

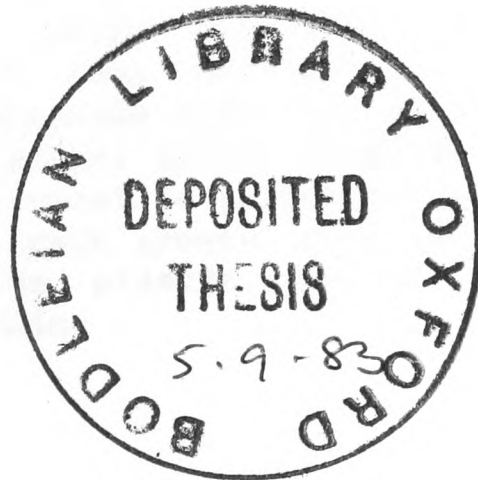
Fracture Studies in Superalloys

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A thesis submitted for the degree of Doctor of Philosophy in the
University of Oxford, Hilary Term, 1983.



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D.Phil Thesis
Hilary Term, 1983.

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ABSTRACT

This research is concerned with the effects of temperature and frequency on the fatigue crack propagation behaviour. The fatigue behaviour at elevated temperature has been reviewed with emphasis on the mechanisms of propagation proposed to explain the effects of temperature and frequency.

The crack propagation characteristics of a single crystal superalloy have been investigated at various temperatures and frequencies. Determination of the fatigue crack propagation rates was combined with the measurement of crack tip plastic zone sizes and observation of the deformation behaviour.

At elevated temperature, the fatigue crack propagation rates were determined to be dependent on the frequency of loading. At low and high crack growth rates increased crack propagation rates were associated with higher cycling frequencies. At intermediate crack growth rates, the highest propagation rates were associated with the lowest frequency.

Selected area channelling pattern techniques were used to determine the plastic zone sizes and revealed that at high frequencies ($>10\text{Hz}$) the plastic zone sizes at elevated and ambient temperature are similar. With decreasing frequency at elevated temperature creep may lead to increased plastic zone sizes but may also affect the crack tip geometry thereby restricting further effects.

At room temperature crack propagation is observed to occur by shear of the γ' precipitate on $\langle 110 \rangle$ $\{111\}$ resulting in $\{111\}$ crystallographic facets on the fracture surface. At elevated temperature ($>600^\circ\text{C}$) however, the crack growth mechanism changes and propagation takes place on $\{100\}$. Observations of the dislocation structure indicate that deformation is concentrated within the γ matrix. It is proposed that the observed crack propagation in the γ matrix on $\{100\}$ arises by the constraints imposed by the surrounding γ' precipitate. A model is presented for the frequency dependence of the intermediate crack growth rate in terms of the observed strain gradients within the plastic zone and the achievement of a critical local fracture strain.

Acknowledgements

Many people have helped me in the course of this research and I should like to express my thanks to them all. In particular I should like to thank the following:

Professor Sir Peter Hirsch F.R.S. for the provision of laboratory facilities.

The Science and Engineering Research Council and the Ministry of Defence for financial support.

My supervisor Dr. J.W. Martin for his advice throughout the project, and Dr. M. Winstone for useful discussions.

The National Gas Turbine Establishment for the provision of material.

The members of the laboratory workshop for their assistance.

The other members of the J.W.M. group especially Ian Baker, John Blind, Bill Clegg, Mike Cowling, Lyndon Edwards and Adrian Hopgood for their help, encouragement and friendship during the course of this project.

Bill Clegg and Adrian Hopgood for proof reading this thesis.

Glynis Edwards for her patient and careful typing of this thesis.

Jeff Crompton

March, 1983.

Preface

This thesis is an account of research carried out in the Department of Metallurgy and Science of Materials at the University of Oxford between October 1979 and March 1983. The work reported is original, and is not substantially the same as any thesis submitted in this or any other university. Where the work of other authors has been included in the text this has been acknowledged and its source given in an alphabetical list of references at the end of the thesis. Part of this work has been presented at the 4th European Conference on Fracture, Leoben, Austria, September 1982 and at the European Conference on High Temperature Alloys for Gas Turbines, Liege, Belgium, October 1982.

Index

Abstract

Acknowledgements

Preface

Chapter 1 : Fracture Studies in Superalloys	1
1.1 Introduction	1
1.2 Crack Characterisation	2
1.2.1 Crack Tip Stresses in an Elastic Body	2
1.2.2 Effects of Strain Hardening	5
1.2.3 Effects of Time Dependent Deformation	6
1.2.4 Summary of Crack Tip Stress Distribution	8
1.3 The Crack Tip Plastic Zone	9
1.3.1 Monotonic Loading	10
1.3.2 Cyclic Loading	15
1.4 Micromechanisms of Fracture	16
1.5 Fatigue Crack Propagation at Ambient Temperature	18
1.5.1 Application of Fracture Mechanics to Fatigue Crack Propagation	18
1.5.2 Threshold and Near Threshold Crack Growth	19
1.5.3 Intermediate Crack Growth	22
1.5.4 High Growth Rates	24
1.5.5 Fatigue Crack Propagation in Superalloys	24
1.6 Elevated Temperature Crack Growth	26
1.6.1 Characterisation of Elevated Temperature Crack Growth	26

1.6.2	Fracture Characteristics of Superalloys at Elevated Temperature	28
1.7	High Temperature Fatigue	29
1.7.1	Frequency Effects	29
1.7.2	Environmental Effects	30
1.7.3	Waveshape Effects	32
1.8	Models for High Temperature Fatigue Crack Growth	34
1.8.1	Environmentally Based Models	35
1.8.2	Creep Based Models	37
1.9	Summary	44
Chapter 2 : Material and Experimental Techniques		46
2.1	Material	46
2.2	Specimen Geometry	46
2.3	Specimen Preparation	48
2.4	Description of the Apparatus	49
2.4.1	Fatigue Machine	49
2.4.2	Operation of the Fatigue Machine	50
2.4.3	Specimen Gripping and Axiality	53
2.4.4	Temperature Control	54
2.5	Measurement of Crack Length	54
2.5.1	Potential Drop Technique	55
2.5.2	Potential Drop Calibration	56
2.6	Crack Growth	58
2.6.1	Fatigue Crack Propagation	58
2.6.2	Measurement of Fatigue Crack Growth Rates	59

2.6.3	Creep Crack Growth	60
2.7	Data Analysis	61
2.8	Plastic Zone Size Measurement	62
2.8.1	Channelling Patterns	63
2.8.2	Effect of Deformation on Channelling Patterns	65
2.8.3	Experimental Determination of Strain Effects	67
2.8.4	Plastic Zone Effects	69
2.9	Metallography	71
2.10	Fractography	71
Chapter 3 : Results		73
3.1	Material	73
3.2	Potential Drop Calibration	73
3.3	Crack Propagation	74
3.3.1	Room Temperature	75
3.3.2	600°C	75
3.3.3	850°C	76
3.3.4	Summary	76
3.4	Fractography	77
3.4.1	Room Temperature	77
3.4.2	600°C	78
3.4.3	850°C	78
3.4.4	Summary	79
3.5	Crack Tip Deformation	79
3.5.1	Effects of Deformation on S.A.C.P.'s	80
3.5.2	Plastic Zone Sizes	80

3.5.3	Strain Distribution within the Plastic Zone	82
3.5.4	Summary	83
3.6	Preliminary Discussion	84
Chapter 4 : Discussion		87
4.1	Plastic Zone Size	87
4.2	Crack Propagation at Low Growth Rates	94
4.3	Crack Growth at High Growth Rates	98
4.4	Crack Growth at Intermediate Growth Rates	102
4.4.1	Room Temperature Crack Growth	102
4.4.2	Elevated Temperature Crack Growth	105
4.4.3	Frequency Dependent Crack Growth	110
Chapter 5 : Summary		116
5.1	Conclusions	116
5.2	Suggestions for Further Work	117
References		

Chapter 1

Fracture Studies in Superalloys

1.1 Introduction

Fatigue failure by the application of an alternating stress or strain to a material generally occurs by the initiation and growth of one or more cracks. In the majority of materials rapid initiation occurs due to the presence of microstructural defects such as grain boundaries or second phase particles. Once initiated, the controlled growth of cracks may occur until the material's fracture toughness is reached, at which point rapid failure occurs.

The presence of a crack within a body alters the local distribution of stress and strain. In most materials yielded zones at the crack tip have been shown to exist, the mechanisms determining crack advance may occur within this plastic zone. If cyclic loading of a crack occurs then, at ambient temperature, the additional possibility of shear decohesion at the crack tip arises and the behaviour is relatively insensitive to the applied loading waveform. However, at elevated temperature a complex situation exists and the mechanism of crack extension is less clear.

With increasing temperature, the crack propagation characteristics of a material become particularly sensitive to the frequency of cycling and to the environment. Many authors consider that localised oxidation effects determine the rate of crack advance. Alternatively, an interaction with time-dependent creep processes

occurring directly ahead of the crack tip has been suggested. The major part of this work is concerned with an investigation of the elevated temperature crack growth behaviour and the crack tip deformation processes which determine crack advance.

Initially, the stress distribution around a crack tip and the resulting failure of material at the crack tip will be briefly reviewed. The mechanisms of fatigue crack growth at ambient and elevated temperature are then considered and finally the specific research undertaken is outlined.

1.2 Crack Characterisation

The rate at which a crack advances is determined, in part, by the stresses and strains. Theoretical treatments of the crack tip stresses and strains can be most easily made for an ideal elastic solid, more comprehensive analyses consider the effects of crack tip plasticity.

1.2.1 Crack Tip Stresses in an Elastic Body

The stresses at the tip of a crack in an elastic body can be obtained by considering the stresses in the X-Y plane of fig. 1.1 and then extending the result to three dimensions. Airy (1862) demonstrated that a stress function ϕ can exist such that the stresses are:

$$\begin{aligned}
\sigma_{xx} &= \frac{\partial^2 \phi}{\partial y^2} \\
\sigma_{yy} &= \frac{\partial^2 \phi}{\partial x^2} \\
\tau_{xy} &= -\frac{\partial^2 \phi}{\partial x \partial y}
\end{aligned}
\tag{1.1}$$

The Airy stress function ϕ can be shown (e.g. Broek, 1978) to satisfy the requirements for the equilibrium of stresses and the compatibility of strains, but it is difficult to derive a function which also satisfies the appropriate boundary conditions. A solution was proposed by Westergaard (1939) in terms of a complex function ψ , applicable to a body containing a sharp crack and subject to mode I loading (see fig. 1.2). Westergaard applied the complex function to the specific case of an infinite sheet under a biaxial tensile stress σ , containing a crack of length $2a$ (fig. 1.3). The appropriate choice of the complex function allows the crack tip stresses to be given in polar co-ordinates as:

$$\begin{aligned}
\sigma_{xx} &= \frac{\sigma\sqrt{\pi a}}{\sqrt{2\pi r}} \cos\theta \left(\frac{1-\sin\theta\sin 3\theta}{2} \right) + \text{additional terms} \\
\sigma_{yy} &= \frac{\sigma\sqrt{\pi a}}{\sqrt{2\pi r}} \cos\theta \left(\frac{1+\sin\theta\sin 3\theta}{2} \right) + \text{additional terms} \\
\tau_{xy} &= \frac{\sigma\sqrt{\pi a}}{\sqrt{2\pi r}} \sin\theta \cos\theta \cos 3\theta + \text{additional terms}
\end{aligned}
\tag{1.2}$$

If the point (r, θ) is sufficiently close to the crack tip, the

additional terms of equation (1.2) are negligible.

The Westergaard solution can be extended to the more common case of uniaxial tension by superimposing a pressure of $-\sigma$ in the X direction (fig. 1.3). At large values of r this does however lead to σ_{xx} becoming zero but does not affect the value of the stresses close to the crack tip.

The group of terms $\sigma/\pi a$ in equation (1.2) were labelled by Irwin (1958) the stress intensity factor, K . For an infinitely sharp crack in an infinitely wide elastic plate, the stress intensity factor for mode I opening can be expressed as:

$$K_I = \sigma/\pi a \quad (1.3)$$

For a body whose dimensions are finite this becomes:

$$K_I = Y\sigma/\pi a \quad (1.4)$$

where Y is a correction factor determined by the specific specimen geometry.

The stress intensity factor can be used to characterise the stress field around the crack tip. For mode I opening equations (1.2) can be rewritten to give:

$$\sigma_{xx} = \frac{K_I}{\sqrt{2\pi r}} \cos\theta \left(1 - \frac{\sin\theta}{2} \sin 3\theta\right)$$

$$\sigma_{yy} = \frac{K_I}{\sqrt{2\pi r}} \cos\theta \left(1 + \frac{\sin\theta}{2} \sin 3\theta\right) \quad (1.5)$$

$$\tau_{xy} = \frac{K_I}{\sqrt{2\pi r}} \sin\theta \sin 3\theta$$

$$\sigma_{zz} = 0 \quad \text{for plane stress}$$

$$\sigma_{zz} = \nu(\sigma_{xx} + \sigma_{yy}) \quad \text{for plane strain}$$

The equations developed for the crack tip stresses from linear elasticity predict infinite stresses at the crack tip. In real materials this cannot occur since the material will either fracture or plastically deform. If the region of plastic flow is small the stress intensity factor K may adequately characterise the crack tip stresses. When yielding occurs the stress distribution within the plastic zone may, to a first approximation, be considered as the yield stress. The size of the plastic zone may be estimated in terms of K , the various estimates are considered in section 1.3. However, if the material strain-hardens on deformation this simplified stress distribution may be further modified.

1.2.2 Effects of Strain Hardening

Hutchison (1968) and Rice and Rosengren (1968) have produced theoretical treatments of the stresses and strains ahead of a crack tip in a material deforming by rate-insensitive power-law strain

hardening as described by

$$\sigma = k\epsilon^N \quad (1.6)$$

where σ is the true stress, ϵ the true strain, k a constant and N the work hardening exponent. For this situation the results may be given in terms of the J integral.

The J integral, as defined by Rice (1968a), may be regarded as an elastic-plastic fracture parameter that will accommodate extensive plasticity. Although originally developed for non-linear elastic materials, Rice (1968b) has demonstrated the validity of the concept when extended to elastic-plastic materials. In the limit of small scale yielding the J integral can be shown to be equivalent to the strain energy release rate, G , for a linear elastic body (e.g. Knott, 1973).

The maximum stresses associated with time independent plastic deformation at the crack tip have been given by Hutchison (1968) and Rice and Rosengren (1968) as,

$$\sigma(r, \theta) = A\sigma(\theta) \frac{1}{r^{1/N+1}} \quad (1.7)$$

where A is the amplitude of the stress field, determined by the value of the J integral, and $\sigma(\theta)$ is an angular function given by Hutchison (1968) or Rice and Rosengren (1968).

1.2.3 Effects of Time Dependent Deformation

If time dependent deformations occur at the crack tip, expressions similar to equation (1.7) can be developed in which the

amplitude A is a function of both the applied load and the time of loading. Riedel (1978) has considered the situation of a crack subject to mode III loading in which the surrounding material deforms by power-law creep such that

$$\dot{\epsilon} = B\sigma^n \quad (1.8)$$

where $\dot{\epsilon}$ is the strain rate, σ the applied stress, n the steady state creep exponent and B a temperature dependent constant.

Analysis of the strain distribution at the crack tip shows that the creep strains, ϵ_{cr} , have a stronger singularity than the elastic strains ϵ_{el} :

$$\epsilon_{cr} \propto \frac{1}{r^{n/n+1}} \quad (1.9)$$

$$\epsilon_{el} \propto \frac{1}{r^{1/N+1}} \quad (1.10)$$

Riedel's analysis also enables the definition of a plastic zone in which the accumulated creep strain is in excess of the elastic strain (fig. 1.4). Riedel and Rice (1980) have extended this analysis to the mode I loading situation enabling the definition of a characteristic time for the transition from small scale creep to extensive creep of the whole specimen as

$$t_1 = \frac{K^2 (1-\nu^2)}{E(n+1) J^*} \quad (1.11)$$

where ν is Poisson's ratio and J^* is the time dependent J integral which can be determined either experimentally or analytically

(Sadananda and Shahinian, 1981d).

A more extensive analysis of the time dependence of stress relaxation at the crack tip has been provided by Bassani (1981) and McClintock and Bassani (1981). They have demonstrated that time dependent stress relaxation can lead to the development of specific regions at the crack tip in which regions dominated by either elastic, time independent plastic, primary creep or secondary creep exist. Such relaxation may occur by cavitation damage or by power law creep, leading to the interpretation that several deformation mechanisms may be operating at the crack tip. The rate of crack tip failure may then be determined by their interaction in an extending region of plastic deformation.

1.2.4 Summary of Crack Tip Stress Distribution

Various theoretical treatments of the stresses and strains at the crack tip have been made. In most materials the stress distribution predicted by linear elasticity is modified by plastic flow. The stresses ahead of the crack in a semi-infinite plate of finite thickness subjected to mode I loading have been described by Boyd (1972) and are illustrated in fig. 1.5.

σ_{yy} : the stress parallel to the applied loading (fig. 1.5a) attains a maximum value ahead of the crack tip but then falls to the level of the applied stress. The stress varies through the thickness and is a maximum in the mid-plane.

σ_{xx} : the stress in the crack plane and perpendicular to the crack length (fig. 1.5b) is zero at the crack tip but reaches a maximum value at a position corresponding to the maximum of σ_{yy} before

decreasing to a level of approximately $\sigma_{yy}/3$.

σ_{zz} : the stress parallel to the crack front and in the crack plane (fig. 1.5c and d) is zero at the edge but increases towards the mid-plane.

If the octahedral shear stress is given by

$$\tau_{oct} = \frac{1}{3} \sqrt{(\sigma_{xx} - \sigma_{yy})^2 + (\sigma_{yy} - \sigma_{zz})^2 + (\sigma_{zz} - \sigma_{xx})^2} \quad (1.12)$$

it can be seen to be highest at the plate surface and at the crack tip since σ_{xx} and σ_{yy} may be zero. From fig. 1.5 it can be seen that since the three principal stresses have their maxima at the mid-plane a maximum in the mean stress σ_m ($=(\sigma_{xx} + \sigma_{yy} + \sigma_{zz})/3$) will occur in the mid-plane slightly ahead of the crack tip.

1.3 The Crack Tip Plastic Zone

The linear elasticity treatment of crack tip stress predicts infinite values of stress at the crack tip. In most materials this does not occur because the material plastically deforms. The degree of the plastic deformation at the crack tip will be reflected by the size of the plastic zone.

Several theoretical estimates of the plastic zone size have been made. Under conditions of cyclic and time dependent deformation, modifications to the monotonic loading solutions may be made. Some of the estimates made for monotonic loading will be considered initially.

1.3.1 Monotonic Loading

An initial estimate of plastic zone size can be made by using the value of σ_{yy} from equation (1.5), (see fig. 1.6) with $\theta=0^\circ$ this gives,

$$\sigma_{yy} = \frac{K_1}{\sqrt{2\pi r}} \quad (1.13)$$

The plastic zone size can be obtained from the distance over which this stress exceeds the yield stress σ_y , thus giving an initial estimate r_1 as:

$$r_1 = \frac{1}{2\pi} \frac{K_1^2}{\sigma_y^2} \quad (1.14)$$

However, since the stress represented by the shaded region in fig. 1.6 will be supported by the material immediately ahead of the plastic zone r_1 , equation (1.14) will give an underestimate of the plastic zone size. This stress causes more material to yield so that a larger plastic zone size r_2 results (fig. 1.7). Rice (1967) has suggested that the increased size can be given by,

$$r_2 \approx 2r_1 = \frac{1}{\pi} \frac{K_1^2}{\sigma_y^2} \quad (1.15)$$

An alternative approach was developed by Irwin (1960) who maintained that since larger displacements and a lower material stiffness are obtained when plastic yielding occurs at the crack tip, the crack tip plasticity results in the crack acting as if it were longer than its actual size. Consequently, Irwin proposed a plastic zone correction factor r_p^* such that the crack length a , in equation

(1.3), is replaced by $(a+r_p^*)$ giving,

$$K = \sigma \sqrt{\pi(a+r_p^*)} \quad (1.16)$$

Irwin chose

$$r_p^* = r_1 = \frac{1}{2\sqrt{2}} \frac{1}{4\pi\sqrt{2}} \frac{K^2}{\sigma_y^2} = \frac{1}{6\pi} \frac{K^2}{\sigma_y^2} \quad (1.17)$$

Equations (1.16) and (1.17) imply that the stress intensity factor K must be determined by an iterative process. Although the use of a plastic zone correction factor allows a more adequate calculation of K , r_p^* was not meant to be an estimate of the true plastic zone size, but it is frequently used as such.

Dugdale (1960) postulated a model which assumes that yielding occurs in a narrow strip ahead of the crack (fig. 1.8). The crack of length $2a$, with yielded zones at the crack tip, is replaced by a totally elastic crack of length $2(a+r_3)$, where r_3 is the plastic zone size; plus a crack of length $2(a+r_3)$ which is prevented from opening over the distance r_3 by restraining stresses equal to the yield stress σ_y . The value of r_3 is then determined such that the stress at the crack tip $\pm(a+r_3)$ is finite. This gives:

$$r_3 = \frac{\pi K^2}{8 \sigma_y^2} \quad (1.18)$$

Although this model is useful for calculating the plastic zone size, some physical significance is lost by the requirement of a zero stress intensity factor at $(a+r_3)$.

More precise predictions of the plastic zone size consider the state of stress, that is whether it is plane stress or plane strain,

and the yield criterion, Tresca or von Mises. The crack tip stresses (equations (1.5)) can be rewritten in terms of principal stresses to give:

$$\begin{aligned}\sigma_1 &= \frac{K}{\sqrt{2\pi r}} \frac{\cos\theta(1+\sin\theta)}{2} \\ \sigma_2 &= \frac{K}{\sqrt{2\pi r}} \frac{\cos\theta(1-\sin\theta)}{2}\end{aligned}\tag{1.19}$$

$$\sigma_3 = 0 \text{ for plane stress}$$

$$\sigma_3 = \nu(\sigma_1 + \sigma_2) = \frac{2\nu K \cos\theta}{\sqrt{2\pi r}} \text{ for plane strain}$$

The use of these equations in conjunction with the von Mises yield criterion enables the boundary of the plastic zone as a function of the angle θ to be given as:

$$r_p(\theta) = \frac{K^2}{4\pi\sigma_y^2} \left[\frac{3\sin^2\theta + (1-2\nu)^2(1+\cos\theta)}{2} \right]\tag{1.20}$$

for plane strain

$$r_p(\theta) = \frac{K^2}{4\pi\sigma_y^2} \left[\frac{1+3\sin^2\theta+\cos\theta}{2} \right]$$

for plane stress

This is equivalent to the estimate of the plastic zone size r_1 and for $\theta=0^\circ$ the plane stress value of $r_p(\theta)$ is that given by equation (1.14). However, for plane strain conditions the plastic zone size is given by:

$$r_p(\theta=0) = \frac{(1-2\nu)^2}{2\pi} \frac{K^2}{\sigma_y^2} \quad (1.21)$$

This value is approximately nine times smaller than the plane stress value. Thus, the estimate of the plastic zone size r_1 applies accurately to the plane stress condition but considerably overestimates the size of the plane strain plastic zone.

The effect of the plane strain constraint upon the plastic zone size can be fully appreciated by consideration of the variation of the plastic zone size with the angle θ , see fig. 1.9. In plates of sufficient thickness the plastic zone size would vary throughout the thickness as shown in fig. 1.10. McClintock and Irwin (1965) suggested that sufficient constraint to produce a plane strain plastic zone that was nine times smaller than the plane stress plastic zone would not be obtained. Their estimate of the constraint results in a plane strain plastic zone which is approximately one-third of the size of the plane stress plastic zone. This allows two further estimates of the plane strain plastic zone size to be made:

$$r_4 = r_1 = \frac{1}{3} \frac{K^2}{6\pi \sigma_y^2} \quad (1.22)$$

$$r_5 = r_2 = \frac{1}{3} \frac{K^2}{3\pi \sigma_y^2} \quad (1.23)$$

So far we have only considered the theoretical treatments of the plastic zone size for an elastic-perfectly plastic solid. If the material strain hardens the result may be further modified.

Estimates of the plastic zone size made by taking into account the hardening characteristics of the material (Rice and Tracey (1973);

Tracey (1971 and 1976)) show that the plane strain plastic zone is not symmetrical about the axis at 90° to the crack plane. The lobes of the plastic zone are found to be rotated forward towards the crack plane so that the maximum dimension of the plastic zone is at $\theta \approx 70^\circ$.

Rice and Rosengren (1968) found that the maximum distance from the crack tip to the elastic-plastic boundary was determined to be at $\theta=100^\circ$. For $N=0.05$, the plastic zone size at $\theta=0^\circ$ was given by,

$$r(\theta=0^\circ) = 0.007 \frac{K^2}{\sigma_y^2} \quad (1.24)$$

The influence of time dependent deformation on the plastic zone size has been estimated by Riedel (1978) and Riedel and Rice (1980). It has been shown that accumulated creep strain ahead of the crack tip may be in excess of the elastic strain distribution. By equating the elastic and creep strains a creep plastic zone in mode I loading may be defined by,

$$r_{cr} = \frac{1}{2\pi} \frac{K_i^2}{E^2} \frac{(n+1)^2 E^n B t^{2/n-1} F_{cr}(\theta)}{2n\alpha_n(n-1)} \quad (1.25)$$

where K_i is the initial stress intensity factor, E is Young's modulus, B and n are defined by equation (1.8), t is the time under load, α_n is a constant approximately equal to unity and $F_{cr}(\theta)$ is an angular function. Fig. 1.11 shows the variation in shape of the creep zone obtained by varying n , for both plane strain and plane stress conditions.

In a similar manner, Vitek (1977) has considered the time dependent development of a crack tip plastic zone. Computer simulations have been used to demonstrate that creep deformation at

the crack tip may lead to an increase in plastic zone size. The crack tip relaxation has also been used to evaluate the rate of creep crack growth.

1.3.2 Cyclic Loading

So far the plastic zone size due to monotonic loading has been considered. However, under cyclic loading Rice (1967) has postulated the existence of a plastic zone associated with reversed plasticity. The size of this zone can be estimated using a superposition argument.

Tension loading creates a plastic zone of size r_p (fig. 1.12a). On unloading the stress increment necessary to cause yielding will, to a first approximation, be twice the tensile yield stress. Hence a smaller plastic zone, of size r_c , associated with reversed loading will arise (fig. 1.12b). Consequently, under cyclic loading both a cyclic plastic zone, where reversed plasticity occurs, and a monotonic plastic zone may exist (fig. 1.12c). Simple estimates of the cyclic plastic zone can be obtained by replacing the yield stress, in the monotonic estimates of the plastic zone size, by twice their values. Thus, a cyclic plastic zone size, r_c , that is approximately one quarter of the monotonic plastic zone size will be predicted.

Thus it can be seen that under small scale yielding many theoretical expressions of the extent of crack tip plasticity have been made. The experimental determinations of crack tip plasticity, often show limited agreement with the theoretical estimates, (Lankford et al., 1977). This may reflect either the limited sensitivity of some of the measurement techniques or the idealisations inherent in the theoretical models.

1.4 Micromechanisms of Fracture

A generalised classification of the mechanisms of fracture at ambient and elevated temperature has been given by Ashby (1979) and is represented in fig. 1.13.

At low temperatures where plastic flow is independent of the temperature and time cleavage and brittle intergranular fracture may occur at a stress lower than that required for slip. Alternatively, slip may lead to the pile up of dislocations at second phase particles which then themselves fail in a brittle manner. In this situation fracture may occur without general plasticity when the elastic energy released on crack extension is sufficient to provide the surface energy of the fractured material (Griffiths, 1920). The crack extension will then be characterised by a critical potential energy release rate, G_c , equal to the surface energy of the fractured material produced by crack extension, 2γ .

If extensive plastic flow occurs before fracture it is possible that holes are nucleated at inclusions by either decohesion of the particle/matrix interface or fracture of the particle (Goods and Brown, 1979). Once nucleated the holes grow until they coalesce to give a fracture path. If holes are not nucleated, material may become mechanically unstable and deformation will be localised in a neck or shear band. Eventually the cross section will reduce to zero and failure occur.

With increasing temperature material flow may be time dependent and the flow stress dependent on the strain rate. Holes may then nucleate at inclusions within the grains and grow as the material creeps in a similar manner to low temperature hole growth at

inclusions. However, since plasticity now occurs at lower stresses nucleation may be delayed to larger strains. Further, stabilisation of the flow may occur and postpone the coalescence of voids.

At lower stresses, a transition to intergranular failure occurs. Grain boundaries may slide, wedge-cracks or voids may grow on boundaries normal to the tensile axis and thereby influence the subsequent failure.

Numerous models of the mechanism of void nucleation and growth have been provided. For example, nucleation may be controlled by grain boundary sliding which is in turn limited by power-law creep (Crossman and Ashby, 1975). Alternatively, growth may occur by local diffusion at a rate controlled by power-law creep of the surrounding grains (Beeré and Speight, 1978).

If diffusion is rapid, that is at high temperatures, and the stresses low, power-law creep may be negligible and voids may grow by diffusion causing the grain boundaries to move apart. This type of failure mechanism is not frequently encountered.

Thus it can be seen that crack advance may be determined by various mechanisms of failure at the crack tip. The specific micromechanism will be dependent on the material and the crack tip deformation. Failure in a brittle manner may occur after limited plastic deformation whereas hole growth may require more extensive plasticity. Although the basic mechanisms of failure persist under cyclic loading the continuing oscillation of the level of stress may lead to a controlled rate of crack extension. The phenomenon of fatigue will now be considered with particular reference to the influence of temperature.

1.5 Fatigue Crack Propagation at Ambient Temperature

In many materials the initiation of a crack occurs rapidly. They have been observed to occur along slip planes, in grain boundaries, within second phase particles and at the interface between the matrix and second phase particles. In some cases pre-existing cracks may arise from the processing history of the material, consequently fatigue crack propagation is of considerable importance.

Initial propagation often occurs along crystallographic planes (referred to as stage I by Forsyth, 1960), but then continues in a plane perpendicular to the maximum principal stress (stage II). In some cases cracks initiated below the fatigue limit may become non-propagating; for example, inclusions may crack, but the stress required to extend the crack into the matrix may be greater than the applied stress. Alternatively, cracks may initiate at a notch or surface flaw but may become non-propagating as they grow out of the notch stress field. Since fatigue crack growth may occur under low levels of applied stress, the crack growth response has often been characterised in terms of linear elastic fracture mechanics parameters.

1.5.1 Application of Fracture Mechanics to Fatigue Crack Propagation

Providing that small scale yielding occurs, the propagation rate of a microcrack may be described by linear elastic fracture mechanics. Under normal ambient temperature conditions it is now well established that the crack growth rate per cycle (da/dN) is a function of the alternating stress intensity factor ΔK , where ΔK is the difference

between the maximum and minimum stress intensities for each cycle, that is $\Delta K = K_{\max} - K_{\min}$. Paris and Erdogan (1963) originally proposed that the two could be related through a power law of the form:

$$\frac{da}{dN} = C(\Delta K)^m \quad (1.26)$$

where C and m are constants. More recently it has been shown that the law applies only over a limited range of ΔK , and that more generally a sigmoidal relationship exists between da/dN and ΔK (Beevers et al., 1975). As shown in fig. 1.14 three regions may be identified:

- A: Stage I, a low growth rate region where da/dN decreases rapidly with decreasing ΔK to a threshold value ΔK_{th} .
- B: Stage II, a mid-region where the Paris relation holds, and m is normally between 2 and 4.
- C: Stage III, a high growth rate region, with an increased value of m, obtained as the maximum stress intensity factor, $K_{\max}$, approaches the critical stress intensity for static fracture, K_c .

It is convenient to discuss the fatigue behaviour in terms of these three regimes.

1.5.2 Threshold and Near Threshold Crack Growth

At low values of ΔK (stage I in fig. 1.14) deviations in the crack growth rates predicted by equation 1.26 are observed. The crack growth rate per cycle, da/dN , decreases rapidly with decreasing ΔK , thus leading to a threshold value, ΔK_{th} , for crack propagation. The form of the growth rate curve in this region has been shown to be

sensitive to variables such as the microstructure, the ratio of maximum to minimum stress intensity factor $R (=K_{min}/K_{max})$, the environment and the load history (Ritchie, 1977).

At low growth rates microstructurally sensitive growth often manifests itself as intergranular facets or as transgranular facets having marked crystallographic features. Observations in both f.c.c. materials (Garrett and Knott, 1975) and b.c.c. materials (Rieux et al., 1979) have shown that the transgranular facets may not be simple fracture along slip planes. Models involving slip on more than one system have been proposed to explain the crystallographic nature of transgranular facets, for example two intersecting $\{111\}$ systems leading to $\{100\}$ planes of separation in copper (Neumann, 1974) and aluminium (Garrett and Knott, 1975). Recently, Rieux et al. (1979) have considered that multiple slip may accomodate large shear offsets and that the crack plane is then determined by the intersection of these offsets.

In the threshold regime, it is often observed that higher values of R lead to crack growth being maintained to lower values of ΔK . This has been interpreted (Ritchie et al., 1980) in terms of the compressive stresses developed in the crack tip plastic zone.

