The transition from elastic to plastic behaviour was very clear in all these prestrained specimens so there was no uncertainty in evaluating the yield stress. The yield stress curve for these prestrained specimens, although showing a small but steady rise with decreasing temperature, always lay well below the curve for the 10 at.%Ta alloy, whereas the single, unprestrained, yield stress at

77 K lay well above it.

Also shown in table 7 are the compressive and tensile yield stresses from virgin and prestrained specimens of the 10 and 60 at.%Ta alloys. Even allowing for the effect of prestraining on the yield stress, a very considerable difference between tension and compression is noticeable.

3.3.3 Nb - Mo alloys (fig. 22).

All the tensile data for the Nb-Mo alloys in fig.22 was obtained from room temperature prestrained specimens. Although the yield stress shows more scatter than was usually found, e.g. in the compression testing, it is possible to see that all the alloys exhibited an increasing reyield stress with decreasing temperature. Along with this, the reyield stress at any temperature increased with the amount of solute. In addition, because the reyield stress rose fastest with decreasing temperature in the low concentration alloys, all the yield stresses converged on going towards the lowest temperatures. Two points must, however, be borne in mind :-

(a) The effect of the room temperature prestrain (table 7). Because of the initial experimental difficulties in obtaining stable deformation in tension, yield stresses were acquired for various virgin specimens and these may be compared to the reyield stresses found with the prestrained specimens. In each case it can be seen that prestraining has reduced the 'yield' stress as was the case in compression (see also "stress-strain" section 2.3.3 for more details of the effect of prestraining on the 1 at.%Mo alloy).

(b) Although the evidence is slight, because few tests were done, prestrained specimens of the 1 and 3 at.%Mo alloys seem to show a lower

reyield stress in tension than in compression (table 7). There appears to be a smaller effect for the 5 and 11 at.%Mo alloys.

3.4 Thermal stress - compression.

3.4.1 Introduction.

The temperature dependence of the flow stress can be determined by deforming a specimen at T_1 and then reyielding it at a different temperature, T_2 , where the temperature difference is not too great, and measuring the difference in the stress required for plastic deformation. By summing these stress differences found in the temperature change tests for a series of successive temperature intervals, $T_1 - T_2$, $T_2 - T_3$... $T_{n-1} - T_n$, the temperature dependence of the flow stress between T_1 and T_n can be determined. Furthermore, if T_1 is in the athermal stress region, i.e. zero thermal stress, then the summed stress difference values will give the absolute value of the thermal stress.

As was mentioned in the "Experimental Method" section the majority of the temperature change tests were for changes to a lower temperature. This was in order to follow earlier practice (e.g. Bowen and Taylor, ref.8) where 'downward' changes were used to obviate the possibility of dislocation re-arrangement on reyielding and an associated reyield load drop. However, about 15% of the changes in this work were for increases in temperature. These tests were done with all the alloys, except the 2 at.%Mo alloy, and although they were not done for all possible temperature changes for each alloy, all possible changes were covered by tests on one alloy or another. No reyield load drops were found and the stress differences produced lay always within the scatter found for the downward changes. The majority of temperature change tests were repeated, usually more than twice. In most cases the scatter in the results was better than $\pm 6\%$ around the mean value. More generally, the size of the stress change between any two temperatures

was not found to be dependent on the strain at which the change was made, although no systematic studies were made. Likewise, the stress change between any two temperatures did not appear to depend on the temperatures at which the specimen had previously been deformed.

A very good correlation was observed between the temperature dependence of the flow and yield stresses such that the difference between the yield stress at two temperatures equalled the stress difference produced by doing temperature change tests between those temperatures. Usually the discrepancy between the two types of stress differences was less than $\pm 5\%$ of the value of the yield stress (see table 8), although the 5 at.%Mo and the 4 at.%Ta alloys were rather more variable, in some cases $\pm 10\%$. However, at 77 K there was poorer agreement, especially in the more concentrated Ta and Mo alloys.

Although the absolute value of the thermal stress for the Nb-Ta alloys may be found to be a good approximation by assuming that their thermal stress was zero at 373 K, i.e. 373 K is in the athermal plateau; this was not realistic for the Nb-Mo alloys. In order to calculate the thermal stress for these alloys, use was made of the close relationship between temperature dependence of the yield and flow stresses described above. Assuming that this relationship extended to higher temperatures, the difference in yield stress between 373 K and the athermal plateau at 915 K was taken as the value of the thermal stress at 373 K. This makes use of the 915 K yield stresses of Jax (4) who studied Nb-Mo alloys of up to 6 at.%Mo. For the 11 and 16 at.%Mo alloys the 915 K yield stresses have been calculated by assuming that the linear relationship between the yield stress and concentration found by Jax for alloys with less than 6 at.%Mo holds for more concentrated alloys. It is these thermal stresses which are considered below. For all the compression work the thermal

stress for Nb has been taken from the results of Bowen and Taylor (8) who measured the thermal stress by the same method which has been used in this work.

3.4.2 Nb - Ta alloys (fig. 23 and 24).

The addition of Ta to Nb produced at all temperatures only a slight rise in the thermal stress curve over that found for Nb. The hardening caused by the Ta increased as the temperature fell and as the amount of Ta increased, although the effect of initial solute additions was greater, relatively, than for subsequent additions (see fig.24). It was also noticeable that the rate of hardening with decreasing temperature was not constant, for between approximately 120 K to 180 K the rate was less than at higher or lower temperatures.

3.4.3 Nb - Mo alloys (fig. 25 and 26).

The effect of Mo alloying additions was markedly different to that produced by Ta. Whereas the thermal stress curves for the Nb-Ta alloys slowly diverged as the temperature fell, those for the Nb-Mo alloys initially diverged and then converged, with the thermal stress for the lower concentration alloys equalling at 77 K that of Nb to within $\pm 5\%$. Generally, the greater the Mo additions, the greater the amount of hardening, although the actual amount of hardening was temperature dependent (fig. 26). The behaviour of the 1 at.% Mo alloy below 145 K was slightly contrary to these trends. The temperature at which maximum hardening occurred decreased slightly as the solute concentration rose, e.g. from 225 K to 150 K as the Mo level rose from 1 to 16 at.%. For the 11 and 16 at.% alloys the thermal stress always appeared to be greater than for the less concentrated alloys, even below 77 K (fig. 25), with no indication of any convergence at 0 K. It will be discussed later whether this is

because the thermal stresses for both these alloys are too large as a consequence of the assumptions made initially.

3.5. Thermal stress - tension (fig. 27).

3.5.1 Introduction.

No temperature change tests were done in tension for temperatures between 298 K and 77 K. A consequence of this is that the tensile thermal stress at 77 K is not known so it is not obvious how to present and compare the results of the temperature change tests below 77 K.

Various alternatives were considered :-

(a) Assume the thermal stresses in tension and compression at 77 K are equal. This would, however, result in a striking conflict between the thermal stresses for Nb in tension and compression between 63 K and 77 K (see section 3.5.2 below).

(b) In compression the difference in the yield stress between 298 K and 77 K was found to be a reasonable approximation to the difference in thermal stress between those two temperatures. One could therefore use the difference between the tensile yield stress at 77 K and the plateau stress of Jax (see 3.4.1 above) as the thermal stress at 77 K. The drawback of this method is that the yield stress for many of the alloys was rather poorly determined because of the marked tendency to neck at yield.

(c) In compression the close relationship between the yield and thermal stresses was not drastically affected at 77 K by the use of room temperature prestrained specimens (table 8). As most of the tension specimens were prestrained at room temperature, one could use the reyield stress and Jax's plateau stress as a means of determining the thermal stress. In tension, however, the prestrain reduced the 77 K

'yield' stress by a greater amount than was the case in compression.

Although it is not totally satisfactory, method (b) has been used to present the results, but the actual results of the temperature change tests are also given in table 9. It should be kept in mind, however, that no matter which of the above methods is used the only effect is to alter the relative separation of the thermal stresses for the various Nb-Mo alloys and not to alter their relative positions. The position is less clear for the Nb-Ta alloys (see below).

3.5.2 Niobium.

The thermal stress curve in fig. 27 was made up with data from temperature change tests with 10 K sized jumps for temperatures above 20 K, and 5 K jumps below 20 K. All changes made were to lower temperatures. The stress change for each and every temperature interval was determined at least six times. The size of the stress change was not found to depend on the total strain or the temperatures at which the specimen had previously been deformed.

The feature which stands out is the lack of any temperature dependence of the flow stresses between 77 K and 40 K. Below 40 K the thermal stress rose smoothly with decreasing temperature. This behaviour is very similar to the temperature dependence of the yield stress.

3.5.3 Nb - Ta alloys.

Although it was not feasible to determine the thermal stress curve for the 10 at.%Ta alloy because of the shortage of specimens, duplicate tests were possible for the 60 at.%Ta alloy. The resulting curve for the 60 at.%Ta alloy showed a thermal stress steadily rising with decreasing temperature at the same rate as for the Nb-Mo alloys.

3.5.4 Nb - Mo alloys.

Although the temperature change tests were on average repeated twice for each alloy for each temperature jump, the curve obtained for the 11 at.%Mo alloy should be regarded with caution as it was determined from specimens which were slowly necking down. So far as it was possible to say for the other specimens, the stress change did not appear to depend on the strain or the previous deformation temperatures. It should also be noted that the 77 K base for the 3 at.%Mo alloy has been fixed by interpolating between the 1 and 5 at.%Mo alloys as no 77 K 'yield' stress had been determined.

The thermal stress curves for the alloys were very similar, showing a smooth rise with decreasing temperature. The rate of increase was approximately the same for all the alloys even though the 1 at.%Mo alloy deformed by a mixture of primary and anomalous slip whilst all the others showed only primary slip. No solution softening was found. Larger amounts of solute produced greater thermal stresses. In all four alloys the temperature dependence of the reyield and flow stresses were similar.

4. Strain rate sensitivity.

4.1 Introduction (fig. 28).

For the majority of alloys tested here no difficulty was found in establishing the stress change brought about by changes in the strain rate. In these cases a simple extrapolation procedure was used, akin to that employed in determining the yield stress (fig. 28(a)). However, for the 11 and 16 at.%Mo alloys deformed in compression at the lower temperatures, and for Nb and most of the alloys tested in tension

the character of the stress changes accompanying the strain rate change was rather less simple. In these cases various 'stress changes' were measured, and these are defined in fig. 28 (b).

4.2 Compression.

4.2.1 Niobium (fig. 29 and 30).

The curve for Nb shown in fig. 29 and 30 is, with one slight difference, that of Bowen and Taylor (8) for 0.3 at.ppm N₂. The difference is the inclusion of the strain rate sensitivity at 145 K which was determined in the course of this work. The effect of the inclusion of this extra data is to broaden the peak in the curve at 112 K. The behaviour of the specimen was in accordance with that found by Bowen and Taylor, i.e. no overshoots and no dependence of the stress changes on the strain.

4.2.2 Nb - Ta alloys (fig. 29).

The effect of Ta additions on the strain rate sensitivity of Nb was relatively small, except perhaps at 77 K. At high temperatures, after an initial reduction on first alloying, increased concentration raised the sensitivity. Below approximately 150 K the effect was reversed, with there being a reduction in the sensitivity with increasing solute concentration. At 77 K, however, the initial effect of alloying was to increase the sensitivity, the opposite of its effect at high temperature.

The size of the stress changes were found to be independent of the strain for all the alloys. There was a slight tendency for an increase in strain rate to produce marginally larger stress changes than were found for decreases in the strain rate. Occasionally strain

rate changes were made on specimens which were being used in temperature change tests. The changes in stress found in these cases were always characteristic of the temperature at which the rate changes had been made, irrespective of the total strain or of the temperatures at which the specimen had previously been deformed. The strain rate sensitivity shown in fig. 29 is the average of all the values determined at any one temperature. With one exception, the changes in strain rate produced only a monotonic rise or fall in the stress, c.f. fig. 28 (a). The sole exception was the 10 at.%Ta alloy deformed at 63 K after straining at 298 K and 77 K, when stress overshoots occurred for both increases and decreases in the strain rate, cf. fig. 28 (b). In this case the value used for fig. 29 was the peak value for the upward change, i.e. for an increase in strain rate - see fig. 28 (b).

4.2.3 Nb - Mo alloys (fig. 30 - 36).

The presentation and interpretation of the results for the Nb - Mo alloys is greatly complicated by two factors which must be noted before the particular strain rate sensitivity - temperature relationship shown in fig. 30 can be appreciated fully (fig. 30 shows the sensitivity at 5% shear strain, with peak stress values ($\Delta\tau_p$ - cf. fig. 28 (b)) used for the 11 and 16 at.%Mo alloys when overshoots appeared - see below). The first of these two factors was the appearance of stress overshoots accompanying strain rate changes for the 11 and 16 at.%Mo alloys when they were being deformed at or below 77 K or 175 K respectively. The second factor was common to all the alloys : between 77 K and 175 K (or 77 K and 373 K for the 16 at.%Mo alloy) the strain rate sensitivity increased with increasing strain. Both these points are discussed more fully below.

With these constraints in mind, several trends are discernible. Leaving aside for the moment the low temperature peaks for the

11 and 16 at.%Mo alloys, the effect of alloying was to reduce the strain rate sensitivity at low temperatures, i.e. around 100 K, and to raise it at high temperatures. Initial additions of solute, however, produced a change at both temperature extremes opposite to that found for subsequent solute additions, e.g. at 290 K, it was not until 3 at.%Mo had been added before the sensitivity was raised back to the value shown by pure Nb. Only the 11 and 16 at.%Mo alloys were deformed to any extent below 77 K and both showed a very marked narrow peak in their strain rate sensitivities at about 40 K - 50 K. The data for the 11 at.%Mo alloy was obtained from specimens which had been deformed first at higher temperatures ; the 16 at.%Mo alloy used virgin specimens.

If the simplifications employed in producing fig. 30 are removed, a more complex picture appears. Consider first the dependence of the strain rate sensitivity on the strain at or above 77 K. The effect is illustrated in fig.31 where the sensitivity of the 11 at.%Mo alloy is shown at 5% and 20% shear strain (peak sensitivity if transients present). The behaviour of all the other alloys was very similar, although for the 16 at.%Mo alloy between 113 K and 158 K the effect can only be inferred from the first few rate changes because of the very early onset of zonal glide (see fig. 10). The other simplification applied only to the 11 and 16 at.%Mo alloys, which exhibited stress overshoots at certain temperatures. The character of the overshoots depended on the temperature, strain and the direction of the rate change. Although the illustrations are taken from the 16 at.%Mo alloy (virgin specimens) the 11 at.%Mo alloy (prestrained specimens) behaved in the same fashion.

(a) The temperature dependence at a fixed strain, 5% shear. The overshoots which began to appear at 158 K became more pronounced as the temperature decreased, reaching the point at 77 K when the

stress change found on altering the strain rate was totally transient, lasting only a fraction of a second, and the steady state sensitivity was zero (fig.32 and 35). As the temperature was reduced further, the overshoots became less marked, although at 20.4 K the stress oscillated quite noticeably on the upward changes (fig.36).

(b) Between 50 K and 77 K, the flow behaviour after a change in the strain rate depended on the strain (fig.35). The peak sensitivity values for the upward and downward changes were not affected but in both cases the steady state sensitivities decreased with increasing strain (fig. 33 and 34). Whereas at 77 K the overshoots lasted only a fraction of a second, those at, say, 58 K (fig.35) lasted much longer. Below 50 K the strain rate sensitivity was independent of the strain.

(c) The peak sensitivities for increases in strain rate were greater at all temperatures than those for decreases in strain rate (fig. 34).

(d) Below 50 K the overshoots were less in evidence for the downward changes (fig. 34 and 35). For the upward changes, the overshoots were more rapid, more like small load drops.

(e) The behaviour of the 11 at.%Mo alloy paralleled the above except that overshoots appeared only at lower temperatures, below 77 K. The similarities can be seen by comparing the flow behaviour on changing the strain rate for the two alloys (fig. 35 and 36).

4.3 Tension.

4.3.1 Introduction.

As was noted in the "Experimental Method" section, two types of strain rate changes were made during tensile deformation. The 10 x rate changes, usually between 10^{-4} s^{-1} and 10^{-3} s^{-1} , are

referred to as being changes between medium (m) and fast (f) strain rates, and vice versa, i.e. m - f and f - m changes. Likewise the $2\frac{1}{2}$ x rate changes are called medium to slow rate changes, 10^{-4} s^{-1} to $4 \times 10^{-5} \text{ s}^{-1}$, i.e. m - s or s - m changes.

The 'error bars', where present, in fig. 37 and 38, indicate the range of the strain rate sensitivity found at that temperature for the alloy in question, unless otherwise stated.

4.3.2 Niobium (fig. 37 - 39).

The response of Nb to changes in the strain rate falls into three groups based on the temperature of deformation, viz. 77 K - 50 K, 40 K - 30 K, and 20 K - 4.2 K (fig. 39). This grouping by temperature and appearance closely parallels the temperature dependence of the sensitivity itself (fig. 37), although the match is not perfect, especially at the higher temperatures.

(a) 77 K - 50 K: It was difficult to determine meaningful strain rate sensitivities over this range of temperatures, for both the nature of the response, e.g. the presence or absence of overshoots, and the size of the stress changes were found to be dependent on the strain. Generally, the sensitivity, as measured by the peak stress difference for upward changes ($\Delta\sigma_p$ - cf. fig. 28) and as shown in fig. 37, increased with strain. Increases in strain rate almost invariably produced a greater stress change than was found for decreases in strain rate. Prior deformation at a higher temperature did not appear to affect subsequent deformation at a lower temperature, although no systematic work was done.

(I) 77 K : Rate changes ($\times 10$) in the serrated flow region had little effect on the flow stress. Although increasing the strain rate produced a very small increase in the stress, approximately 0.25 kg/mm^2 (axial), the stress fell almost instantaneously back to its previous 'level'. Equivalent behaviour was found on decreasing the strain rate. Only once smooth flow had appeared was there any permanent stress rise, although stress overshoots of various sizes were always present. The sensitivity increased with strain until approximately 6% (axial) when the changes stabilised at the value shown in fig. 37 ($\Delta\tau_p \uparrow$).

(II) 70 K : No permanent flow stress changes were observed for strains up to 6% (axial) even though by then the flow was smooth. Only overshoots were found. The stress changes ($\Delta\tau_p \uparrow$) were much smaller than those at 77 K or 60 K.

(III) 60 K : The behaviour was very similar to that at 77 K although the flow was rather more irregular.

(IV) 50 K : Flow was very erratic. Occasionally a decrease in the strain rate produced a rise in the flow stress, after an initial fall. Only at larger strains was it possible to unambiguously determine the strain rate sensitivity.

(b) 40 K - 30 K : Flow was perfectly smooth with 'ideal' strain rate changes, cf. fig. 28 (a). No overshoots were observed. The sensitivity was independent of prior deformation temperatures, strain, sense of the rate change (i.e. f - m or m - f), or the size of the rate change ($2\frac{1}{2} \times$ or $10 \times$).

(c) 20 K - 4.2 K : Overshoots began to appear again at 20 K for f - m and m - f changes. The prominence of these overshoots increased as the temperature was decreased, because of the progressive lowering of the steady state strain rate sensitivity. At 10.5 K stress oscillations were seen after increases in the strain rate (but m - f changes only), whereas an apparently negative steady state strain rate sensitivity was found after decreases in strain rate (f - m changes only). At 8.25 K, massive load drops, approximately 11 kg/mm^2 (axial) in size, followed increases in strain rate. No evidence for twinning, adiabatic slip, shear zones, necking, coarse slip or grip slip was found on the specimen after deformation. The strain rate sensitivity (from $\Delta\sigma/\dot{\epsilon}$) was independent of strain at all temperatures.

When $2\frac{1}{2} \times$ rate changes were performed at 16 K and 10.5 K, no overshoots or stress oscillations were observed. The stress change was equal for increases or decreases in the strain rate, and the strain rate sensitivity was the same as that calculated from the $10 \times$ changes using the peak stress change for upward changes ($\Delta\sigma/\dot{\epsilon}$). It is these values which are shown in fig. 37. The value at 4.2 K is from unpublished work by Taylor.

The strain rate sensitivity curve in fig. 37 and 38 has been 'dotted in' between 77 K and 50 K because of the nature of the flow behaviour accompanying strain rate changes. As shown, the low value found at 70 K has been 'drawn over'.

4.3.3 Nb - Ta alloys (fig. 37 and 40).

Although there are many similarities between the two alloys, it should be noted that a major difference between them was that the 10 at.%Ta alloy deformed primarily by anomalous slip whilst the

60 at.%Ta alloy showed only primary glide, at least at the higher test temperatures (see section 5.3.2). Further to this, it can be seen from the tensile tests at 77 K that the strain rate sensitivity depended on which slip system was operating. Virgin specimens of the 10 at.%Ta alloy deformed by anomalous slip and had at large strains a sensitivity approximately half that of a prestrained specimen which showed only primary glide (the sensitivity of the virgin specimen increased with strain). When the sensitivities at 77 K in tension and in compression are compared, they are found to be the same for each alloy so long as the specimens were deforming by the same slip system. Other more general observations are summarised below :-

(a) The presence of stress overshoots and the instances when an increase in strain rate produced larger peak stress changes than found for a decrease in strain rate were closely linked together for both alloys. Overshoots were found for all m - f and f - m changes made between 77 K and 30 K (fig.40 - see (b) for behaviour below 30 K). In all these cases peak stress changes were larger for the upward changes. With the $2\frac{1}{2}$ x rate changes, no overshoots were observed at or below 30 K, the stress changes being equal for increases or decreases in the strain rate. Changes made above 30 K behaved in a fashion similar to the 10 x rate changes. As was found for the Nb, the strain rate sensitivity was independent of the size of the rate change if the peak stress difference was used, when necessary, in the calculations. It is these values which have been used in fig. 37 : see below for the strain dependence.

(b) The m - f changes made on both alloys strained below 30 K were followed by large load drops and unstable or oscillatory flow. This type of behaviour was found also for the s - m changes at 13 K

with the 60 at.%Ta alloy. These load drops were associated with necking or shear zones.

(c) The dependence of the peak and steady state strain rate sensitivities on the strain during stable flow were slightly different for the two alloys. For the 10 at.%Ta alloy the peak sensitivity between 77 K and 50 K increased with strain. At 77 K and 60 K, though, the sensitivity was indeterminate at low strains because of the serrated and irregular nature of the flow. Below 50 K no dependence on strain was observed. The strain dependence of the steady state sensitivity paralleled that of the peak sensitivity at all temperatures (but see below), the effect of this being that the overshoots did not become more prominent as the strain increased or the temperature fell. The sole exception was at 29 K when there was a marked increase in the prominence of the overshoots at all strains.

For the 60 at.%Ta alloy, the peak and steady strain rate sensitivities from tests between 77 K and 52 K showed little dependence on strain so long as the strain was less than 5% (axial). At 42 K, and 52 K for strains greater than 5% (axial), the steady state sensitivity decreased with strain making the overshoots more prominent. This is in addition to the general trend that the overshoots became more dominant as the temperature fell to 30 K.

(d) Some 60 at.%Ta alloy specimens were deformed at 77 K without a room temperature prestrain, and rate changes on these specimens resulted in overshoots as in the case of prestrained specimens.

4.3.4 Nb - Mo alloys (fig. 38 and 41).

The general trend seen in fig. 38 was for the strain rate sensitivity to decrease at all temperatures as the Mo content rose.

However, there are several qualifications and other observations to be borne in mind :-

(a) The Nb - Mo alloys were very prone to early necking, with their stability decreasing as the Mo concentration rose. Because of this it was possible to make only a few strain rate changes and accordingly it could not be determined whether there was any dependence of the sensitivity or the overshoots on the strain. The 11 at.%Mo alloy almost invariably started to neck just after the start of the deformation so the values given for the strain rate sensitivity must be only approximate.

(b) Overshoots were found for all 10 x rate changes made on prestrained specimens (fig. 41). Their prominence tended to increase as the temperature decreased. At low temperatures, 20 K - 30 K, oscillatory flow and apparent negative strain rate sensitivity appeared. This was, with one exception (5 at.%Mo deformed at room temperature and then at 22 K), always associated with the necking of the specimen. If the specimen was not necking down 'ordinary' overshoots were observed, i.e. like fig. 28 (b), e.g. 1 or 11 at.%Mo alloys at 22 K after room temperature prestrain. In the only test below 20 K, m - f rate changes produced large load drops from the formation of shear zones, e.g. 1 at.%Mo at 17 K after a room temperature prestrain.

(c) Some rate changes were made during the deformation of a few of the early tests on virgin crystals which necked on yielding. In all cases no overshoots were seen, even after approximately ten rate changes (see fig. 41). Such tests were done only on the 3 at.%Mo alloy at 77 K, the 5 at.%Mo alloy at 70 K and 60 K and the 11 at.%Mo alloy at 60 K. This behaviour was in contrast to that found for the Nb-Ta alloys.

(d) The $2\frac{1}{2}$ x rate changes produced slight overshoots when the temperature was at or above 50 K, or 60 K for the 11 at.%Mo alloy. At lower temperatures no overshoots were found, even at temperatures as low as 8 K.

(e) The strain rate sensitivity calculated from the $2\frac{1}{2}$ x rate changes (peak values if overshoots present) was the same as that evaluated from the peak stress change for m - f rate changes. Increases in strain rate (10 x or $2\frac{1}{2}$ x when overshoots were present) always produced larger stress changes than were found for decreases in strain rate. When no overshoots were found the strain rate sensitivity was independent of the sense of the rate change.

(f) Because the crystals in any nominal composition group have a range of compositions, any comparison between the sensitivity at 77 K for tension and compression cannot be very exact when based on measurements taken from different crystals. When the (probable) compositional differences are allowed for, the strain rate sensitivities in tension at 77 K for the 3 and 5 at.%Mo alloys are very similar to their compression values, although poorer agreement was found for the 11 at.%Mo alloy. Good agreement was found also for the 1 at.%Mo alloy irrespective of whether it was deforming in tension by primary glide alone or a combination of primary and anomalous slip.

5. Slip Morphology,

5.1 Introduction.

The information presented in this section was gathered by examining crystals which had been deformed in one of two ways. The first method simply involved examining the specimen at the end of a test. This approach did not allow a very accurate determination

of the operative slip systems because of the large strains involved nor was it ideal in that the surface contained the sum of all the slip traces from the deformation. This prevented any strain dependence of the structure being seen. However, the method did avoid the problem found in incremental strain studies, especially those done at low temperatures, namely that unloading the specimen and warming it up to room temperature for inspection may affect the subsequent deformation and slip structure on redeformation at the lower temperature. This was especially noticeable with specimens deformed at or below 77 K, for on reyielding after a room temperature excursion they frequently deformed at a considerably higher stress than previously. The second method used was to strain the specimens incrementally. In this a specimen was deformed approximately 5% (shear), examined, deformed another 5%, and the process repeated to a maximum strain of 20 - 25% (shear). The disadvantages of this method were really the converse of those of the first. Attempts were made to polish the crystals after each 5% strain in order to look at the slip structure formed in just one strain interval. These were unsuccessful for the chemical polish never completely eliminated the previous slip structure or gave a really good surface finish, especially in those alloys which had coarse slip lines.

It was routine practice to examine all specimens deformed in tension whether they had been deformed at only one or at several temperatures. Whilst not all the crystals were given a full multi-face surface analysis, in all cases at least half the surface of each crystal was examined thoroughly. About half to three-quarters of specimens deformed in compression at a single temperature were given a full multiface surface analysis, but those deformed at several temperatures were usually examined only briefly as a check.

The accuracy of the multiface surface analysis was limited by two main factors, namely the large strains at which most specimens were examined, typically 20 - 40% (shear), and the inherently wavy nature of the slip traces in the alloys, especially on or near the top face. However, it was possible to obtain accuracies typically within 5° in the assignment of the primary slip plane pole to a specific (hkl) plane, and of $7^\circ - 10^\circ$ for the secondary slip systems. With specimens deformed to smaller strains, especially those in tension showing more crystallographic slip, e.g. anomalous slip, higher degrees of accuracy were achieved, e.g. $1^\circ - 2^\circ$ for the anomalous plane and other major slip planes. There was the additional complication in specimens with anomalous slip in that some of the minor systems operated over only a very small percentage of the surface but it was nevertheless possible to obtain good accuracy in their analysis.

As the slip morphology of bcc metals and alloys depends upon the orientation of the surface one examines, the orientation of the specimens in the following photographs is referred to by means of the angle θ . This is the angle by which the specimen has been rotated around its longitudinal axis from the position where the [111] direction, the primary Burger's vector, is contained within the surface, i.e. for $\theta = 0^\circ$ or 180° one is looking at the side face of the crystal (the side face so far as primary $(\bar{1}01)$ slip is concerned) and for $\theta = 90^\circ$ one is looking at the top face. In all cases the stress axis is parallel to the longest edge of the photograph. It should also be noted that the (hkl) 'lines' beside some of the figures are provided merely to identify the various slip systems. They are only schematic and are not meant accurately to represent the trace of that plane at that particular surface orientation.

5.2 Compression.

5.2.1 Niobium (fig. 42 - 46).

The few Nb specimens which were deformed in compression showed clearly a transition in slip system as the temperature was lowered. At 145 K slip was almost exclusively on the primary plane ($\bar{1}01$) and was very fine over the whole of the specimen. After 2% axial strain some traces of anomalous slip were seen, but by 19% axial strain only the fine primary slip was visible. However, when virgin crystals were deformed at 113 K, fine evenly distributed slip was clearly visible on the ($0\bar{1}1$) plane after 2.4% axial strain (fig. 42 - the triangular marks are spurious post-test artefacts). There was also a small amount of very fine slip on the primary plane, together with some activity on (011). It was not possible to detect the 'dead band' for anomalous slip.

At 77 K there was a marked change in the slip morphology (fig. 43 - 46). No longer fine, the anomalous slip appeared in discrete, well separated, straight slip lines, accurately parallel to ($0\bar{1}1$), completely encircling the specimen and dominating the slip structure. With increasing strain there was a strong tendency for clusters of these lines to form giving coarse bands of anomalous slip with little activity between them (fig. 43 - 46 cover axial strains of 2.6% - 8.3%). Irrespective of the strain, there was always a dead band in the anomalous slip lines which corresponded to an apparent Burger's vector $\approx 5^\circ$ from $[011]$ lying on the great circle joining $[011]$ and $[\bar{1}11]$. However, the exact position of the dead band varied from slip line to slip line, so that the apparent Burger's vector had a range of $\pm 10^\circ$ around this average value.

As well as slip on the anomalous system, activity was present on three other $\{110\}$ systems. The most prominent of these was (101) $[\bar{1}11]$. This was found over the whole of the specimen's surface at all strains and was always in the form of strong straight slip lines or bands. Although these slip lines always terminated on an anomalous slip band, they varied in that some were quite short, extending only between adjacent $(0\bar{1}1)$ bands (e.g. fig. 45 and 46) whilst others would cover a quarter of the specimen's circumference before ending (e.g. like those in fig. 44). The only effect of strain was generally to increase the number of the (101) slip lines. At all strains the dead band was very clear, indicating $[\bar{1}11]$ as the operative Burger's vector.

The glide on the other two $\{110\}$ planes was in marked contrast to the strong straight bands found on the $(0\bar{1}1)$ and (101) planes. Slip on the primary plane $(\bar{1}01)$ was visible at all strains but always in the form of a weak wavy background (e.g. fig. 43). Although found at all surface orientations of the specimen, it was not present uniformly over the specimen, hence its absence from the other photographs. Traces of glide on the (011) plane were extremely fine and were confined to within $\pm 15^\circ$ of its top and side face (assuming an operative Burger's vector of $[\bar{1}\bar{1}1]$). These were almost always present as short slip segments adjacent to the anomalous slip bands. As the strain increased they disappeared.

5.2.2 Nb - Ta alloys (fig. 47 - 54).

As has been seen in the previous sections, Nb - Ta alloys do not show dramatic property changes over Nb. This type of behaviour may also be seen in the alloys' slip morphology. Consider first of all slip in the 10 at.%Ta alloy at various temperatures and strains (see 58, 60, 289 for the slip morphology of Nb).

At room temperature the 10 at.%Ta alloy has a 2 - 3 stage work hardening curve similar to Nb (fig. 4). One specimen (312/5) was strained incrementally to 7, 12, 18 and 24% (shear) to allow the relationship between the slip structure and the work hardening curve to be seen. At the start of stage I (7% shear strain) fine primary slip, $(\bar{1}01)$ $[111]$ was seen on both the top and side faces, that on the top surface being reminiscent of 'orange peel' (fig. 47 and 48). As the strain on stage I increased, the overall morphology was little altered, only becoming slightly stronger, i.e. more distinct. However, just before the work hardening rate increased at the end of stage I, at 12% shear strain, secondary slip on (011) $[\bar{1}\bar{1}1]$ began to form in bands parallel to the $(\bar{1}01)$ plane. These were only visible near the primary's side face. By 18% shear strain, after the increase in the work hardening rate, these bands of secondary slip had begun to break out of their originally discrete and localised bands and to spread over the surface (fig. 50). In many places the original bands had disappeared in the uniform secondary slip. The top face, however, had no visible secondary slip, only primary slip traces of an irregular nature, as before, but rather stronger (fig. 49). Further straining merely resulted in the secondary slip on the side face spreading out away from that surface and the total elimination of the original banded structure (fig. 51 - 24% shear strain). The secondary slip remained much coarser than the rather fine primary slip.

At lower temperatures the three-stage work hardening curve was replaced by one which was nearly linear (fig. 4). At low strains, however, the slip structures were rather similar, e.g. at 175K and room temperature figs. 52 and 53 show the top and

side faces after 4.7% shear strain at 175 K. Again there was fine, straight slip on the side face and irregular traces on the top face. As the strain increased to 16% (shear) little change was visible in the slip structure. Above 16% shear strain some rather restricted secondary slip began to appear on the side face of the primary system. By 40% shear strain, however, the secondary slip was visible over the whole of the surface. As at room temperature, the secondary slip was much coarser than the primary which had altered little in appearance with the increasing strain. This pattern was typical of slip on specimens deformed down to 113 K. Indeed, it was not easy to say from looking at the slip line structures at low strains at what temperature the specimen had been deformed.

Specimens deformed at 77 K exhibited a range of behaviour. Virgin crystals twinned, with $(\bar{1}21)$ and $(\bar{2}11)$ as the major twin planes, together with a small amount on (211) . Prestraining at room temperature suppressed the twinning, but it was a little unclear precisely what effect it had in promoting the appearance of primary or anomalous slip. As stated previously, the 77 K reyield stress for both 1 at.%Mo and 10 at.%Ta showed a fair degree of scatter (see tables 5 and 6). For the 1 at.%Mo alloy (see section 5.2.3 below) this may be related to the presence or absence of anomalous slip. However, for the 10 at.%Ta alloy, none of the specimens which were examined after only the 77 K deformation (and the room temperature prestrain) showed any anomalous slip (but see below). Rather, they had deformed on the primary system (except perhaps 297/22 - see below). Most specimens were subsequently deformed at lower temperatures where some showed a mixture of anomalous and primary slip (297/21, 22 and 312/6, see table 5) whilst others continued to deform on the primary system (297/17, 18 and 20). Unfortunately two of the

three specimens which did show anomalous slip after deformation at the lower temperatures were not examined after straining at 77 K so it is not known what slip system was operating at 77 K. The one specimen that was examined, 297/22, appeared to be free of any slip traces even after 4% shear strain at 77 K and 1.7% at room temperature. Nevertheless, there does appear to be some correlation between the amount of room temperature prestrain and the subsequent appearance, or non-appearance, of anomalous slip since the three specimens which did show anomalous slip at the lower test temperatures had the smallest room temperature prestrains.

When anomalous slip did appear it was in the form of strong straight slip lines distributed uniformly over the specimen together with primary slip, also in the form of strong slip lines, and some twinning. On analysing specimen 312/6 the dead band in the $(0\bar{1}1)$ slip lines corresponded to an effective Burger's vector of $[011] \pm 5^\circ$, i.e. $\pm 5^\circ$ along the great circle passing through $[011]$ and $[\bar{1}11]$. It was also noteworthy that the axial rotation of this specimen during deformation was towards the pole of the $(0\bar{1}1)$ plane (fig. 55- arrow No.1). In those specimens which showed only primary slip the morphology was rather akin to that at room temperature or 175 K at low strain, being only slightly coarser and with no tendency to form coarse slip bands even after 20% shear strain at 77 K.

No incremental straining studies were done on the 4 at.%Ta alloy. The specimens were examined at large strains, above 25% (shear), and always contained extremely fine slip. This was especially so at the lower temperatures, e.g. 158 K and 113 K. It was extremely difficult at these temperatures to make out the slip traces at all, but at 113 K there was a small amount of clear, very straight, slip parallel to the trace of the $(0\bar{1}1)$ plane. It was present on two

opposing faces of the specimen, each region covering approximately 15% of the crystal's circumference. At 77 K there was only primary slip but the specimen had been prestrained at room temperature by 9% (shear) to suppress twinning on $(\bar{2}11)$ and (211) .

The slip structure of the 60 at.%Ta alloy was examined after incremental tests at room temperature, 175 K, 113 K and at 77 K after a 4% (shear) prestrain at room temperature. As was mentioned in the "Experimental Method" section it was difficult to obtain a reasonable surface finish for this alloy and the surface was not particularly smooth (fig. 54). For this reason fine slip traces may not be visible. This might be the case at room temperature, for after 12% shear strain no slip traces were visible at all, although at 175 K, 113 K and 77 K they were quite clear. At these lower temperatures only primary glide was observed. Although the tests at 175 K and 113 K were strained up to 13% (shear) and that at 77 K to a total strain of 25% (shear), the slip structure was apparently independent of strain or temperature. The slip was always moderately fine, although noticeably coarser than for the 10 at.%Ta alloy. Fig. 54 shows slightly branched slip on a face half-way between the top and side faces for deformation at 175 K. On the top face the traces were rather wavier and less well defined than the ones in the picture. Those on the side face were always fine and straight.

5.2.3 Nb - Mo alloys (fig. 56 - 83).

The Nb - Mo alloys exhibited a wide variety of slip morphologies. To study these, some alloys, viz. 1 and 11 at.%Mo, were examined after incremental straining at room temperature, 175 K and 77 K. The other alloys, viz. 2, 5 and 16 at.%Mo, were looked at only at the end of their deformations, so the strains were variable and

rather large. Consider the slip morphology for each composition as a function of temperature.

To a large extent the 1 at.%Mo alloy behaved very similarly to the 10 at.%Ta alloy, and this is seen clearly at room temperature. As with the 10 at.%Ta alloy, there was a 2 - 3 stage work hardening curve and a very similar development of the slip structure. Here the specimen was examined after 6.2, 11.6, 17.2 and 23.2% shear strain (see fig. 6 for the stress-strain curve for the specimen in question, 302/22). At the start of stage I there was fine 'orange peel' on the top face and a fine slip structure on the side face (fig. 56 and 57). The slip lines on both faces became stronger as the strain in stage I increased, but by 11.6% shear strain fairly coarse secondary slip on (011) $[\bar{1}\bar{1}1]$ had begun to appear in bands parallel to the trace of the $(\bar{1}01)$ plane. The number of bands increased with increasing strain, although it was always slightly less than in the case of the 10 at.%Ta alloy. By 17% shear strain the bands were regularly spaced along the side face although none had yet broken out. A further increase in the strain was accompanied by a rise in the work hardening rate. Associated with this was the appearance of general secondary slip on the side surface where some of the secondary slip bands had begun to break through the primary slip bands (fig. 58). Not all, however, had done so by this strain (fig. 59). This change in the work hardening rate occurred at a larger strain than for the 10 at.%Ta alloy, 17% as opposed to 13% shear strain. On the top surface the slip traces were still 'orange peel' in character but stronger now than at the lower strains (fig. 60).

On lowering the temperature to 175 K a distinctly different slip morphology appeared. At low strains, 2.7% (shear), slip near the side face was virtually invisible, whilst on the top face narrow,

distinct, wavy slip bands approximately parallel to the trace of the $(\bar{1}01)$ plane had formed (fig. 61). Secondary slip on $(011) [\bar{1}\bar{1}1]$ was present over the whole of the specimen. With increasing strain the distinct nature of the slip bands on the top face disappeared and were replaced by a more uniform and far less easily identified structure (fig. 62 - 16.4% shear strain). In contrast, the increasing strain made the slip lines on the side face more easily visible, revealing a uniform structure of fine straight slip traces with a small amount of secondary slip (fig. 63).

This form of primary slip morphology was also seen at 113 K after 12.5% shear strain, i.e. fine slip on the side face and well defined wavy bands on the top face although no slip was observed on the (011) plane. In addition to these dominant primary slip traces there were numerous fairly strong and very straight slip lines which were accurately parallel to the trace of the $(0\bar{1}1)$ plane (fig. 64). Their dead band, which was not well defined, corresponded to an effective Burger's vector of approximately $[011]$. These regularly spaced anomalous slip lines were found over the whole specimen.

At 77 K, several types of deformation were observed as was the case also with the 10 at.%Ta alloy. Virgin crystals twinned, with $(\bar{2}11)$ and (211) as the major twin planes. Prestraining at room temperature suppressed the twinning but as already seen (e.g. in table 6 and stress-strain section 2.2.3) the 77 K reyield stress showed considerable scatter and the operating slip systems varied from primary alone to primary and anomalous together (fig. 65). There did not appear to be a simple relationship between the size of the prestrain and the appearance of anomalous slip. Rather, one can say only that those specimens which had a 77 K reyield stress below approximately 31 kg/mm^2 were likely to show anomalous slip whereas for larger values

only primary slip was found. In either case the primary slip usually appeared in coarse, well spaced bands (fig. 66). The anomalous slip took the form of clear, well spaced slip 'lines' accurately parallel to the trace of the $(0\bar{1}1)$ plane. They were not present over the whole of the crystal but only on two opposing regions each region covering approximately one-quarter of the crystal's circumference. It was not possible to find the dead band for the $(0\bar{1}1)$ slip lines but $[011]$ was parallel to the surface in that region of the crystal which did not show any of these slip lines. The final orientation of specimens with mixed primary and anomalous slip indicated an axial rotation towards neither the $(0\bar{1}1)$ nor the $(\bar{1}01)$ pole but rather a compromise between the two with a bias towards the $(0\bar{1}1)$ pole (fig. 55 - note that the specimens had been deformed at temperatures below 77 K as well).

As mentioned above, no incremental tests were done on the 2 and 5 at.%Mo alloys, observations being made only at large strains. Given this limitation, only general comments may be made. Consider first the 2 at.%Mo.

At room temperature the slip structure was generally similar to that in the 1 at.%Mo alloy, but some differences were apparent. On the top face the slip traces were still reasonably fine but they were also identifiable as separate traces rather than as having constantly been criss-crossing each other (fig. 67 - note the different magnification). On the side face, fine straight traces were present and some bands of secondary slip were just beginning to form parallel to the trace of the $(\bar{1}01)$ plane, although the work hardening rate had not yet increased (fig. 7). Although this behaviour was very like that for the 1 at.%Mo alloy, the secondary slip appeared at a larger strain than in the more dilute alloy, 14% as opposed to 11% (shear). Lowering the temperature, e.g. to 175 K, had little effect on the

'scale' or distribution of the slip. However, on deformation at 77 K there was a marked change in the morphology. Virgin crystals twinned on the $(\bar{2}11)$ and $(\bar{1}21)$ planes. A room temperature prestrain suppressed the twinning, and on reyielding at 77 K, these specimens had coarse primary slip bands, especially on the top face, even after relatively large strains, 18% (shear) in fig. 68 and 69. Secondary slip on (011) $[\bar{1}\bar{1}1]$ was visible between the primary slip bands.

In the 5 at.%Mo alloy the trend, noticeable in the more dilute alloys, viz, that of similarities at higher temperatures and differences at lower ones, was seen to develop further. After 35% shear strain at room temperature slip was still moderately fine on both the top and side faces with that on the top face again clearly consisting of separate slip bands (fig.70). Decreasing the temperature initially produced no marked change in the slip morphology from that found in the 2 at.%Mo alloy, certainly for temperatures down to 175 K. However, at 145 K a very different morphology appeared. Individual slip bands were clearly visible all over the specimen, those on the top face being the most prominent and irregular (fig. 71 - 15% shear strain). Moving towards the side face the slip traces tended to group together rather than appear as individual bands. Deformation at lower temperatures, e.g. 77 K, resulted in coarse slip bands akin to those seen in the 2 at.%Mo alloy. Usually no room temperature prestrain was needed at 77 K to suppress twinning, but the alloy's behaviour was a little variable in this respect.

No anomalous slip was observed in the 2 and 5 at.%Mo alloys in tests down to 77 K. However, in one temperature change test (room temperature-77 K - 20.4 K) on the 5 at.%Mo alloy, a considerable number of very straight slip lines parallel to the trace of the $(0\bar{1}1)$ plane were found on two opposing faces of the specimen. Their dead band

corresponded to an effective Burger's vector approximately 5° above $[011]$, lying on the great circle joining $[011]$ and $[\bar{1}11]$. This strongly suggests that they are genuine anomalous slip lines. Given the sensitivity of anomalous slip to the size of the room temperature prestrain it should be noted that before deformation at 20.4 K the specimen had already been strained 11.2% (shear).

An increase in Mo level in the alloy to 11 at.% resulted in further changes in the slip line structure. This is clearly seen in specimens deformed incrementally at room temperature to 5.5, 7.9 and 14.8% shear strain. At low strain, 5.5%, the top face contained relatively straight and narrow but branching slip bands (fig.72). As the strain increased the surface between the initial bands filled up uniformly with other slip traces so that by 14.5% shear strain the surface had very little apparent structure at all (very reminiscent of the 10 at.%Ta alloy deformed 4.7% (shear) at 175 K, fig.52). Continued straining, up to 28% (shear), produced little or no change. The side face contained closely spaced bands of primary slip and increasing strain again resulted in the slip spreading between the bands and producing a surface virtually devoid of features. Little secondary slip was observed.

At 175 K the initial development of the slip structure was very similar to that at room temperature. Narrow bands formed on the top surface (fig. 73 - 7.3% shear strain) and with increasing strain fresh bands formed between them (fig. 74 - 12.1% shear strain) until the surface was uniformly covered and little surface structure was discernible. A similar sequence of events occurred on the side face. However, in specimens which were strained to 28% (shear) without interruption the slip structure was dominated by coarse, well spaced, primary slip bands with some secondary slip between the bands. This trend towards coarser slip bands at lower temperatures was quite marked,

especially at 113 K and 77 K. At 77 K, broad, well separated, yet branching and irregular bands dominated the surface (fig. 75 - 3.5% shear strain). Only towards the side face did the bands spread out to give a more uniform coverage (fig. 76). Further straining, up to 28% (shear), produced only a slight broadening of the bands.

The trend of the above observations for the 1 - 11 at.% Mo alloys, at low strains, has been for 'discrete' slip bands to appear at higher temperatures as the Mo content increased. In the 16 at.% Mo alloy, coarse bands were visible at all temperatures despite the relatively large strains at which they were examined, typically 13 - 21% (shear). No twinning at yield was found in virgin crystals deformed at temperatures as low as 8 K. However, at 8 K plastic flow was irregular with several large load drops, typically equivalent to a stress change of 26 kg/mm^2 (shear). Despite this there was no sign of twinning on the specimen. At 20.4 K the specimen twinned after 8% shear strain but smooth flow reappeared immediately after the twin drop at the same stress level as before. At room temperature slip bands were present on both top and side faces (fig. 77 and 78). On the top surface there was a moderate degree of branching and irregularity, especially in the narrower bands. Reducing the temperature to 175 K, then 77 K, and finally 20.4 K, resulted in less branching and straighter, more parallel, bands (fig. 79 - 81). However, the basic scale of the slip structure on either face was not altered to any great extent. At 8 K the slip bands were virtually all parallel with very little branching and few deviations, even on the top face (fig. 82 and 83). At all temperatures very little secondary slip was seen. The little that was found always appeared on the specimen's primary slip side face.

As has been seen above, an increase in the Mo concentration or a decrease in the temperature has tended to produce a coarser slip

structure. However, there does not appear to be a simple relationship between the thermal stress and the slip structure at low strains as has been proposed by Statham (73). For example, consider the 1 at.%Mo alloy at 113 K (fig. 64) or the 5 at.%Mo alloy at 145 K (fig. 71) with the 11 at.%Mo alloy at 175 K (fig. 73 and 74). The values of the thermal stresses were very similar but the resulting slip morphologies were rather different. These and other differences between the slip morphology found in the course of this study and the work of Statham (73) will be considered in the discussion section.

5.3 Tension.

5.3.1 Niobium (fig. 84 and 85).

A total of eighteen Nb specimens were deformed in tension at temperatures between 77 K and 4.2 K (those specimens deformed below 20 K had always been deformed previously at a higher temperature). Every specimen was examined after the deformation, half of them being given a full surface analysis. The strains at which they were examined ranged from 1.8% to 12.3% (axial), the average value being about 8% (axial). Approximately half the specimens were deformed at several temperatures. In all these specimens, irrespective of the temperature(s) of deformation or the strain, several common features were found : -

(1) The totally dominant slip lines were accurately parallel to the trace of the $(0\bar{1}1)$ plane. They were always extremely fine and straight and evenly distributed over the entire surface of the specimen. Although fig. 84 and 85 were taken at 77 K and 40 K at strains of 1.8% and 3% (axial) respectively, they could have been taken on any one of the specimens.

(2) Most of the specimens also have some straight slip lines on (101). These were either in the form of long coarse single bands, some $40^\circ - 80^\circ$ long, or short coarse bands only about 5° long, i.e. the specimen could be rotated by α° before a single continuous slip band under observation disappeared. The (101) slip was always present over the whole of the specimen's circumference. However, it was not at all common, some specimens containing only half-a-dozen or so bands, e.g. after 10.9% (axial) strain at ten temperatures between 77 K and 5.5. K, or 1.8% (axial) strain at 77 K. There was also a marked tendency for the (101) bands to appear near the specimen's grips.

(3) The majority of the specimens also contained some very fine slip on (011). This was in the form of very short slip lines, typically 2 - 4 μm long. It was only visible on two opposing faces of the specimen in such a way that the (011) slip lines were approximately perpendicular to those of the anomalous slip. These short (011) slip lines were often very closely spaced along some of the anomalous slip lines, e.g. some 1 - 2 μm apart.

(4) No $(\bar{1}01)$ slip was observed at all.

(5) The dead band for the $(0\bar{1}1)$ slip lines always corresponded to an effective Burger's vector approximately $6^\circ - 11^\circ$ 'above' [011], lying on the great circle joining [011] and $[\bar{1}11]$. Although it was easy to find, it did vary slightly ($\pm 3^\circ$) over the length of the specimen.

(6) The dead band for the (101) slip was always very clear and indicated that the operating Burger's vector was $[\bar{1}11]$.

As was described in the stress-strain section some specimens deformed below 8 K underwent a series of large load drops corresponding to stress changes of up to 20 kg/mm^2 (axial). These specimens were carefully examined after the test but the slip morphology was exactly the same as that described above. There were no extra coarse slip bands, e.g. on $(0\bar{1}1)$, no unusual slip systems activated, e.g. $(\bar{1}01)$, no shear zones, no sign at all of 'pull out' of the specimen from the grips, and no twins (except in one specimen which had four tiny twins near one grip).

5.3.2 Nb - Ta alloys (fig. 86 - 89).

When virgin crystals of the 10 at.%Ta alloy were deformed at 77 K in compression they twinned. The switch to tension produced a marked change in behaviour with the specimens deforming by glide at 77 K and 60 K. The slip structure was dominated at both temperatures by numerous straight, evenly distributed slip bands on the $(0\bar{1}1)$ plane set against a background of wavy and fairly weak slip on the primary system $(\bar{1}01)[111]$ - see fig. 86. The dead band in the $(0\bar{1}1)$ slip lines corresponded to an effective Burger's vector approximately $8^\circ \pm 3^\circ$ above $[011]$, lying on the great circle between $[011]$ and $[\bar{1}11]$. Whilst these two slip systems appeared over the whole of the specimen's surface, slip on (011) was also present but was very restricted in its operation, only appearing over about 10% of the total surface and then only on two opposing faces of the specimen. The slip traces were generally fairly weak (fig.87). Slip on (101) was very rare and it was found over an even smaller range than in the case of the (011) slip. In contrast to this anomalous slip in virgin crystals, a specimen prestrained 0.45% (axial) at room temperature and then reyielded at 77 K showed only fine primary slip (specimen 362/2, fig. 14).

At 50 K the specimen twinned. Coarse bands of anomalous slip were found on a background of primary slip (fig. 88), along with twins. Those specimens deformed at lower temperatures, viz. 42 K and 29 K, were prestrained 0.5% (axial) at room temperature but they twinned almost immediately on reyielding. However, after this twinning, smooth deformation followed. In both cases anomalous slip bands on a background of the primary slip were found along with some glide on (101) - see fig. 89. No (011) slip traces were found. The effective Burger's vector of the anomalous slip was as found at 77 K, i.e. about 6° above [011]. However, although anomalous slip was common, large areas of the surface of the twinned specimens showed only primary slip and twins.

When looking at the 60 at.%Ta alloy, it was not easy to say unequivocally what slip systems were operating. As has been noted before, it was not found possible to obtain a good surface finish on the specimens. Further, all the specimens were prestrained by 0.6% (axial) at room temperature. Many were then reformed at several temperatures, and not just one, because of the shortage of specimens. Those which were cut from crystal 377 and deformed at several temperatures starting at 77, 62, 51 and 42 K showed identical slip structures in that they all contained fine primary slip visible over the whole specimen. However, two other specimens (336/3 and 339/1) were deformed to 7% axial strain at 52 K and 42 K respectively after a 0.6% axial prestrain at room temperature. Their surfaces were almost entirely devoid of any visible slip lines except for some very straight and strong ones accurately parallel to the trace of the $(0\bar{1}1)$ plane. These traces were visible over only a very restricted range of the specimens' surface, not more than 5° .

5.3.3 Nb - Mo alloys (fig. 90 - 96).

It has already been seen that in order to postpone premature necking in the Nb - Mo alloys it was found necessary to prestrain the specimens. This resulted in a change in the slip morphology (see below). However, for the 1 at.%Mo alloy the effect was rather more complex.

Although a switch from compression to tension often suppresses twinning, when virgin crystals of the 1 at.%Mo alloy were deformed at 77 K or 70 K they twinned on (112), ($\bar{1}12$) and ($1\bar{1}2$). However, one specimen yielded and only twinned after approximately 0.5% axial plastic strain (specimen 326/4 : see "stress-strain" section 2.3.3 and fig.16). In both these cases there was, along with the twins, a considerable amount of slip on the ($0\bar{1}1$) plane, with some on ($\bar{1}01$) and (101) and a little on (011). Slip bands on the ($0\bar{1}1$) plane were always very straight and were present over the entire surface of the specimen (fig.90 and 91). The dead band in these ($0\bar{1}1$) slip bands again corresponded to an effective Burger's vector about 6° above [011]. The primary slip and that on (101) were very much background to the anomalous slip.

When a room temperature prestrain was used to suppress twinning, the slip systems which operated on redeformation at 77 K or below were found to be very sensitive to the size of this prestrain. Those prestrained less than 0.6% (axial) at room temperature always showed a mixture of anomalous and primary slip after straining at 77, 40 or 20 K. Larger prestrains, even if only 0.8% (axial), produced only fine primary slip on reyielding at 77, 60 or 30 K. In those specimens which did show anomalous slip there was always a background of primary slip along with some wavy slip on (101) and (011) irrespective of temperature. In contrast, the appearance of the anomalous slip lines was temperature dependent, although care must be taken in interpreting the results because of the different strains. In going from 77 K (fig. 92) to

40 K (fig. 93 and 94) and then 22 K (fig. 95 and 96), the anomalous slip bands become much coarser with fewer bands being generated between the main ones. In all three cases it was not possible to locate accurately the dead band for the anomalous slip for there was no visible $(0\bar{1}1)$ slip in a band $\pm 15^\circ$ around the surface orientation which contained $[011]$. However, it was possible to confirm that there were no dead bands when any of the $\langle 111 \rangle$ vectors were parallel to the surface.

With the higher Mo content alloys, i.e. those containing 3 at.% Mo or more, virgin crystals necked rapidly after yield and their slip structures were always one of coarse primary slip bands and conjugate slip. To gain some stability the specimens were prestrained at room temperature, usually by 1 - 2% (axial). The slip structure was reduced in all cases to a fairly fine and homogeneous primary slip with no strong bands at all. There was little variation in this observation between the different temperatures and alloy compositions. No anomalous slip was seen in any specimens.

Discussion.

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Discussion.

1. Quality of the materials used.

The methods and equipment used in this work to make high purity Nb single crystals were virtually identical to those employed by Garrett-Reed and others (8, 288, 326). These authors used various methods to determine the purity of their crystals, e.g. mass spectrometric analysis, resistivity ratio, and thermodynamic calculations on the solubility of interstitial solutes. Although the answers produced by the different methods were not always consistent with each other, it was concluded that the probable levels of impurities were approximately 1 at.ppm interstitials, 50 at.ppm Ta, and 5 at.ppm other substitutional solutes (but see also Reed (331) where the total interstitial solute concentration in Nb crystals prepared in a similar manner was estimated to be rather larger).

No attempt was made to measure the impurity levels in the Nb or alloy single crystals which were used in this present work. As the deformation behaviour of Nb is known to be sensitively dependent on the quantity of interstitial solute (e.g.8), the observed close similarity and behaviour between the Nb prepared for the present work and that prepared by the authors mentioned above, has been taken to indicate that the Nb crystals used in all the various studies contain similar quantities of impurities. No such comparison can be made for the Nb-Ta and Nb-Mo alloys, as no comparable work has been published. However, an idea as to the level of interstitial solute can be obtained by looking at the effect of Ta or Mo solute additions on the thermodynamics of interstitial solubility in Nb. Reference to gas-metal equilibrium studies shows that Ta additions marginally increase the solubility of nitrogen, whereas Mo additions cause a rapid decrease in nitrogen's solubility (332 - 334). Thus on

thermodynamic grounds it is to be expected that interstitial solute levels would be of the same order as those found in the UHV annealed Nb, i.e. a few atomic ppm.

One of the variables determining the interstitial concentration is the final annealing temperature. In this work the highest annealing temperature reached was limited by a fuse in series with the main crystal. This fuse was intended to melt when the main crystal was approximately 100 K below its solidus. This requirement fixes the ratio of their diameters, cf. Stefan's law. After the anneal, though, it was noticed that the fuse was always tapered towards its centre where it had melted, i.e. its diameter had been preferentially reduced by evaporation in the centre. Thus crystals set to be annealed 100 K below their solidus were really annealed at some lower temperature. It can be estimated using Stefan's law that most crystals only reached a maximum temperature some 150 K - 250 K below their solidus. This was, however, only the temperature of the bulk of the crystal for the ends were held in relatively large Nb grips and therefore would have been cooler. This was reflected in the slight taper found at the ends of each crystal. It might also be expected that the interstitial concentrations would be larger than that of the bulk crystal. The only crystal in which these end effects appeared to be noticeable was 295, the 4 at.%Ta alloy, for specimens taken from near its ends gave atypical results (see "stress-strain" section 2.2.2). Since the anneal given to crystal 295 was in any event suspect because of a component failure in the furnace, it seems likely that this crystal was annealed at a lower temperature than usual with the result that end effects would have extended further from the grips and into the material usually used for testing. It was also found that the end one or two specimens from this crystal were rather richer in solute

than the bulk of the material (see "alloy characterisation" section).

Earlier work using the same electron beam floating zone furnace as was used here has shown that Nb - Mo zone refined alloy single crystals were both microscopically homogeneous and of a uniform composition along their length (320). The only work done here on the homogeneity of the alloys after a UHV anneal was a determination of the composition along the length of crystals 295 and 297, 4 and 10 at.%Ta alloys respectively. In both cases there was a rise in the Ta content for the one or two end specimens, this being more marked for the 4 at.%Ta alloy. However, compositional fluctuations along the main body of the crystal were small. Certainly no atypical results were found in any of the alloy crystals, Nb - Ta or Nb - Mo, which could be attributed to compositional fluctuations, with the exception of the 4 at.%Ta alloy mentioned above. Of more importance is the spread in the actual composition of crystals in nominally the same compositional group (table 3). This must especially be kept in mind when interpreting the tensile results because of the use of data from several crystals whereas the data on compression comes from just one crystal per alloy.

2. Materials deforming by primary glide.

2.1 Thermodynamics of glide.

The thermodynamic model for thermally activated glide is outlined briefly in the appendix. Although some useful insights may be gained from the model's application, due caution should be exercised because of probable infringements of the model's assumptions and also the non-ideal behaviour of some of the alloys, e.g. the appearance of overshoots.

2.1.1 Compression (fig. 98 - 105).

The activation volumes (fig. 98, 99) have been calculated from the strain rate sensitivities shown in fig. 29 and 30. Thus the activation volumes for the Nb - Mo alloys are those determined at 5% shear strain : activation volumes for the Nb - Ta alloys are independent of strain. Further to this, the 11 and 16 at.%Mo alloys showed very marked overshoots at low temperatures ; peak stress changes for increases in strain rate ($\Delta\tau_p$) were used to calculate the strain rate sensitivity and hence the activation volume.

The difference between the effects of the two solutes is most marked (fig. 98, 99). Additions of Ta produced relatively little change in the form or size of the activation volume except at high thermal stresses when the broad peak in $v^* - \tau^*$ found for Nb was suppressed. There was also a slight, though very consistent, increase in the activation volume with increasing solute concentration at all thermal stress levels. Additions of Mo, on the other hand, rapidly brought about a radical alteration in the behaviour of Nb. Whilst the 1 at.%Mo alloy behaved like the Nb - Ta alloys, i.e. monotonic, smooth dependence on stress and no peak in the activation volume at high stresses, increasing Mo levels rapidly raised the level of the $v^* - \tau^*$ in addition to producing small peaks at large thermal stresses. These peaks became more prominent in the more concentrated alloys.

The differences between the two alloy systems is also very noticeable in the dependence of the activation enthalpy, ΔH , on the thermal stress (fig. 100 and 101). The Nb - Ta alloys show a ΔH decreasing smoothly and monotonically with increasing stress. Increased Ta content always raised the level of the $\Delta H - \tau^*$ curve at all stresses. The effect of Mo additions was rather different. As was seen with the activation volume, the 1 at.%Mo alloy behaves in a similar manner to

the Nb - Ta alloys, but for greater amounts of solute, ΔH , whilst decreasing with increasing stress, does not do so smoothly at all. Rather, the curves tend to show a shoulder or inflexion after the initial decrease in ΔH . Indeed, for the 16 at.%Mo alloy, there is a peak in the $\Delta H - \tau^*$ curve.

The thermodynamic analysis may be taken one stage further to calculate ΔH_0 , the absolute height of the barrier. To do this it is necessary to know the activation volume at (effectively) zero thermal stress. Since this has not been determined for these alloys, a relatively arbitrary cut-off has been taken at $140b^3$ for the Nb - Ta alloys and $200b^3$ for the Nb - Mo alloys. The arithmetic consequence of this arbitrary step is that the absolute value calculated for ΔH_0 calculated for each alloy may well be incorrect, as will be the absolute numerical difference between the alloys. However, since the $v^* - \tau^*$ and $\Delta H - \tau^*$ curves show that at any particular stress increasing solute concentration increases the value of ΔH and v^* in both alloy systems, it would not be expected that this cut-off procedure would alter the relative values of ΔH_0 (assuming that the maximum activation volume is not dependent upon the solute concentration). Likewise, the choice of the value for the maximum activation volume will not affect the arithmetic dependence of ΔH_0 on stress for any particular alloy, provided that the assumptions made in determining ΔH_0 are valid.

The result of this analysis shows that the alloys fall into two groups, each with a different stress dependence for ΔH_0 , and ΔH (fig. 102). The first group consists of the 1 at.%Mo alloy and all three Nb - Ta alloys. For these alloys, ΔH_0 depended little, if at all, on the stress, except that there is a slight increase at low stresses in the more dilute Nb - Ta alloys. The second group contains the 2 - 16 at.%Mo alloys, although a distinction should be drawn between

the 11 and 16 at.%Mo alloys, and the more dilute Nb - Mo alloys. Nevertheless, the second group as a whole is marked by a discontinuity in the $\Delta H_0 - \tau^*$ curve along with an inflexion or plateau in the $\Delta H - \tau^*$ curve. At high stresses, ΔH_0 is independent of stress, although this is less true for the 16 at.%Mo alloy. At low stresses, although ΔH_0 is lower than at high stresses, the exact stress dependence is a little unclear because there are only two data points in this region for each of the 2 - 5 at.% Mo alloys. Because of this, it is uncertain whether ΔH_0 is independent of, or possibly slowly increasing with, stress. For the 11 and 16 at.%Mo alloys the variation in ΔH_0 at low stresses is again a little unclear, although the dependence is relatively slight. The transition to the larger ΔH_0 at high stresses is rather more protracted than in the case of the more dilute alloys. Although the step in the $\Delta H_0 - \tau^*$ plot for the 2 - 5 at.%Mo alloys appears at greater thermal stresses as the concentration increases, the temperature at which it occurs is relatively constant, being between 175 K and 225 K. For the 11 and 16 at.%Mo alloys the step starts to appear at approximately 160 K.

In interpreting the results the stress dependence of ΔH_0 and ΔH should be considered first as this sheds considerable light on the dominant thermodynamic factors. For the first group of alloys, viz. 1 at.%Mo and all three Nb - Ta alloys, ΔH_0 is independent of stress. Both this, and the smooth decrease of ΔH with increasing stress, accord well with the idea of a single thermally activated step being rate determining over the whole range of thermal stresses investigated here, as envisaged in the model outlined in the appendix. This simple explanation obviously does not hold for the second group of alloys, 2 - 16 at.%Mo, where ΔH_0 increased markedly above a particular thermal stress, yet was otherwise independent of the stress.

To understand first of all the behaviour of the 2 - 5 at.%Mo alloys, take the case where there are two possible thermally activated rate controlling processes operating in parallel, i.e. the 'slower' process will be rate limiting because the two mechanisms are in some way dependent upon each other's operation. In fig. 103 the obstacle back stress versus area slipped is sketched for these two different mechanisms, with the curve ADG representing Mechanism One (M1) and the curve A'YG' Mechanism Two (M2).

At a stress τ_1 , M1 requires no thermal activation, all the work necessary to surmount the barrier being supplied by the stress. M2, though, still requires thermal activation and will be the rate determining step. At a stress τ_2 both M1 and M2 need thermal activation to get over the barrier. However, at this stress it is assumed that the amount of work needing to be done by thermal activation is greater for M2 than for M1, i.e. the work necessary for thermal activation for M1 corresponding to the area (CDE) is less than that required for M2, the area (C'YE'). Thus, other factors being equal (but see below), M2 will still be rate limiting. At a low stress, τ_3 , one can envisage the situation being reversed. Thus ΔH for M1, represented by the area (BDF) will be greater than ΔH for M2, area (B'YF'). So, other factors being equal as before, M1 will now be the rate determining step.

At a stress somewhere between τ_2 and τ_3 there will be a change as to which mechanism is rate controlling. However, one of the assumptions made in allowing an evaluation of ΔH_0 by the graphical integration method used here is that only one rate controlling process is present over the whole stress range in question. If this is not the case then it is possible to envisage how the step in the $\Delta H_0 - \tau^*$ can appear, although it requires one to pass over simpler models in order to obtain the desired effect.