Schmidt and Paris (1973) proposed an explanation in terms of Elber's original concept of crack closure (Elber, 1970). The tensile plastic deformation left in the wake of a propagating fatigue crack may result in contact of the opposite faces of the crack during part of the loading cycle (see fig. 1.15). Such closure may reduce the effective ΔK at the crack tip to a value

$$\Delta K_{eff} = K_{max} - K_{op} \quad (1.27)$$

where K_{op} is the lowest stress intensity at which the crack is fully open. Thus, if K_{op} is constant ΔK_{eff} will increase as R increases and the observed ΔK_{th} will decrease.

The presence of corrosion products on the fracture surfaces produced in the near threshold regime suggests that environmental action may also be significant. The results of the effect of environment are, however, somewhat contradictory. Some studies on alloy steels indicate that ΔK_{th} is lower in vacuum than in air, a result that has been attributed to a higher K_{op} in air (Suresh et al., 1981). Other workers have observed higher values of ΔK_{th} in vacuum compared to those in air (Beevers, 1977). This has been interpreted as being due to the aggressive nature of the testing atmosphere resulting in either dissolution of the crack tip or embrittlement of the material ahead of the crack tip.

It has been further suggested that as ΔK is lowered, crack growth may occur by a mode II shear process rather than a mode I process (Ohtsuka et al., 1975; McEvily, 1977 and Tomkins, 1979). More recently Minakawa and McEvily (1982) have associated the threshold behaviour with a combined mode II and mode I loading in which the shear mode of crack growth is associated with dislocation motion on one active slip plane. Evidence for this model arises from the observations of Davidson (1980) on crack tip motion under alternating loads. If, however, this is the case the relevance of crack closure to the near threshold fatigue crack growth process will not be as significant as with a fully mode I mechanism.

1.5.3 Intermediate Crack Growth

The presence of a pattern of striations on a fatigue fracture surface was first reported by Zappfe and Worden (1949), and the formation of striations has subsequently been widely characterised as the accepted mechanism of crack growth in the intermediate region. Programmed loading (e.g. Forsyth and Ryder, 1961) has shown that each striation is produced by one stress cycle, although every stress cycle does not necessarily produce a striation. The mechanism of striation formation is not fully established, but it is generally accepted to involve alternate blunting and resharpening of the crack tip by plastic flow. Two alternative proposed mechanisms are illustrated in fig. 1.16. Essentially in the model due to Laird (1967) the crack tip area is continually increased during the loading part of the cycle and sharpened during the unloading part. In the model of Tomkins and Biggs (1969) the crack tip is initially blunted under a small tensile load and then, on further tensile loading, fracture occurs at the centre of the blunted crack. More recent work, for example Wanhill (1975) and Laird and de la Veaux (1977), has not produced agreement on the exact mechanism involved.

Most quantitative models of fatigue crack propagation have been based on the striation mechanism of growth. Tomkins (1975) and Irving and McCartney (1977) have reviewed some of these models and have shown that they could broadly be divided into two categories; those based on material damage accumulation and those linked to crack tip opening displacement.

Damage accumulation theories have been based on the progressive attainment of some critical condition ahead of the crack tip at which

point crack advance occurs. This condition has been variously taken to depend on the critical fracture strength being reached with cycling (Head, 1953), accumulated cycle displacement in the plastic zone (Weertman, 1966) and more recently the accumulation of cyclic strain within the crack tip plastic zone (McClintock, 1962; Tomkins, 1968; Antolovich et al., 1975). The concept of damage accumulation recieved a set-back from work by Garrett and Knott (1976) which showed growth rates in an aluminium alloy to be independent of annealing treatment applied to the strained crack tip.

The second type of model is based on the achievement of a critical displacement or strain at the crack tip on each cycle in previously undamaged material. The geometrical changes which must occur at the crack tip during loading led McClintock (1967) to suggest that the crack tip opening displacement (CTOD) should be some fraction of the crack growth rate per cycle, da/dN . Tomkins and Wareing (1977) have shown that the relationship between the crack growth rate, the stress intensity range ΔK and the CTOD can be expressed as,

$$\frac{da}{dN} = \frac{CTOD}{2} = A \frac{\Delta K^2}{2E\sigma_y} \quad (1.28)$$

where A is a constant <1 , E is Young's modulus and σ_y the yield stress. Generally the value of da/dN is observed to be less than the $CTOD/2$ leading Tomkins and Wareing (1977) to suggest that equation (1.28) offers an upper bound to continuum controlled fatigue crack propagation.

All models for striation growth predict a value of the exponent m in the Paris equation (equation (1.26)) of between 2 and 4. In some cases higher values than this are found and Lindley et al. (1975) have

produced considerable evidence that large departures from this range of values are associated with the presence of additional static fracture modes such as cleavage, intergranular cracking or microvoid coalescence. Similarly, crack growth rates exceeding the CTOD/2 have been observed (Jablonski et al., 1977; Lloyd and Wareing, 1979), again leading to the assumption of additional mechanisms of failure.

1.5.4 High Growth Rates

In stage III of fig. 1.14 a rapid acceleration in the fatigue crack growth rate is observed as the material's fracture toughness, K_{IC} , is approached. For higher values of R this behaviour is approached at lower values of ΔK . The static modes of failure dominate at these high growth rates, thus crack extension is often found to be discontinuous and involves cracking ahead of the main crack tip followed by subsequent ligament rupture.

1.5.5 Fatigue Crack Propagation in Superalloys

The ambient temperature fatigue crack propagation characteristics of superalloys are generally found to be dominated by the tendency to planar slip usually found in these alloys. Gell and Leverant (1968b) found that cracks in single crystals of Mar M 200 propagated preferentially on $\{111\}$ planes in directions parallel to $\langle 110 \rangle$. At low growth rates Purushothamen and Tien (1978) found that controlled propagation occurred on alternating slip planes, giving a fracture surface containing large crystallographic facets.

In polycrystalline material, transitions from crystallographic crack propagation to transgranular stage II propagation have been

observed (Runkle and Pelloux, 1979; Sadananda and Shahinian, 1981d; King, 1982). It has been suggested that the transition in behaviour is determined by the extent of crack tip plasticity. Runkle and Pelloux suggest the transition is determined by the ratio of the cyclic plastic zone size to the grain size, whereas Sadananda and Shahinian consider the monotonic plastic zone size to be important.

At small values of ΔK or large grain sizes, cracking is restricted to specific planes within a grain. At large ΔK or small grain sizes the deformation is more continuous and stage II crack propagation ensues. King (1982) has reported a transition from crystallographic fracture to striation growth when the estimated cyclic plastic zone size approaches the grain size.

The mechanism by which crystallographic failure occurs has been variously suggested to be related to cleavage or to be due to intense slip activity. The resemblance of the crystallographic fracture facets to cleavage facets observed in b.c.c. and h.c.p. material has led some authors (e.g. Sadananda and Shahinian, 1979) to relate the two failures. Duquette and Gell (1971) have observed that environmental effects can occur, with facets produced by failure in vacuum showing more ductility than those observed after failure in air. This has led them to propose that the dislocation dipole structure generated at the crack tip effectively increases oxygen adsorption resulting in a localised reduction in the material's cleavage strength. Once the crack tip stresses exceed this reduced value, localised failure occurs.

Although similarities exist in the appearance of crystallographic and cleavage facets, the observations of Gell and Leverant (1968a) indicate that failure is accompanied by intense plastic deformation.

The existence of striations on the fractured facets implies a controlled cyclic crack growth rate well below that usually associated with cleavage fracture. Gell and Leverant have consequently considered the failure process to be related to the dislocation structure generated during fatigue. Cyclic deformation promotes weakening of the slip planes and separation of the glide plane occurs under the influence of the crack tip shear and normal stresses (fig. 1.15).

In view of the extensive use of superalloys at elevated temperature, much work has been carried out into the high temperature crack growth characteristics. This behaviour will now be considered.

1.6 Elevated Temperature Crack Growth

At elevated temperature slow crack extension may occur under a steady applied load, creep crack growth, as well as under cyclic loading and a combination of the two. In many circumstances creep crack growth under steady loading involves the development of creep failure in material ahead of the crack tip.

1.6.1 Characterisation of Elevated Temperature Crack Growth

The time dependent occurrence of creep deformation ahead of the crack tip by either power-law creep or cavitation may be accompanied by considerable stress relaxation. Thus, the time dependent development of a plastic zone may give rise to violation of linear elastic fracture mechanics predictions of small scale yielding at the crack tip. Under these conditions the applicability of the linear elastic stress intensity factor to characterising the crack tip stresses may be limited.

Numerous parameters have been investigated in an attempt to correlate creep crack growth rates, (see e.g. Pilkington, 1979; Sadananda and Shahinian, 1981b). In addition to the linear elastic stress intensity factor, parameters such as the crack tip opening displacement, the net section stress, a reference stress chosen such that the displacement rate is the same as that of an uncracked specimen and various derivatives of the non-linear elastic J integral have been used.

During creep crack growth, time-dependent derivatives of the J integral have been successfully used (Sadananda and Shahinian, 1978) to correlate crack growth. Sadananda and Shahinian (1979) have proposed the use of a cyclic value of the J integral ($\Delta J = J_{\max} - J_{\min}$) to characterise high temperature fatigue crack growth rates. This interpretation must, however, be limited since the J integral is defined for situations of monotonic loading (Rice, 1968a) and has little meaning for conditions of reversed plastic flow. Also, if the load is applied rapidly, the instantaneous stress-strain field may be close to that defined by linear elastic fracture mechanics.

In the time immediately after application of the load, some stress relaxation may occur the extent of which will govern the applicability of the use of K. For material with a low creep ductility fracture may occur rapidly, limiting the spread of plasticity. Consequently the crack growth characteristics may be adequately correlated with the linear elastic stress intensity factor. Sadananda and Shahinian (1981c) have shown that the crack growth rates of numerous superalloys can be correlated with K, whereas for more ductile materials, such as stainless steel, a better correlation is obtained with the non-linear elastic J integral.

1.6.2 Fracture Characteristics of Superalloys at Elevated Temperature

When material is deformed at high homologous temperatures structural changes may occur. For example, holes or cracks may nucleate and grow inside the solid; the grain size may increase, by grain growth, or decrease, by dynamic recrystallisation. In general, the crack growth characteristics are most commonly affected by void growth in the vicinity of the crack tip. As the temperature is raised the tendency for intergranular crack growth increases and in superalloys intergranular cavities and cracks have been observed ahead of the main crack (Floreen, 1980). Crack extension then occurs by linkage of the main crack and the cavitated grain boundary giving a fracture surface characteristic of void coalescence. However, Pedron and Pineau (1982) have noted brittle intergranular fracture in Inconel 718 and suggest that environmental interactions are important.

In addition, superalloys have been observed to show faceted crack growth at elevated temperature (Leverant and Gell, 1975; Sadananda and Shahinian, 1981a). Leverant and Gell have shown that in single crystals the facets represent crystallographic fracture on $\{111\}$ slip planes. In contrast, Sadananda and Shahinian have identified the facets in Udimet 700 fractured at 850°C as $\{100\}$ cleavage planes. Subsequently, Sadananda and Shahinian (1981d) have proposed models based on the planar slip characteristics.

For growth along $\{111\}$ slip planes (fig. 1.17a) cracks form on the preferential slip plane ahead of the main crack until cracks form on a more favourably oriented slip plane. In the case of crack growth along the cleavage plane (fig. 1.17b) it is proposed that the concentration of slip along specific slip bands leads only to crack

tip blunting. Microcracks then nucleate on the cleavage plane and link with the main crack or the crack extends by a simple cleavage mechanism.

Cyclic loading at high homologous temperature may lead to an influence of high temperature deformation mechanisms. Although the fatigue crack growth rates at elevated temperature vary with ΔK in a manner similar to that found at low homologous temperature (section 1.5.1), significant variations in behaviour may be obtained. This behaviour will now be considered and the mechanisms that have been proposed to explain the response will be discussed.

1.7 High Temperature Fatigue

The interpretation of the fatigue behaviour of materials at elevated temperature is often complicated by the varying responses of the material to the temperature, the frequency of cycling, the environment and the waveform. With increasing temperature fatigue lifetimes generally decrease and crack growth rates increase.

1.7.1 Frequency Effects

Organ and Gell (1971) have reported that the fatigue lifetimes of Udimet 700 at 760°C are affected by frequency: at frequencies below approximately 10Hz lifetimes were found to decrease significantly, fig. 1.18. Similarly, Leverant and Gell (1975) have reported a frequency dependence of the lifetime of Mar M 200 single crystals with a transition in the behaviour occurring below approximately 1Hz.

The effect of frequency on the crack propagation rates at elevated temperature has also been extensively investigated, for

example Ohumura et al. (1973), Scarlin (1977). Ohumura et al., using a cobalt base superalloy HS188, found that at high frequencies ($>1\text{Hz}$) crack growth was usually transgranular and was relatively insensitive to the frequency. However, lowering the frequency resulted in an increase in crack propagation rate and a change to intergranular crack growth (fig. 1.19). Similar results have been reported by Jablonski et al. (1977) on a series of solid solution strengthened alloys and more recently Gabrielli and Pelloux (1982) have observed the effect in Inconel X-750 in both air and vacuum. In addition, Gabrielli and Pelloux showed that as the frequency decreased the crack growth rate tended to the constant load creep crack growth rate.

Although Scarlin (1977) observed increases in the elevated temperature crack growth rates of both wrought Nimonic 105 and cast IN 738LC with decreasing frequency, differences were observed in their response to changes in environment. In wrought material Scarlin observed that the growth rates in air were always greater than those obtained in vacuum. However, in cast material the growth rates were lower in air than in vacuum and at low frequencies ($<10^{-4}\text{Hz}$) were generally accompanied by interdendritic crack branching.

1.7.2 Environmental Effects

At elevated temperature, environmental interactions may influence the initiation and growth of cracks. Coffin (1972) has reported that the high strain amplitude fatigue lifetimes of A286 in vacuum at 593°C show little sensitivity to the frequency of cycling. However, the low cycle fatigue crack growth rates of the alloy (Solomon and Coffin, 1972) showed increases at low frequencies (fig. 1.20). Tomkins and

Wareing (1977) have shown that if the endurances are calculated from the crack growth rates, they compare favourably with the lifetimes in air but grossly underestimated those in vacuum. This has led them to suggest that the environmental interaction is predominantly associated with crack initiation.

Metallographic evidence for the incidence of oxidation at crack tips has been given by McMahon and Coffin (1970) and Chaku and McMahon (1974) who observed the preferential oxidation of surface connected grain boundaries. However, under high cycle fatigue conditions Gell and Leverant (1973) observed that oxide blocking of the crack tip occurred so that, although initiation may be enhanced, the subsequent propagation may be retarded and failure may occur from cracks nucleated internally. Similarly, Wareing (1975) has shown that the low strain amplitude fatigue lifetimes in air are consistent with high strain amplitude lifetimes obtained in vacuum.

The influence of the testing atmosphere on the elevated temperature crack growth rates has been extensively investigated by Floreen and Kane (1979). They have shown that small additions of oxygen (~100p.p.m.) to an inert atmosphere can increase crack growth rates to values which are close to those obtained in air. Also, Sadananda and Shahinian (1980a) have shown that, at elevated temperature, evacuation of the test chamber effects an immediate decrease in the crack growth rate.

James (1976) has suggested that the increased growth rates at elevated temperatures may be attributed primarily to environmental interactions. Whilst a comparison of the growth rates of 304 stainless steel at 538°C in vacuum to those at room temperature in air supports this hypothesis, a comparison of growth rates in the same environment

shows that the temperature effect remains. Similarly, Sadananda and Shahinian (1981a) have found that whilst the growth rates of Udimet 700 at 850°C were higher in air than in vacuum, increasing the temperature increased the growth rates both in vacuum and in air.

1.7.3 Waveshape Effects

In addition to the effect that reductions in the frequency have on fatigue lifetimes and crack growth rates, both hold times and asymmetric cycling may influence the elevated temperature fatigue behaviour.

In a study of the effects of tensile hold times during the high temperature high strain amplitude cycling of 20/25 Nb stainless steel, Tomkins and Wareing (1977) noted that although crack initiation and early propagation proceeded in the normal stage I and II manner, eventually intergranular crack growth occurred through a pre-cavitated region. Similarly, Lloyd (1979) has reported that although reductions in the frequency may give slight increases in the elevated temperature crack growth rates of 316 stainless steel, more significant increases can be given by incorporating a tensile hold time into the signal. Under these conditions unstable intergranular crack growth occurred at a rate in excess of the estimated CTOD/2 per cycle and with a value of $m=10$ in the Paris equation (equation (1.26)) that was characteristic of that for creep crack propagation.

Michel and Smith (1980) have shown that in aged solution annealed 316 stainless steel 16 minute hold times gave rise to unstable intergranular crack growth. Material adjacent to the crack tip showed grain boundary cavitation suggesting that intergranular failure

occurred by cavity coalescence. Hold time effects on 316 and 304 stainless steel are also observed to be dependent on microstructure (Lloyd and Wareing, 1979). In general, ageing reduces the effect of hold times in both solution annealed and 20% cold worked material; Lloyd and Wareing postulate that this is due to the precipitation and coarsening of grain boundary carbides retarding grain boundary sliding.

Hold time effects have also been observed in numerous superalloys. Pedron and Pineau (1980) have shown that 10 second hold times can increase the crack growth rates of Inconel 718 above the estimated CTOD per cycle and that growth rates approaching the calculated monotonic plastic zone size per cycle can be obtained. Similarly, increased growth rates have been reported by Sadananda and Shahinian (1981d) in Inconel 718 and Udimet 700 and Pelloux and Huang (1981) in LC Astroloy and HIP'ed Astroloy, the latter occurring in both vacuum and air.

Although these investigations show intergranular failure during hold time cycling the appearance of the fracture surfaces differ. Pedron and Pineau (1982) report that Inconel 718 exhibits brittle intergranular fracture and suggest that the environment may affect grain boundary ductility, whereas the intergranular failure of Pelloux and Huang (1981) is characteristic of grain boundary void coalescence. The measurement of crack growth rates during the hold times themselves shows differing results: Pedron and Pineau have found the crack growth to be below that associated with static loading whereas Pelloux and Huang observed hold time growth rates in excess of the normal creep crack growth rates.

A further complication in the interpretation of elevated

temperature crack growth data may arise from a dependence on the rate of loading. Both Pedron and Pineau and Pelloux and Huang have shown that the majority of crack extension is associated with the loading transient of the hold time waveform. However, Min and Raj (1979) and Baik and Raj (1982b) have observed that increased growth rates may be obtained by asymmetrical cycling in which the tensile strain rate is less than the compressive strain rate. Their analysis, which considers that unequal grain boundary sliding during loading and unloading will lead to a net build up of damage cannot, however, easily explain the dependence of the crack growth rate on reductions in the frequency of symmetrical loading waveforms.

It can therefore be seen that at elevated temperature, time dependent phenomena may be important in determining the crack growth processes. Both environmental and creep processes may influence the crack growth rate and various models have been proposed by which these processes affect the fracture behaviour.

1.8 Models for High Temperature Fatigue Crack Growth

The models proposed to explain the high temperature fatigue crack growth behaviour can be broadly divided into two classes: those in which increased crack growth rates arise from environmental interactions and those in which creep processes are considered to be important.

1.8.1 Environmentally Based Models

Environmental enhancement of fatigue crack growth rates at elevated temperature has been considered to be principally caused by localised corrosion or by localised embrittlement of the material. Oxidation of grain boundary carbides (Coffin, 1973) may lead to microcrack initiation giving rise to a localised stress concentration. Similarly, crack tip oxidation may lead to the localised depletion of the aluminium and titanium rich γ' precipitate (McMahon and Coffin, 1970) which may alter the mechanical properties of the material at the crack tip thus aiding crack advance.

Although dissolution of material may result in crack advance, Scarlin (1979) and Hoffelner and Speidel (1980) have considered that greater effects may result if alterations to crack tip plasticity occur. Achter (1968) has shown that small partial pressures of oxygen are sufficient to effect a monolayer coverage of freshly exposed surfaces, and this has been used as an explanation of the differences in the crack growth rates in air and vacuum. At elevated temperature the effect may be increased so that either damage accumulation or the attainment of a critical crack tip opening displacement may be enhanced and increased growth rates result.

A more general view of the high temperature interaction has been given by Sadananda and Shahinian (1980b) who consider a change from cycle-dependent growth to time-dependent growth to occur. The time dependence may arise from either environmental or creep interactions with a threshold stress intensity for the interaction existing. If cycling is above the threshold value, time-dependent effects may contribute directly to crack advance. If it is below the threshold

value, the influence will be limited, but cumulative effects may occur. The exact value of the threshold value will be determined not only by the temperature and frequency but also by the rate of crack propagation.

An alternative mechanism by which environmental influences may occur is localised embrittlement of material at the crack tip. Such an interaction requires several sequential processes following the application of the load (fig. 1.21),

- (i) transport of gas to the reactive surface,
- (ii) adsorption of the gaseous material and production of the embrittling species,
- (iii) diffusion of the damaging species ahead of the crack tip, and
- (iv) embrittlement of the material.

Although this mechanism has been proposed by Wei and Simmons (1981) to explain hydrogen embrittlement, it is equally applicable to the suggestions of embrittlement by oxygen diffusion along grain boundaries (McMahon and Coffin, 1970). In all cases, for embrittlement to be a possible mechanism, it must occur at a rate such that the rate of diffusion is greater than, or equal to, the rate of crack growth, that is

$$\sqrt{D} > \sqrt{\nu \frac{da}{dN}} \quad (1.29)$$

where D is the appropriate diffusion coefficient, ν is the frequency and da/dN the crack growth rate per cycle. Floreen and Kane (1980) have pointed out that although specific measurements of the diffusion coefficients of complex superalloys are not available, extrapolated values suggest that this condition may be satisfied. However, such a

treatment may be considered to be an oversimplification since it does not involve the time dependence of the processes prior to diffusion occurring. Also, the presence of an oxide layer at the crack tip may act as a barrier to further oxidation so that freshly exposed metal would be required to enable the embrittling process to continue.

An alternative explanation of the environmental influence has been proposed by Raj (1982) who has considered that oxygen diffusion ahead of the crack tip may give rise to the production of CO at grain boundary carbides. The accompanying increase in pressure may increase cavitation at the crack tip. A similar mechanism may arise if crack tip oxidation results in vacancy injection into the material (Hancock, 1976) enabling the formation of grain boundary voids. In either case, the increased growth rate must arise from the interaction of the crack with an array of grain boundary voids, and not specifically from an environmental interaction.

1.8.2 Creep Based Models

The increased crack growth rates and decreased lifetimes observed at high temperature and low frequency are usually accompanied by a transition from transgranular to intergranular failure. One of the most common explanations of this effect has been to assume that plastic strain accumulates as a result of creep deformation or stress relaxation. Several mechanisms have been proposed for a creep-fatigue interaction in terms of grain boundary cavities and cracks under cyclic loading. For this situation two points must be considered,

- (1) the effects of waveform and frequency on the nucleation and growth of cavities,

(ii) the interaction between a crack and a distribution of cavities.

Raj and Ashby (1975) have treated cavity nucleation as a solid state transformation in which vacancies coalesce to form cavities. Using classical Becker-Doring nucleation theory they found that void nucleation would most probably occur heterogeneously at second phase particles. The analysis of Raj (1978) has shown that the formation of stable vacancy clusters should be possible provided a certain incubation period and threshold stress are exceeded. If grain boundary sliding occurs, stress concentrations may arise at grain boundary particles and thus decrease the incubation period for cavity nucleation. Raj's calculations show that stress concentrations sufficient to nucleate cavities at particles may only arise at high strain rates ($\sim 10^{-2} \text{ s}^{-1}$ for copper). This forms the basis of the specific mechanism proposed by Min and Raj (1978) for the effect of hold times on the elevated temperature fatigue behaviour.

In cyclic loading, Min and Raj postulate that cavities will nucleate if the reduced incubation period is less than the duration of the loading transient. An estimate of the time constant for relaxation of the stress concentration, τ , can be made from

$$\tau = 0.1 (1-\nu) \frac{kT}{G} \frac{f_b d p^2}{\Omega \delta D_B} \quad (1.30)$$

where ν is Poisson's ratio, k Boltzmann's constant, T the absolute temperature, G the shear modulus, f_b the area fraction of second phase particles in the grain boundary, d the grain size, p the average particle size, Ω the atomic volume, δ the grain boundary thickness and D_B the grain boundary diffusion coefficient.

If nucleated, reversal of the stress may cause the cavities to sinter and disappear so that, if they are to interact with a crack, some cavity growth must occur before the stress is reversed. Given these conditions Min and Raj propose that the maximum effect will be observed using a waveform consisting of a short compressive hold time followed by rapid stress reversal to an extended hold time allowing cavity growth.

The sliding properties of grain boundaries have also been used to explain the deleterious effects of asymmetric cycling on the high temperature crack growth rates and fatigue lifetimes (Min and Raj, 1979; Baik and Raj, 1982a and b). Since grain boundary sliding is a shear process, it will occur equally in tension and compression, and if the rate of straining is equal little net damage will accrue. If however, the tensile strain rate is less than the compressive strain rate (slow-fast cycling) more sliding occurs in tension than compression. This may produce stress concentrations at grain boundary triple points thus aiding cavity nucleation, and the possible formation of wedge cracks (fig. 1.22).

In an Al-5%Mg alloy Baik and Raj (1982b) have used the strain rate sensitivity of grain boundary sliding, determined by internal friction experiments, to predict the critical strain rate for the formation of wedge cracks. The use of slow-fast cycling promoted wedge type cavitation whereas tensile hold cycles favoured the occurrence of cavitation uniformly across the grain facet.

An alternative approach to modelling the high temperature crack growth behaviour has been to consider the effect of the dislocation structure on the fracture at elevated temperature and low frequencies. With increasing temperature and decreasing frequency a more

homogeneous dislocation structure is usually observed, for example Gell and Leverant (1973) in single crystal Mar M 200 and Clavel et al. (1980) on Waspaloy. Under conditions where homogeneous slip is encountered but creep effects are small increased transgranular crack growth rates may be obtained due to the decrease in slip reversibility (Hornbogen and Zum Gahr, 1976). If conditions arise where creep processes become dominant, intergranular creep failure may occur giving intergranular crack propagation.

However, Fournier and Pineau (1977) and Clavel and Pineau (1978) have shown that deformation of Inconel 718 at high temperature and low strain rates results in inhomogeneous plastic deformation and twin formation. Consequently Clavel and Pineau have suggested that the tendency to intergranular failure may be enhanced at the intersection of a deformation band and grain boundary. Clavel and Pineau (1982) have observed alterations to the elevated temperature crack growth of Waspaloy by prior cyclic deformation at room temperature. Under short tensile hold-time conditions, intergranular failure is then obtained. With more extended tensile hold times the crack growth reverts to being transgranular.

Similar effects have been observed in material that had been pre-strained before high temperature annealing, e.g. Dyson et al. (1976) in Nimonic 80A, Saegusa et al. (1980) and Kikuchi et al. (1982) is Astroloy. Dyson et al. consider that pre-deformation at room temperature may introduce intense slip bands which intersect grain boundaries and effect decohesion of the grain boundary or a grain boundary particle. The applied stress, τ , for crack nucleation at a particle of length p can be given by (Smith and Barnby, 1967)

$$\tau = \frac{2\gamma G}{M(1-\nu)d} \frac{p}{(d+p)^{1/2}} \quad (1.31)$$

where γ is the surface energy of the matrix material, G the shear modulus, ν Poisson's ratio and d the length of the dislocation pile up. An alternative treatment by Kikuchi et al. (1982) considers that interface decohesion occurs due to the incompatibility of deformation between the hard grain boundary particle and the matrix.

Although grain boundary cracks and cavities may be required for intergranular failure, their presence alone is not sufficient for increased crack growth rates. For these to be obtained linkage of the main crack and the grain boundary damage must occur.

Numerous analyses are available which consider that crack advance under static loading occurs by the linkage of the crack with an array of grain boundary cavities which grow in response to the elastic stress distribution ahead of the crack tip. Nix et al. (1977) consider that cavity growth is controlled by power law creep of the surrounding matrix whereas Wilkinson (1981) and Wilkinson and Vitek (1982) consider diffusion controlled growth of a single cavity adjacent to the crack tip.

During cavity growth, the build-up of a localised displacement will result in crack tip stress relaxation. Raj and Baik (1980) have used numerical methods to show that the resulting stress redistribution may give rise to non-uniform cavity growth. Crack extension then occurs as a series of small jumps determined by the time taken for the cavities to reach a critical size.

Whilst crack extension may occur by the growth of a cavity until linkage with the crack occurs, an effect may also arise due to a

decrease in the effective flow stress σ_f' ,

$$\sigma_f' = \sigma_f \left[1 - \frac{p_c^2}{\lambda_c^2} \right] \quad (1.32)$$

where σ_f is the material flow stress, p_c the cavity size and λ_c the cavity spacing. However, as Tomkins and Wareing (1977) point out, large values of (p/λ) would be required to produce significant changes in the effective flow stress.

Tomkins and Wareing suggest that under cyclic loading a more likely condition for an interaction, will be when the crack tip displacement, δ , effects cavity linkage. For an acceleration in crack growth rate to occur, the increment of crack advance per cycle must be related to the damage spacing, that is

$$\frac{\delta}{2} = (\lambda_c - p_c) \quad (1.33)$$

During tensile hold time cycling Lloyd (1979) and Michel and Smith (1980) have observed crack growth rates in excess of the estimated value of $\delta/2$ per cycle. Recently Lloyd and Wareing (1979) have rewritten equation (1.33) in terms of the grain boundary carbide size, p , and spacing, λ , as

$$\frac{\delta}{2} \approx \alpha(\lambda - p) \quad (1.34)$$

where α is a positive integer reflecting the ease of nucleation of cavities.

Satisfaction of equation (1.33) may lead to the possibility of unstable crack advance, but may only represent an upper bound for the

prediction of crack growth rates. Damage spacing below $\delta/2$ may equally represent an interaction between a fatigue crack and discrete intergranular cavitation. Lloyd and Wareing therefore consider that four main types of creep-fatigue behaviour occur,

- (i) Non-interactive; crack propagation is either transgranular, even in the presence of grain boundary damage, or intergranular when no grain boundary damage is present.
- (ii) Interactive; intergranular failure with a damage spacing of greater than $\delta/2$.
- (iii) Stable; transgranular or interactive.
- (iv) Unstable; intergranular failure occurring with a damage spacing of less than $\delta/2$.

The analyses of Min and Raj (1979) and Baik and Raj (1982a) for the formation of grain boundary cavities and cracks rely, to some extent, on a degree of asymmetry of loading to produce local stress concentrations, and are therefore not able to explain simply the observations of increased crack growth rates with reductions in frequency. Ohnuma et al. (1973) suggested the transition in behaviour to be associated with a critical strain rate within the plastic zone. The elastic strain rate $\dot{\epsilon}_t$ at which the transition is observed can be given by

$$\dot{\epsilon}_t = \frac{4\sigma_{yc}}{E} v_t \quad (1.35)$$

where σ_{yc} is the cyclic yield stress, E Young's modulus and v_t the transition frequency. Comparison of the calculated values of $\dot{\epsilon}_t$ with the strain rate observed for intergranular crack propagation in tensile tests showed good agreement.

1.9 Summary

At elevated temperatures crack growth rates are observed to be dependent on frequency, environment and waveform. At frequencies greater than approximately 1Hz transgranular crack growth rates show little sensitivity to frequency. With decreasing frequency, crack growth rates increase significantly with an accompanying transition to intergranular failure. Variations in the waveform may give rise to more significant increases in the crack growth rates with unstable crack growth reported. Models based on environmental and creep interactions have been proposed but none can satisfactorily explain the behaviour.

Enhanced crack tip corrosion at elevated temperature may give rise to dissolution of material, but may require the presence of bare metal or rupture of the existing oxide layer, processes which may be dependent on crack tip plasticity. Preferential grain boundary diffusion of species leading to embrittlement has also been suggested and yet the effects of temperature and frequency are maintained in inert atmospheres, in single crystal material (Gell and Leverant, 1973) and plasticity dependent processes have been observed to occur adjacent to the crack tip (Michel and Smith, 1980).

The relaxation of crack tip stresses by cavitation may give rise to linkage of the main crack with arrays of grain boundary cavities, thus leading to increased growth rates. In this situation, it has been suggested (Min and Raj, 1979) that the rate at which stress relaxation occurs may impose constraints on the loading waveform. Similarly, stress relaxation will lead to the redistribution of crack tip stresses allowing failure mechanisms to extend over greater distances

(Bassani, 1981; Raj and Baik, 1980).

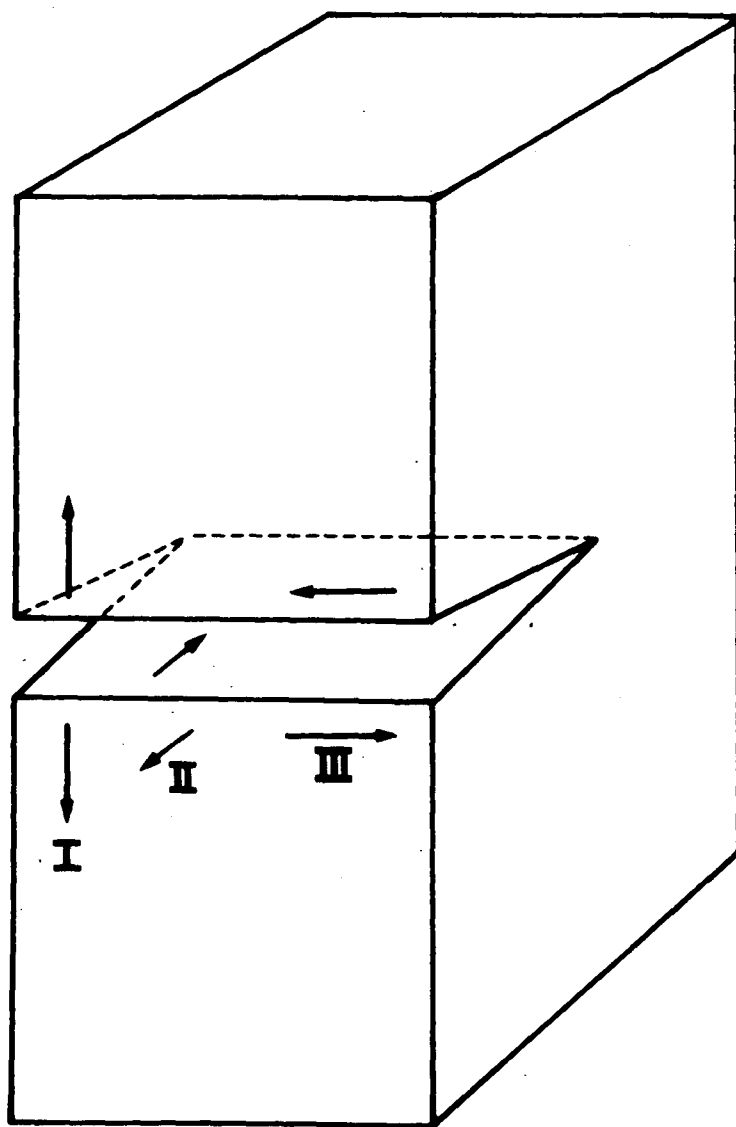
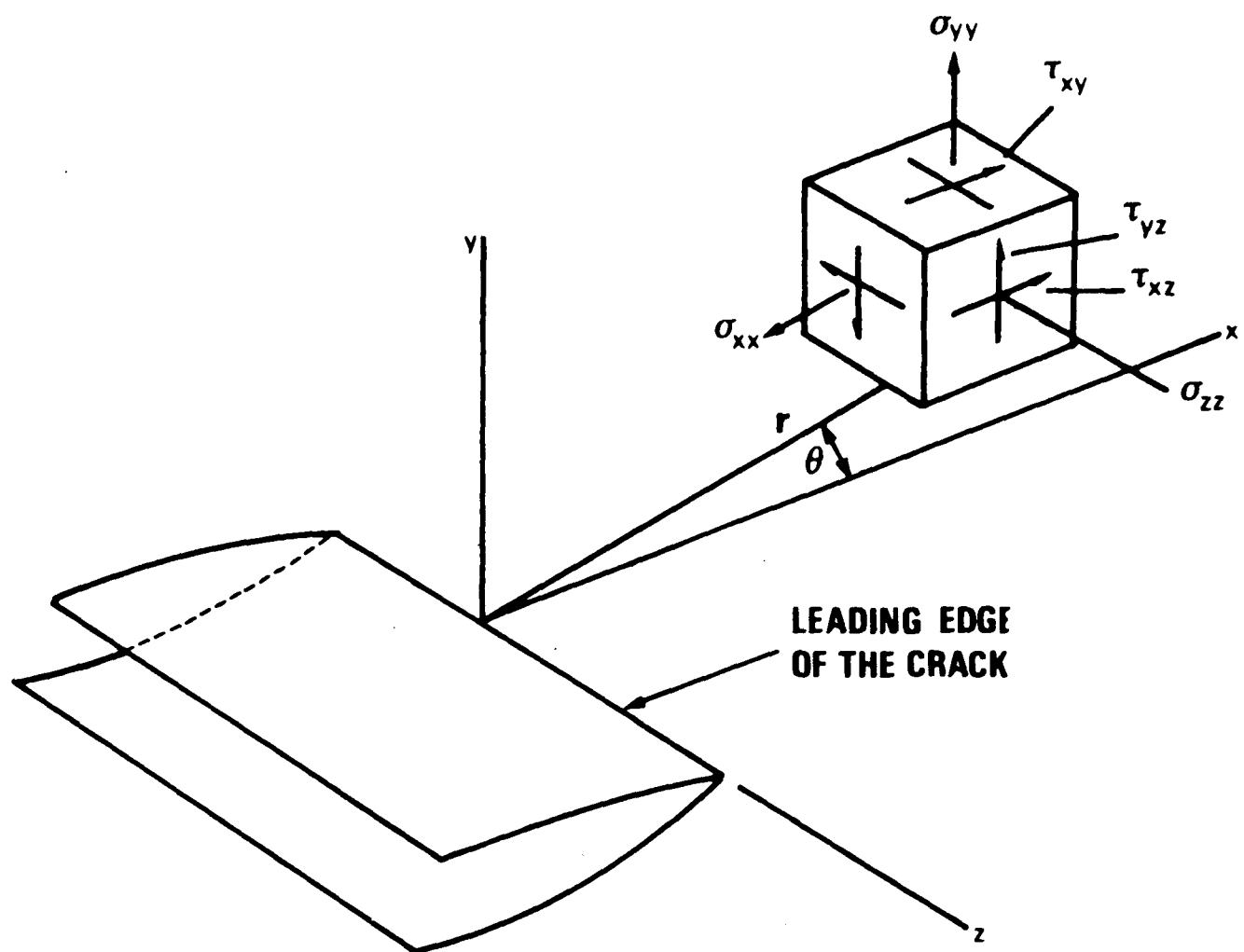
To investigate the temperature and frequency dependence of fatigue crack growth rates it was decided to study the crack growth behaviour of a nickel based superalloy. To eliminate the possible ambiguity of interpretation associated with studies conducted on polycrystalline material it was decided to use single crystals of Mar M 002. A study of the temperature and frequency dependence of the crack growth rates in conjunction with a detailed examination of the crack tip plastic zone was planned to help distinguish the interactions between the crack growth mechanisms and environmental and creep processes.

Figure 1.1

Co-ordinate system for the crack tip stresses.

Figure 1.2

Modes of crack loading.



- I OPENING MODE**
- II SLIDING MODE**
- III TEARING MODE**

Figure 1.3

Mode I crack under biaxial stress.

Figure 1.4

Distribution of elastic and creep strains ahead of
crack tip.

(After Riedel, 1978).

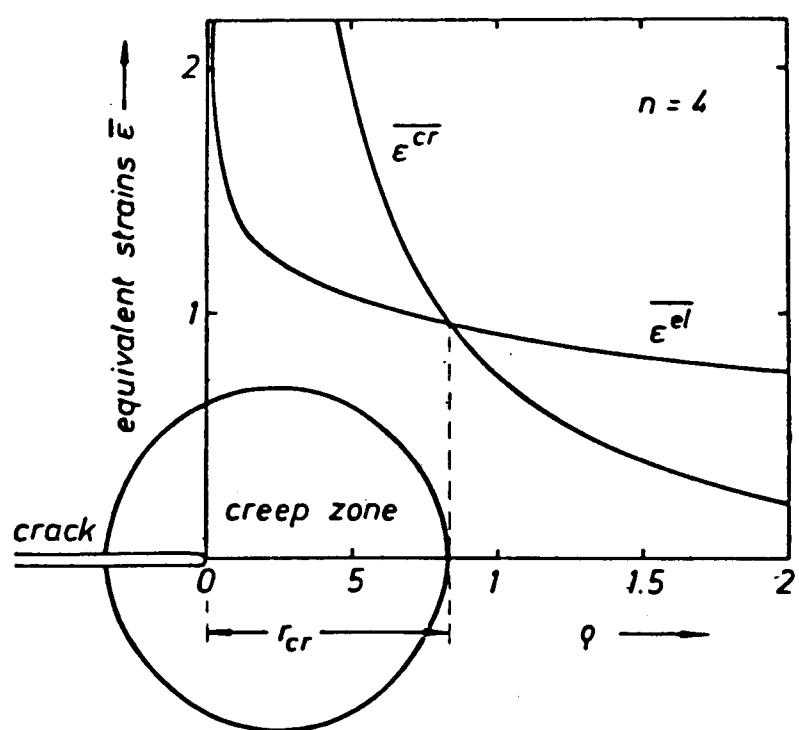
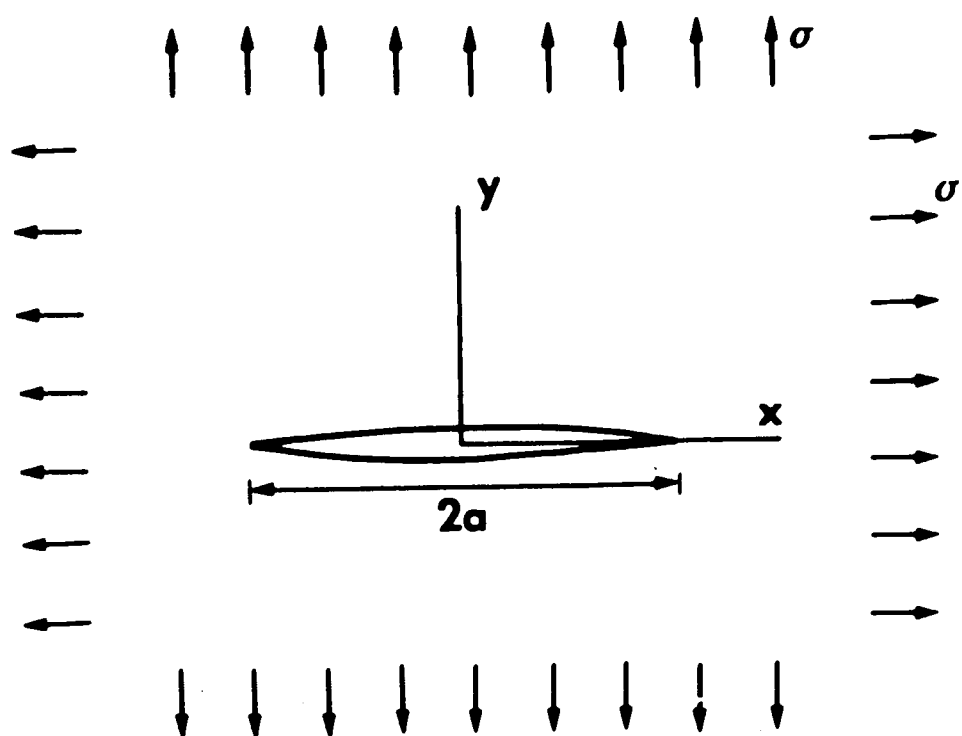


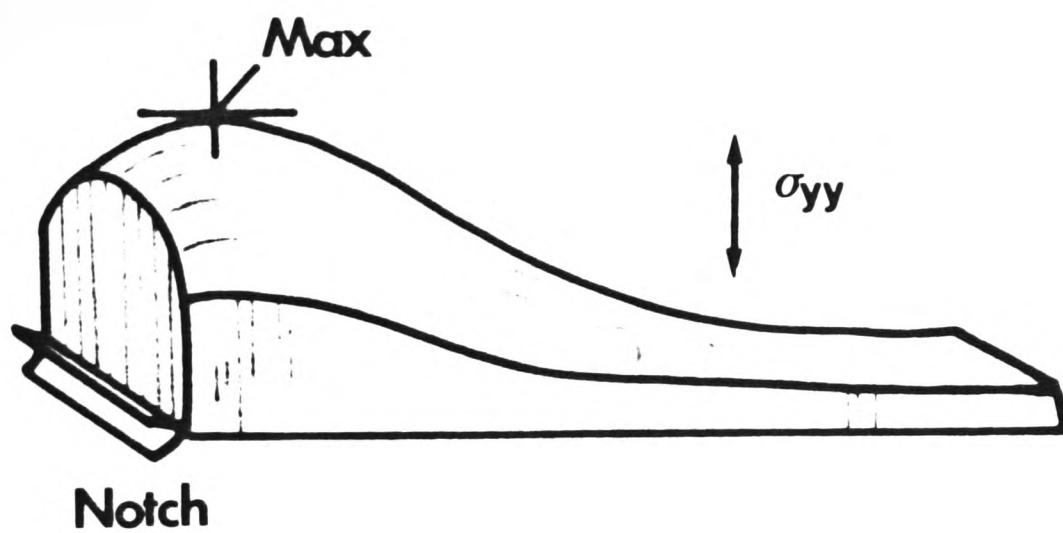
Figure 1.5

Variation of crack tip stresses with position
ahead of the crack tip.

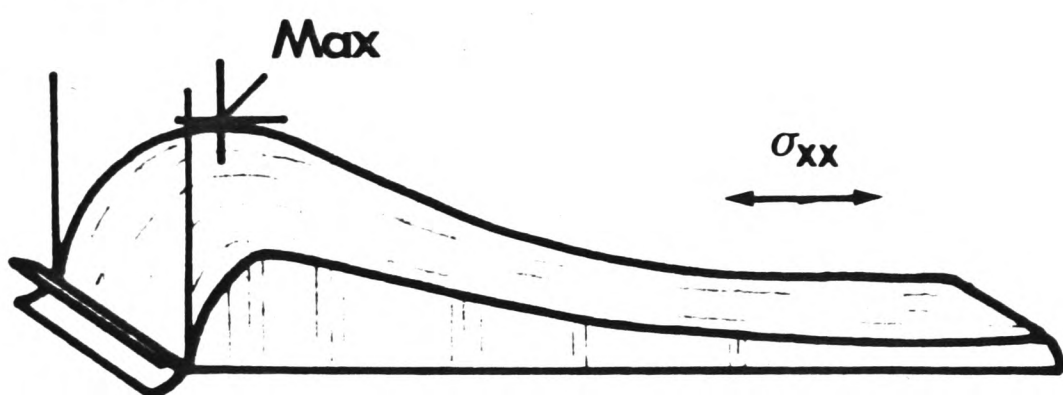
(After Boyd, 1972).

- a) σ_{yy}
- b) σ_{xx}
- c) σ_{zz}
- d) σ_{zz} against thickness for thin and thick
plate.

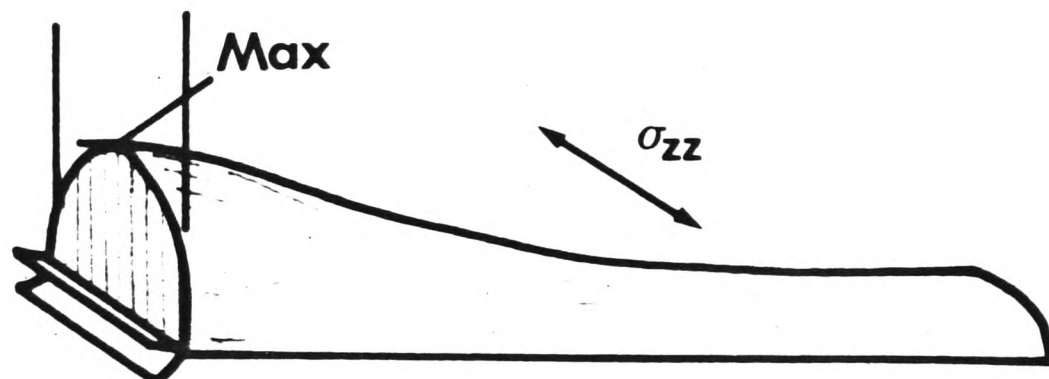
a)



b)



c)



d)

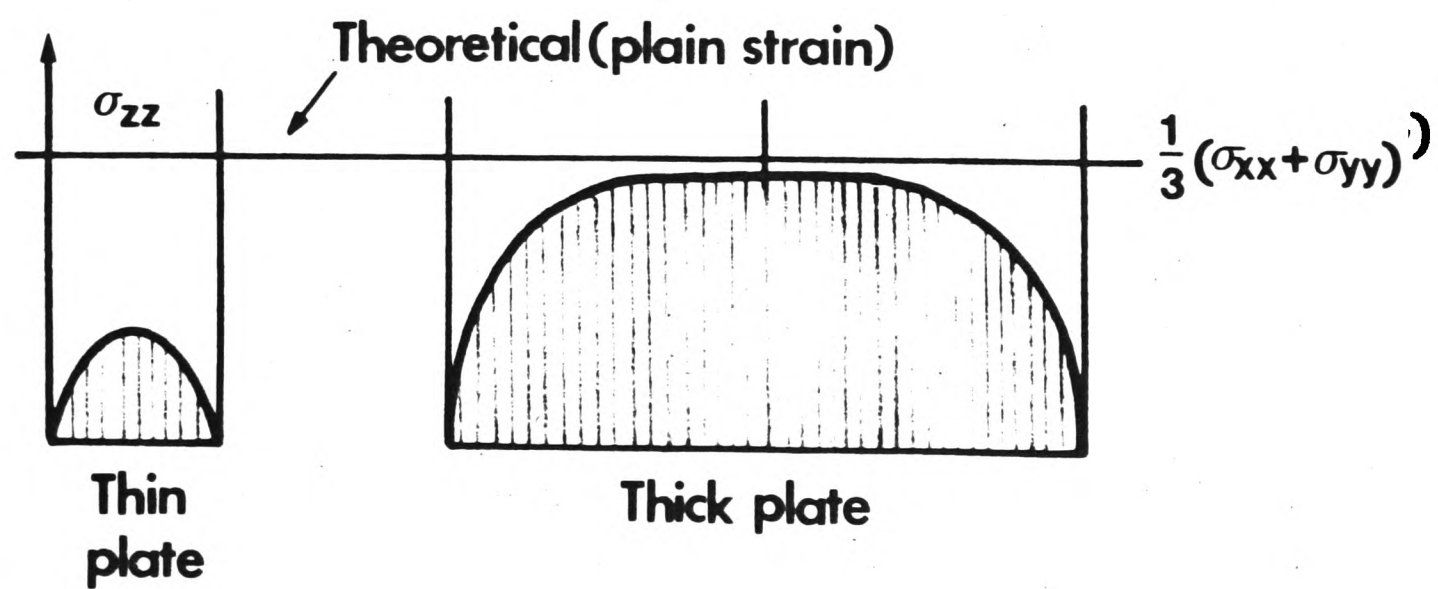


Figure 1.6

First estimate of the crack tip plastic zone size.

Figure 1.7

Second estimate of the crack tip plastic zone
size.

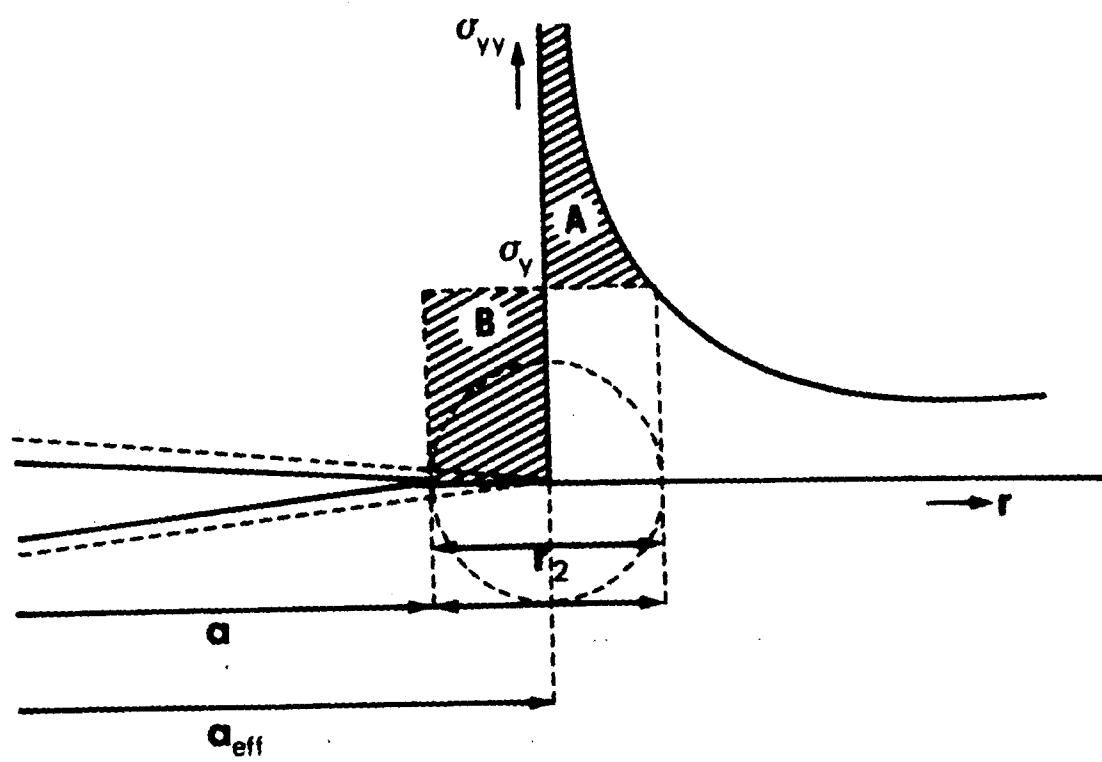
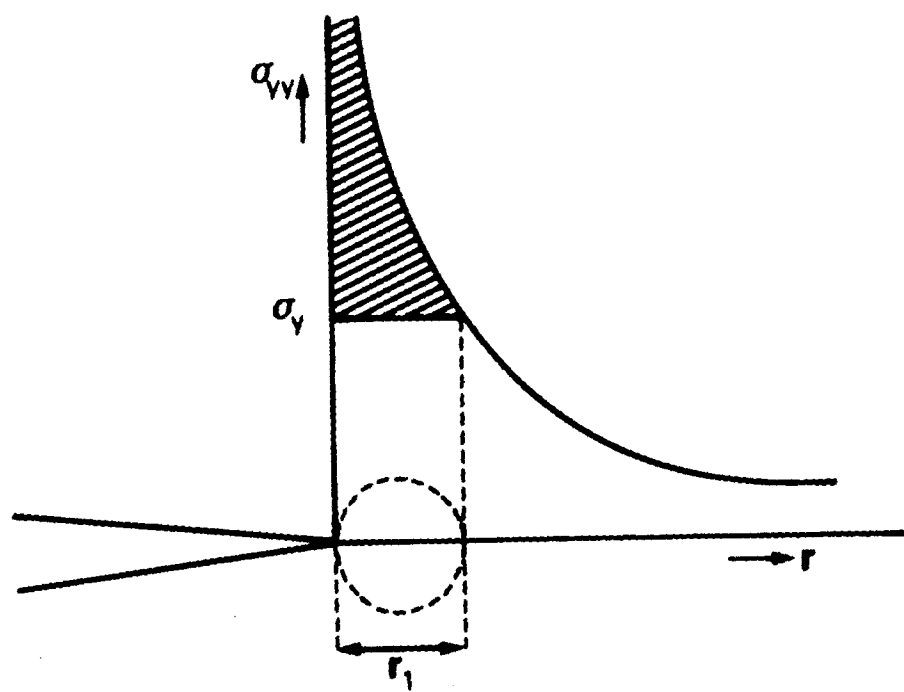


Figure 1.8

Dugdale's model for the crack tip plastic zone
size.

Figure 1.9

Variation of the plastic zone size for plane strain
and plane stress.

(After Broek, 1978).

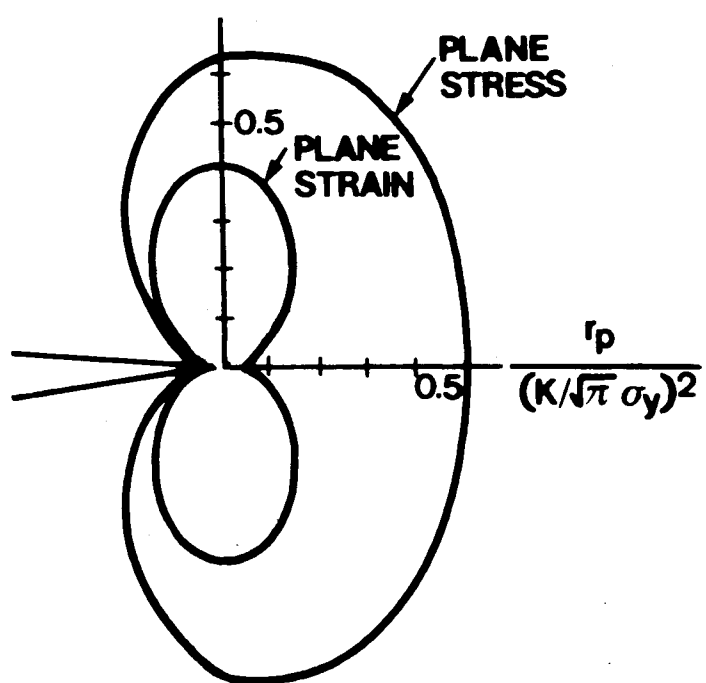
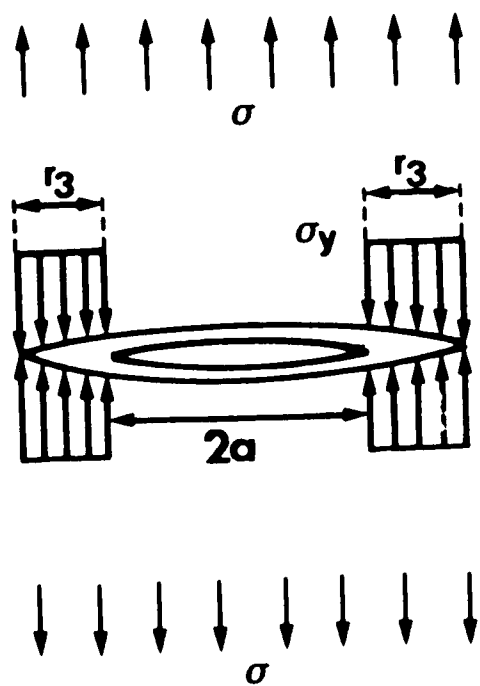


Figure 1.10

Effect of specimen thickness on the plastic zone.

Figure 1.11

Variation of the angular functions $F_1(\theta)$ (dashed line) and $F_{Cr}(\theta)$ (solid line) for plane strain (upper half) and plane stress (lower half) with varying creep exponent.

(After Riedel and Rice, 1980).

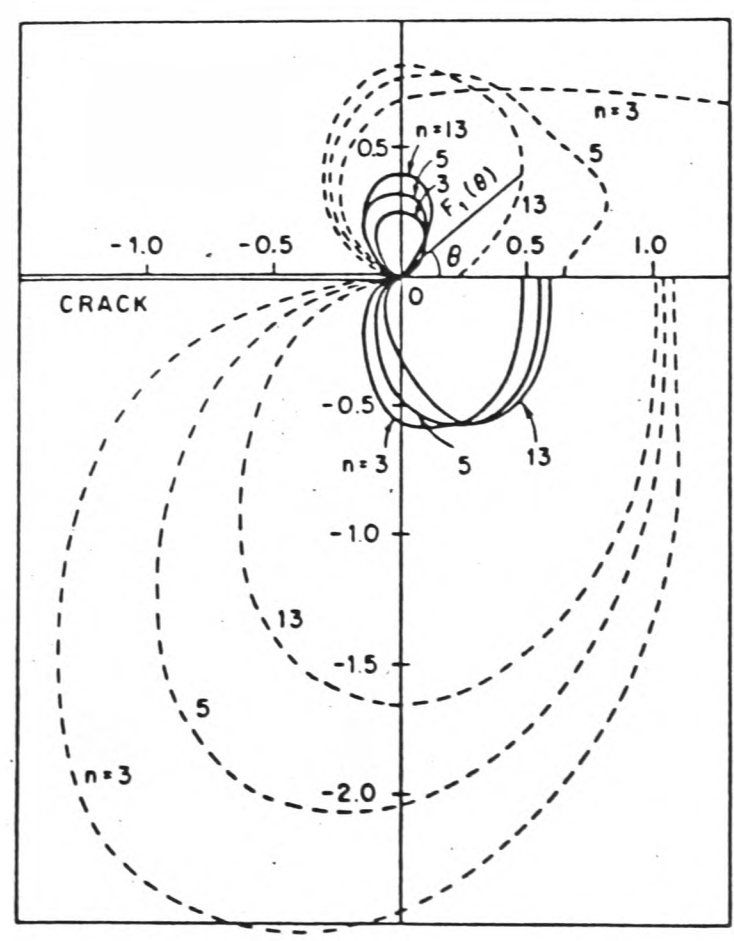
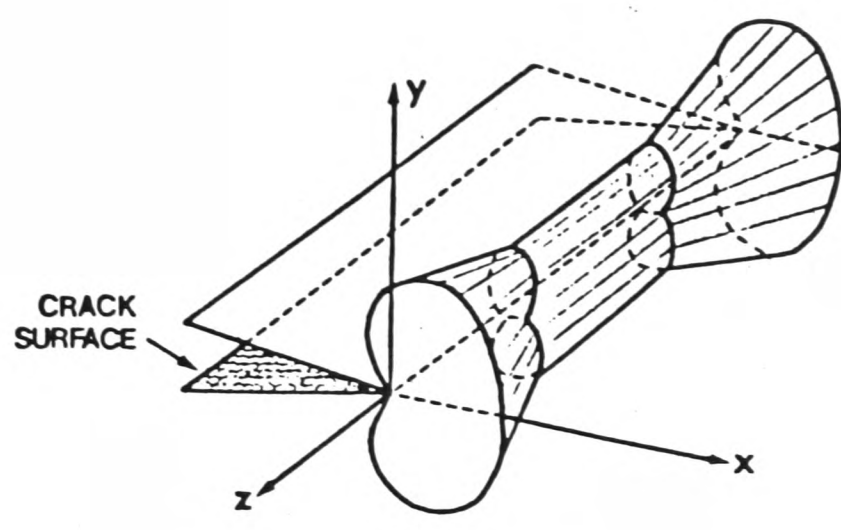


Figure 1.12

Development of the cyclic plastic zone.

(After Rice, 1967).