The simplest criterion to determine the switch over might be expected to be that of the activation enthalpy. However, as will be shown below, this does not produce the observed step in the $\Delta H_0 - \tau^*$ plot. To understand why this is not so and to see how the step may be explained it is first necessary to outline why a simple ΔH criterion is not sufficient. At τ_c , fig.103, ΔH for the two mechanisms are equal, i.e. area $(\alpha D \beta)$ equals area $(\alpha' Y \beta')$. But a consequence of the graphical integration method used to evaluate the integral in the expression for ΔH_0 (see appendix) is that ΔH_0 also appears to be equal at this stress for both mechanisms :-

$$\Delta H_0 (M1) = (\alpha D \beta) + (A \alpha \beta G) = (ADG)$$

$$\Delta H_0 (M2, \text{apparent}) = (\alpha' Y \beta') + (A \alpha \beta G)$$

$$\text{but } (\alpha' Y \beta') = (\alpha D \beta)$$

$$\therefore \Delta H_0 (M1) = \Delta H_0 (M2, \text{apparent})$$

Above τ_c , e.g. at τ_2 , M2 is rate controlling. Then :-

$$\Delta H_0 (M2, \text{apparent}) = \Delta H + b \int_0^{\tau^*} A^* d\tau^*$$

$$= (C' Y E') + \left[(A \alpha \beta G) + (\alpha' C' E' \beta') \right]$$

$$\text{but } (C' Y E') + (\alpha' C' E' \beta') = (\alpha' Y \beta') = (\alpha D \beta)$$

$$\text{therefore } \Delta H_0 (M2, \text{apparent}, \tau > \tau_c) = \Delta H_0 (M1, \text{actual})$$

Hence no step or discontinuity appears in the $\Delta H_0 - \tau^*$ plot. The clue which suggests the explanation is to be found in the behaviour of the $\Delta H - \tau^*$ curves, fig. 101, and it is best exemplified in the 2 at.%Mo alloy. After decreasing sharply as the thermal stress increases from zero, ΔH reaches a pseudo plateau between approximately 3-7 kg/mm²

before decreasing further with increasing stress. All the other Nb - Mo alloys show this characteristic to a greater or lesser extent (except the 1 at.%Mo alloy which behaves like the Nb - Ta alloys). In fig. 104 the stress dependence of ΔH for M1 and M2 is sketched. The observed behaviour of the 2 at.%Mo alloy suggests that the transition between M1 and M2 as to which is rate controlling is delayed until the stress reaches τ_b . The consequence of this delay in the transition is to produce a step in the $\Delta H_0 - \tau^*$ plot as may be seen from the following considerations (fig. 105). At τ_b , with M1 rate controlling ($\Delta H (M1) < \Delta H (M2)$) :-

$$\Delta H_0 (M1, \tau < \tau_b) = (XDZ) + (AXZG)$$

but when the transition to M2 occurs, the stress is observed to increase at approximately constant ΔH . Thus :-

$$\Delta H_0 (\tau > \tau_b) = (P'YQ') + \left[(AXZG) + (X'P'Q'Z') \right]$$

$$\text{i.e. } \Delta H_0 (\text{apparent}, \tau > \tau_b) = \Delta H_0 (M1, \tau < \tau_b) + (X'P'Q'Z')$$

Hence a step appears in the $\Delta H_0 - \tau^*$ plot as a consequence both of the delayed transition between M1 and M2 and of the graphical integration method used here to evaluate the work done by the thermal stress. Thus at stresses above the transition, ΔH_0 as shown in fig.103 is only an apparent ΔH_0 which does not correspond to the actual ΔH_0 for M2 or M1.

Several points can be brought forward to help explain why the M1 - M2 transition is 'delayed' for the 2 - 5 at.%Mo alloys. A more realistic criterion governing the transition is which mechanism requires the largest stress so that the specimen can continue to deform

at the applied strain rate. From the simple rate equation :-

$$\dot{\gamma} = \dot{\gamma}_0 \exp(-\Delta G/kT)$$

one can explain the observed behaviour by postulating different $\dot{\gamma}_0$, the pre-exponential factor, for the two mechanisms (M2 having a larger $\dot{\gamma}_0$ than M1). For the two mechanisms to generate the same strain rate at the transition point ΔH (M1) would have to be smaller than ΔH (M2), as envisaged in fig. 104. A complementary interpretation is to note that M1 and M2 are not independent of each other. As will be outlined below, one can associate edge /mixed and screw dislocation motion with M1 and M2 respectively. A more general form of the rate equation is :-

$$\dot{\epsilon} = p_s b / \bar{v}_s + p_e b / \bar{v}_e$$

where p_s , p_e are the mobile densities for screw and edge dislocations respectively, and v_s , v_e are the velocities for the same. If the terms of the equation are interdependent, e.g. as in the case of an expanding loop where p_s depends on v_e and vice versa, then it becomes difficult to see intuitively what mechanistic criterion should be applied to determine when the transition between edge and screw dislocation as to which is rate controlling should take place.

With pure bcc metals, such as Nb, the transition to screw controlled dislocation glide occurs almost as soon as the thermal stress rises above zero. For the alloys under discussion here, the transition occurs at a thermal stress greater than that found for the pure metal. Although the screw dislocations are not rate controlling at these stresses ($\tau < \tau_b$), they still require thermal activation to enable them to glide. Thus one may expect them to show some variant of the edge-screw dislocation interdependence observed in the pure metals, especially as the stress approaches the transition point, τ_b .

Several examples of the inter-related nature of dislocation motion have been noted or observed in in-situ straining experiments in the case when screw dislocation motion is rate controlling.

(1) The screw dislocation density will depend on the free path length of the edge dislocations, and vice versa.

(2) The velocity of a screw dislocation depends upon its length, amongst other factors (81). The faster and the further the edge dislocations move, the faster the screw dislocations can move.

(3) It has been shown in in-situ experiments that there are several dislocation multiplication mechanisms, e.g. from superjogs (81). The velocity of the edge-mixed dislocation produced depends on the separation of the screw dislocations it generates by its own motion. This separation will, in turn, depend on the velocity at which the screw dislocations which are being generated move apart.

Given these interconnected relationships, it does not seem obvious what mechanistic criterion should be taken in order to predict when the M1 - M2 transition will occur.

Finally, consider $\Delta H_0 - \tau^*$ for the 11 and 16 at.%Mo alloys. The discontinuity appears at a much larger stress than is found for the 2 - 5 at.%Mo alloys. In conjunction with this, the strain rate sensitivity for the 11 and 16 at.%Mo alloys is split into two separate peaks (fig. 30). This corresponds to a peak in the $v^* - \tau^*$ curve at high thermal stresses (fig. 99), unlike the other alloys which show a continuously decreasing activation volume with increasing stress. The peak activation volume occurs at the same thermal stress as that at which ΔH_0 appears to become independent of stress again after the step in the $\Delta H_0 - \tau^*$ curve.

These observations may be understood qualitatively by considering the case of a thermal barrier which may be overcome by a screw dislocation in one of two different ways (M2 and M3). The mechanism which requires the lowest stress to allow the applied strain rate to be matched will be the one which operates. A model developed by Duesbery (41) examined this phenomenon by investigating the stress dependence of various mechanisms, e.g. different dislocation configurations and transformation paths, whereby a screw dislocation could surmount the Peierls barrier. The calculation indicated that since the stress dependence of the various mechanisms was different, each mechanism would operate over a particular stress range. The model was applied by Statham (101) to explain the peak found in the $v^* - \tau^*$ curve for concentrated Nb-Mo and Nb-Re alloys (see also ref. 218 for Nb-W alloys). If it is assumed that $\dot{\gamma}_0$ is different for the two possible mechanisms, M2 and M3, then the ΔH for M2 and M3 at the transition stress will not be equal for the same reason as was outlined for the M1 - M2 transition. A similar argument can be developed to explain the appearance of the step in the $\Delta H_0 - \tau^*$ plot and the peak in the $v^* - \tau^*$ curve.

No step in the $\Delta H_0 - \tau^*$ plot is found at low thermal stresses for the 11 and 16 at.%Mo alloys. This may be because no tests were done at a low enough thermal stress to allow M1 to be rate determining, hence no M1 - M2 transition would be observed. However, a simplistic extrapolation of the thermal stress at which the ΔH_0 step appeared for the 2 - 5 at.%Mo alloys might lead one to expect that the transition should be capable of being observed. It must be remembered, though, that the absolute value of the thermal stress for all the Nb-Mo alloys depends upon the use of plateau yield stresses of Jax (4) to allow an estimate of the thermal stress between the

plateau at 915 K and the start of tests in this work at 373 K. Since Jax only determined yield stresses for alloys up to 6 at.%Mo it was necessary to extrapolate his data to 11 and 16 at.%Mo. If the linear extrapolation used is not valid then the absolute thermal stress for the 11 and 16 at.%Mo alloys will be wrong, but in what sense the quoted values would be incorrect is unknown.

The thermodynamic analysis used so far indicates that different rate controlling processes operate in the various alloys as the thermal stress rises. What other evidence can be brought forward to support the assertions made above that M1 can be associated with non-screw dislocation motion, and M2 and M3 with variants of screw dislocation glide?

It has been accepted for several years that the low temperature properties of pure bcc metals are governed by the large Peierls stress for screw dislocations and their consequential low mobility. This has been demonstrated both directly, e.g. in-situ straining experiments (81, 265), and indirectly, e.g. atomistic dislocation core models (37). Other evidence to support this position has been reviewed several times (e.g. 78, 79, 198).

The addition of substitutional solute to group $\overline{VA}$ metals has been found, usually, to increase the thermal and athermal stresses. Electron microscopy has shown that such alloys contain, amongst other structural changes, a larger density of longer and straighter screw dislocations than would be found in the pure base material at the same temperature (14, 137). This has been taken to show that the solute further greatly reduces the already low screw dislocation mobility. However, substitutional solute also has a marked effect on the velocity of non-screw dislocations. This has been inferred by Foxall (137) from observations made on the size and shape of glide loops in Nb-Mo alloys as a function of temperature and Mo content. Indeed, this

indirect evidence was taken to suggest that the edge-screw dislocation velocity ratio, v_e/v_s , is reduced by substitutional alloying (14,80,137).

More direct evidence as to the effect of substitutional solute comes from in-situ deformation studies on Nb-Ta, Mo (263) and Fe-Si (258, 261). With Nb-Mo and Fe-Si alloys, which both show strong solution hardening, the velocities of both screw and non-screw dislocations were reduced by alloying. However, the effect was greater for non-screw dislocations, i.e. v_e/v_s was reduced, but only for alloys containing 2 at.%Mo or more ; 1 at.%Mo had little effect on the velocity ratio. Decreasing temperature increased the velocity ratio for all the alloys. The critical temperature was also raised, e.g. 5 at.%Mo increased T_c to 300 K, approximately 60 - 100 K above that for Nb. In contrast, Ta additions to Nb had little effect on the dislocation velocity, either absolute or relative.

Transitions in the rate controlling mechanism as the temperature, and hence the thermal stress, alters will reflect the relative ease of motion of the various dislocation processes. For pure bcc metals like Nb, the transition to screw dislocation control occurs just above zero thermal stress. If the velocity ratio, v_e/v_s , is reduced by alloying, screw dislocations will not become rate controlling until a larger thermal stress can be applied. More solute will further reduce v_e/v_s , thus further increasing the thermal stress at which the non-screw - screw dislocation transition takes place. Another consequence of screw and non-screw dislocations being thermally activated is that an increase in the height of the barrier will increase the temperature at which the thermal stress falls to zero.

The disproportionate increase in the barrier faced by edge dislocation motion at low thermal stresses in Nb-Mo alloys may be explained in part by noting that, at least in linear elasticity theory, the principal edge dislocation - solute atom interaction is through the size misfit

effect, although the effect from the modulus difference is not insubstantial (339). Within the framework of linear isotropic elasticity theory, screw dislocations do not interact with misfitting solute atoms as the screw dislocation does not have a dilational field. If second order elastic effects are included, however, an interaction between a screw dislocation and a centre of dilation does appear (109) ; otherwise the modulus effect is the main means of interaction. Since the Nb-Mo alloys have a far larger size misfit parameter than the Nb-Ta alloys (5.8×10^{-2} as opposed to 5.8×10^{-4} : see table 10) it would be expected that the edge dislocation's velocity would be reduced by a larger amount in the Nb-Mo alloys, notwithstanding any contribution from the modulus effect. The strengths of the modulus interactions for screw dislocations in the two alloy systems are far closer together, the Nb-Mo modulus misfit parameter being only a factor of 1.6 larger than that in the Nb-Ta system (0.548 and 0.339 respectively : see table 10). However, some authors (291) use different elastic moduli, and the relative sizes of the interactions depend on which elastic constants are considered relevant. Nevertheless, the difference between the alloy systems is never so great as that found for the edge dislocation - size misfit interaction. The net result of these various solute - dislocation interactions will be to reduce the velocity ratio, v_e/v_s , in the Nb-Mo alloys to a greater extent than in the Nb-Ta alloys. These simple rationalisations agree qualitatively with the observations made in the in-situ straining experiments.

The ideas set out above agree very well with the observations made in the present work. The first group of alloys, 1 at.%Mo and all three Nb-Ta alloys, showed relatively little hardening at high temperatures, suggesting the lack of a strong solute dislocation interaction. The lack of any discontinuity in $\Delta H_0 - \tau^*$ or $\Delta H - \tau^*$, coupled with the

observations of Ikeno (263) from in-situ straining experiments, suggest that in these alloys the non-screw - screw dislocation transition, M1 - M2, occurs at a very low thermal stress and that the whole of the low temperature domain is controlled by screw dislocation glide as the rate limiting factor. Greater amounts of Mo increase the high temperature hardening and push the M1 - M2 transition to a larger thermal stress so that the step in the $\Delta H_0 - \tau^*$ plot and the inflexion in the $\Delta H - \tau^*$ curve become apparent in the 2 - 5 at.%Mo alloys.

The step in the $\Delta H_0 - \tau^*$ plot for the 11 and 16 at.%Mo alloys has already been discussed in terms of a change between two mechanisms (M2 - M3) for a screw dislocation to overcome the Peierls barrier. Such a change would not be detectable by such methods as in-situ deformation.

Another consequence of the non-screw - screw dislocation transition is that it can go part way towards explaining the very large activation volumes found at low thermal stresses. The activation volume associated with thermally activated screw dislocation glide by double kink nucleation is relatively small, at most $50 - 100 b^3$ (41), and the model cannot explain the large activation volumes of $100 - 500 b^3$ found for some of the alloys. However, at these stress levels, non-screw dislocation glide is thought to be rate controlling. In current solution hardening theory, the size and nature of the obstacle facing the dislocation is seen as being temperature dependent in itself. At 0 K the dislocation interacts with each individual solute atom. At higher temperatures where thermal activation is present the effective obstacles are groups or clusters of atoms and the stress required to pass them is less than that at absolute zero (143). The more concentrated the solid solution, the larger the clusters/groups, and the greater the amount of solution hardening. If the non-screw dislocations in bcc solid solutions are governed at low thermal stresses by this bowing-out/break-through model,

activation volumes far greater than those associated with double kink nucleation on screw dislocations are to be expected. This may also be rationalised on the basis of fig. 103. As the total area under the curve ADG, ΔH_0 (M1), is greater than the area A'YG', ΔH_0 (M2), and, given that the transition stress, τ_b , is small compared with the maximum stress required to operate M2, then the activation volume at low thermal stresses must be far larger than it is when the stress is near the transition point, τ_b . Thus the activation volume will vary rapidly with stress at low stresses, $\tau < \tau_b$.

2.1.2 Tension (fig. 106).

The activation volumes have been calculated from the strain rate sensitivities given in fig. 38, i.e. from maximum stress changes for increases in strain rate. The nature of the overshoots observed in the majority of the rate changes is discussed below in section 4.

The activation volumes at these low temperatures and high stresses are all small, although they increase slightly with greater amounts of solute, as is also seen in compression. The stress dependence is very slight, especially for the more dilute Nb-Mo alloys and the 60 at.%Ta alloy. Both the size and the weak stress dependence at these high stresses are to be expected if screw dislocation glide is controlled by double kink nucleation (41). The size of the activation volumes agrees fairly well with those determined in compression at the same temperature (77 K). No maxima are found in the $v^* - \sigma^*$ or SRS - T curves, unlike the case of the 11 at.%Mo alloy in compression. It is not immediately obvious why the SRS - T peak is absent in tension. However, it is possible that its appearance is sensitively dependent on composition, for the nominally 11 at.%Mo crystals used in the tension tests had a lower Mo content than those tested in compression (see table 3 and 4).

2.2. Yield and thermal stresses.

2.2.1 Compression.

The experimental results for the alloys show clearly that the temperature dependence of the yield and flow stresses are the same for any particular alloy. It can be seen also that the absolute value of the thermal stress, determined from temperature change tests, equals the temperature dependent part of the yield stress to within $\pm 10\%$ (table 8). The slightly greater spread in the results at 77 K can be attributed to the necessity in the temperature change tests to warm the specimen back up to room temperature when making the 113 K - 77 K temperature change. This temperature excursion could allow dislocation relaxation and rearrangement and it is therefore difficult to obtain a true measure of the difference in the thermal stress between 113 K and 77 K. The spread is also caused in part by the sensitivity of the 77 K reyield stress to the amount of prestrain in the cases when it was necessary to prestrain specimens at room temperature to suppress twinning, even when only primary glide was found after the 77 K deformation.

This good correlation between the yield and flow stress supports the idea that in these alloys yielding and plastic deformation are controlled by the same mechanism, whether that be screw dislocation glide, or, for some of the alloys at higher temperatures, edge dislocation motion. The idea is further supported by observations from in-situ straining experiments on metals and alloys deforming by primary glide. Yielding at low temperature (i.e. below T_c for the material) is associated with the start of general screw dislocation motion and flow is dominated by their slow propagation (81).

This close similarity between the temperature dependence of the yield and flow stresses is observed for Nb only at temperatures of 170 K or above (8). In marked contrast, at lower temperatures the temperature dependence of the flow stress is greater than that of the yield stress, e.g. at 113 K the yield and thermal stresses are 13.7 and 17 kg/mm² respectively. As will be discussed later the lack of direct correspondence between the yield and thermal stresses (determined from temperature change tests) can be attributed to a difference in the dislocation glide mechanism for specimens at low temperatures depending upon their prior deformation, if any. If virgin Nb specimens are deformed at 113 K, anomalous slip occurs (fig. 42). However, small amounts of prestrain at a higher temperature before deformation at 113 K will suppress the anomalous slip and only primary glide will be seen, eg. in temperature change tests. Thus the thermal stress determined from temperature change tests, i.e. the temperature dependence of the flow stress, will reflect the large temperature dependence of the stress required to move primary screw dislocations, whilst the yield stress at the same temperature will show the low temperature dependence of the stress to initiate and propagate anomalous slip. Precisely this breakdown in the relationship between the yield and flow stresses is seen in the alloys when they begin to show anomalous slip.

Whilst it is possible to state unequivocally that no solution softening occurs in the yield or thermal stress of the Nb-Ta alloys (fig. 19, 23 and 24), it is not possible to be quite so definite for the Nb-Mo alloys deformed in compression. At its simplest this is because the various curves for the 1 - 5 at.%Mo alloys converge at low temperature and the differences between them become very small, within experimental error. If there is no solution softening at

these low temperatures, 113 K - 77 K, then the effect on the temperature dependent part of the yield stress and the flow stress of the increase in the Mo concentration from 1 to 5 at.% is virtually zero - see especially fig. 25 and 26. This is despite the considerable effect on the yield stress produced by the addition of 1 at.%Mo to Nb (fig. 20). This last point is discussed further in section 5.2 - the effect of alloying on anomalous slip.

To deal with these points in more detail one should look first at the variation of the thermal stress for the 1 - 3 at.%Mo alloys (fig.26). The softening, such as it is, lies within $\pm 5\%$ of the thermal stress itself, i.e. within the $\pm 10\%$ expected variation. As discussed above the 113 K - 77 K temperature change test may produce a misleading value for the difference in the thermal stress between the two temperatures because of the necessity to warm the specimen to room temperature. Effects such as dislocation rearrangements will occur for all the alloys although it is expected that they would be less important in the more concentrated alloys because of the greater long range (athermal) stresses present. An absence of hardening is also to be seen in the yield stress for the 1 - 3 at.%Mo alloys (fig.20). Leaving aside the yield stresses at 77 K, because of their dependence on the room temperature prestrain, it can be seen that the yield stress for the 1 at.%Mo alloy exceeds that of the 2 at.%Mo alloy at 113 K and 145 K and equals that of the 3 at.%Mo alloy at 113 K. The behaviour of the 2 at.%Mo alloy may be explained in part by noting that the 2 at.%Mo crystal 300 had a rather different orientation to the majority of the crystals, with $\chi = + 15^\circ$ as opposed to $+ 3.5^\circ$ for the 1 at.%Mo alloy and 0° for the alloy crystals on the whole. It has been observed previously that Nb and Ta crystals, doped with nitrogen and deformed at low temperatures, which have large positive χ values show less solution hardening of the yield stress than do

centre triangle orientation crystals containing the same amount of solute (8, 20). If the same effect occurs for substitutional alloys then it might be expected that the relative separation between the curves for the 1 and 2 at.%Mo alloys would be reduced, or even possibly reversed, at low temperatures when orientation effects are at their most prominent. (Such a reversal does occur for the Nb-300 at.ppm N₂ alloys, ref.8). At high temperatures when orientation effects are absent the expected dependence on concentration is observed. The behaviour of the 3 at.%Mo alloy at 113 K appears to reflect the virtual absence of hardening in the thermal stress at 113 K for alloys containing 3 at.%Mo or less.

Since the discussion of possible solution softening based on merely the yield and flow stresses is not clear-cut, it is worthwhile considering the behaviour in the light of the thermodynamic considerations of the previous section. It was concluded for the 1 - 5 at.%Mo alloys that above a critical thermal stress the rate controlling step was that of double kink nucleation (M2) on screw dislocations (the critical stress depends on the solute concentration, but it is considerably lower than the stresses at which this possible softening occurs - see fig.102). If a softening mechanism does come into prominence as the temperature falls then one would expect the activation enthalpy to decrease faster with increasing stress than would be the case if the softening mechanism was not present. In consequence the $\Delta H_0 - \tau^*$ plot as evaluated in this work would not show a ΔH_0 independent of stress, but rather a ΔH_0 which would decrease with increasing stress. This assumes that the softening mechanism has a progressively greater effect on the activation barrier the lower the temperature, which seems reasonable given that no softening or reduction in the barrier height is observed at high temperatures, but as the temperature falls the effect slowly becomes apparent. As this type of behaviour is not observed in practice,

it can be concluded that the dependence of the thermal stress on temperature and concentration as seen in fig. 25 and 26 is intrinsic to the Nb-Mo system. It is interesting also to note that at 77 K the activation enthalpies for the 1 - 3 at.%Mo alloys are all identical (fig. 101). Further, fig. 26 shows that the reduction in the hardening of the thermal stress is observed also in the 11 and 16 at.%Mo alloys which, as is discussed below, would not be expected to show any softening if interstitials were present. This further supports the idea that the property is intrinsic to the alloys.

The absence of solution softening in these high purity binary alloys when deformed in compression at or above 77 K supports the view that in group $\bar{V}$ A based solid solutions of centre triangle orientation the presence of a second solute is necessary before softening can occur (1, 10, 11). This is further brought out when the yield stress - temperature curves obtained by Statham (73) for zone refined centre triangle Nb-Mo alloy single crystals are examined. Being only zone refined, the alloys would have contained a larger concentration of interstitial solute than the crystals used in the present work. Statham's 2 and 5 at.%Mo alloys had a larger yield stress at high temperatures than do the equivalent alloys studied here. At 77 K the position is reversed with the UHV annealed alloys having the greater yield stress. The more concentrated alloys do not show this switch-over, as would be expected from the observations of Shields on Ta-Re-300 at.ppm N_2 (14). Once enough Re had been added to eliminate softening further additions produced a ternary alloy which was stronger than the base binary Ta-Re alloy. In Nb-Mo system, approximately 5 at.%Mo is required before solution softening is eliminated at 77 K (1). At high temperatures, both substitutional and interstitial solute always produce hardening irrespective of whether they are present separately or together (224).

2.2.2 Tension.

It is difficult to draw firm conclusions from the yield and thermal stress results of the tensile tests because of the need to prestrain the majority of the specimens to suppress premature necking at low temperatures. The prestrain reduces the yield stresses of the virgin crystals (table 7) presumably by increasing the density of edge and mixed dislocations at the low temperature reyield. However, the effect on the 60 at.%Ta alloy is drastic and produces the impression that it is 'weaker' than the 10 at.%Ta alloy. The Nb-Mo alloy shows no solution softening down to 20 K, the lowest temperature at which any reyield stresses were determined. This is in line with their behaviour in compression at the higher temperatures.

The temperature dependence of the flow stress (fig. 27) also shows no solution softening, although the basis for the 77 K base line is suspect. The similarity of the temperature dependence of the yield and flow stresses again suggests that the yielding and flow are controlled by the same mechanism, namely screw dislocation glide.

2.2.3 Solution hardening models.

The behaviour of the two alloy systems are very different although both are controlled at low temperature by screw dislocation glide. It must be expected, therefore, that different models will be required in order to explain fully their behaviour.

The simplest model which can rationalise the behaviour of the Nb-Ta alloys is that the Ta additions simply raise the height of the screw double kink nucleation barrier by a small amount. The screw dislocation velocity will be slightly decreased but in most other respects the alloy will behave like the base material, Nb (but see the "anomalous slip" section for more discussion on this point). This is seen in

the dependence of the activation volume and enthalpy and ΔH_0 on the thermal stress, and the temperature dependence of the thermal stress itself (fig. 98,100,102 and 24 respectively). This rationale is also consistent with the properties of the alloys' constituents, e.g. virtually zero size difference, similar elastic constants (μ differs by 50% - see table 10) and the same outer electronic configuration (both group V A metals.)

The Nb-Mo system poses a more difficult problem. The solute has a different atomic size, moderately different elastic constants and a different outer electronic structure (a group VI A metal). The thermodynamic analysis has shown that the activation parameters are strongly affected by Mo additions, to the extent that by 11 at.%Mo there is an additional change at low temperature in the mechanism governing the glide of the rate limiting screw dislocations. This behaviour is generally interpreted by means of linear-elastic dislocation models and changes in stacking fault energy with alloying for certain transformation paths (e.g. 41,101). Alternatively, a more sophisticated approach has been to use computer simulation of screw dislocation motion. Alloying affects the interatomic potential and this in turn affects the screw dislocation's motion (37). Although in the last account everything does depend upon the interatomic forces, etc., the current models are based on empirical potentials and dislocation motion at 0 K and thus they cannot predict or explain observations except in the most general terms, especially in the presence of thermal activation. However, the progressive effect of Mo additions on the interatomic potential are clear. The 1 at.%Mo addition produced very little change, as was also the case with Ta additions, but larger quantities of Mo result in the general behaviour becoming more and more unlike that of Nb, cf. the activation volumes and enthalpies.

The zero hardening around 77 K is seen simply as a facet of the way Mo alters the interatomic potential. If the data collected in tension does show that the physical properties are different in tension and compression, then these differences may be explained by noting that the computer simulations have indicated that screw dislocation motion is sensitive to the sense of the stresses normal to the slip plane (48).

Many quantitative theories have been developed to explain the effect of solute on the low temperature deformation of bcc alloys. However, these have tended to treat the solute either merely as a barrier, e.g. to sideways kink motion (147, 148, 160), or alternatively as the source of softening by reducing the double kink nucleation energy, e.g. by means of a torque exerted on the screw dislocation by the solute atom if the elastic constants of the alloys' constituents are different (150, 262, 310, but see also 152). The models show either hardening at all temperatures or softening at low temperatures and hardening at high temperatures. None of the models sheds much light on the complex behaviour of the Nb-Mo alloys, probably because they cannot satisfactorily consider the changes brought about in the core of the screw dislocation by the addition of a solute which significantly alters the interatomic potential.

2.3 Slip morphology and work hardening.

The role of solute and temperature in determining the slip morphology of the alloys can be rationalised, in part, by a consideration of the effect of these factors on the stability of screw dislocation arrays and the ease of cross slip. If the stress required for deformation is divided into its component parts (8) then :-

$$\tau = \tau_{\mu} + \tau_{\mu}(\gamma) + \tau^* + \tau^*(\gamma)$$

where τ_{μ} , $\tau_{\mu}(\gamma)$ are the long range athermal stresses from, for example, solute additions and work hardening respectively, and τ^* , $\tau^*(\gamma)$ are the short range thermal stresses from the basic Peierls mechanism and work hardening respectively. Because the smallest stable distance between two parallel screw dislocations of the same sign is proportional to $1/\tau_{\mu}$ (e.g. 236), groups of screw dislocations are not stable in pure bcc metals (341). As alloying increases τ_{μ} , it allows the screw dislocations to move closer together and form stable arrays, e.g. glide bands. Alloying also affects the capture cross-section of parallel screw dislocations of opposite signs as this is proportional to $1/(\tau - \tau_{\mu}(\gamma))$ - see ref.88 and 236. It is also to be expected that lower temperatures or an increase in the Peierls stress, e.g. from alloying, will reduce the ability of the screw dislocation to cross-slip.

The effect of a small increase in τ_{μ} over that for Nb is, on the above rationale, expected to be relatively slight. The observations on the 4 and 10 at.%Ta and 1 at.%Mo alloys bear this out. At high temperatures, e.g. room temperature, the slip structure and its dependence on strain are very similar to those of Nb (60). Fine slip and easy cross-slip dominate stage I (fig. 47 and 57). Secondary slip bands, parallel to the trace of $(\bar{1}01)$, form towards the end of stage I, and the increase in the work hardening rate to that of stage II is coincident with the (011) secondary slip breaking out of these narrow bands to cover the whole surface (e.g. fig.51 and 59). The increase in the length of stage I for specimens of this orientation from approximately 10% shear in Nb (8) to 14 and 17% shear in the 10 at.%Ta and 1 at.%Mo alloys respectively can be ascribed to the greater dislocation density found in the alloys (e.g.73,83)

causing a greater amount of latent hardening of the secondary slip systems. Indeed it is interesting to note that it is the expected secondary slip system, $(011)[\bar{1}\bar{1}1]$, which is observed, whereas an unexpected system is found for Nb (58). The greater dislocation density will likewise increase the stage I work hardening rate, as observed, because the work hardening in this stage is primarily through elastic interactions between individual dislocations (80).

The lack of any marked coarsening of the slip structure at lower temperatures (fig. 52 and 61) again reflects the small changes produced by the solute. The average step height is increased over that for Nb (Nb slip lines are virtually invisible at low temperatures, e.g. 158 K) but the relatively homogeneous structure indicates that slip bands are not stable and that dislocation motion is relatively uniform throughout the body of the crystal.

To follow the changes wrought in the more concentrated Nb-Mo alloys, it is first necessary to consider in more detail the effects of solute on dislocation behaviour at yield and during deformation.

Although the initial movement of, and multiplication from, the grown-in dislocations and those produced by preyield edge - mixed dislocation motion will be uniform throughout the crystal, there will be a tendency for slip to become localised in the more concentrated alloys. The preyield dislocation distribution, whilst being, on average, uniform, will have local fluctuations and regions or distributions which are more favourable for dislocation multiplication. Once a screw dislocation group has been generated in such a region the internal stresses it generates (236) will further aid the motion of dislocations in that region (340). As the stress levels rise, either because of increasing amounts of solute or lower temperatures, the importance of these locally favoured sites increases, for once they

start to operate they will prejudice the initiation of other sites by removing the necessity for them to do so : strain is being generated and only when work hardening has increased the stress to a sufficient level will it be necessary for further slip band formation to occur. (The situation is somewhat analogous to nucleation in phase transformations. At high temperatures few nuclei are formed, but once the activation barrier is overcome, they grow rapidly into large particles. At low temperatures many nuclei are formed, but each can only grow into a small particle). Thus higher stresses will be accompanied at 'yield' by coarser slip lines. In addition to these considerations, increasing amounts of solute will stabilise larger screw dislocation groups so one could expect, on a simplistic interpretation, coarser slip bands at the start of deformation as the solute content increases. Cross-slip will also be progressively impeded, both as the amount of solute increases or the temperature decreases, so slip bands could be expected to be less wavy. This inhibition of cross-slip can be rationalised by noting that although the shear stress resolved onto the cross-slip plane relative to that on the primary system is unaffected by the level of the stress, the absolute difference increases with increasing stress, thus biasing slip away from the cross-slip plane.

Once the initial slip bands have been formed they may either grow or, alternatively, fresh slip bands may nucleate. In the former case the slip bands may grow laterally, i.e. dislocation activity occurs mainly at the edge of the band, or they can carry the increasing strain without expansion, i.e. the work hardening in the band is not sufficient to necessitate dislocation generation at its edges. If fresh bands are nucleated they will have to form and grow between the already existing bands and against the internal stresses generated by these bands (180). Nevertheless, for some alloys the surface can quickly be covered with

slip bands (e.g. 73) although the strain per band may well remain constant during deformation at a given temperature and composition (202, 204). The balance between these two processes, growth or nucleation, will depend on the temperature and solute concentration, and the strain. The ease of cross-slip must also be taken into consideration for this will play a role in determining how easy it will be for a band to expand. Low temperatures will favour less wavy slip and slip bands, and a simplistic extrapolation of this would suggest that slip would therefore be confined more strongly to its original slip bands.

Thus in an alloy with a small to moderate τ_p , e.g. 2 - 5 at.%Mo alloys, only small dislocation groups would be stable. Although at high temperatures individual slip bands nucleate at yield, they are close together and fresh bands easily nucleate between them and fill up the surface quickly with increasing strain. However, some band structure is retained (fig. 67 and 70). At lower temperatures the nucleation of fresh bands is more difficult. Thus, although the inter-band spacing is probably greater at yield than at the high temperatures (cf. 202), the surface does not fill up so fast with freshly nucleated bands (e.g. fig. 71). The larger stress will also restrict cross-slip, so producing less wavy bands (e.g. fig. 68, 69 and 71).

The 11 at.%Mo alloy shows the above trend continuing, viz. coarser slip appearing at the start of deformation at higher temperatures than previously (fig. 72, 73). However, the nucleation of fresh bands is still easy enough at 290 K and 175 K for the individual slip bands to disappear with increasing strain. Only at 77 K do the dynamics of yielding and the difficulty of fresh nucleation, along with the difficulty of cross-slip, produce a strong slip band structure (fig. 75) and enable it to retain its identity with increasing strain.

The most extreme effects are seen in the 16 at.%Mo alloy. The difficulty of band nucleation, both at yield and during flow, are noticeable even at room temperature (fig. 77 - 21% shear strain). The effect of lower temperatures is to virtually eliminate fresh band nucleation, which did occur at room temperature, and through limiting cross-slip, to promote extremely straight slip bands, e.g. fig. 82 - 14.5% shear strain at 8 K.

The work hardening rate increased with the amount of solute, e.g. the linear hardening rate increased from 13 to 17 to 20 to 23 kg/mm² as the Mo content rose from 1 to 16 at.% (the values are only approximate both because they depend slightly on the temperature and because of end effects, etc. on the compression specimens). This may be rationalised by noting that alloying increases the density of dislocations, especially primary screw dislocations, and decreases the volume of the crystal in which they move (the slip bands). Thus, whether the dislocations move co-operatively or singly, the interactions between them will be more frequent, thus increasing the hardening rate.

Although the effect of decreasing temperature or increasing amounts of solute, especially with a strong hardener such as Mo, appears to be the same, viz. coarser slip, it seems that a distinction should be made as to how the effect is brought about. The temperature affects band nucleation and thus the dynamics of yielding, whereas solute affects the stability and, hence, the size, of the screw dislocation groups. Both affect the ease of cross-slip. At one extreme of coarse slip, high temperatures and large amounts of solute, it is the stability of large screw dislocation groups which produces coarse slip. Lowering the temperature merely imposes an extra constraint that fewer bands will be nucleated initially. At the other extreme, low temperatures and small amounts of solute, the slip structure is controlled by the difficulty

of nucleating slip bands. As the temperature rises this constraint is relaxed and the individual bands form closer together in the first place, and easy band nucleation and cross-slip leads to the surface being rapidly covered with slip. Because of the means by which the temperature and solute control the slip structure, it seems likely that any effort to 'describe' the structure solely by reference to the thermal stress (180) would be unsuccessful. Indeed, as has been pointed out before, the 1 at.%Mo alloy shows anomalous slip at a thermal stress where the 16 at.%Mo alloy is showing coarse primary slip bands.

All the above discussion is based on the results obtained from the compression tests. All the prestrained tension specimens showed fine primary slip except in the region of necks when the slip was often very coarse. On the rationale stated above, once the relatively homogeneous structure of the high temperature prestrain has been formed and slip is fairly evenly distributed throughout the crystal then localisation of slip is likely to be more difficult as the important position held by the restricted number of favourable nucleation sites will have been diminished because of the large number of such sites now present. Hence the slip structure will remain fine at low strains.

3. Low temperature instabilities.

3.1 Niobium.

The appearance of instabilities in the low temperature deformation of Nb, and other metals, is not a new phenomenon (343, 345). The appearance of large load drops, up to approximately a quarter of the flow stress in size, and extremely localised shear deformation have been attributed to heat generated by dislocation motion locally raising the temperature of the specimen. This is made possible by the low heat

capacity and thermal conductivity found at temperatures approaching absolute zero. The load drops and the shear deformation have been taken as fairly general characteristics of this form of instability. However, the observations in this work appear to show some novel features of the phenomenon.

Specimens deformed at or just below 20 K and subject to a 10x increase in strain rate (1.2×10^{-4} to $1.2 \times 10^{-3} \text{ s}^{-1}$) showed overshoots after the rate increase (see fig. 39). At lower temperatures the overshoots developed into oscillatory flow. At 8.25 K the 10x rate increase was followed by a large load drop, approximately one third of the flow stress in size, although immediately after the load drop the specimen deformed in an oscillatory manner at a stress only just below that before the rate increase. Between 8.25 K and 10.5 K a 10x decrease in the strain rate to the base rate (1.2×10^{-3} to $1.2 \times 10^{-4} \text{ s}^{-1}$) produced an initial decrease in the stress, but this was rapidly followed by a rise in the stress such that the steady state flow stress was larger than at the higher strain rate. Deformation at 5 K, even at the base strain rate ($1.2 \times 10^{-4} \text{ s}^{-1}$), after deformation at higher temperatures, proceeded by a series of large load drops. However, in none of the cases was any inhomogeneous deformation whatsoever found. On the contrary, very fine homogeneous anomalous slip was seen over the entire specimen in each and every case. The above observations suggest that the instabilities were not produced by a localised temperature rise in one part of the specimen, but rather were caused by a temperature rise for the specimen as a whole. For this to be the case the temperature profile around a group of moving dislocations must overlap with other sets of dislocations, i.e. a homogeneous temperature distribution in the crystal. The characteristic width of this temperature profile around an active slip band is approximately $(\bar{k}R/2h)^{\frac{1}{2}}$ - see table 11

for the meanings of the symbols, also ref. 342. This gives a characteristic width of approximately 2500 μm at 4.2 K. The separation of anomalous slip bands is about 1 - 5 μm . In addition, the time required for any heat generated by one group of dislocations to be dissipated to adjacent groups must be much smaller than the time observed for the load drops to occur. This too can be shown to be the case here (342).

Deformation under these conditions has been analysed by Kubin (342, 344). The phenomenon may be described by three parameters - α , v_r and ξ , dimensionless terms representing the activation volume, a thermomechanical factor and the strain rate respectively (see table 11 and ref. 342 for a full explanation of the terms). Three types of behaviour can be expected (see fig. 107) :-

(A) Stable: Thermal equilibrium is maintained between the specimen and the temperature bath. The reduced parameters lie outside the lower dashed line.

(B) Unstable: Heat generated by dislocation motion produces a temperature rise which allows easier dislocation motion. Deformation is unstable and load drops are seen. The reduced parameters lie within the area bounded by the stability boundary.

(C) Stable, but at a different temperature: The reduced parameters lie between the stability curve and the lower dashed line. On yielding, the temperature of the specimen rises and the stress falls. However, the reduction in stress reduces the heat output, and it is possible to establish an equilibrium where the temperature of the specimen is raised by a small but constant amount over that of the temperature bath.

As the boundary between regions (C) and (B) is approached, the flow after yielding becomes more oscillatory as it is more difficult for the specimen to stabilise at its new, higher, temperature. The oscillations merely reflect the coupling and feed back between the heat generating mechanism and the temperature.

Although Kubin's analysis is for yielding and subsequent deformation at a fixed applied strain rate, the analysis should be applicable to the case when the strain rate is increased during deformation, for, like yielding, the extra dislocation activity will increase the previous steady state heat flux. As the nominal deformation temperature falls, the pulse of extra heat generated during the rate increase will become more important, as is seen clearly in fig. 39 for temperatures at or below 20 K. At 20 K there was a very slight overshoot after the rate increase, i.e. the increase in the strain rate has raised the equilibrium temperature of the specimen. By 10.5 K one is fairly near the instability boundary and the stable temperature rise produced after the oscillations have settled out is sufficient to allow easier deformation at the faster strain rate. Thus when the strain rate is reduced to the base rate ($1.2 \times 10^{-4} \text{ s}^{-1}$), the instantaneous effect is for the load to fall. However, heat generation decreases and the temperature rapidly falls back to the nominal bath temperature, or closer to it. The deformation load then needs to rise to a greater value than was required at the higher strain rate. From the strain rate sensitivity and the slope of the stress - temperature plot at 10.5 K it can be seen that the temperature rise produced was approximately 0.6 K. At 8.25 K it would seem that the instability boundary has been crossed for a large load drop occurs after the rate increase. However, the flow was oscillatory after the load drop and a decrease in the strain rate produced a load rise as at 10.5 K. At 5 K, however,

flow was unstable, load drops occurring sequentially.

To see how well the numerical analysis of Kubin models the observed behaviour, the dimensionless parameters were calculated (see table 11 and fig. 107 where the calculated points for the 10x base rate, $1.2 \times 10^{-3} \text{ s}^{-1}$, have been included). Given the uncertainty in such parameters as the surface heat exchange coefficient (see comments in Kubin (342)), good general agreement is found. At 30 K the model predicts stable deformation, as in fact occurs. From the instability criterion (Kubin (342) - eqn.12) :-

$$\alpha \xi \leq \frac{\left(\frac{\beta - 1}{\beta} \right) v_r \xi - 1}{(1 + v_r \xi / \beta)^3}$$

instability would be expected to appear between 8 K and 10 K, very much in line with the observations. At intermediate temperatures, oscillations are expected in conjunction with a stable rise in the specimen's temperature, which was again observed.

3.2 Effect of alloying.

Alloying will affect at least three important parameters which determine the onset and nature of the stress - temperature instabilities. The first effect is that the thermal conductivity will be reduced. Secondly, the heat capacity will be altered. Whilst Ta has a very similar heat capacity to Nb this is not so for Mo where the value is rather smaller. The heat capacity of the alloys would be expected to follow a 'law of mixtures' so that the Nb-Mo alloys would have a lower heat capacity than Nb. The third factor is that both Ta and Mo, but especially Mo, promote coarser slip than found in Nb at

these low temperatures. They also promote the tendency for necking to occur early in the deformation. This may well affect the tendency for localised heating and shear deformation, especially given the reduction in the heat capacity and thermal conductivity. The expected consequence of the changes outlined above will be that the instabilities and oscillatory flow should appear at higher temperatures than was found in Nb, especially in the more concentrated Nb-Mo alloy.

To illustrate that this is what does happen consider the flow behaviour after changes in the strain rate at 22 K for the Nb-Mo alloys deformed in tension (fig. 41). The same trends appear in the Nb-Ta alloys (fig. 40). The 1 at.%Mo alloy was just stable, with some oscillations if the specimen was necking. The 3 at.%Mo alloy showed considerable oscillations and an apparent negative strain rate sensitivity because of the temperature changes produced in the specimen. The effect is even more apparent in the 5 at.%Mo alloy. The 10 at.%Mo alloy appeared to behave differently (but see the 30 K behaviour). This may simply be a reflection of whether or not the slip is already localised by necking. When the load drops did occur in these alloys they were usually associated with shear deformation. Only occasionally were they connected with necking on its own. When necking cannot occur, as in compression, the trend is a little clearer (fig. 35 and 36). The 11 at.%Mo alloy showed a small overshoot at 20.4 K whereas the 16 at.%Mo alloy did so at 30 K, whilst showing complex oscillations at 20.4 K after the increase in strain rate.

Whilst it is relatively easy to identify the temperature at which oscillations appear, although it does depend on the amount of inhomogeneous deformation, it is not so easy to say at what nominal deformation temperature the effect of specimen temperature rises ceases to play a part. This difficulty is caused by the presence of overshoots after an increase in

the strain rate at temperatures at which no thermal instabilities are thought to occur. In Nb the two regions are separated by a temperature gap of 30 K - 40 K. In the alloys overshoots are found at all temperatures at or below 77 K. However, on the basis of the compression behaviour, the 16 at.%Mo alloy appears to show no thermal instabilities at 42 K so overshoots at or above 30 K - 40 K in the 1 - 11 at.%Mo alloys would be expected not to have a thermal origin, unless the specimen was necking. Approximately the same conclusions would be expected to hold for the Nb-Ta alloys given the temperatures at which the oscillations appear.

More indirect evidence as to whether the division between the two 'types' of overshoots occurs between 30 K - 40 K can be gleaned from looking at the flow behaviour after the $2\frac{1}{2}$ x rate changes ($1.2 \times 10^{-4} - 4.8 \times 10^{-5} \text{ s}^{-1}$) for the Nb-Ta alloys. For both the alloys deformed at or above 40 K the $2\frac{1}{2}$ x rate changes mirrored the 10 x rate changes as regards the presence of overshoots. At lower temperatures their behaviours diverged for the $2\frac{1}{2}$ x rate changes cease to show any overshoots, whilst the 10 x rate changes continue to do so. This can easily be explained by recalling the various strain rates involved and the relative size of the rate changes. This behaviour suggests that the division between the two mechanisms producing overshoots lies at about 30 K - 40 K. This line of reasoning is harder to support in the Nb-Mo alloys but the slight evidence that there is certainly does not contradict this rationale.

4. Strain rate change effects (fig.35,36,39 - 41).

There are very few reported instances of overshoots appearing after a strain rate change for bcc materials (see 8) although they have been extensively studied in fcc metals (e.g. 346,347). Leaving aside the

behaviour of the alloys below 30 K - 40 K and that of Nb below 20 K (see last section for a discussion of these temperature regimes), any explanation must take account of several factors :-

(a) The overshoots occur irrespective of whether deformation is by primary glide or anomalous slip.

(b) They occur over a wide temperature range, possibly 77 K - 40 K, although for the 16 at.%Mo alloy they occur from 150 K to 40 K.

(c) The character of the overshoots is not affected by the strain, except in the case of Nb and the 16 at.%Mo alloy (fig. 39 and 35 respectively).

(d) The overshoots are reversible, i.e. the up and down changes are mirror images of each other.

Whilst it is possible to suggest formal reasons which could account for the overshoots, e.g. changes in the mobile dislocation density or the work hardening rate, inertial effects or a dependence of velocity on stress which is not single valued, the lack of any specific model means that it is virtually impossible to develop anything more than a speculative argument. None of the work done here discriminates between the possible relative contribution of, say, the first two of the above suggestions. For example, it is tempting to ascribe the effect to a change in the mobile dislocation density. However, this raises the problem that since in-situ deformation work has suggested that all dislocations are mobile at low temperatures (8), overshoots imply an increase or decrease in the dislocation density. Whilst this may not be a conceptual difficulty in the case of small overshoots, e.g. 1 at.%Mo alloy at 77 K, fig.41, when the overshoots are such that no steady state rise in the flow stress is observed for a 10 x increase in strain rate, e.g. 16 at.%Mo alloy at 77 K, fig.41, the implication is that the dislocation density must increase or decrease

by a factor of 10 at each strain rate change, and must do so rapidly, For an increase in strain rate the overshoot for the 16 at.%Mo alloy lasts for less than one second. On the other hand, a change in the work hardening rate could be used to explain the small overshoots but such a model would find it difficult to explain the apparently zero steady state strain rate sensitivity and very short-lived overshoots of the 16 at.%Mo alloy at 77 K.

The 11 and 16 at.%Mo alloys show two peaks in the strain rate sensitivity - temperature plot. This has been interpreted as showing the presence of two different rate determining mechanisms operating over different temperature ranges. The most extreme forms of the overshoots have been seen for these alloys in the vicinity of the minimum in the strain rate sensitivity - temperature plot, i.e. where the transition between the two rate controlling mechanisms occurs. It is tempting to suggest that the rate changes in this vicinity take the specimen to and fro between the two mechanisms, thus producing fast overshoots as the rate limiting step switches between them. However, overshoots are also found in these alloys well away from the supposed transition temperature. This suggests that in these cases other factors, e.g. mobile dislocation density etc., must be playing a part in the overshoot. It is interesting to note that Statham (101) observed a zero strain rate sensitivity for similar composition Nb-Mo alloys at the same temperature as the zero steady state strain rate sensitivity in this work. However, he did not observe any overshoots at that temperature or any other. This may be attributed to the equipment that he was using, which was a much softer machine with a far slower load recording system than has been used here. In addition, the cross head speed was altered by changing the speed of the motor, whereas in this work the rate changes have been made through magnetic clutches.

The specific behaviour of Nb, and 10 at.%Ta alloy deformed at 77 K and 60 K, are rather different to the rest of the alloys even though they too show overshoots. All showed anomalous slip without any complicating factors introduced by prestraining. They will be discussed in more detail in the next section, although the comments made in this section about the overshoots will apply to that aspect of their behaviour, despite the other differences which are present.

5. Materials deforming by anomalous slip.

5.1 Niobium.

Although anomalous slip was first observed some fifteen years ago, very little work has been reported on its behaviour in Nb below 77 K (71, 101, 348). The present work with Nb, concentrating as it does on precisely this temperature range, sheds some new light on the subject. Because the exact nature of anomalous slip is not totally clear it is best to start by considering what deductions can be made from the new observations without any reference to a particular dislocation model.

The most striking observation, and arguably the most important, is the total absence of any temperature dependence of the yield and flow stresses between 77 K and 50 K, coupled with the reappearance of a temperature dependence at and below 40 K (fig. 21 and 27). Similar observations have been made (101, 348) but in both cases it has been interpreted as a region of low temperature dependence rather than an athermal region. In the work of Statham (101) it should be noted that only a limited number of yield stresses were determined, whilst in the work of Nagakawa (348), flat specimens with a tapered central region were used, producing a far larger scatter than was observed in the present work. Tests were also done at few temperatures. The presence

of this athermal region bars any thermodynamic analysis based on the type of model outlined in the appendix. It is implicit in such a model that as the temperature falls, the amount of work done by the applied stress must increase, as must the stress itself. The presence of an athermal region implies that a non-thermally activated mechanism is controlling the stress levels. Only at or below 40 K does this 'conventional' mechanism of thermal activation reappear, making possible a thermodynamic analysis. It is also to be noted that the temperature dependence of the yield and flow stresses are the same, implying that the same mechanism controls yielding and flow in the crystals. This is contrary to the behaviour in compression at or above 59 K (8), but it is in accord with other low temperature tensile deformation (348).

This interpretation has important consequences with regards to an understanding of the strain rate sensitivity. If deformation is controlled by an athermal mechanism, any strain rate sensitivity observed cannot be related in any simple way to a thermally activated process of the kind envisaged in the appendix. This is reflected in the unusual behaviour observed between 77 K and 50 K when the strain rate was altered ; e.g. zero sensitivity at low strains at 77 K, a dependence on strain, poor definition, irregular flow and overshoots. The strong dependence on strain found at 77 K in tension is paralleled by observations made in compression (ref. 8, fig. 3b). Although in that work no strain rate changes were made below 2% axial strain (i.e. when the sensitivity in tension was zero) the strain rate sensitivity increased rapidly at larger strains. This type of behaviour was not observed at higher temperatures in compression, where the sensitivity was reasonably independent of the strain (8), nor was it found in tension at or below 40 K. It is only at 40 K, when the yield and thermal stresses regain their temperature dependence,

that the change in strain rate produces normal transients (cf. fig.28a), free from overshoots and irregular flow. The difference between the behaviour at 50 K and 40 K is most marked (fig. 39). The simplest conclusion is that thermally activated dislocation glide only reappears as rate controlling at 40 K. The activation parameters calculated from the above model for temperatures between 77 K and 50 K are not meaningful as the model is not valid.

The appearance of load drops and irregular flow is also restricted to the athermal region, strongly suggesting that the two features are related. For load drops to appear, the plastic strain rate must be capable of increasing very rapidly. It has been estimated that the average strain rate during a load drop is 1 s^{-1} , whilst the applied strain rate is approximately 10^{-4} s^{-1} (59). This implies that either the mobile dislocation density or the velocity of the dislocations, or both, can increase very rapidly. If flow is athermal, i.e. flow is not controlled by thermal activation over short range barriers, then once the stress is sufficient to overcome the largest athermal barrier, there is effectively no obstacle to the deformation mechanism. Thus dislocations could accelerate very rapidly to a large velocity. Alternatively, if the limiting athermal barrier is the generation rather than purely the movement of dislocations then once the stress is sufficient to overcome the generation barrier both the mobile dislocation density and the velocity would increase very rapidly. In such a case where the yield stress is controlled by the stress required to operate the dislocation generating mechanism, then once the mechanism operates the dislocations produced will be 'overstressed', i.e. subject to a stress in excess of that required to move them at a velocity sufficient to match the applied strain rate. Even if the motion of the dislocations thus generated involves a thermally activated step, and hence a 'wait'

at each activation site, the excess stress on the dislocations is sufficient to reduce the activation barrier to such an extent that the waiting time at each activation site is reduced to the point where the dislocation's velocity is sufficient not to make it the rate limiting, and hence stress determining, process in the deformation. However, as the temperature is lowered the velocity of the thermally activated dislocations will decrease, for although the extra work required to be done by the stress to operate the athermal generation mechanism is the same as at the higher temperatures, the available thermal energy has been reduced and the waiting time at each activation site before the dislocation can overcome the barrier will increase. Thus, even though the same increase in the mobile dislocation density may occur at yield, the velocity of the dislocations will not increase to such an extent, hence the plastic strain rate cannot reach such a large value and the load drops will become less marked, as was observed. At some critical temperature, between 50 K and 40 K, there will be a switch as to which mechanism requires the greater stress to operate so that the applied strain rate can be matched. As the temperature is further decreased thermally activated dislocation motion becomes rate controlling, the yield and thermal stresses regain their temperature dependence and a normal strain rate sensitivity will be in evidence. A similar argument can be used to explain why no load drops are observed when anomalous slip occurs at temperatures above 77 K, i.e. when the yield stress is again temperature dependent and the stress is controlled by some rate limiting thermally activated step.

Several other points can be made to support the idea that dislocation generation, rather than motion per se, controls the yield stress. Deformation of virgin crystals between 77 K and 60 K was almost always accompanied by preyield load drops, sometimes occurring

at a stress some 30% below the macroscopic yield stress. If the preceding argument holds, then the presence of load drops implies a burst of dislocation activity at a stress above that necessary for unhindered dislocation motion. The movement of edge - mixed dislocation segments at stresses well below those necessary to move screw dislocations has been well established by in-situ deformation studies. However, such pre-macroyield dislocation activity is a gradual affair, occurring over a range of stresses (e.g. 272,284). In a large scale test such as a tensile test it does not reveal itself by sudden burst of activity ; rather it occurs as a gradual microstrain. If the above argument is valid, then the presence of these load drops implies that the stress at which they occur is already greater than that necessary to move the dislocations. What limits the deformation is the generation of dislocations, not their motion. As the temperature decreases towards the transition point, 40 K - 50 K, the stress necessary to produce dislocation motion becomes increasingly similar to that necessary to generate the dislocations. Hence the stress range below the macroyield over which it would be possible to have rapid bursts of dislocation activity, would be reduced. Below the transition temperature no load drops would be expected. This is as observed. The observations of Garrett-Reed (59) that a light prestrain or surface abrasion suppresses the load drops is also in accord with the above rationale, for an increase in the initial number of sources will reduce the occurrence of explosive bursts of dislocation activity, i.e. the load drops.

The disappearance of the load drops with increasing strain can be attributed to the effect of work hardening shortening the dislocation free path. This will restrict the amount of strain generated in each load drop and hence the size of the load drop. Whether the load drops actually cease at large strains or whether they become too small for the equipment to detect is a debateable point. An alternative interpretation is that if the number of sources increases with strain,

then since the velocity of the dislocations will now be less after their generation than was the case before, the load drops should become smaller as the strain increases. It is also interesting to note that the work hardening rate, and hence mechanism, is independent of temperature over the whole range 77 K to 8 K.

A similar insensitivity to temperature is to be found in the slip line structures of tensile specimens orientated in the centre of the stereographic triangle (fig. 84 and 85). The fine, homogeneous anomalous slip, together with the extremely limited amount of slip on the (101) and (011) planes at all temperatures is in marked contrast to the behaviour in compression at 77 K (fig. 43-46) with its coarse slip bands and large scale activity on all four {110} planes. Similar differences between tension and compression have been observed in Vanadium (23, 196). Notwithstanding this, the yield stress at 77 K is the same in tension and compression. Even in compression, however, coarse lines and multiple slip activity are not necessary, e.g. at 113 K (fig. 42). The effective Burger's vector of the anomalous slip, approximately 6° above [011] and independent of temperature, is in good accord with other work (e.g. 289). However, a surprising discrepancy exists between the present work and that of Garrett-Reed (289) in respect of the Burger's vector operating on the (101) plane. The observations made by Garrett-Reed were that an apparent minimum in the contrast of the slip lines was found in a position corresponding to an effective Burger's vector of $[\bar{1}01]$. This tied in nicely with his TEM studies which showed that screw dislocation networks were formed on all four {110} planes and thus one might expect all four {110} planes to have equivalent $\langle 110 \rangle$ type Burger's vectors. The present work has unambiguously determined the dead band of the (101) slip bands to be due to the slip of dislocations with the $[\bar{1}11]$ Burger's vector at all temperatures, i.e. normal conjugate

dislocation glide. This raises the difficulty of explaining why the activity on the $(101)[\bar{1}11]$ system gives rise to extremely coarse crystallographic slip bands whilst other $\{110\} \langle 111 \rangle$ systems have fine wavy traces at these low temperatures. It also raises the problem of why no primary traces are found. It must be concluded that the mechanisms governing anomalous slip are only weakly dependent, if at all, on the presence of slip activity on any other slip systems taking place over long distances.

Any attempt to explain anomalous slip must obviously take account of its special features, e.g. co-operative dislocation motion, majority of strain being carried by two low stress systems, etc. Whilst some of the early models could 'explain' certain aspects of the deformation, they fell short in that other predictions of the theories ran counter to observations (see literature review for a full description : also ref. 294, 297, 298). More recent work has concentrated on the observed dislocation structures and in particular the interaction between primary $[111]$ and conjugate $[\bar{1}11]$ screw dislocations and the extensive twist walls that are rapidly formed during deformation (289). Much attention has centred on the torque which exists at the crossing point of two $\langle 111 \rangle$ screw dislocations (301, 355). This acts in the common plane of the two dislocations, $(0\bar{1}1)$ in this case. Because the torque acts so as to turn the screw dislocations out of their pure screw orientation over a short distance near the junction, various models have used the torque as a way of reducing the stress required for slip. This might be by aiding double kink nucleation at the crossing point (348) or in the vicinity of a $\langle 100 \rangle$ junction dislocation (301 - from in-situ work on Mo). An alternative interpretation arrived at after extensive TEM work on anomalous slip in Nb is that the torque can bend the screw dislocation so that it acquires a degree of edge-mixed dislocation character (289). Since this can move at a much smaller stress than the screw

dislocation, under suitable conditions this edge-mixed dislocations segments will be able to glide at a lower axial stress than a primary screw dislocation. As with all the above models based on the effect of the torque, slip is confined to the $(0\bar{1}1)$ plane as the dislocations must move together in their common plane. The last variation has the attraction that it is immediately obvious that any factor which alters the v_e/v_s velocity ratio will affect the onset of anomalous slip, e.g. solute and temperature.

Several difficulties have to be considered before the conclusions arrived at from the mechanical tests can be properly related to the interpretations placed on dislocation structures. The first point is whether there is any significant difference between tension and compression, e.g. as seen in the fine and coarse slip at 77 K (fig. 84 and 43). As it happens, the only temperature at which a direct comparison can be made is at 77 K. The yield stresses are the same and from the form of the temperature dependence of the yield stress it seems likely even in compression that the yield stress is almost independent of temperature by 77 K (8). The difference between the temperature dependence of the flow stresses in tension and compression below 77 K has already been commented on. It should be noted that no temperature change tests were made in compression over solely the range 77 K - 63 K. The lowest start temperature was 112 K. Since the yield stress is still temperature dependent at 112 K and the slip is very fine (fig. 42) it is just possible that different dislocation structures are formed which produce a temperature dependence in the flow stress below 77 K (see also below).

The difference in the scale of the visible slip traces between tension and compression must reflect a change in the dislocation distribution. It is implicit in the work of Garrett-Reed (289) that

there is no fundamental difference, except that of band separation, between the dislocation structures in those specimens which exhibited fine or coarse anomalous slip in tension at 77 K. Whether the same structures are to be found at temperatures below 77 K is a debateable point. At higher temperatures, e.g. 158 K, different dislocation structures were found, e.g. no twist walls and a more homogeneous structure (289). However, a degree of caution must be exercised since only a few specimens were examined. This notwithstanding, it seems unlikely that there are several distinct mechanisms which can produce anomalous slip, each operating over a different temperature range. Any change in slip morphology or general dislocation structures should therefore be taken as reflecting a change in factors which are secondary to the basic mechanism which generates slip on the $(0\bar{1}1)$ plane. Therefore in the following discussion it will be taken that the important rate and stress controlling factors are unaffected by changes in the form of testing, i.e. tension or compression.

The second difficulty lies in deciding whether the proposed dislocation mechanisms are sufficient to produce continuous anomalous slip or whether they are merely a necessary step in the process. Whilst they may allow the dislocations to glide on the $(0\bar{1}1)$ plane, little attention has been paid to explaining how the twist walls appear or how they are sustained during deformation. This may best be approached by introducing some of the conclusions reached from the mechanical tests.

A pertinent question to ask is whether any of the suggested mechanisms are athermal. The two variants which use the couple between the crossed screw dislocations to reduce the double kink nucleation energy are clearly not going to be athermal for they still envisaged a rate limiting thermally activated step in the movement of the dislocations,

be it double kink nucleation itself or sideways kink propagation (348). The third proposal, in which the torque bends part of the screw dislocation into edge-mixed orientation, seems at first sight to have the same property for although the motion of edge dislocations is considerably easier than for screw dislocations and has a lower temperature dependence, it is still a thermally activated process (e.g. 272, 273). Thus any criterion for yielding based on edge-mixed dislocation motion as being the rate and stress determining step will inevitably predict a temperature dependent yield stress. In addition, the resistance of the screw dislocation to being bent by the torque at the junction will increase as the temperature falls, i.e. the screw dislocation will become stiffer because the decreasing amount of thermal energy will confine it more firmly to the bottom of the Peierls potential.

However, it is possible to interpret this model slightly differently. As stated (289), a necessary precursor to large scale motion of the edge-mixed dislocation segment is that a sufficient length of the $\langle 111 \rangle$ screw dislocation has been bent out of the screw orientation. This is envisaged as occurring by a reduction in the spacing of the dislocations in the $(0\bar{1}1)$ plane, which can occur in one of two ways. Either the dislocations in the network glide on the $(0\bar{1}1)$ plane or more screw dislocations are actually added to the network, e.g. by some multiplication mechanism. The yield stress would then be associated with a mechanism which creates favourable conditions for the network as a whole to move. One of the conclusions drawn from the mechanical tests was that the limiting factor in the athermal region could be the start of dislocation generation, or the initial formation of a generating mechanism, rather than the motion of dislocations per se. If this was to be the case then in the athermal region the stress required

to yield the specimen would be greater than that necessary to move edge dislocations. The consequence of this 'overstressing' of the edge-mixed dislocations would be very much as anticipated in the preceding discussion, i.e. load drops. As this envisages a sequence of consecutive steps in the initiation of anomalous slip, the step requiring the largest stress will control the yield stress. The reappearance of a temperature dependence in the yield stress between 40 K - 50 K can therefore be ascribed to the appearance of the thermally activated edge-mixed dislocation as the rate and stress controlling factor. The athermal generation mechanism will still operate but the limiting step will be different.

It seems therefore that the proposed mechanisms as they stand cannot account for the athermal region between 50 K and 77 K. If a generating mechanism is controlling deformation in this region, it is, unfortunately not at all obvious what this step actually entails.

5.2 The effect of alloying.

The appearance of anomalous slip in the 1 at.%Mo and 4 and 10 at.%Ta alloys, represents the first observation of this mode of deformation in any substitutional solid solution based on a group $\bar{V}$ metal. There is only one other report of anomalous slip in any substitutional alloy and that is in two Mo based alloys (194). This is in itself a 'problem' for Mo is not thought to exhibit anomalous slip as found in group $\bar{V}$ metals (199, 294). Notwithstanding the absence of previous observations, the depression of the temperature at which anomalous slip first appears with increasing solute concentration is in accord with studies made on interstitial solid solutions, e.g. Nb or V doped with O or N (e.g. 8, 23, 71). Although these studies followed the effect of interstitials by varying their concentration whilst studying deformation at a fixed

temperature, usually 77 K, it is clear that the anomalous slip is displaced to lower temperatures and larger stresses by increasing amounts of solute.

As with the last section, the effect of solute on anomalous slip, and deformation generally, will be discussed first of all from the standpoint of external properties, e.g. yield stresses, etc., before moving on to see whether any implications can be drawn as to the operative dislocation mechanism.

The slip line structure shows that the fundamental aspects of anomalous slip are unaltered, eg. effective Burger's vector close to $[011]$, crystallographic $(0\bar{1}1)$ slip, etc. However, the anomalous slip lines are much coarser, more akin to Nb deformed in compression. A similar effect has been seen with O_2 doped V or Nb (8, 23, 71) or electron irradiated Nb (348). It is also noticeable that far more activity is present on other $\{110\}$ systems, e.g. fig. 86 and 87, in particular on the primary system, which is rarely observed in Nb except in compression - fig. 43. This must not be confused with primary slip from the room temperature prestrain (the 10 at.%Ta alloy shown in fig. 86 and 87 was not prestrained ; the other pictures of anomalous slip in alloys are from prestrained specimens, with the exception of fig. 64, 88, 90 and 91).

One of the most striking effects of solute addition is the very large increase in the yield stress produced by initial solute additions at temperatures where anomalous slip is present in Nb, i.e. below approximately 150 K (fig. 19 and 20). The important change brought about by even the smallest amounts of Mo or Ta used in this work was the suppression of anomalous slip at these temperatures. Although some anomalous slip was present at 113 K with the 4 at.%Ta and 1 at.%Mo alloys, primary slip was always dominant, e.g. fig. 64.

As has already been discussed, the yield and flow stresses of the alloys have been taken to be controlled by the glide of primary screw dislocations, cf. in-situ deformation studies, similar temperature dependence of flow and yield stresses, etc. This is not so for Nb and is reflected in the difference between the temperature dependence of the yield and flow stresses. If, as has been assumed, the temperature dependence of the flow stress represents the temperature dependence of the stress necessary for screw dislocation glide, then the lower temperature dependence of the yield stress below approximately 150 K, and its eventual athermal behaviour, characterises a slip mechanism which operates at a far lower stress. Once that low stress mechanism is suppressed and screw dislocation glide is reinstated as rate and stress controlling, the stress required to deform the specimen will have to increase considerably to the level associated with screw dislocation glide. Thus, as is seen, the temperature dependence of the yield and flow stresses of the dilute alloys are very close to that for the flow stress for Nb, i.e. the curve which describes the temperature dependence of screw dislocation glide in pure Nb. If anomalous slip can be induced to form at 77 K in compression (table 6), it can be seen that the difference between the temperature dependence of the yield and flow stresses once again appears. Further solute additions cause a far smaller increase in the yield stress because they are merely hardening the primary screw dislocation glide and not causing any drastic change in the rate controlling mechanism.

The difference between the stresses necessary for anomalous slip and primary glide is well illustrated by the effect of a room temperature prestrain on the 77 K reyield stress for the 1 at.%Mo alloy deformed in compression (table 6). Although the exact dependence on the size of the prestrain is unclear, it was possible to obtain a mixture of primary and

anomalous slip at stresses as low as 44 kg/mm^2 (axial), only marginally above the 113 K yield stress. Primary slip alone was never observed at stresses less than 60 kg/mm^2 (axial). It is interesting to note that the 77 K yield stress in tension for virgin crystals was 44 kg/mm^2 (axial).