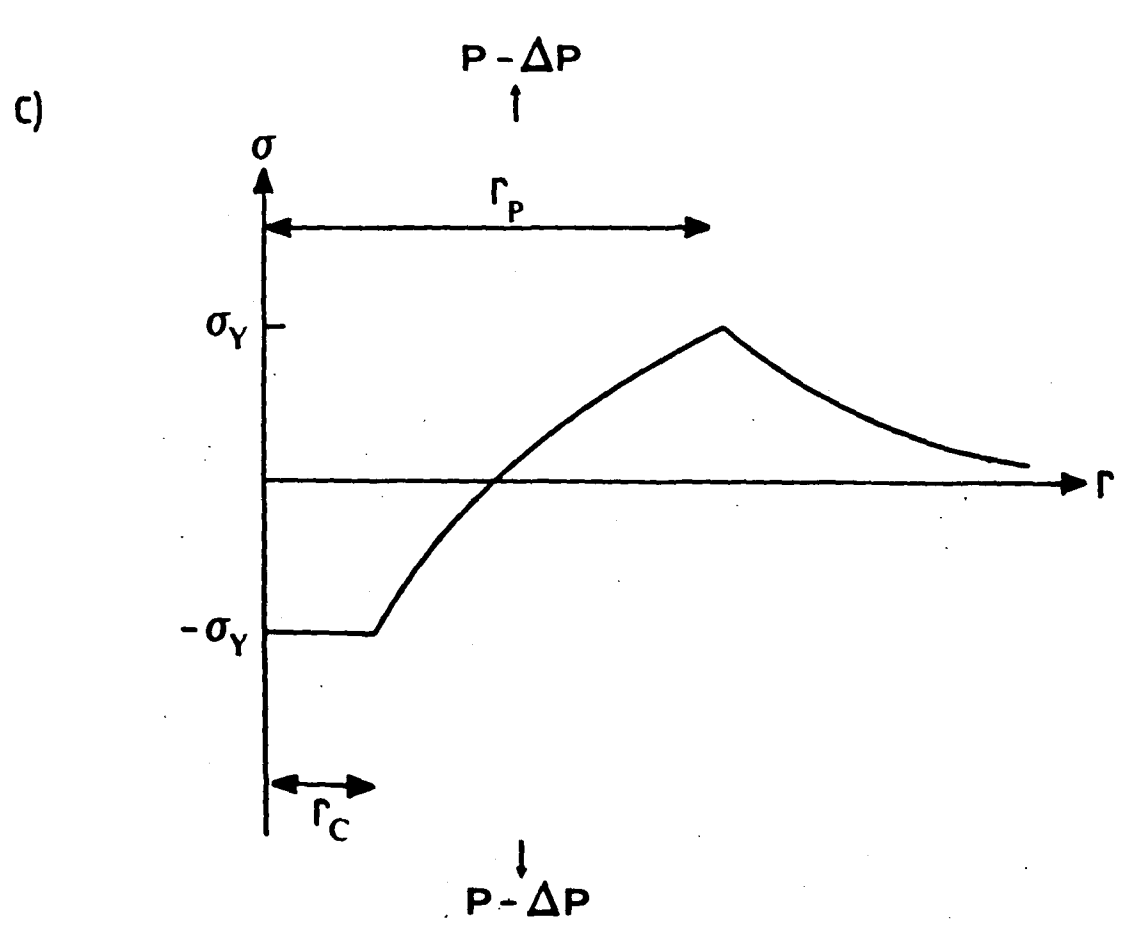
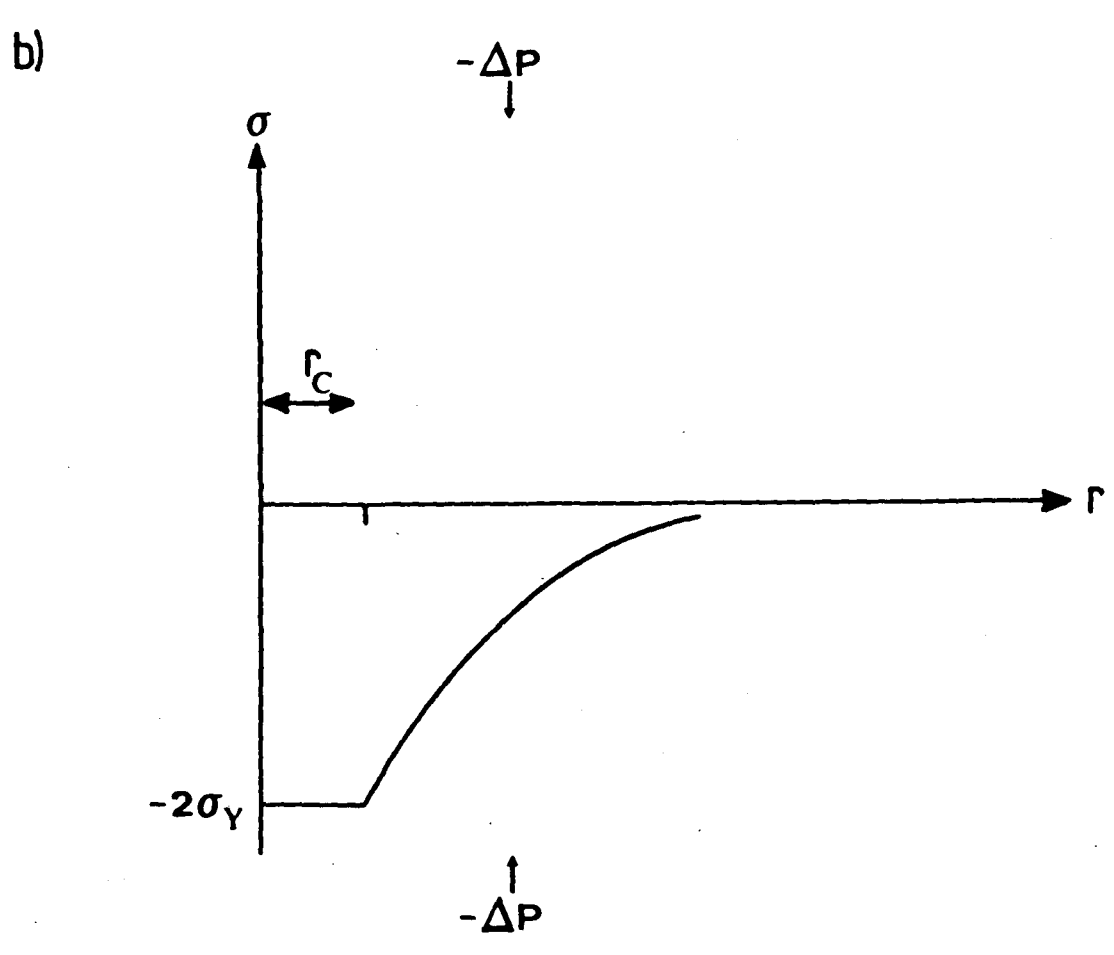
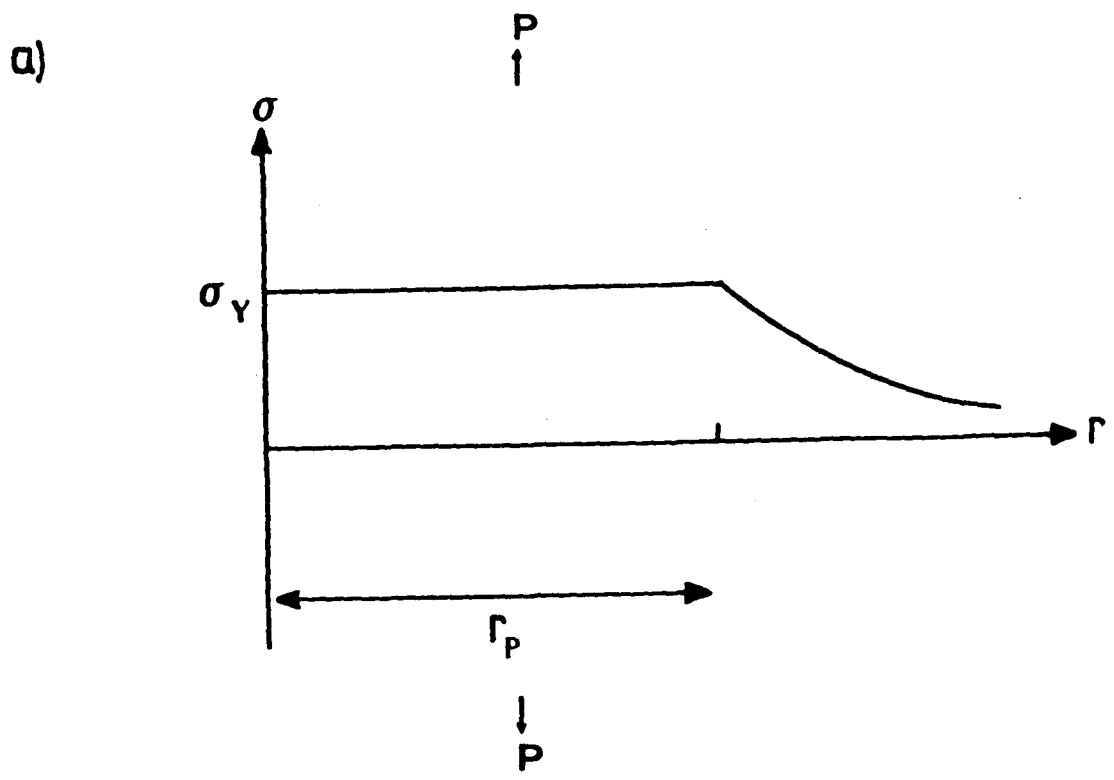


Figure 1.13

Broad classification of the fracture mechanisms at
low and high homologous temperature.

(After Ashby, 1979).

Figure 1.14

Relationship between the fatigue crack propagation
rate per cycle (da/dN) and the cyclic stress
intensity (ΔK).

(After Beevers et al., 1975).

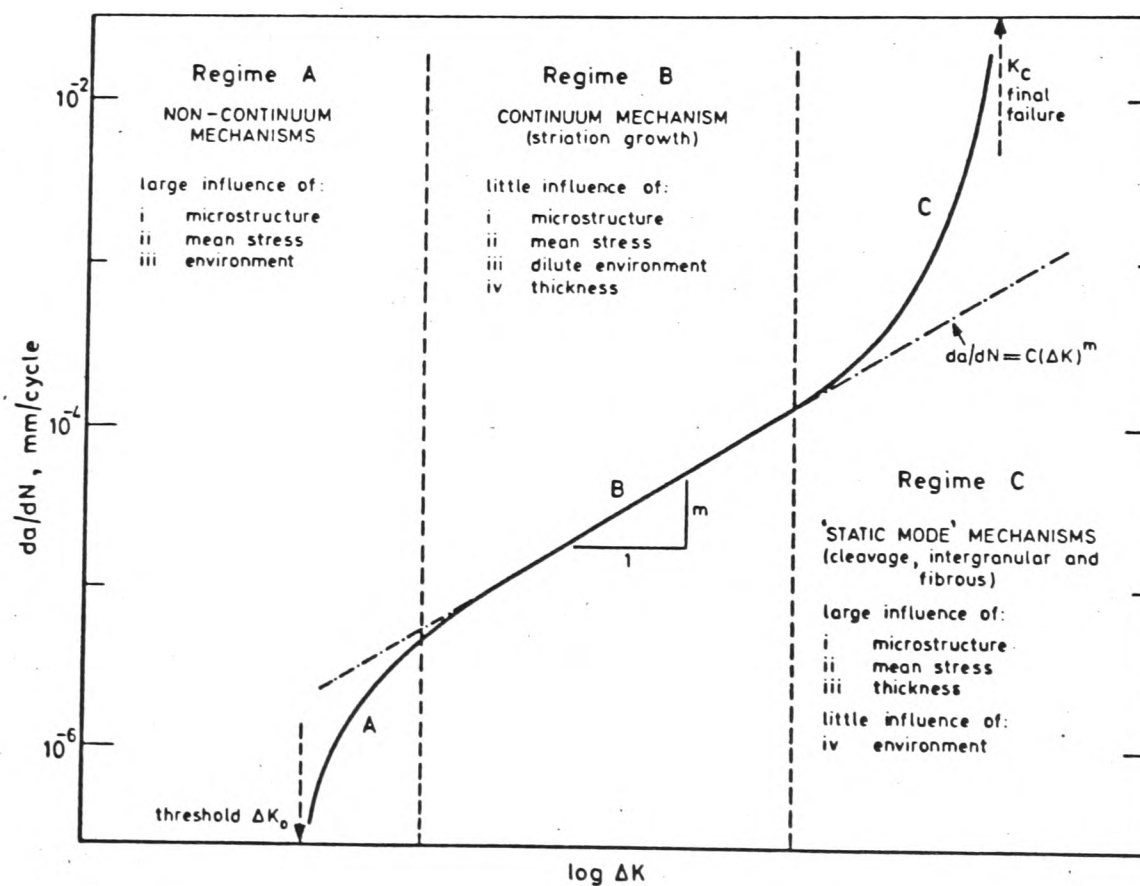
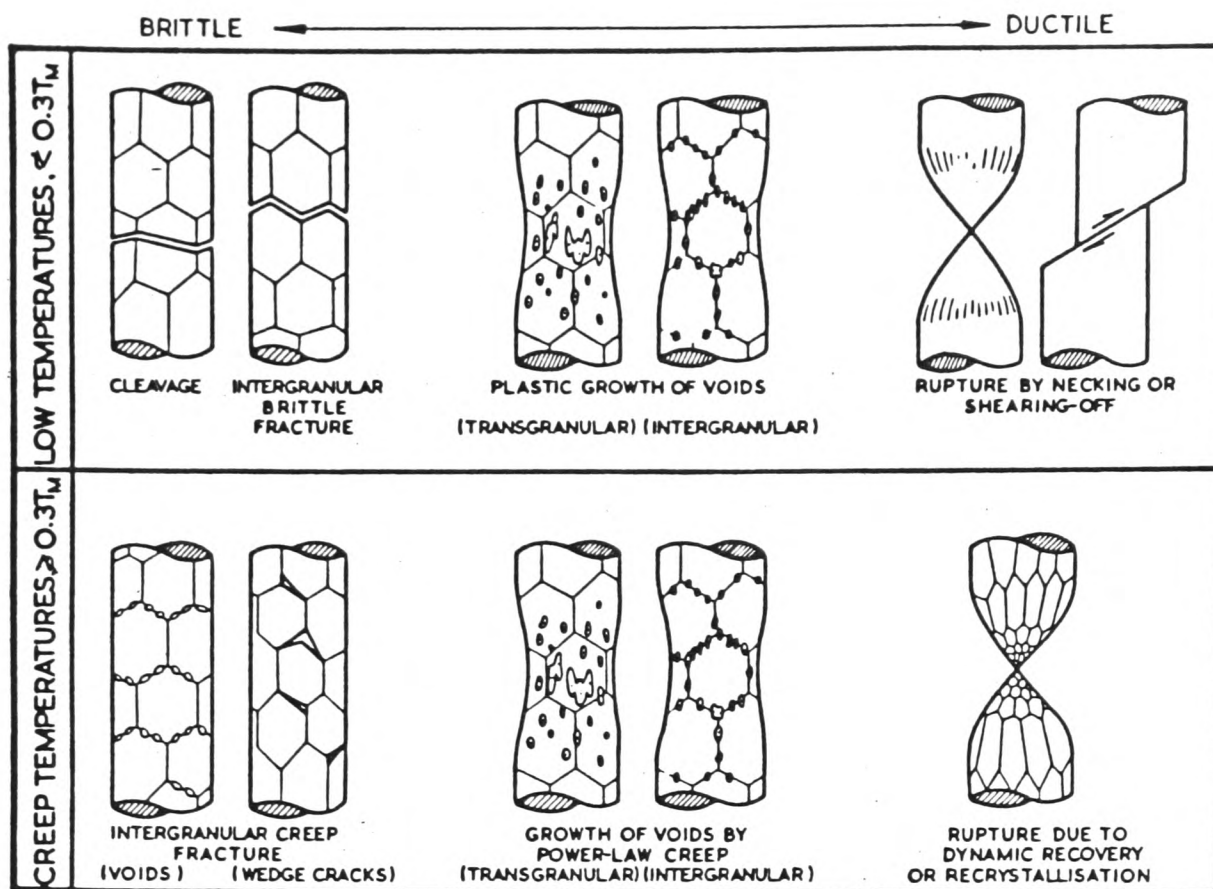


Figure 1.15

Schematic representation of the effect of crack
tip closure on cyclic stress intensity.

(After Suresh and Ritchie, 1982).

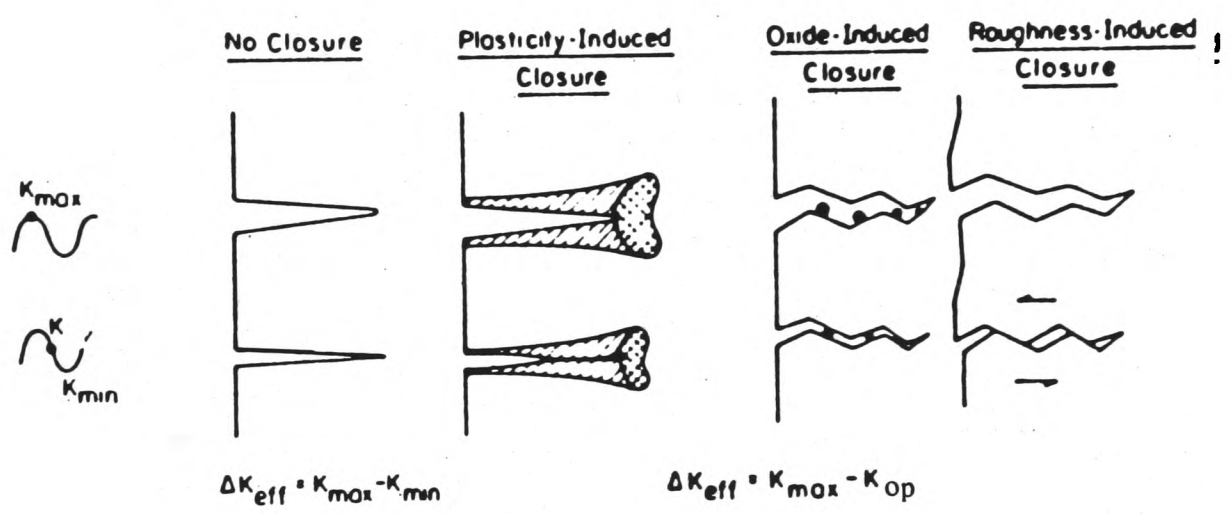
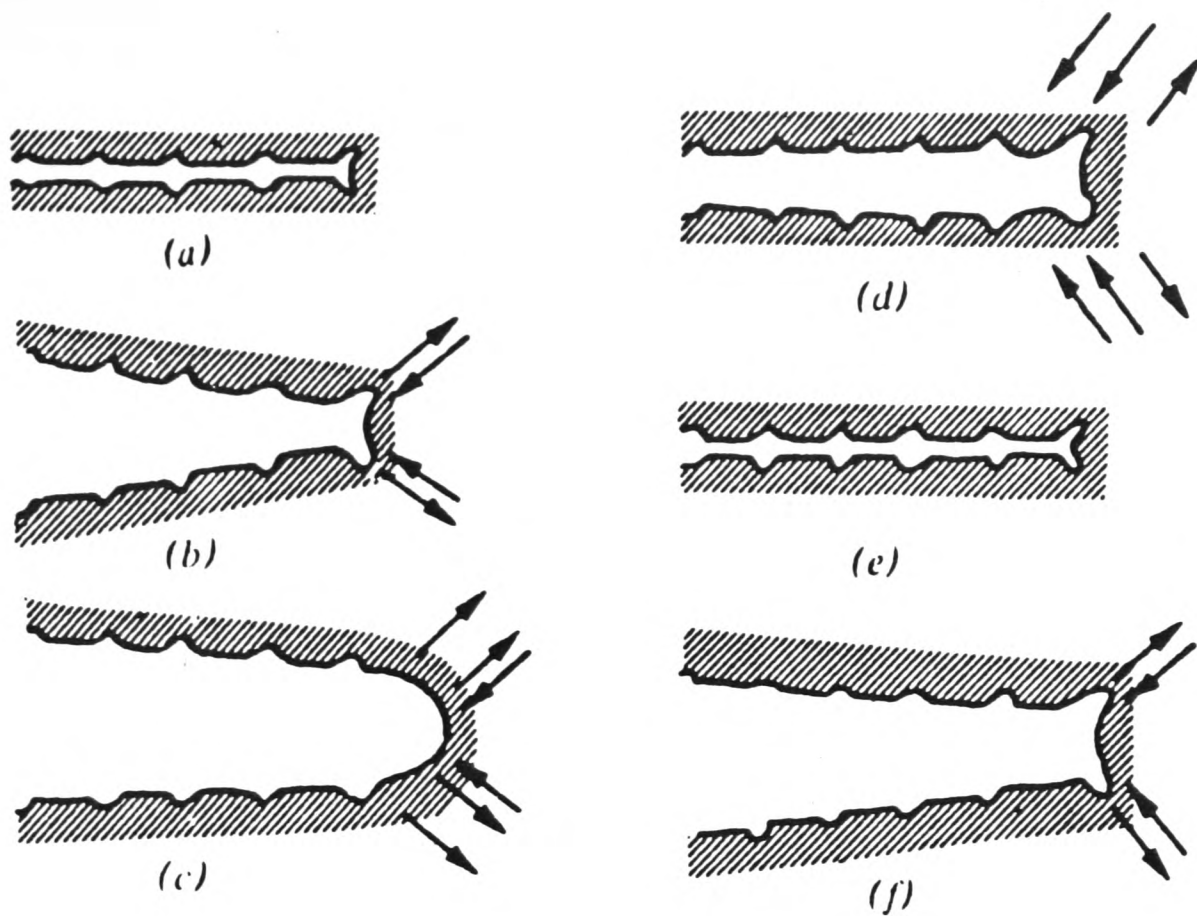
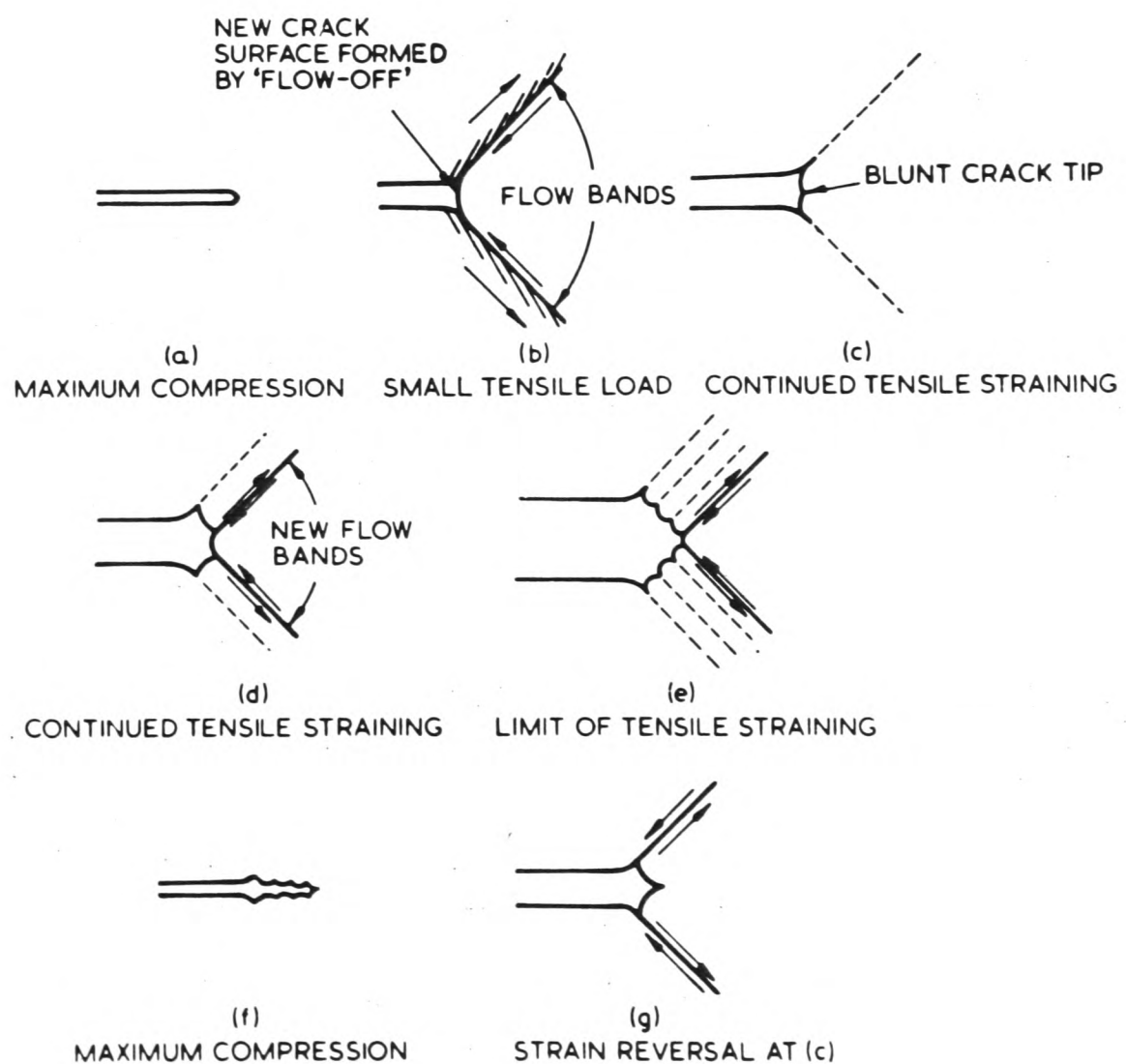


Figure 1.16

Schematic representation of the mechanisms for striation growth suggested by (a) Laird (1967) and (b) Tomkins and Biggs (1969).



(A)



(B)

Figure 1.17

Schematic representation of the proposed mechanisms of crack growth in superalloys,

- a) fracture by the nucleation of small cracks on the slip plane,
- b) fracture as a result of crack nucleation on the cleavage plane.

(After Sadananda and Shahinian, 1981d).

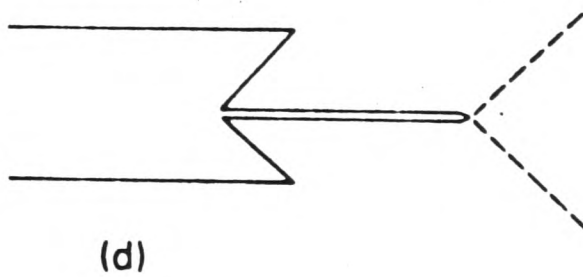
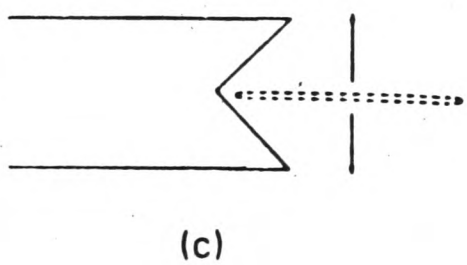
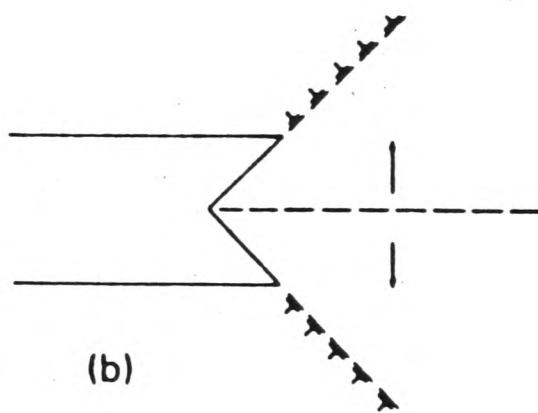
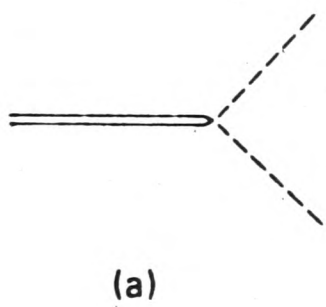
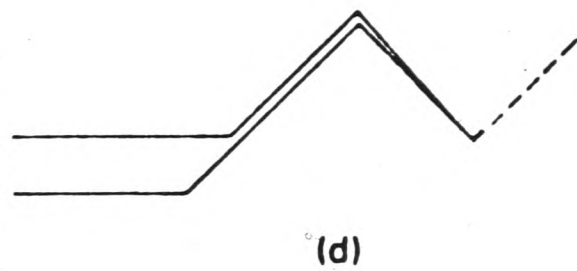
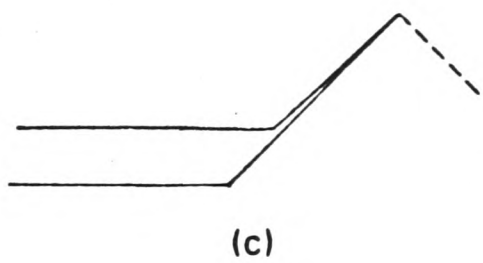
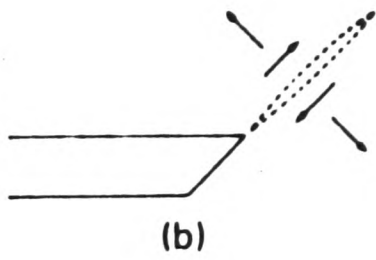
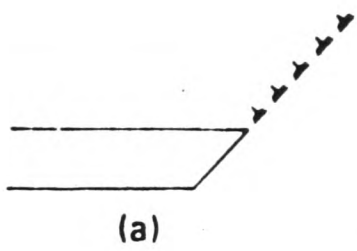


Figure 1.18

Effect of frequency on the elevated temperature fatigue lifetimes of Udimet 700.

(After Organ and Gell, 1971).

Figure 1.19

Effect of frequency on the elevated temperature fatigue crack propagation rate and fracture path.

(After Ohnuma et al., 1973).

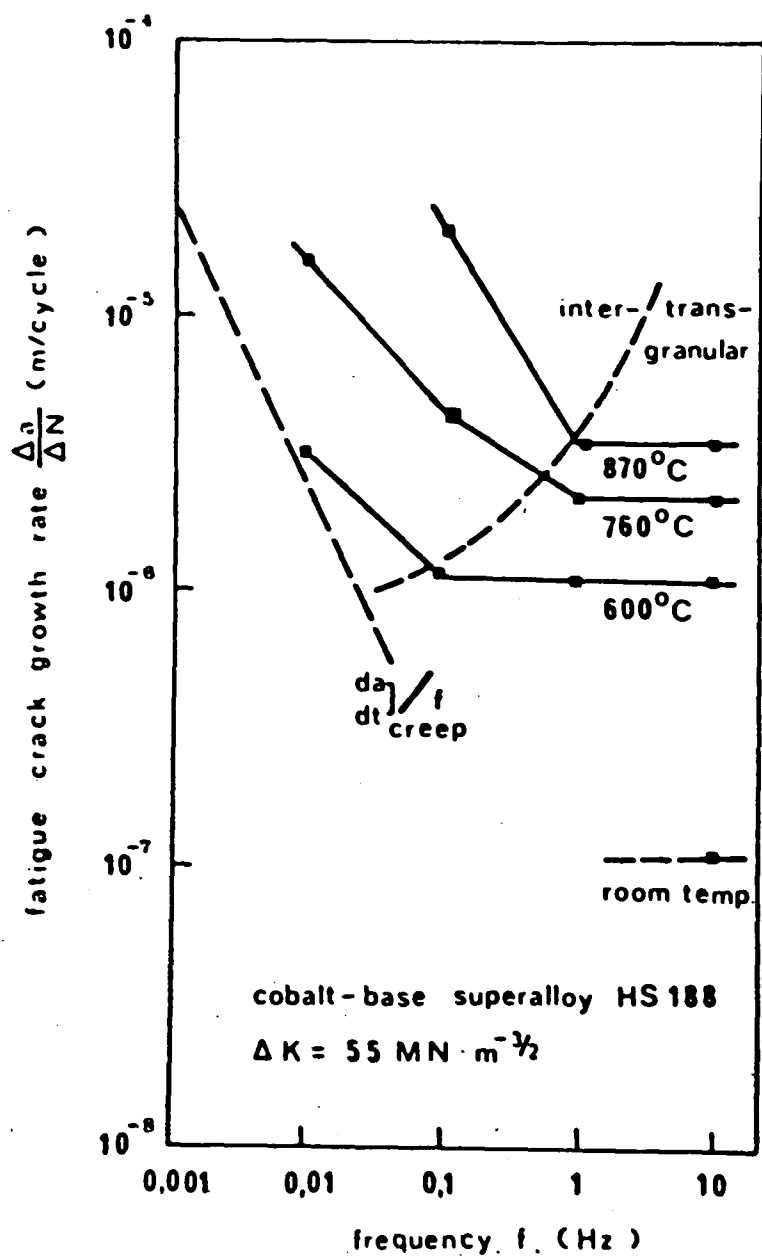
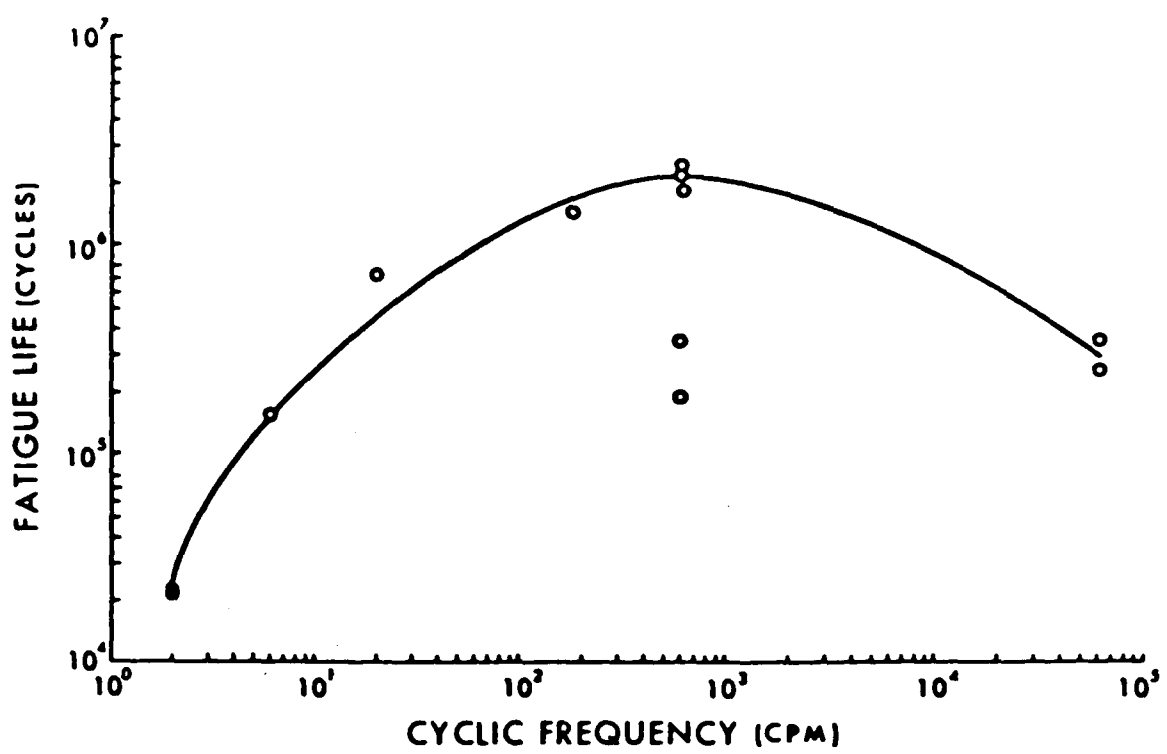


Figure 1.20

Effect of frequency and environment on the low cycle fatigue crack propagation of A286.

(After Solomon and Coffin, 1972).

Figure 1.21

Proposed sequence for embrittlement of material ahead of the crack tip.

(After Wei and Simmons, 1981).

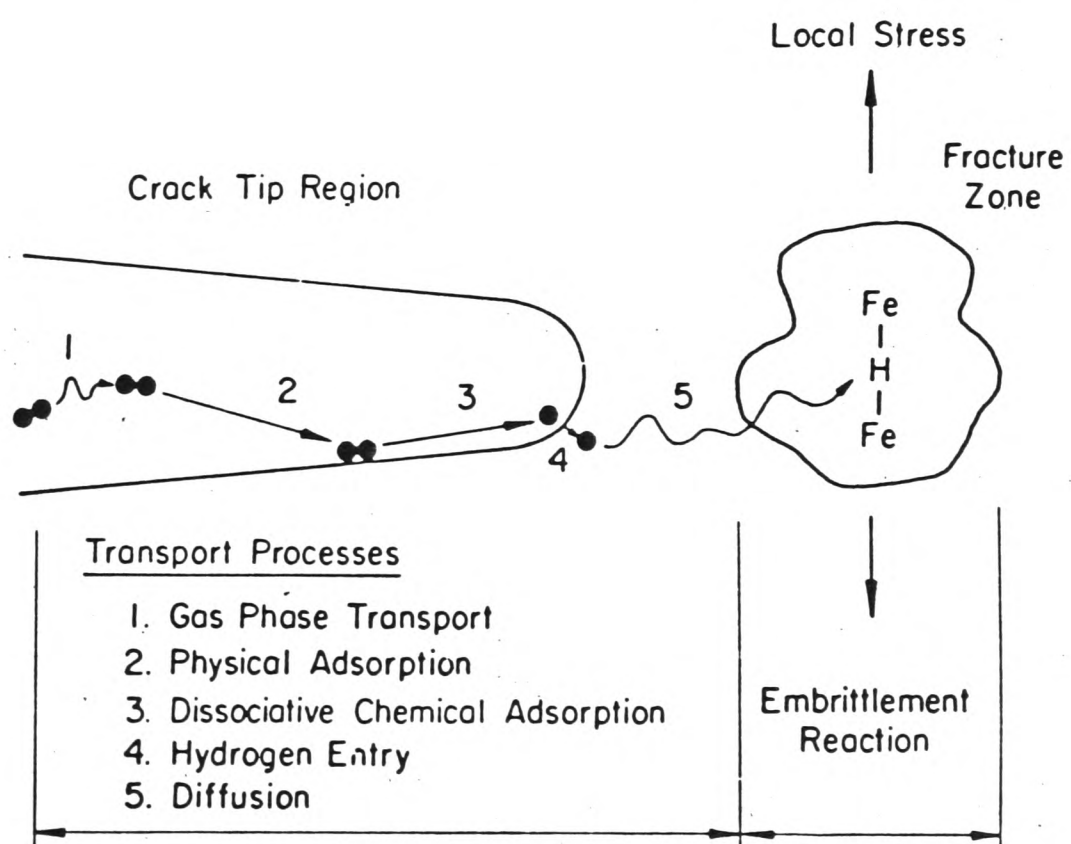
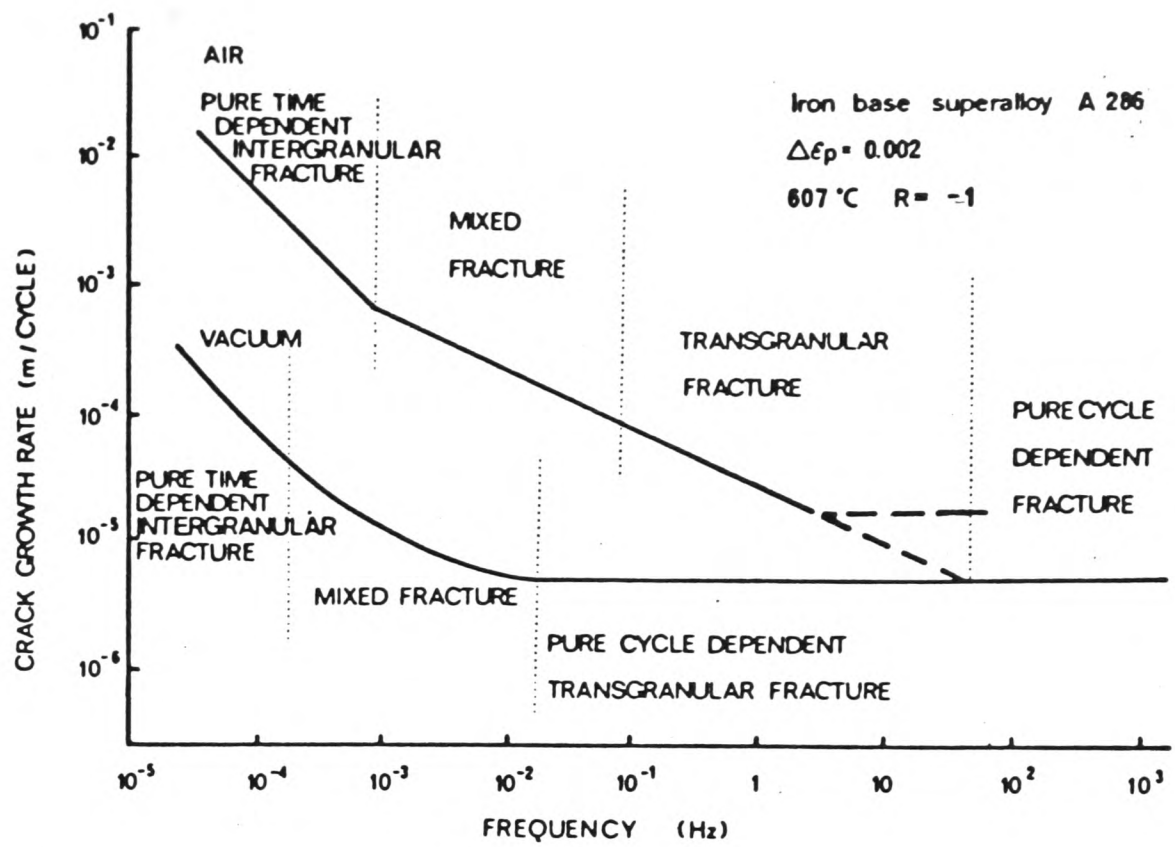
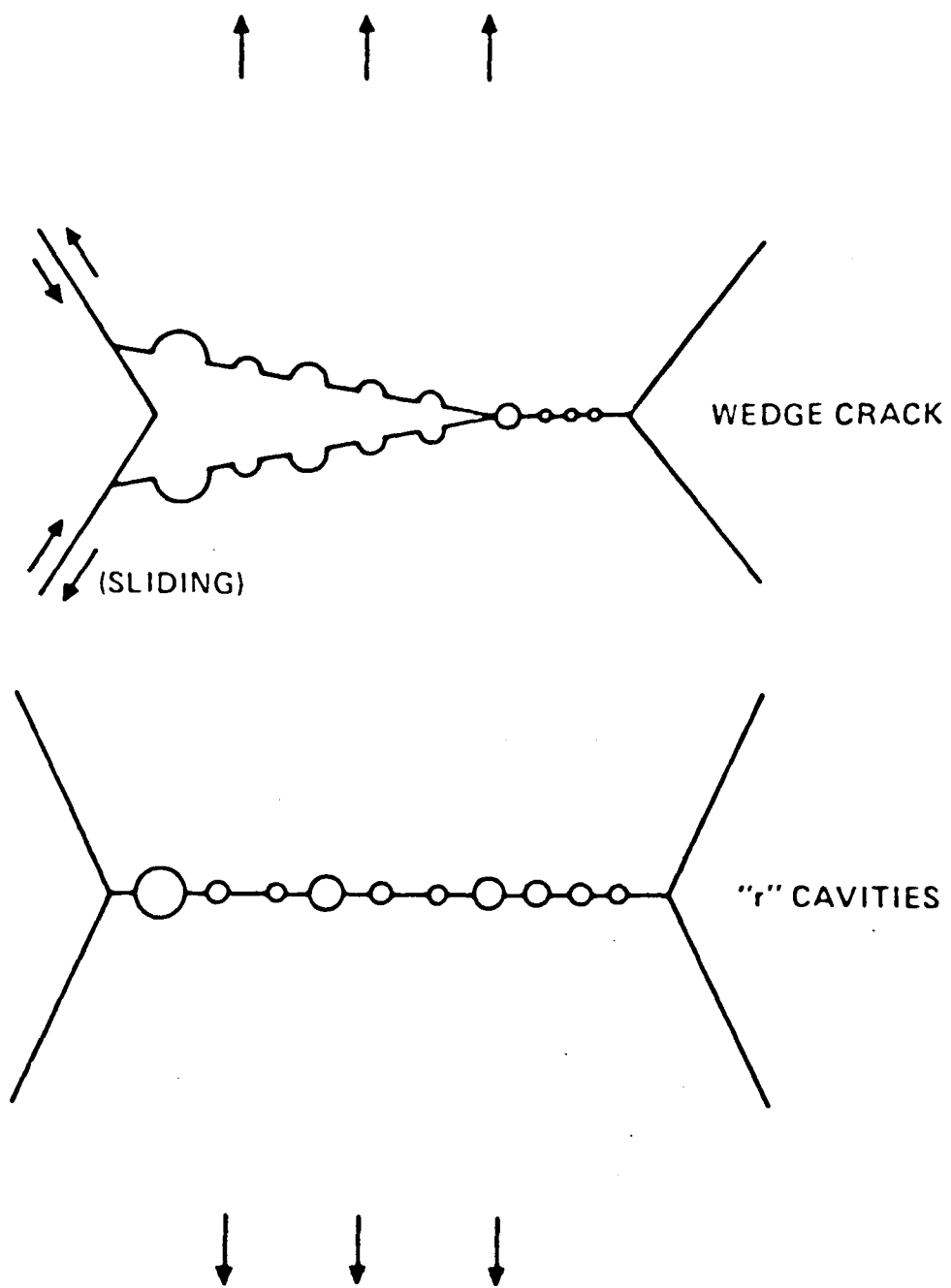


Figure 1.22

- a) Grain boundary sliding giving rise to wedge cracking.
- b) Cavity nucleation on the grain boundary.

(After Baik and Raj, 1982).



Chapter 2

Material and Experimental Techniques

2.1 Material

The material used for this work was single crystal Mar M 002, a nickel based superalloy, the composition of which is given in Table 2.1. The single crystals were produced by The National Gas Turbine Establishment using a modified directional solidification process (see e.g. Sims and Hagel, 1972; Meetham, 1981) which gives a single crystal having a growth direction within approximately 5° of a $\langle 100 \rangle$ lattice direction. However the orientation in the plane normal to the growth direction is uncontrolled. The material was heat treated for 1 hour at 1100°C , followed by an ageing treatment of 16 hours at 860°C .

The resulting microstructure consisted of approximately 60 volume percent of γ' of $0.5\mu\text{m}$ edge dimension, a small volume fraction of spherical γ' of approximately $150\text{\AA}$ radius, together with about 6 volume percent of a γ/γ' eutectic. A number of 'script' carbide particles were also present.

2.2 Specimen Geometry

Numerous specimen geometries and sizes have been used to study fatigue crack propagation at both ambient and elevated temperatures, e.g. compact tension (C.T.), single edge notch (S.E.N.) and centre cracked tension (C.C.T.) specimens. In view of the complex stress

distribution associated with the mode of loading of S.E.N. tension specimens, several alternative relationships between the stress intensity factor, K , and the applied load exist. For pin-loaded S.E.N. tension specimens the compliance function due to Brown and Srawley (1966) is commonly used. However, the ratio of crack length to specimen width, a/W , for which this calibration is applicable is limited. For S.E.N. tension specimens subject to a loading configuration which restricts the rotation of the specimen ends there are numerous expressions relating the applied load to the crack tip stress intensity (Hams, 1967; Sih, 1973; James, 1976; Rooke and Cartwright, 1976; British Standards Institution, 1977; Gray, 1977). Attempts to utilise these expressions as control parameters in the present work revealed not only a large degree of scatter within any one expression, but also a lack of correlation between expressions.

Centre cracked tension (C.C.T.) specimens do not suffer from the ambiguity of stress analysis inherent in the S.E.N. specimens since any Mode III loading of the crack is restrained by the specimen itself. Further, the crack tip stress intensity factor has been determined to be valid for a wide range of a/W (Isida, 1955). A further advantage of this type of specimen geometry is the separation of the point of loading and the crack plane, which allows the small area of the crack plane to be heated to high temperatures whilst permitting the use of conventional materials for the loading assembly.

The specimen geometry used in the work was C.C.T., with specimen dimensions in accordance with those given by A.S.T.M. E647-78T (1978). Stress intensity factors were determined from the expression due to

Feddersen (1967):

$$K = \frac{P (\pi a)^{1/2}}{BW \sec(\pi a/W)} \quad (2.1)$$

where K is the stress intensity factor,

P is the load,

B is the specimen thickness,

W is the specimen width and

a is the half crack length.

2.3 Specimen Preparation

The as received material was in the form of cylinders of approximately 40mm in diameter and 110mm in length, with a tapered end approximately 40mm in length. The material used was initially checked for the presence of "stray" grains. One end of the block was mechanically ground and polished, section 2.9, and etched in a solution of 10% hydrochloric acid in methanol, the conditions for which are given in Table 2.2. This enabled a visual examination of the carbide structure to be made allowing the presence of any "stray" grain boundaries to be determined.

The orientation of each block was determined using the Laue back reflection technique, thus allowing specimen blanks to be produced with faces parallel to {100}. Some specimens were also prepared with the vertical faces parallel to {110} and {210}.

Specimen blanks were rough cut to be oversize by 1 to 2mm using a Wickman spark machine. The specimen blanks were ground to size using

an Elliot grinding machine fitted with a coarse abrasive wheel operating at 2670r.p.m. Material was removed in steps of 0.01mm. A plentiful supply of cooling water was required and the specimens were frequently reversed during the grinding process to minimise the possibility of distortion.

A central notch was obtained by spark machining on a Wickman Erodomatic machine using a rectangular tool 2mm in width and 0.5mm in thickness in conjunction with an oil jet operating at a pressure of 5lbs/in². The pressurised oil effects removal of any cracked oil products from the central slot allowing the even removal of material throughout the thickness. The notch was extended by spark machining using 38 gauge copper wire operating on a fine setting. This produced a parallel-sided notch with a semi-circular root of small radius. The depth of the notch was controlled to produce a symmetrical notch of known length. The dimensions of the final specimen are shown in fig. 2.1.

2.4 Description of the Apparatus

Fatigue testing was performed under axial loading conditions and crack growth rates determined. Since crack growth rates are thought to be dependent on the temperature of testing and the frequency of loading, both these parameters were varied independently.

2.4.1 Fatigue Machine

Fatigue testing was performed using a Dowty-Mand servo-hydraulic testing machine with a loading capacity of 25kN. Its operation depended upon a closed loop control system using either load or

displacement feedback signals from an actuator capable of performing fatigue tests through a range of dynamic loads. Pressurised hydraulic fluid for the operation of the actuator was supplied from a hydraulic power pack. The cycling frequency and amplitude of the actuator were controlled by a Moog servo-valve.

Modifications to some of the original Dowty electronics had previously been carried out, Cowling (1980). Upon commencement of the work replacement of the remaining Dowty electronics was undertaken. The load control and load measurement units were replaced by solid state systems supplied by Mand Testing Machines Ltd. Also, the frequency generator was replaced by a Mand function generator capable of operating at frequencies of 10^2 to 10^{-5} Hz. In addition a Mand 25kN actuator, with a stroke of ± 25 mm, capable of applying a smooth loading waveform was installed. Load measurement was by means of a Novatech load cell and a peak reading digital voltmeter. The stability of the system was monitored and inherent fluctuations were determined to be better than $\pm 0.5\%$ of the lowest applied load.

2.4.2 Operation of the Fatigue Machine

Prior to operation of the machine the following points were checked:

- (i) The air cooling system or the water cooling system for the oil reservoir was connected and operational.
- (ii) The power pack contained sufficient Shell Tellus 37 oil.
- (iii) All the hydraulic lines were open.
- (iv) The control electronics were switched on and allowed to stabilise for at least 30 minutes.

Before starting the hydraulic power pack the electronics panel (refer to fig. 2.2) was checked according to the following conditions:

- (i) Oscilloscope (A); switched on, Y1 input selected at 1V/cm, Y2 input selected at 50mV/cm.
- (ii) Amplitude measurement unit (B); Y1 output set on position, Y2 output set on ref., amplitude measurement set on load, maximum and minimum loads set for tension (+), tension(+) cycling.
- (iii) Digital voltmeter (C); selected to read load and to operate in normal mode.
- (iv) Load cell amplifier (D); correctly balanced according to the calibration and zero load readings.
- (v) Load trip unit (E); trips set on "warn".
- (vi) Position servo-amplifier (F); cyclic amplitude set on zero, set point adjusted to give zero error signal, unit activated.
- (vii) Load servo-amplifier (G); cyclic amplitude on zero, loop gain set to the value determined by having the minimum "overshoot" of the load trace when a specimen was subjected to a square loading waveform.
- (viii) Servovalve driver (H); integrator off, dither set on 2, damping set at zero.
- (ix) Interlock unit (I); all interlocks satisfied and unit reset if necessary.
- (x) Pump start unit (J); electronics switched on.
- (xi) Limit detector (K); trips set on "warn".
- (xii) Function generator (L); appropriate waveshape, load function and required frequency selected.
- (xiii) Cycle counter (M); reset to zero, required count rate set, function generator selected.

Before starting the power pack both the specimen and furnace were removed from the loading bars. The hydraulic motor was started by pressing the "pump start" button (panel J) and the Moog valve activated by pressing the "pressure on" button.

The actuator was raised to the top of its stroke using the position servo-amplifier (F) set point control. The specimen and furnace were located in the bottom grip, the load cell re-balanced, and the actuator lowered to allow the top grip to be installed. To ensure axially of loading a small tensile load was applied before rigidly clamping the specimen in position (see fig. 2.3).

At this point fatigue loading of the specimen between known loads was obtained in the following manner:

- (i) The load servo-amplifier (G) error signal was zeroed using the set point control and the load servo-amplifier activated.
- (ii) The voltage equivalents of the loads between which the specimen was to be cycled, $V_{\max}$ and $V_{\min}$, were obtained from tabulated values and levels of $V_{\max}-50\text{mV}$ and $V_{\min}+50\text{mV}$ set on the amplitude measurement unit (B). This effectively restricts the output signal of the amplitude measurement unit to that which is above $(V_{\max}-50)\text{mV}$ and below $(V_{\min}+50)\text{mV}$.
- (iii) Using the cyclic amplitude to vary the amplitude of the control voltage and the set point to adjust the mean of the control voltage, the signal was adjusted until the load trace on the oscilloscope gave a vertical line 2cm in height and symmetrical about the mean. This signal corresponded to cycling between the loads given by $V_{\max}$ and $V_{\min}$. For low frequency testing ($<1\text{Hz}$) the peak reading digital voltmeter was used to set the loads.
- (iv) The valve bias on the servovalve driver and the set point on

the load servo-amplifier were adjusted such that zero error was given on the load servo-amplifier, thus preventing long term variation in the control signal.

2.4.3 Specimen Gripping and Axiality

To facilitate the application of high loads a clamping grip was designed and manufactured in which the specimen was rigidly held. This consisted of split specimen grips which could be securely bolted together. A central locating pin was used to ensure correct positioning of the specimen.

As transfer of excessive heat from the furnace to the actuator or the load cell would have resulted in large fluctuations in the control signal, water cooling of the loading system was incorporated above and below the specimen. The water cooling was observed to have no effect on the temperature distribution of the specimen.

On installation of the actuator, alignment of the loading system was found to be necessary. The axiality of the loading system was tested using a strain-gauged sheet of material identical in dimensions to the centre cracked panels used for the crack growth tests. Four strain gauges, Micro-Measurements specification EA-06-256BG-120, were symmetrically positioned about the centre-line of the specimen and in the mid-plane. The specimen was clamped in an identical arrangement to that used for the crack growth tests. The response of the strain gauges to known changes in load was monitored using a strain gauge bridge (Jones, 1976) and a strain gauge amplifier. Adjustments to the alignment were made by adjusting the position of the large nuts locking the machine top plate to the four posts of the loading frame.

After each adjustment it was necessary to insert and clamp the specimen as before. Adjustments were carried out until the alignment, as indicated by the output of the strain gauges, was within 0.5%.

2.4.4 Temperature Control

In order to heat the specimen, a special furnace to surround the test-piece was designed and manufactured. This consisted of a silica tube 40mm in length with an internal rectangular cross-section of 8mm x 40mm. The silica tube was wound with 1mm diameter Nichrome wire whose separation was set at 0.5mm. Thermal insulation was provided by enclosing the furnace tube in an asbestos box containing alumina powder.

Control of the test temperature was effected using a thyristor phase angle controller which enabled the test temperature to be maintained to better than $\pm 1\text{K}$ over the length of a test. The temperature distribution over the specimen was checked by monitoring the output of thermocouples attached to the specimen. The temperature distribution within the crack plane was observed to be less than 2K of the central temperature, variations within a distance of 5mm on either side of the crack plane were less than 5K of the central temperature. During testing, movement of the furnace was prevented by the use of clamps attached to the machine frame.

2.5 Measurement of Crack Length

A number of different methods exist which enable the length of a crack in a body to be determined, e.g. optical detection, methods which utilise the compliance change of a specimen containing a crack

(Floreen and Kane, 1979), acoustic emission (Westlake, 1969), back face strain (Deans and Richards, 1979) and ultrasonic techniques (Gerberich and Hartbower, 1969). A further technique is that of D.C. potential drop, which utilises the disturbance caused in the potential field around a crack, or other discontinuity, when a current is passed through the specimen. Due to its accuracy and reproducibility this method was used in the present work.

2.5.1 Potential Drop Technique

If a current is passed through a specimen then as the crack extends the resulting decrease in cross sectional area causes an increase in the specimen's resistance. If a constant current is used the change in potential measured between two points on either side of a crack can be used to correlate the crack extension.

The experimental circuit used for monitoring the potential across the crack is shown schematically in fig. 2.4. A direct current of 20 amps. was obtained from a Farnell power supply capable of providing a current stable to 1 part in 10^4 . The current was applied by means of copper plates securely clamped between the specimen and the loading assembly. So that current passed only through the specimen, an insulated loading bar was used, which could be screwed onto the actuator piston rod. Insulation was provided by "Tufnol" inserts which, due to their lower tensile strength, were in regions of compression.

Platinum wire 0.15mm in diameter was used as potential leads. These were spot welded to the specimen using a S.L.E.E. D.C. spot welder operating at 60V and a welding pressure of 7. The potential

leads were attached to the centre line of the specimen, 1.5mm on either side of the crack plane to give a greater sensitivity and were prevented from coming into contact with other areas of the specimen by sheathing with alumina tube (see fig. 2.5). Noise associated with junctions external to the potential measuring circuit was minimised by connecting the platinum wire directly to the measuring circuit.

The potential differences developed across the specimen were monitored by a digital voltmeter capable of measuring microvolts and a chart recorder having a maximum sensitivity of 0.5mV full scale deflection. To allow the most sensitive range of the chart recorder to be used a voltage back-off supply was included in the circuit.

2.5.2 Potential Drop Calibration

Due to the difficulties of applying the available theoretical calibrations, for example those of Johnson (1965) and Gilbey and Pearson (1966), relating crack length and the potential developed across the crack, an experimental calibration was used in the present study.

After attaching the current and potential leads to the specimen, the potential drop across the uncracked specimen, V_0 , was measured. A central notch was introduced into the specimen and extended in small symmetrical increments, as previously described. The potential characteristic of the flaw in the specimen geometry used, V_a , and the length of the flaw, $2a$, were measured after each increment. Although the thickness of the spark machined notch is greater than that of a crack, McIntyre and Priest (1971) have shown that this leads to small errors in the measured crack length.

Calibrations were determined at each of the test temperatures used. At elevated temperature an e.m.f. due to the thermocouple action between the specimen and the potential probe may be generated. If the junction characteristics of both potential leads are identical, the net thermal e.m.f. across the leads will be zero. However, in practice identical junction characteristics are difficult to obtain and therefore the contribution of the thermal e.m.f. to the output potential should be accounted for. Therefore, at elevated temperature, values of both the potential across the flaw, V_T , and the thermal e.m.f., V_{th} , were determined. The potential characteristic of the flaw length under consideration, V_a , is then given by

$$V_a = V_T - V_{th} \quad (2.2)$$

The initial potential drop across the specimen was about 4mV and the magnitude of the thermal e.m.f. generated was typically 100 μ V. At small flaw lengths this may be a significant proportion of the potential V_a , however measurements indicated that the thermal e.m.f.'s were stable to better than $\pm 0.1\%$ of the initial potential drop over periods exceeding one week.

From the experimentally determined values of V_a and a/W a computer program was used to determine the relationship between V_a and a/W . The computer program is listed at the back of this work. The relationship between V_a and a/W was used to determine the value of the crack length during crack growth for the specific value of the potential drop observed across the crack.

2.6 Crack Growth

Crack growth rates were investigated at different temperatures and frequencies over a range of cyclic stress intensities.

2.6.1 Fatigue Crack Propagation

Fatigue crack propagation tests were performed in C.C.T. specimens machined in accordance with the A.S.T.M. specification E647-78T (1978). Determination of the relationship between the crack growth rate and the cyclic stress intensity factor, ΔK , is most commonly achieved by either constant cyclic load amplitude testing or constant ΔK loading of a growing crack.

The former technique requires complex data processing to provide the crack growth rate at a given ΔK since a particular ΔK is only applied over a short crack length. Wei et al. (1979) have shown that considerable variation in fatigue crack growth rates can arise with this method due to the choice of the interval of crack length measurement and the associated data processing procedure. Similarly, Edwards (1981) observed a large degree of scatter with constant load amplitude testing which obscured the differences in the fatigue crack propagation rates of some aluminium alloys. To overcome these difficulties crack growth is often determined under conditions of constant applied cyclic stress intensity.

The application of a constant ΔK is normally achieved in practice by either appropriate specimen design or by decreasing the applied load as the crack extends. The latter method was used in this work.

A constant ΔK was maintained by manually decreasing the applied

load. The value of the applied load for a specific crack length was obtained from tabulated values calculated for the specific specimen geometry used here. The small load reductions needed to maintain a constant ΔK were made at 0.1mm intervals.

Crack growth rates at different levels of applied ΔK were obtained by making larger changes in the applied load until the new ΔK was obtained. Typical changes in load were 5-10% of the previously applied loads. Crack growth was continued until a steady state growth rate typical of the new ΔK level was obtained.

The effect of R ratio ($R=K_{\min}/K_{\max}$) on the crack growth rate was investigated by independently varying R; within any one test the R ratio was maintained at a constant value of 0.1 or 0.25.

2.6.2 Measurement of Fatigue Crack Growth Rates

A fatigue pre-crack was initiated in a centrally notched specimen at room temperature by applying a ΔK of 19.8MPa/m at an R ratio of 0.1, under a sinusoidal loading waveform with a frequency of 10Hz. The pre-crack was allowed to extend by approximately 1mm at which point it was considered to have grown out of the notch tip stress field and growth rates were considered to be valid. Ambient temperature crack growth rates were obtained using a triangular loading waveform under conditions of constant ΔK and R ratio loading. At any given level of ΔK , crack growth was measured over a length of approximately 1mm with at least 10 crack length measurements made to determine the crack growth rate.

After initiation the ΔK was systematically reduced to enable low growth rates, $<10^{-6}$ mm/cycle, to be recorded. The applied ΔK was then

increased and higher growth rates recorded. At the highest crack growth rates constant ΔK could not be maintained and growth rates were recorded under constant applied load amplitude.

High temperature crack growth rates were determined in a similar manner. After initiation of the fatigue pre-crack the specimen was heated to the required test temperature and allowed to equilibrate for 12 hours before growth rates were measured. A small tensile load was applied to the specimen during heating. Its value was selected such that the value of the stress intensity at the crack tip was below the lowest applied stress intensity at room temperature. At high temperatures a range of frequencies was used to determine the effect of the frequency of loading on the crack growth rate.

2.6.3 Creep Crack Growth

Creep crack growth rates were measured under constant applied load using Mand creep machines fitted with an automatic beam levelling facility.

The specimens used for creep crack growth were similar to those used for fatigue crack growth, except that 10mm pin holes were used and an 11mm starter notch introduced. A sharp crack tip was formed by pre-cracking in air at room temperature at a ΔK of 15MPa/m with $R=0.1$, until the total crack length was 12mm.

The load was applied to the specimen by means of a cantilever beam with a 10:1 ratio, the furnace used has been described and the crack length was measured using the potential drop technique. The values of the potential drop across the crack were recorded at preset time intervals using a Normalair Garrett datalogger. The maximum

sensitivity of the datalogger was 10 μ V with preset data acquisition at time intervals between 1 minute and 1 hour. A continuous record of the output was made using a chart recorder having a maximum sensitivity of 0.5mV.

2.7 Data Analysis

The data obtained from the crack under constant ΔK conditions gave the length of the crack, a , and the corresponding number of loading cycles, N . The crack growth rate was obtained by the use of a linear regression analysis based on the values of a and N .

Crack growth under constant applied load amplitude (high fatigue crack growth rates) and constant applied load (creep crack growth) was analysed by the use of a computer program included in the back of this work. The growth rate is given by calculating the slope of the straight line between adjacent points on the a against N or a against t curve. This may be expressed as:

$$\frac{da}{dN} = \frac{a_{i+1} - a_i}{N_{i+1} - N_i} \quad (2.3)$$

$$\frac{da}{dt} = \frac{a_{i+1} - a_i}{t_{i+1} - t_i} \quad (2.4)$$

Since the computed growth rate is an average rate over the increment $(a_{i+1} - a_i)$ of crack extension, the average crack length was used to calculate the appropriate stress intensity.

2.8 Plastic Zone Size Measurement

The accurate determination of plastic zone sizes is generally limited by two parameters; first the minimum detectable level of strain associated with the technique, and secondly the area of material with which this strain level is associated. Numerous techniques exist for determining the plastic zone size, see for example Lankford et al. (1977) and Blind (1982). Many, such as brittle lacquer and strain gauges (Kwum and Fine, 1980), may only demonstrate the existence of plastic deformation at a free surface. The plane strain plastic zone has been investigated by techniques such as etching (Hahn et al., 1972), microhardness (Bathias and Pelloux, 1973) and recrystallization of the plastic zone (Iino, 1976). Although successful, these methods are generally limited to specific materials or are of limited sensitivity. For example, microhardness indentations may be large relative to the plastic zone size, and often only detect relatively high levels of strain.

Stickler et al. (1971) have demonstrated that plastic deformation may affect channelling patterns. The patterns, obtained with a scanning electron microscope, may be obtained for small volumes of material, with typical surface diameters of approximately 15 microns. In addition, Lankford et al. (1977) report the detection of levels of strain of approximately 0.4%. Thus, due to the greater sensitivity, the present work used a channelling pattern technique to investigate the crack tip plastic zone.

2.8.1 Channelling Patterns

Channelling patterns can be used to give crystallographic information about a specimen and are obtained by adjusting the normal operating conditions of a scanning electron microscope. The patterns were first observed by Coates (1967) and arise because the electron intensity backscattered from the crystal is a function of the angle of incidence of the electron beam with respect to the crystal lattice. This effect has been qualitatively explained in terms of anomalous absorption by Booker et al. (1967) and quantitative theories have been given by several authors (Hirsch and Humphreys, 1970; Reimer et al. 1971).

Contrast can thus be produced by changing the relative orientation of the incident beam and the crystal lattice giving rise to a channelling pattern consisting of a geometrical array of bands and lines determined by the crystallography of the specimen (see fig. 2.6). Electron channelling patterns (E.C.P.'s) may be obtained from large areas of a single crystal or, by suitable adjustment of the operating conditions of the microscope, patterns may be generated from small selected areas of a specimen, selected area channelling patterns (S.A.C.P.'s), thus allowing polycrystalline material to be used.

In practice, the channelling pattern contrast is obtained by varying the angle at which the incident electron beam strikes the specimen surface. Various methods have been devised to enable rocking of the electron beam over small areas of the specimen (e.g. Coates, 1967; van Essen and Schulson, 1969; van Essen, Schulson and Donaghay, 1970) each involving changes to the normal operating conditions of both the lens currents and scan coils. The method used in the present

work is illustrated in fig. 2.7.

The deflection coil used during the normal scanning operation (scanning coil in fig. 2.7) is deactivated and deflection of the beam is effected at point A in fig. 2.7 by a coil positioned just below the second condenser lens (rocking coil in fig. 2.7). The current in the second condenser lens is adjusted so that the electron beam is focussed on the front focal plane of the objective lens (B in fig. 2.7). The objective lens focal length is set so that the image of the object located at the rocking coil is focussed on the specimen. Hence, variations in the rocking coil current give rise to rocking of a parallel electron beam about a given point on the specimen surface.

Due to the restrictions imposed upon the size of the final electron beam the point about which the beam is rocked has a finite size. The factors governing the minimum area from which a pattern may be obtained have been discussed by Joy and Booker (1972) and more recently by Schulson (1977) and are strongly determined by the inherent spherical aberration of the objective lens (Newbury, 1972). This phenomenon causes rays that are at different distances from the optic axis to focus at different points along the axis creating a disc of least confusion on the specimen surface of radius Δr (see fig. 2.7) which can be given (Thornton, 1968, pg.36) by

$$\Delta r = c_s \beta^3 \quad (2.5)$$

where c_s is the spherical aberration coefficient of the lens and β is the angle that a ray makes with the optic axis. The size of the spot on the specimen surface is thus a function of the angle of rock and of the distance between the specimen and the objective lens.