It is assumed that the room temperature prestrain suppresses anomalous slip by building up a dislocation structure which impedes the critical step in the generation of anomalous slip. If the prestrain is small enough then anomalous slip can 'break through', otherwise it is suppressed. Under favourable conditions load drops can still be observed (fig. 16 specimen 370/3 : 77 K). However, this rationale must be a simplification for it seems that the anomalous slip was more 'tolerant' of a room temperature prestrain when being deformed in compression; e.g. whilst a 2% axial strain in compression need not eliminate anomalous slip in 1 at.%Mo alloy at 77 K (table 6), a 1% axial strain in tension invariably caused suppression.

The displacement of anomalous slip to lower temperatures and larger stresses as the solute content increases can be qualitatively rationalised by the simplistic argument that one needs to recreate conditions equivalent to those for Nb. Decreasing temperatures will reduce the velocity of both types of dislocations but it will increase the velocity ratio, v_e/v_s . Increasing amounts of solute will also reduce the dislocation velocity but it will alter the velocity ratio in the opposite sense to the temperature. Thus equivalent conditions, at least in so far as relative velocities are concerned, will be found at lower temperatures and higher stresses when solute is present. Although this rationale 'fits' the observations, certain points indicate that it is a simplification. In the previous discussion on Nb, the presence or absence of load drops was linked to the operation of an athermal flow mechanism. The scanty evidence from virgin 10 at.%Ta alloy

specimens is that load drops occur whilst the yield stress is still temperature dependent (the temperature dependence of prestrained specimens may be due to the presence of primary slip). This would suggest that whilst the same basic mechanism is operating, other factors have altered the relative 'importance' of the steps leading to the generation of anomalous slip. In contrast, interstitial alloying causes a progressive suppression of anomalous slip and the load drops (Nb - 0 at 77 K, ref.71).

Although the 1 at.%Mo alloy showed anomalous slip at 113 K, no anomalous slip was observed in prestrained 3 at.%Mo alloy specimens after tensile deformation at temperatures down to 30 K. On the other hand, a 5 at.%Mo alloy specimen deformed at room temperature, 77 K and 20.4 K did show some limited anomalous slip after the 20.4 K deformation. It can only be suggested that the anomalous slip is affected by the nature of the dislocation arrangements introduced by straining at higher temperatures than those at which anomalous slip could possibly occur in virgin crystals.

Given the inconclusive conclusions that were reached in the discussion of the dislocation mechanisms in Nb, it is not surprising that little, if anything, constructive can be deduced with respect to the dislocation mechanisms in the alloys other than to say that the fundamental aspects of the mechanism are not affected by alloying, even though the external conditions under which it operates will be different to those of Nb.

Summary of the main conclusions

1. Niobium (4.2K - 77K).

- (a) Niobium deforms by anomalous slip over the temperature range 4.2K - 77K.
- (b) The yield and flow stresses in tension are independent of temperature between 50K and 77K. No thermodynamic analysis is possible in this temperature range. Deformation in this athermal plateau is distinguished at low strains by load drops changing to highly irregular flow as the temperature falls towards 50K.
- (c) Below 50K, the deformation regains its temperature dependence. Plastic flow is now smooth (but see below for behaviour below 20K).
- (d) It is concluded that there must be several possible rate limiting steps in anomalous slip, each controlling the deformation over a particular temperature range. The current models do not seem adequate to explain the observations, in particular the athermal plateau between 50K and 77K. Suggestions are made as to the importance of dislocation multiplication mechanisms being rate limiting in the athermal region, with a temperature dependence reappearing at lower temperatures when edge dislocation motion is rate limiting.

2. Effect of solute on primary glide.

- (a) Nb-Ta alloys: The effect of Ta additions to Nb are, in general, slight, cf. the small increases in the activation volume, ΔH and the thermal stress. This is in accord with the close similarity between the two elements, e.g. atomic size and chemical group.
- (b) Nb-Mo alloys: Molybdenum additions produce a more complex change than is found with Ta as the solute. A thermodynamic analysis

offers certain insights into the strong interaction between the solute and dislocations, e.g. the displacement to larger thermal stresses of the transition to screw dislocation glide as being rate controlling, and the change in the transformation path for thermally activated screw dislocation motion at low temperatures for the 11 and 16 at. % Mo alloys. However, it does not provide an answer to why Mo does not increase the thermal stress at all temperatures (although no solution softening is observed). Such observations suggest that the obstacles, as seen by the dislocation, alter with temperature.

3. Effect of solute on anomalous slip.

Both solutes depress the temperature at which anomalous slip first appears, e.g. to 113K for 1 at. % Mo or 77K for 10 at. % Ta. Many of the characteristics of anomalous slip in Nb are unaltered, e.g. load drops, effective Burger's vector and crystallographic slip. However, it was not possible to ascertain whether there was an athermal plateau in the yield or flow stresses. The effect of the solute appears to be to alter the relative importance of the various possible rate determining steps, and the stress required to operate them, but without altering their fundamental character.

4. Effect of solute on slip morphology.

The changes brought about by the two solutes were very much in accord with the scale of their effect on the mechanical properties. Tantalum additions produced relatively little change, whilst Mo additions produced, in the extreme, very coarse straight slip bands. The behaviour can be rationalised through a consideration of two factors, viz the stabilisation of screw dislocation arrays (glide bands) by a large athermal stress, and the effects of temperature on the initial nucleation of glide bands.

5. Overshoots were observed after changes in the strain rate for a variety of experimental conditions:-

- (a) Niobium deformed between 50K and 77K. The exact nature of these were sensitively dependent on the temperature and the strain, but in all cases they appeared to be closely linked to the athermal nature of the deformation. Thus they are not related in any simple way to a model of thermally activated dislocation motion.
- (b) Niobium deformed at or below 20K. Overshoots, first visible at 20K, became more prominent as the temperature decreased, developing into complex oscillations and apparently negative strain rate sensitivity at 10K, and then large load drops at 8K. These may all be explained by reference to the heat generated during deformation raising the temperature of the specimen during the rate changes.
- (c) The alloys deformed below ~30K. The cause is as in 5(b) above, but because deformation in the alloys is less homogeneous than in Nb and because the thermal conductivity is lower, temperature rises are more localised and there is a marked tendency towards shear zones being formed.
- (d) The alloys deformed between ~40K and 77K. A variety of overshoots were observed. It is not immediately obvious how they should be analysed. For the 11 and 16 at. % Mo alloys the overshoots extended to higher temperatures and may be connected with the change in the rate controlling process found in these more concentrated alloys.

Appendix.

Thermodynamics of dislocation glide.

The deformation of bcc metals and alloys is controlled by the thermally activated motion of dislocations. The only exceptions are when the temperature is sufficiently high for deformation to be taking place in the athermal region, or when Nb is deforming by anomalous slip between 77 K and 50 K, and at absolute zero. With these few exceptions, the passage of dislocations through the crystal is controlled by the interaction of the dislocations with localised obstacles. These localised obstacles are of such a height and extent that they may be overcome by a combination of the effective stress and thermal fluctuations (the effective stress is the difference between the applied stress, τ_a , and the stress necessary to move the dislocations over long range internal stresses, τ_μ : i.e. $\tau^* = \tau_a - \tau_\mu$). The interaction between a dislocation and one such obstacle is characterised by a Gibbs free energy profile G_b - fig. 97(a). Under an effective stress, τ^* , the interaction profile is changed to G , which includes the obstacle free energy and the work done by the effective stress. The back stress from the obstacle, τ_b , is defined as :-

$$\tau_b = \frac{1}{b} \left(\frac{\partial G_b}{\partial A} \right)_1 \quad (1)$$

and this is shown schematically in fig. 97(b). In the absence of any effective stress the equilibrium and metastable positions for the dislocation are A_1 and A_2 , whilst under stress ($\tau^* > 0$) these positions are modified to A^*_1 and A^*_2 . For the dislocation to move from A^*_1 to A^*_2 the co-operation of the effective stress and a thermal fluctuation is required.

In the approach outlined below, it is assumed that the obstacle back stress, hence G_b , does not depend upon the applied stress (linear elastic obstacle - ref. 221, 223, 351).

Consider a dislocation moving isothermally and reversibly from its equilibrium position A^*_1 to the activated position A^*_2 . The Gibbs free energy of activation is :-

$$\Delta G = b \int_{A^*_1(T, \tau^*)}^{A^*_2(T, \tau^*)} \tau_b(A, T) dA - bA^* (\tau_a - \tau_\mu) \quad (2)$$

$$\Delta G = \Delta G_i(T, \tau^*) - bA^*\tau^* \quad (3)$$

where ΔG_i is the changing free energy associated with the local barrier and the second term is the net work done by the effective stress during activation. A^* , the activation area, equals $A^*_2 - A^*_1$. It is useful to rewrite this equation in a different form. After Li (350) :-

$$\Delta G = \Delta G_o(T) - b\bar{A}\tau^* \quad (4)$$

where

$$\bar{A}(T, \tau^*) = \frac{1}{\tau^*} \int_0^{\tau^*} A^*(T, \tau^*) d\tau^* \quad (5)$$

and

$$\Delta G_o(T) = b \int_{A_1(T)}^{A_2(T)} \tau_b(A, T) dA \quad (6)$$

ΔG_o is the total reversible isothermal work required to overcome the barrier at zero effective stress. This equation follows from a consideration of the various areas under the $\tau_b - A$ plot, fig.97(b).

To link this 'single dislocation-barrier' activation energy to conventional rate theory, averaged over large numbers of dislocations,

it is necessary to assume that there is only one rate limiting step controlling the deformation. This being so then the ΔG in the conventional rate equation :-

$$\dot{\gamma} = \dot{\gamma}_0 \exp (-\Delta G/kT) \quad (7)$$

can be equated with that derived above. $\dot{\gamma}_0$ is proportional to the product of the rate at which a dislocation attempts to overcome an obstacle and the strain produced by a successful fluctuation. It also contains the mobile dislocation density. These equations may now be manipulated to relate the theoretical quantities with experimentally determinable ones. From eqn.(7) :-

$$\Delta G = - kT \ln (\dot{\gamma}/\dot{\gamma}_0)$$

$$\left(\frac{\partial \Delta G}{\partial T} \right)_{\tau^*} = - k \ln (\dot{\gamma}/\dot{\gamma}_0) - kT \left(\frac{\partial \ln(\dot{\gamma}/\dot{\gamma}_0)}{\partial T} \right)_{\tau^*}$$

$$\text{but } \Delta S = - \left(\frac{\partial \Delta G}{\partial T} \right)_{\tau^*}$$

$$\text{so } T (\Delta S)_{\tau^*} = kT \ln (\dot{\gamma}/\dot{\gamma}_0) + kT^2 \left(\frac{\partial \ln(\dot{\gamma}/\dot{\gamma}_0)}{\partial T} \right)_{\tau^*}$$

$$\text{but } \Delta G = - kT \ln (\dot{\gamma}/\dot{\gamma}_0)$$

$$\Delta G + T (\Delta S)_{\tau^*} = \Delta H = kT^2 \left(\frac{\partial \ln(\dot{\gamma}/\dot{\gamma}_0)}{\partial T} \right)_{\tau^*}$$

now if $\tau^* = \tau^*(T, \dot{\gamma}/\dot{\gamma}_0)$ then :-

$$\left(\frac{\partial \ln(\dot{\gamma}/\dot{\gamma}_0)}{\partial T} \right)_{\tau^*} \left(\frac{\partial T}{\partial \tau^*} \right)_{\dot{\gamma}/\dot{\gamma}_0} \left(\frac{\partial \tau^*}{\partial \ln(\dot{\gamma}/\dot{\gamma}_0)} \right)_T = - 1$$

$$\text{so } \Delta H = - kT^2 \left(\frac{\partial \tau^*}{\partial T} \right)_{\dot{\gamma}/\dot{\gamma}_0} \left(\frac{\partial \tau}{\partial \ln(\dot{\gamma}/\dot{\gamma}_0)} \right)_T$$

The first term in brackets is the slope of the effective stress - temperature curve and it may be obtained from the slope of the experimental plot at the appropriate temperature. The second term in brackets is related to the activation volume as :-

$$v^* = bA^* = \left(\frac{\partial \Delta G}{\partial \tau^*} \right)_T$$

This may be seen from eqns. (4) and (5). After substituting from eqn.(7):-

$$v^* = kT \left(\frac{\partial \ln(\dot{\gamma}/\dot{\gamma}_0)}{\partial \tau} \right)_T$$

$$v^* = kT \left(\frac{\partial \ln \dot{\gamma}}{\partial \tau} \right)_T - kT \left(\frac{\partial \ln \dot{\gamma}_0}{\partial \tau} \right)_T$$

If the pre-exponential factor, $\dot{\gamma}_0$, is unchanged during a strain rate change test (351) then :-

$$v^* = kT \left(\frac{\partial \ln \dot{\gamma}}{\partial \tau} \right)_T$$

Replacing the differentials by discrete changes one obtains :-

$$v^* = kT \left(\frac{\Delta \ln \dot{\gamma}}{\Delta \tau} \right)_T$$

which may be obtained from the strain rate change tests.

The total height of the barrier, ΔG_0 , can be evaluated from the ΔH - τ^* and $v^* - \tau^*$ data. Rearranging eqn. (4) :-

$$\Delta G_0 = \Delta G + b \int_0^{\tau^*} A^* d\tau^*$$

If ΔS is put equal to zero

$$\Delta H_0 = \Delta H + \int_0^{\tau^*} v^* d\tau^*$$

ΔH is known at each value of τ^* and the integral can be evaluated

graphically : it is the area under the $v^* - \tau^*$ curve up to a particular value of τ^* . The result of such an analysis is the plot in fig.102.

A much fuller analysis of the problem of thermally activated dislocation glide can be found in ref. 221 - 223, 350 - 353.

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Table 1.

Nb chemically polished in 70 : 30 conc.HNO₃ (analar) :

55% HF (commercial)

or * 60 :40 conc. HNO₃ (analar) : 48% HF (aristar)

Ta chemically polished in 2 : 2 : 1 48% HF : conc.HCl : conc.HNO₃

Mo electropolished in 1 : 7 solution of conc.H₂So₄ : CH₃OH

* The change became necessary when 55% HF was withdrawn from production soon after the work began. A 70 : 30 mixture using the 48% HF did not give as good a polish as the solution using 55% HF.

Table 2.

Boiling water	373 K
room temperature	290 K
s-1 chlorobenzene	225 K
s-1 acetone	175 K
s-1 ethanol	158 K
s-1 n-pentane	145 K
s-1 isopentane	113 K
liquid nitrogen	77 K
s-1 nitrogen	63 K
liquid hydrogen	20.4 K
liquid helium	4.2 K

Table 3.

Nominal Composition at. %	Crystal number	As made up composition at. %	Composition ex density at. %	Composition ex microanalysis	290 K shear yield stress kg/mm ²
1 Mo	302	1.2		1.1 ± 0.2*	1.75
	332	1.1			1.8
	326	1.0			-
	370	1.5			2.4
	375	1.5			2.5
2 Mo	300	1.9		1.8 ± 0.2*	2.8
	331	2.7			4.1
3 Mo	368	2.7			4.4
	322	2.7		2.8 ± 0.2*	4.6
	287	4.6		4.4 ± 0.2*	7.5
5 Mo	308	4.6		4.6 ± 0.2*	7.6
	346	5.3			7.8
	371	5.7			8.5
	361	9.1			12.4
11 Mo	309	9.7		9.4 ± 0.3*	14.0
	373	10.5			14.4
	292	11.0		10.9 ± 0.3*	15.0
16 Mo	335	11.1			16.4
	311	19.0		16.7 ± 0.4*	22.1

* only 1 specimen

(CONTINUED ON THE NEXT PAGE)

Table 3 - continued.

Nominal Composition at.%	Crystal number	As made up composition at.%	Composition ex density at.%	Composition ex microanalysis	290 K shear yield stress kg/mm ²
4 Ta	295	3.7	3.7 ± 0.5#		1.1
	362	10.0			1.3
10 Ta	312	9.8	12.9 *		1.3
	297	10.5	11.2 ± 1.0#	11.6 ± 0.3	1.8
	336	48.5	57.5 +		5.0
	377	51.8			5.5
60 Ta	339	48.5	57.5 +		5.7
	316	48.7	59.7 *		6.6
	296	49.5	61.0 ± 1.0		7.2

* only 1 specimen

+ only 2 specimens

depends upon the position in the crystal

Table 4.

The number of specimens from each crystal tested in
compression or tension.

Alloy group	Crystal	Compression	Tension	Comments.
1 Mo	302	24	-	
	332	-	5	
	326	1	3	
	370	-	5	
	375	-	4	
2 Mo	300	21	-	
	331	-	4	
3 Mo	368	-	4	
	322	10	3	
	287	21	-	13 used in part II *
5 Mo	308	2	5	
	346	-	5	
	371	-	4	
	361	-	5	
	309	3	3	
11 Mo	373	-	4	
	292	25	-	12 used in part II*
	335	-	4	
16 Mo	311	19	-	

* These crystals were grown and partially tested for the part II examination in the Honour School of Natural Science, Metallurgy and Science of Materials.

(CONTINUED ON THE NEXT PAGE)

Table 4 (continued).

The number of specimens from each crystal tested in
compression or tension.

Alloy group	Crystal	Compression	Tension	Comments
4 Ta	295	17	-	13 used in part II*
	362	-	4	
10 Ta	312	3	4	
	297	22	-	
	336	3	1	
	377	-	4	
60 Ta	339	-	2	
	316	2	3	
	296	13	-	
Nb	6 crystals	6	21	8 used in part II*
				crystal no.'s 354,355, 356,357,358,369

* These crystals were grown and partially tested for the part II examination in the Honour School of Natural Science, Metallurgy and Science of Materials.

Table 5.

10 at.%Ta
crystals 297 and 312.

Specimen	$\dot{\epsilon}$ s ⁻¹	290 K shear yield stress kg/mm ²	290 K strain shear %	77 K shear reyield stress kg/mm ²	temperatures at which deformed	slip systems
297/3	4×10^{-4}	1.9	3.2	33.6	77	P
297/8	4×10^{-4}	1.8	4.0	33.5	77	P
297/17	1.2×10^{-4}	1.9	5.0	26.4	77 - 63 - 4.2	P
297/18	1.2×10^{-4}	2.1	5.0	30.9	77 - 63 - 4.2	P
297/20	1.2×10^{-4}	1.7	5.0	28.6	77 - 4.2	P
297/21	1.2×10^{-4}	1.9	3.6	29.0	77 -20.4	P + A
297/22	1.2×10^{-4}	1.9	3.4	32.3	77-60-40-20	P + A
312/6	1.2×10^{-4}	1.3	3.0	29.8	77- 54	P + A

P = Primary system

A = Anomalous system

Table 6.

crystal 302 : 1 at.%Mo

Specimen number	strain rate s^{-1}	290 K shear yield stress kg/mm ²	290 K prestrain shear %	77 K shear reyield stress kg/mm ²	slip system	work hardening after yield	load drops at yield ?
3	4×10^{-4}	1.8	3.9	31.0	P	negative	yes
12	4×10^{-4}	1.7	4.6	32.3	P	negative	yes
17	1.2×10^{-4}	1.9	3.3	23.4	P + A	positive	no
18	1.2×10^{-4}	1.8	3.0	28.3	P + A	zero	no
19	1.2×10^{-4}	1.8	3.1	30.8	P	negative	yes
20	1.2×10^{-4}	1.7	4.3	21.8	P + A	positive	no
21	1.2×10^{-4}	1.7	3.2	30.3	P + A	negative	no
23	1.2×10^{-4}	1.7	4.6	31.0	P + A	negative	no

P = Primary slip

A = Anomalous slip

Table 7.

77 K yield or reyield stresses : kg/mm² (axial)

composition	crystal	Compression		Tension	
		virgin	prestrained	virgin	prestrained
	370				30.0†
1 Mo	302		40† - 60#		
	326			40.8†	
	332			46.0*	34.0#
2 Mo	300	47.0*	56.0#		
3 Mo	322		56.0		
	331				38.0-41.0
	287	66.2	55.2		
5 Mo	308			60.1	
	346				51.2
11 Mo	292	80.0	74.1		
	309			76.3	64.6
	297	36.6*	52.8-67.8†		
10 Ta	312		59.6†	44.2†	
	362				36.5#
60 Ta	296				
	316			62-54	30.5

† slip on anomalous and primary systems

slip on primary system only

* twinned

Table 8.

1 Mo		2 Mo		3 Mo		5 Mo		11 Mo		16 Mo		4 Ta		10 Ta		60 Ta	
T(K)	$\Delta\tau$	5%	$\Delta\tau$	5%	$\Delta\tau$	5%	$\Delta\tau$	5%	$\Delta\tau$	5%	$\Delta\tau$	5%	$\Delta\tau$	5%	$\Delta\tau$	5%	$\Delta\tau$
298	+0.29	0.1	+0.19	0.2	0	0.2	+0.56	0.4	+0.1	0.7	-0.5	1.1	-0.17	0.1	+0.23	0.1	+0.78
225	+0.39	0.18	+0.81	0.25	-	-	+1.2	0.57	+0.4	1.0	+1.0	1.4	-0.16	0.19	-0.09	0.2	-0.36
175	+0.23	0.37	+0.31	0.4	-	-	+0.29	0.7	-0.8	1.3	+0.1	1.7	-	-	-0.02	0.45	-0.8
158	+0.47	0.5	+0.4	0.51	-0.4	0.6	-0.96	0.75	-0.55	1.4	-1.1	1.8	+0.78	0.55	+0.01	0.6	+0.1
145	+0.94	0.6	+0.35	0.6	-0.5	0.7	-1.72	0.8	-0.5	1.5	-0.9	2.0	-	-	+1.22	0.8	-0.8
113	+2.0 *	0.8	-0.08	0.8	+0.6	0.96	+0.9	1.1	-0.4	1.6	-1.0	2.1	-1.7 *	0.8	+1.94	1.0	+1.0
77	+0.9 +	1.5	+1.3 +	1.4	+1.1 +	1.4	+2.4	1.6	+1.1	2.0	-1.95	2.4	+2.8 +	1.6	+1.6 +	1.5	+3.5 +
to	to	to	to	to	to	to	to	to	to	to	to	to	to	to	to	to	to
-3.0 "							-2.3 +		-1.8 +						-5.8 #		

+ room temperature prestrain - primary slip

room temperature prestrain - primary and possibly anomalous slip

* some anomalous slip

" room temperature prestrain - primary and anomalous slip

5% = 5% of the yield stress
 $\Delta\tau = (\tau_y - \tau_{plateau}) - \tau^*$

τ_y - τ^* (kg/mm²)

Table 9.

Summed stress differences from temperature change tests below 77 K.

All stresses in kg/mm² (axial)

T(K)	Nb	1 Mo	3 Mo	5 Mo	11 Mo	60 Ta
70	0.2	5.8	10.2	6.4	3.6	7.2
60	0.7	10.2	14.8	10.2	5.8	11.4
50	0.6	15.0	19.0	13.6	7.6	17.8
40	1.2	17.4	21.0	18.0	13.2	22.2
30	6.1	25.0	26.8	22.8	20.2	25.4
20	11.5	30.5	32.2	27.6	23.0	30.5
15	15.5					
13						36.3
10	19.8					
8	24.2	39.7	41.2			
4.2	29.2					

Table 10.

Elastic Constants.

	C_{11}	C_{12}	C_{44}	A	μ	ref.
Nb	24.6	13.4	2.87	0.51	4.69	335
Ta	26.7	16.1	8.25	1.56	6.28	335
Mo	46.96	16.76	10.68	0.71	13.63	336

$$A = 2 C_{44} / (C_{11} - C_{12})$$

$$\mu = 1/3 (C_{11} - C_{12} + C_{44})$$

C_{ij} in units of 10^{11} dynes/cm²

lattice parameters (ref.337)

Nb	3.3007	$\times 10^{-10}$	m
Ta	3.3026	$\times 10^{-10}$	m
Mo	3.1466	$\times 10^{-10}$	m

Misfit parameters

$$\text{Size misfit parameters} = \frac{1}{a} \frac{da}{dc}$$

$$\text{Modulus misfit parameter} = \frac{1}{\mu} \frac{d\mu}{dc}$$

The misfit parameters for Nb-Ta alloys have been taken as linear functions of composition.

For Nb-Mo alloys, C_{ij} is linear with composition for compositions less than 16 at.%Mo (320). The variation of the lattice parameter with composition for Nb-Mo alloys has been calculated from data on atomic volumes (338).

Table 11.

Nomenclature.

R	= specimen's radius	
$\bar{k}$	= thermal conductivity	ref. 345
h	= surface heat exchange coefficient = $10^3 \text{ J/m}^2 \cdot \text{s} \cdot \text{deg}$	cf.ref. 342
E	= combined modulus of specimen and machine = $3 \times 10^{10} \text{ Pa}$	
c	= specific heat capacity	ref. 345
k	= Boltzmann's constant	
$\dot{\gamma}_a$	= applied strain rate	
Reduced strain rate	$\xi = \frac{REc}{2h\tau} \dot{\gamma}_a$	
Reduced activation volume	$\alpha = \frac{v^*\tau}{kT}$	
Reduced thermodynamic parameter	$v_r = \frac{\tau^2 \Delta H}{Ek c T^2}$	
Reduced activation enthalpy	$\beta = \frac{\Delta H}{kT}$	

T (k)	4.2	8	10	20	30
$\Delta H \text{ eV}$	0.016	0.025	0.035	0.048	0.077
c $\text{J/m}^3 \cdot \text{deg}$	3.7×10^3	1.1×10^4	2×10^4	8×10^4	2.2×10^5
$\bar{k} \text{ J/m} \cdot \text{s} \cdot \text{deg}$	8.4		25.2		
α	262	213	200	90	88
v_r	7560	1050	377	23.1	4.47
v_r in units of α	28.8	4.9	1.88	0.26	0.051
ξ	7.1×10^{-4}	2.3×10^{-3}	4.5×10^{-3}	2.2×10^{-2}	6.8×10^{-2}
ξ in units of $1/\alpha$	0.18	0.4	0.9	1.94	6.0

Fig. 1 Initial orientations.

All the alloy crystals lay within the hatched area with the exception of :-

crystal 296 - Nb-60 at.%Ta

crystal 300 - Nb-2 at.%Mo

crystal 326 - Nb-1 at.%Mo

Fig.1

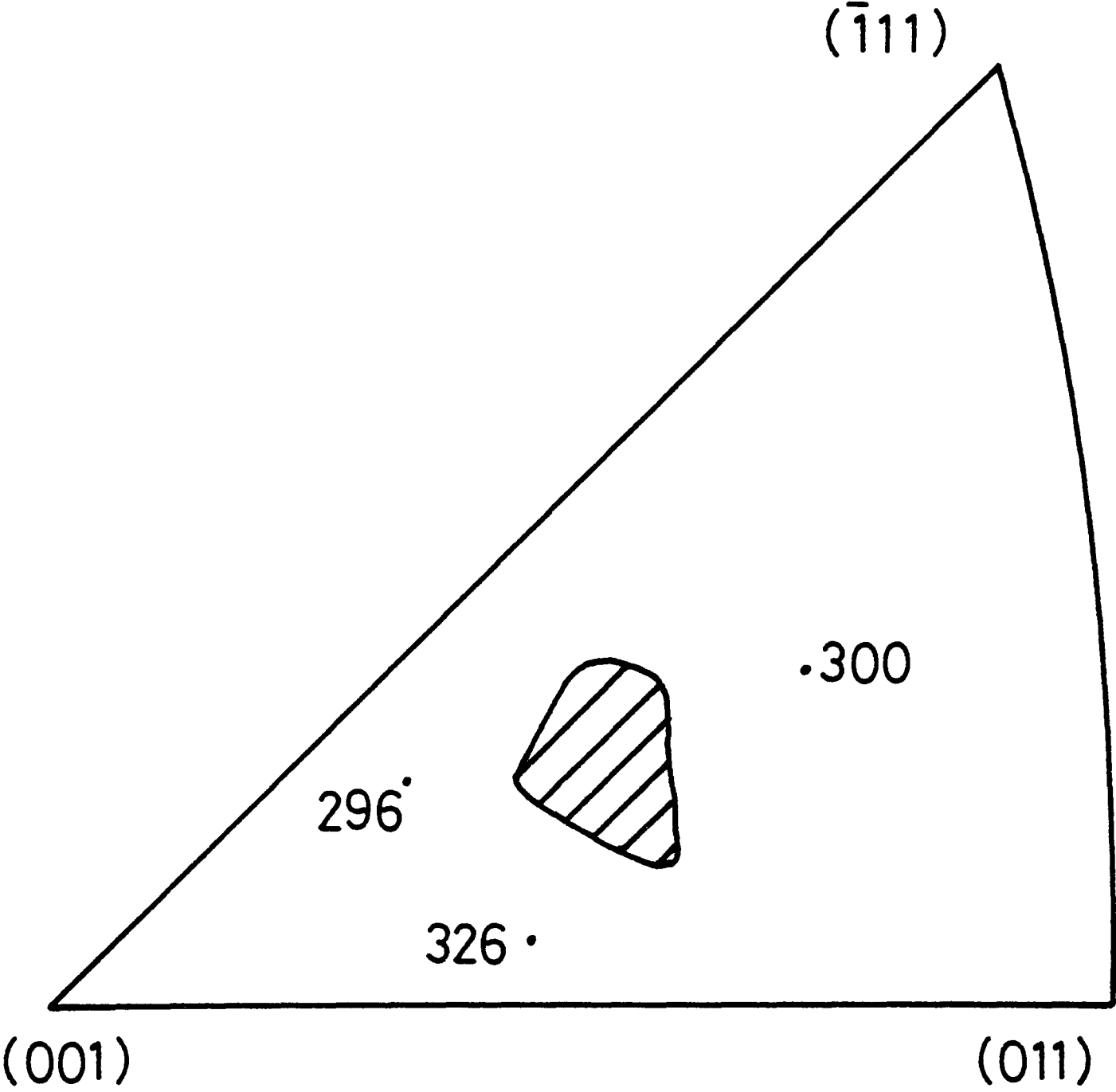


Fig. 2 Shear stress - shear strain for Nb
Compression

Fig.2

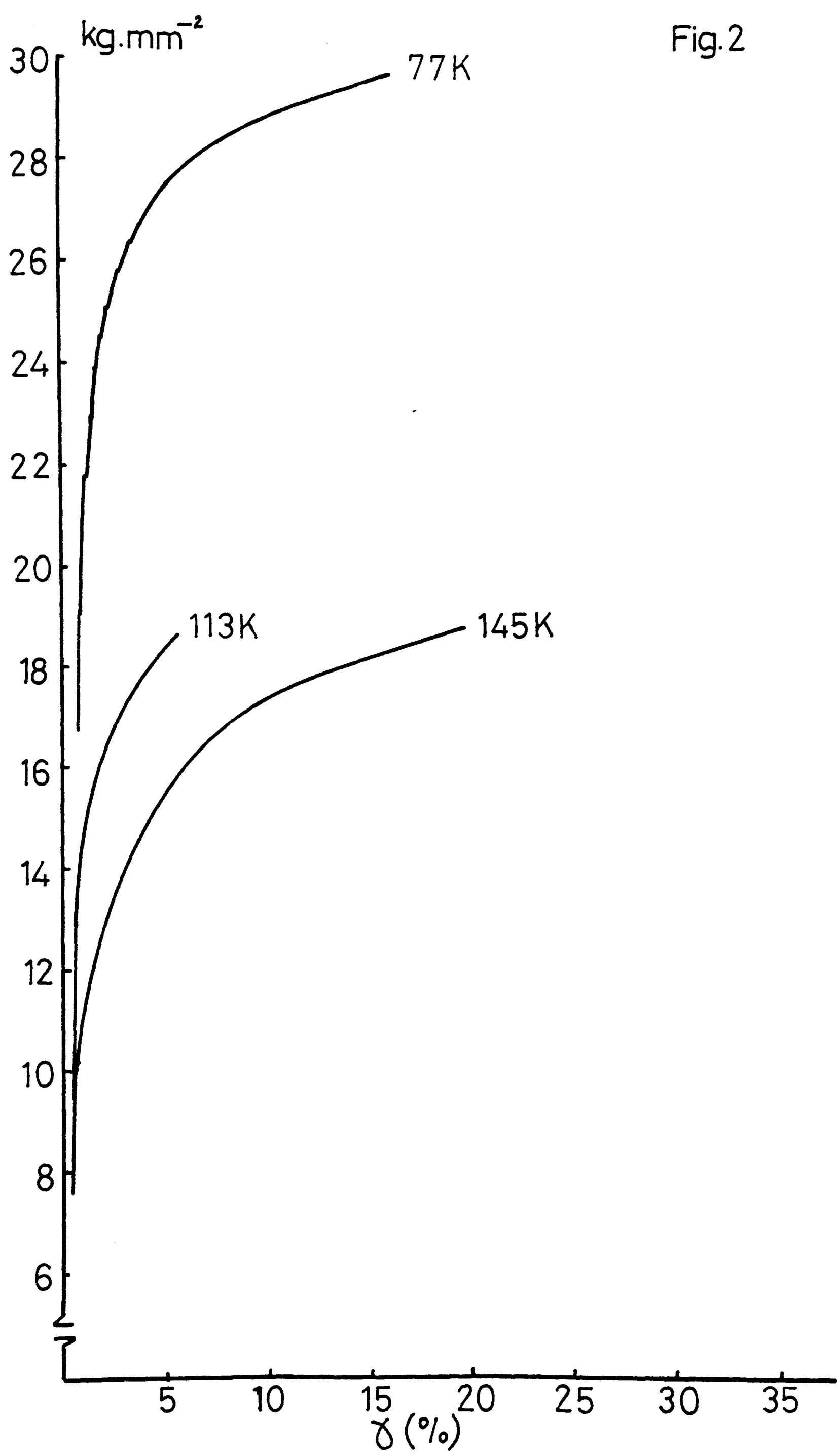


Fig. 3 Shear stress - shear strain for Nb-4 at.%Ta
 Compression

Fig. 3

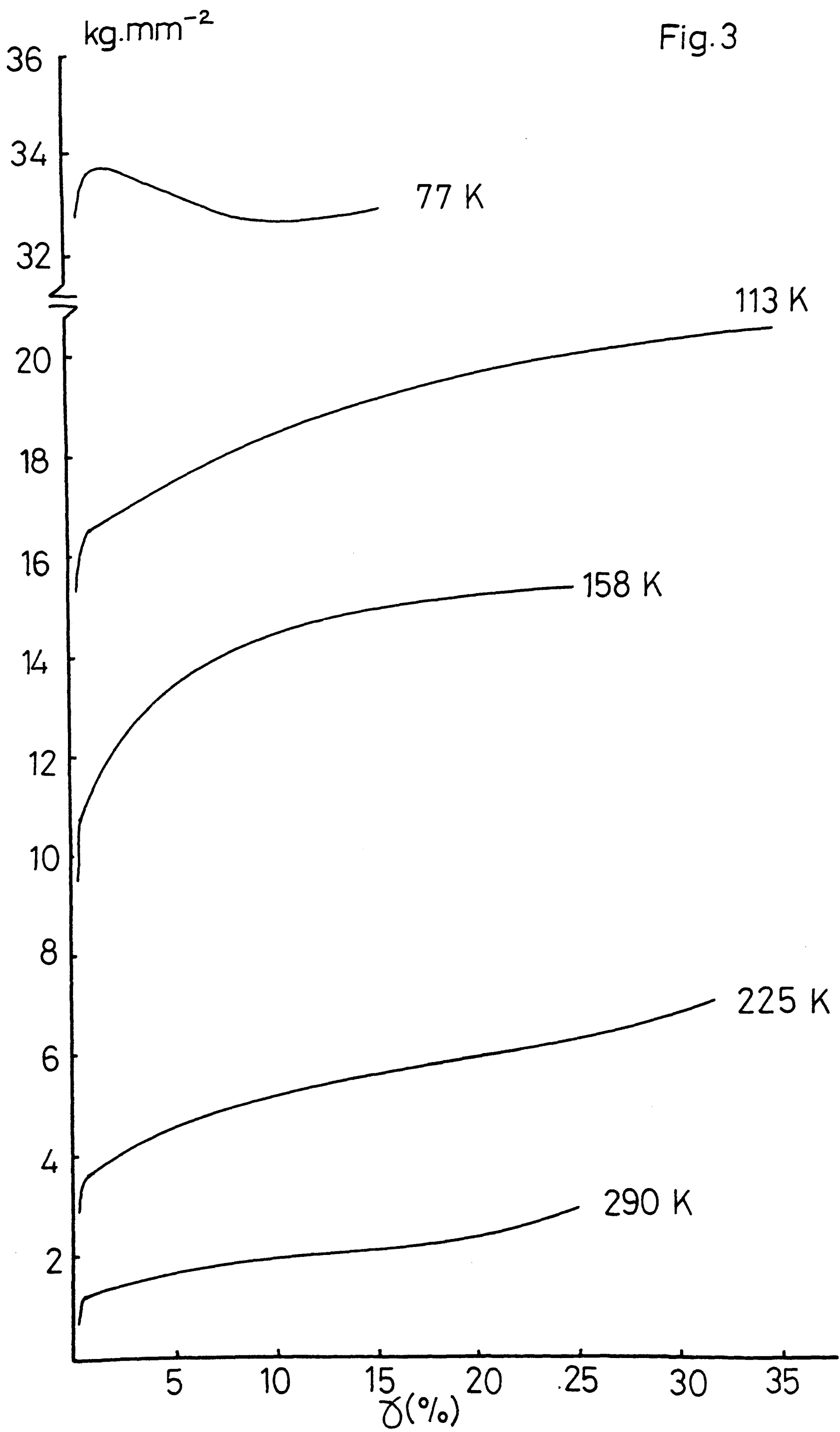


Fig. 4 Shear stress - shear strain for Nb-10 at.%Ta
Compression

Fig.4

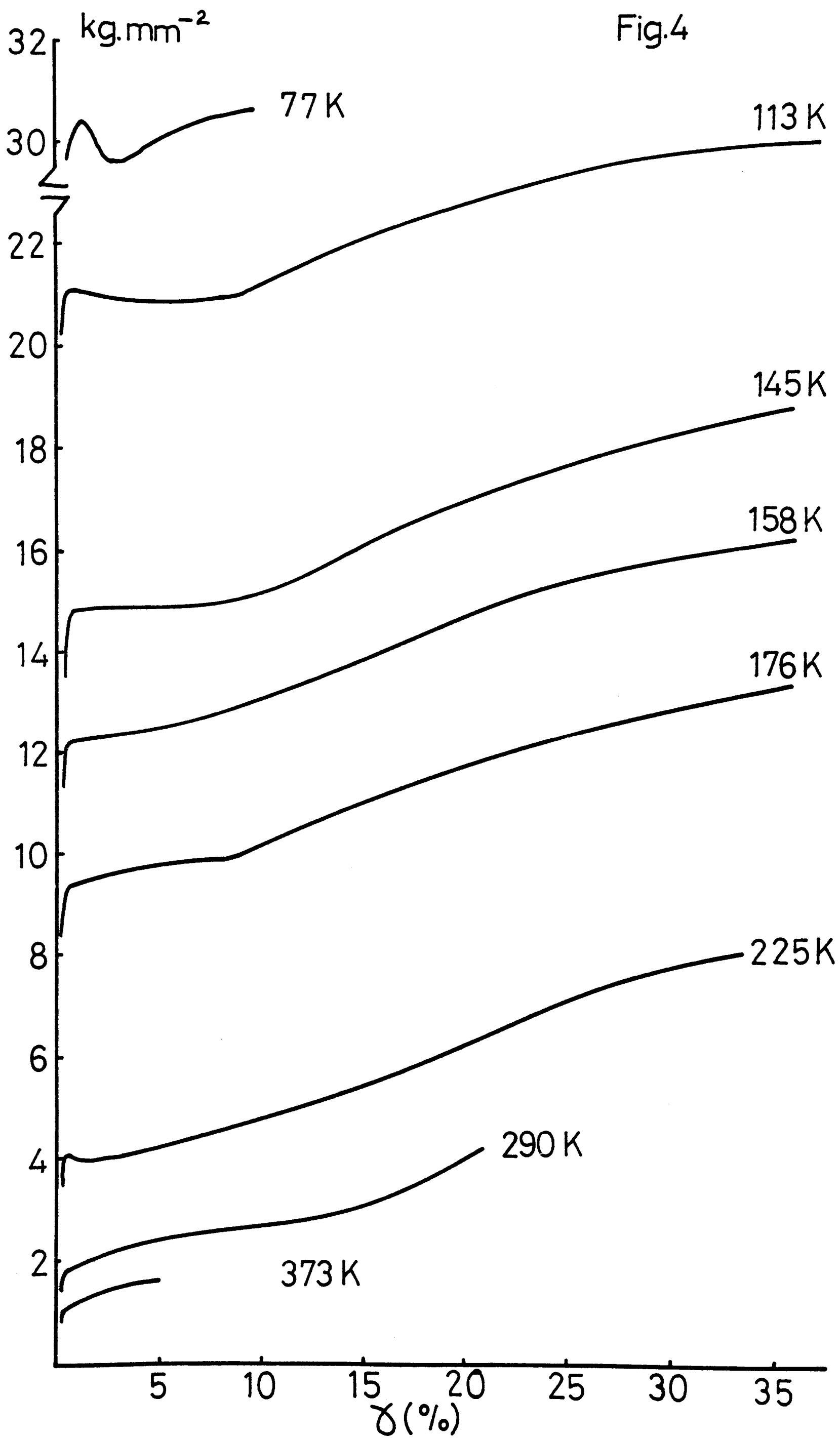


Fig. 5 Shear stress - shear strain for Nb-60 at.%Ta
 Compression

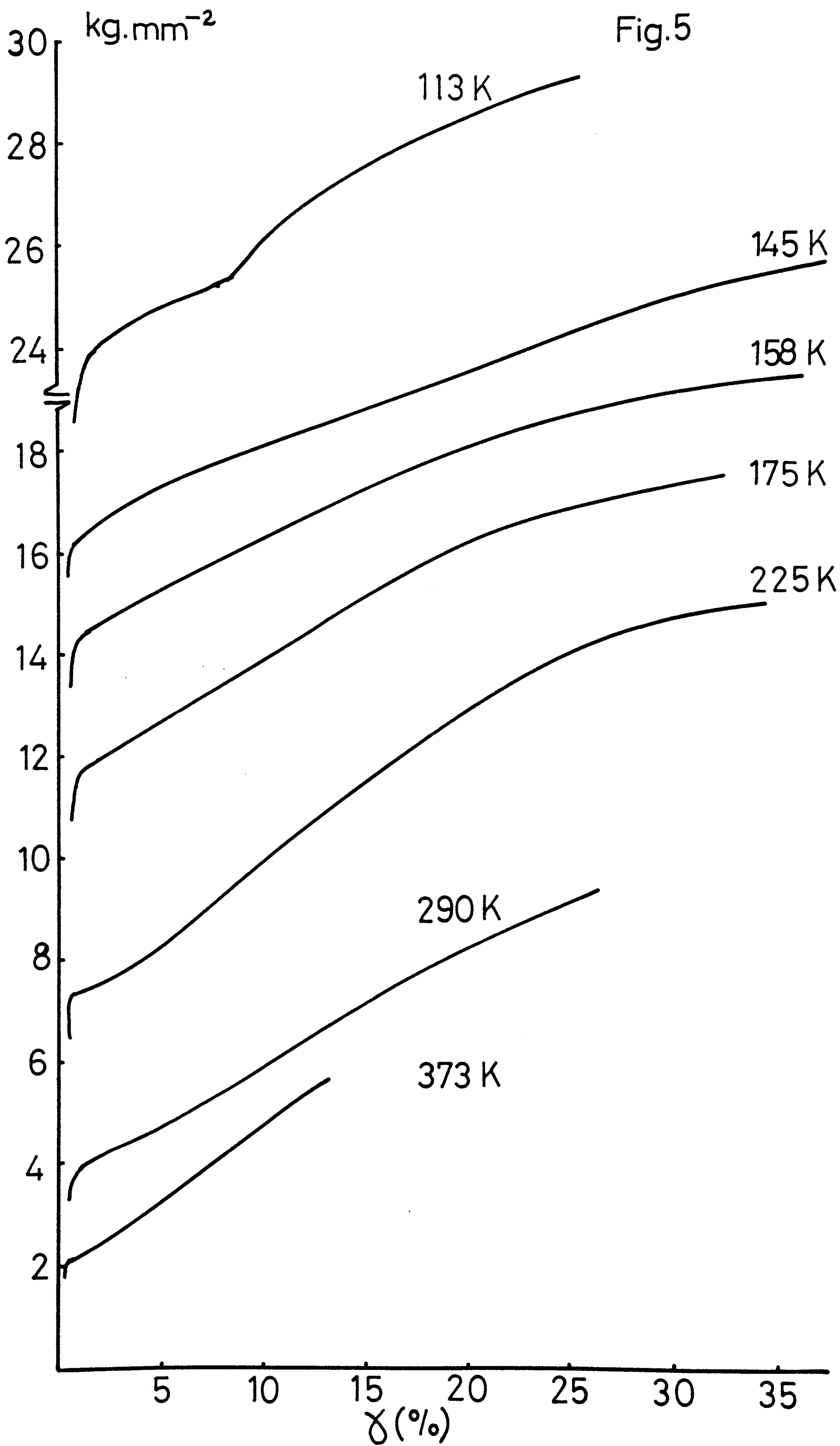


Fig. 6 Shear stress - shear strain for Nb-1 at.%Mo

Compression

Specimen 302/17 was prestrained 2.9% at room temperature

Specimen 302/12 was prestrained 4.5% at room temperature

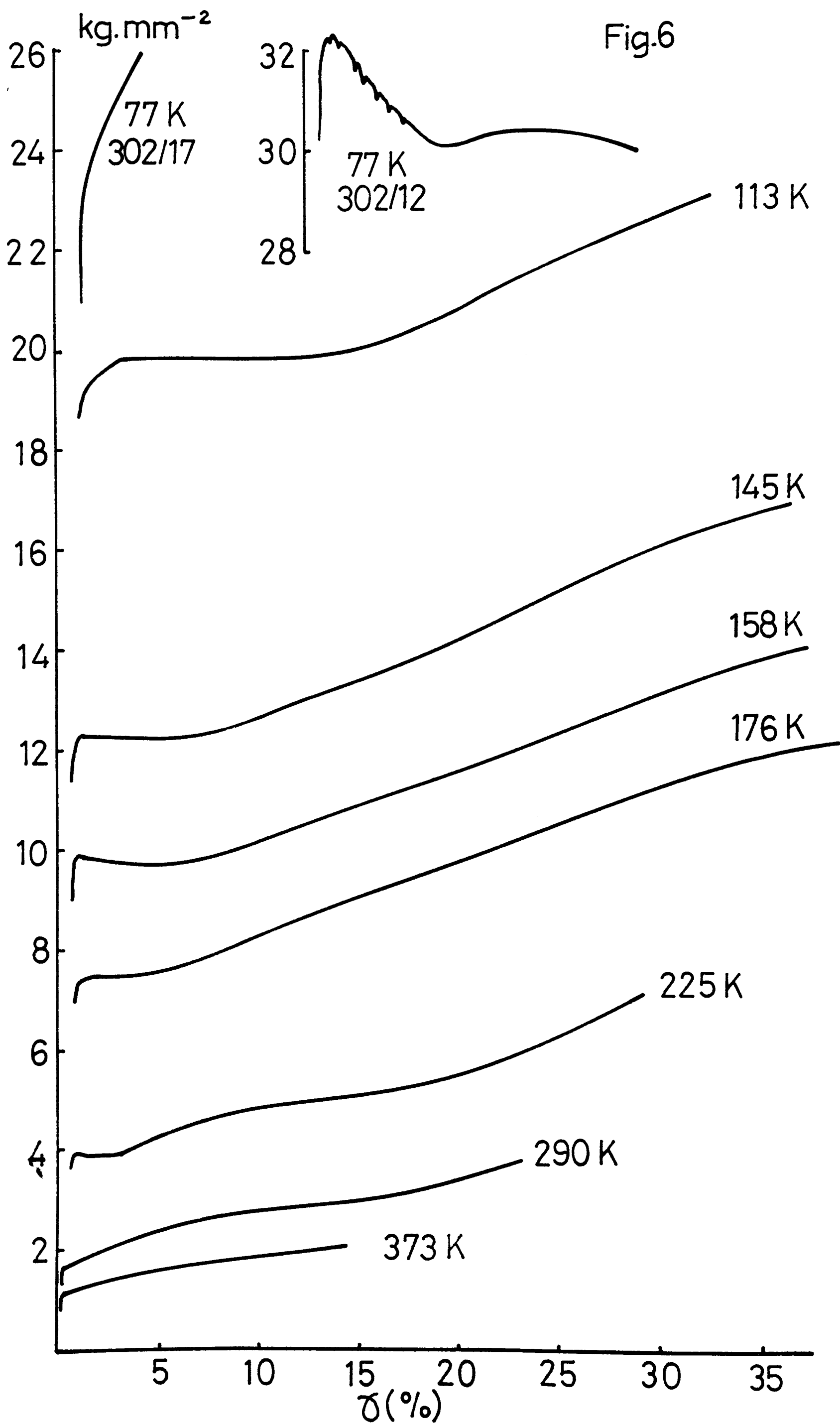
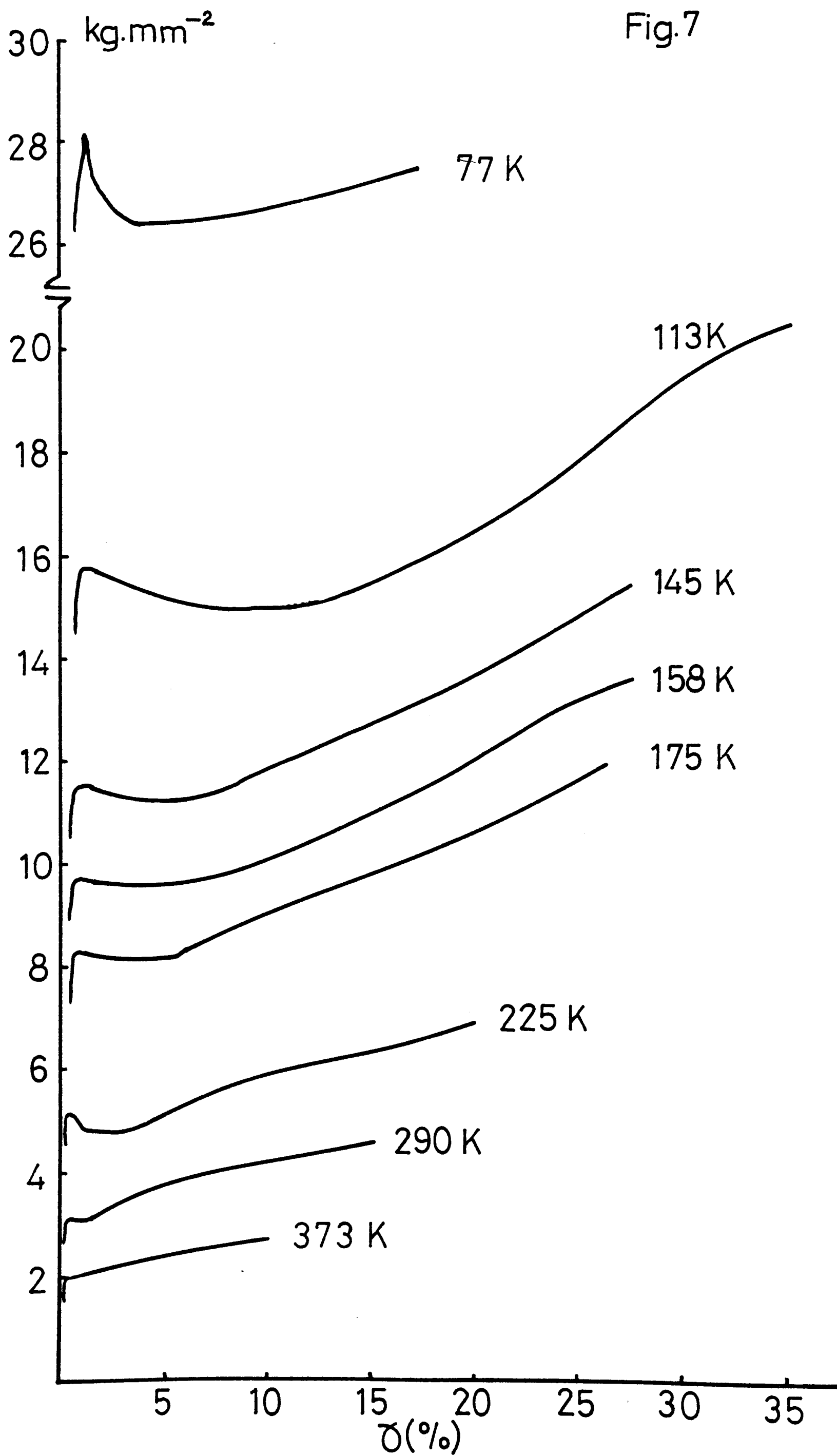


Fig. 7 Shear stress - shear strain for Nb-2 at.%Mo
Compression

Fig.7



[illegible]

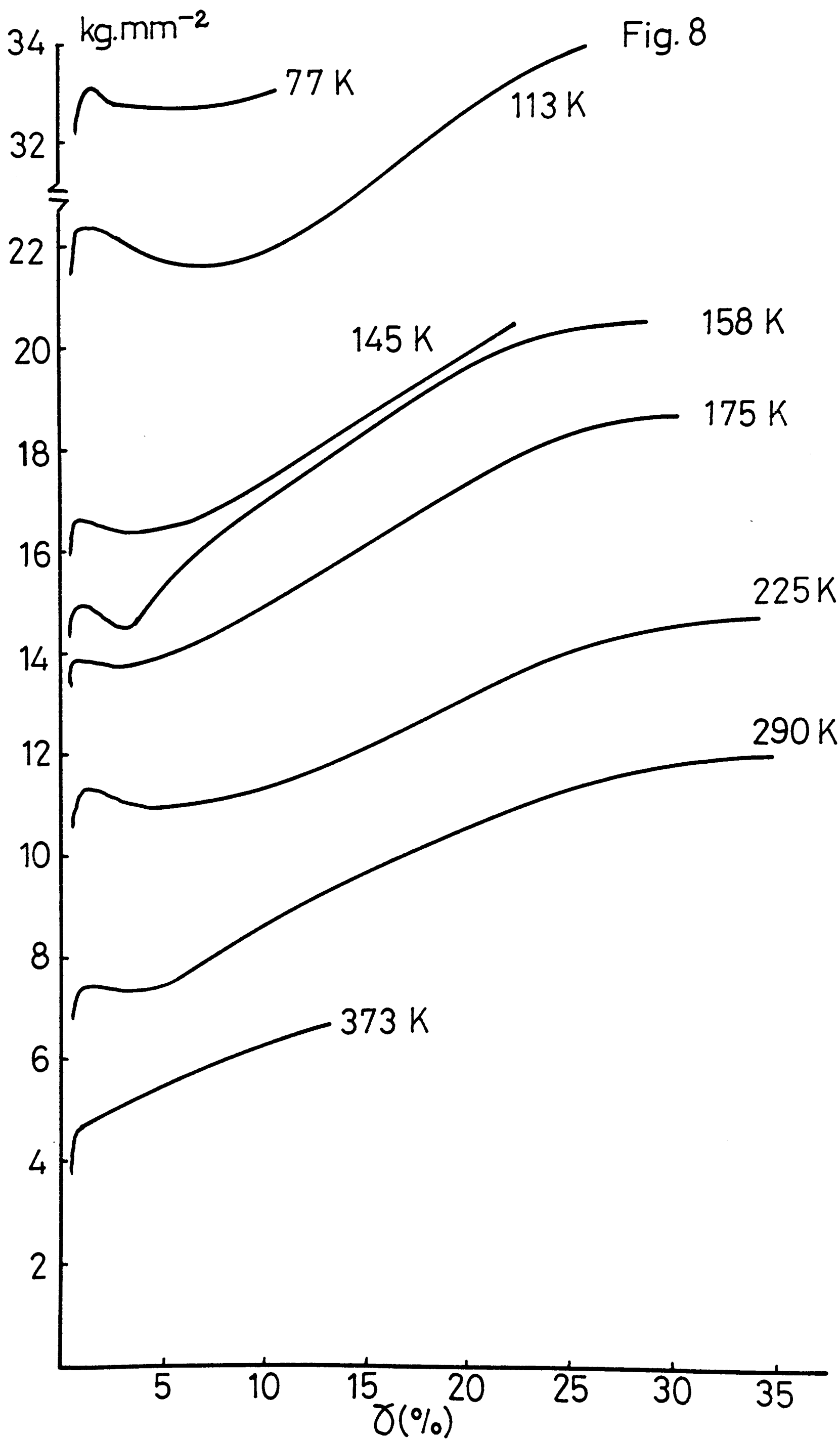


Fig. 9 Shear stress - shear strain for Nb-11 at.%Mo
Compression

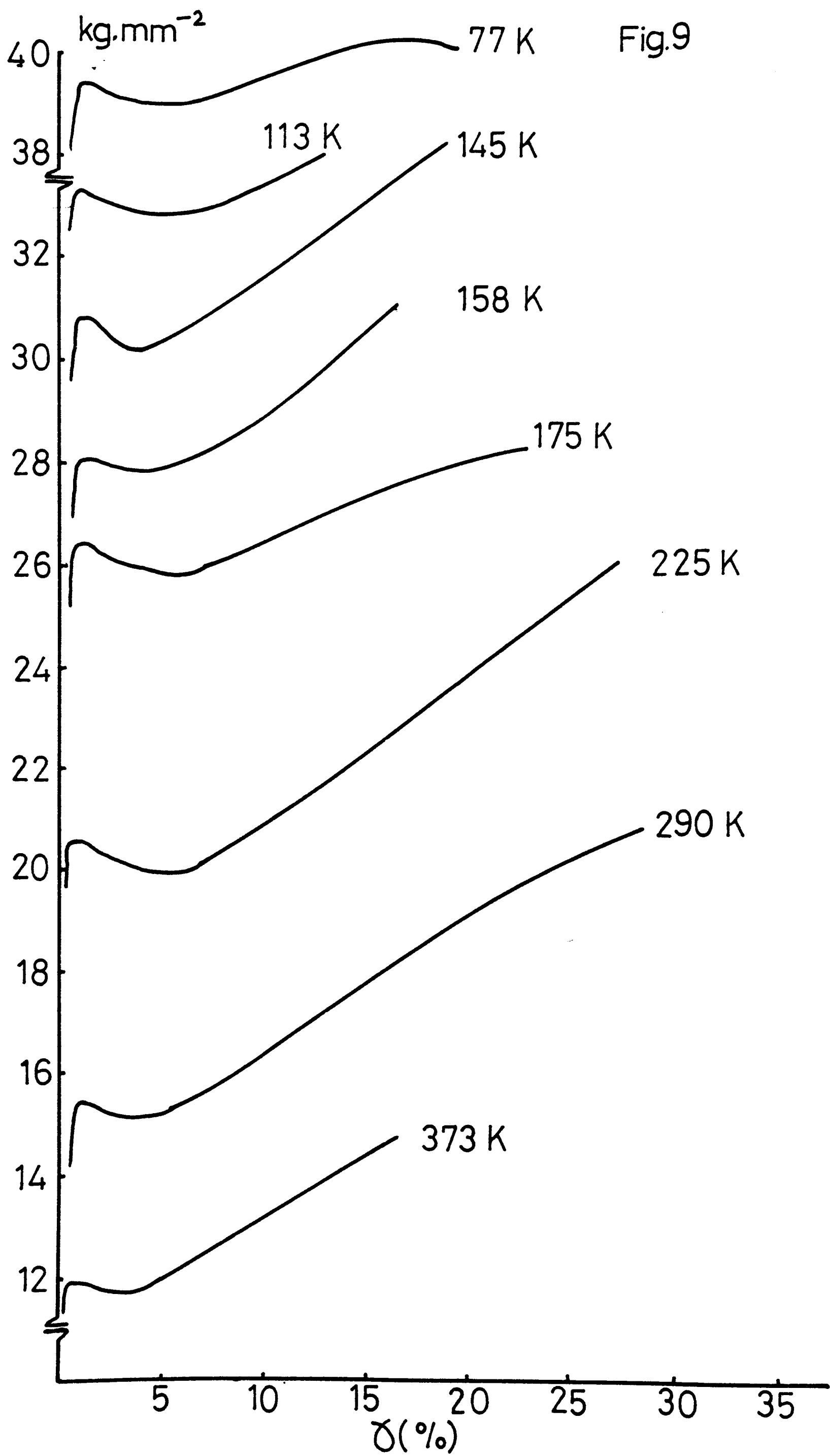


Fig. 10 Shear stress - shear strain for Nb-16 at.%Mo
Compression

Fig.10

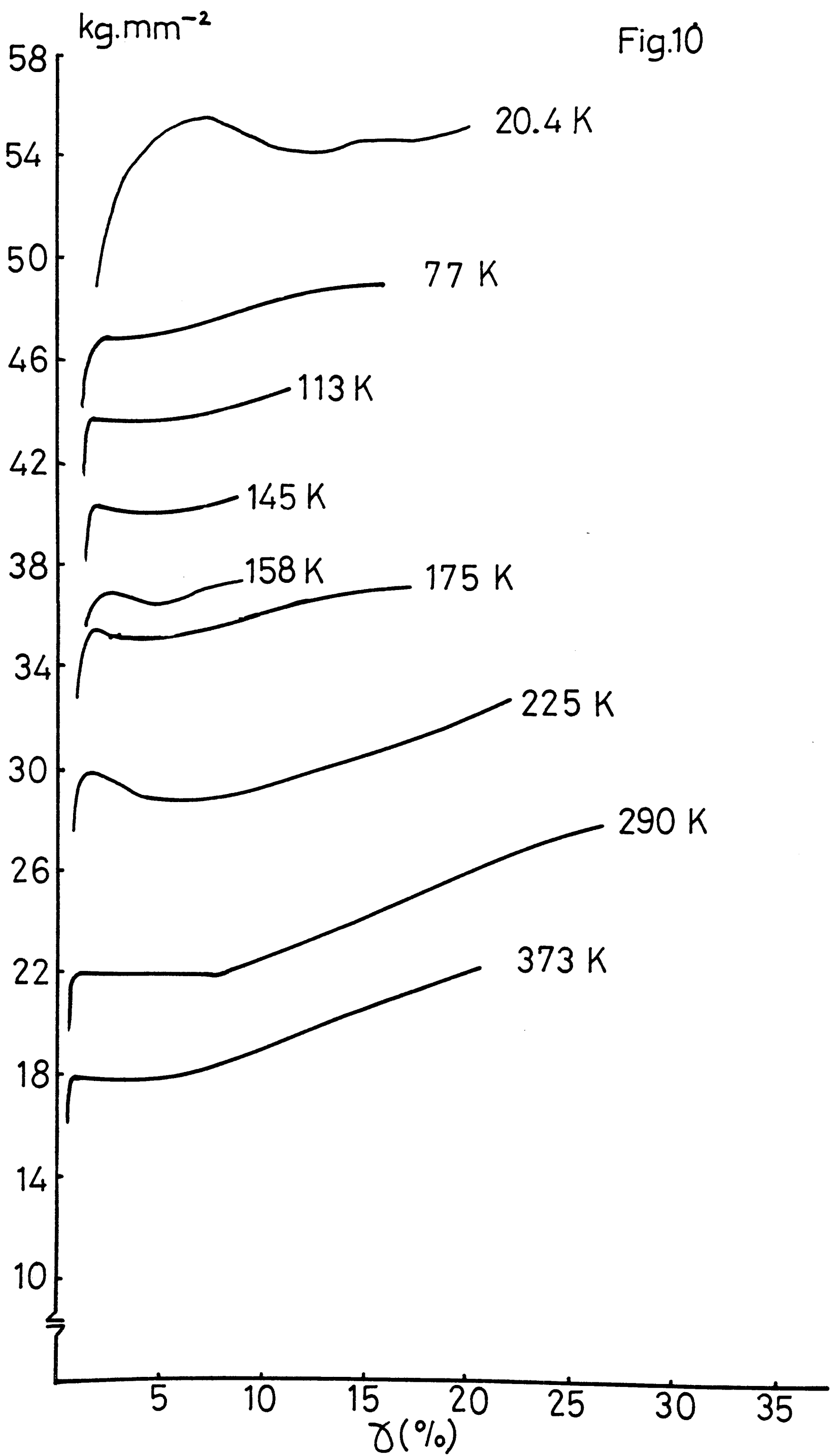


Fig. 11 Axial stress - axial strain for Nb deformed
at 77 K, 30 K and 20 K in tension.

The expansion is the yield region at 77 K.