Since the electron beam penetrates the specimen surface the

interactions which give rise to contrast actually occur within a volume of material termed the interaction volume. The size of this interaction volume depends on the spot size, the accelerating voltage of the electron beam and the atomic number of the material. Although precise values for the depth below the specimen surface from which channelling pattern contrast arises are not known, Hirsch and Humphreys (1969) have calculated that it is of the order of 1000Å for an accelerating voltage of 20kV.

In addition to providing crystallographic information such as the orientation and lattice parameter of a specimen, the characteristics of channelling patterns can be used to provide information concerning the average number of defects within the volume of material sampled by the beam. The deterioration of channelling patterns can be shown to depend on the dislocation density, thus enabling crack tip plastic zones to be investigated.

2.8.2 Effect of Deformation on Channelling Patterns

The effect of deformation on channelling pattern contrast has been investigated by several authors (e.g. Stickler et al., 1971 and Spencer et al., 1974). Stickler et al. have investigated the effect of tensile strain on S.A.C.P.'s obtained from several polycrystalline materials and have correlated the width of individual lines with both the percentage tensile strain and the experimentally determined dislocation density; such a determination relies on a particular orientation being present in the polycrystalline material. However, in general as the specimen is deformed, the contrast of a channelling pattern is observed to decrease and the width of individual lines are

observed to increase so that the higher order lines progressively disappear. A more quantitative treatment of the channelling pattern degradation with dislocation density has been given by Spencer et al. (1974). This allows the determination of a calibration curve of pattern resolution against dislocation density which is applicable to most elements. However, such a calibration is specific to certain electron-optical parameters such as accelerating voltage, spot size and beam divergence.

Despite the difficulties in quantifying the change in the quality of a channelling pattern with deformation, S.A.C.P.'s have been successfully used to assess the deformation in bulk specimens. Stickler et al. (1971) have demonstrated that the strain adjacent to both hardness indentations and creep cracks can be detected. The technique has also been used to study the plastic zone at the tip of fatigue cracks in polycrystalline materials (e.g. Lankford et al., 1977). By experimentally calibrating the S.A.C.P. quality with strain in tapered tensile specimens, Davidson and Lankford (1976) have determined the size of the crack tip plastic zone and have also reported the use of S.A.C.P.'s for the measurement of strain contours within the plastic zone. Their determination was, however, subjective since it relied on judgements of the channelling pattern quality to determine the strain.

More recent investigations (Crompton and Martin, 1980) have used single crystal material to relate the deterioration in channelling pattern quality to the bulk deformation. The use of crystals with a constant orientation enabled measurement of the angular resolution of particular channelling pattern lines as a function of strain. The results show that over the range of strain investigated the resolution

of a particular channelling pattern line can be linearly related to the strain. Although the range of strain studied was not as large as that of Stickler et al. (1971) the higher electron accelerating voltage used, 100kV as opposed to 20kV, the greater stability of the system and the decreased effects of any inhomogeneous deformation due to the influence of grain size resulted in a significant decrease in the scatter of the results.

2.8.3 Experimental Determination of Strain Effects

In order to determine the effect of strain on the S.A.C.P.'s used in the present work a series of specimens deformed in compression were examined.

Bars with dimensions approximately 4mm x 4mm x 100mm were spark eroded from the as received single crystal blocks such that their length was parallel to the crystal growth direction. The bars were then spark machine lathed and finally centreless ground to allow cylindrical compression specimens 2.3mm in diameter and 4.6mm in height to be prepared. By subjecting specimens to uniaxial compression several samples of known strain were obtained, friction effects due to contact between the specimen and the compression plates were minimised by lubricating the two surfaces.

To eliminate errors in interpretation due to "dead zone" effects obtained in compression specimens both transverse sections taken from the mid height, and longitudinal {100} sections were examined.

All channelling pattern work was carried out on a J.E.O.L. 100C electron microscope with an EM-ASID-40 scanning attachment operating at 100kV. Using a small ($<10\mu\text{m}$) carbide particle, a collimated beam in

rocking mode was obtained by adjusting the objective lens current so that the carbide particle exhibited maximum magnification. By moving away from the carbide particle a channelling pattern characteristic of the deformed material was obtained. The signal was collected by a solid state backscattered electron detector operating in a differentiated mode with a time constant of 0.25ms. To optimise the channelling pattern quality small adjustments to the condenser lens current were made to obtain the best line resolution.

Patterns characteristic of the deformed and undeformed material were recorded for rocking angles of both $\pm 10^\circ$ and $\pm 5^\circ$. Increasing the rocking angle increases both the extent of the pattern that is visible and the spot size of the beam. Using a copper mesh having $10\mu\text{m} \times 10\mu\text{m}$ grid openings the optimum channelling pattern conditions for a rocking angle of $\pm 10^\circ$ revealed that one grid opening filled the screen. Hence for a rocking angle of $\pm 10^\circ$ S.A.C.P.'s were generated from a volume with a surface area of approximately $10\mu\text{m}$ diameter. For a rocking angle of $\pm 5^\circ$ the spot size was much smaller than the grid spacings. Since the specimen is positioned close to the objective lens very small spherical aberration coefficients are obtained. Hence for a rocking angle of $\pm 5^\circ$ the spot size may be estimated at 2-3 μm diameter.

Visual examination of the S.A.C.P.'s from the deformed material revealed that strains of approximately 1% could be detected by observing the loss of the higher, typically fourth, order lines. In view of the accuracy and relative ease of this method for detecting strain the loss of higher order lines was used to determine the plastic zone boundary.

The effect of deformation was quantified using a travelling microscope to measure the increase in width of specific lower order

lines with increasing strain. Several measurements of the width of {200} and {220} lines were taken from the photographic negative at varying positions along their length for rocking angles of both $\pm 10^\circ$ and $\pm 5^\circ$. A high degree of reproducibility was found, allowing the level of strain in deformed specimens selected at random to be accurately determined. On the basis of these results, it was determined that measurements of line widths were capable of determining levels of strain to an accuracy of better than $\pm 0.25\%$.

2.8.4 Plastic Zone Effects

To allow the crack tip plastic zone to be studied several crack growth tests were interrupted prior to failure at known values of ΔK . The specimen was rapidly removed from the testing rig so that any relaxation effects not characteristic of the propagating crack were minimised. The crack tip region and surrounding material were cut from the specimen by spark erosion and sectioned to reveal the longitudinal mid plane (fig. 2.8). The mid plane containing the crack tip and plastic zone was mechanically ground and polished and finally electropolished (section 2.9) to allow good channelling pattern quality to be obtained. Throughout the preparation care was taken to preclude additional loading of the crack tip.

The extent of the crack tip plastic zone was determined using S.A.C.P.'s. The digital volt meters (D.V.M.'s) attached to the specimen translation system were used to set up a co-ordinate system the origin of which was taken as the crack tip. The D.V.M.'s had previously been calibrated and found to be accurate to the nearest micron, backlash in the system was minimised by making all readings

after translation in a constant direction. Using this technique it was determined that specific locations on the specimen could be determined to an accuracy of ± 2 microns.

The plastic zone boundary was determined in the following manner. A good quality channelling pattern was obtained at a distance of several millimetres ahead of the crack tip. With the system operating with optimum channelling pattern conditions the crack tip was located by noting the agreement between the previously recorded D.V.M. readings of the crack tip position and the distorted image on the screen. This position was taken as the origin of a co-ordinate system given by the D.V.M. readings (see fig. 2.9). With the y co-ordinate remaining fixed the x co-ordinate of the specimen translation system was adjusted until a S.A.C.P. characteristic of undeformed material was obtained. The specimen translation x co-ordinate was adjusted so that S.A.C.P.'s from material nearer to the crack tip were obtained. Once deformation was detected, by the loss of the higher order lines on the constant orientation channelling pattern, the position, (x,y) in fig. 2.9, was noted. An initial estimate of the x co-ordinate was made using S.A.C.P.'s for a rocking angle of $\pm 10^\circ$; a $\pm 5^\circ$ rocking angle was then used to gain a more accurate value of the boundary. By varying the y co-ordinate and determining the "new" x co-ordinate corresponding to the plastic zone boundary in the same manner, a map of the plastic zone boundary was obtained. For each point (x,y) several estimates were made and the average value of x taken, this gave an accuracy of better than ± 5 microns for each point.

The qualitative method of determining strain used to find the plastic zone boundary, allows values of $\approx 1\%$ strain to be detected so that the boundaries located are strictly 1% strain contours of the

plastic zone. In addition, a quantitative measure of the strain distribution within the plastic zone was obtained. The y co-ordinate was adjusted to correspond to the value of the crack tip, photographic recordings of the S.A.C.P.'s for angles of rock of both $\pm 10^\circ$ and $\pm 5^\circ$ were taken at different distances ahead of the crack tip, as given by the x co-ordinate. Comparison of the measured line width of lower order lines with the calibration of line width against strain, allowed the strain at selected points ahead of the crack tip to be determined.

2.9 Metallography

Specimens for optical metallography and the determination of plastic zone sizes were prepared by initial grinding on wet silicon carbide paper to 800 grade. Mechanical polishing was effected using cotton drill impregnated with $3\mu\text{m}$ or $1\mu\text{m}$ diamond paste. Between each operation of the grinding and mechanical polishing processes the specimen was cleansed by ultrasonic agitation in ethanol.

Specimens for optical metallography were electro-etched with a phosphoric acid solution by passing a platinum wire cathode over the specimen surface. The conditions for electro-etching are given in Table 2.3. Specimens were electropolished for the determination of plastic zone sizes with a Butoxyethanol - Perchloric acid - Acetic acid solution; the conditions are given in Table 2.4.

2.10 Fractography

Fracture surfaces were examined with a J.S.M. 35X scanning electron microscope and a J.E.O.L. 100C scanning-transmission electron microscope operating in secondary electron image mode.

The fracture surfaces were removed from the undeformed portion of the specimen using a Buehler low speed diamond saw. To prevent contamination of the fracture surface during cutting, the fracture surface was coated with collodion solution. This acted as a barrier to any contaminant, and yet could be easily stripped off the fracture surface without altering surface features.

Table 2.1

Compostion of Single Crystal Mar M 002 (wt%)
Co 10; Cr 8.8; Al 5.5; Ti 1.4; W 9.8; Ta 2.5;
Hf 1.5; C 0.015; B 0.012; Zr 0.07; Ni balance.

Table 2.2

Conditions for Macro-Etch

10% HCL in Methanol

Large Stainless Steel Cathode

Room Temp., 4V, 1min - 30secs.

Table 2.3

Conditions for Electro-Etch

10% Phosphoric Acid in water.

Room Temp., 3V, 5 - 10 seconds.

Table 2.4

Conditions for Electropolishing

45% Butoxyethanol, 45% Acetic Acid, 10% Perchloric Acid

Large Stainless Steel Cathode

-20°C

Current Density 1Acm^{-2} , 1min.

Figure 2.1

Schematic representation of the C.C.T. specimen
and its dimensions in mm.

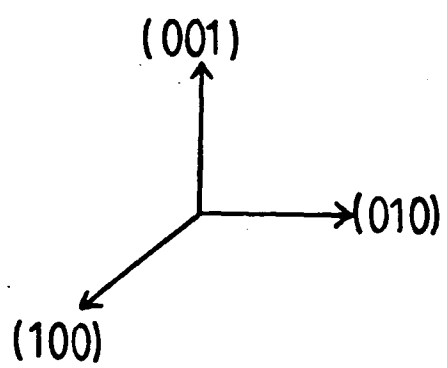
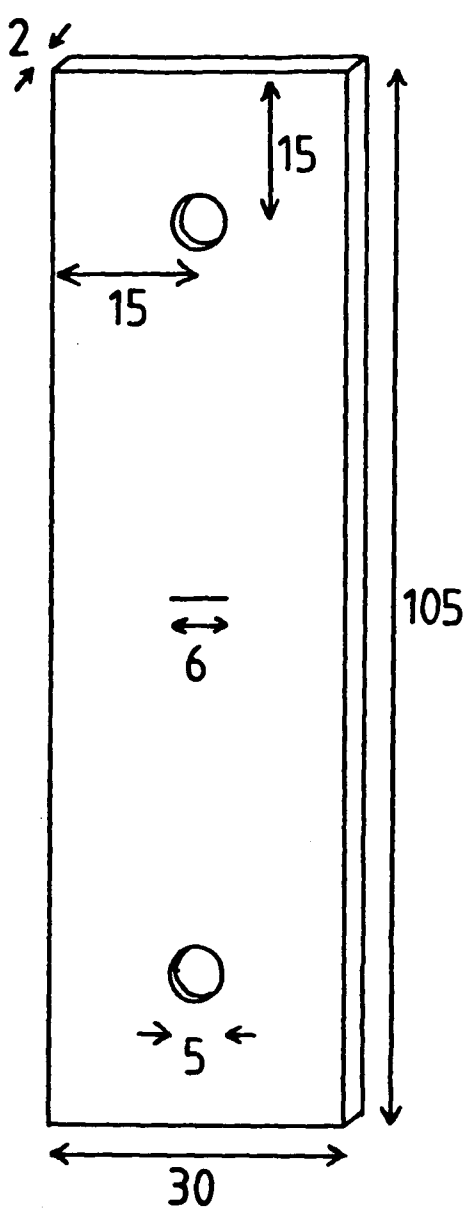


Figure 2.2

Electronics panel of the fatigue machine.

A: Oscilloscope.

B: Amplitude measurement unit.

C: Digital voltmeter.

D: Load cell amplifier.

E: Load trip unit.

F: Position servo-amplifier.

G: Load servo-amplifier.

H: Servovalve driver.

I: Interlock unit.

J: Pump start unit.

K: Limit detector.

L: Function generator.

M: Cycle counter.

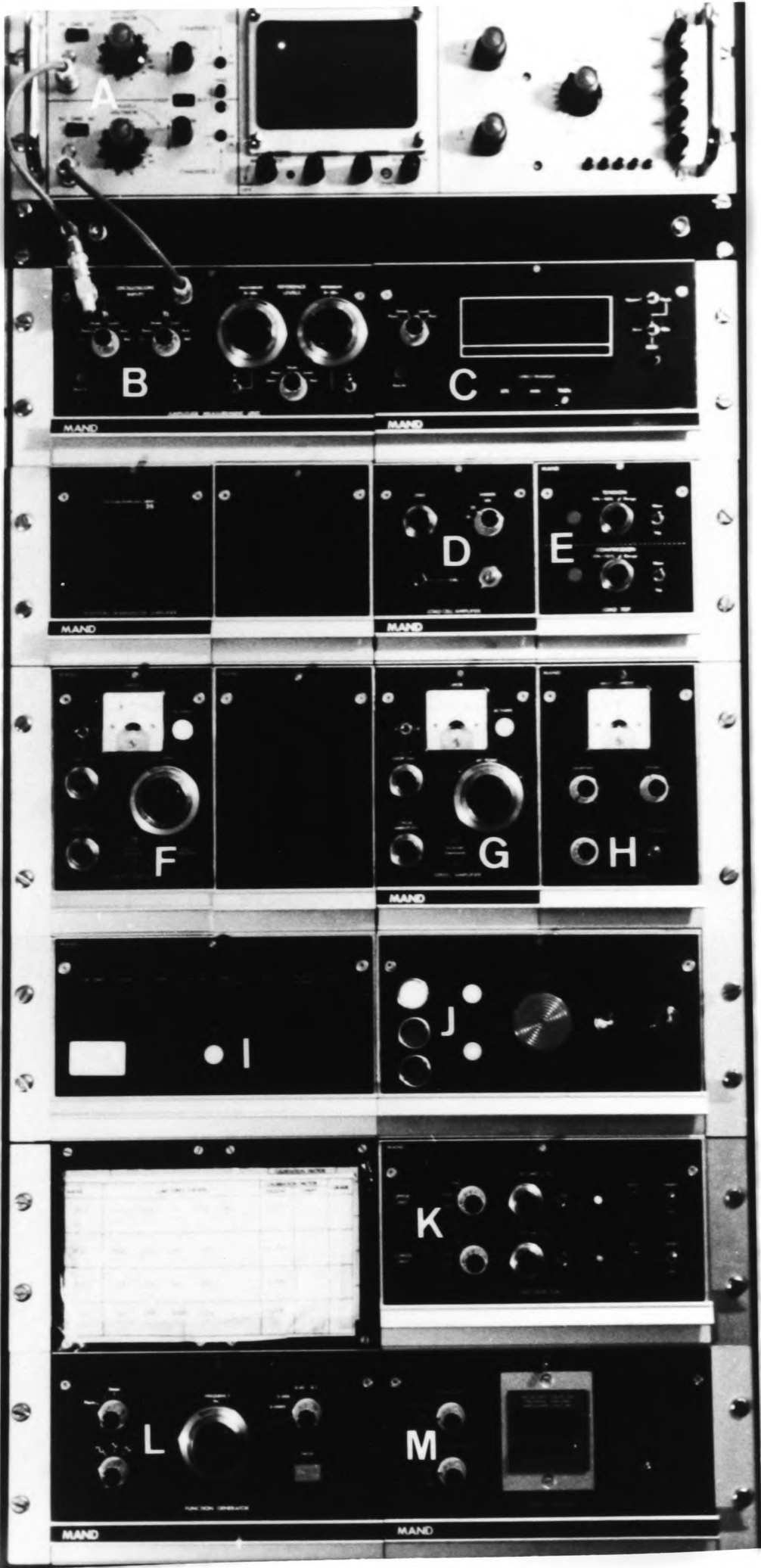


Figure 2.3

Experimental loading assembly and furnace.

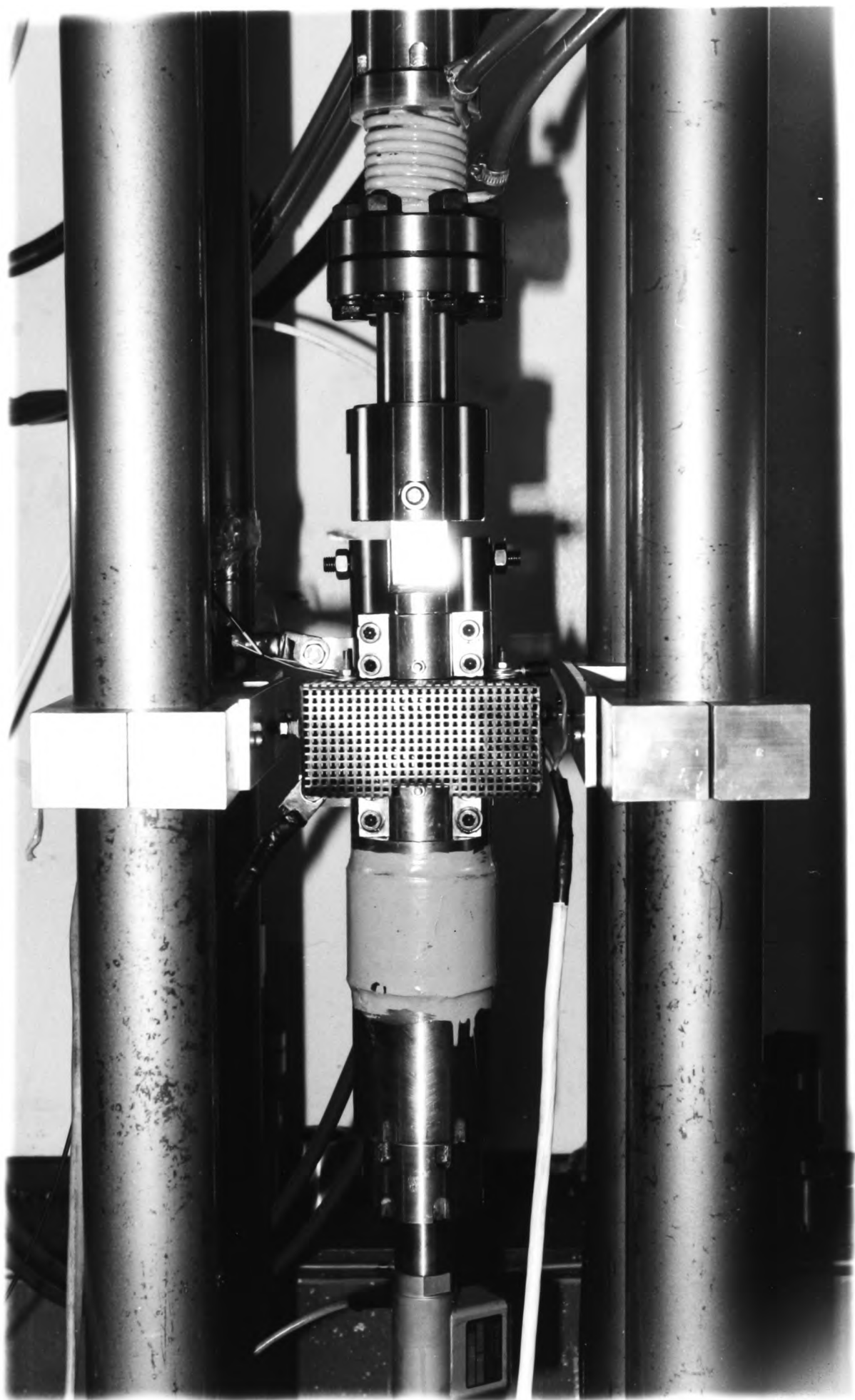


Figure 2.4

Schematic representation of the crack length
measurement apparatus.

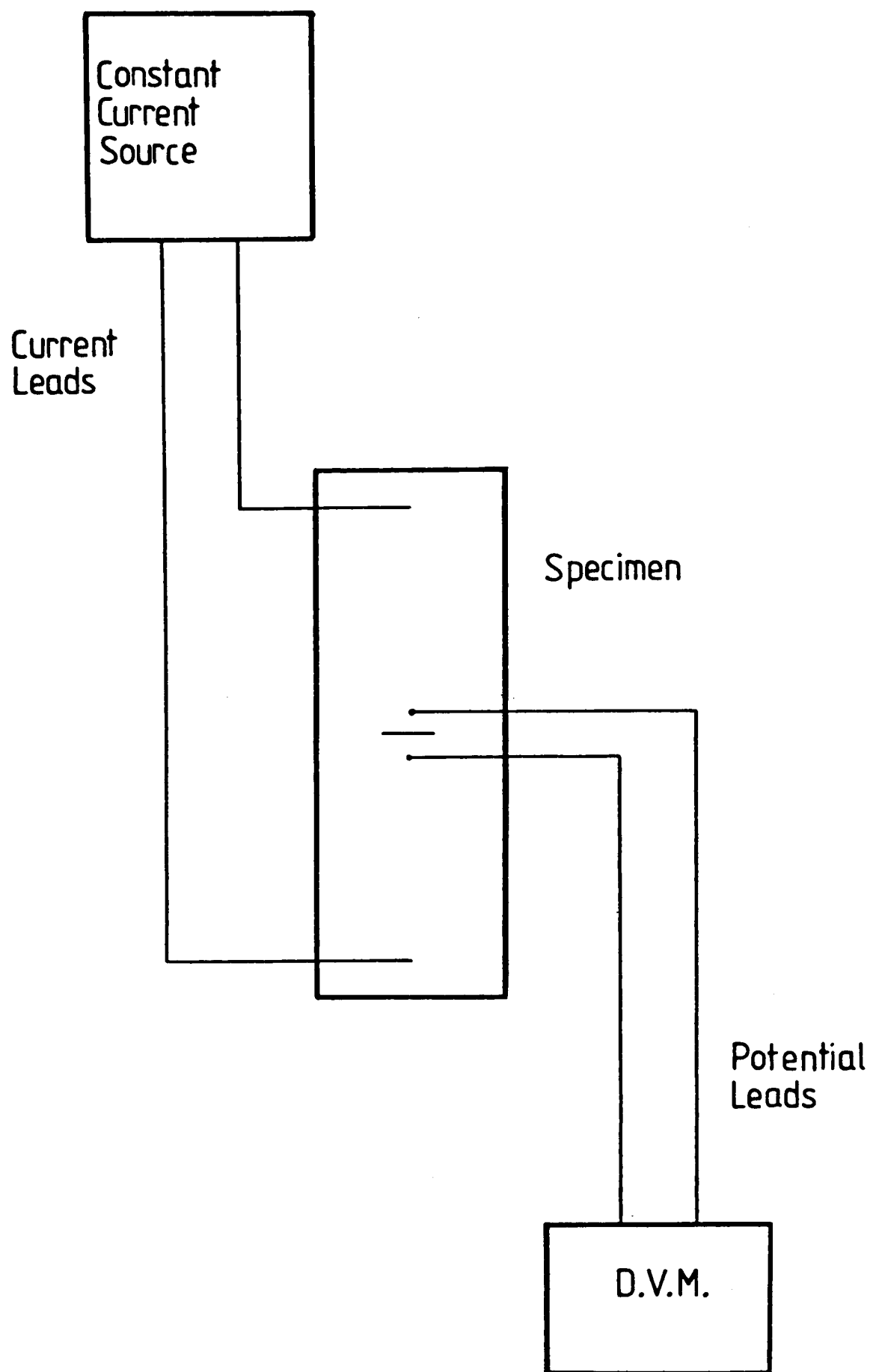


Figure 2.5

Specimen with potential probes attached.

Figure 2.6

{100} selected area channelling pattern from
single crystal Mar M 002.

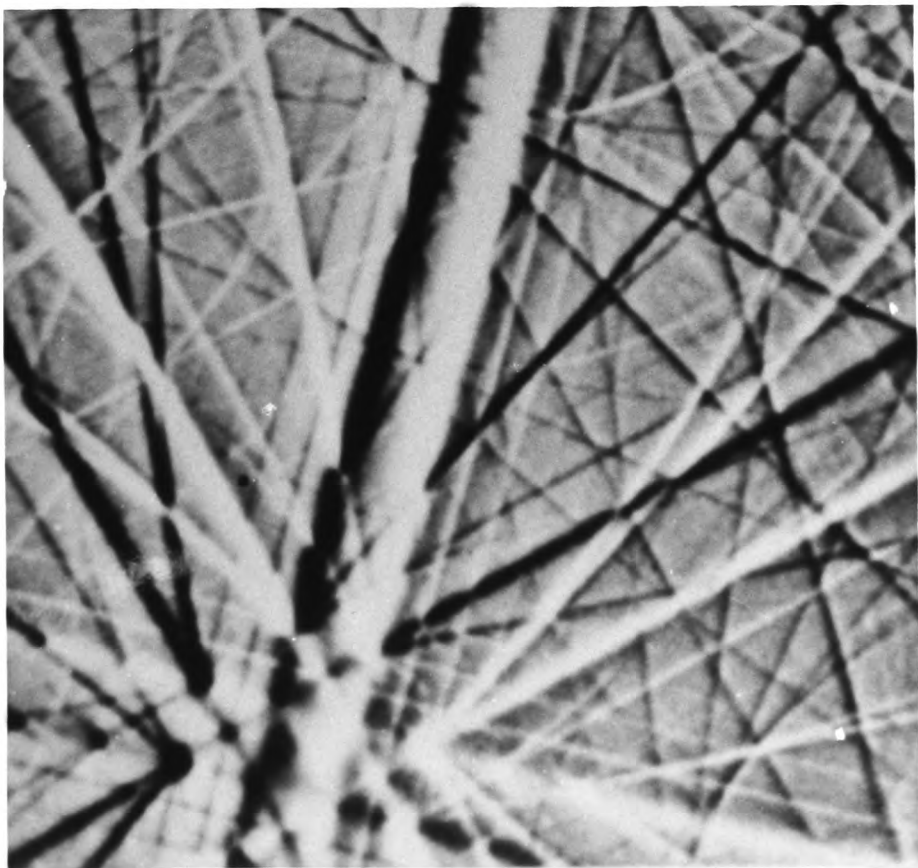
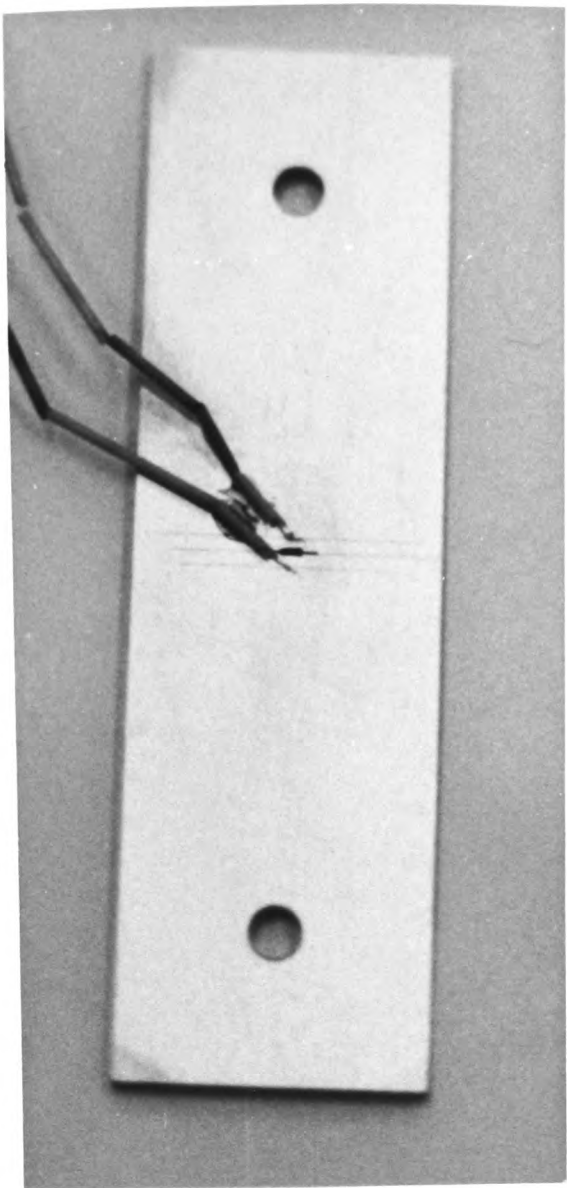


Figure 2.7

Schematic representation of the ray diagram for the production of selected area channelling patterns.

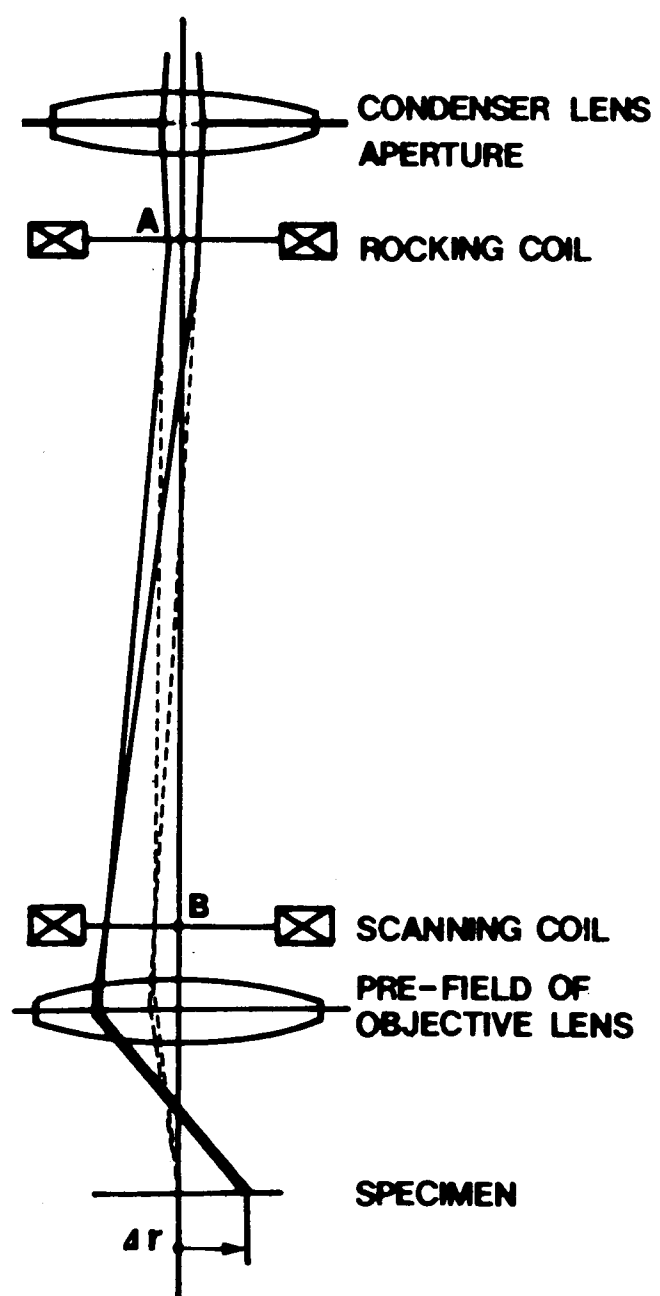


Figure 2.8

Method of sectioning to reveal a mid-thickness longitudinal section through the crack, spark machining carried out along the dashed line.

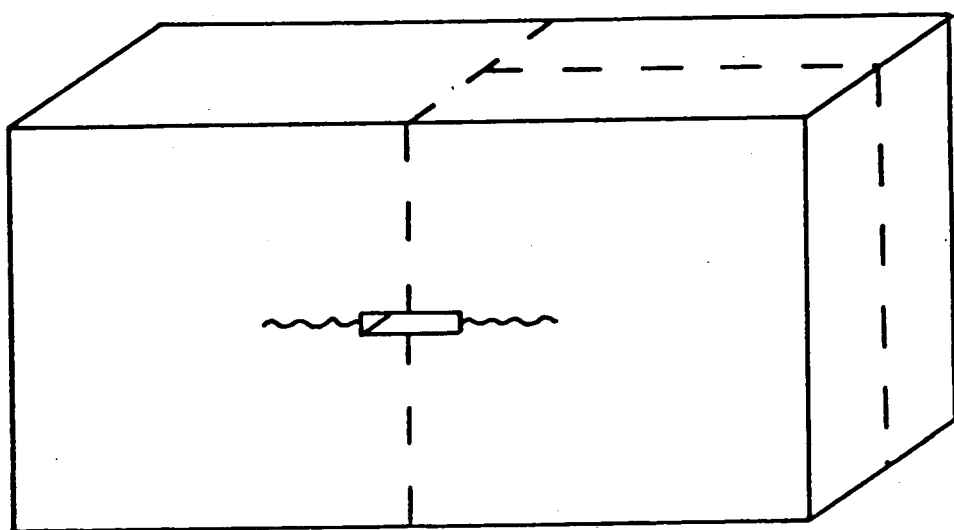
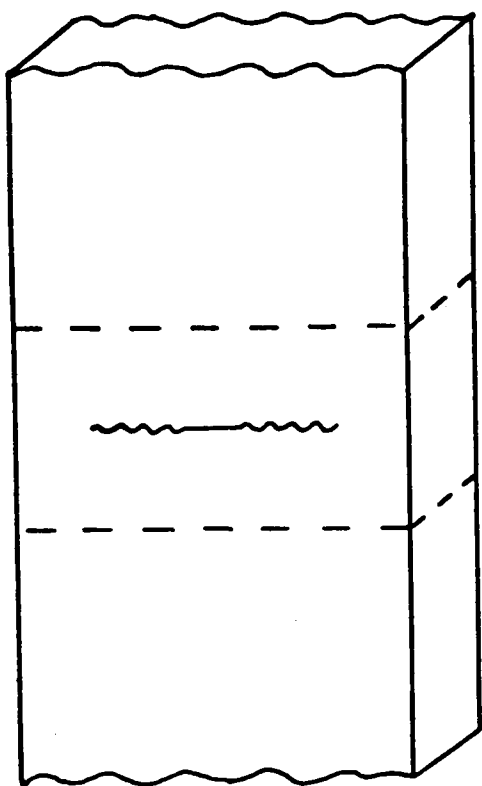
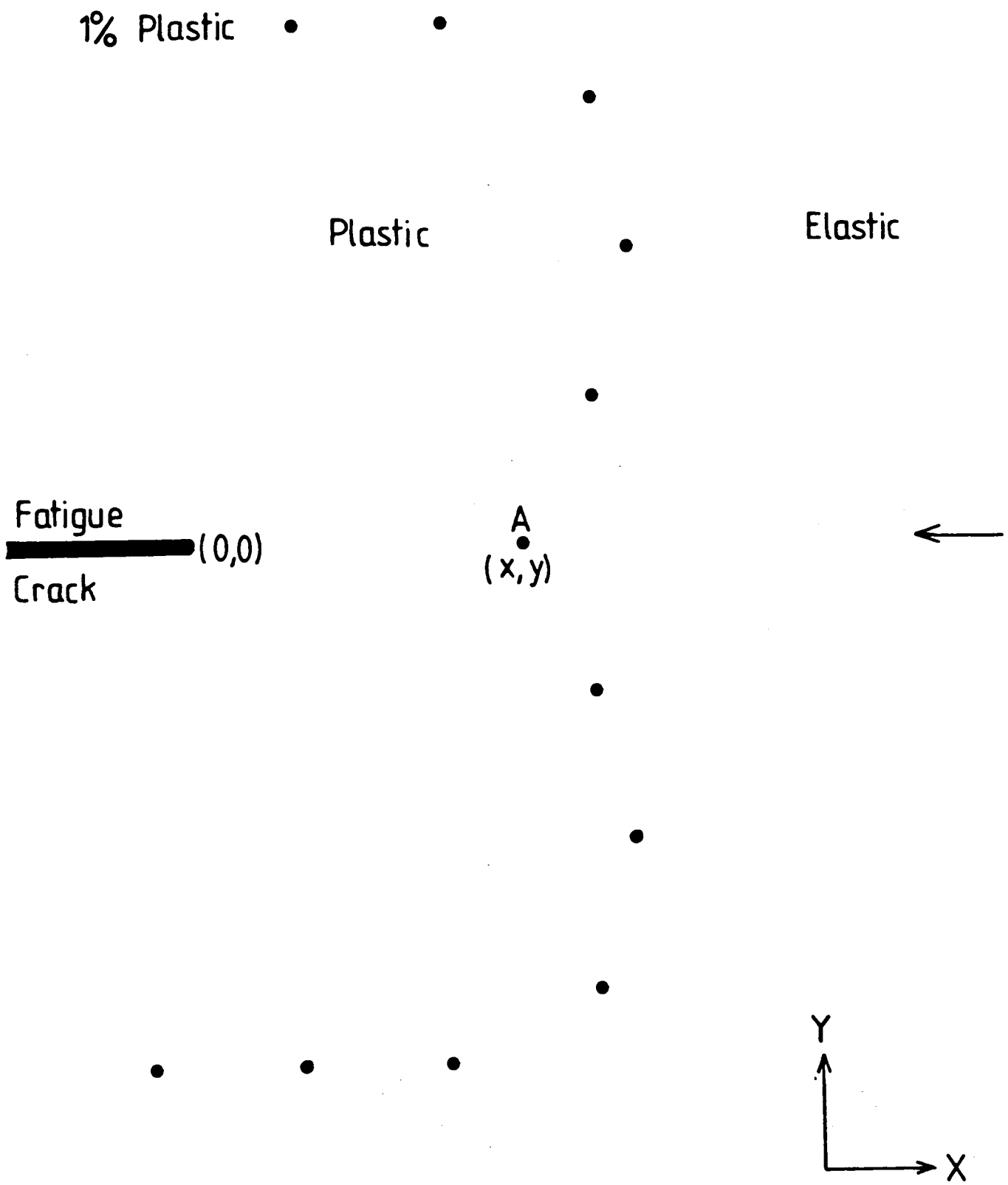


Figure 2.9

Co-ordinate system for measurement of the crack
tip plastic zone.



Chapter 3

Results

3.1 Material

The microstructure of Mar M 002 is typically found to contain a large volume fraction of cuboidal γ' precipitates of approximately $0.5\mu\text{m}$ edge dimension together with some fine spherical γ' precipitates of approximately 150A radius (fig. 3.1). The cube edges are aligned on {100} and the γ' precipitates are coherent with the matrix and although compositionally complex possess an ordered L1₂ structure of the intermetallic phases Ni_3Al or Ni_3Ti (Decker, 1969).

Colonies of γ/γ' eutectic and MC script carbides, where M is either titanium, tantalum, tungsten or hafnium, are usually found in addition to the γ' precipitate (fig. 3.2). Generally the carbides form in the melt and are interdendritically located. The material has a temperature dependent yield stress and the tensile data is given in Table 3.1.

3.2 Potential Drop Calibration

Calibrations for the crack length measurement were obtained in terms of the potential across the crack, V_a , and the ratio of the total crack length to specimen width, $2a/W$. The results for the C.C.T. specimens at room temperature, 600°C and 850°C are given in figs. 3.3 to 3.5 respectively.

With increasing temperature it may be seen that the sensitivity of the technique varies giving a maximum sensitivity of approximately $600\mu\text{V}/\text{mm}$. The polynomial equations developed to describe the variation of V_a with $2a/W$ (section 2.5.2) are given in Table 3.2. In practice several equations of different degree were generated for each set of data, the equations used here were those which gave the best statistical fit to the data.

3.3 Crack Propagation

Crack propagation rates were determined for a range of temperatures and loading rates as described in section 2.6.1. The majority of the tests were conducted on specimens oriented for crack propagation along $\langle 100 \rangle$. Some tests were also conducted in which cracks propagated along $\langle 110 \rangle$ and $\langle 210 \rangle$. In these cases the crack propagation rates lay within the observed experimental values of the $\langle 100 \rangle$ specimens and are included in those results. The results presented refer to symmetrical crack extension as determined by a comparison of fracture surface features such as heat tints and by the measurement of crack lengths on interrupted tests.

The determination of crack growth rates by loading at constant values of ΔK yielded little scatter. A typical experimental plot of the crack length against the number of cycles is given in fig. 3.6. At room temperature and 600°C high correlation coefficients were obtained, generally better than 0.99, whilst at 850°C slightly more scatter was observed with correlation coefficients of better than 0.98 obtained.

3.3.1 Room Temperature

The variation of the fatigue crack propagation rate with ΔK at ambient temperature is shown in fig. 3.7. It may be seen that over the range of growth rates investigated, the influence of the stress ratio, R , is limited to high growth rates ($> 10^{-4}$ mm/cycle). At low growth rates R is observed to have little effect and no significant change in the slope of the da/dN against ΔK curve is observed at the low ΔK 's.

3.3.2 600°C

For a stress ratio of 0.1, the fatigue crack growth rates at 600°C and frequencies of 10Hz, 1Hz and 0.1Hz are presented in fig. 3.8; in addition, a growth rate was determined at $\Delta K=23.4$ MPa/m and a frequency of 0.01Hz this will be referred to later.

It is apparent from fig. 3.8. that the response of the material is dependent on the frequency of loading. At high stress intensities (> 26 MPa/m) the highest growth rates are associated with the highest frequency giving rise to a frequency dependent value of the critical stress intensity K_c .

At low ΔK 's (< 16 MPa/m) a similar frequency dependence is observed with higher growth rates at higher frequencies. In addition, the 10Hz data show a change in the slope of the da/dN against ΔK plot below growth rates of approximately 10^{-5} mm/cycle and the 1Hz data show increasing departures from the 10Hz data towards lower crack growth rates.

At intermediate levels of ΔK , the 10Hz and 1Hz crack growth rates are similar but a slight increase in growth rate is observed as the

frequency is lowered from 1Hz to 0.1Hz. Similar behaviour is observed with $R=0.25$ (fig. 3.9). Again, at both low and high values of ΔK higher crack growth rates are associated with the 10Hz frequency. At intermediate values of ΔK higher crack growth rates are associated with a frequency of 0.1Hz.

3.3.3 850°C

The fatigue crack growth rates for frequencies of 1Hz and 0.1Hz at stress ratios of 0.1 and 0.25 are presented in figs. 3.10 and 3.11 respectively. The general behaviour is similar to that reported at 600°C. At low levels of ΔK significant changes in the slope of the da/dN against ΔK curves may be seen indicating a transition to threshold type behaviour. In the intermediate range of ΔK , the 0.1Hz results again show growth rates above those at 1Hz.

The variation in the creep crack growth rate with stress intensity factor is given in fig. 3.12 together with the data from the fatigue tests at 1Hz plotted on a da/dt basis. Under static loading it may be seen that small increases in stress intensity factor gave rise to much larger increases in the growth rates than is observed under cyclic loading. The growth rates are, however, lower than under cyclic loading. At stress intensities approaching K_c the crack growth rates under cyclic and static loading approach one another.

3.3.4 Summary

At elevated temperatures the fatigue crack growth rates are dependent on the frequency of cycling. This variation may be appreciated by considering fig. 3.13 in which the crack growth rate

per cycle, da/dN , is plotted against frequency for specific values of the cyclic stress intensity, ΔK . At low ΔK (fig. 3.13a) the crack growth rate increases with increasing frequency. At intermediate values of ΔK , however, (fig. 3.13b) the crack growth rate increases as the frequency is decreased from 1Hz to 0.1Hz, and continues to increase at a frequency of 0.01Hz. Finally, at high values of ΔK (fig. 3.13c) an increase in the crack growth rate is observed as the test frequency is increased from 1Hz to 10Hz. This type of behaviour gives rise to a temperature and frequency dependent value of the critical stress intensity factor K_c , the estimated values of which are summarised in Table 3.3.

3.4 Fractography

The characteristics of the fracture surface were not observed to be significantly influenced by the stress ratio used during crack propagation. However, differences in the fracture surfaces at room temperature and elevated temperatures were observed.

3.4.1 Room Temperature

The room temperature fracture surfaces for $R=0.1$ and $R=0.25$ are typically found to contain large crystallographic facets which in one case, extended across the specimen (fig. 3.14). More usually smaller crystallographic facets are observed together with areas in which crack propagation is seemingly parallel to (001), figs. 3.15a and b. At higher magnification, these areas are found to contain smaller crystallographic facets, fig. 3.16. At levels of ΔK approaching K_c both fractured script carbides and crystallographic fracture are seen,

fig. 3.17.

3.4.2 600°C

The fracture surfaces at low and intermediate levels of ΔK are presented in figs. 3.18a to c for the three frequencies used in testing. It is apparent that in all cases the crack propagation is predominantly parallel to (001) with a small amount of {111} crystallographic fracture and script carbide particles also visible. Although the carbides intersect the fracture surface they show little evidence of initiating further failure (fig. 3.19). The finer detail of the areas parallel to (001) is often obscured by oxidation, but striations are often observed, and an example of this is shown in fig. 3.20.

The appearance of the fracture surface described above is maintained to higher levels of ΔK at lower frequencies. At levels of ΔK where the critical stress intensity factor appropriate to the testing conditions is approached, changes in the fracture morphology occur.

Examples of the fracture surfaces at high growth rates are presented in figs. 3.21a to c. Under these conditions the fracture of script carbides is clearly seen and extensive {111} crystallographic failure may also occur in regions adjacent to these particles.

3.4.3 850°C

The major features of the fracture surfaces obtained at 850°C are similar to those described for fracture at 600°C. The low and intermediate levels of ΔK give rise to crack propagation essentially

parallel to (001), figs. 3.22a and b. At high ΔK 's failure is once again associated with the script carbides and some crystallographic failure is in evidence, figs. 3.23a and b. Similarly, the fracture surface produced by creep crack growth predominantly contains regions associated with fracture at script carbide particles, figs. 3.24a and b.

3.4.4 Summary

From the fractographs presented it can be clearly seen that at intermediate levels of ΔK , the fracture appearance is significantly affected by changes in temperature. At room temperature the predominant mode of crack growth is by crystallographic failure. In some cases this may extend over large distances or it may be restricted in size giving rise to the appearance that crack propagation is parallel to (001).

However, at elevated temperatures crystallographic failure is limited in its extent and in many cases crack propagation along (001) is observed to be accompanied by striation markings.

With increasing ΔK the effects of temperature are less marked. In all cases, at stress intensities approaching the appropriate critical stress intensity the fracture surfaces contain large areas of fractured carbides together with some crystallographic failure.

3.5 Crack Tip Deformation

Several tests were interrupted under known conditions of loading to allow the examination of the crack tip region. The size of the crack tip plastic zone and the local strain distribution were

determined using selected area channelling patterns.

3.5.1 Effects of Deformation on S.A.C.P.'s

The effect of strain on the quality of S.A.C.P.'s can be seen in figs. 3.25a and b. It is apparent that in the undeformed specimen (fig. 3.25a) high order lines, typically the 4th order of {200}, are visible. As deformation increases the acuity of the lines diminishes and the higher order lines are lost. From these changes, strains of approximately 1% may be accurately detected.

The determination may be more quantitatively assessed in terms of the angular resolution of the lines. The measurement of the angular resolution of (200) and (220) lines as a function of strain are presented in fig. 3.26. It may be seen that little scatter is associated with the results, thus permitting the accurate detection of strain, in the present work to within $\pm 0.25\%$.

3.5.2 Plastic Zone Sizes

Measurements of the plastic zone size associated with a propagating crack were taken from the mid-section of specimens fatigued with $R=0.1$ under varying conditions of temperature and frequency. At elevated temperature propagation was interrupted at stress intensities at which increased crack propagation rates occur on decreasing the frequency. In addition, one specimen at 600°C and 0.1Hz was interrupted at $\Delta K=45\text{MPa}/\text{m}$ to enable the high growth rate regime to be investigated. At room temperature a specimen was chosen with a ΔK close to that of the 600°C tests.

The plastic zone size obtained at room temperature and

$\Delta K=21.6\text{MPa}/\text{m}$ is shown in fig. 3.27. It is evident that a certain amount of asymmetry is present since the dimensions of the plastic zone directly ahead of the crack tip are not the same as those perpendicular to the crack plane.

In contrast, the plastic zone obtained at 600°C , 10Hz and $\Delta K=23.4\text{MPa}/\text{m}$ (fig. 3.28) shows more symmetry in its dimensions and a maximum size at an angle of approximately 30° to the crack propagation direction. The dimension directly ahead of the crack tip is, however, close to that obtained at room temperature.

The plastic zones for 600°C , $\Delta K=23.4\text{MPa}/\text{m}$ and frequencies of 1Hz and 0.1Hz are shown in figs. 3.29 and 3.30 respectively. It is apparent that at a constant temperature and ΔK , reductions in the frequency give rise to a progressive increase in the plastic zone size.

The influence of the loading rate on the plastic zone size at room temperature was investigated by loading a specimen until the stress intensity reached $24\text{MPa}/\text{m}$ in a time of 50 seconds, that is equivalent to a frequency of 0.01Hz. The plastic zone dimension directly ahead of the crack tip was observed to be within $3\mu\text{m}$ of that associated with fatigue crack growth at room temperature and 10Hz.

At a constant temperature of 600°C and a frequency of 0.1Hz, the effect of loading at $\Delta K=45\text{MPa}/\text{m}$ may be seen in fig. 3.31. A further increase in the plastic zone size is observed, being most evident in the dimension directly ahead of the crack tip.

At 850°C , crack propagation was interrupted at $\Delta K=30.6\text{MPa}/\text{m}$. The plastic zone sizes for frequencies of 1Hz and 0.1Hz are given in fig. 3.32. An increased plastic zone size is, once again, associated with the lower frequency.

Various dimensions of the plastic zone have been used to compare the sizes. For example, it is possible to use the distance at some pre-determined angle θ to the crack plane or to consider the dimension directly ahead of the crack tip. The majority of equations developed to estimate the plastic zone size (section 1.3) are made for a distance directly ahead of the crack tip; this experimentally determined dimension is given in Table 3.4 for each of the loading conditions investigated.

In general the equations which estimate the monotonic plastic zone size, r_p , can be written as

$$r_p = \alpha \frac{K^2}{\sigma_y} \quad (3.1)$$

where α is a constant of proportionality. In the present study, a value of α , calculated from the room temperature data, may be used to estimate the plastic zone sizes at elevated temperature. These estimates are included in Table 3.4. From these values it may be seen that the predicted value at 600°C and 10Hz is close to the experimentally measured value. However, with decreasing frequency an increasing discrepancy becomes apparent. This discrepancy is maintained at 850°C.

3.5.3 Strain Distribution within the Plastic Zone

The variation of plastic strain with the distance directly ahead of the crack tip, d_{pz} , was determined by analysing the deterioration in the S.A.C.P.'s in terms of the strain (fig. 3.26). An example of the deterioration within the plastic zone is shown in fig. 3.33. Each

S.A.C.P. allows the strain associated with a small volume of material having a surface area of $2-3\mu\text{m}$ in diameter to be determined.

The results for the room temperature plastic zones and for those at 600°C are presented in fig. 3.34. It is evident that the specimens deformed at 600°C tend towards the same local value of strain at the crack tip, approximately 14%. However, as the frequency is lowered there is not only an increase in the plastic zone size but a decrease in the gradient of strain within the plastic zone. Further, the level of strain may be seen to increase progressively through the plastic zone up to the crack tip.

The room temperature data show a similar strain gradient to that observed in the 600°C 10Hz specimen, but show a significantly lower maximum strain at the crack tip, approximately 8%.

The strain distribution in the plastic zones at 850°C is shown in fig. 3.35. Similar features are exhibited to those seen in the 600°C data. The data tend to a local crack tip strain of approximately 14% and show a decrease in the strain gradient with decreasing frequency.

3.5.4 Summary

The investigations of the plastic zone size show that its size is a function of the value of ΔK , the temperature and the frequency. The plastic zone size at room temperature may be used to predict closely the plastic zone size at 600°C and 10Hz. However, at the same ΔK and temperature the plastic zone size is observed to increase progressively with decreasing frequency. This trend is maintained as the temperature is raised to 850°C .

The strain distributions within the plastic zone show that

although the room temperature and the 600°C 10Hz specimens have similar strain gradients, the room temperature crack tip strain of approximately 8% is significantly lower than the observed value of 14% at 600°C and 10Hz. As the frequency of testing is decreased or the temperature increased, the local crack tip strain maintains a value of approximately 14%.

3.6 Preliminary Discussion

The effect of deformation on the S.A.C.P.'s in the present work is similar to that reported by Stickler et al. (1971) and Spencer et al. (1974). Both line broadening and the loss of higher order lines are consistent with an increase in the deformation of the specimen. Although the effect is more correctly correlated with dislocation density (Spencer et al., 1974), the present results show that the angular resolution of specific (hkl) lines can be accurately related to the true strain.

Numerous theoretical treatments of the effect of deformation on S.A.C.P.'s have been made (see e.g. Davidson, 1981). A more qualitative explanation can be offered in terms of the contrast mechanism by which S.A.C.P.'s are generated. In an undeformed specimen rocking the beam over the specimen surface gives rise to a variation in the intensity of secondary and backscattered electrons. At the Bragg condition, the beam suffers anomalous absorption and transmission (Booker et al., 1967) and the resulting variation in contrast gives rise to the formation of a channelling pattern. If the specimen is deformed such that a dislocation is introduced a localised bending of the lattice planes will result. Subsequent rocking of the

beam will give rise to satisfaction of the Bragg condition for angles of incidence other than those in a perfect crystal. Consequently the angular width of individual lines will be increased and the pattern quality will degrade. In practice many dislocations are introduced on deformation and the size of the selected area is larger than the individual defect size. An average effect of the number of defects within the sampling volume is then observed, with increasing deformation the effect correspondingly increases.

The results of Stickler et al. (1971) and Spencer et al. (1974) show a larger amount of scatter than the present results. This may, in part, be due to the higher accelerating voltage and greater stability of the systems used in the present study, but may also be associated with their use of polycrystalline specimens. The presence of grain boundaries may result in more inhomogeneous deformation due to the incompatibility of the slip systems across a boundary. In single crystal material, however, such problems do not arise and strains of approximately 1% were readily detectable.

A large amount of work (section 1.6.1) has investigated the applicability of linear elastic fracture mechanics parameters to the characterisation of elevated temperature crack growth. It has been suggested (Sadananda and Shahinian, 1981d) that the achievement of larger plastic zones at elevated temperature may limit the use of linear elastic fracture mechanics. If time dependent relaxation of crack tip stresses and extensive blunting of the crack tip occur the distribution of stresses at the crack tip may well no longer be characterised by K . However, the present work indicates that, even at high temperatures and low frequencies, discrete areas of plasticity may be associated with a growing crack. In these circumstances, the

measurement of crack growth rates at different crack lengths but constant applied stress intensity show that the crack growth rate may be adequately characterised by the stress intensity factor.

Table 3.1

Summary of tensile data for single crystal Mar M 002 at a strain rate
of 10^{-3} s^{-1} , courtesy of N.G.T.E.

Temperature, °C	σ_y , MPa	U.T.S., MPa	ϵ_f , %	N
20	868	975	7.9	0.034
600	904	1141	5.1	0.083
850	771	921	5.3	0.09

where σ_y = 0.01% proof stress

U.T.S. = Ultimate tensile stress

ϵ_f = Elongation

N = work hardening exponent

Table 3.2

Calibration equations for the potential V across a crack of length a for C.C.T. specimens as a function of the test temperature.

Temperature, °C	Equation
20	$a=(4.8 \times 10^{-3} * V^3 - 1.54 \times 10^{-1} * V^2 + 3.1 * V - 3.5)/2$
600	$a=(3.25 \times 10^{-3} * V^3 - 1.19 \times 10^{-1} * V^2 + 2.69 * V - 3.32)/2$
850	$a=(3.81 \times 10^{-3} * V^3 - 1.34 \times 10^{-1} * V^2 + 2.8 * V - 3.58)/2$

Table 3.3

Estimated values of K_C as a function of temperature and frequency.

Temperature, °C	Frequency, Hz	K_C , MPa/m
20	10	30
600	10	35
600	1	39
600	0.1	52
850	1, 0.1, Creep	44

Table 3.4

Summary of plastic zone size directly ahead of the crack tip as a function of temperature, frequency and stress intensity.

Temperature, °C	ν , Hz	$K_{\max}$, MPa $\sqrt{m}$	$r_p(\text{meas})$, μm	$r_p(\text{calc})$, μm
20	10	24	158	
600	10	26	178	170
600	1	26	223	
600	0.1	26	656	
600	0.1	50	1210	
850	1	34	300	402
850	0.1	34	515	

where ν is the frequency,

$K_{\max}$ the maximum applied stress intensity

$r_p(\text{meas})$ the measured monotonic plastic zone size

$r_p(\text{calc})$ the calculated monotonic plastic zone size using equation (3.1) and the yield stress for the particular temperature.

Figure 3.1

Microstructure of single crystal Mar M 002 with cuboidal γ' precipitate aligned along $\langle 100 \rangle$.

Figure 3.2

Microstructure of single crystal Mar M 002 showing γ/γ' eutectic and script carbide phases.

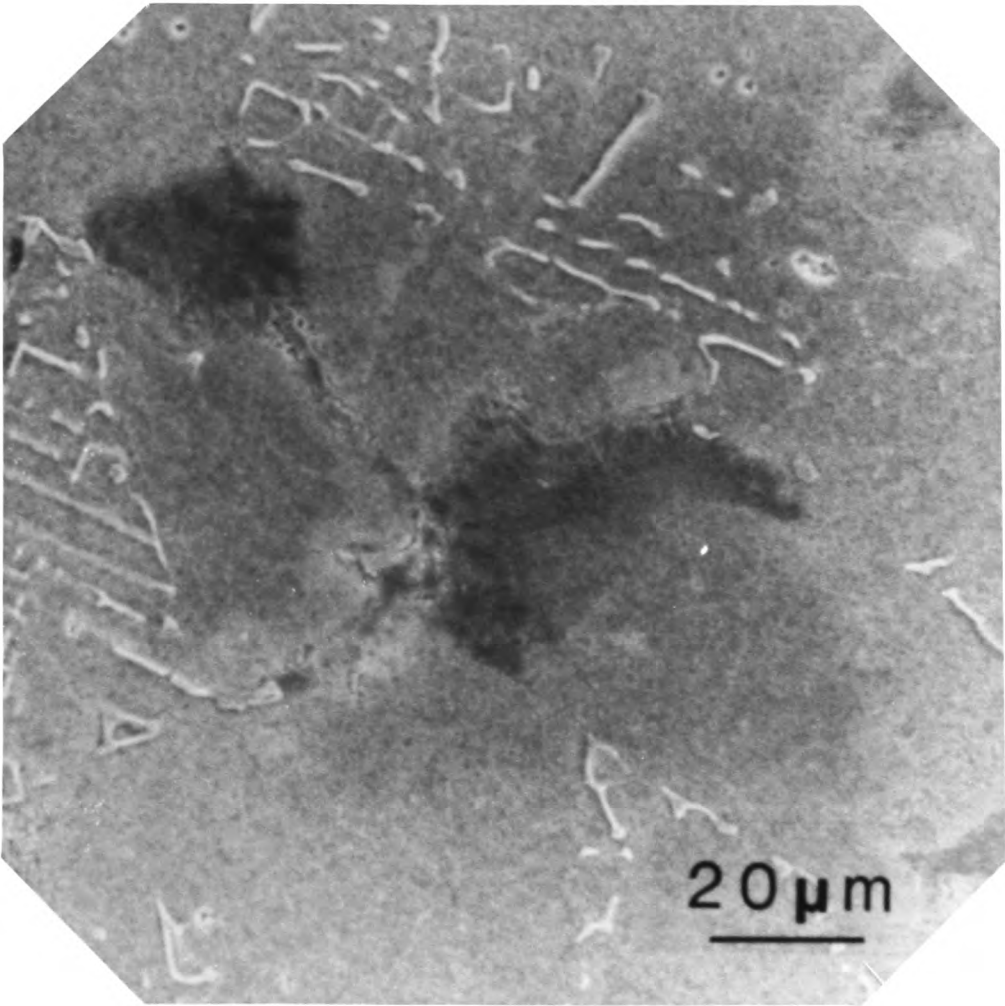
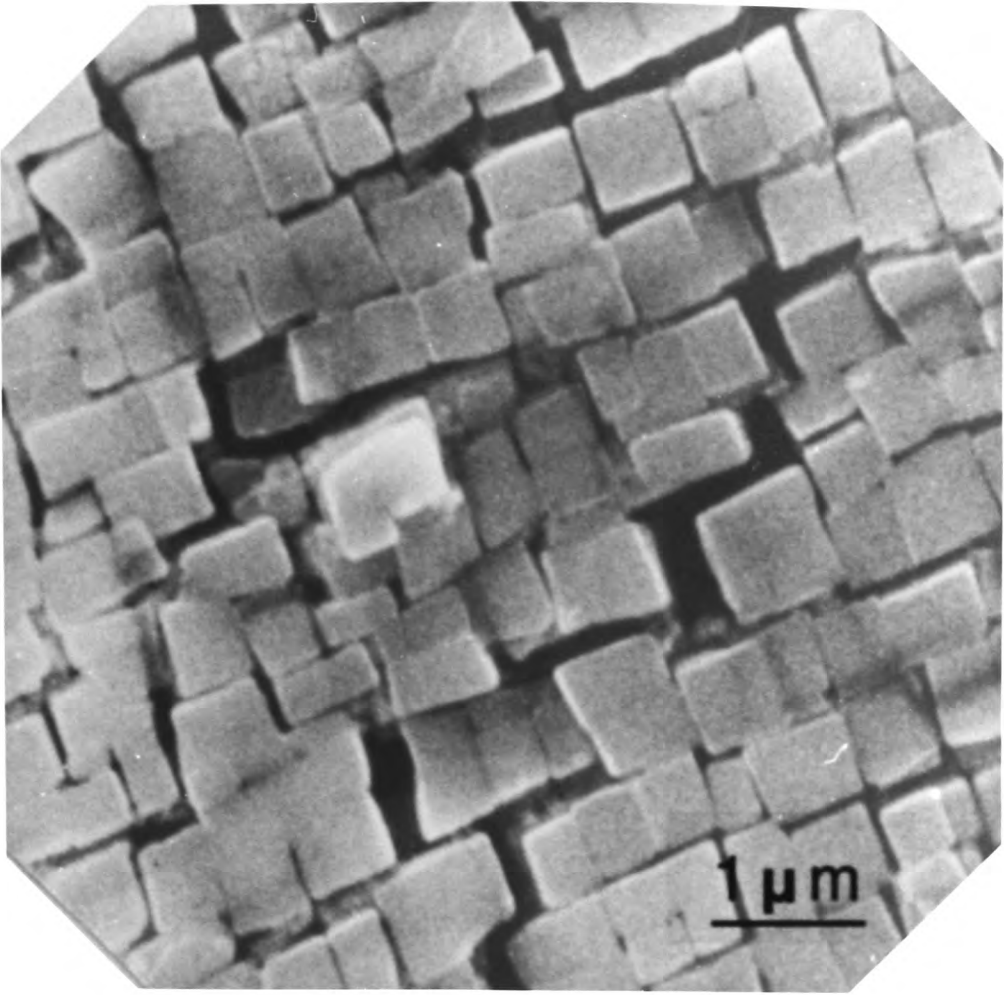


Figure 3.3

Calibration curve of the potential across the crack, V_{2a} , against the ratio of crack length to specimen width, $2a/W$, for C.C.T. specimens at room temperature.