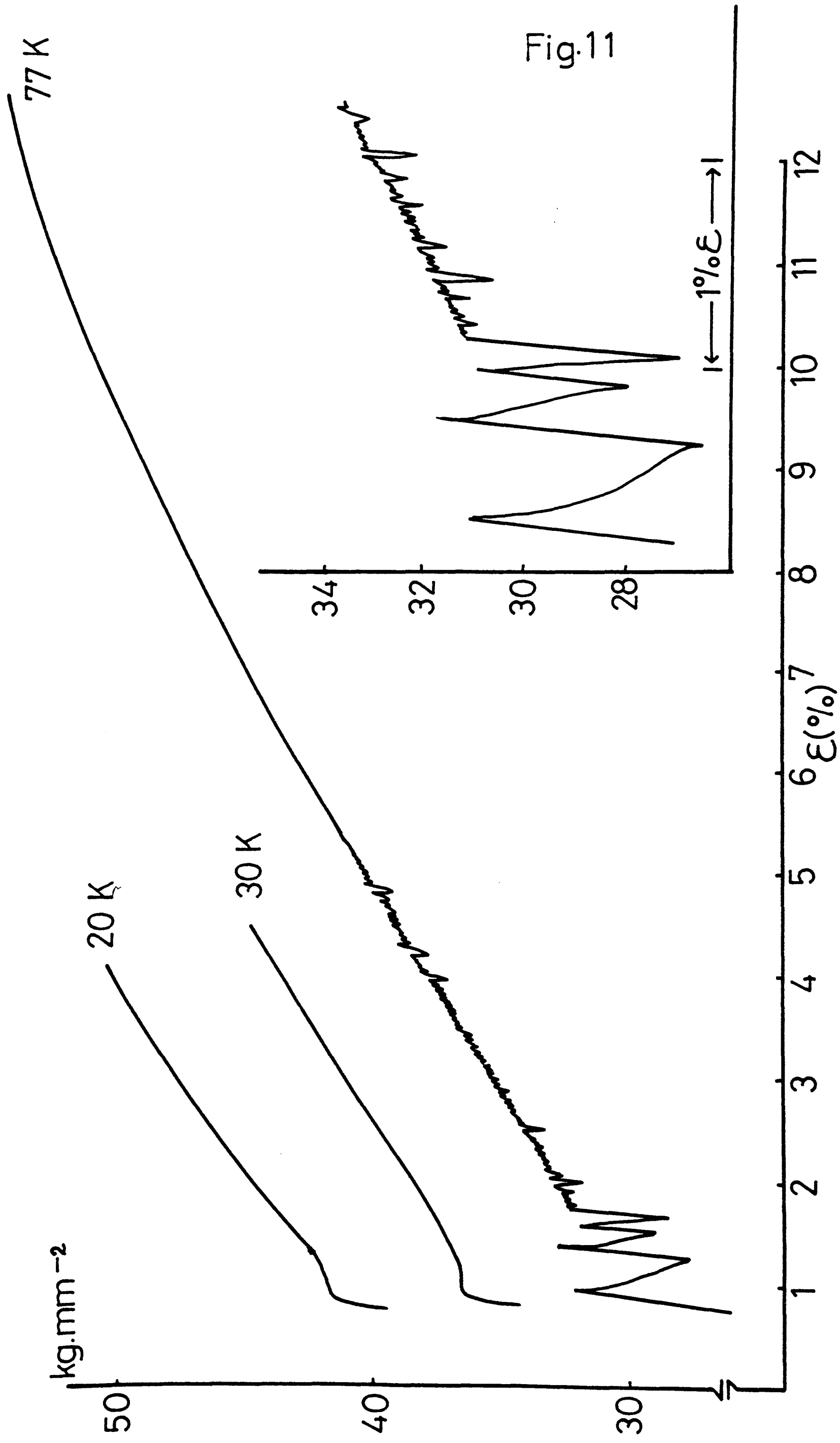


Fig. 12 Axial stress - axial strain for Nb
 deformed at 70 K and 60 K in tension

Fig.12

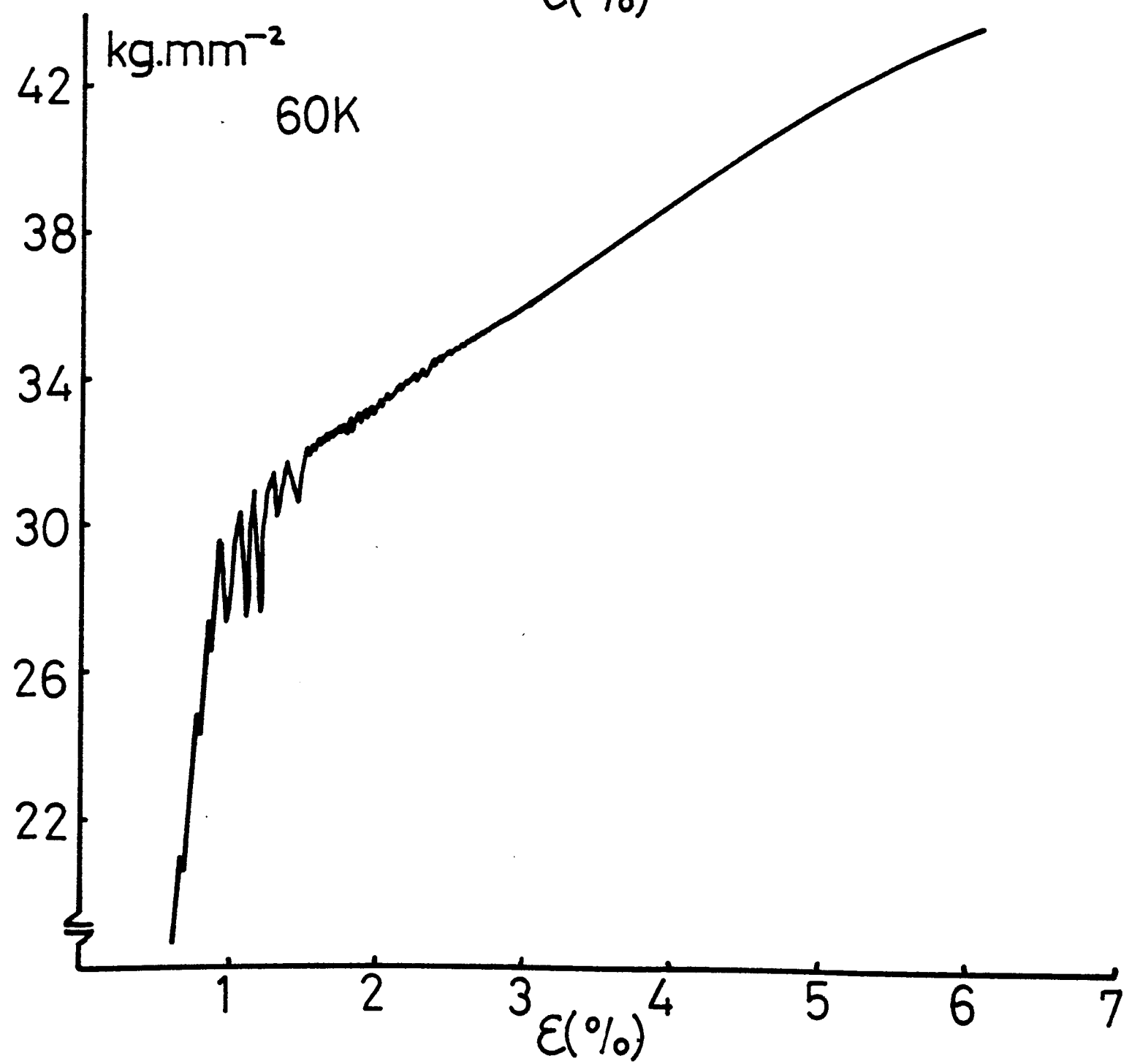
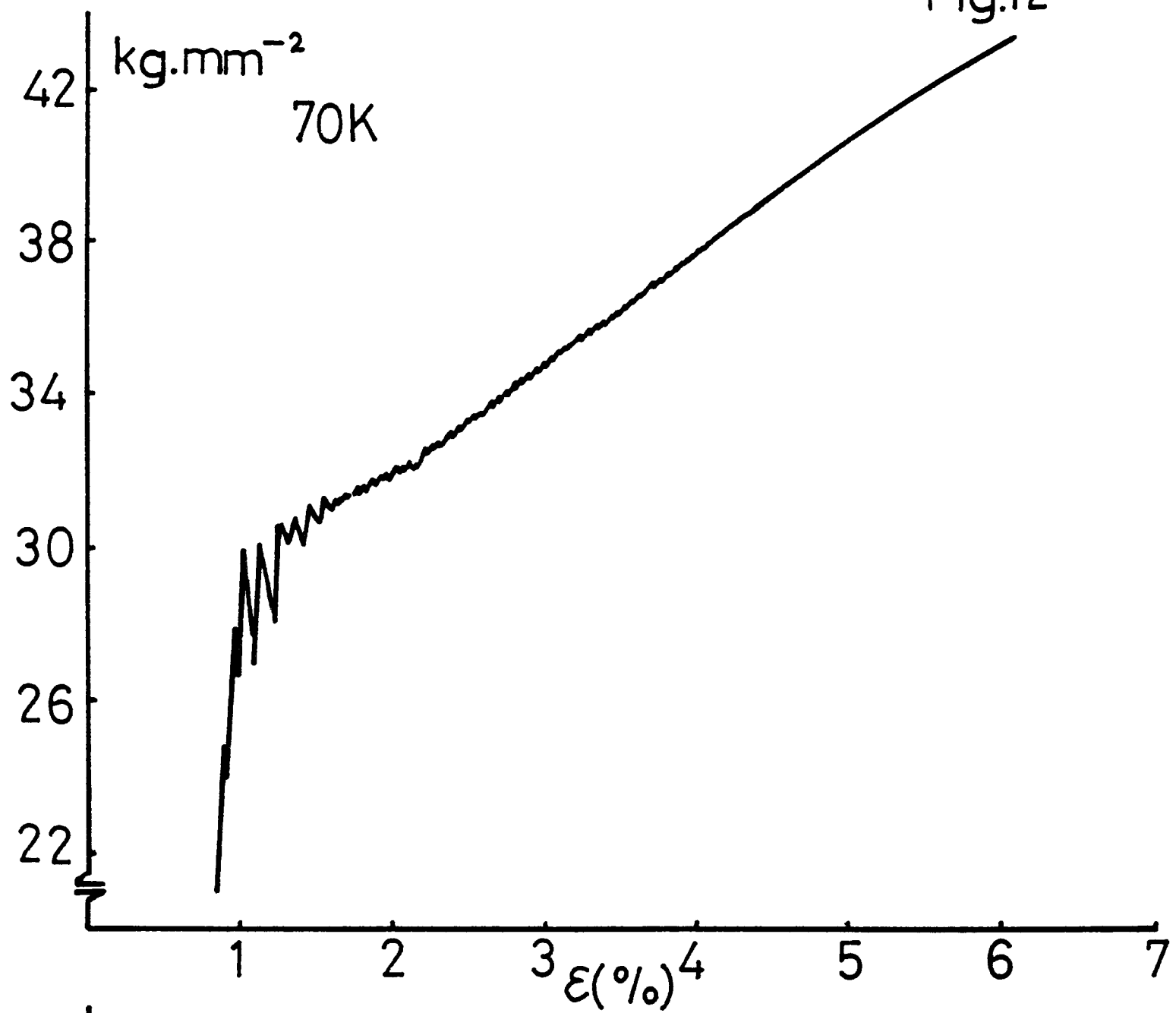


Fig. 13 Axial stress - axial strain for Nb deformed
 at 50 K and 40 K in tension

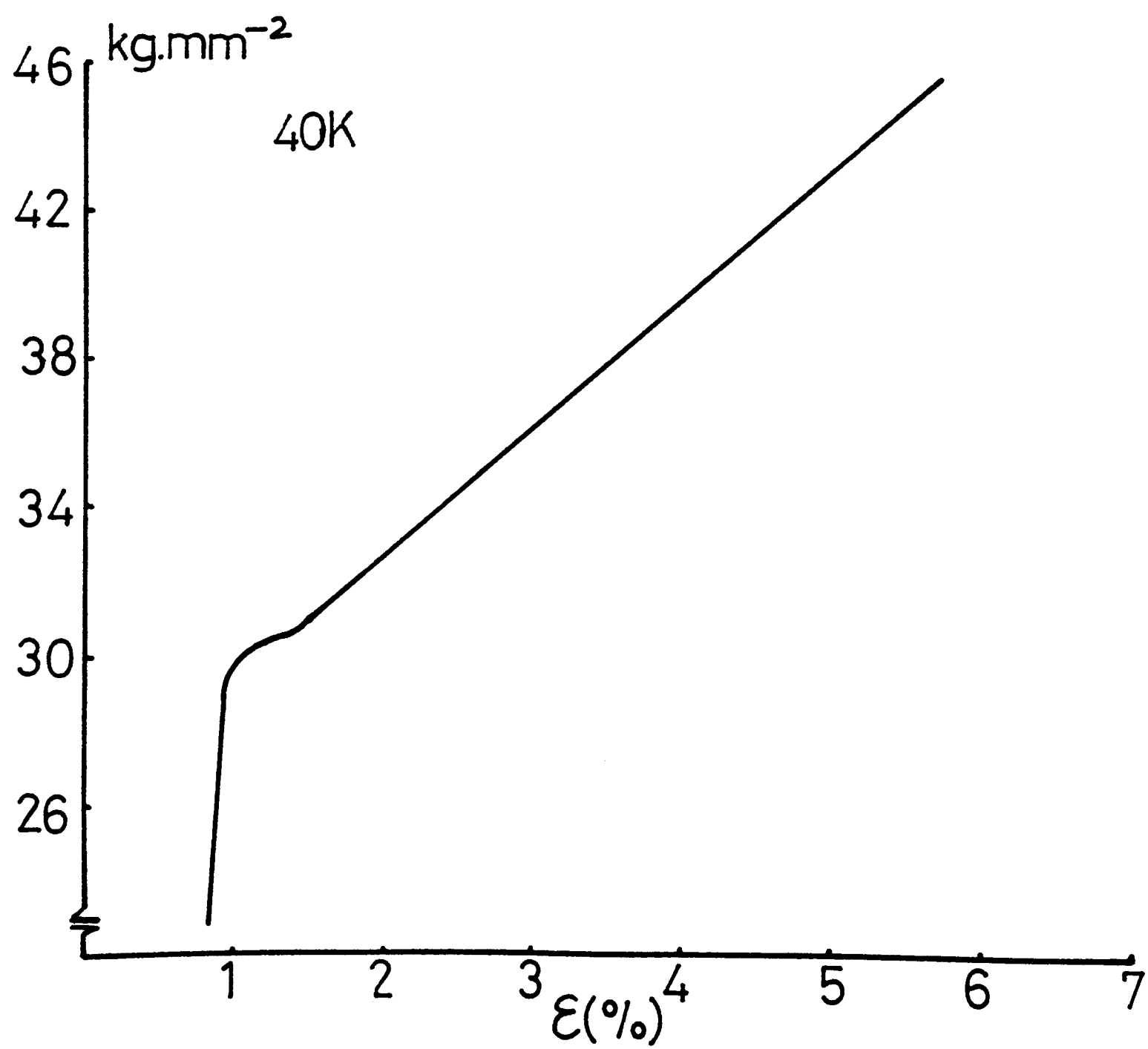
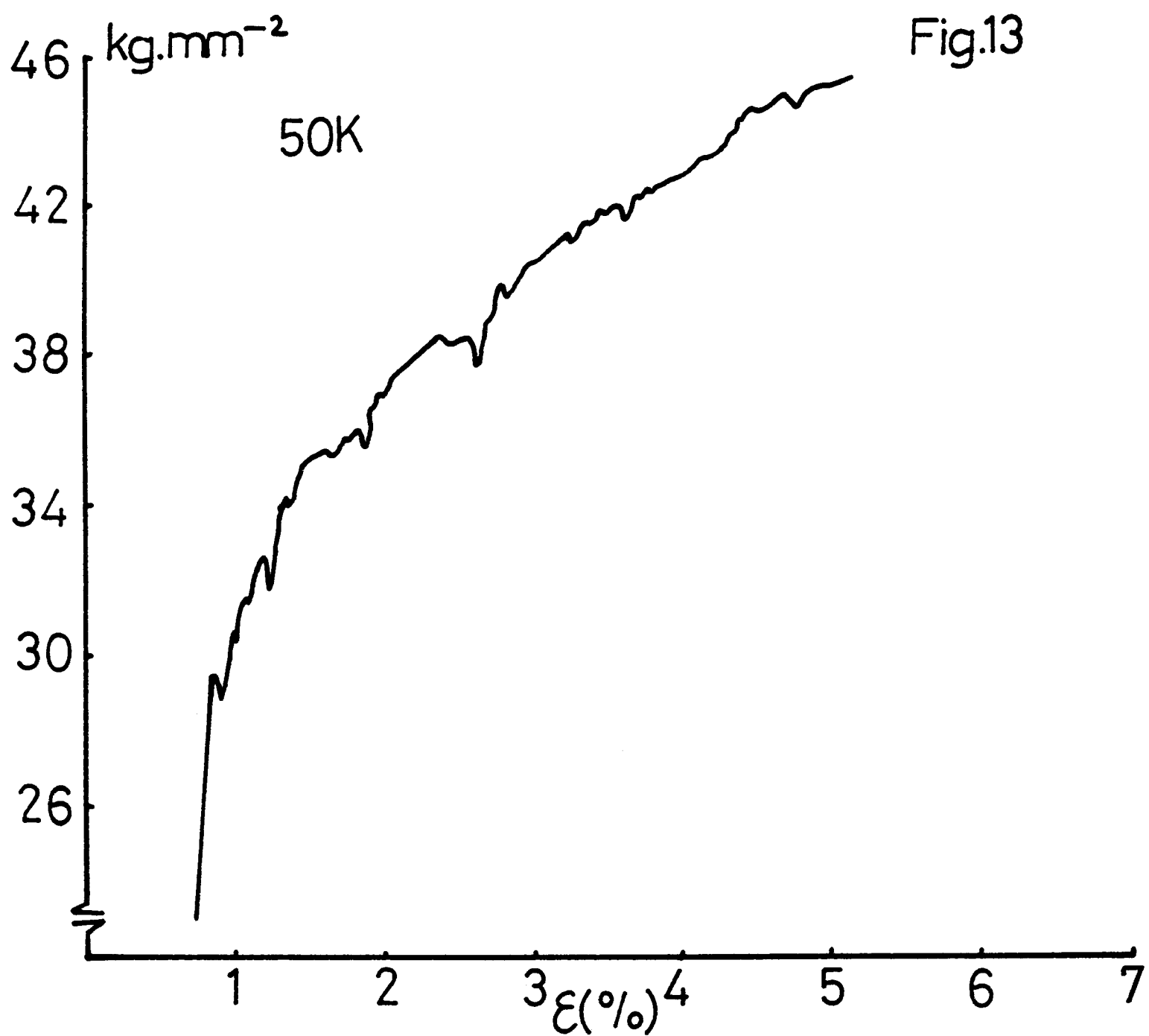


Fig. 14 Axial stress - axial strain for Nb-10 at.%Ta
deformed at 77 K and 60 K in tension.
Specimen 362/2 was prestrained at room temperature
by 0.6% before being deformed at 77 K.

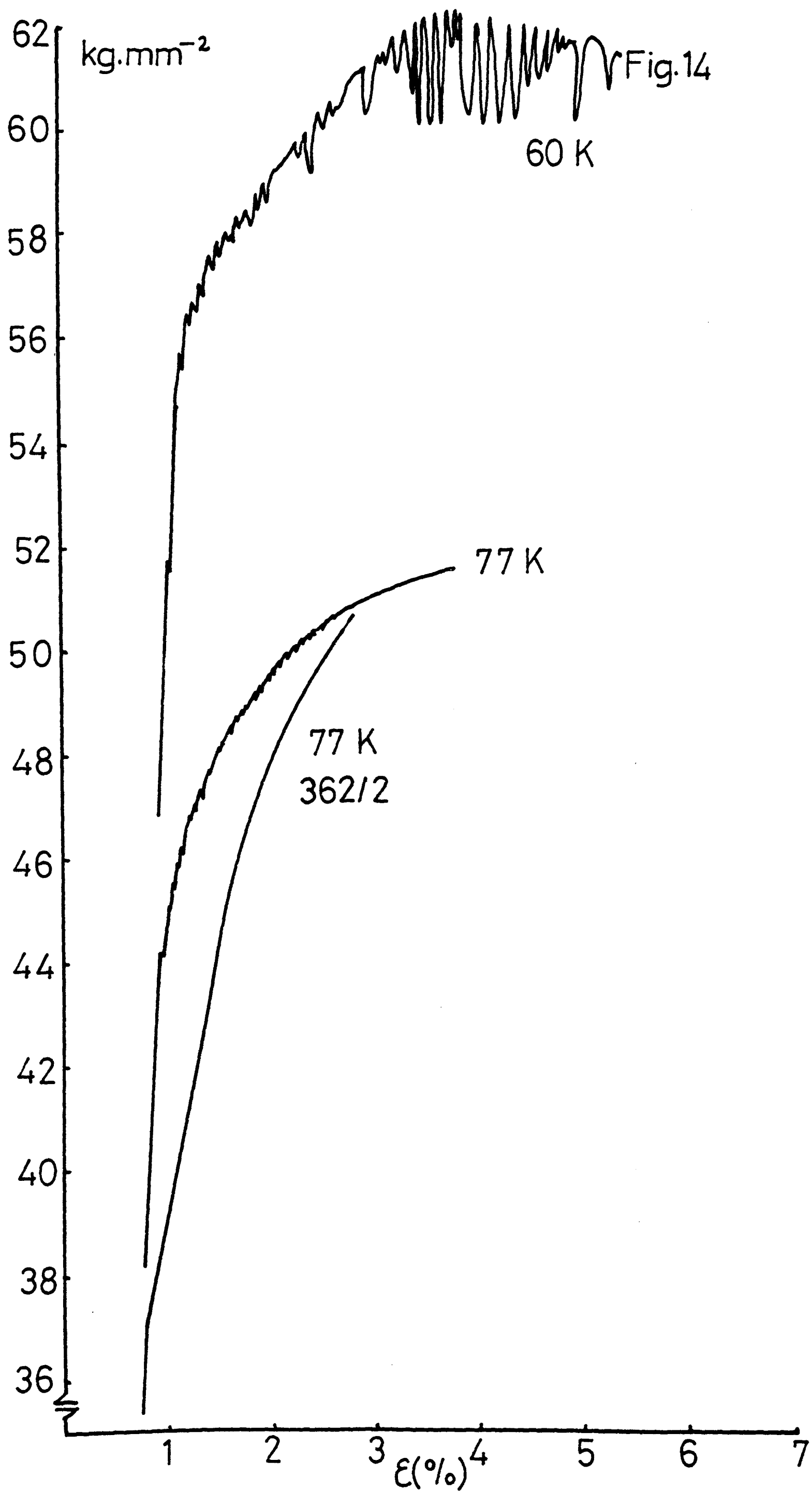


Fig. 15 Axial stress - axial strain for Nb-60 at.%Ta
Tension

Fig. 15

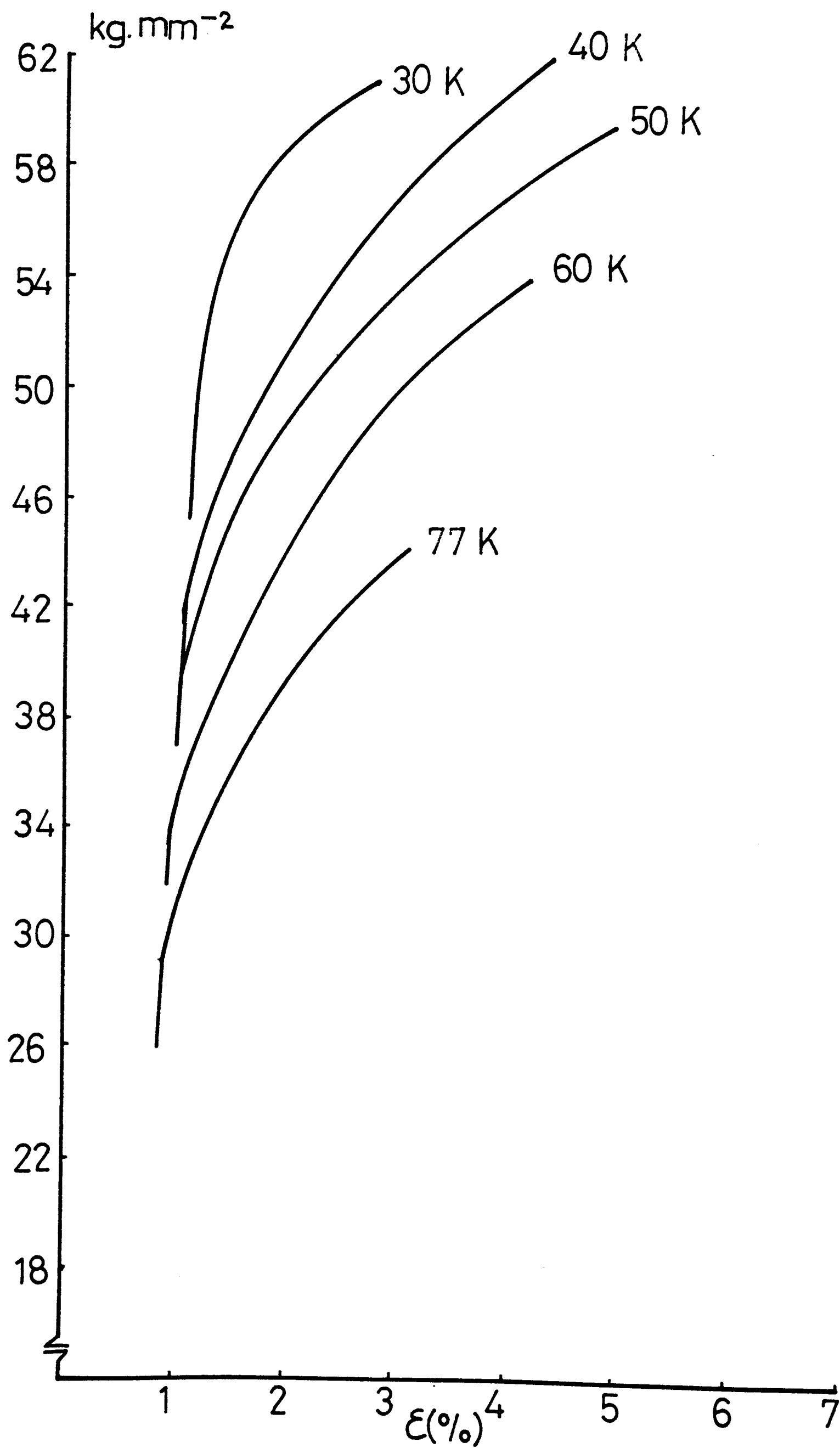


Fig. 16 Axial stress - axial strain for Nb-1 at.%Mo

Tension

Specimen 326/6 was not prestrained before deformation at 77 K

Specimen 370/3 was prestrained 1% at room temperature

Specimen 332/1 was prestrained 1.5% at room temperature

Fig.16

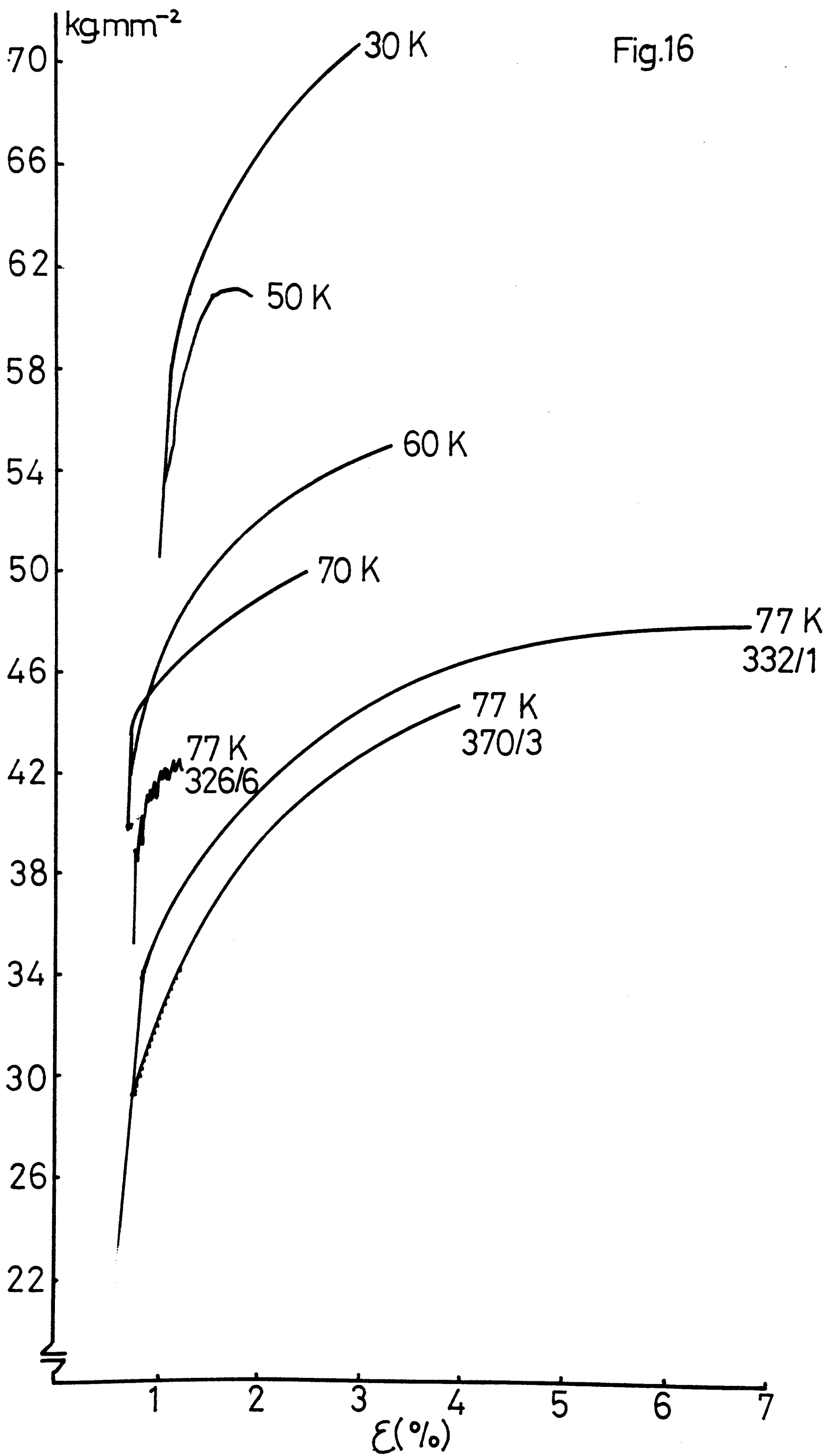


Fig. 17 Axial stress - axial strain for Nb-3 at.%Mo
Tension

Fig.17

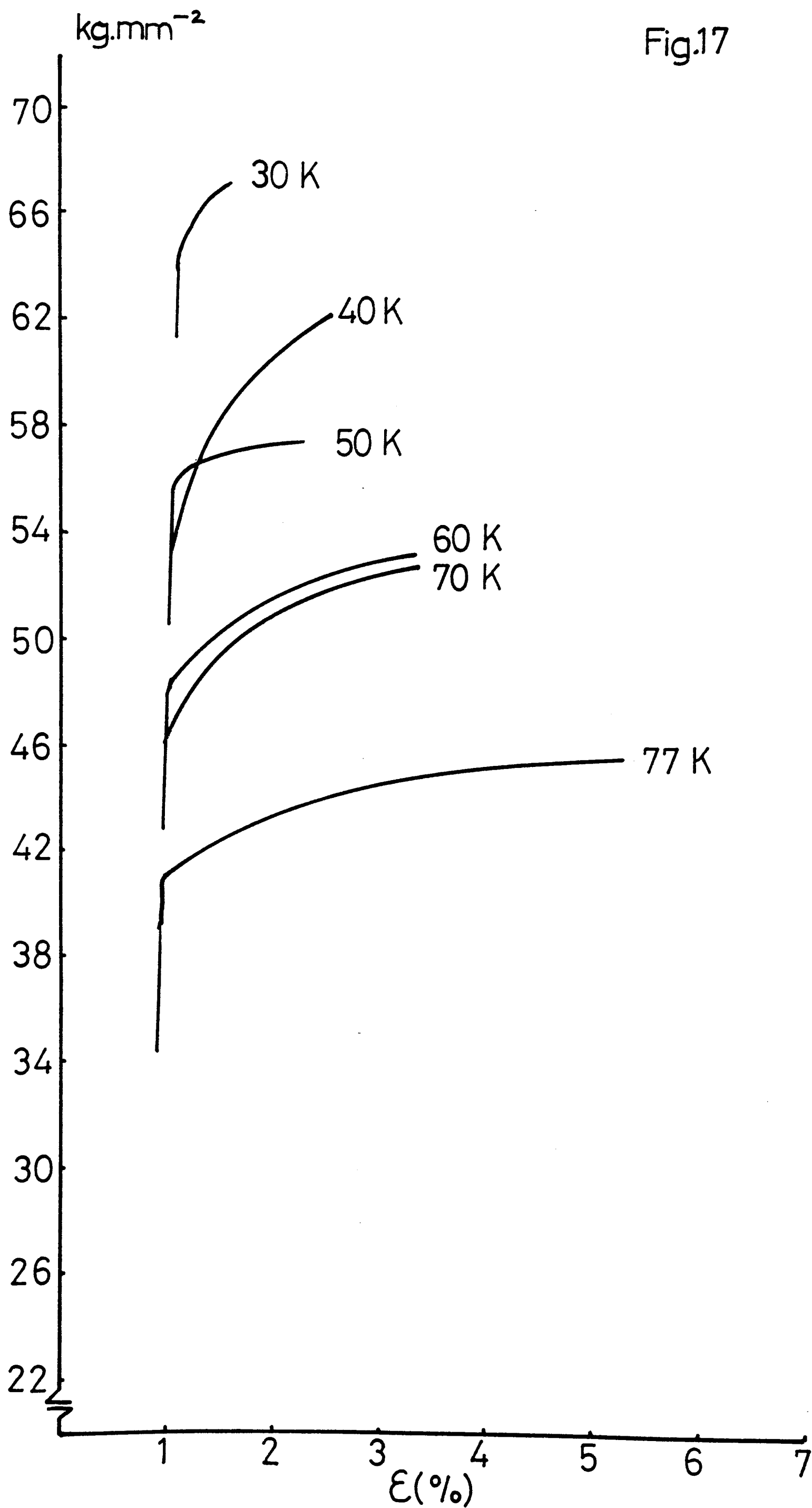


Fig. 18 Axial stress - axial strain for Nb-5 at.%Mo
Tension

Fig.18

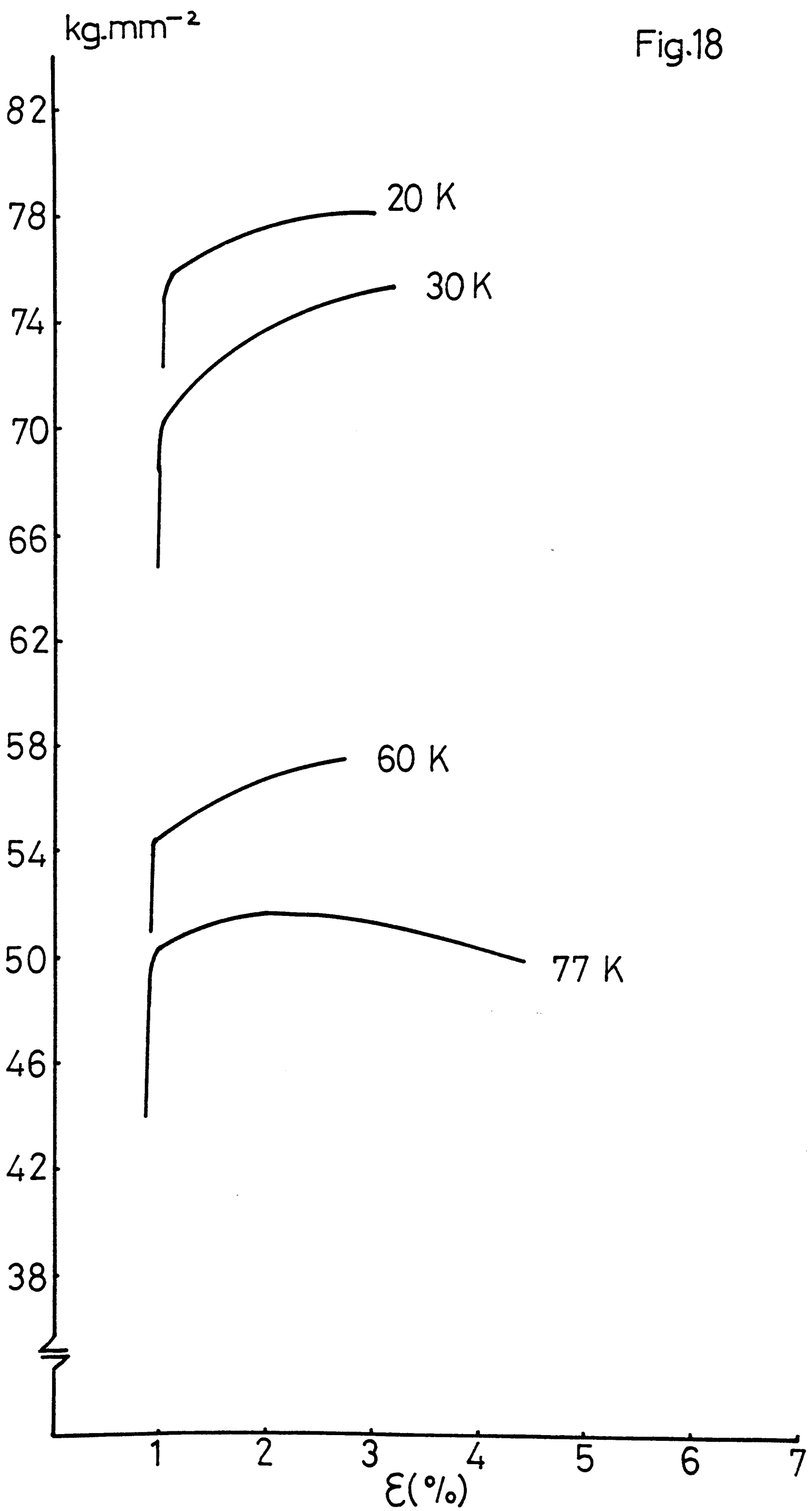


Fig. 19 Shear yield stress - temperature for Nb and Nb-Ta alloys in compression.

The dashed section below 113 K indicates that the yield stresses at 77 K were determined on prestrained specimens.

Fig.19

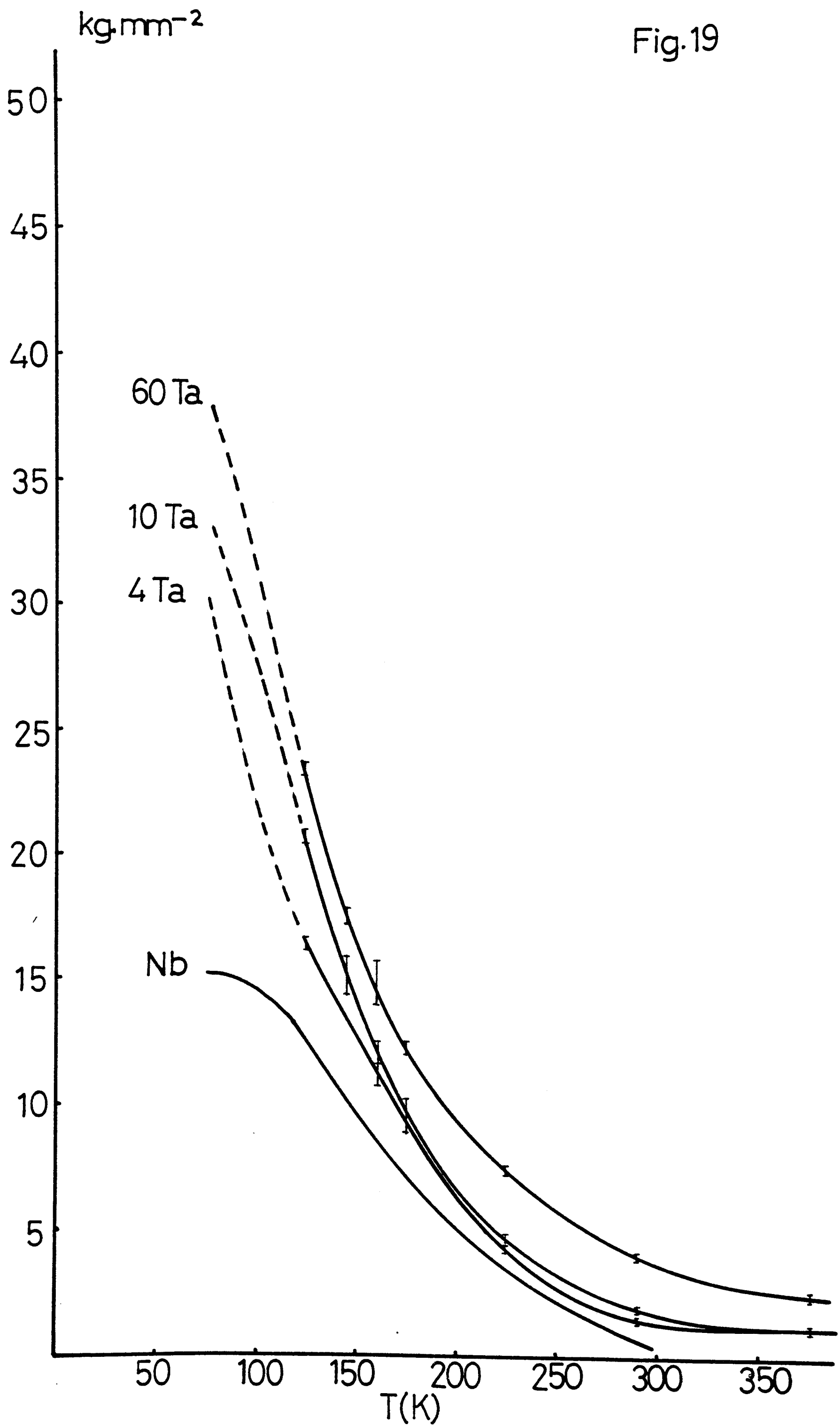


Fig. 20 Shear yield stress - temperature for Nb and Nb-Mo alloys in compression.

The dashed section below 113 K indicates that the yield stresses at 77 K were determined on prestrained specimens.

Fig. 20

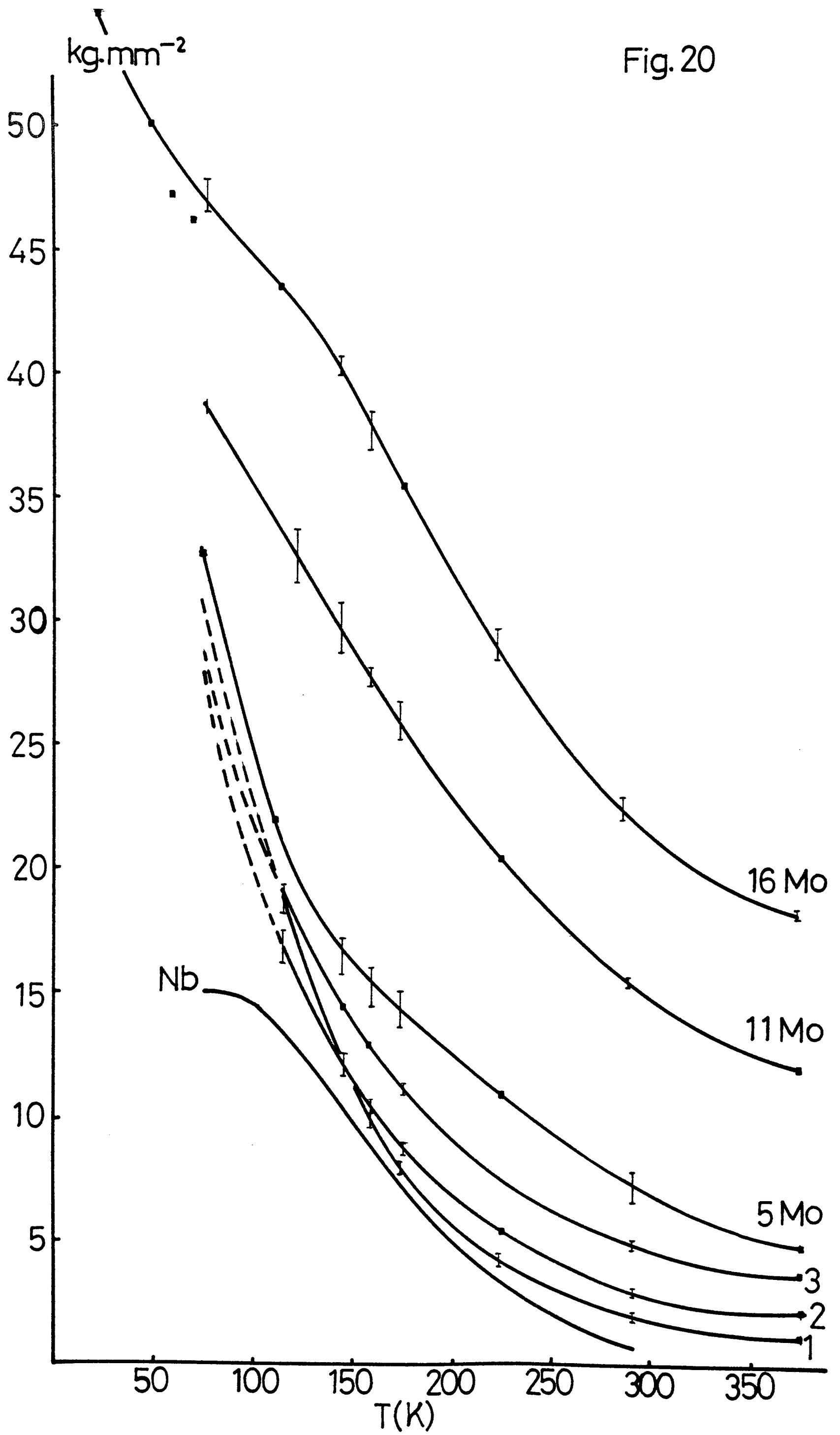


Fig. 21 Tensile axial yield stress - temperature for
Nb and Nb-Ta alloys.

The dashed line indicates that the yield stresses
for the Nb-10 at.%Ta specimens at 30 K ~ 50 K were
determined on prestrained samples.

All the 60 at.%Ta alloy specimens were prestrained.

Fig. 21

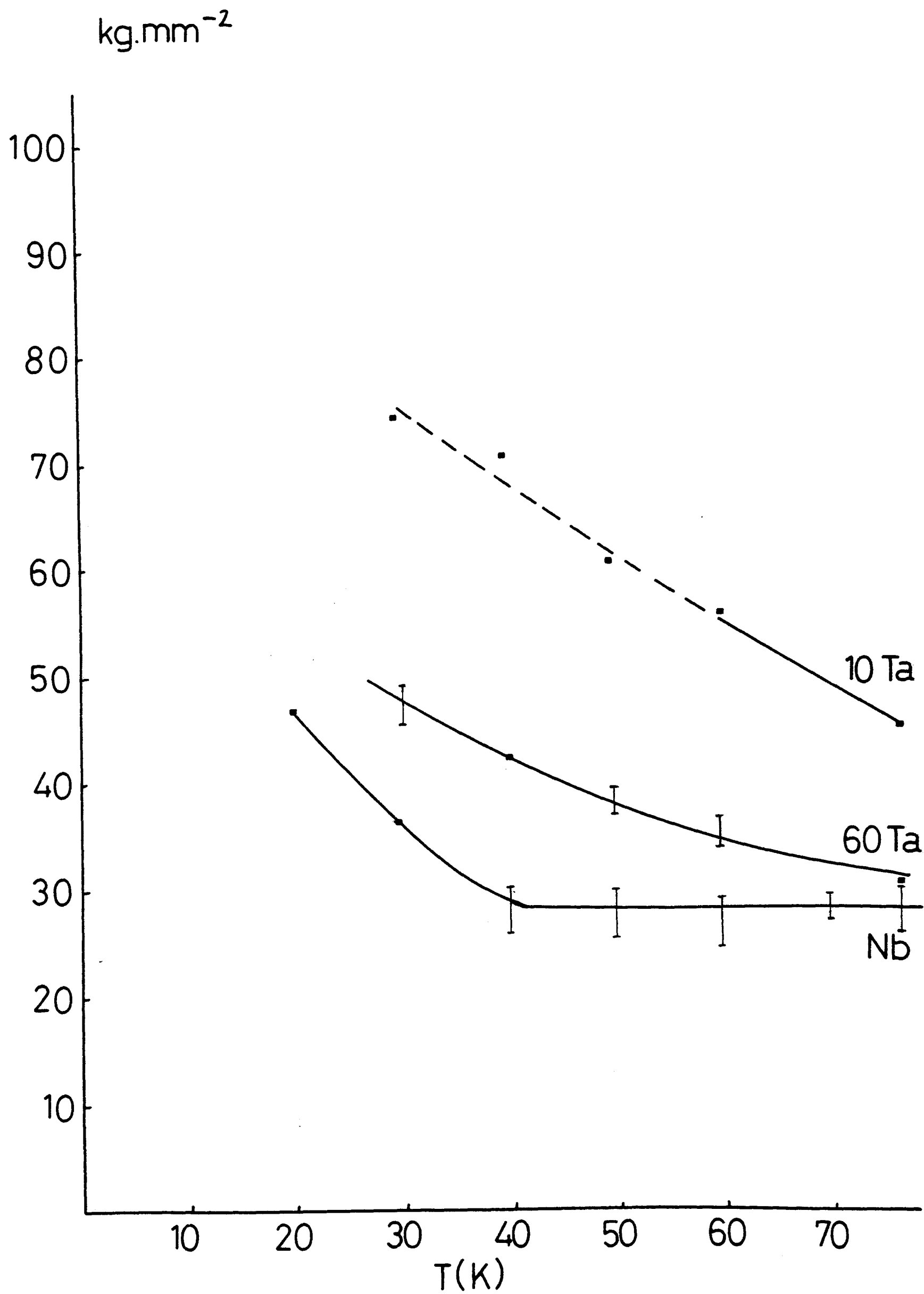


Fig. 22 Tensile axial yield stress - temperature for
Nb and Nb-Mo alloys

Fig. 22

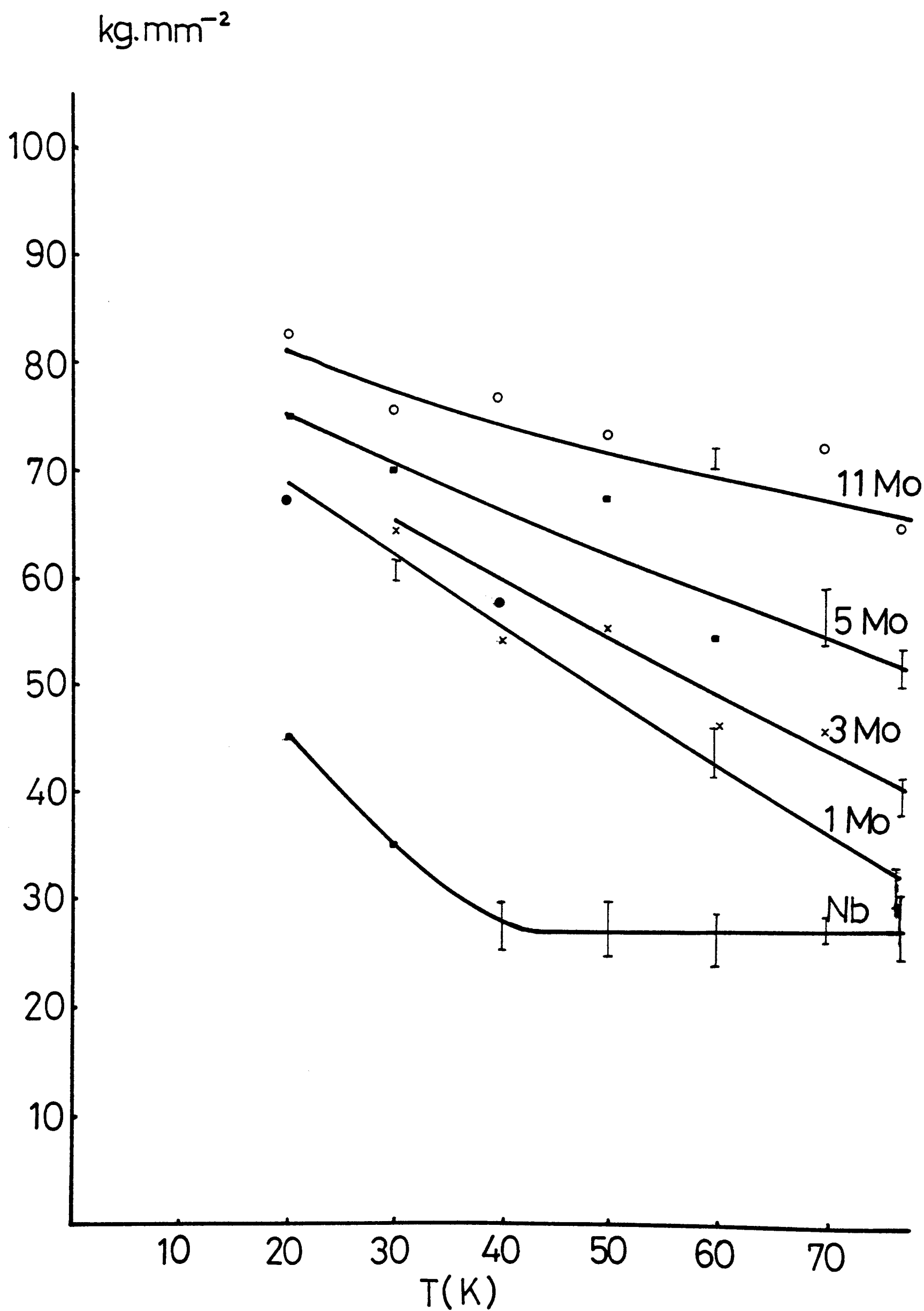


Fig. 23 Thermal shear stress - temperature in compression
for Nb and Nb-Ta alloys

Fig. 23

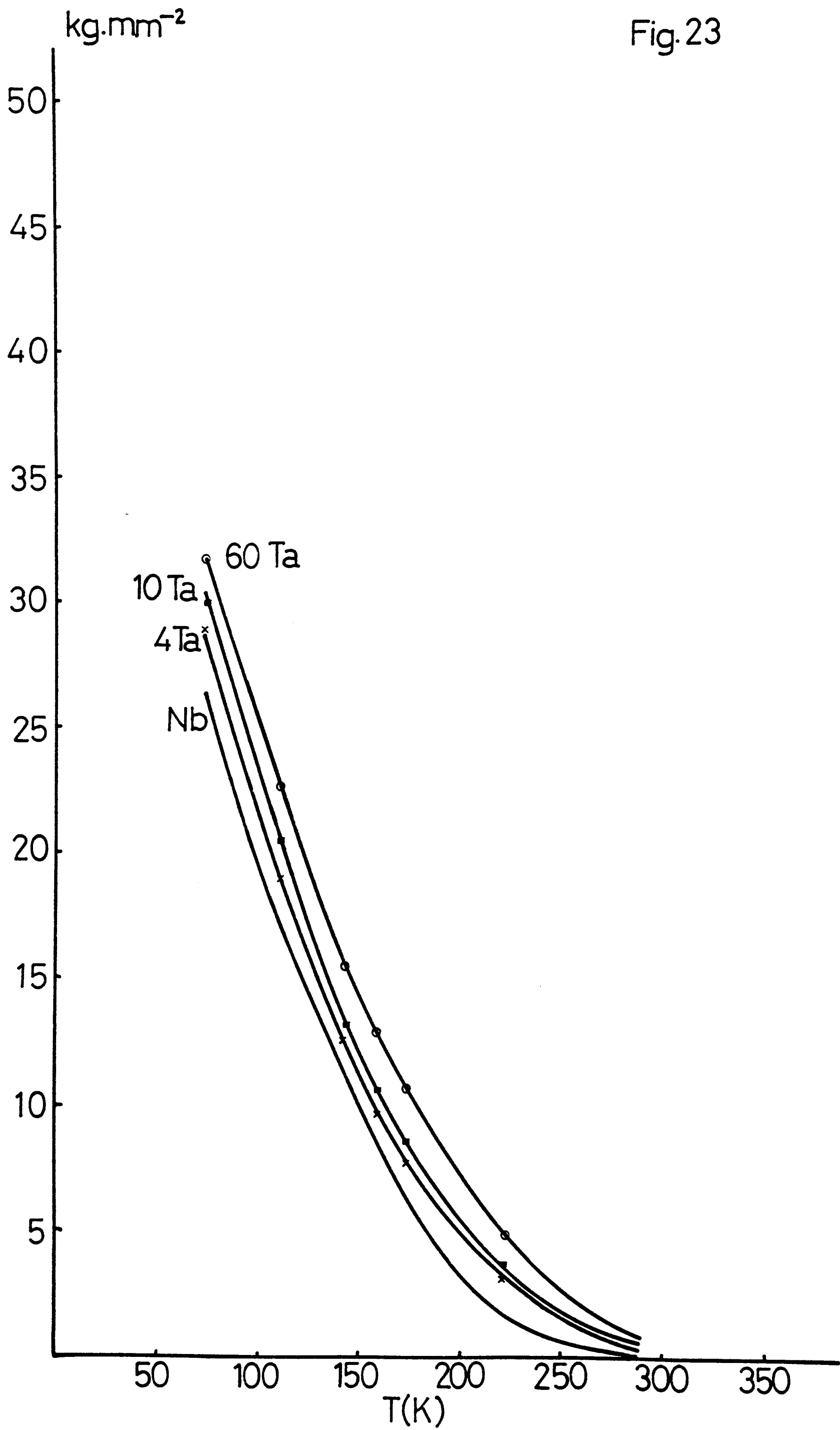


Fig. 24 The difference between the thermal shear stress
of the Nb-Ta alloys and that for Nb.

$$\tau^* (\text{alloy}) - \tau^* (\text{Nb})$$

Fig. 24

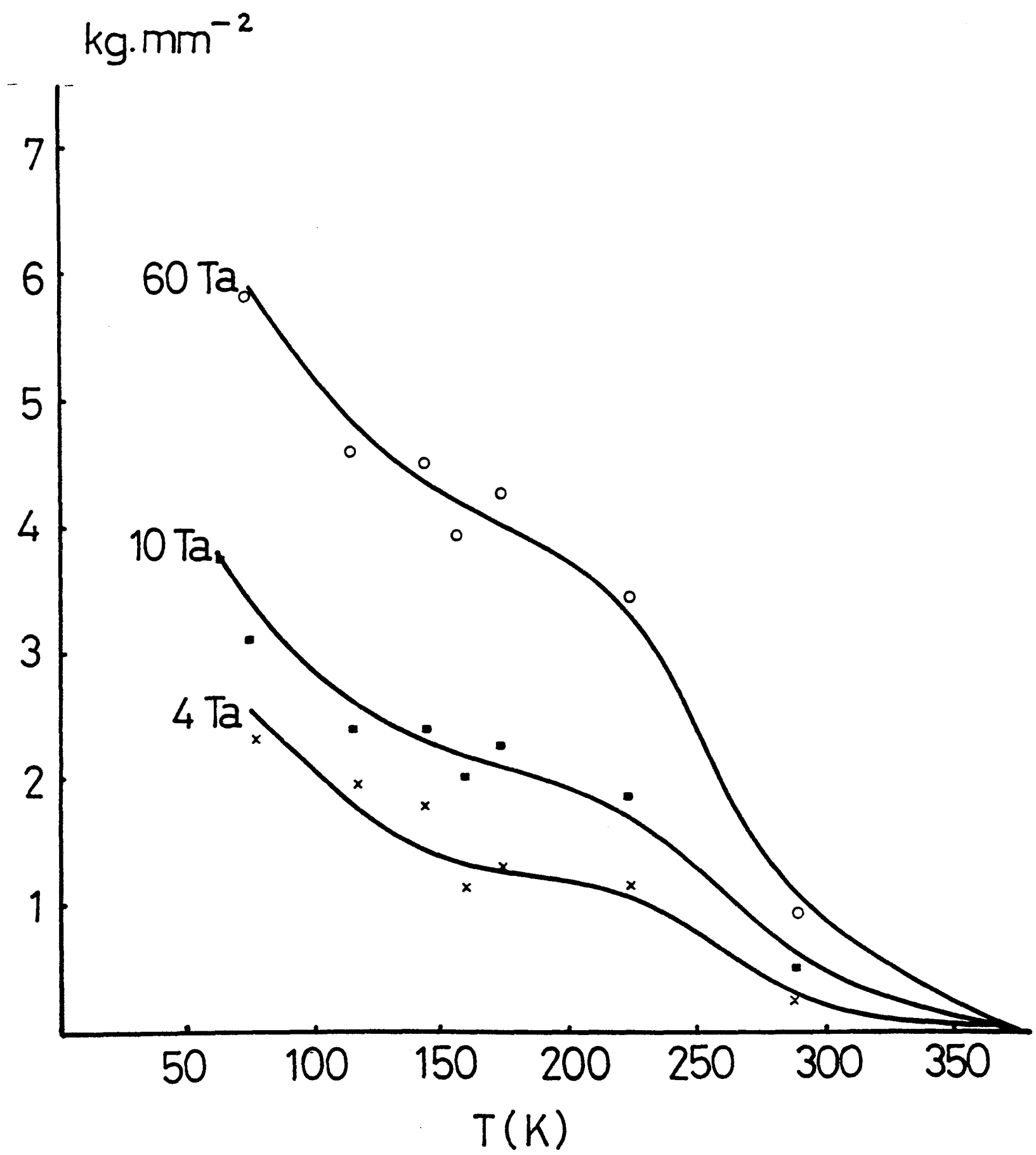


Fig. 25 The thermal shear stress for the Nb-Mo alloys
in compression.

The curve for the 2 at.%Mo alloy has been omitted
for clarity.

The curves for the 1 and 3 at.%Mo alloys have been
omitted below 113 K for clarity.

Fig.25

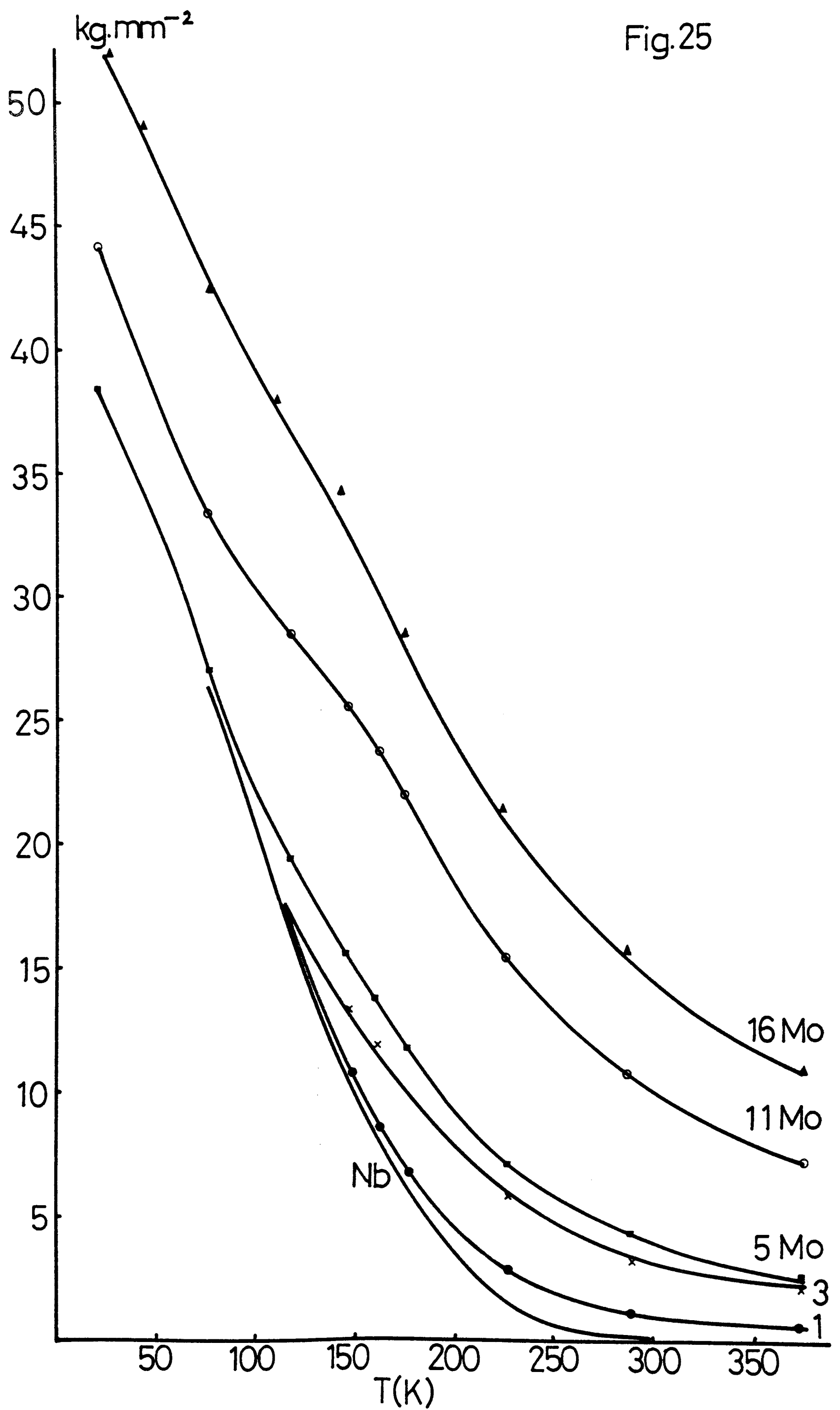


Fig. 26 The difference between the thermal shear stress
for the Nb-Mo alloys and that for Nb.

$$\tau^* (\text{alloy}) - \tau^* (\text{Nb})$$

Fig. 26

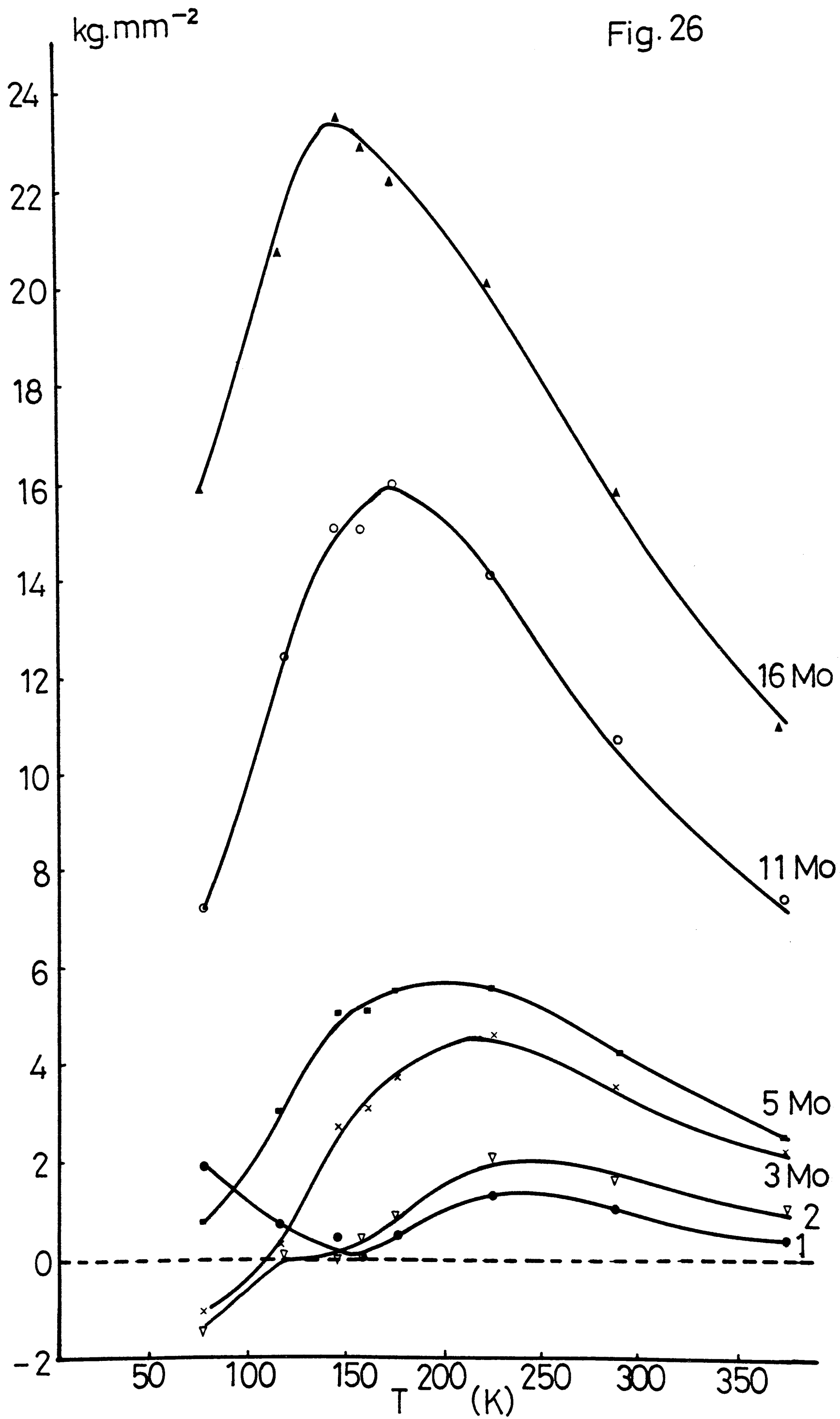


Fig. 27 Thermal stress (axial) - temperature for Nb
and the alloys in tension

Fig. 27

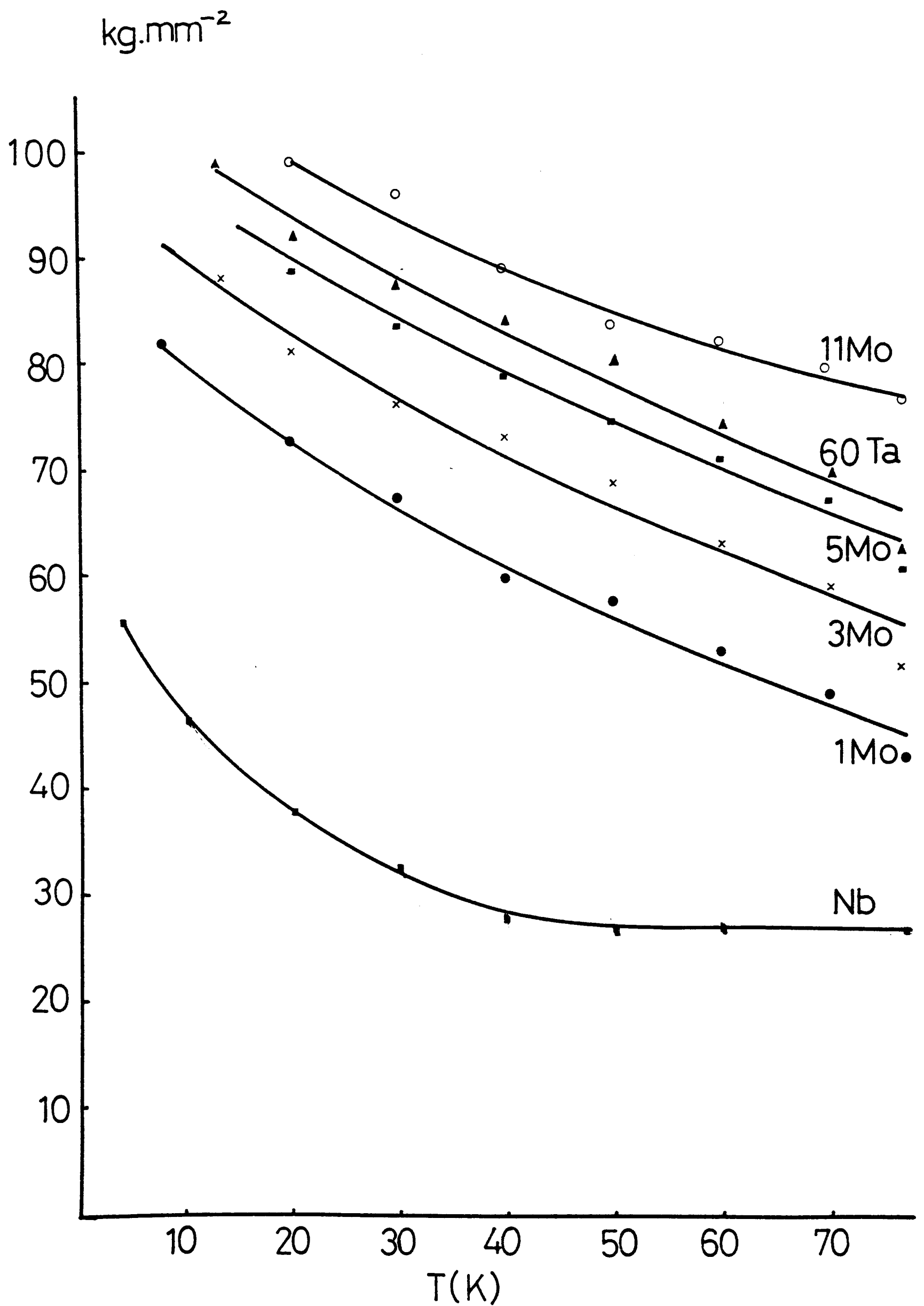


Fig. 28 (a) Definition of terms for a "normal" strain rate change.

(b) Definition of terms when overshoots are present.

$\Delta\tau_{\rho}^{\uparrow}$: maximum stress difference for an increase in strain rate.

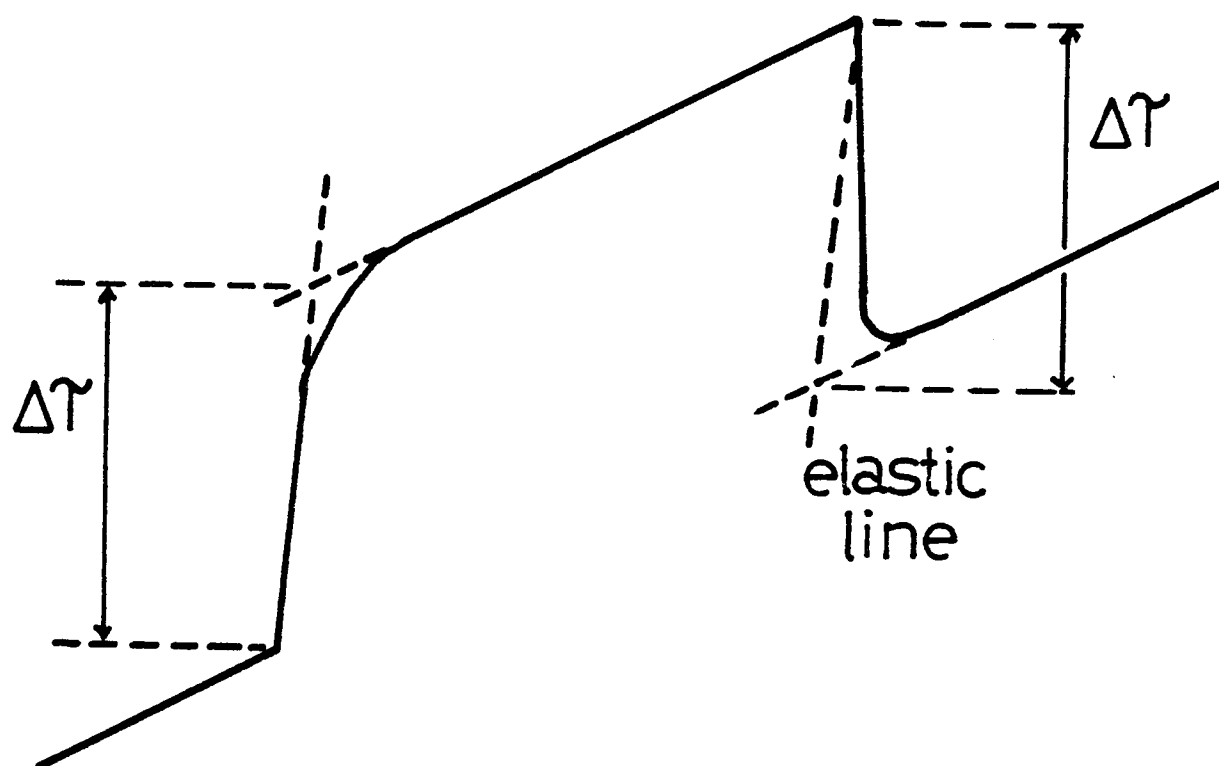
$\Delta\tau_s^{\uparrow}$: "steady state" stress change for an increase in strain rate.

$\Delta\tau_p^{\downarrow}$: maximum stress difference for a decrease in strain rate.

$\Delta\tau_s^{\downarrow}$: "steady state" stress change for a decrease in strain rate.

Fig. 28

(a)



(b)

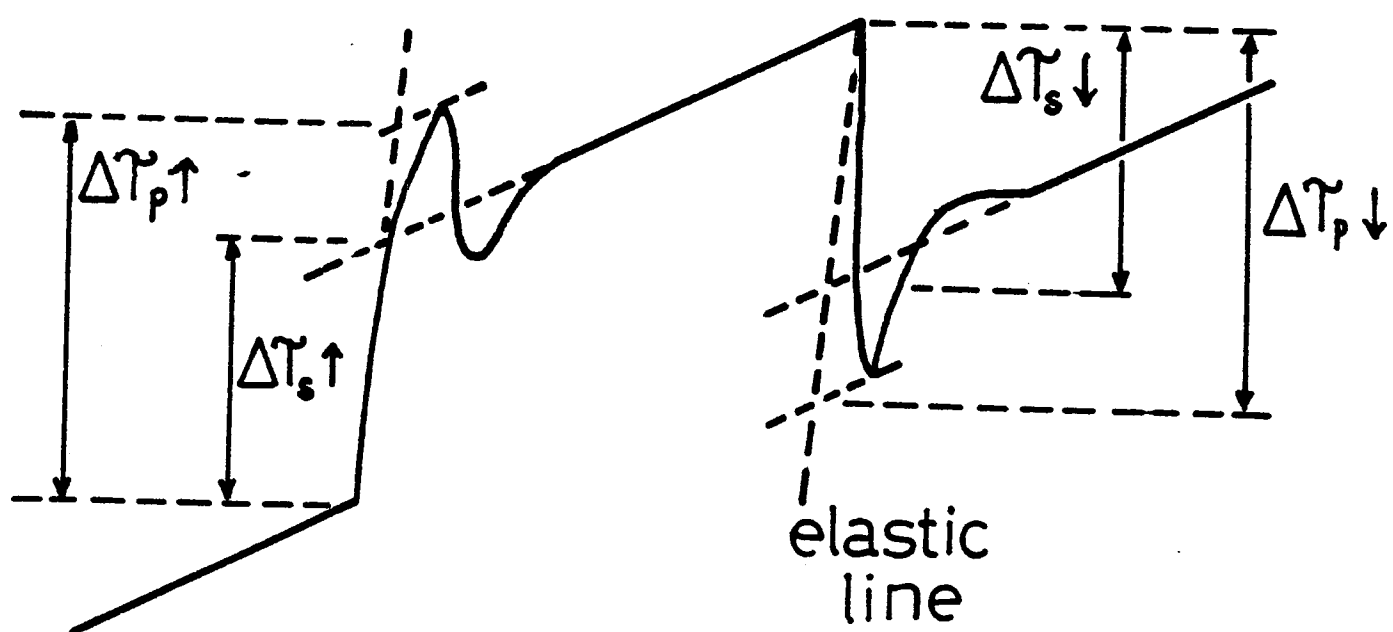


Fig. 29 The strain rate sensitivity, $\frac{\Delta \tau_{\sim}}{\Delta \ln \dot{\gamma}}$,
for Nb and the Nb-Ta alloys.

Fig. 29

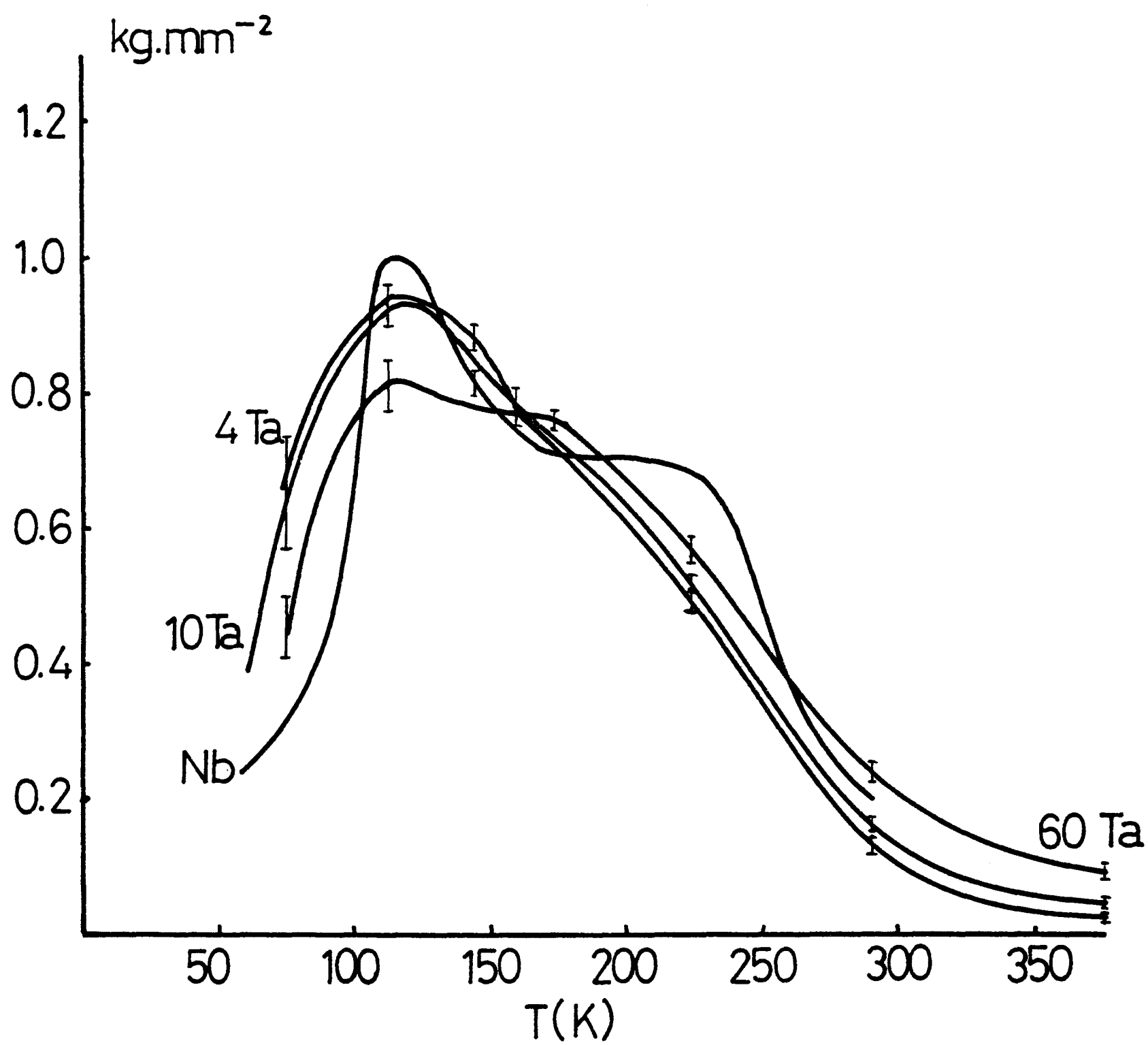


Fig. 30 The strain rate sensitivity, $\frac{\Delta \tau}{\Delta \ln \dot{\gamma}}$, for Nb and the Nb-Mo alloys deformed in compression.

If overshoots were present, then the strain rate sensitivity was calculated from $\Delta \tau_p$ † (see Fig.28).

If the strain rate sensitivity increased with strain, then the value at 5% strain was used in the calculation.

Fig. 30

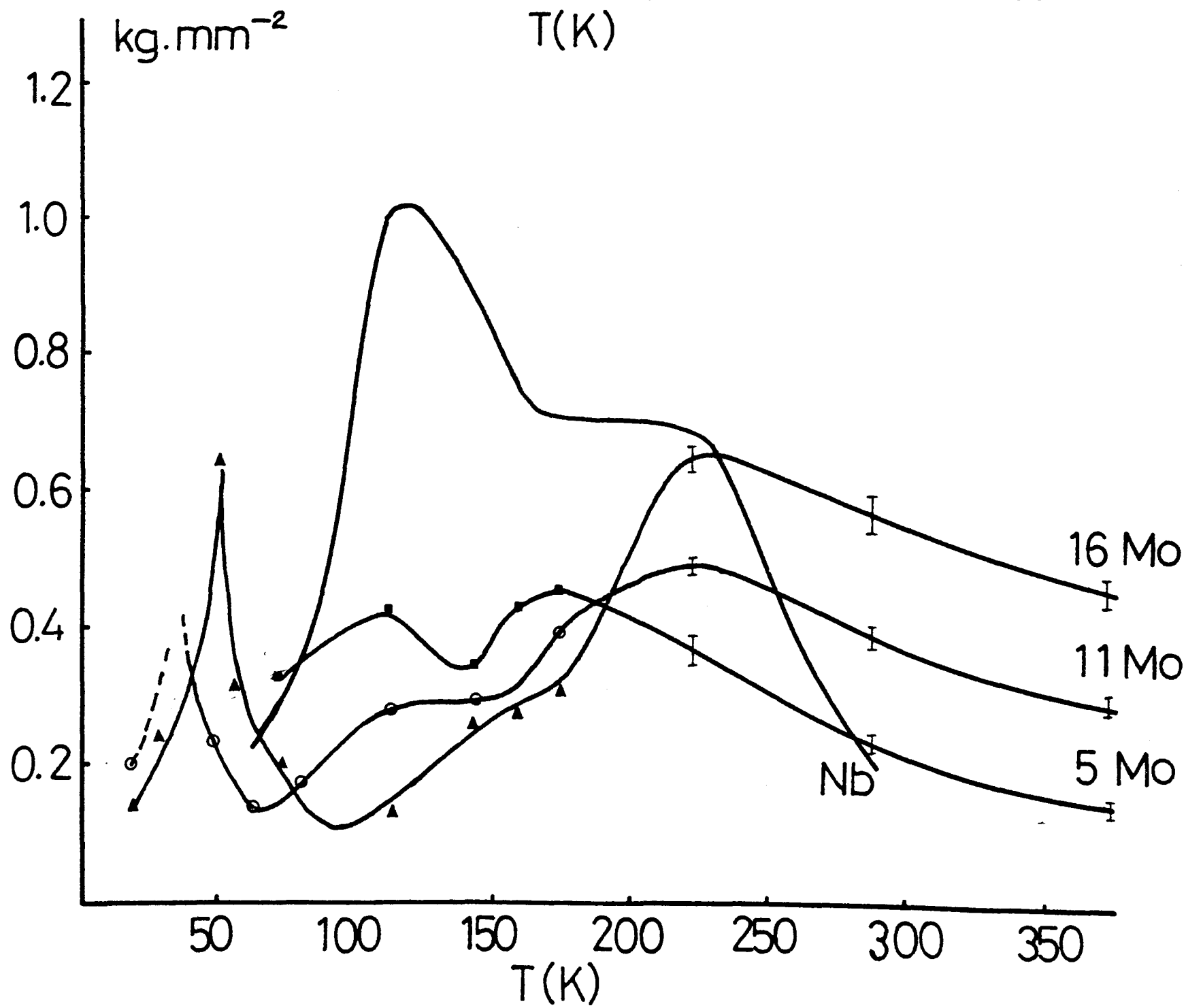
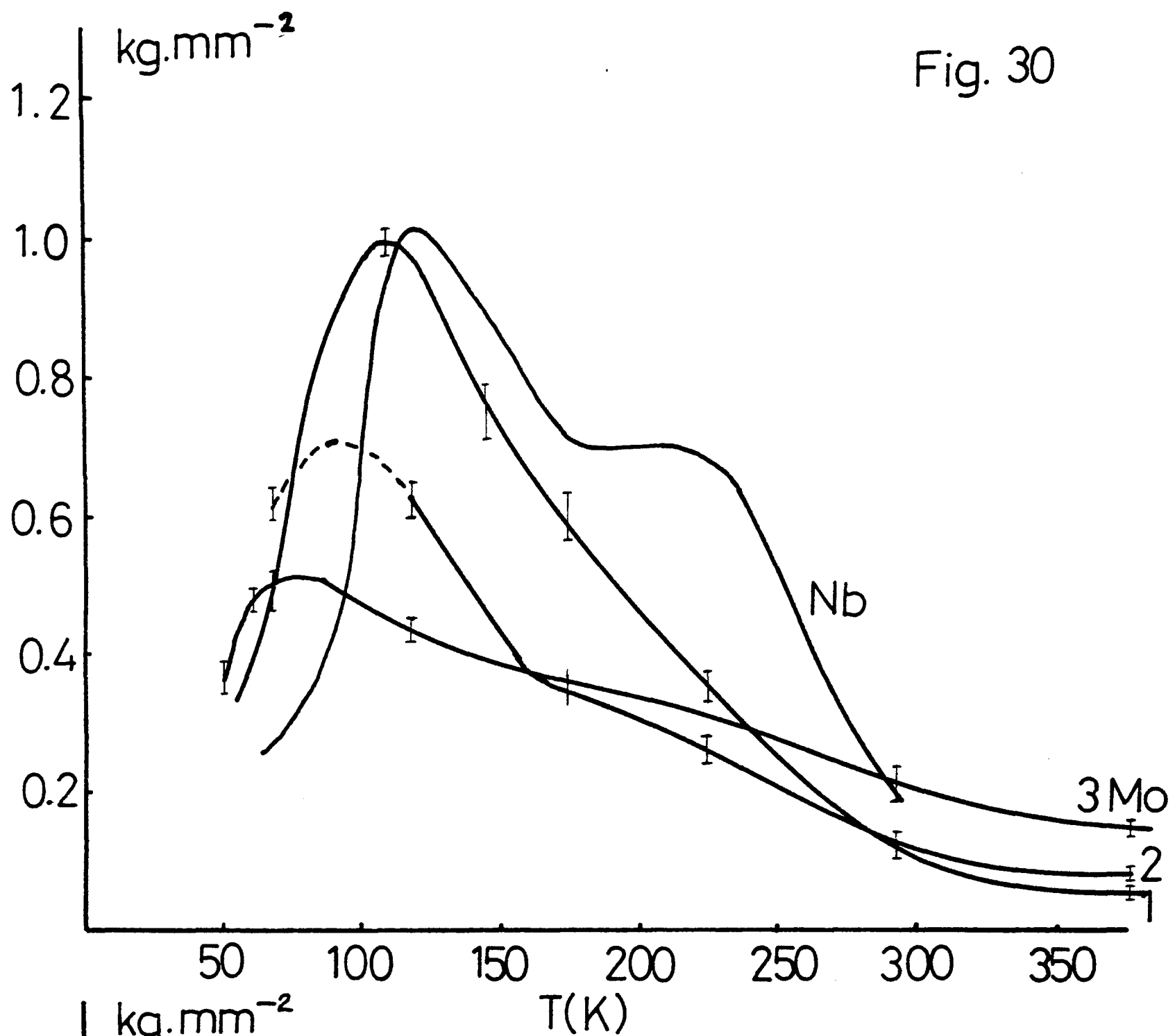


Fig. 31 Strain rate sensitivity for Nb-11 at.%Mo in compression at two strains.

- (a) The dash line is the strain rate sensitivity at 20% strain.
- (b) The solid line is the average strain rate sensitivity, or that at 5% strain when such a dependence appears.

Fig. 32 Strain rate sensitivity for Nb-16 at.%Mo in compression.

- (a) The solid line represents the strain rate sensitivity at 4% strain (when a strain dependence occurs) for increases in strain rate. If overshoots were present, $\Delta\tau_p$ was used in the calculation.
- (b) The dashed line represents the steady state strain rate sensitivity, calculated from $\Delta\tau_s$, at 4% strain when overshoots were present.

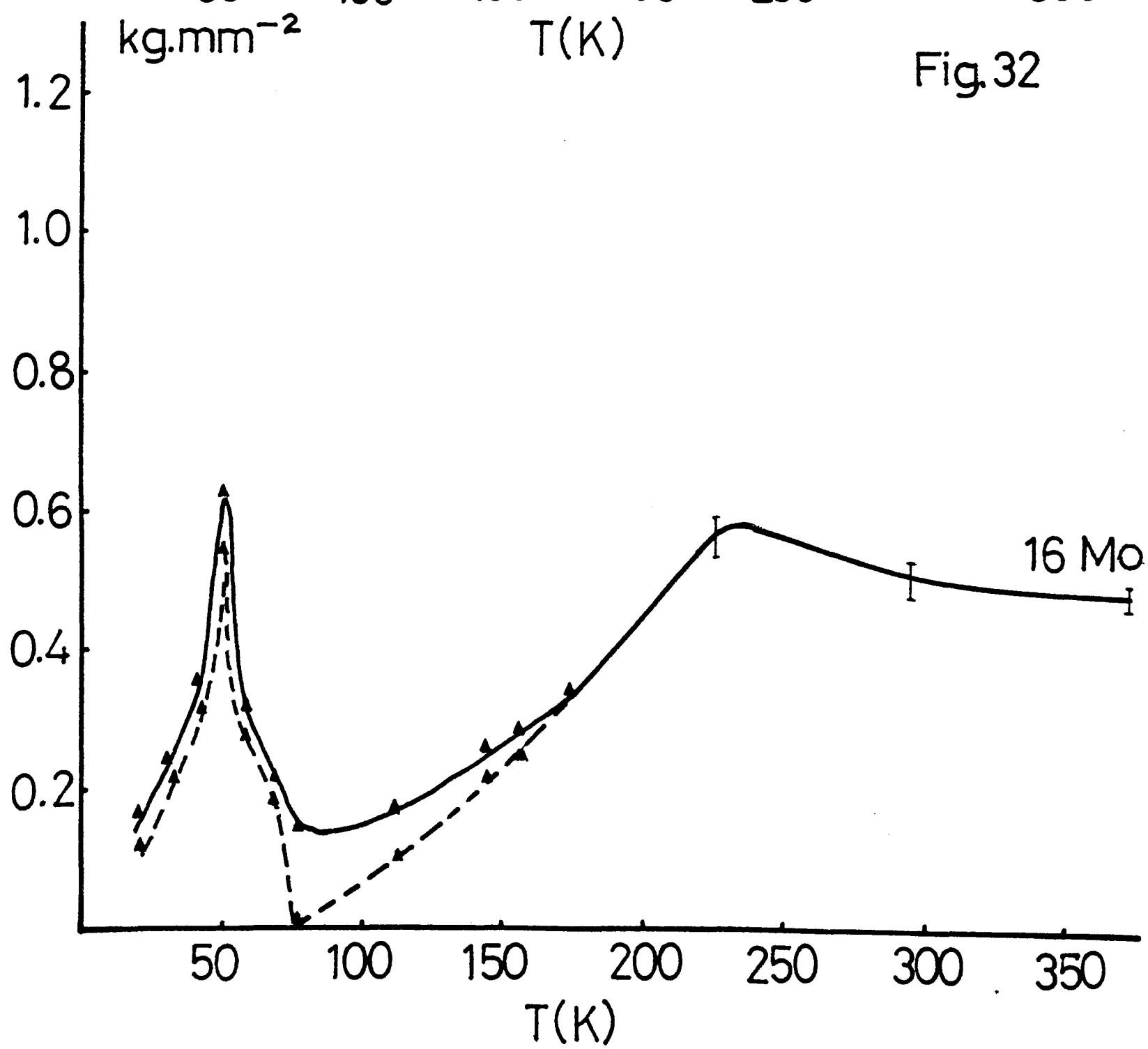
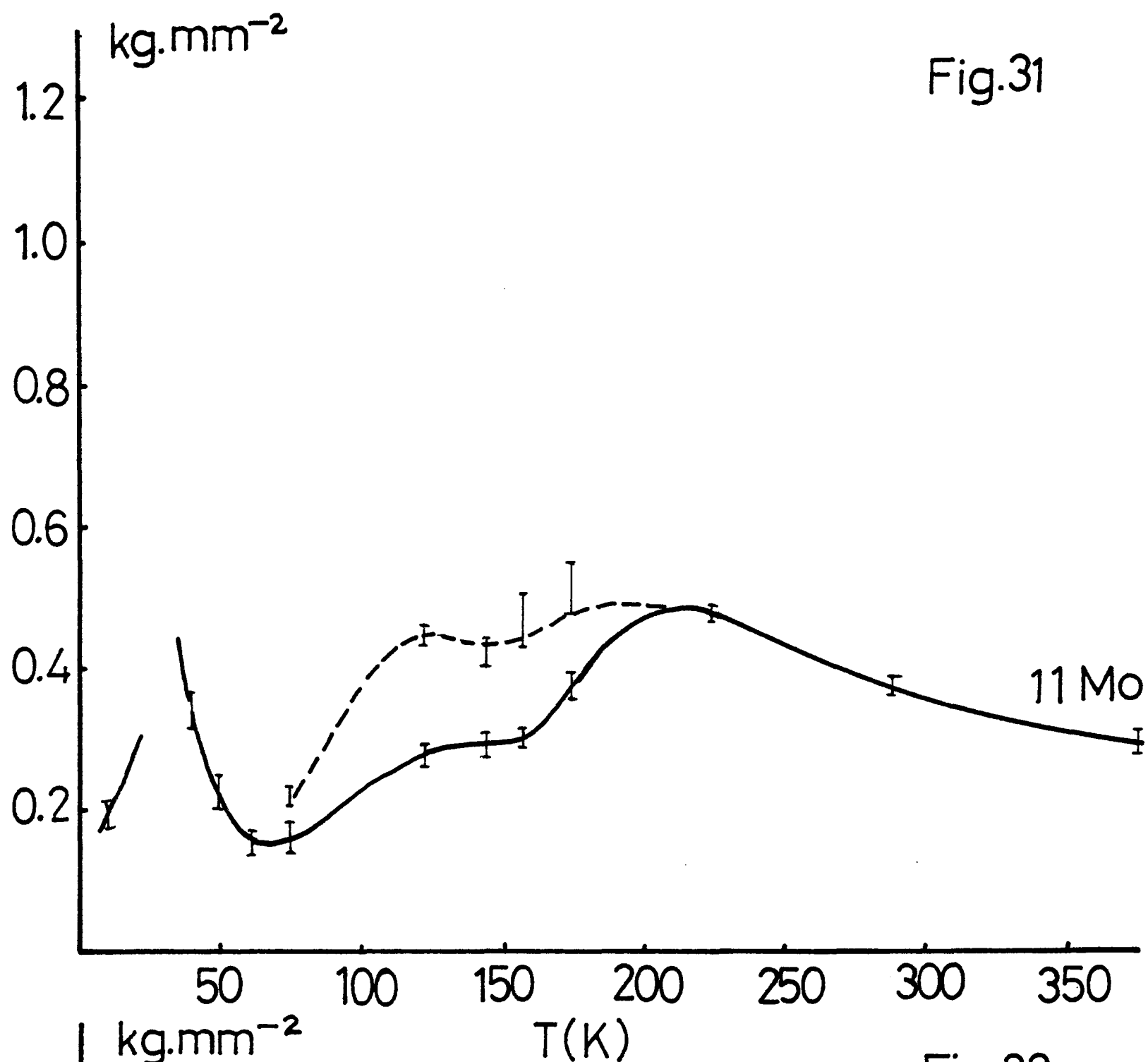


Fig. 33 Strain rate sensitivity for Nb-16 at.%Mo in compression.

curve (a): derived from $\Delta\tau_p \uparrow$

curve (b): derived from $\Delta\tau_s \uparrow$. A strong dependence on strain was observed at 60 K and 70 K.

- (b) 1 - 22% strain
- (b) 2 - 15% strain
- (b) 3 - 12% strain
- (b) 4 - 8% strain
- (b) 5 - 5% strain

Fig. 33

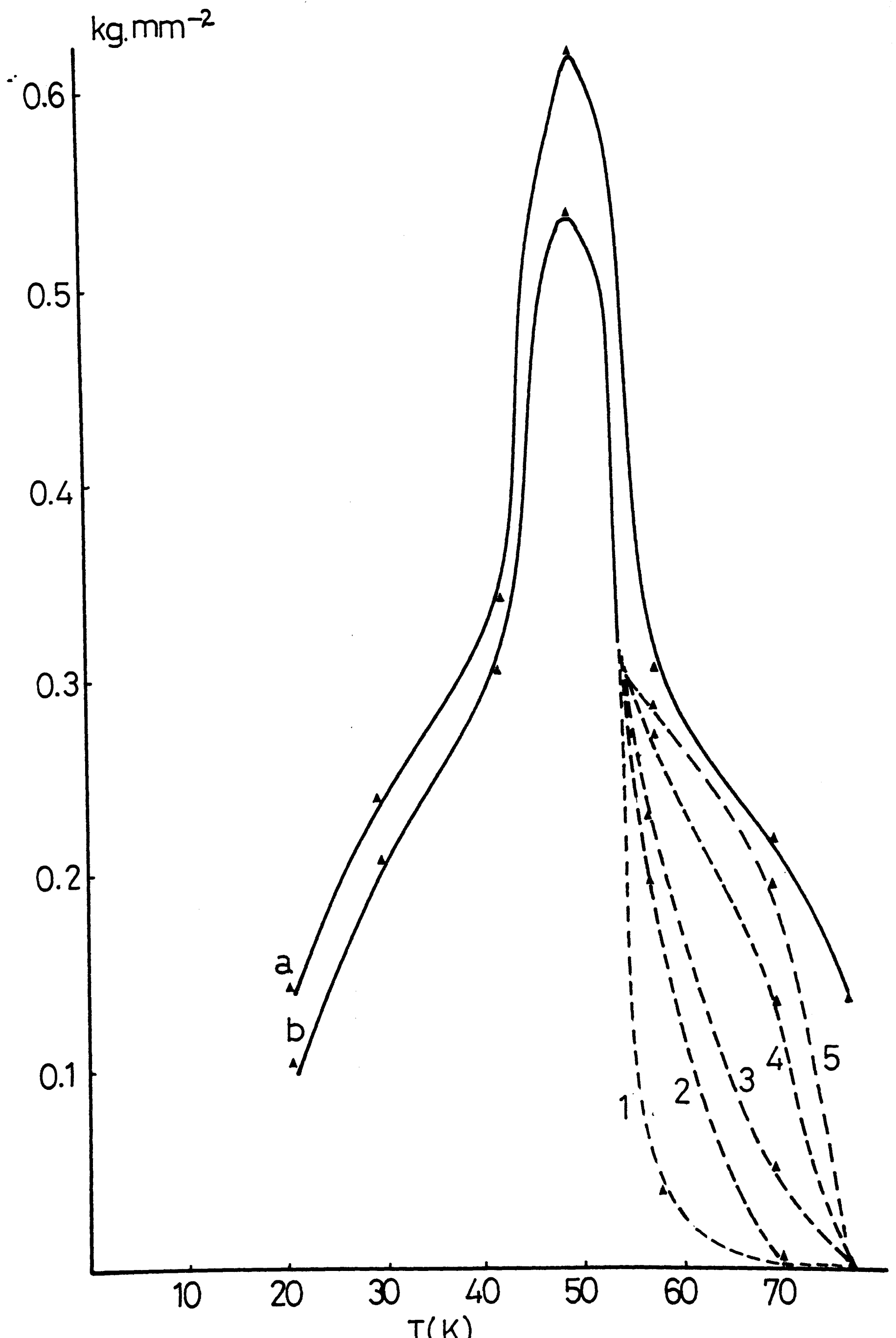


Fig. 34 Strain rate sensitivity for Nb-16 at.%Mo in compression.

curve (a): derived from $\Delta\tau_p \downarrow$

curve (b): derived from $\Delta\tau_s \uparrow$. A strong dependence on strain was observed at 60 K and 70 K.

- (b) 1 - 26% strain
- (b) 2 - 20% strain
- (b) 3 - 14% strain
- (b) 4 - 9% strain
- (b) 5 - 6% strain

Fig. 34

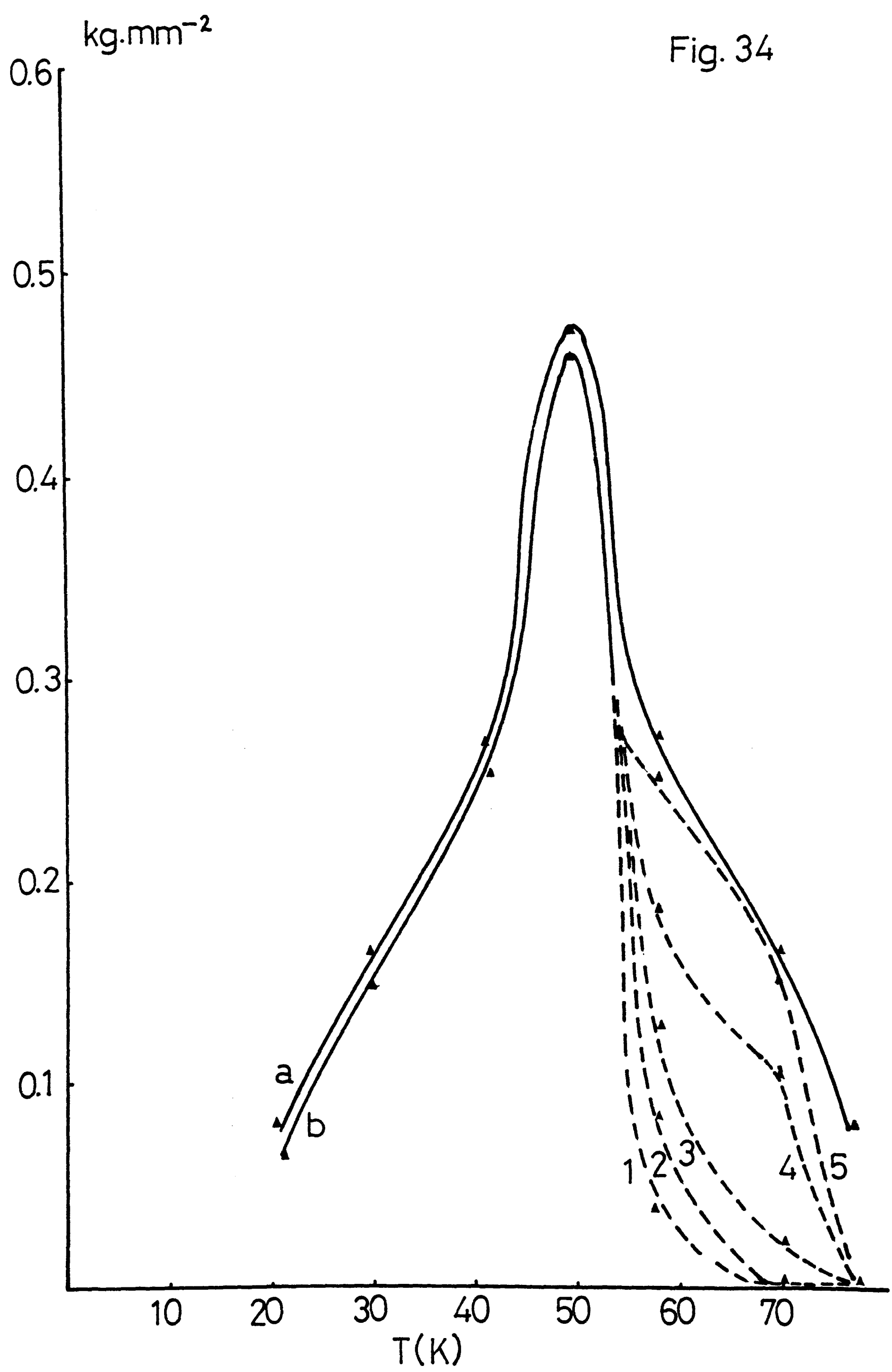
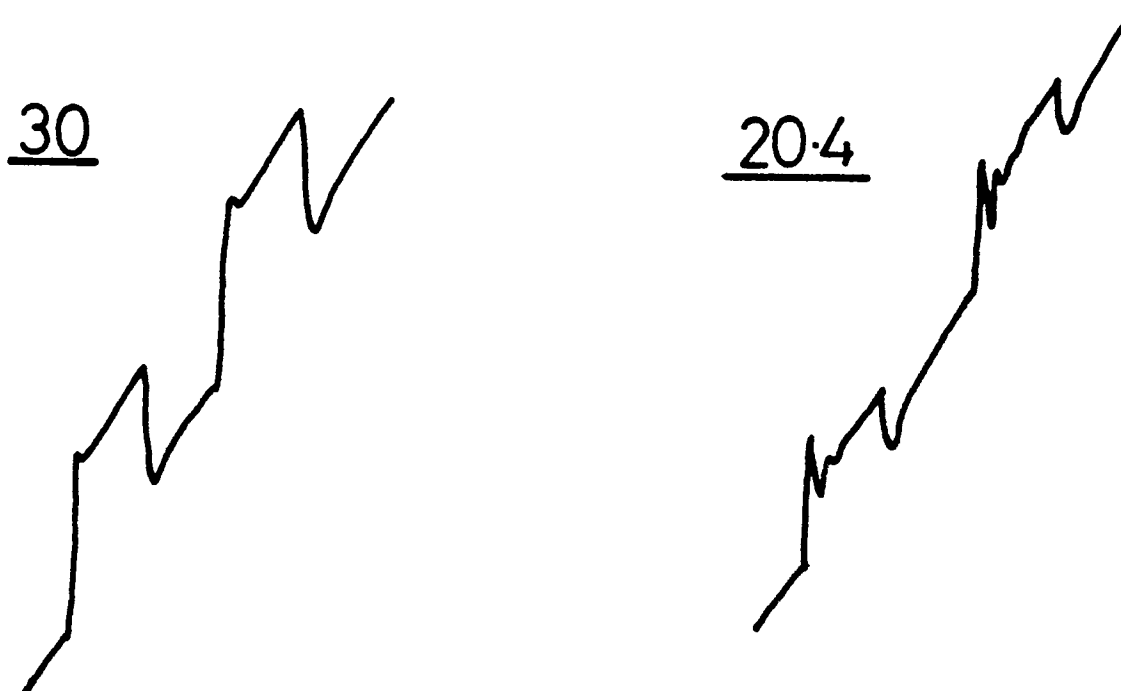
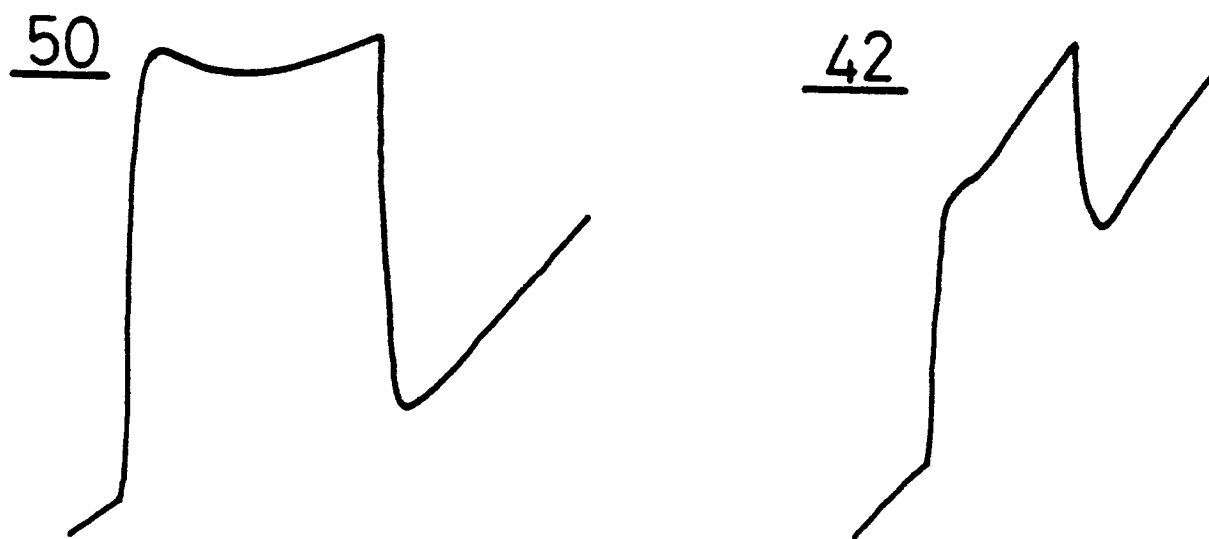
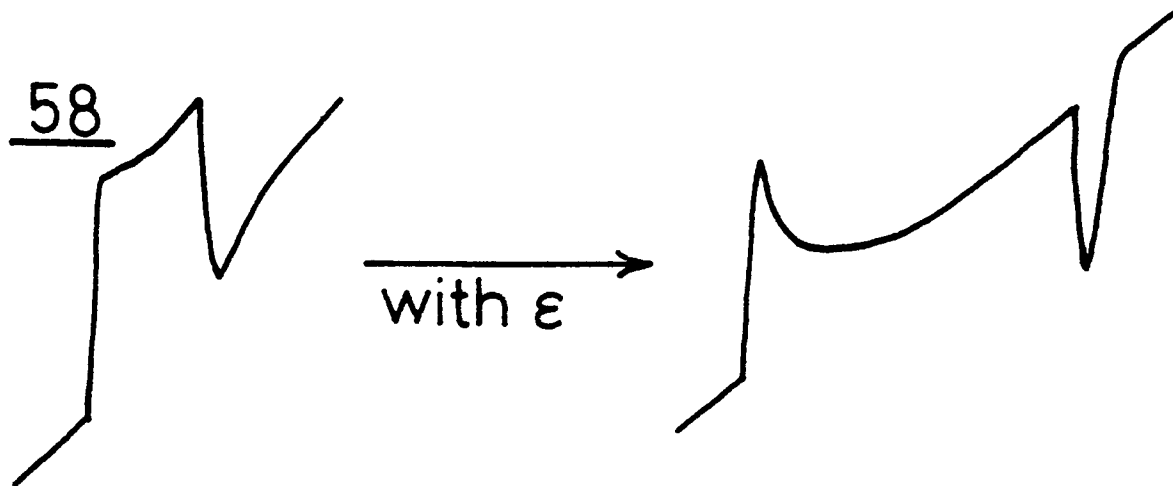
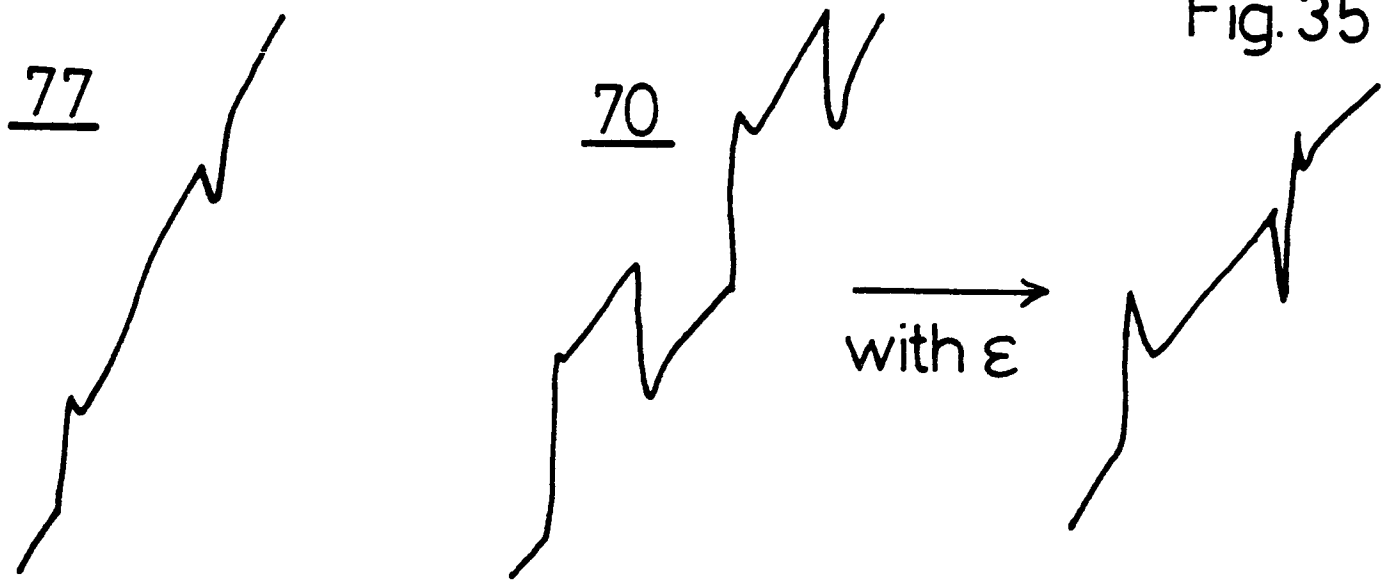


Fig. 35 Trace of the stress-strain behaviour during a strain rate change in compression for virgin specimen of Nb-16 at.%Mo

An approximate scale is at the foot of the page.

Fig. 35



0.5 kg.mm⁻²
(shear)

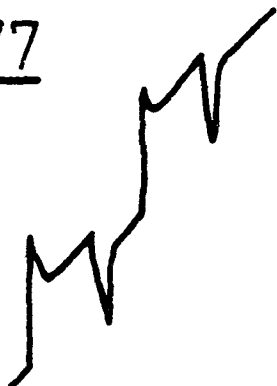
Fig. 36 Traces of the stress-strain behaviour during a strain rate change in compression for Nb-11 at.%Mo. The scale is the same as that in Fig.35.

All the specimens had been prestrained, as indicated below. The underlining indicates the temperature which is shown on the facing page.

290	-	<u>77</u>	K
290	-	<u>61</u>	K
290	-	61	- <u>49</u> K
290	-	77	- <u>41</u> K
290	-	77	- <u>20.4</u> K

Fig. 36

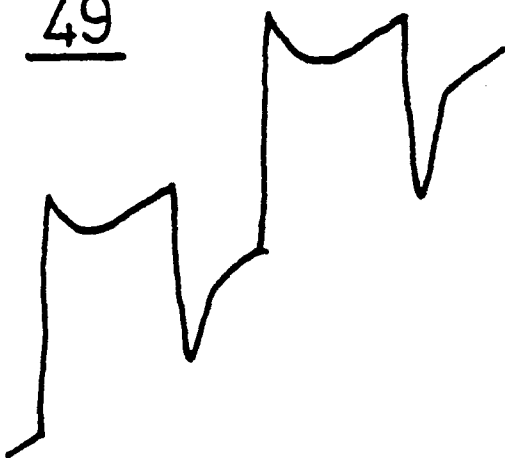
77



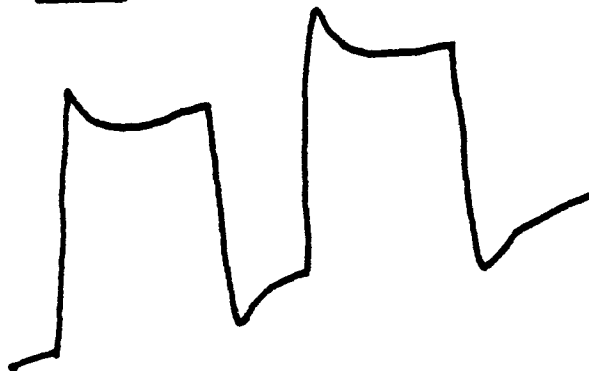
61



49



41



20.4



Fig. 37 Strain rate sensitivity, $\frac{\Delta \sigma}{\Delta \ln \dot{\epsilon}}$, in tension for Nb and the Nb-Ta alloys.

The dotted line for Nb between 77 K and 40 K reflects the uncertainty in the strain rate sensitivity.

The "error bars" at these temperatures are merely to give an indication of the strain rate sensitivity for increases in the strain rate at arbitrary strains.

Fig.37

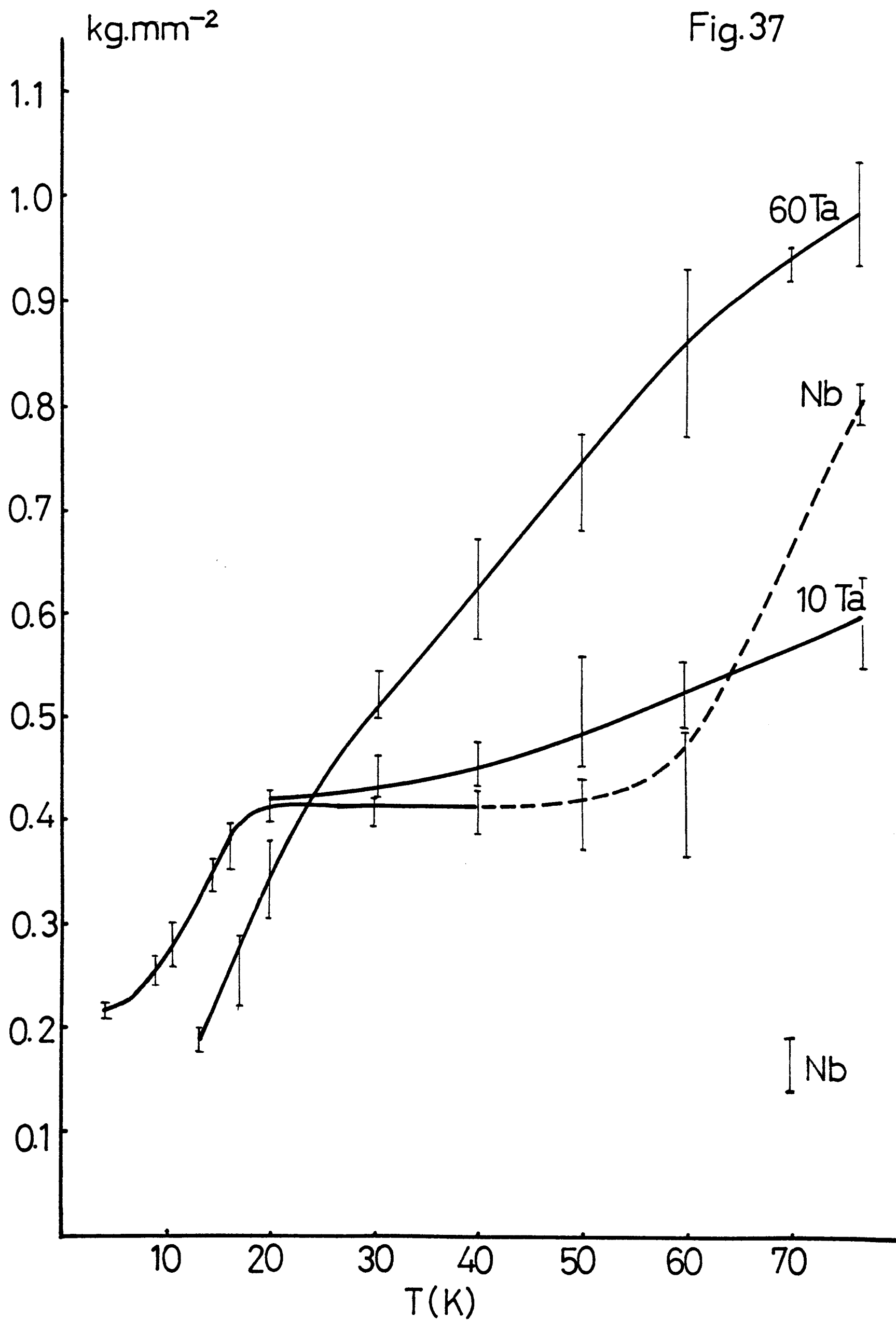


Fig. 38 Strain rate sensitivity, $\frac{\Delta \sigma}{\Delta \ln \dot{\epsilon}}$, in tension for

Nb and the Nb-Mo alloys.

See Fig. 37 for comment on the Nb curve.

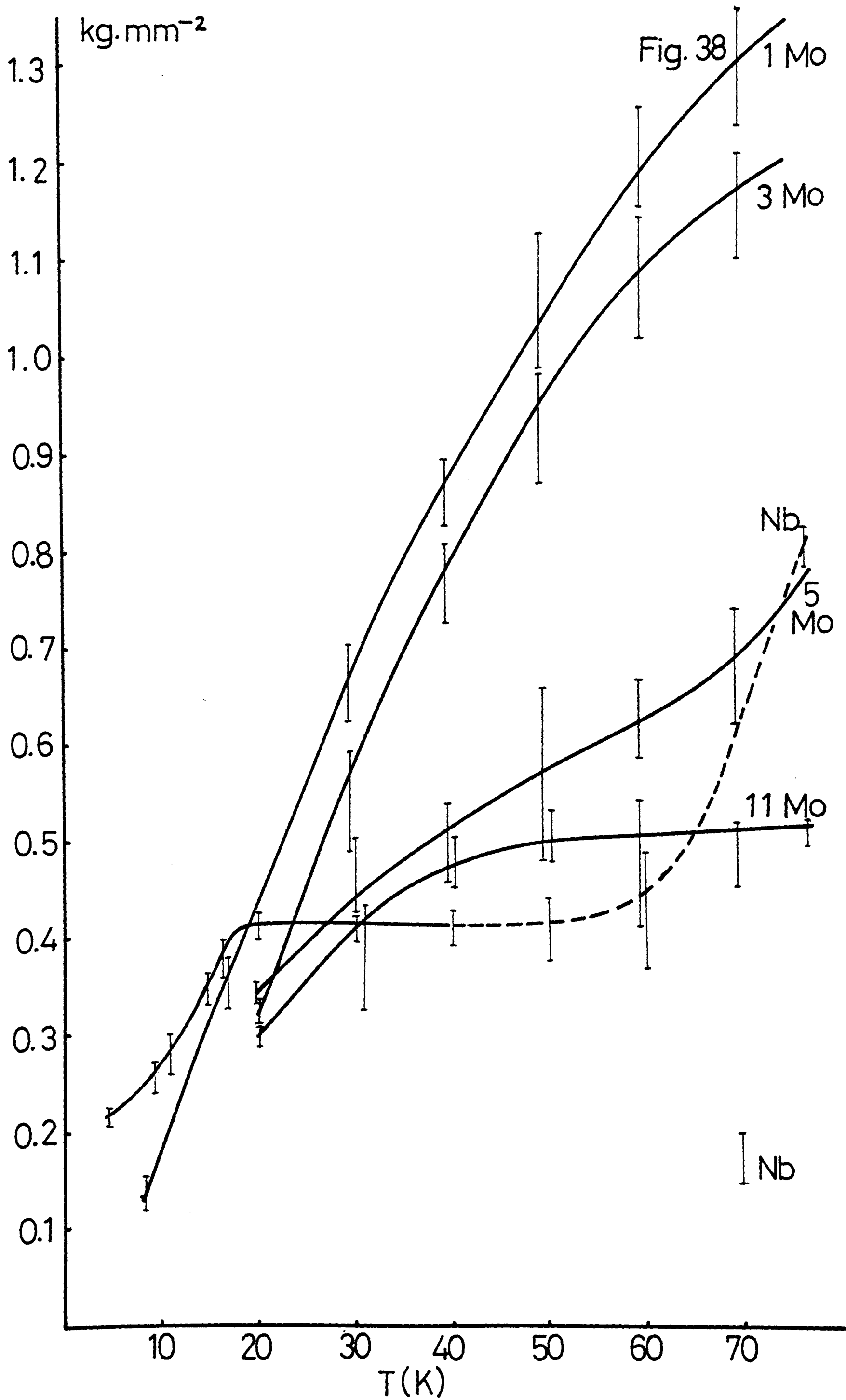


Fig. 39 Traces of the stress-strain behaviour during strain rate changes in tension for Nb.

An approximate scale is at the top of the page.

The size of the strain rate change is indicated at each temperature when other than the standard 10 x change was made.

Some of the specimens were strained at higher temperatures. The underlined temperature is the one shown in the figure.

40 - 30 - 20 - 16 K
70 - 60 - 50 - 40 - 30 - 20 - 15 K
50 - 40 - 30 - 20 - 14.5 K
40 - 29 - 19 - 10.5 K
50 - 40 - 30 - 20 - 9.75 K
30 - 20 - 15 - 8.25 K

Fig. 39

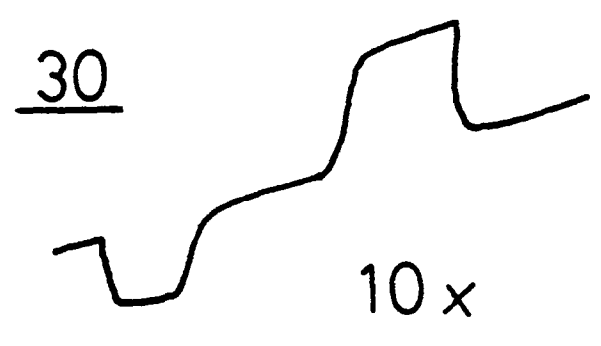
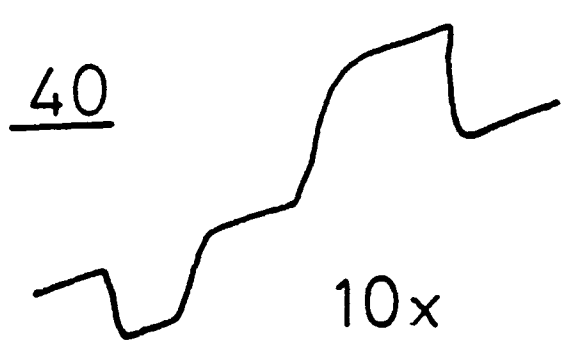
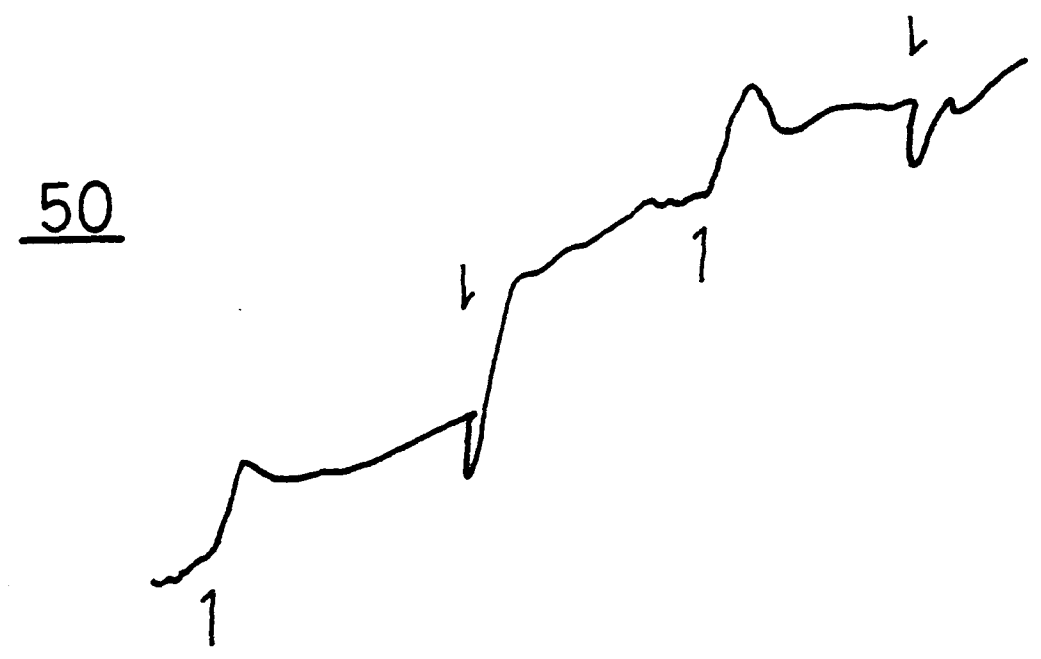
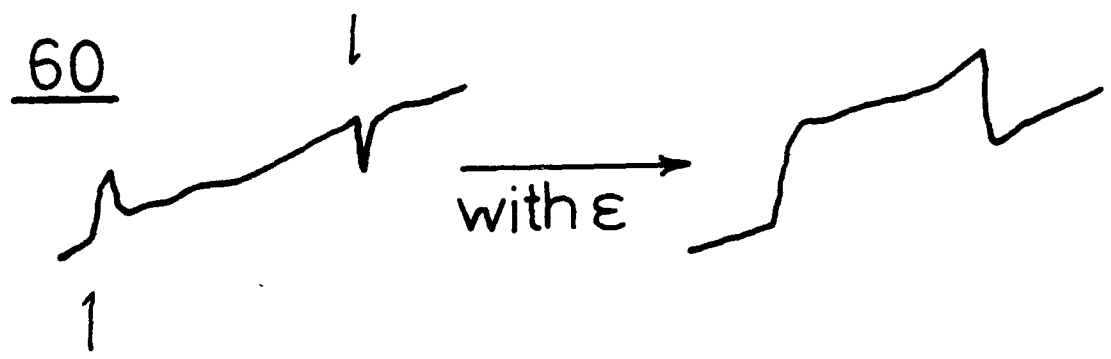
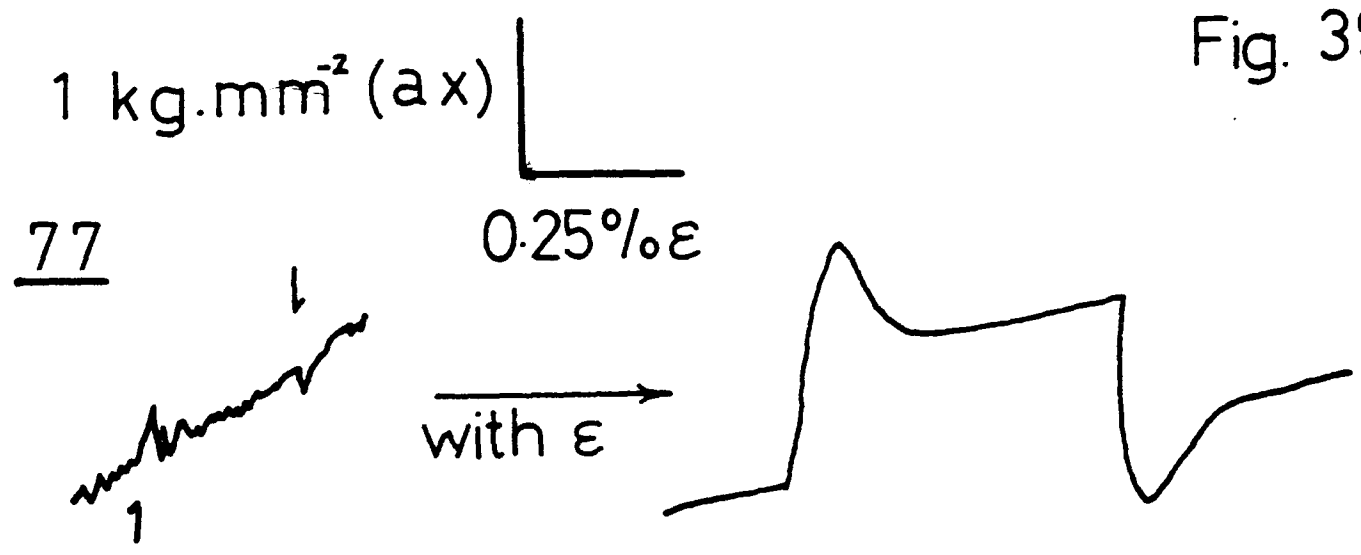
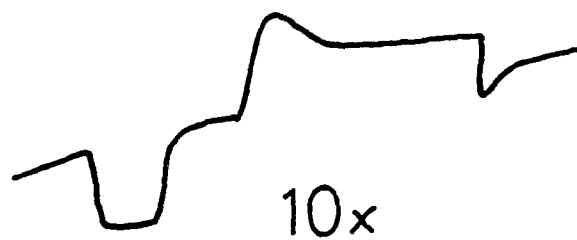


Fig. 39
cont'd

20



16



10x

2½x

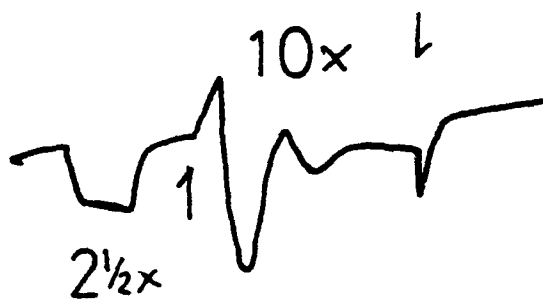
15



14.5



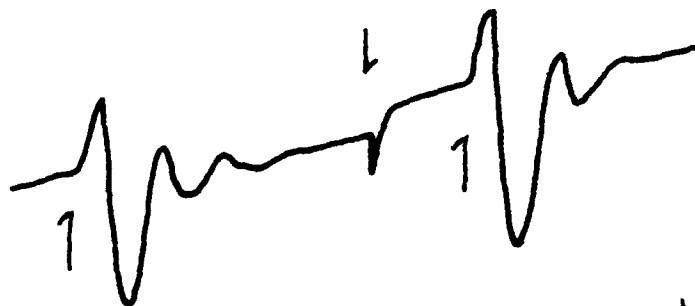
10.5



10x

2½x

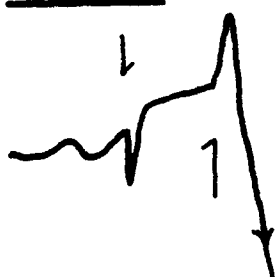
9.75



10x

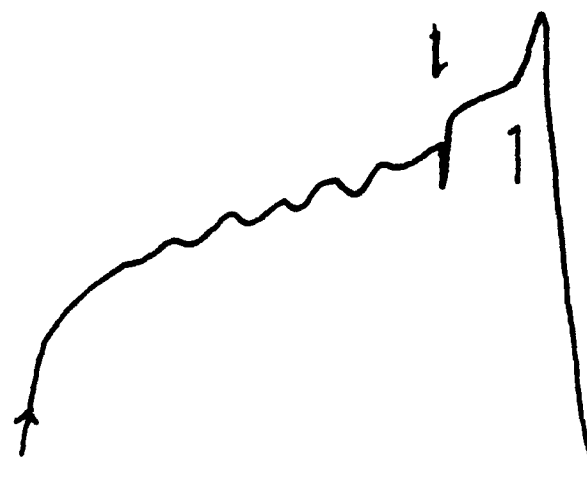
1

8.25



10x

1



10x

1

Fig. 40 Traces of the stress-strain behaviour during
strain rate changes in tension for :-

(a) Nb-10 at.%Ta

(b) Nb-60 at.%Ta

See Fig. 39 for general comments and the scale.

The previous temperatures of straining were,
where applicable :-

(a) 60 - 50 K
 290 - 40 K
 290 - 40 - 30 K

(b) 290 - 77 K
 290 - 77 - 72 K
 290 - 62 K
 290 - 62 - 50 K
 290 - 62 - 50 - 40 K
 290 - 40 - 30 K
 290 - 40 - 30 - 20 K

Fig.40(a)

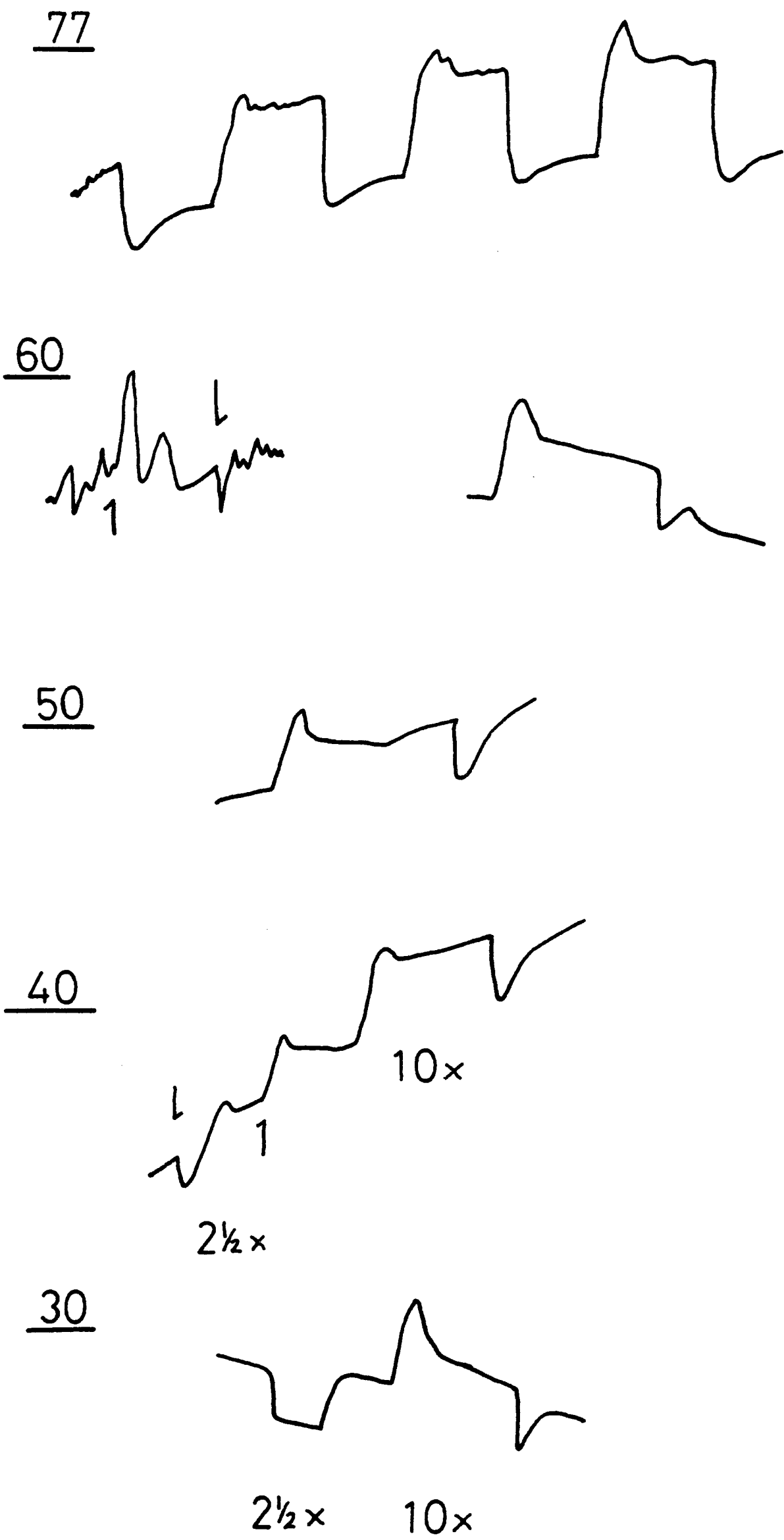


Fig. 40(b)

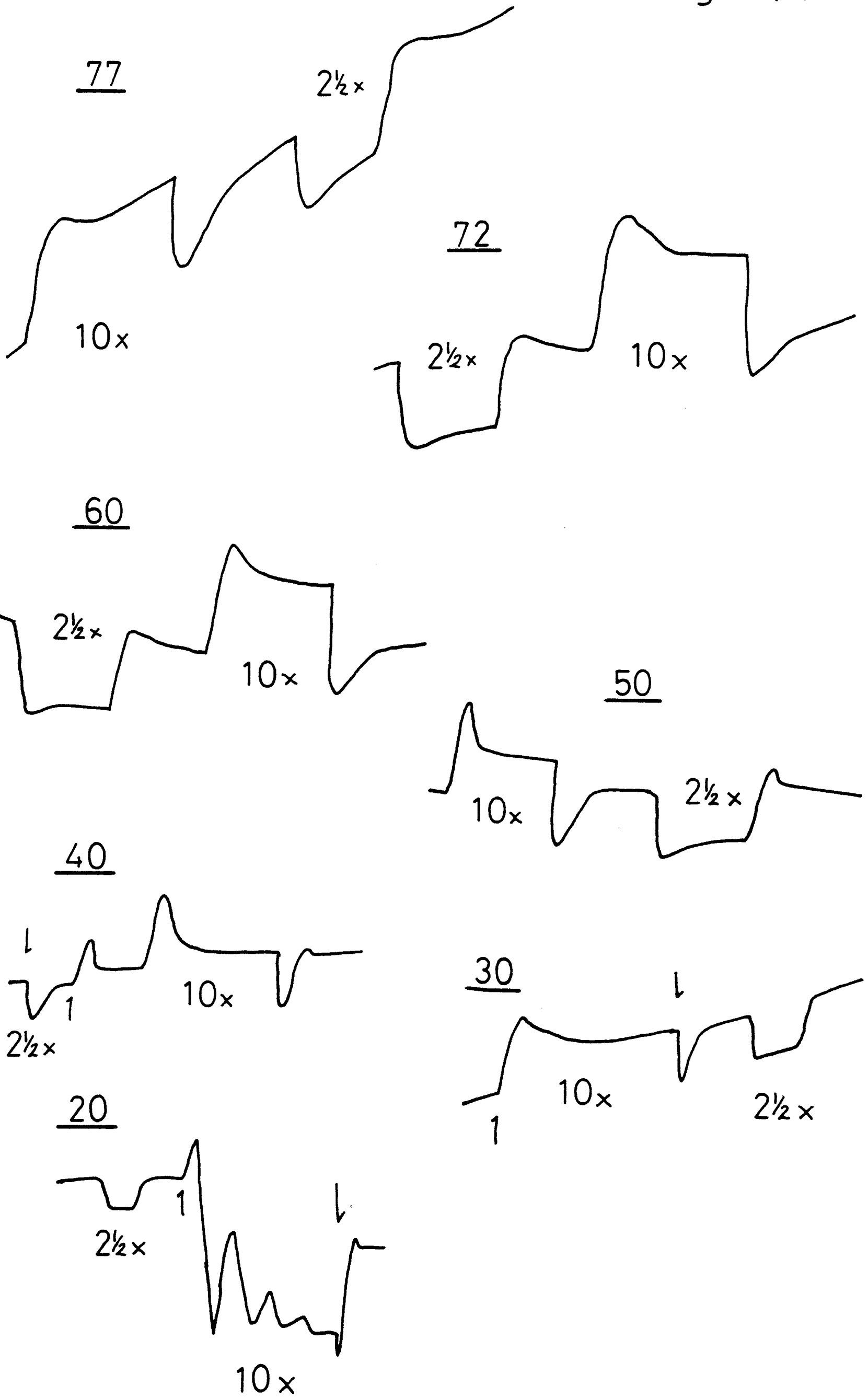


Fig. 41 Traces of the stress-strain behaviour during strain rate changes in tension for :-

- (a) Nb-1 at.%Mo
- (b) Nb-3 at.%Mo
- (c) Nb-5 at.%Mo
- (d) Nb-11 at.%Mo

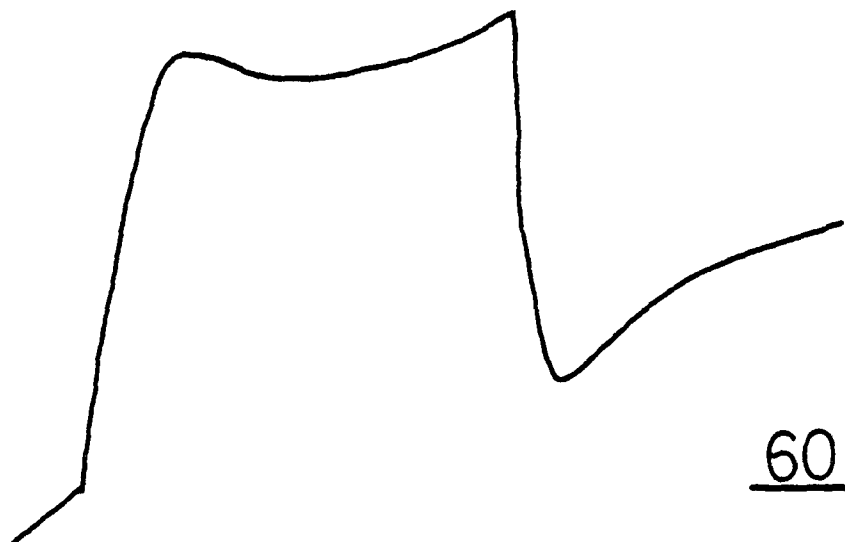
See Fig. 39 for general comments and the scale.

The previous temperatures of straining were :-

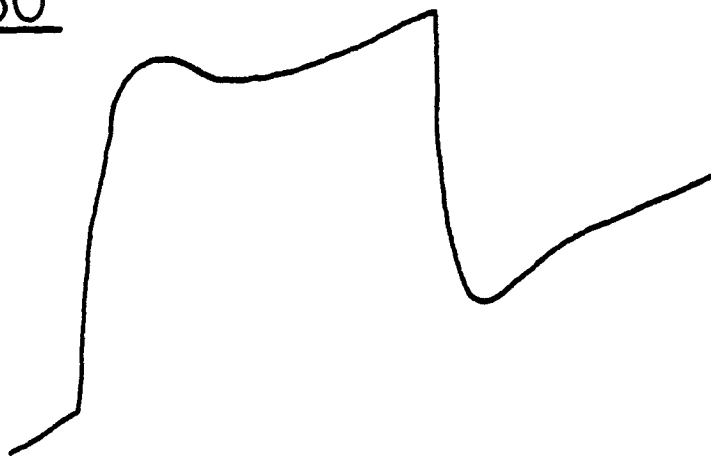
- (a) All Nb-1 at.%Mo specimens were prestrained at 290 K.
- (b) The specimens illustrating 77 K - 40 K were prestrained at 290 K. The temperatures for the other specimens were :-
$$290 - 40 - \underline{30} \text{ K} \quad \text{and}$$
$$290 - 40 - \underline{30} - \underline{20} \text{ K}$$
- (c) The specimens illustrating 77 K, 50 K, 30 K and 20 K were prestrained at 290 K. The others were :-
$$290 - 70 - \underline{60} \text{ K} \quad \text{and}$$
$$290 - 70 - \underline{60} - \underline{40} \text{ K}$$
- (d) The specimens illustrating 77 K, 60 K, 50 K and 20 K were prestrained at 290 K. The others were :-
$$290 - 70 - 50 - \underline{40} \text{ K}$$
$$290 - 40 - \underline{30} \text{ K}$$

Fig.41(a)

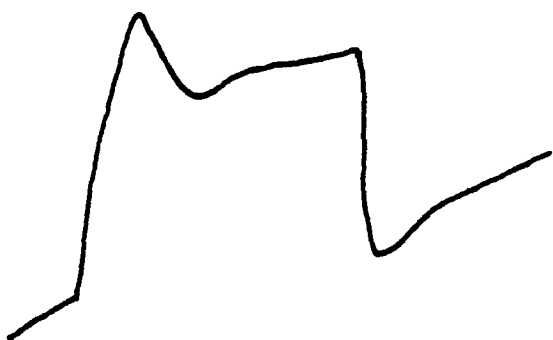
77



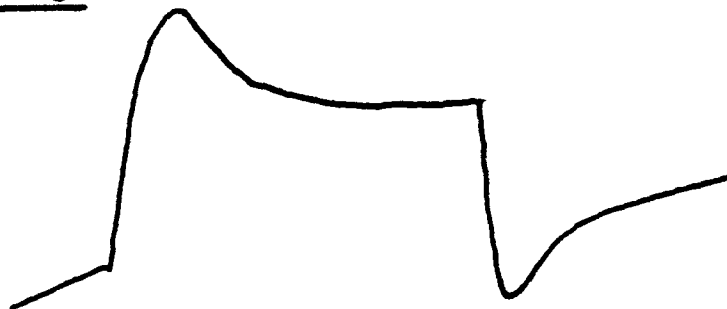
60



50



40



30



22

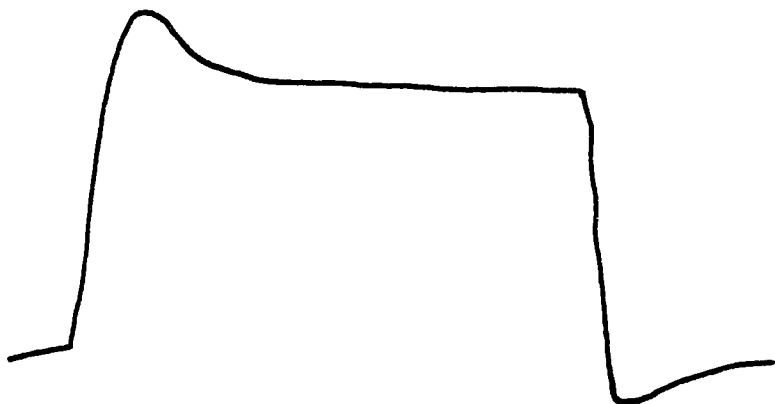


10x

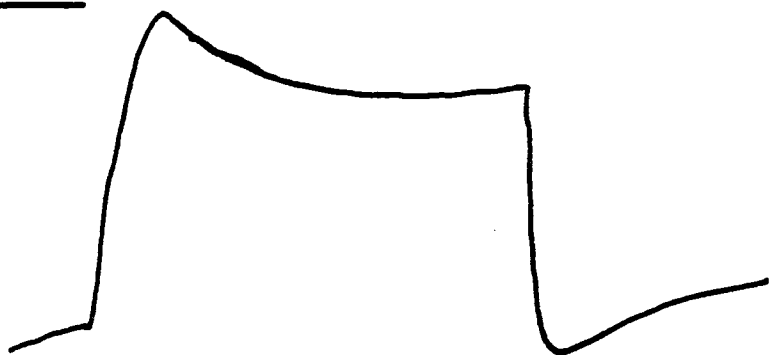
2½x

Fig.41(b)

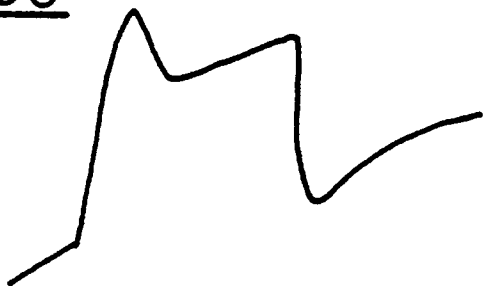
77



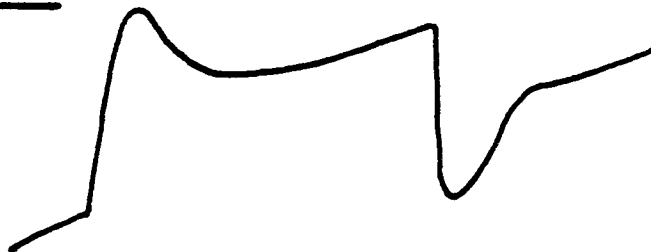
61



50



40



30



20

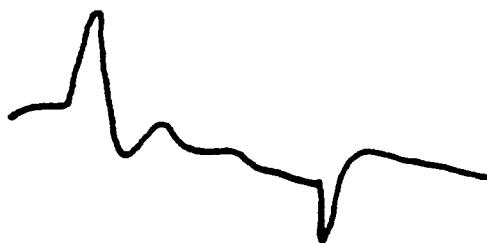
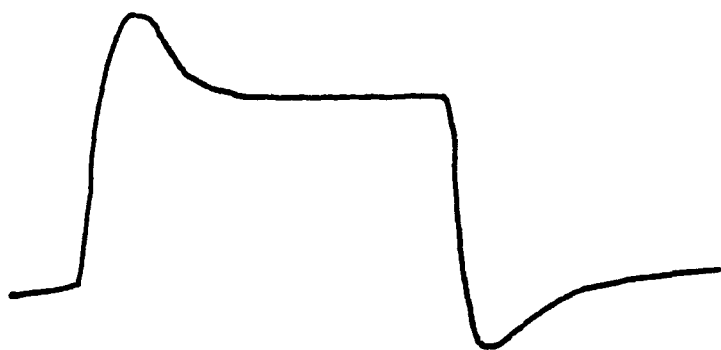
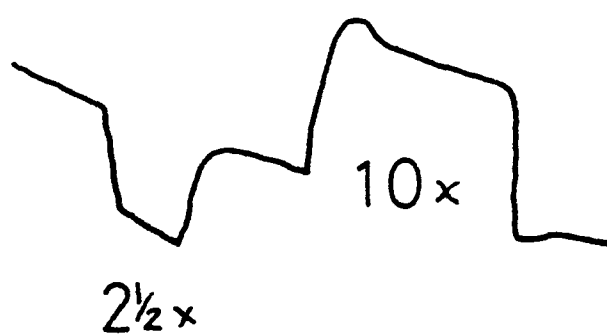


Fig.41(c)

77



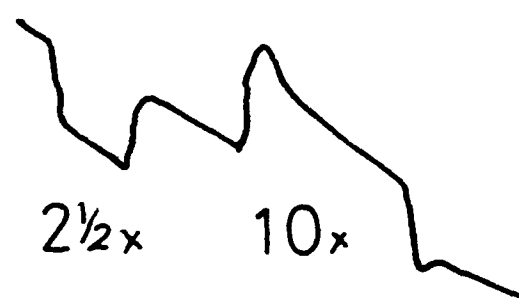
60



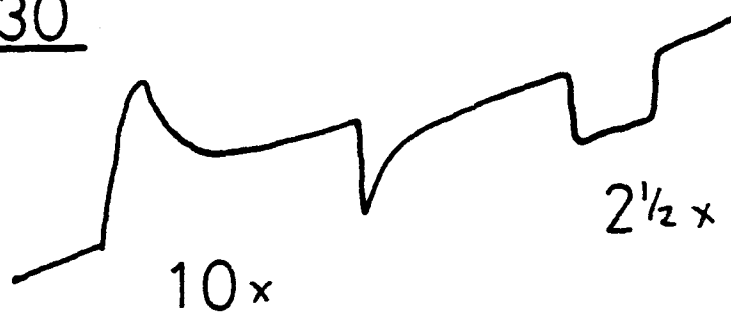
50



41



30

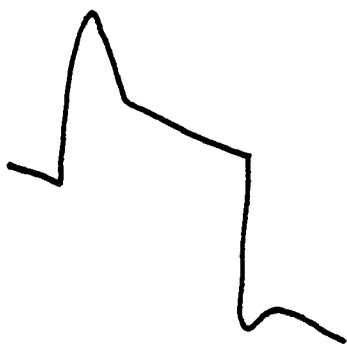


20

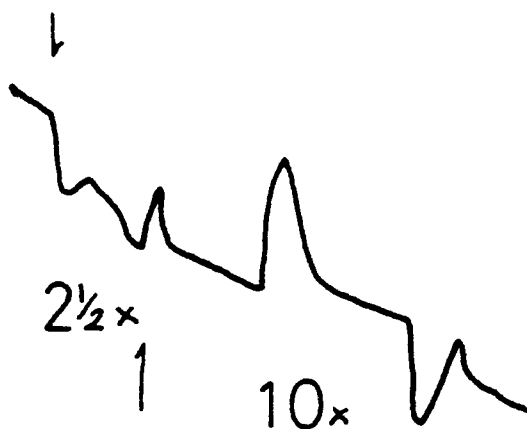


Fig.41(d)

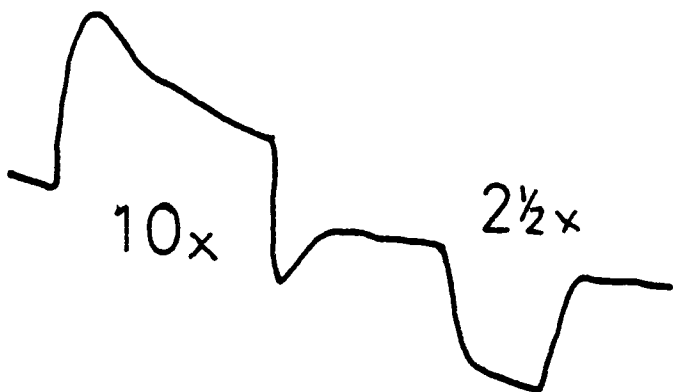
77



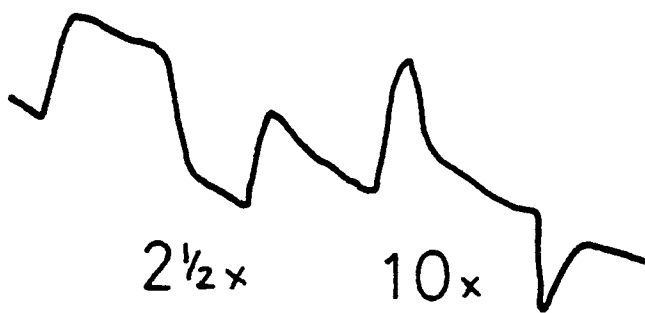
60



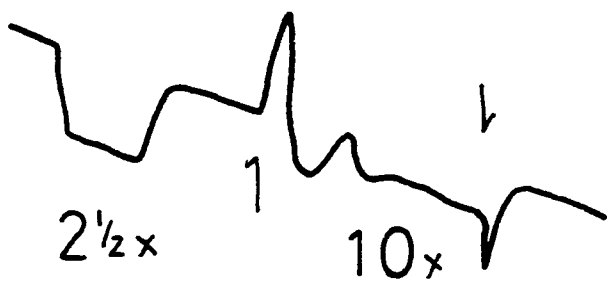
50



40



30



20

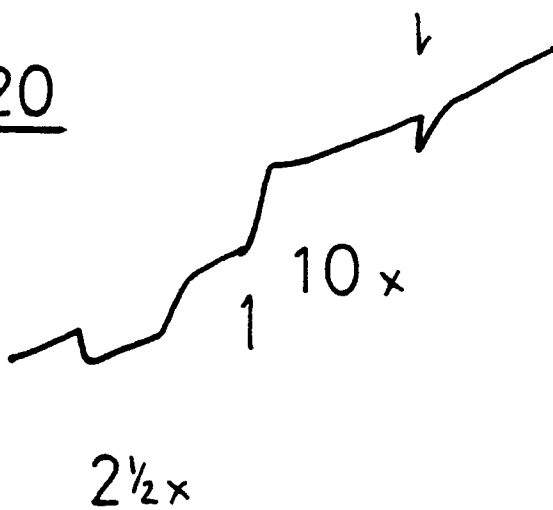


Fig. 42 Nb at 113 K - Compression

$$\epsilon = 2.4\%$$

$$\theta = 73^\circ$$

Fig. 43 Nb at 77 K - Compression

$$\epsilon = 2.6\%$$

$$\theta = 64^\circ$$

Fig. 44 Nb at 77 K - Compression

$$\epsilon = 5.7\%$$

$$\theta = 86^\circ$$

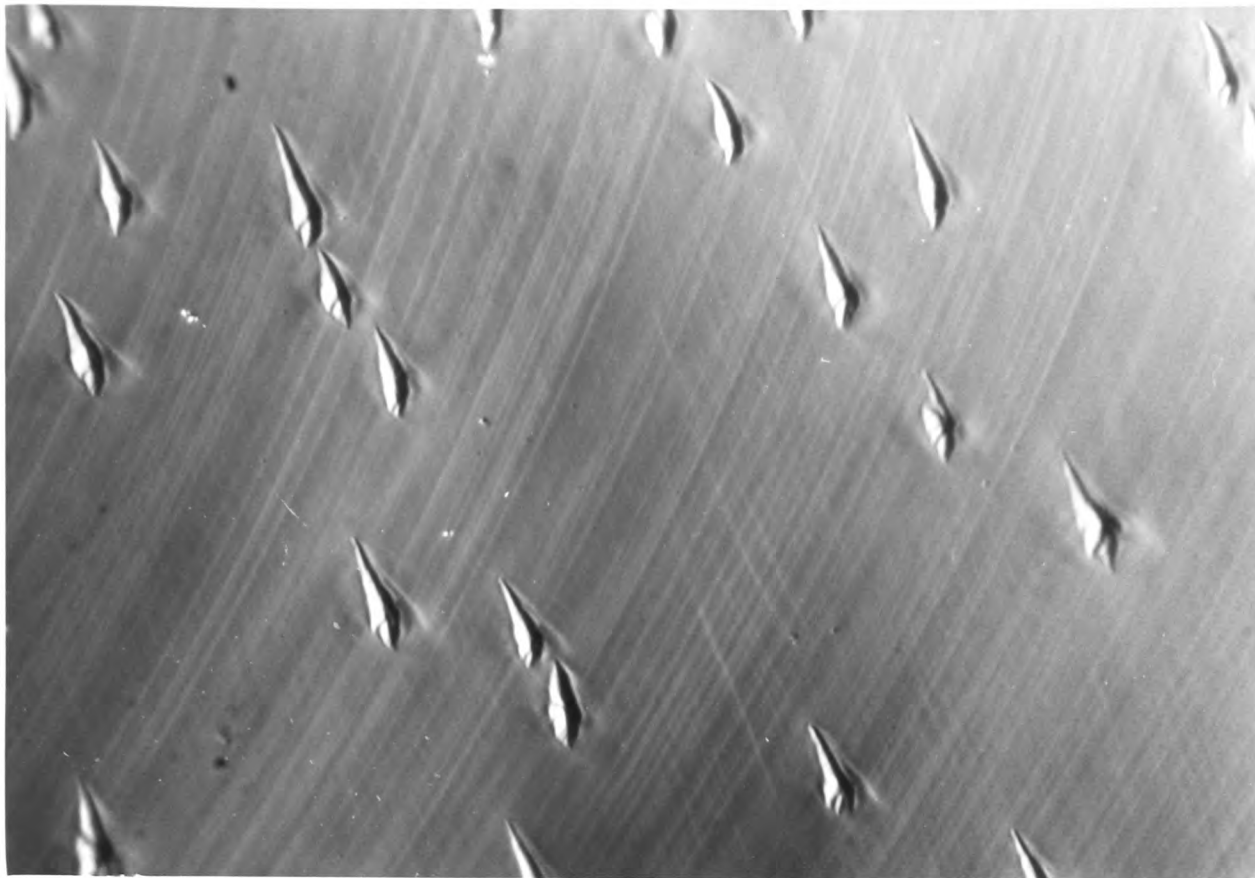


Fig. 42

(011)
 $(0\bar{1}1)$

50 μ m

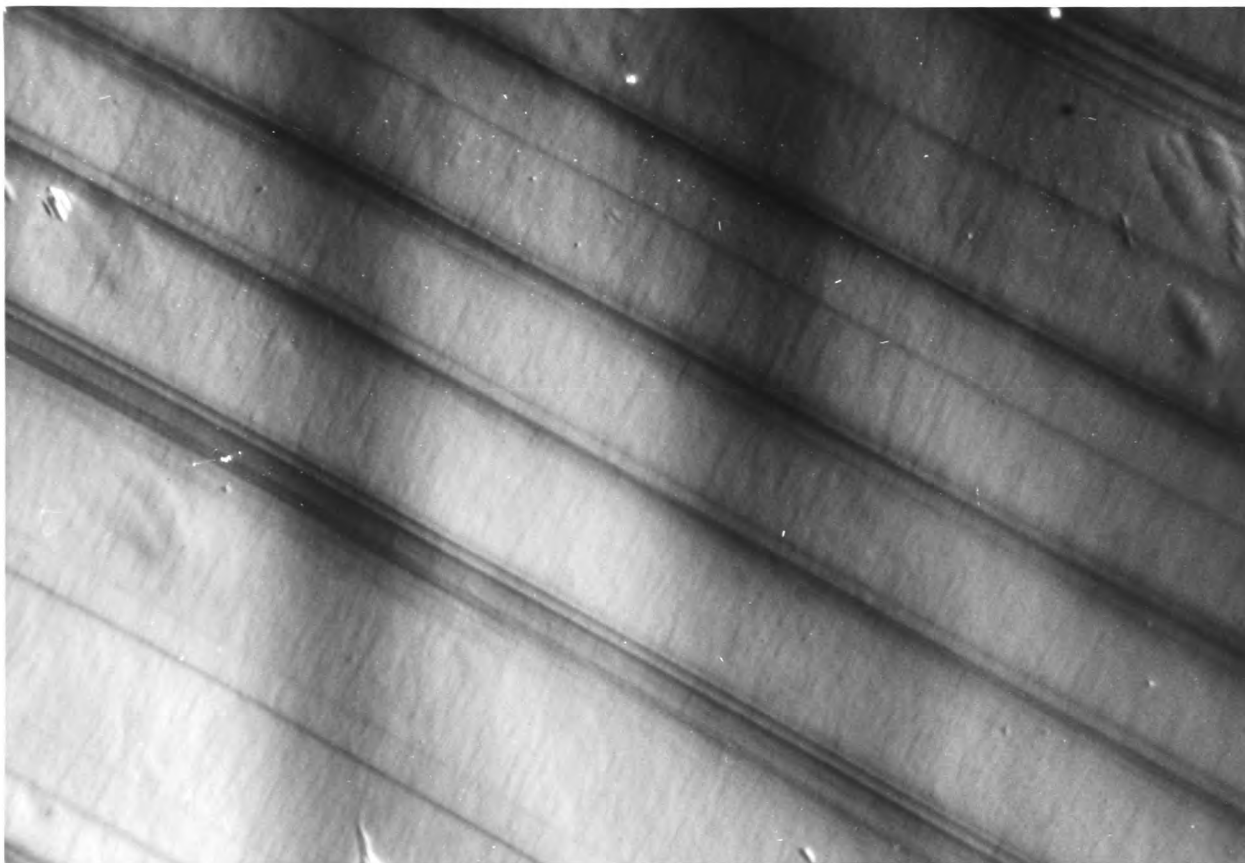


Fig. 43

$(0\bar{1}1)$
 $(\bar{1}01)$

50 μ m

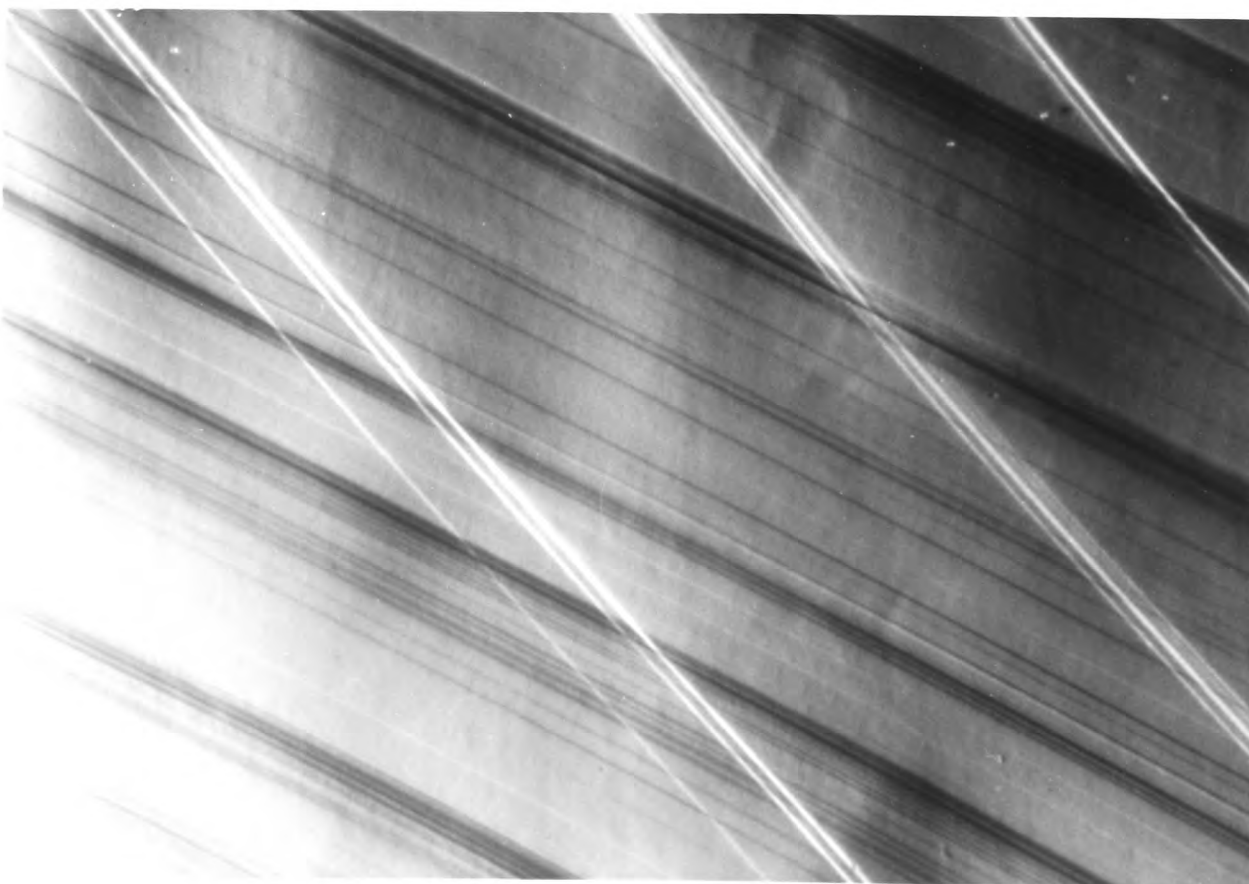


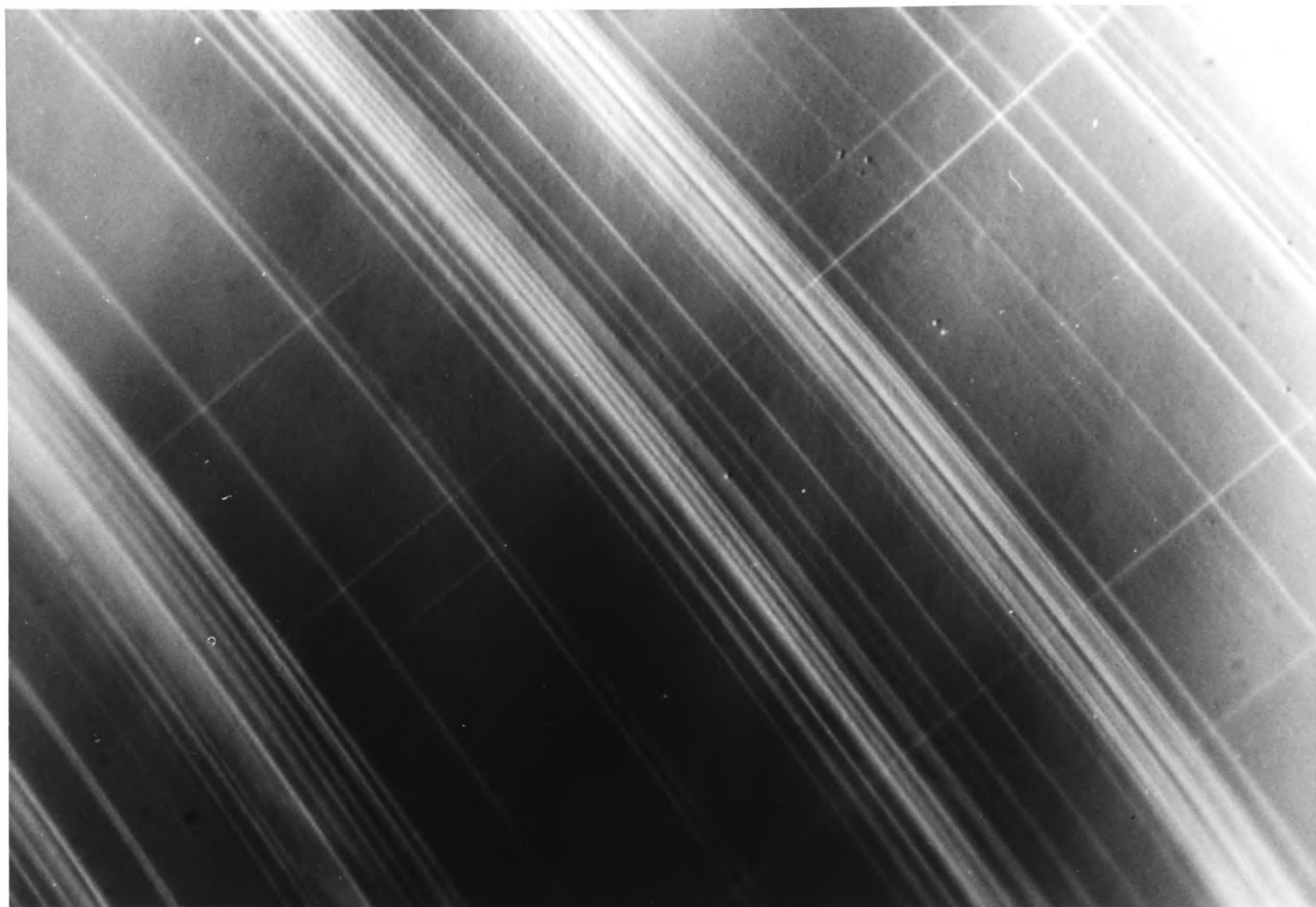
Fig. 44

(101)
 $(0\bar{1}1)$

50 μ m

Fig. 45 Nb at 77 K - Compression
 $\epsilon = 8.3\%$
 $\theta = 1^\circ$

Fig. 46 Nb at 77 K - Compression
 $\epsilon = 8.3\%$
 $\theta = 103^\circ$



50 μm

(101) $\searrow$ (0 $\bar{1}$ 1)

Fig. 45



50 μm

(101) $\searrow$ (0 $\bar{1}$ 1)

Fig. 46

Fig. 47 Nb-10 at.%Ta at 290 K - Compression

γ = 6.7% Crystal 312

θ = 92°

Fig. 48 Nb-10 at.%Ta at 290 K - Compression

γ = 6.7% Crystal 312

θ = 14°

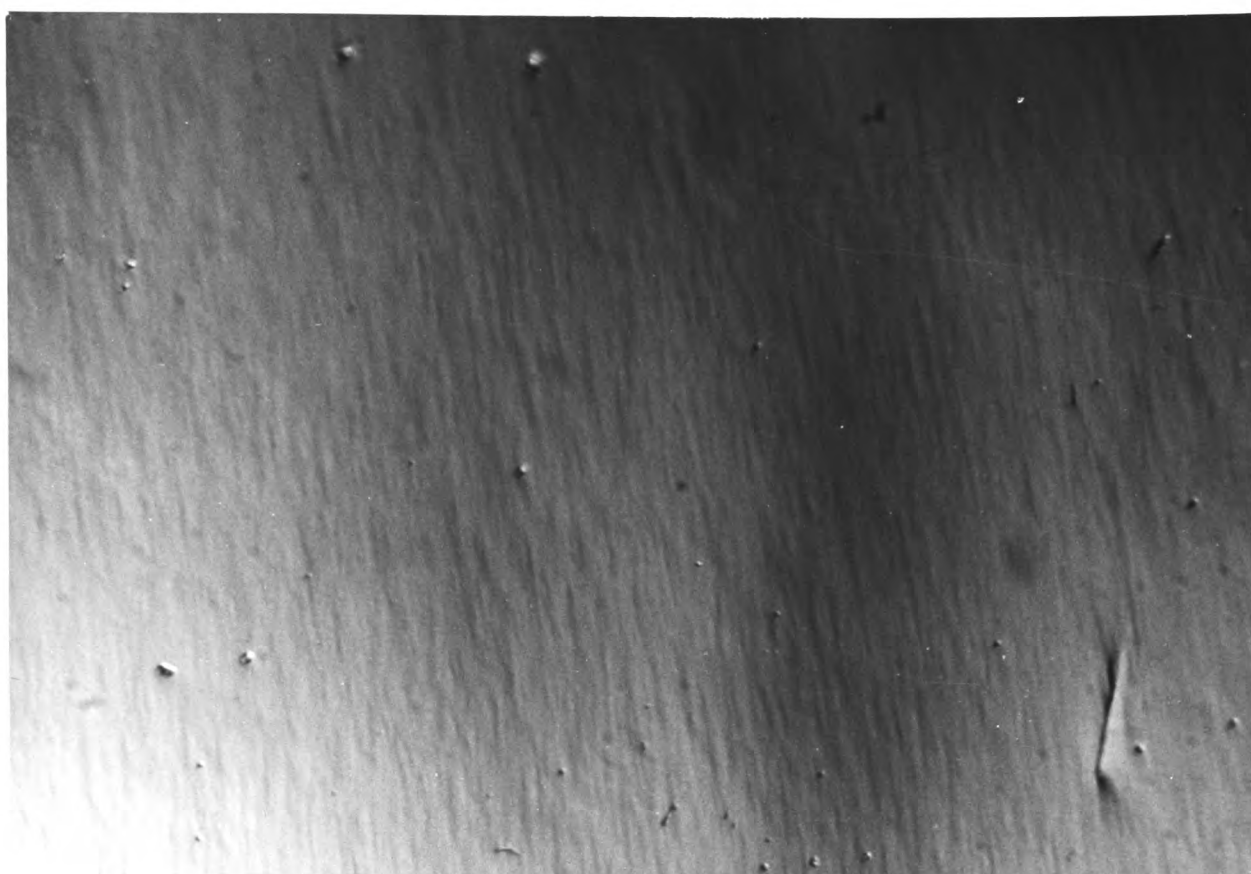


Fig.47

50μm

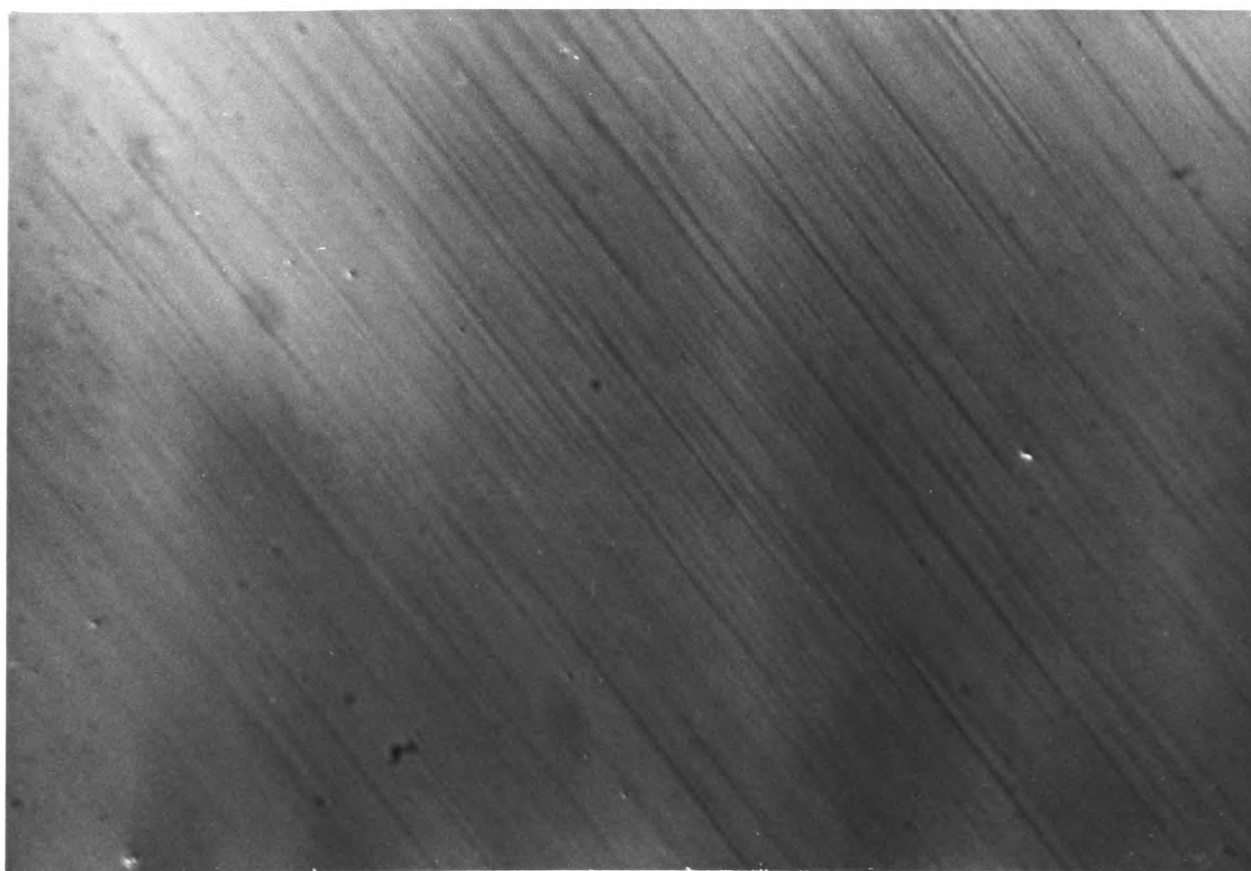


Fig.48

50μm

Fig. 49 Nb-10 at.%Ta at 290 K - Compression

$\gamma = 17.9\%$ Crystal 312

$\theta = 78^\circ$

Fig. 50 Nb-10 at.%Ta at 290 K - Compression

$\gamma = 17.9\%$ Crystal 312

$\theta = 10^\circ$

Fig. 51 Nb-10 at.%Ta at 290 K - Compression

$\gamma = 23.6\%$ Crystal 312

$\theta = 5^\circ$

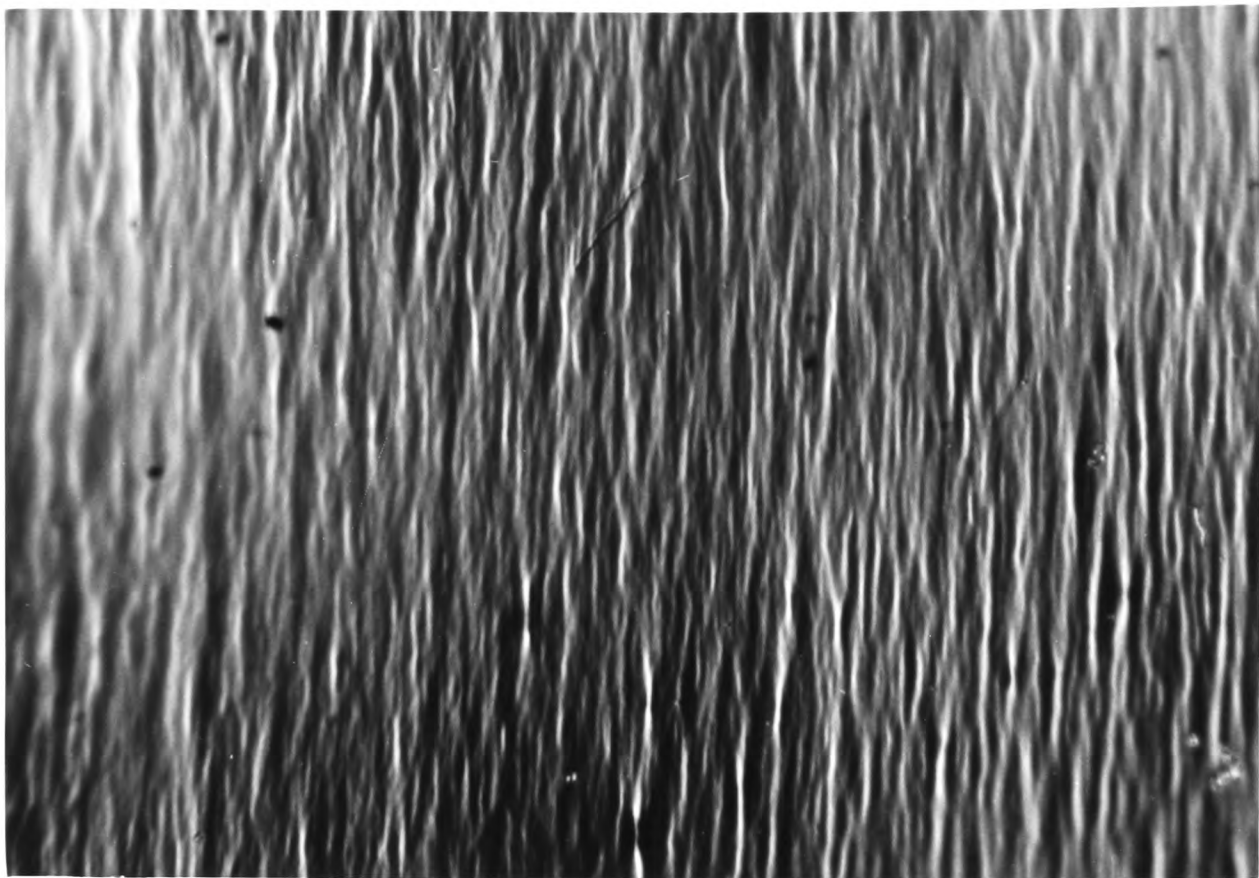


Fig.49

50μm

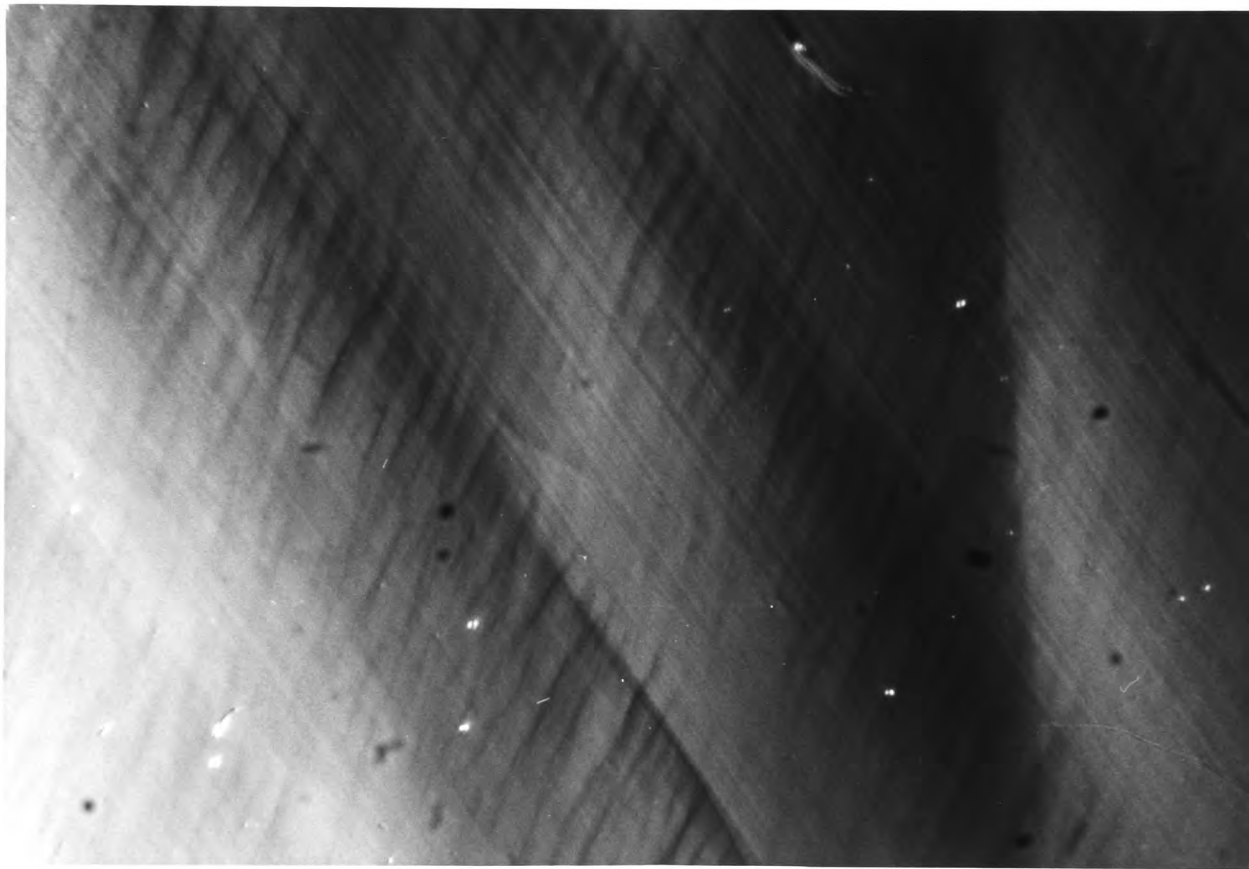


Fig.50

(011)
($\bar{1}$ 01)

50μm

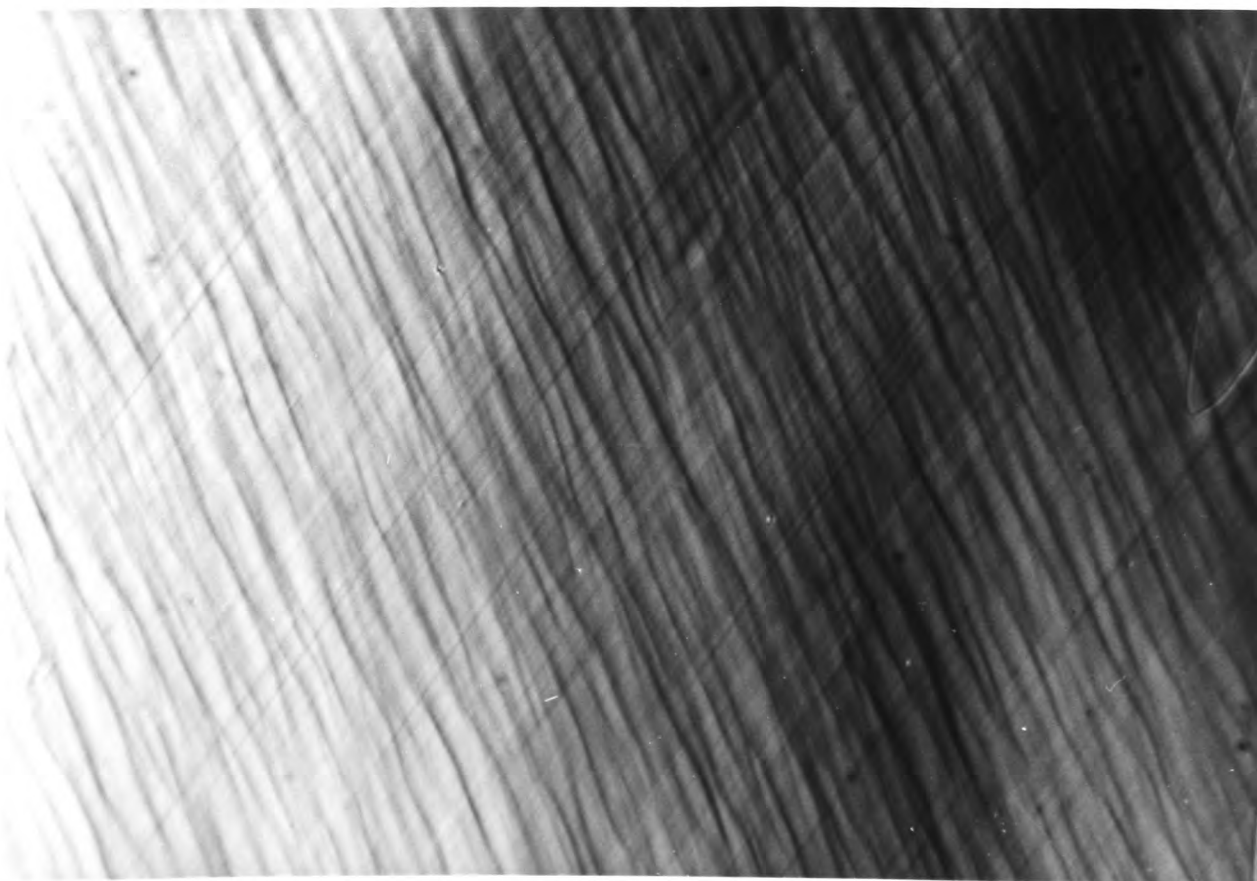


Fig.51

($\bar{1}$ 01)
(011)

50μm

Fig. 52 Nb-10 at.%Ta at 175 K - Compression

γ = 4.7% Crystal 312

θ = 88°

Fig. 53 Nb-10 at.%Ta at 175 K - Compression

γ = 4.7% Crystal 312

θ = 9°

Fig. 54 Nb-60 at.%Ta at 175 K - Compression

γ = 7.1% Crystal 316

θ = 137°



Fig. 52

50 μ m

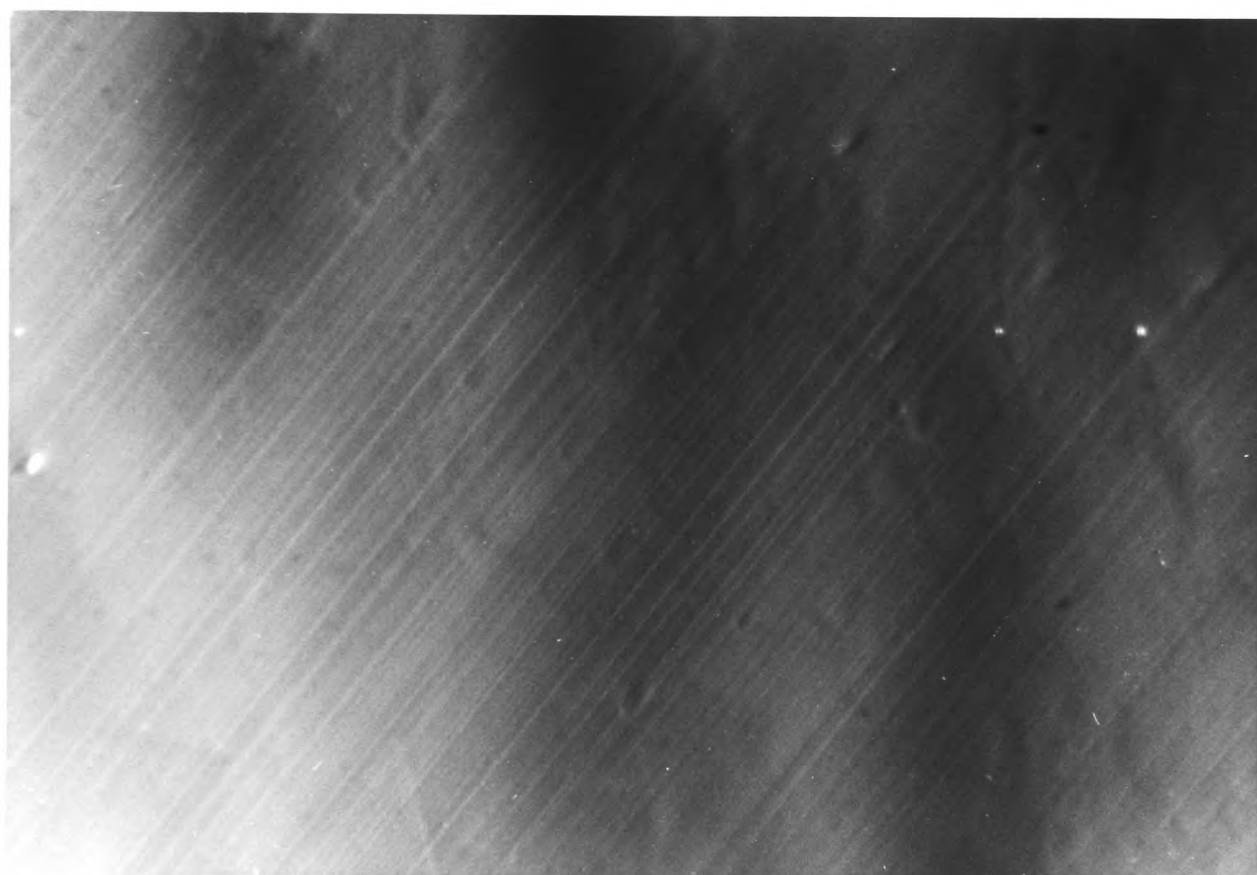


Fig. 53

50 μ m

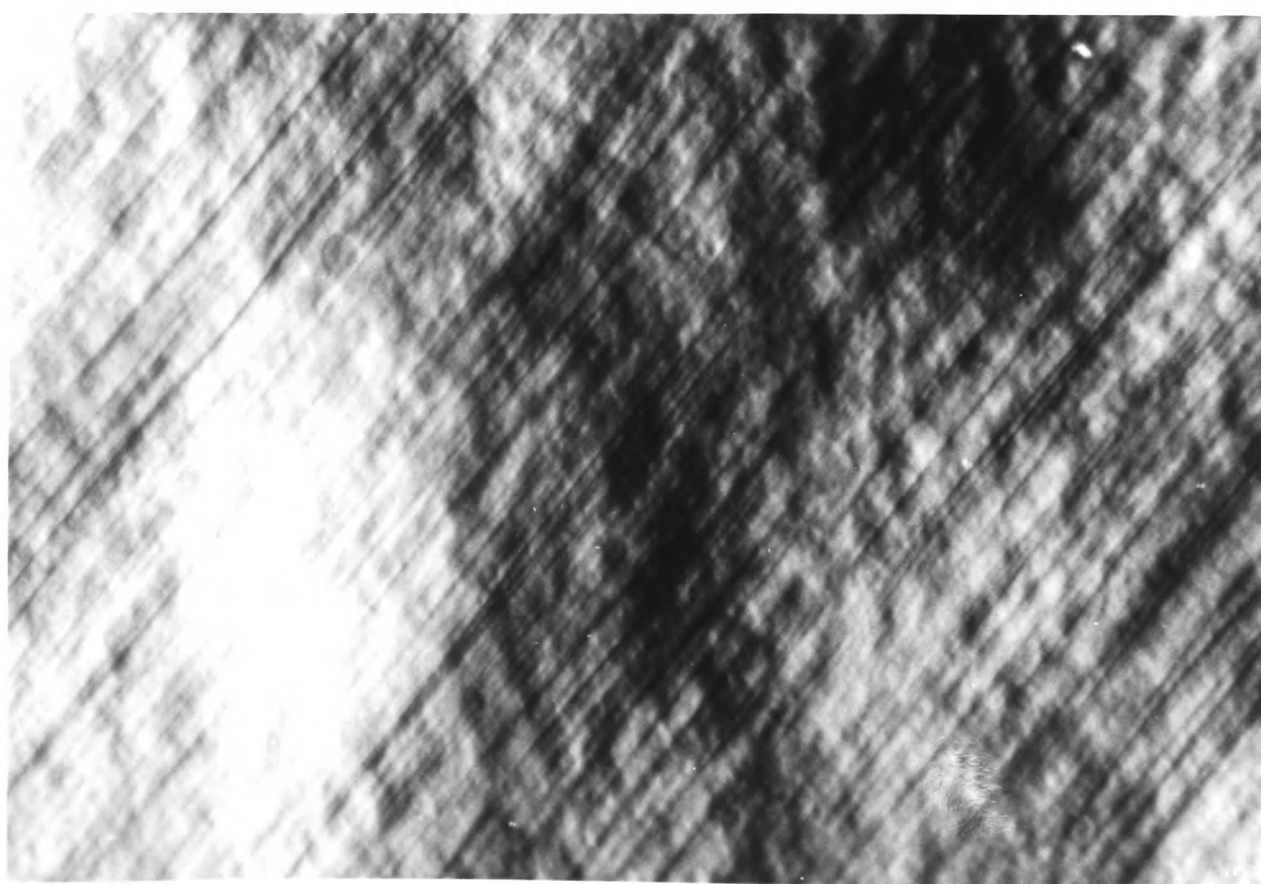
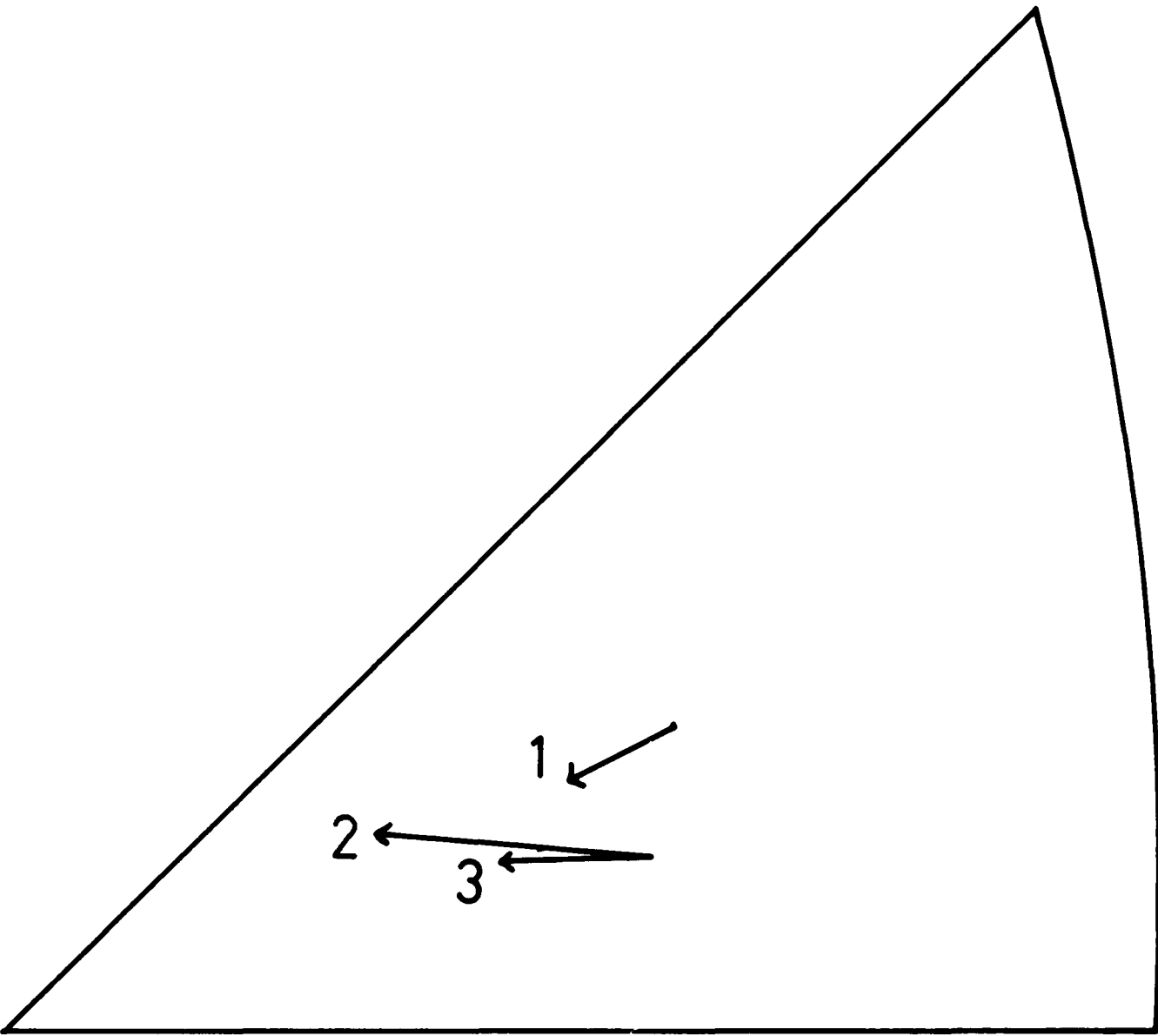


Fig. 54

50 μ m

Fig. 55 Axial rotations of specimens deforming by
anomalous glide in tension.

Fig. 55



- | | | |
|----|--------|------------|
| 1: | 312/6 | 10 at.% Ta |
| 2: | 302/18 | 1 at.% Mo |
| 3: | 302/21 | 1 at.% Mo |

Fig. 56 Nb-1 at.%Mo at 290 K - Compression

$\gamma = 6.2\%$ Crystal 302

$\theta = 80^\circ$

Fig. 57 Nb-1 at.%Mo at 290 K - Compression

$\gamma = 6.2\%$ Crystal 302

$\theta = 15^\circ$



Fig. 56

50 μ m



Fig. 57

50 μ m

Fig. 58 Nb-1 at.%Mo at 290 K - Compression

γ = 23% Crystal 302

θ = 18°

Fig. 59 Nb-1 at.%Mo at 290 K - Compression

γ = 23% Crystal 302

θ = 2°

Fig. 60 Nb-1 at.%Mo at 290 K - Compression

γ = 23% Crystal 302

θ = 86°

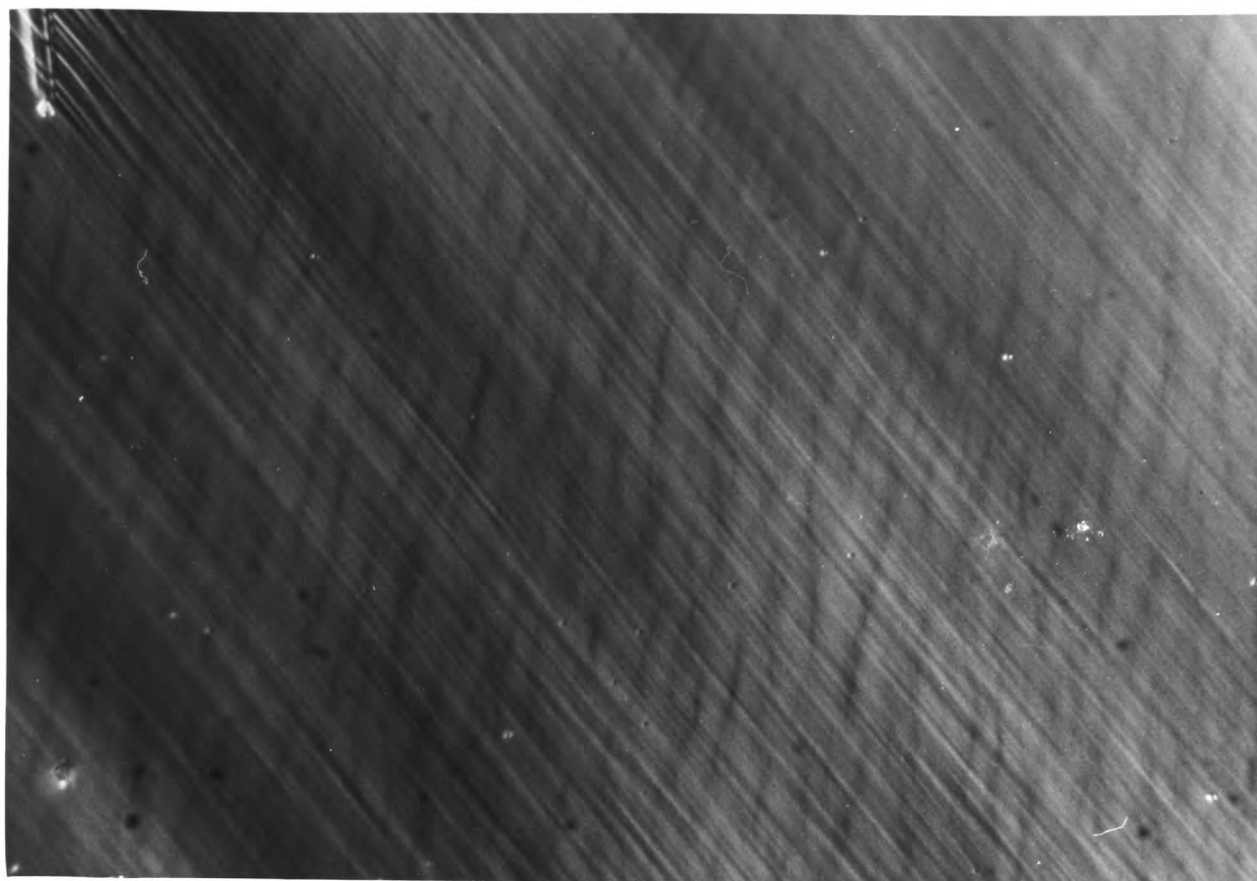


Fig. 58

$(\bar{1}01)$
 (011)

50 μ m

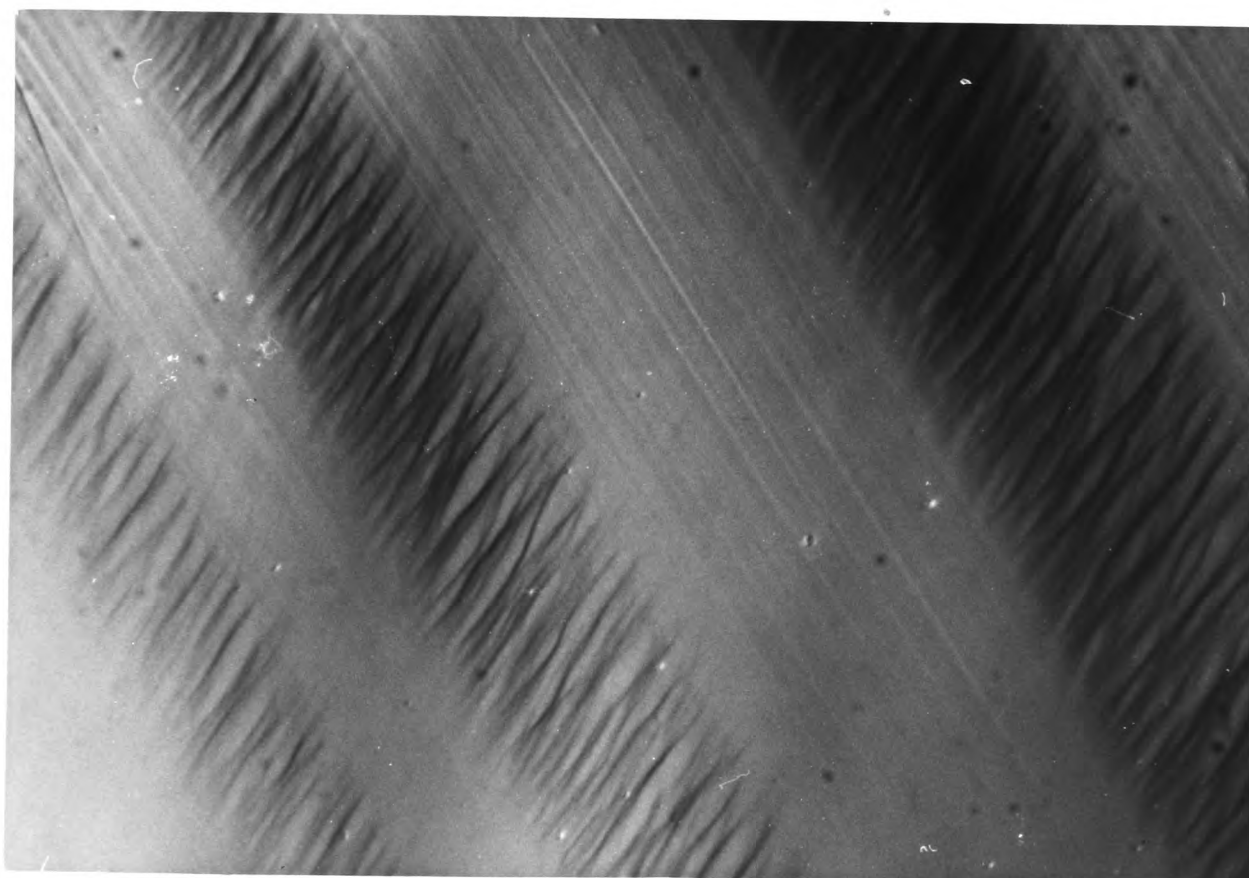


Fig. 59

$(\bar{1}01)$
 (011)

50 μ m



Fig. 60

50 μ m

Fig. 61 Nb-1 at.%Mo at 175 K - Compression
 $\gamma = 5.6\%$ Crystal 326
 $\theta = 83^\circ$

Fig. 62 Nb-1 at.%Mo at 175 K - Compression
 $\gamma = 16.4\%$ Crystal 326
 $\theta = 76^\circ$

Fig. 63 Nb-1 at.%Mo at 175 K - Compression
 $\gamma = 16.4\%$ Crystal 326
 $\theta = 12^\circ$



Fig. 61

$(\bar{1}01)$
 (011)

50 μm



Fig. 62

50 μm

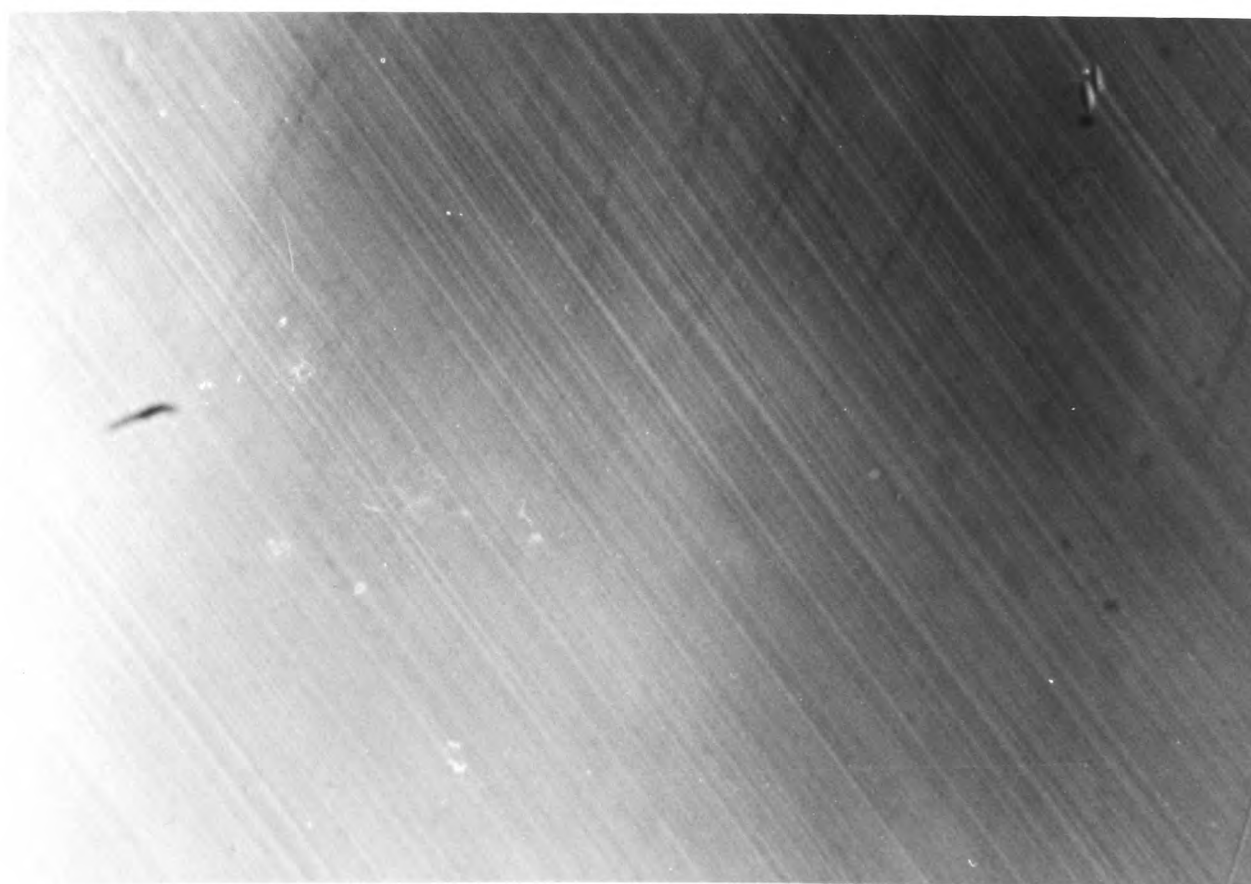


Fig. 63

$(\bar{1}01)$
 (011)

50 μm

Fig. 64 Nb-1 at.%Mo at 113 K - Compression

$\gamma = 12.5\%$ Crystal 302

$\theta = 92^\circ$

Fig. 65 Nb-1 at.%Mo at 77 K (prestrained) - Compression

ϵ at 290 K = 2.3% Crystal 302

ϵ at 77 K = 3.2%

$\theta = 42^\circ$

Fig. 66 Nb-1 at.%Mo at 77 K (prestrained) - Compression

ϵ at 290 K = 2.3% Crystal 302

ϵ at 77 K = 5.9%

$\theta = 30^\circ$



Fig. 64

$(0\bar{1}1)$
 $(\bar{1}01)$

50 μm

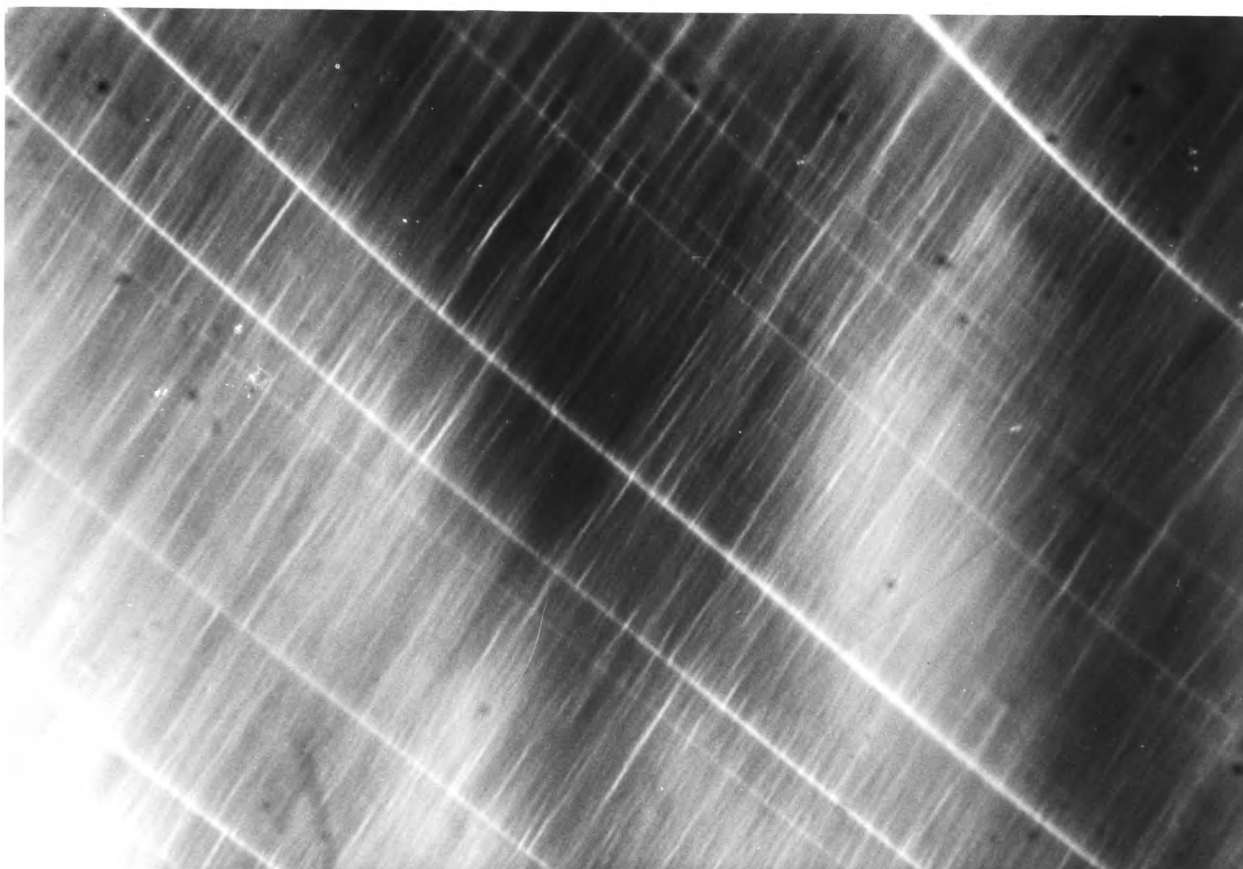


Fig. 65

$(0\bar{1}1)$
 $(\bar{1}01)$

50 μm

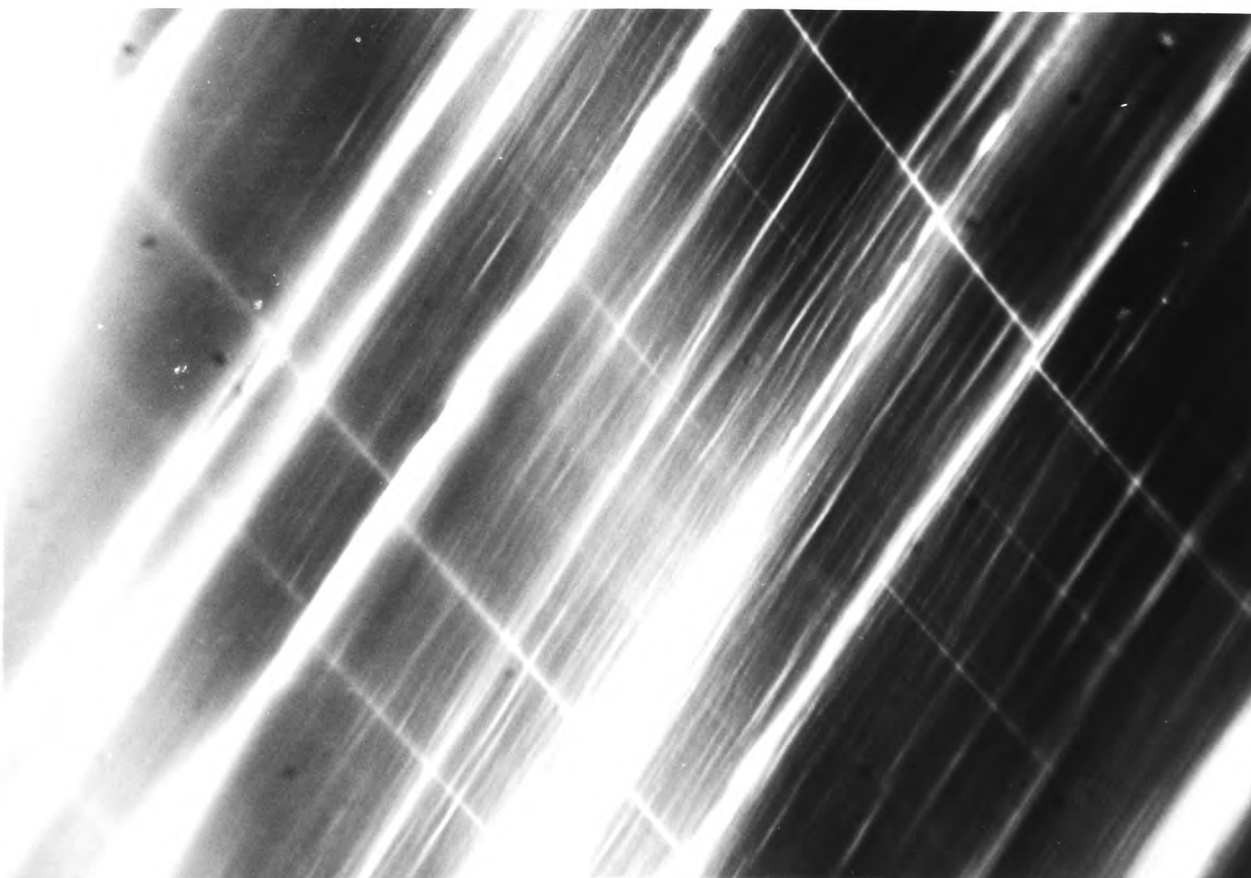


Fig. 66

$(0\bar{1}1)$
 $(\bar{1}01)$

50 μm

Fig. 67 Nb-2 at.%Mo at 290 K - Compression

γ = 14% Crystal 300

θ = 114°

Fig. 68 Nb-2 at.%Mo at 77 K (prestrained) - Compression

γ at 290 K = 3.5% Crystal 300

γ at 77 K = 15.0%

θ = 59°

Fig. 69 Nb-2 at.%Mo at 77 K (prestrained) - Compression

γ at 290 K = 3.5% Crystal 300

γ at 77 K = 15.0%

θ = 172°

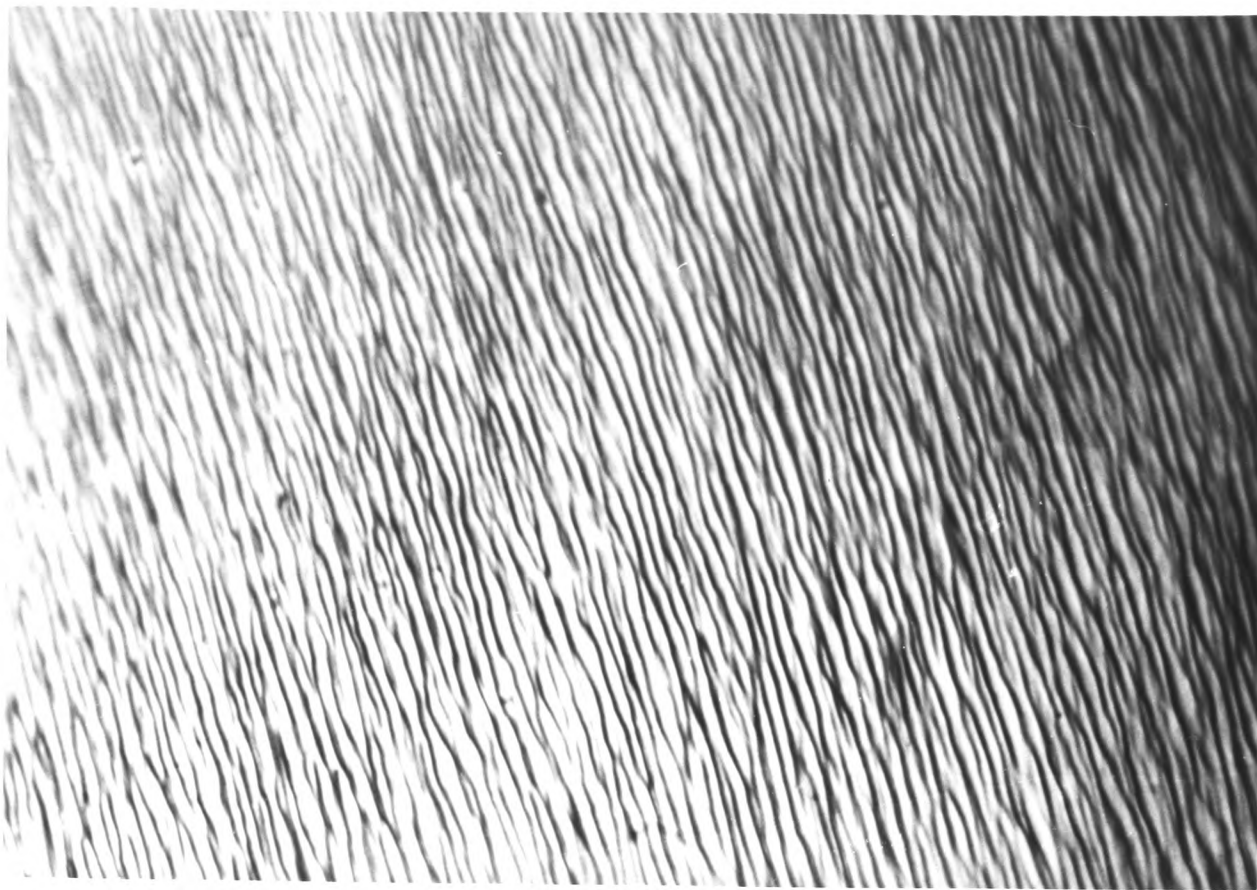


Fig. 67

50 μ m

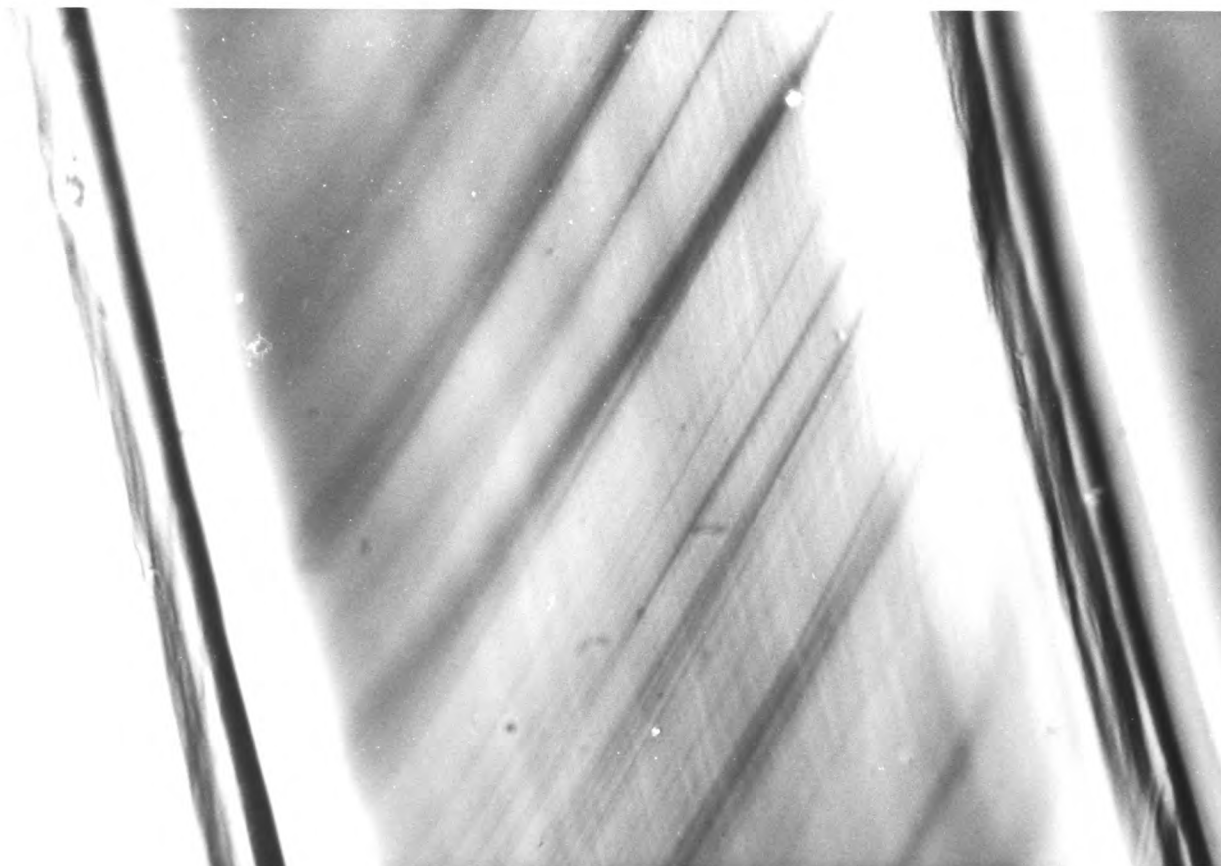


Fig. 68

$(\bar{1}01)$
(011)

50 μ m

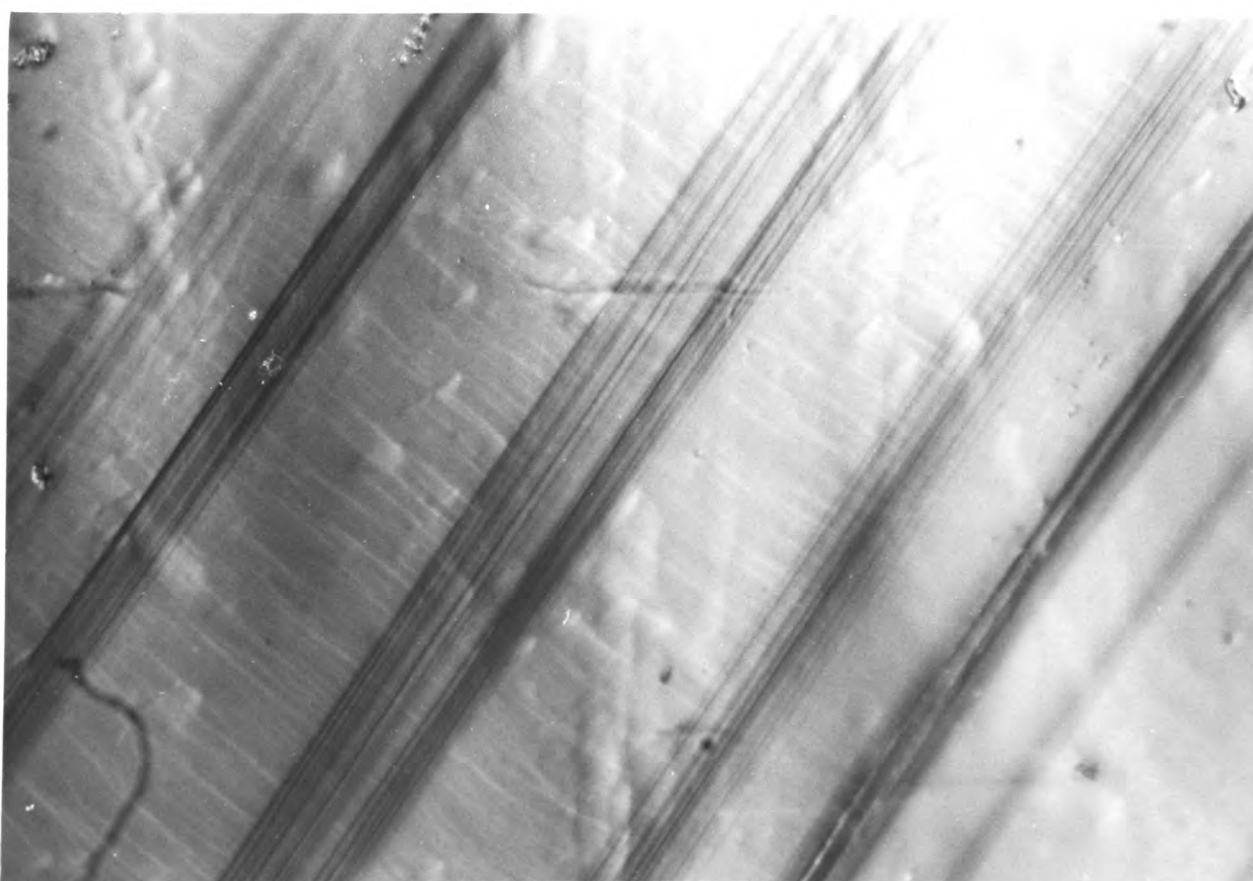


Fig. 69

(011)
 $(\bar{1}01)$

50 μ m

Fig. 70 Nb-5 at.%Mo at 290 K - Compression

γ = 35% Crystal 287

θ = 80°

Fig. 71 Nb-5 at.%Mo at 145 K - Compression

γ = 15.1% Crystal 287

θ = 90°

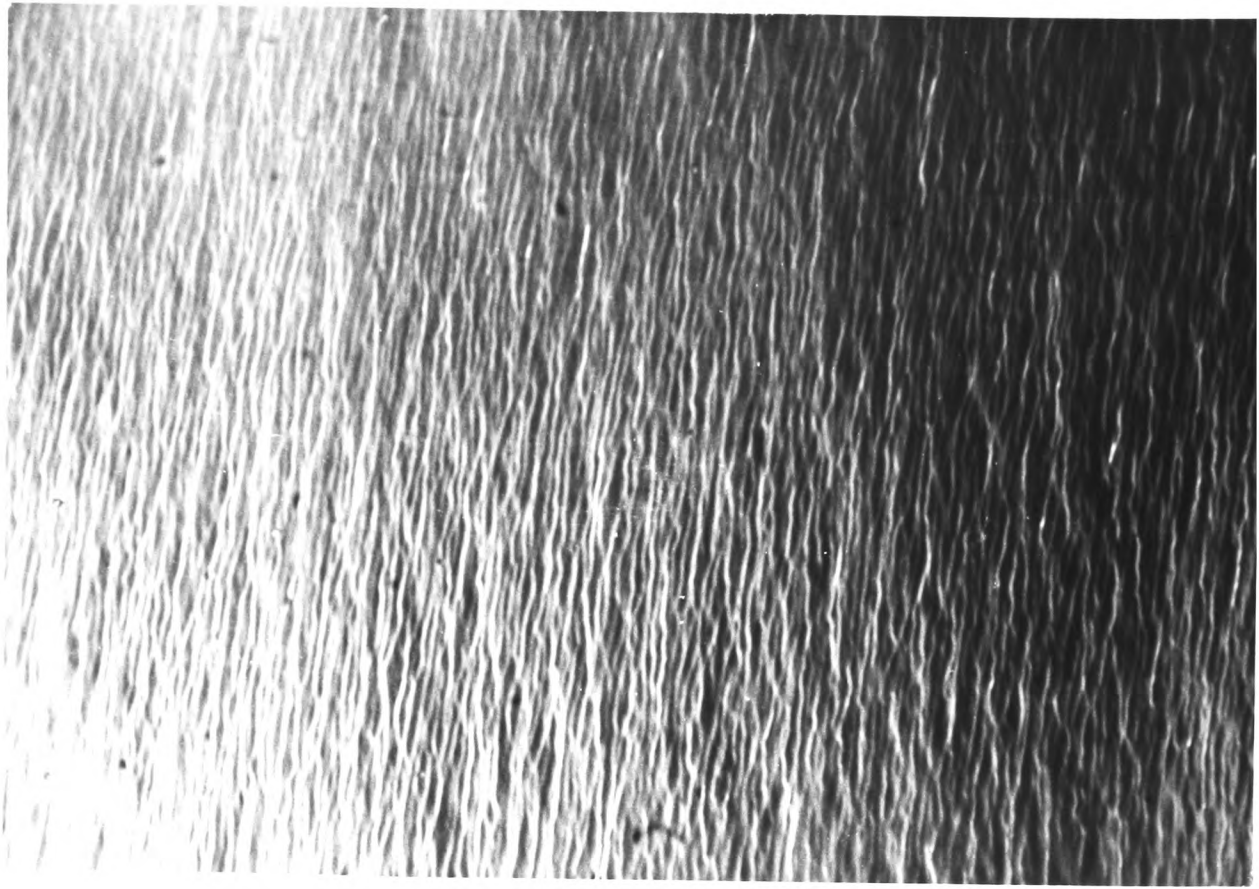


Fig. 70

50 μ m

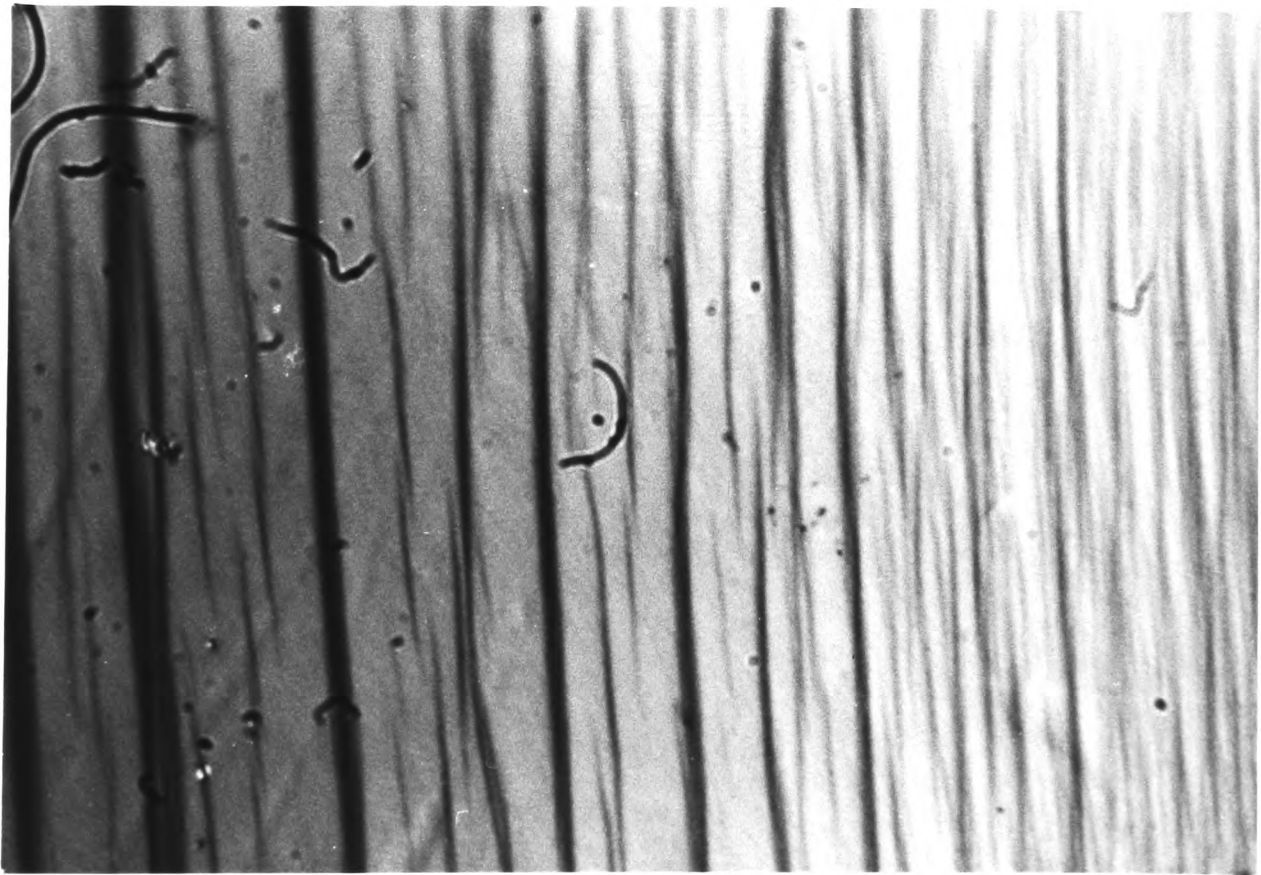


Fig. 71

50 μ m

Fig. 72 Nb-11 at.%Mo at 290 K - Compression
 $\gamma = 5.5\%$ Crystal 309
 $\theta = 70^\circ$

Fig. 73 Nb-11 at.%Mo at 175 K - Compression
 $\gamma = 7.3\%$ Crystal 309
 $\theta = 89^\circ$

Fig. 74 Nb-11 at.%Mo at 175 K - Compression
 $\gamma = 12.1\%$ Crystal 309
 $\theta = 80^\circ$

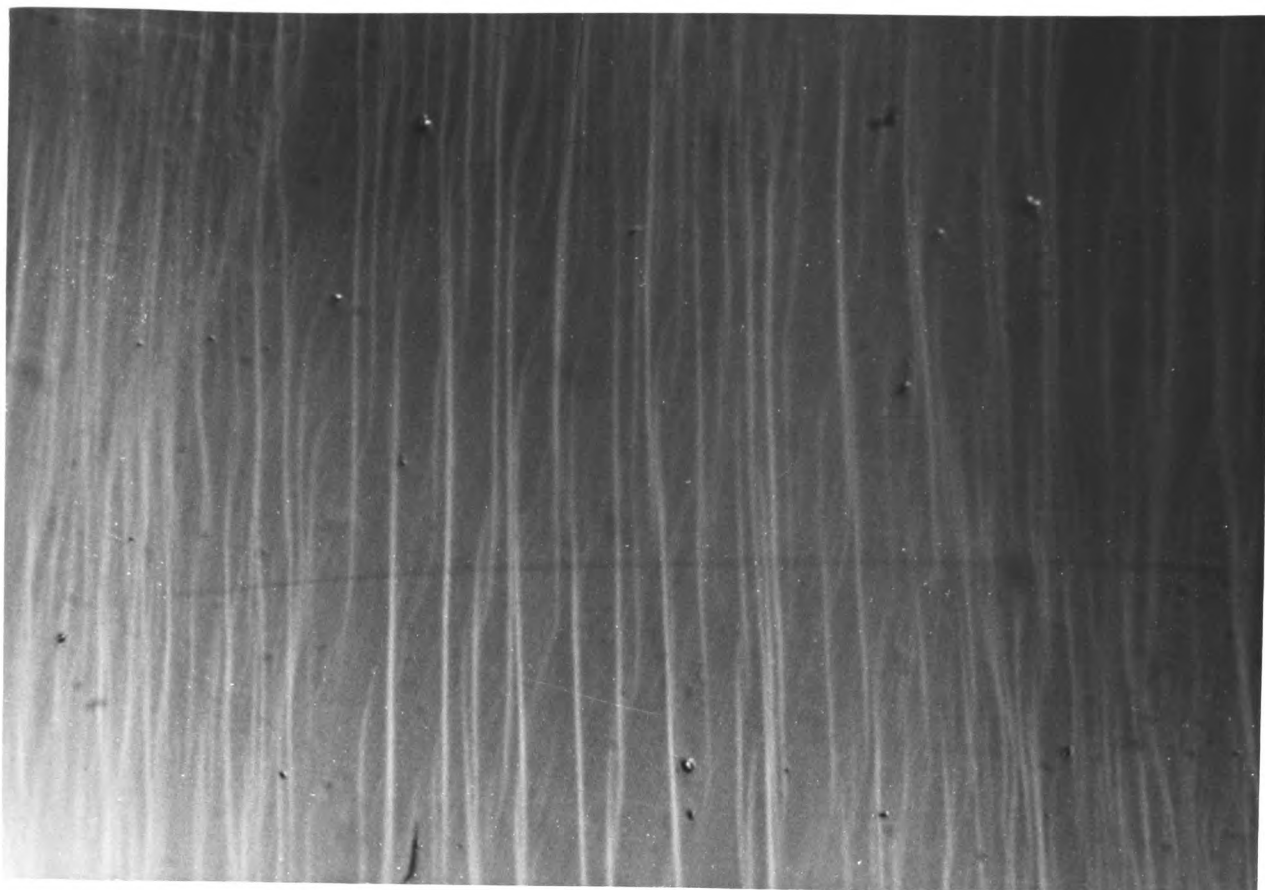


Fig. 72

50 μ m



Fig. 73

50 μ m



Fig. 74

50 μ m

Fig. 75 Nb-11 at.%Mo at 77 K - Compression

$\gamma = 3.5\%$ Crystal 309

$\theta = 105^\circ$

Fig. 76 Nb-11 at.%Mo at 77 K - Compression

$\gamma = 3.5\%$ Crystal 309

$\theta = 11^\circ$



Fig. 75

50 μ m

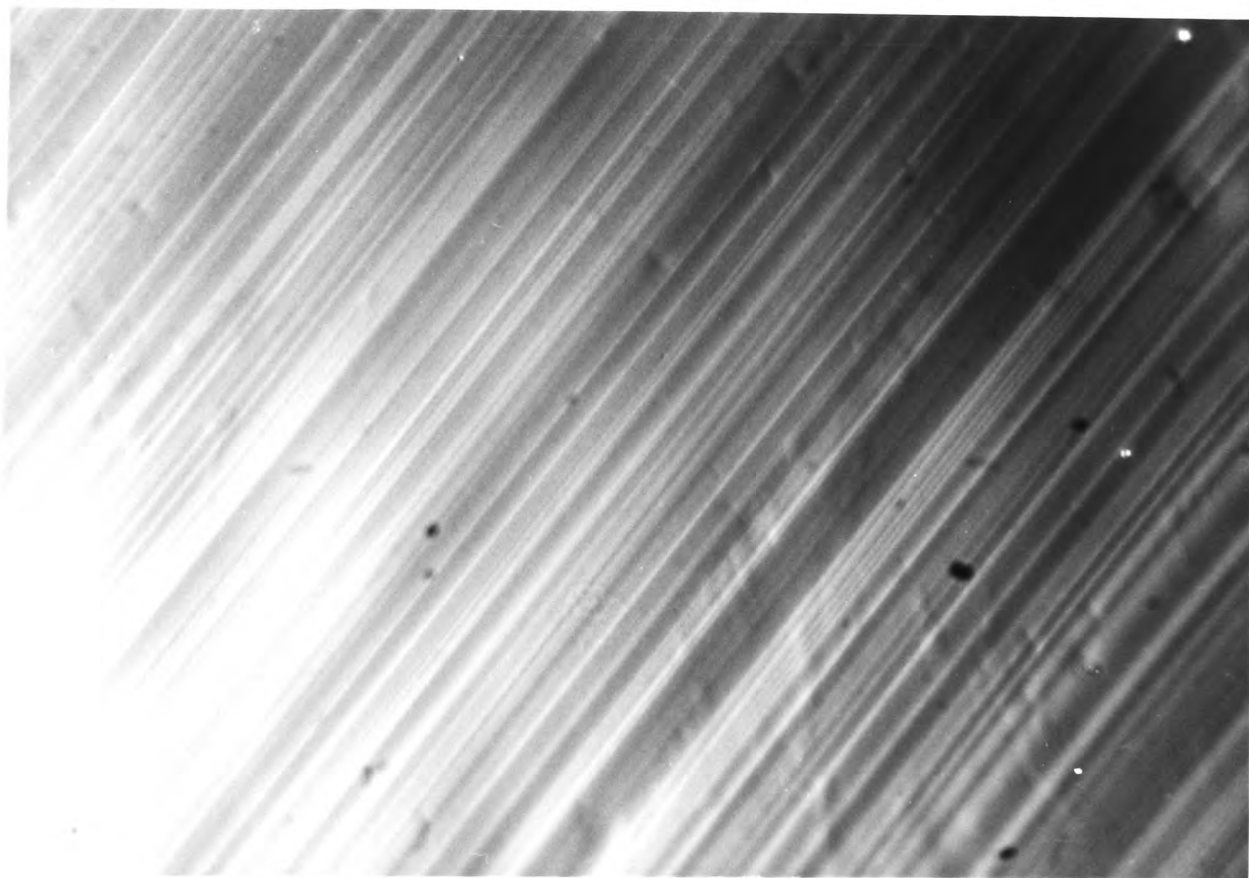


Fig. 76

50 μ m

Fig. 77 Nb-16 at.%Mo at 290 K - Compression

$$\gamma = 21.1\%$$

$$\theta = 94^\circ$$

Fig. 78 Nb-16 at.%Mo at 290 K - Compression

$$\gamma = 21.1\%$$

$$\theta = 11^\circ$$



Fig. 77

50 μ m

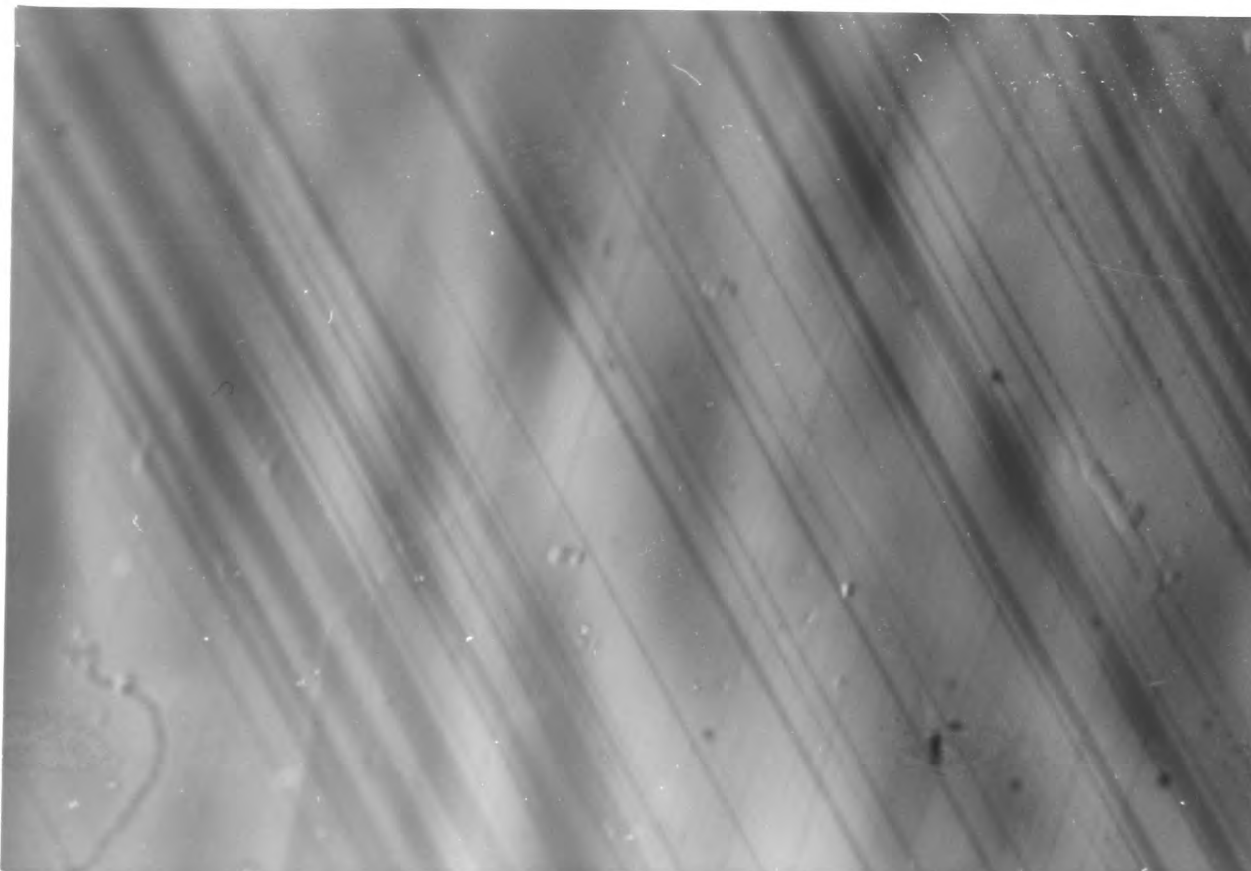


Fig. 78

50 μ m

Fig. 79 Nb-16 at.%Mo at 175 K - Compression

$$\gamma = 14.6\%$$

$$\theta = 91^\circ$$

Fig. 80 Nb-16 at.%Mo at 77 K - Compression

$$\gamma = 13.3\%$$

$$\theta = 99^\circ$$

Fig. 81 Nb-16 at.%Mo at 20.4 K - Compression

$$\gamma = 16.7\%$$

$$\theta = 80^\circ$$



Fig. 79

50μm

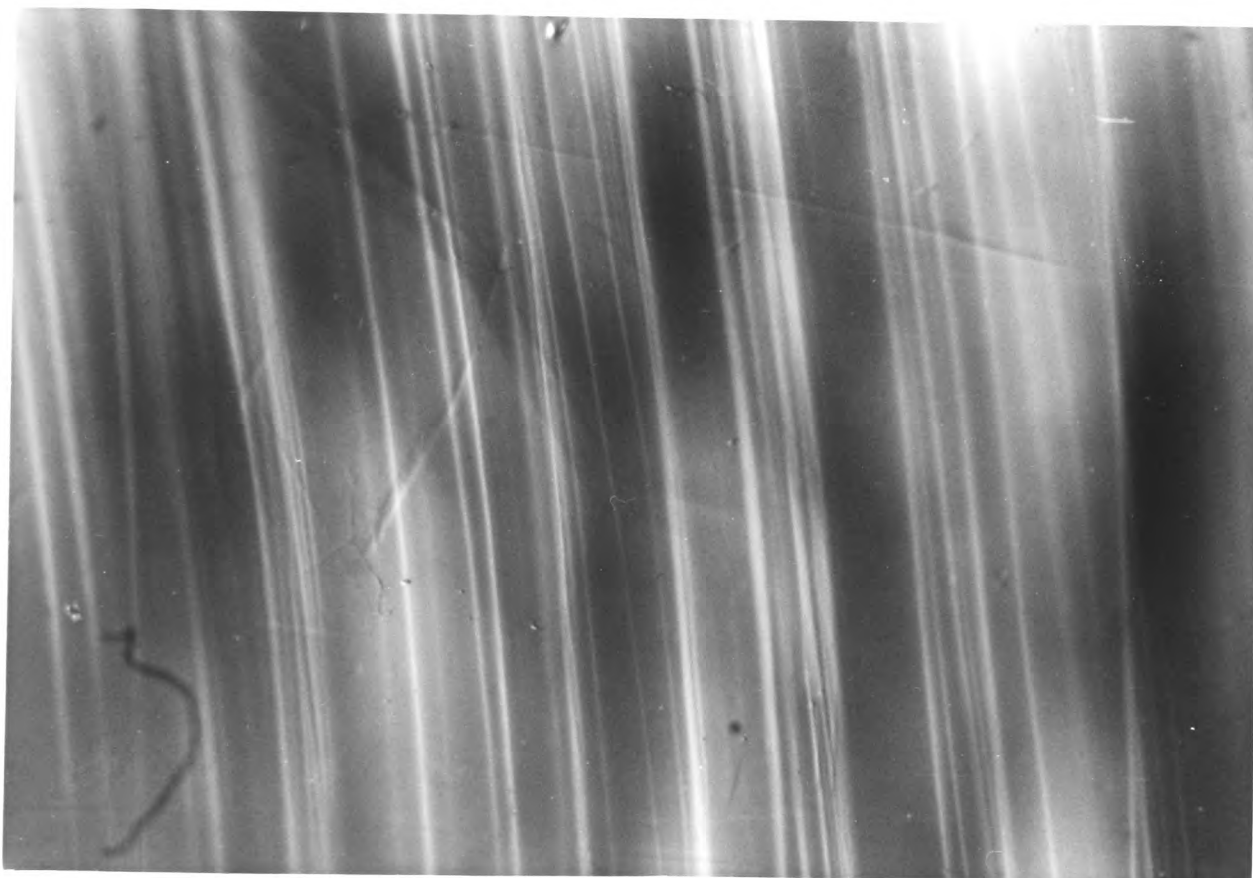


Fig. 80

50μm

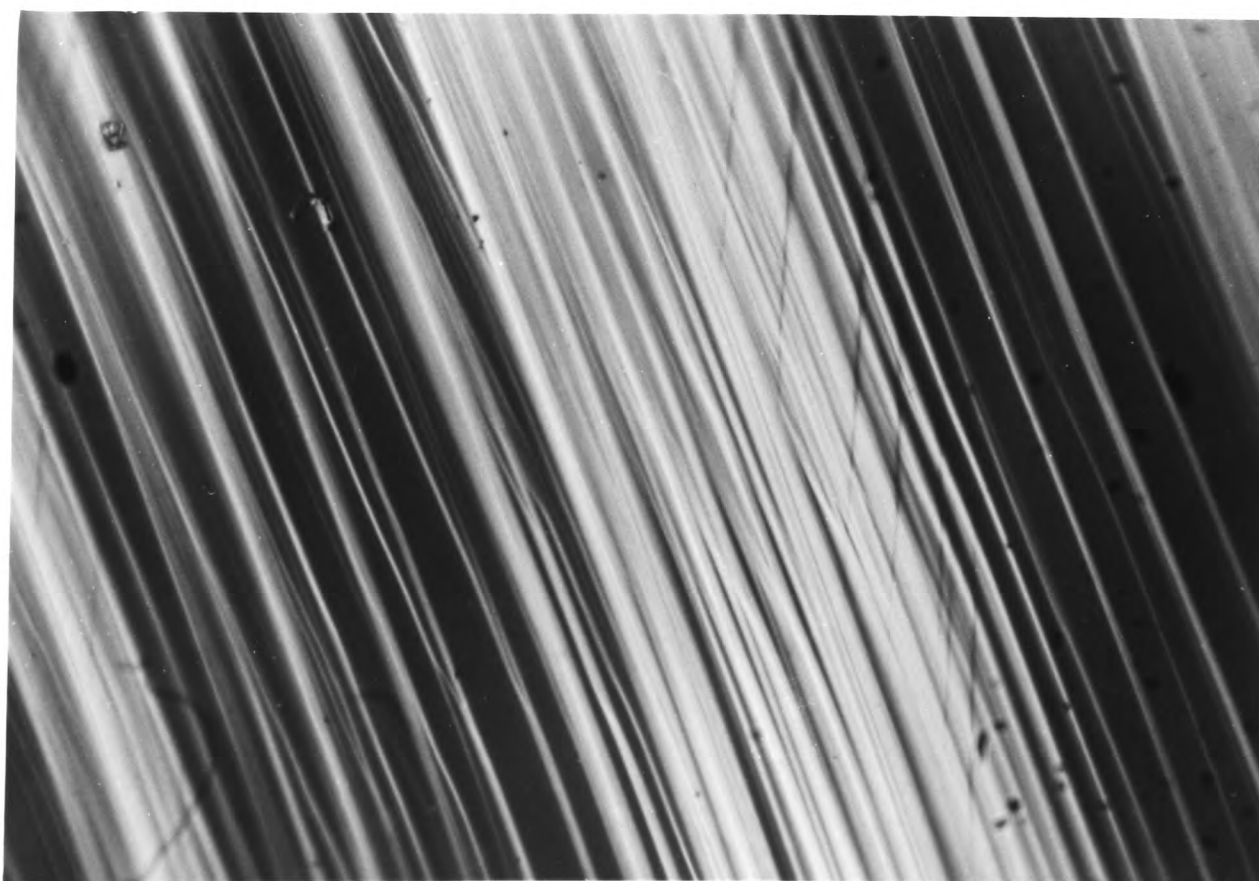


Fig. 81

50μm

Fig. 82 Nb-16 at.%Mo at 8 K - Compression

$$\gamma = 14.5\%$$

$$\theta = 87^\circ$$

Fig. 83 Nb-16 at.%Mo at 8 K - Compression

$$\gamma = 14.5\%$$

$$\theta = 35^\circ$$

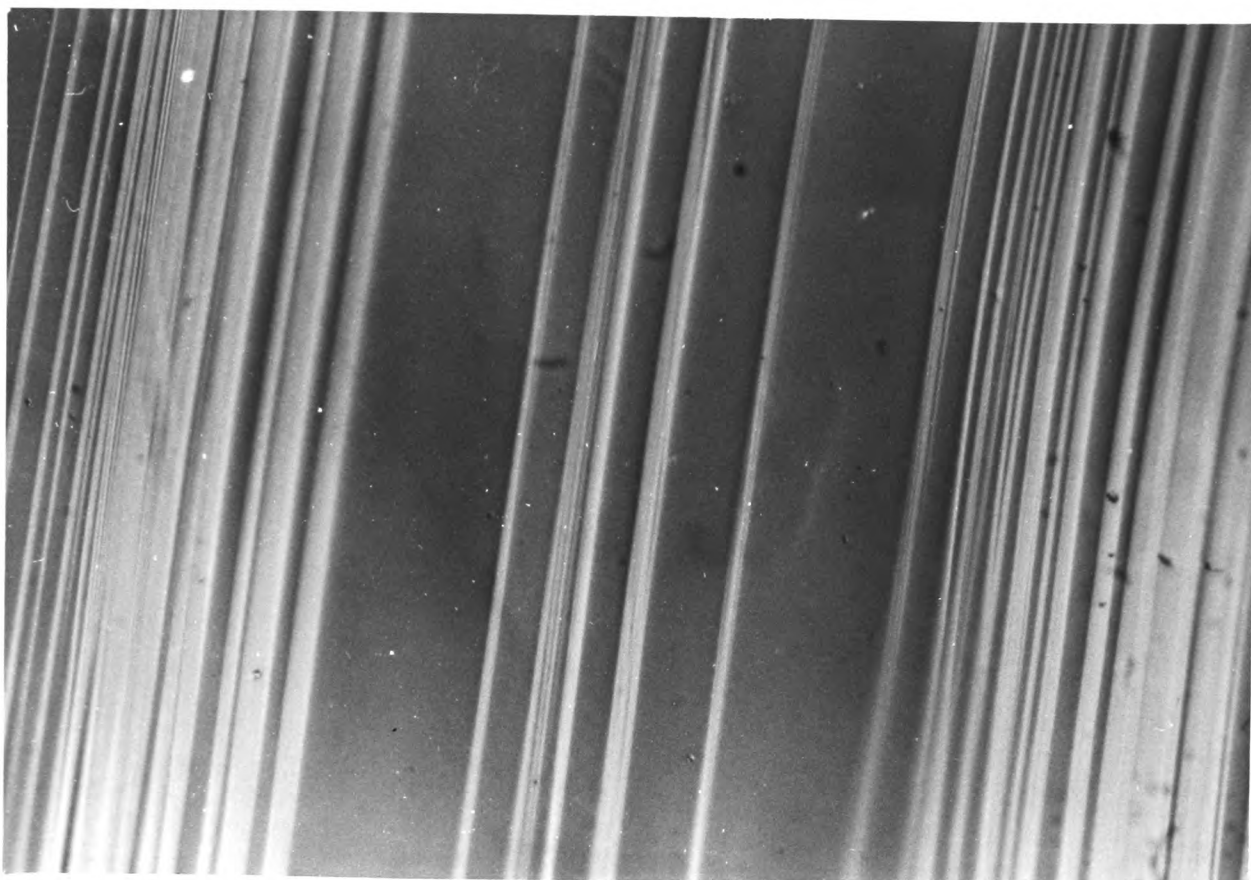


Fig. 82

50 μ m

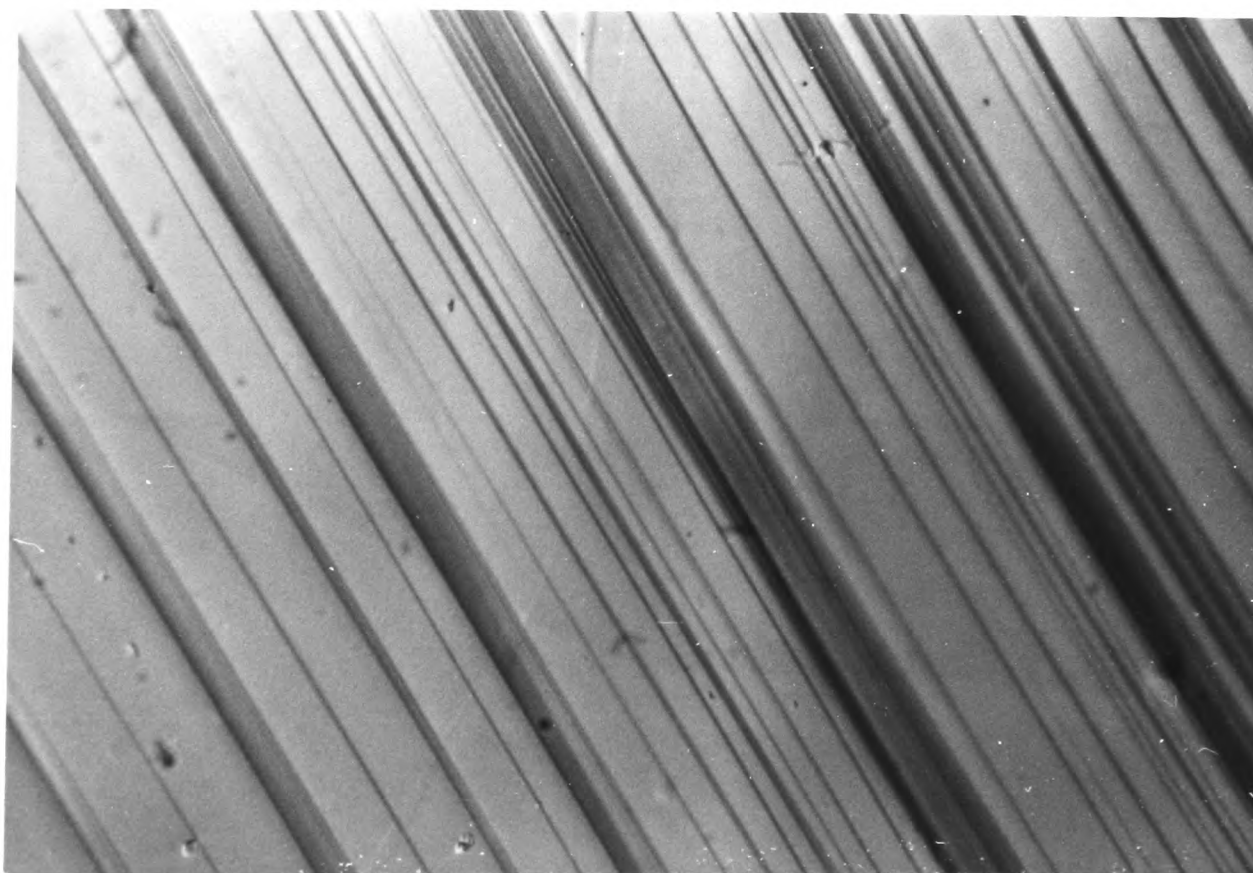


Fig. 83

50 μ m

Fig. 84 Nb at 77 K - Tension

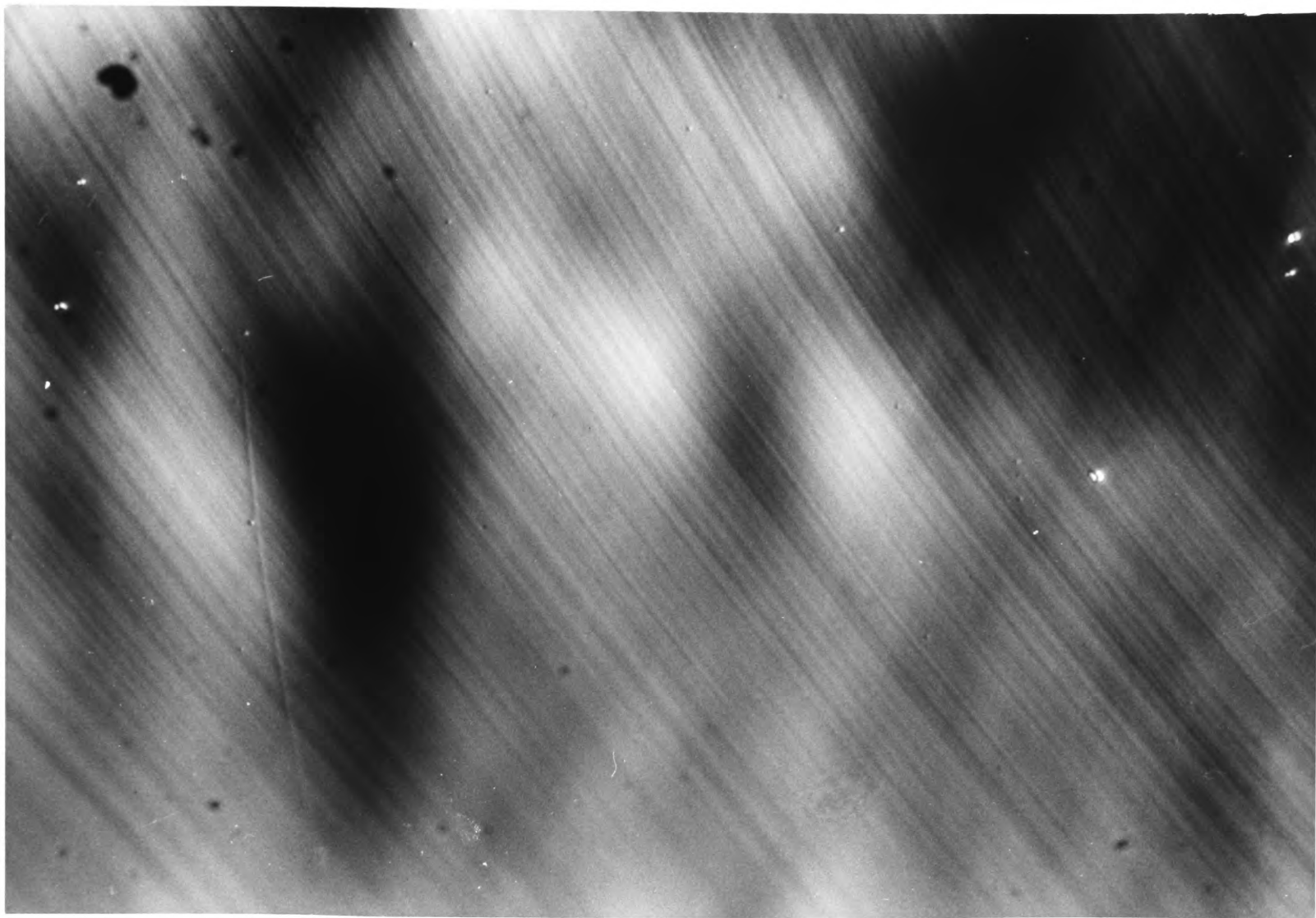
$$\epsilon = 1.8\%$$

$$\theta = 174^\circ$$

Fig. 85 Nb at 40 K - Tension

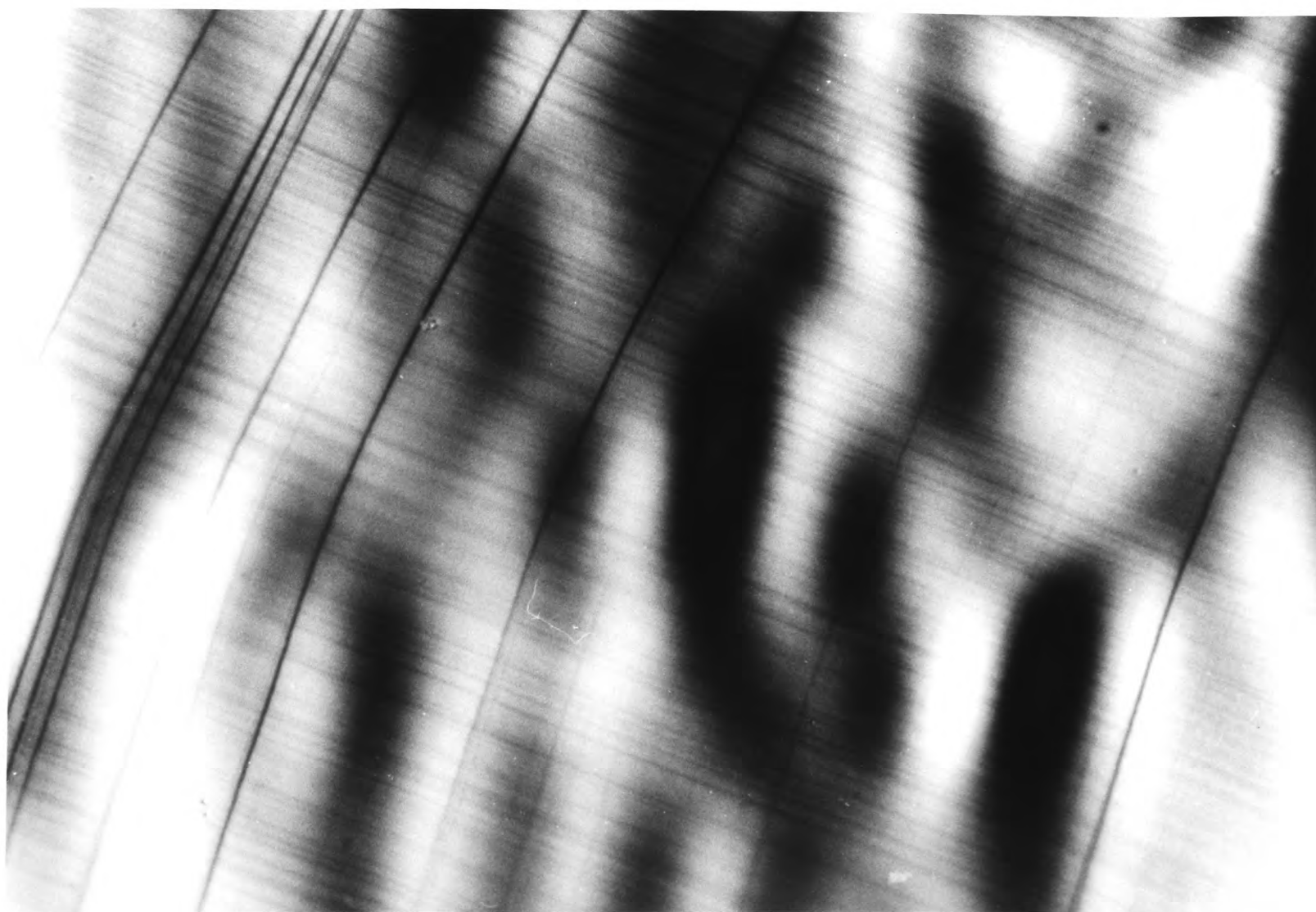
$$\epsilon = 3.0\%$$

$$\theta = 144^\circ$$



50 μm

Fig.84



50 μm

(101) (0 $\bar{1}$ 1)

Fig.85

Fig. 86 Nb-10 at.%Ta at 77 K - Tension

$\epsilon = 4.0\%$ Crystal 312

$\theta = 141^\circ$

Fig. 87 Nb-10 at.%Ta at 77 K - Tension

$\epsilon = 4.0\%$ Crystal 312

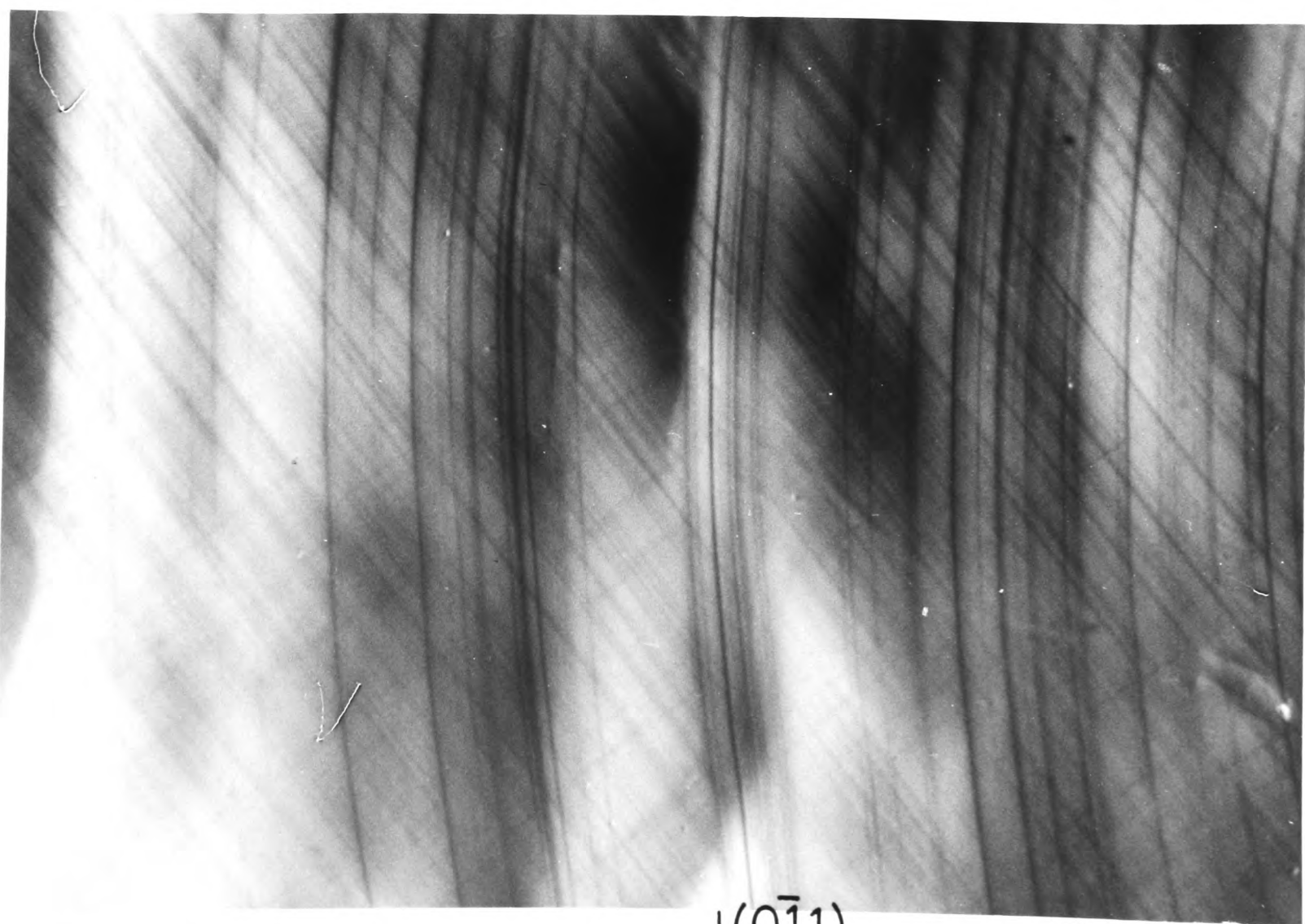
$\theta = 168^\circ$



50 μm

$(0\bar{1}1)$ $(\bar{1}01)$

Fig.86



50 μm

$(0\bar{1}1)$
 (011) $(\bar{1}01)$

Fig.87

Fig. 88 Nb-10 at.%Ta at 50 K - Tension

ϵ = 9.4% Crystal 312

θ = 153°

Fig. 89 Nb-10 at.%Ta after straining at 290 K - 29 K - 22 K - Tension

ϵ at 290 K = 0.4% Crystal 362

ϵ at 29 K = 3.2%

ϵ at 22 K = 3.6%

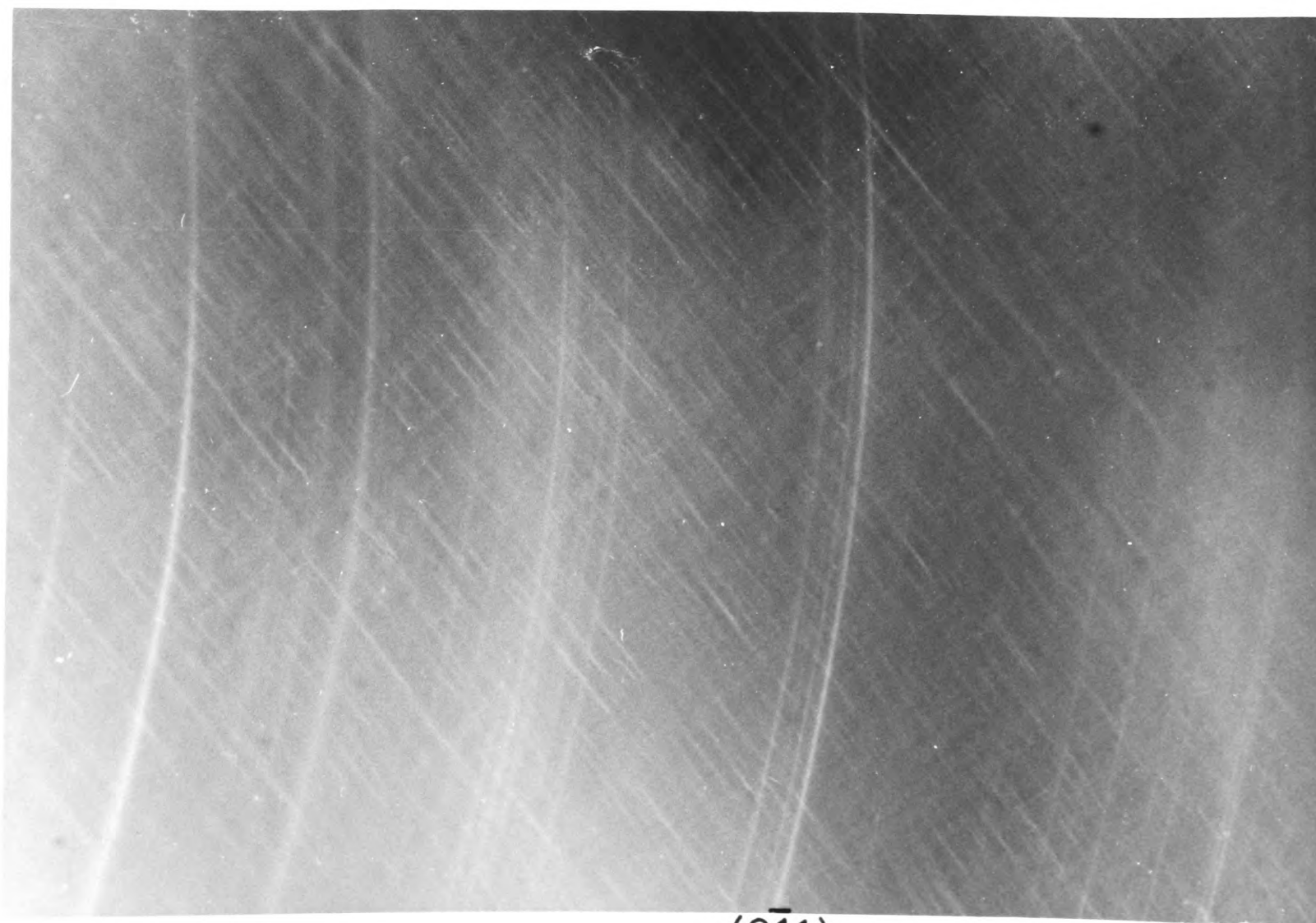
θ = 166°



50 μm

$(0\bar{1}1)$ $\wedge$ $(\bar{1}01)$

Fig. 88



50 μm

(101) $\begin{matrix} | \\ (0\bar{1}1) \\ | \end{matrix}$ $(\bar{1}01)$

Fig. 89

Fig. 90 Nb-1 at.%Mo at 77 K - Tension
 $\epsilon = 0.9\%$ Crystal 326
 $\theta = 20^\circ$

Fig. 91 Nb-1 at.%Mo at 77 K - Tension
 $\epsilon = 0.9\%$ Crystal 326
 $\theta = 59^\circ$

Fig. 92 Nb-1 at.%Mo at 77 K (prestrained) - Tension
 ϵ at 290 K = 0.5% Crystal 370
 ϵ at 77 K = 3.8%
 $\theta = 143^\circ$

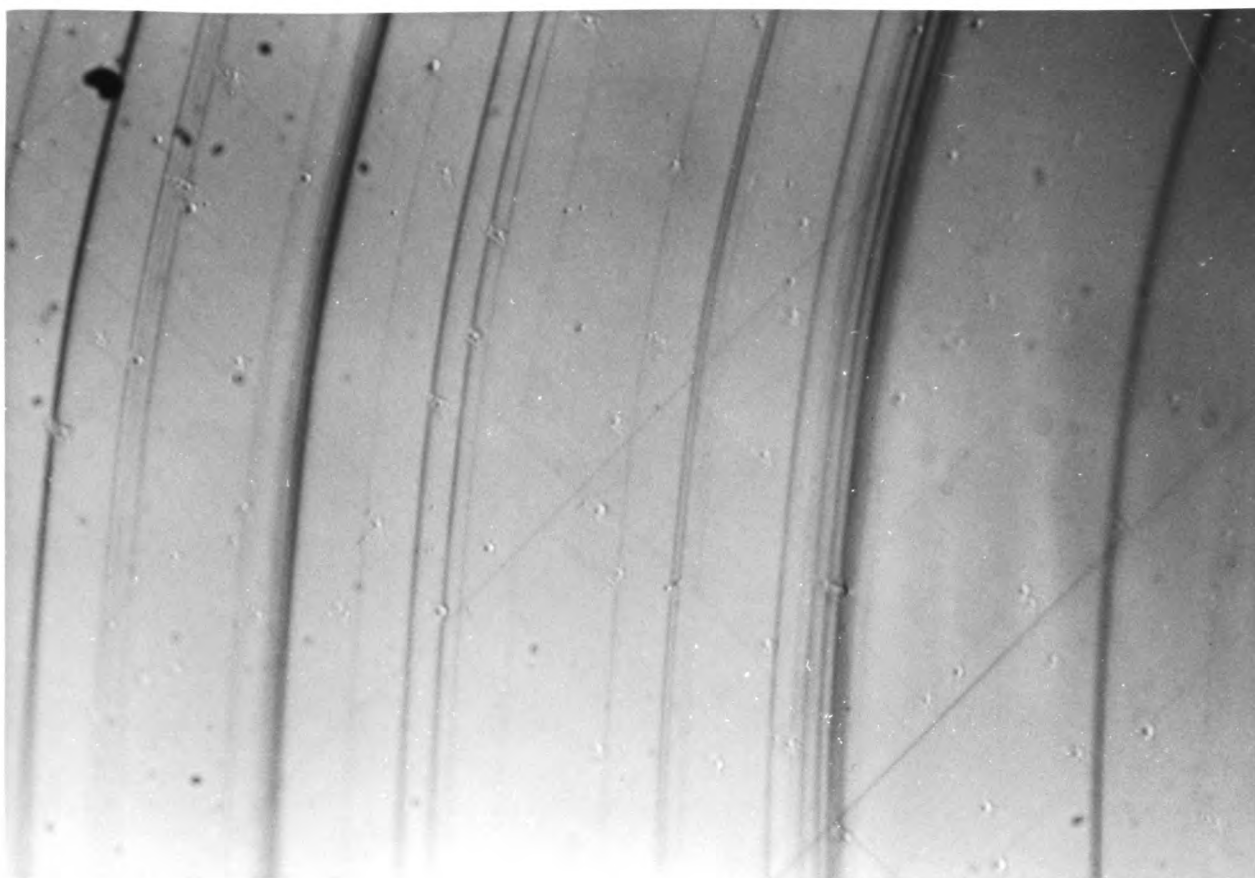


Fig.90

(101)
 $(\bar{1}01)$
 $(0\bar{1}1)$

50 μm



Fig.91

$(0\bar{1}1)$
 $(\bar{1}01)$

50 μm



Fig.92

$(\bar{1}01)$ (011)
 $(0\bar{1}1)$

50 μm

Fig. 93 Nb-1 at.%Mo at 40 K (prestrained) - Tension

ϵ at 290 K = 0.5%

Crystal 370

ϵ at 40 K = 5.4%

θ = 15°

Fig. 94 Nb-1 at.%Mo at 40 K (prestrained) - Tension

ϵ at 290 K = 0.5%

Crystal 370

ϵ at 40 K = 5.4%

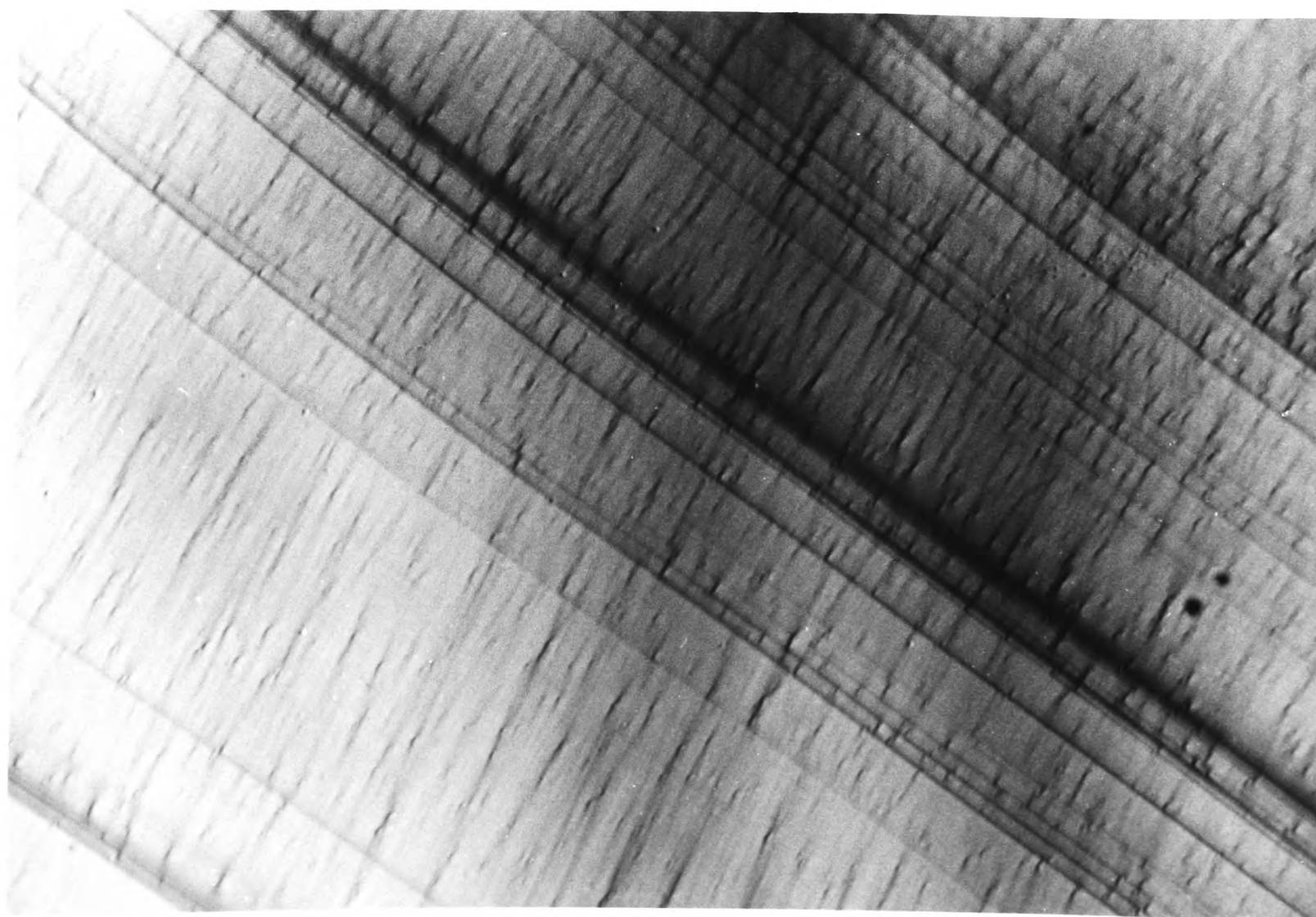
θ = 44°



50 μm

$(\bar{1}01)$
 $(0\bar{1}1)$ (101)

Fig.93



50 μm

$(\bar{1}01)$ $(0\bar{1}1)$

Fig.94

Fig. 95 Nb-1 at.%Mo at 22 K (prestrained) - Tension

ϵ at 290 K = 0.6% Crystal 375

ϵ at 22 K = 7.2%

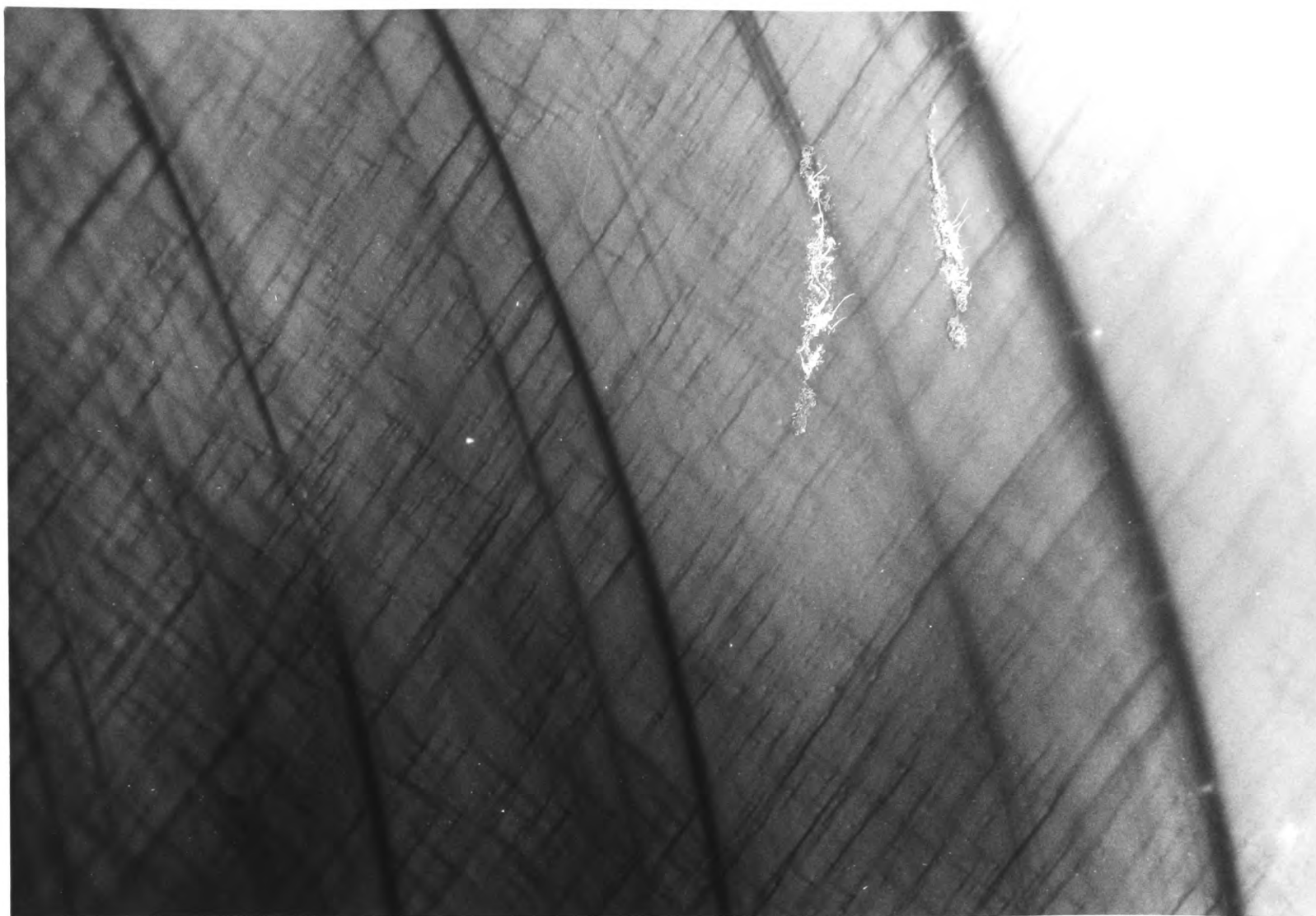
θ = 30°

Fig. 96 Nb-1 at.%Mo at 22 K (prestrained) - Tension

ϵ at 290 K = 0.6% Crystal 375

ϵ at 22 K = 7.2%

θ = 82°



50 μm

$(0\bar{1}1)$
 $(\bar{1}01)$ (101)

Fig.95



50 μm

$(\bar{1}01)$ $(0\bar{1}1)$

Fig.96

Fig. 97 (a) The free energy barrier profile for
thermally activated dislocation motion.

(b) The back stress-area slipped profile for
the free energy barrier in (a).

Fig.97

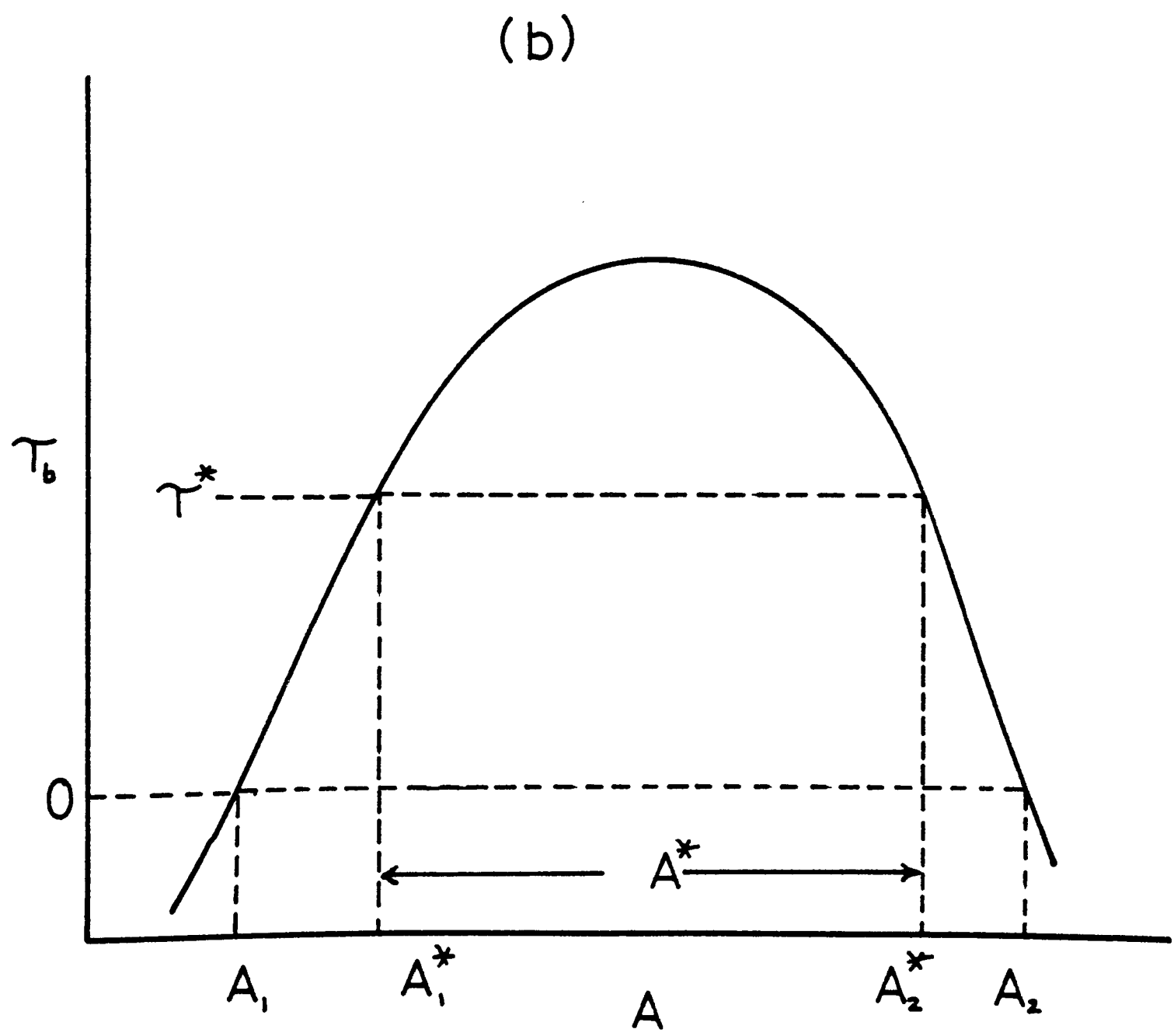
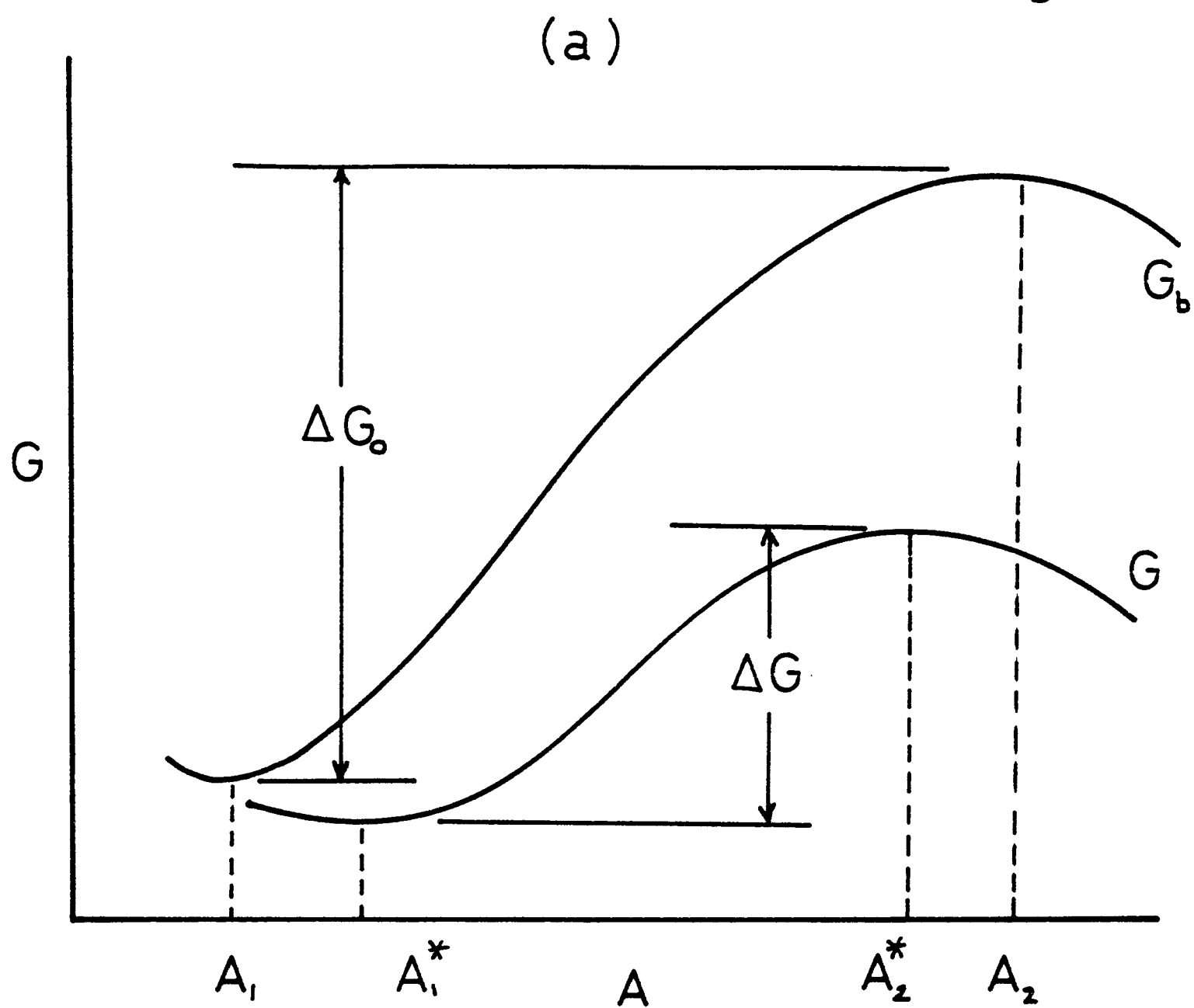


Fig. 98 Activation volumes in compression for Nb and the
Nb-Ta alloys.

The curve for Nb-4 at.%Ta is omitted for clarity.
However it lies just below the curve for Nb-10 at.%Ta
at all stresses.

Fig.98

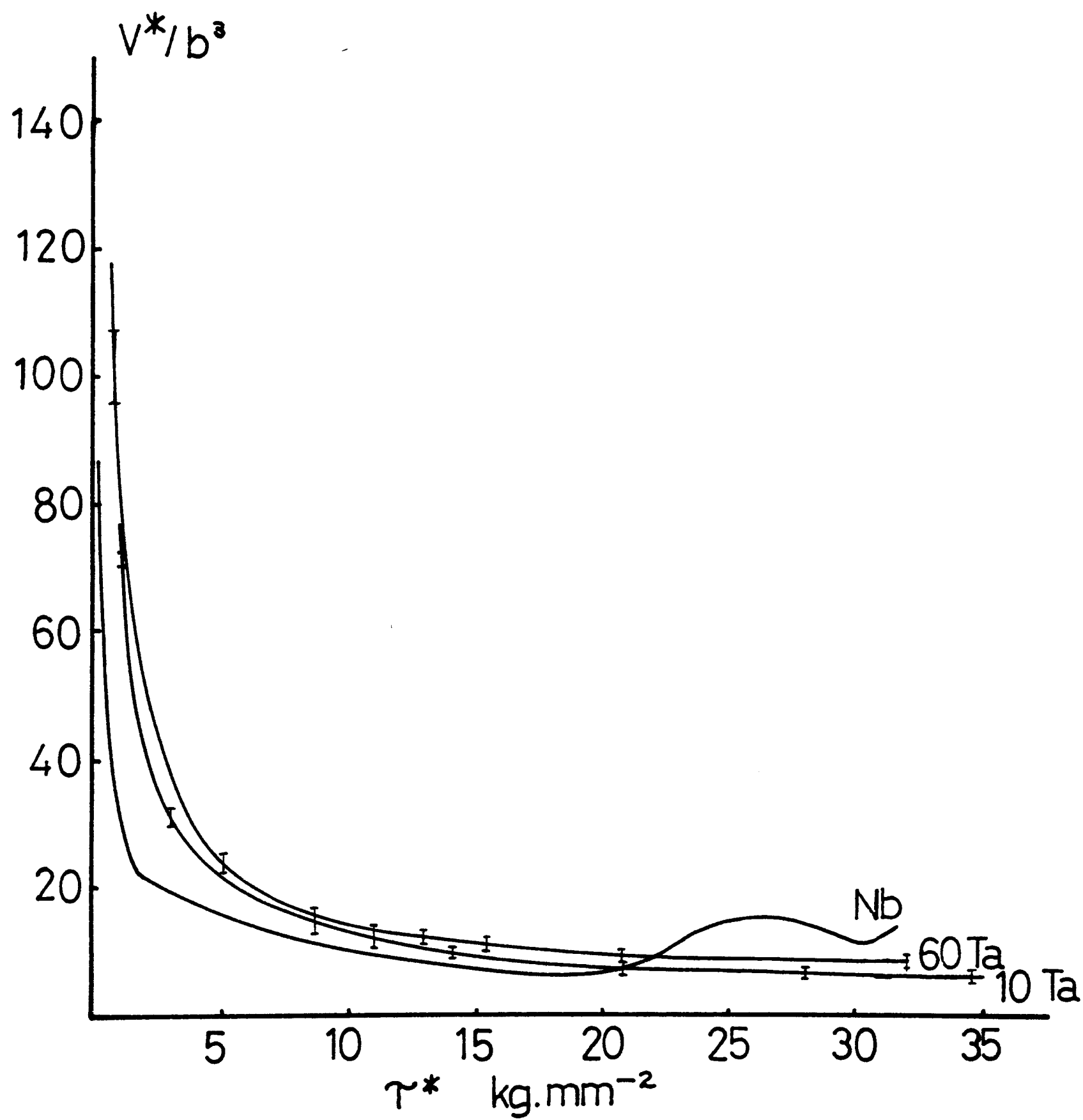


Fig. 99 Activation volumes in compression for Nb and the
Nb-Mo alloys.

- | | |
|----|---------------|
| 1. | Nb |
| 2. | Nb - 1 at.%Mo |
| 3. | Nb - 2 at.%Mo |
| 4. | Nb - 3 at.%Mo |
| 5. | Nb - 5 at.%Mo |
| 6. | Nb -11 at.%Mo |
| 7. | Nb -16 at.%Mo |

Fig.99

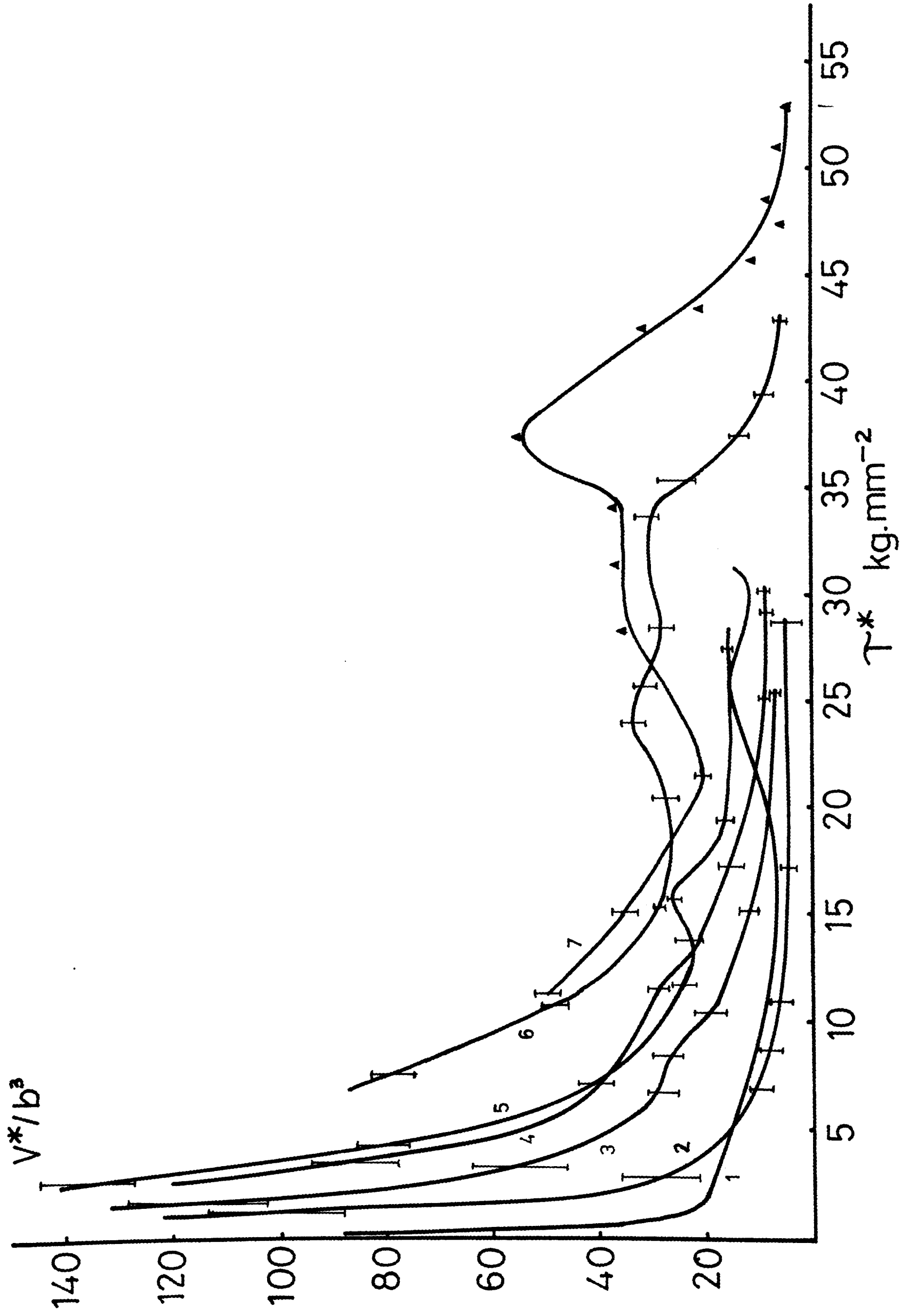


Fig. 100 Activation enthalpy - thermal stress in
compression for Nb and Nb-Ta alloys.

Fig.100

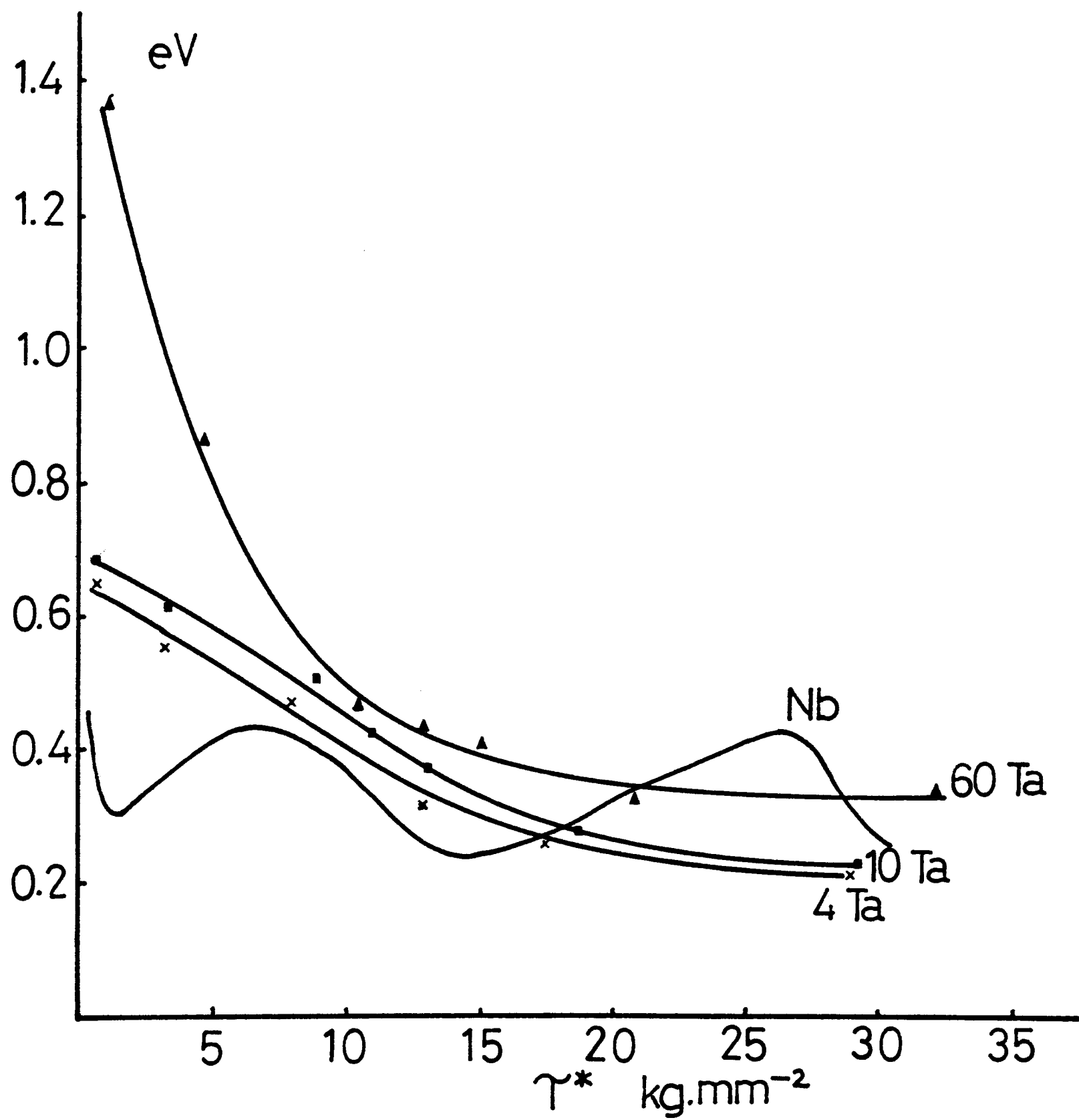


Fig. 101 Activation enthalpy - thermal stress in compression
for Nb and the Nb-Mo alloys.

Fig.101

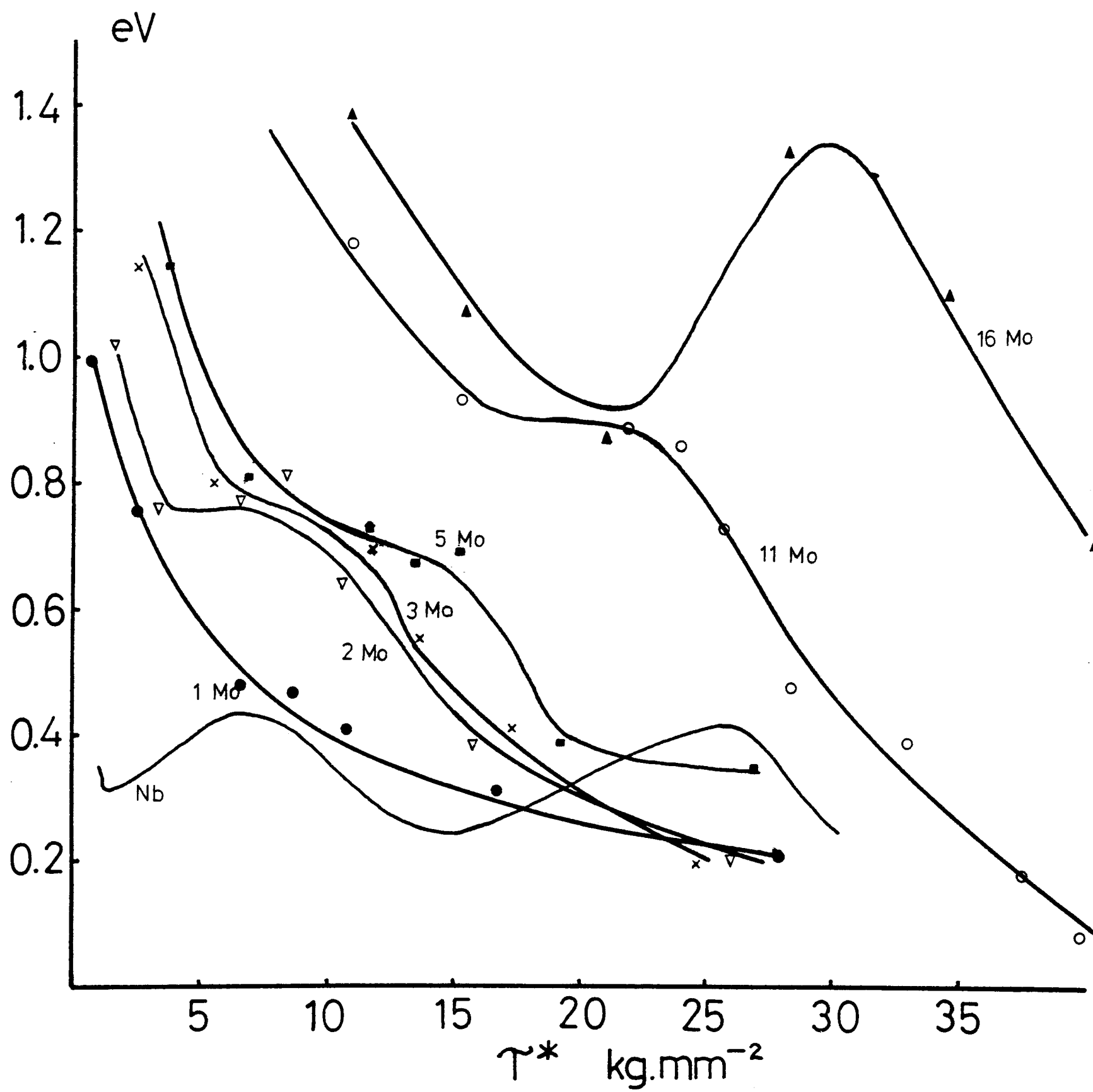


Fig. 102 Total activation enthalpy (ΔH_0) - thermal stress
in compression for Nb-Ta and Nb-Mo alloys.

Although no error bars have been included in the figure for reasons of clarity, the spread in the ΔH_0 values is $\pm 5\%$ for the 11 and 16 at.%Mo alloys and $\pm 10\%$ for the other alloys. This represents the spread in the actual experimental results and it is not an error bar arrived at statistically.

Fig.102

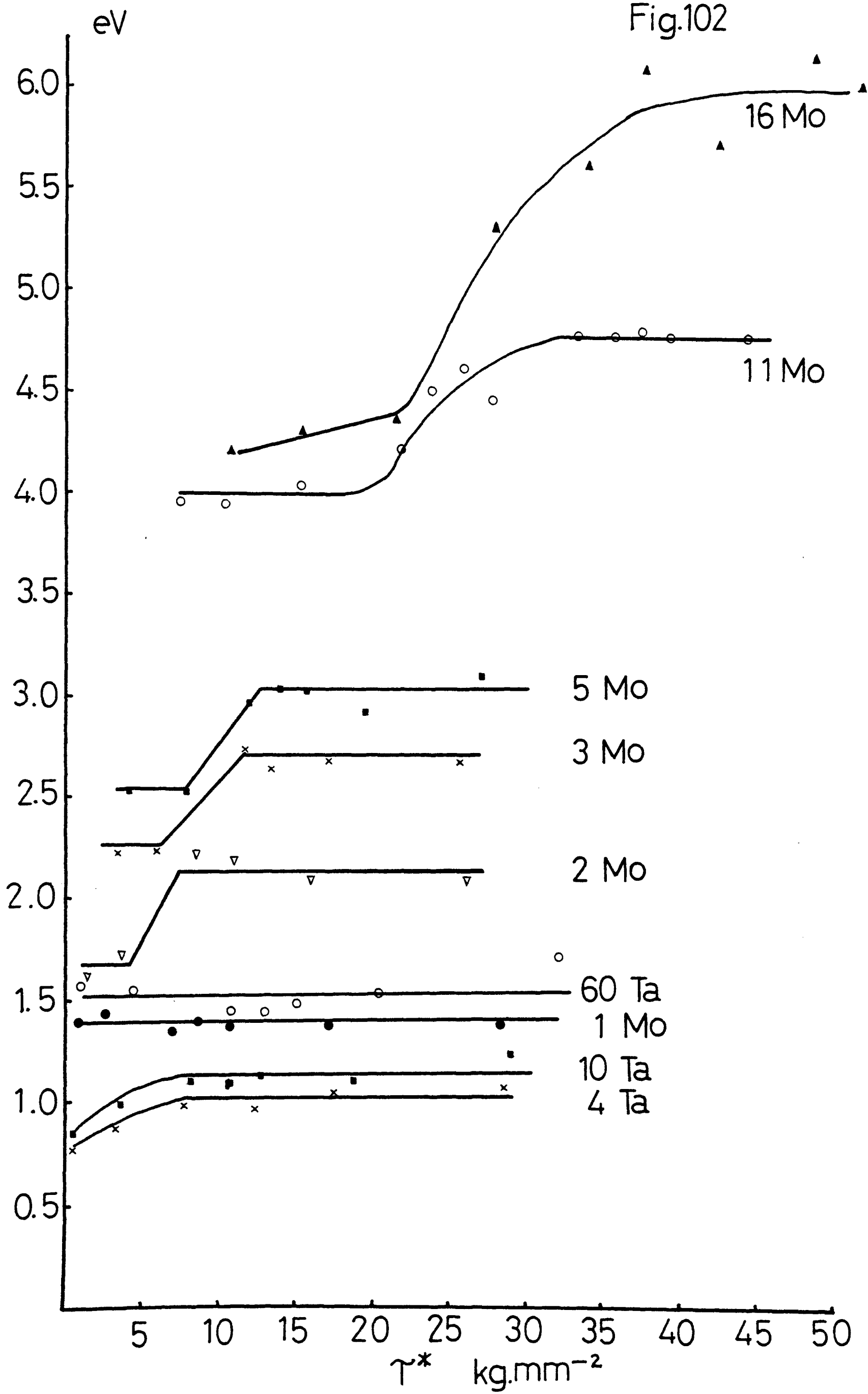


Fig. 103 Schematic back stress-area slipped for the
two postulated mechanisms, M1 and M2.

Fig.103

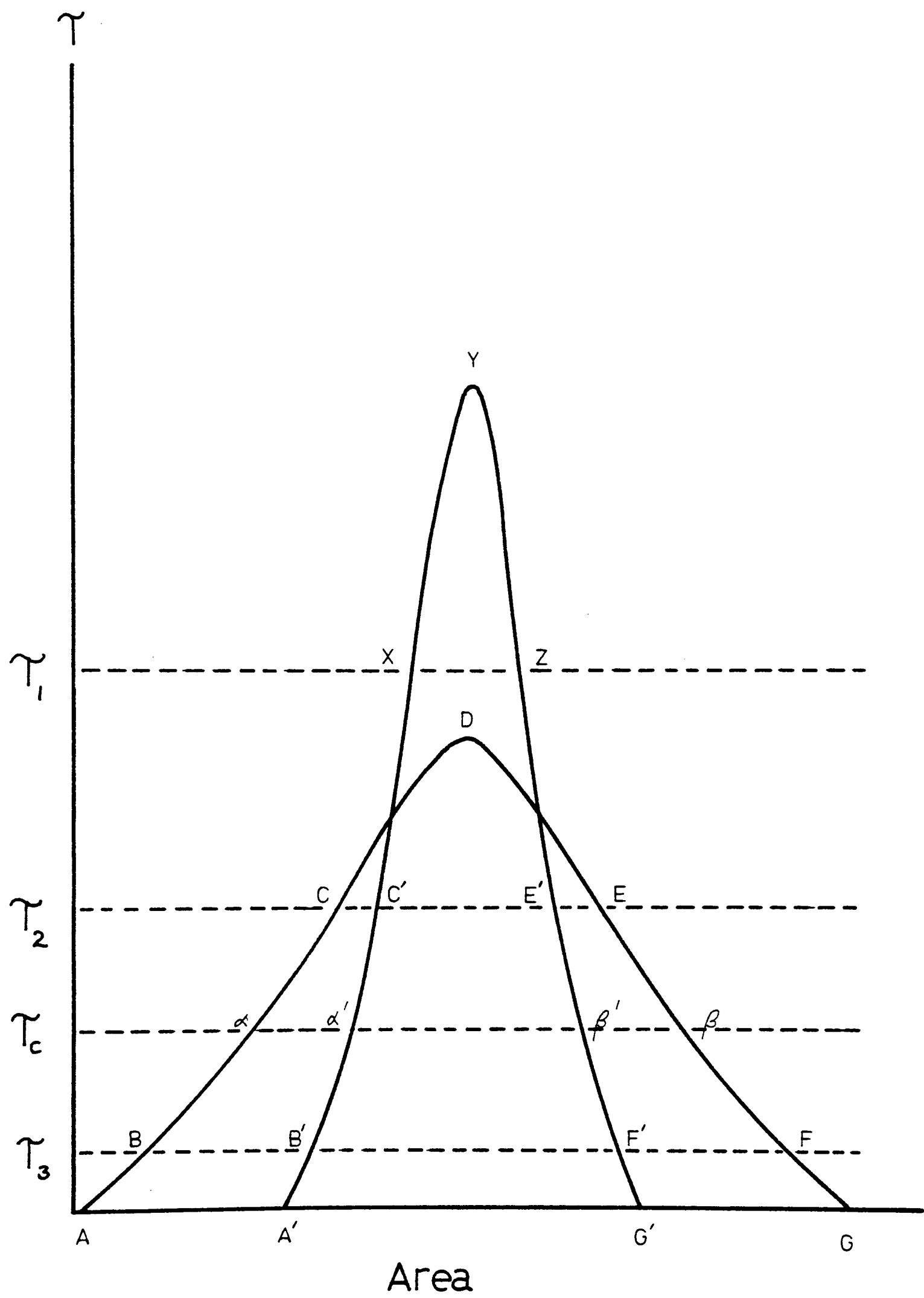


Fig. 104 Schematic activation enthalpy-stress for two mechanisms, M1 and M2.
The solid line indicates the actual behaviour.
The full variation of each mechanism with the stress is continued by the dashed lines.

Fig. 105 Schematic back stress-area slipped in the vicinity of the M1 - M2 transition point.

Fig.104

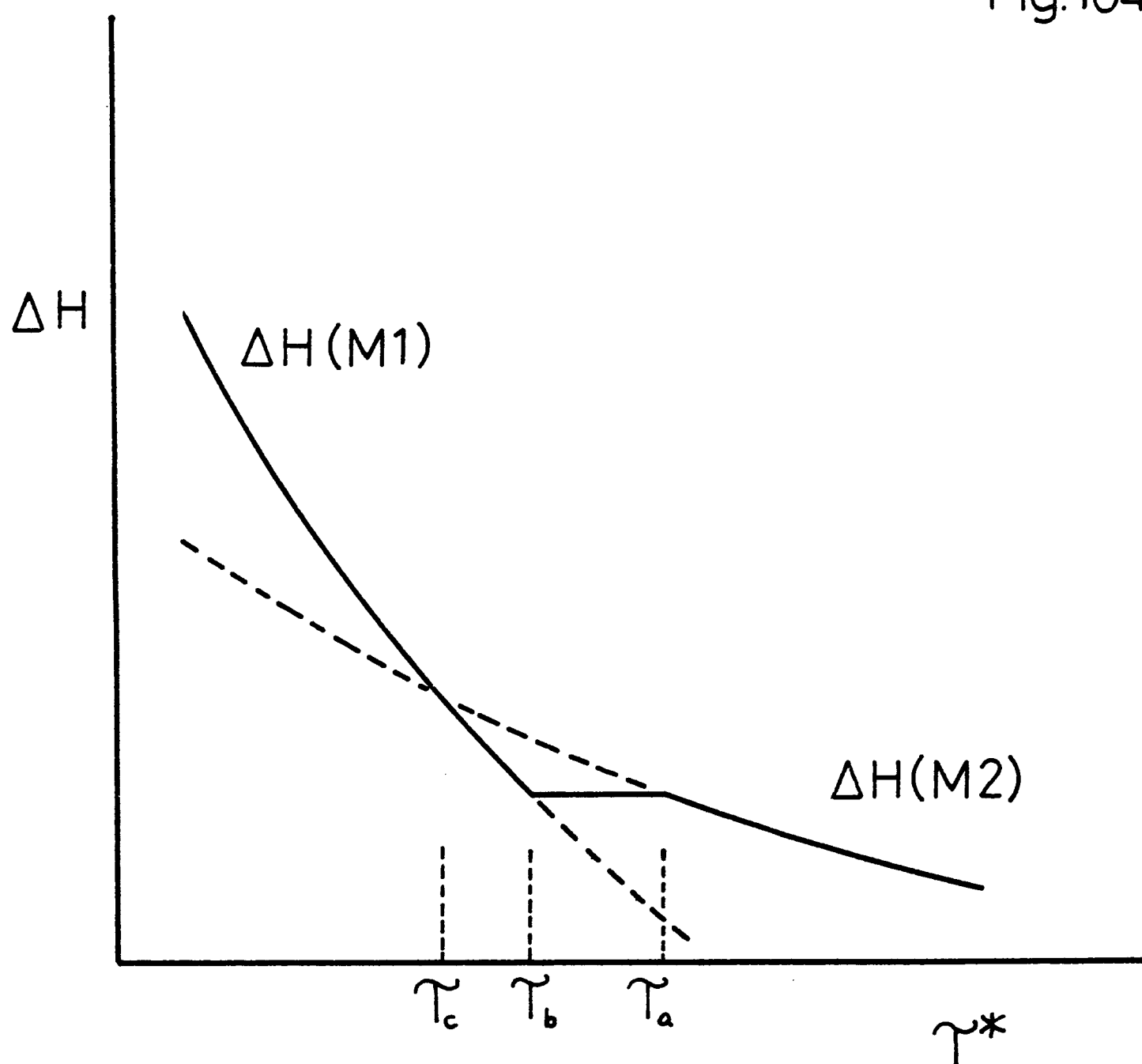


Fig.105

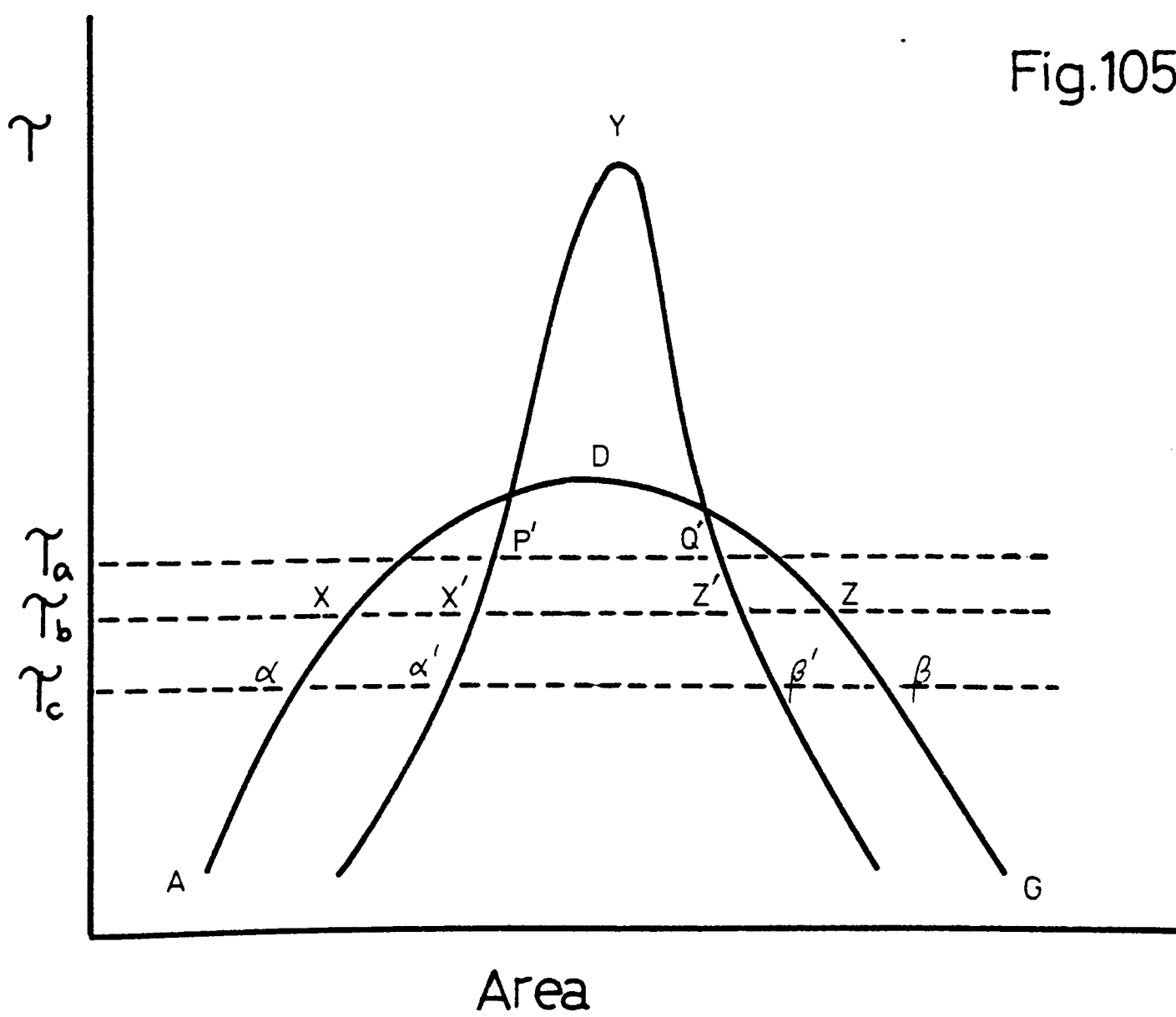


Fig. 106 Activation volume-stress in tension for
Nb and the Nb-Ta and Nb-Mo alloys.

Fig.106

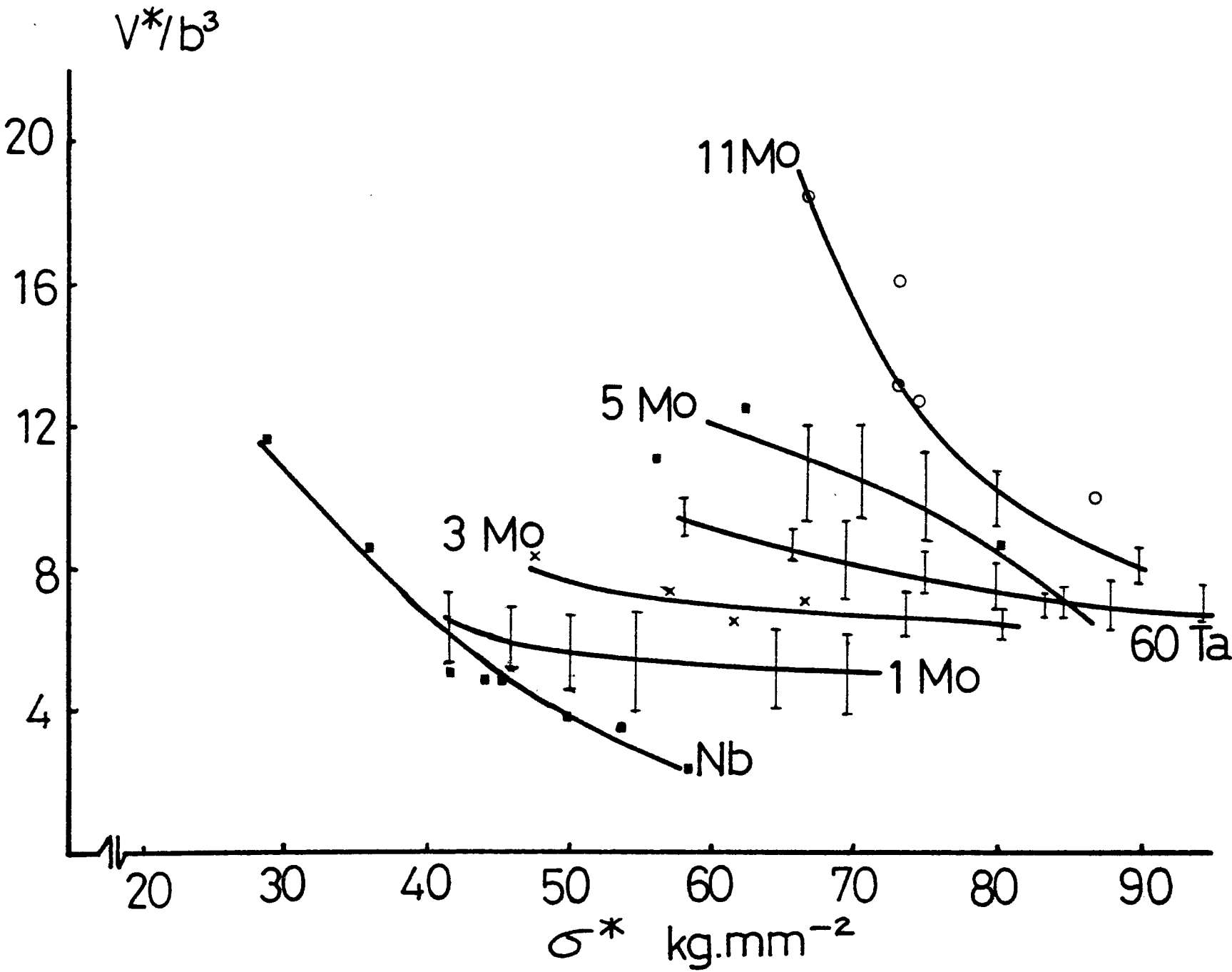


Fig. 107 Reduced thermomechanical parameter-reduced
strain rate for thermal instability analysis.

Point 1	-	30 K
Point 2	-	20 K
Point 3	-	10 K
Point 4	-	8 K

Fig.107