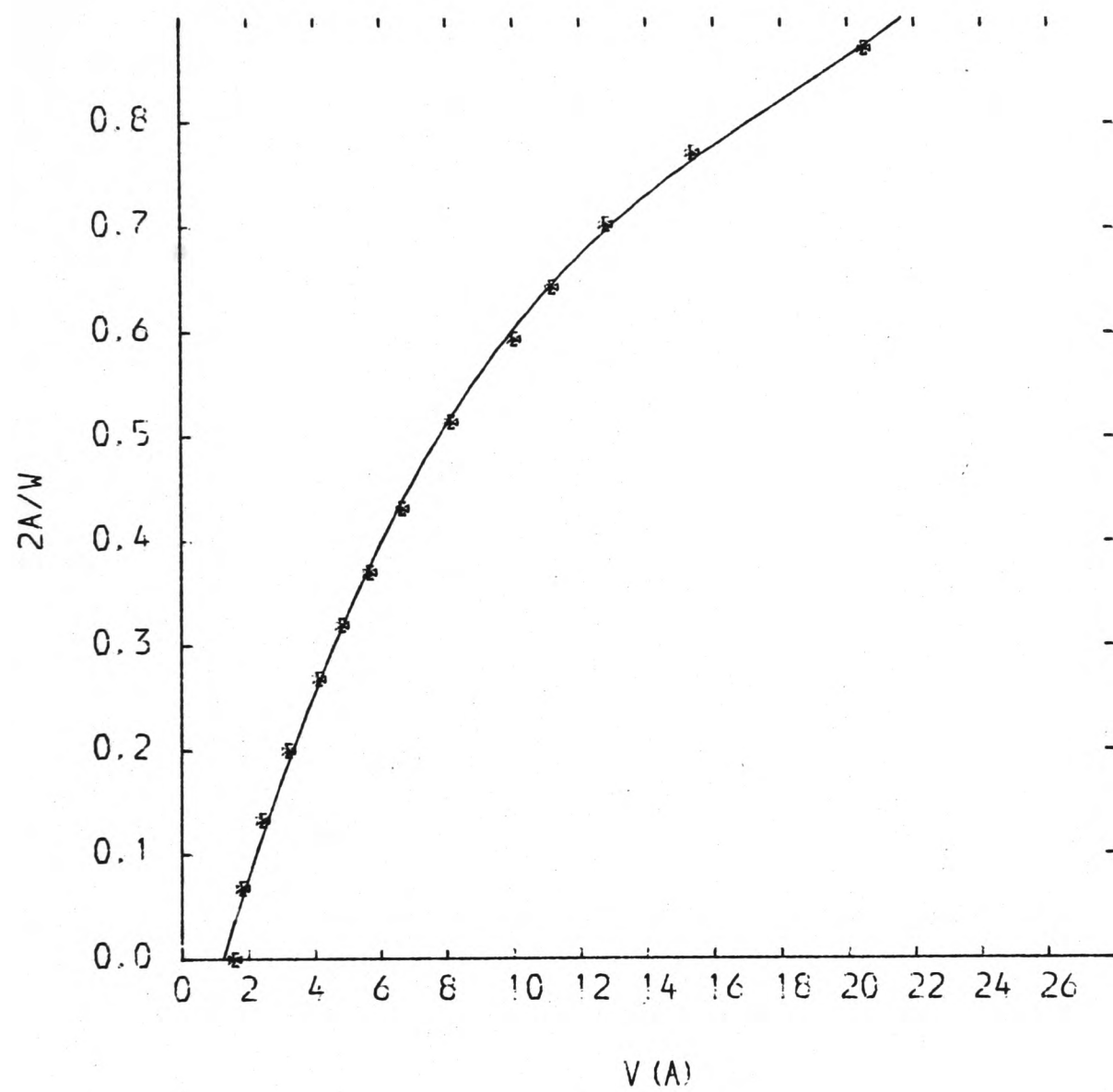


Figure 3.4

Calibration curve of the potential across the crack, V_{2a} , against the ratio of crack length to specimen width, $2a/W$, for C.C.T. specimens at 600°C.

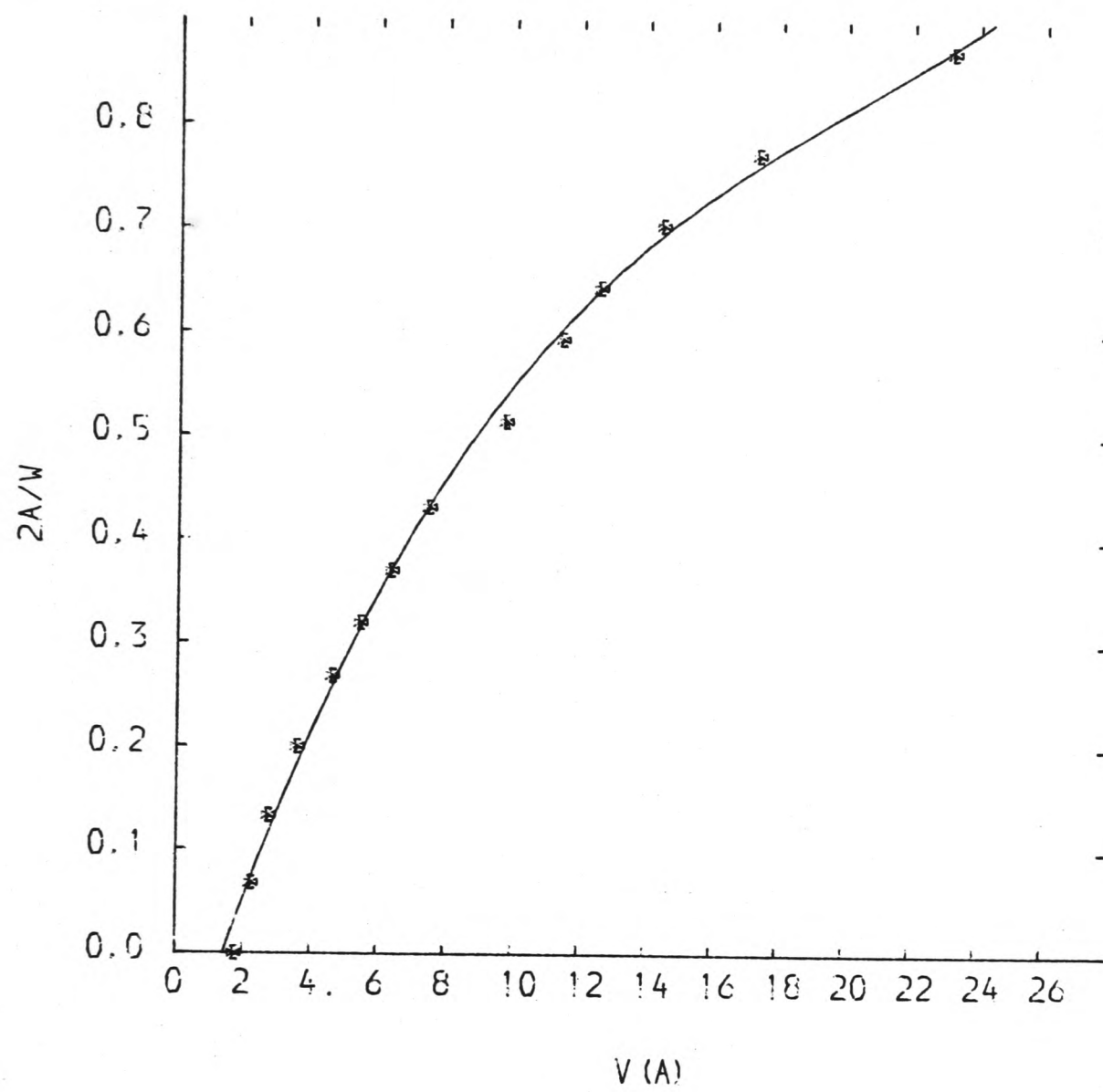


Figure 3.5

Calibration curve of the potential across the crack, V_{2a} , against the ratio of crack length to specimen width, $2a/W$, for C.C.T. specimens at 850°C.

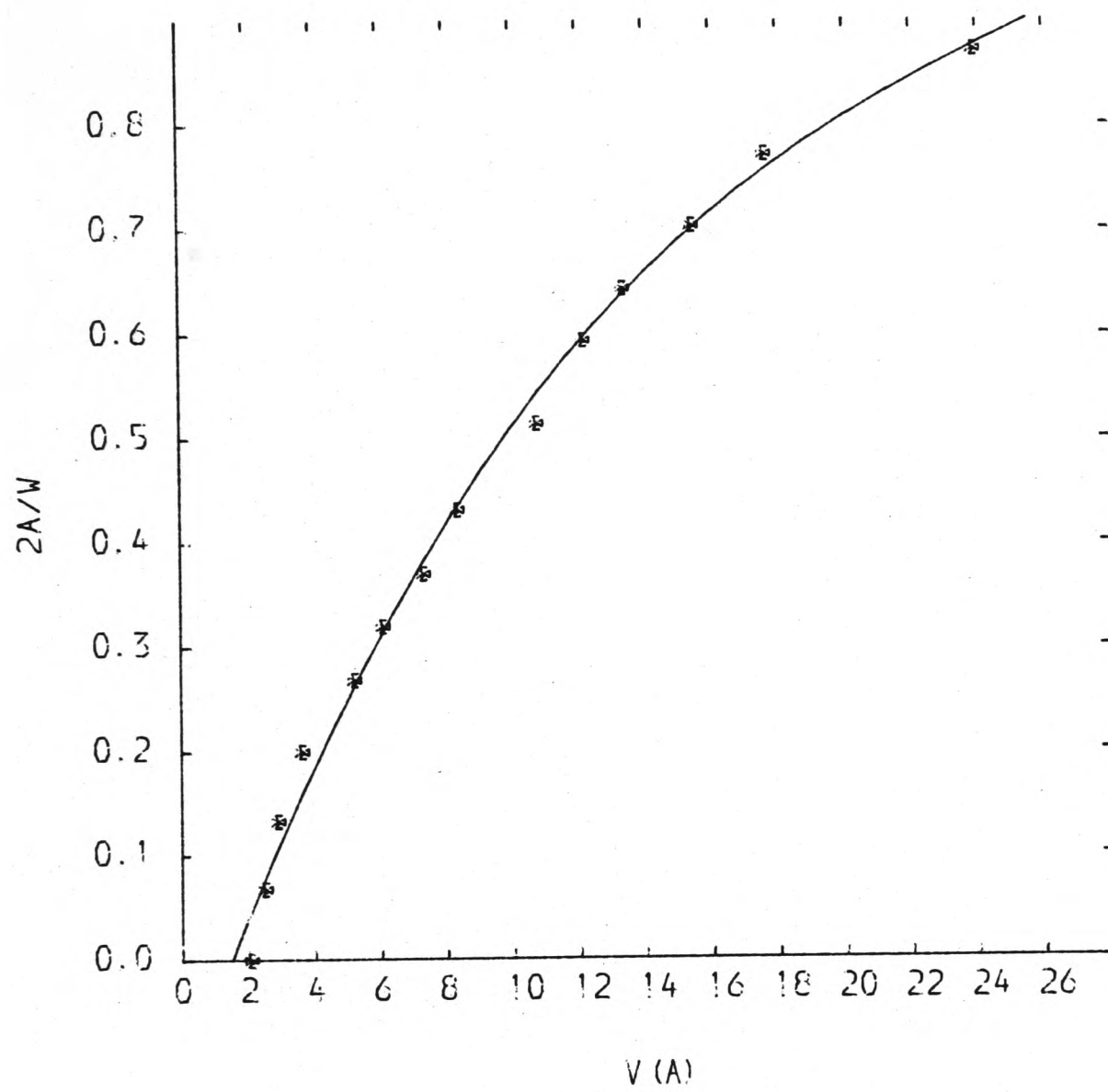


Figure 3.6

Typical experimental plot of half crack length, a ,
against number of cycles, N , at 600°C , 1Hz , $R=0.1$
and $K_{\text{max}}=34\text{MPa}/\text{m}$. Correlation coefficient =
 0.9946 .

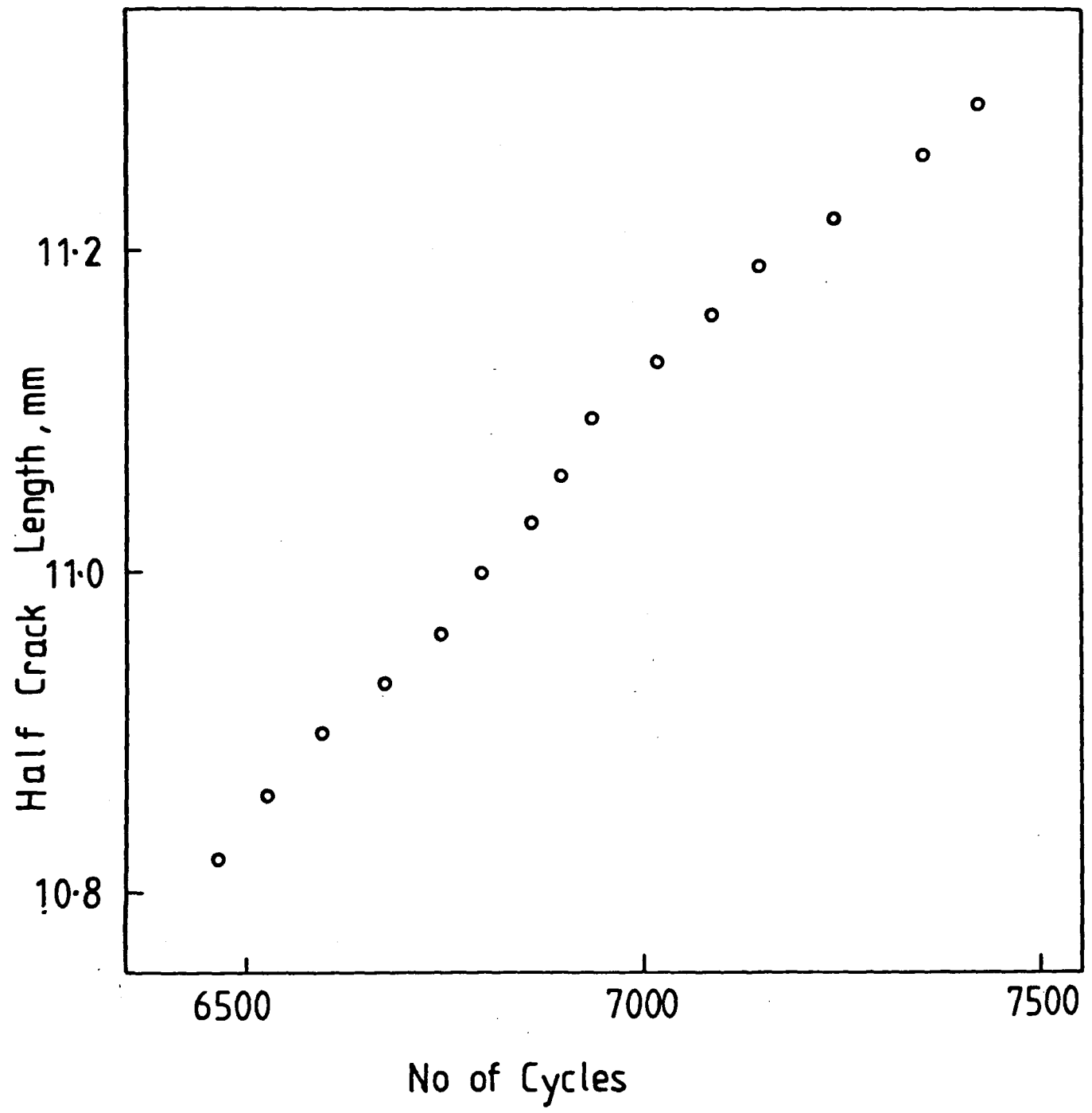


Figure 3.7

Crack growth rate against ΔK at room temperature
for $R=0.1$ and 0.25 .

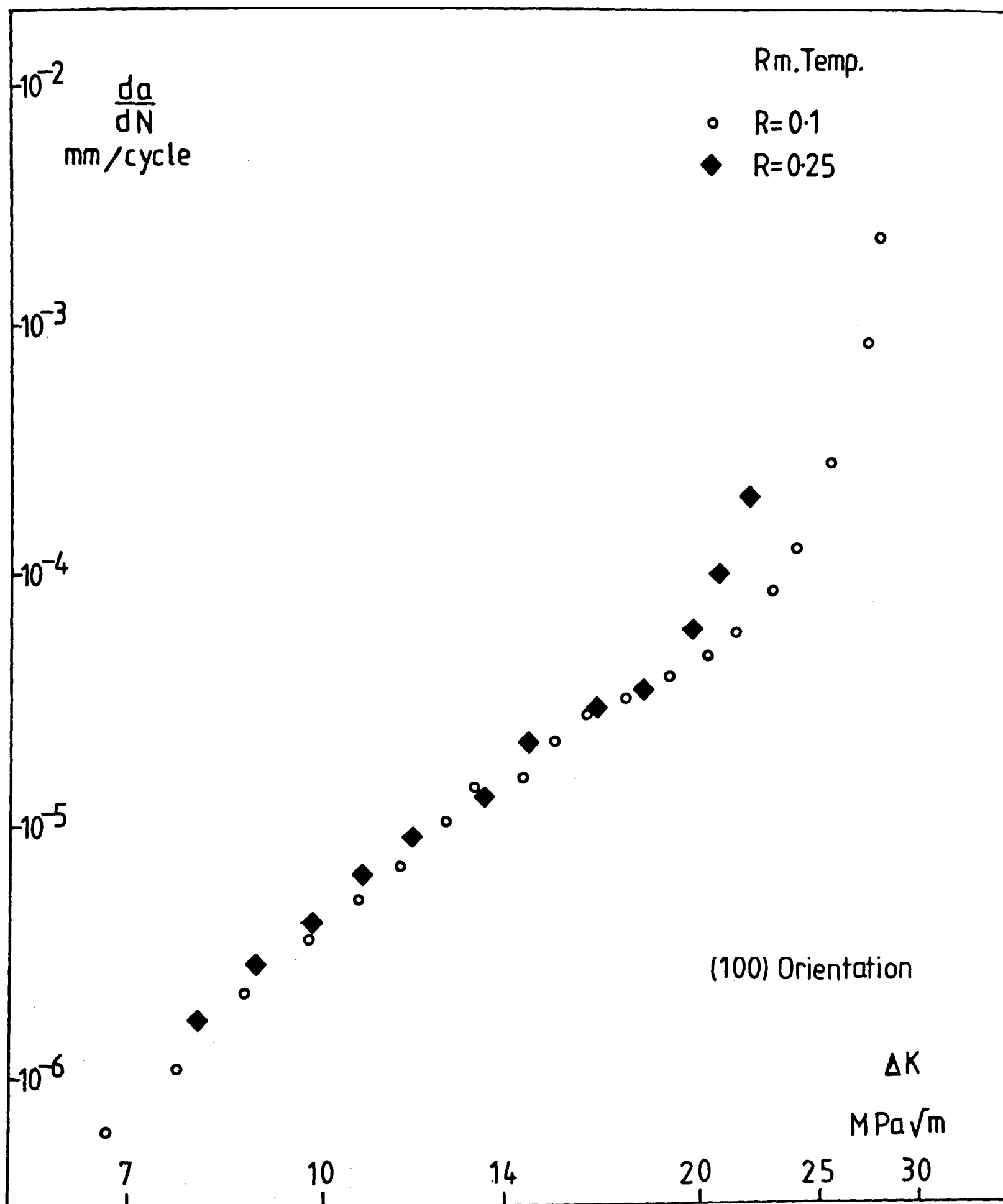


Figure 3.8

Crack growth rate against ΔK at 600°C and
frequencies of 10Hz, 1Hz and 0.1Hz with R=0.1.

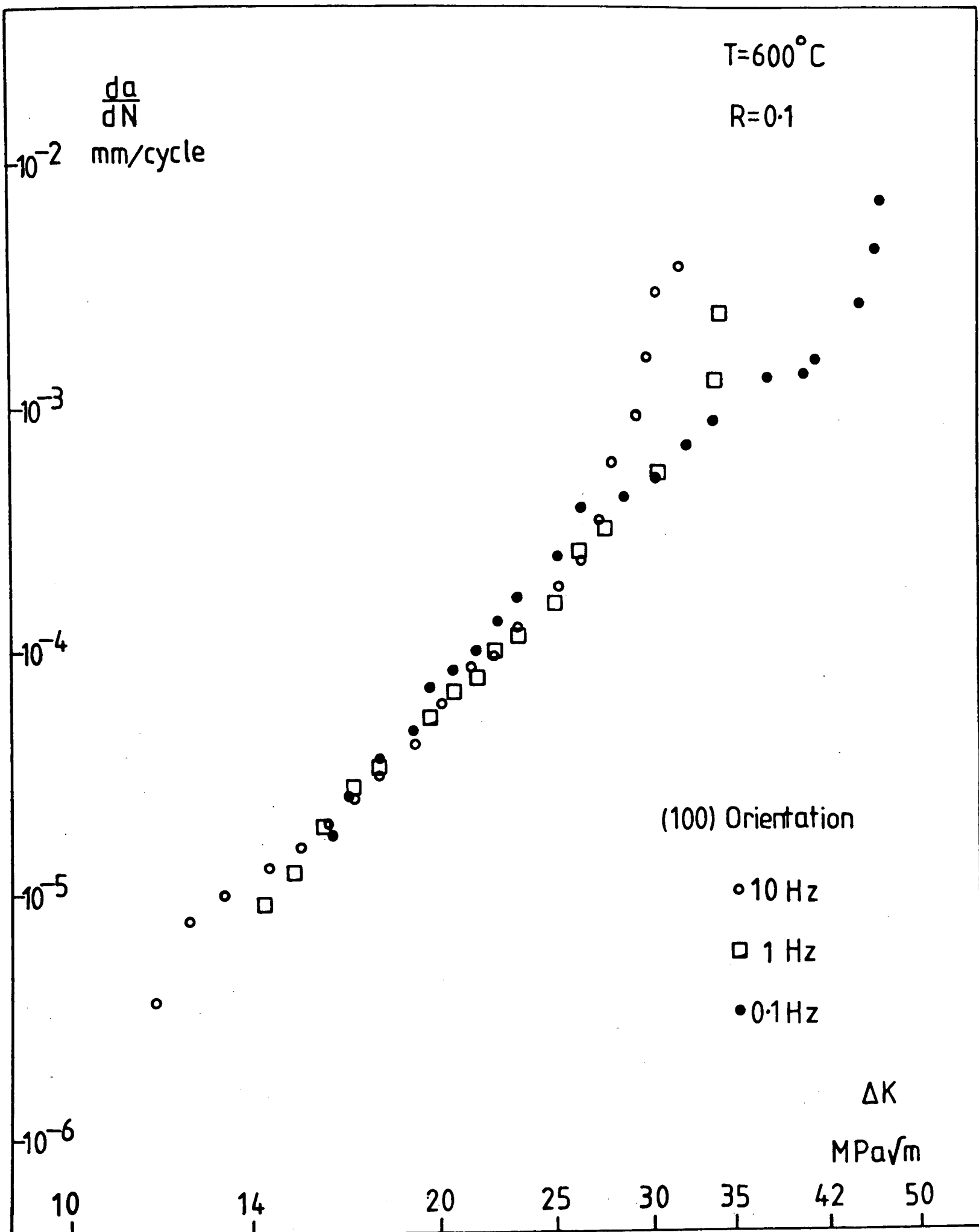


Figure 3.9

Crack growth rate against ΔK at 600°C and frequencies of 10Hz and 0.1Hz with $R=0.25$.

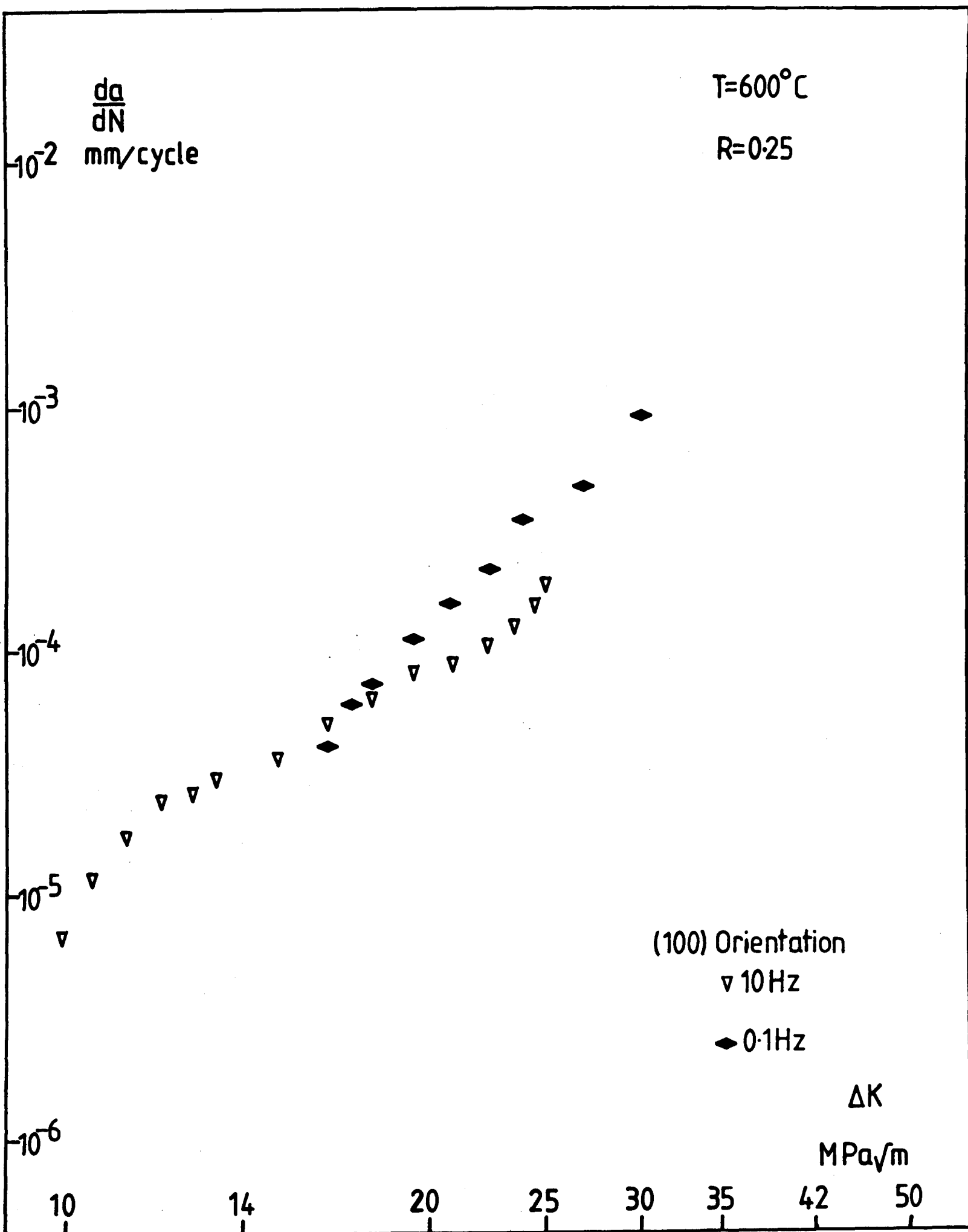


Figure 3.10

Crack growth rate against ΔK at 850°C and
frequencies of 1Hz and 0.1Hz with R=0.1.

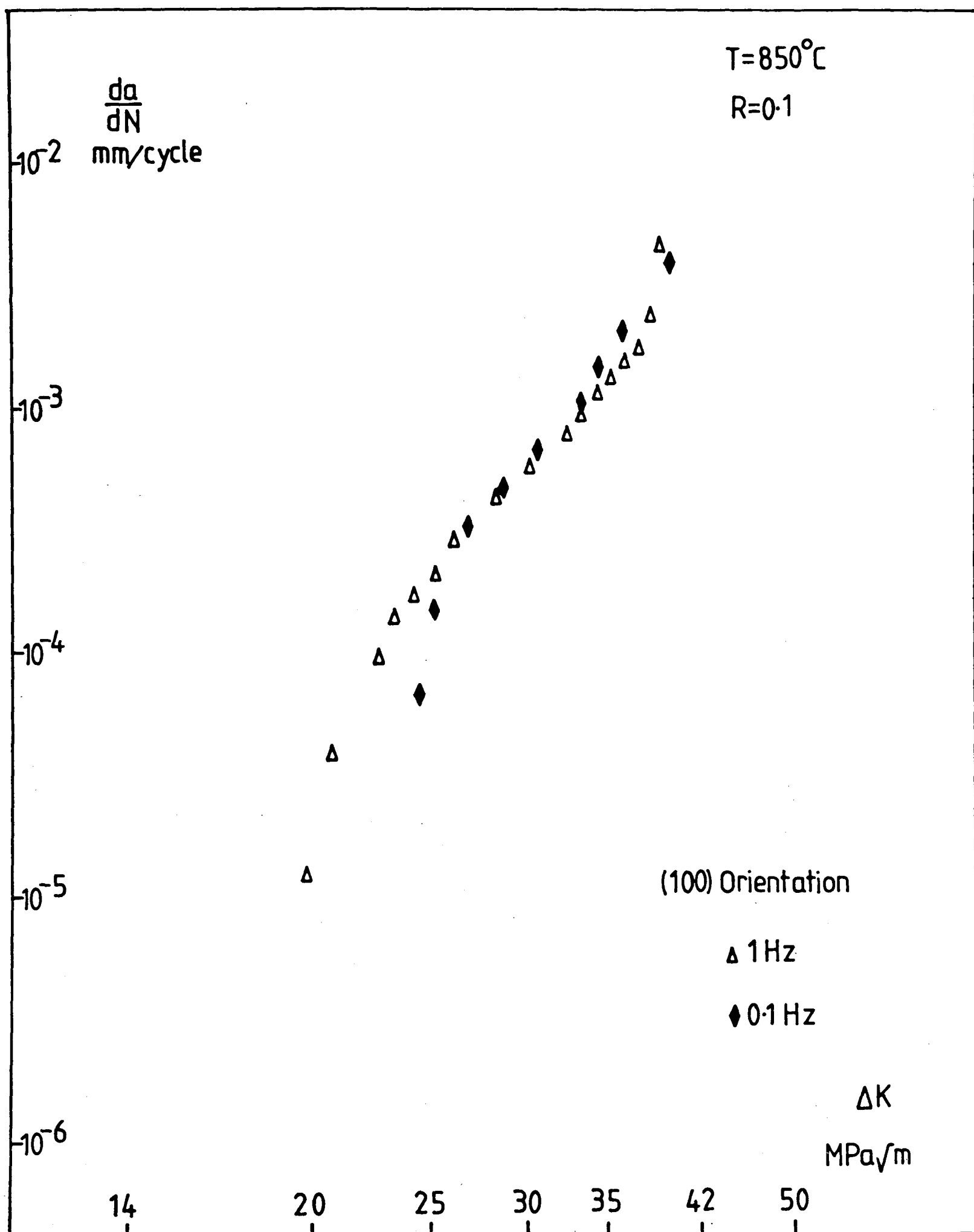


Figure 3.11

Crack growth rate against ΔK at 850°C and frequencies of 1Hz and 0.1Hz with $R=0.25$.

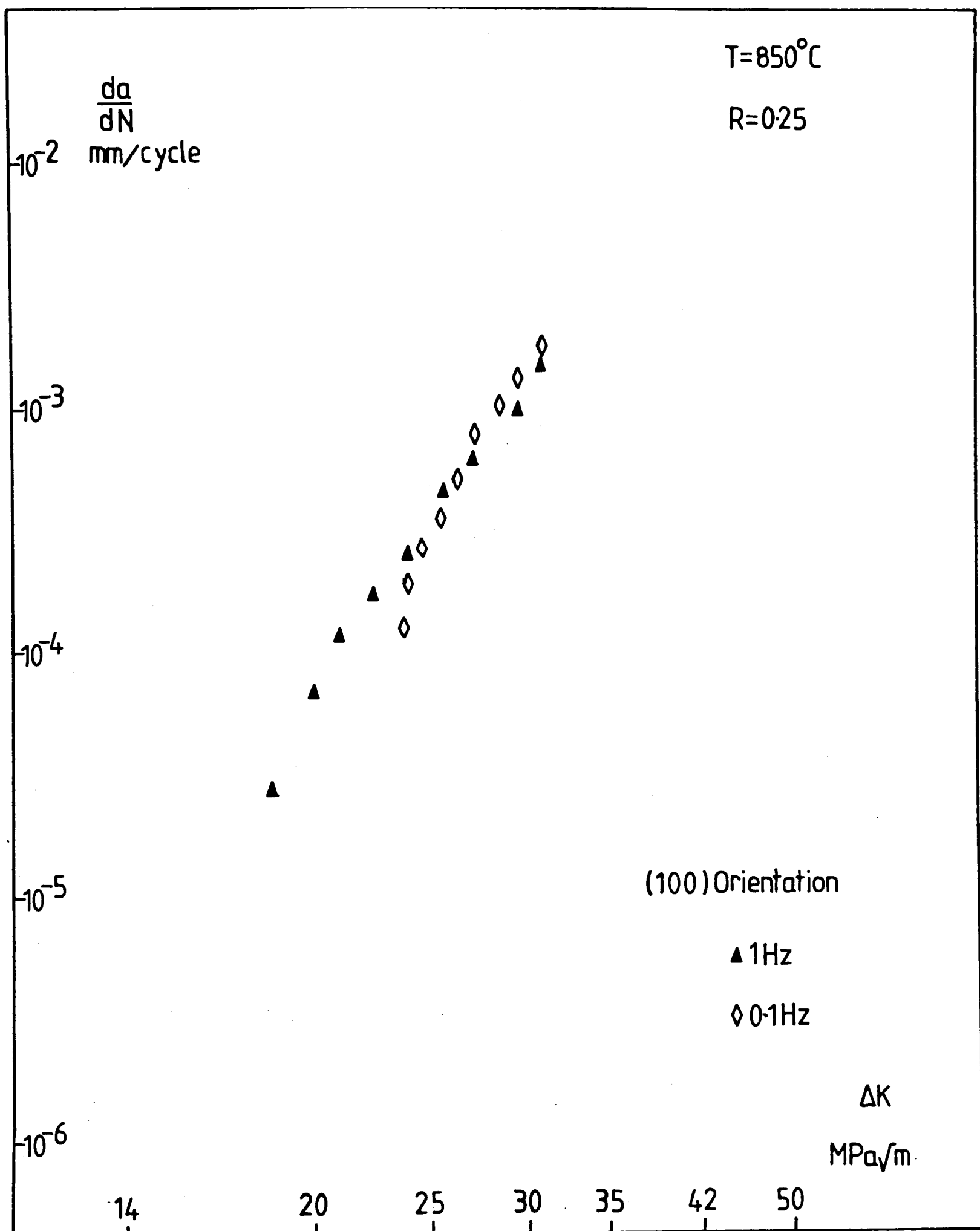


Figure 3.12

Crack growth rate per second against ΔK (1Hz) or K
(static) at 850°C.

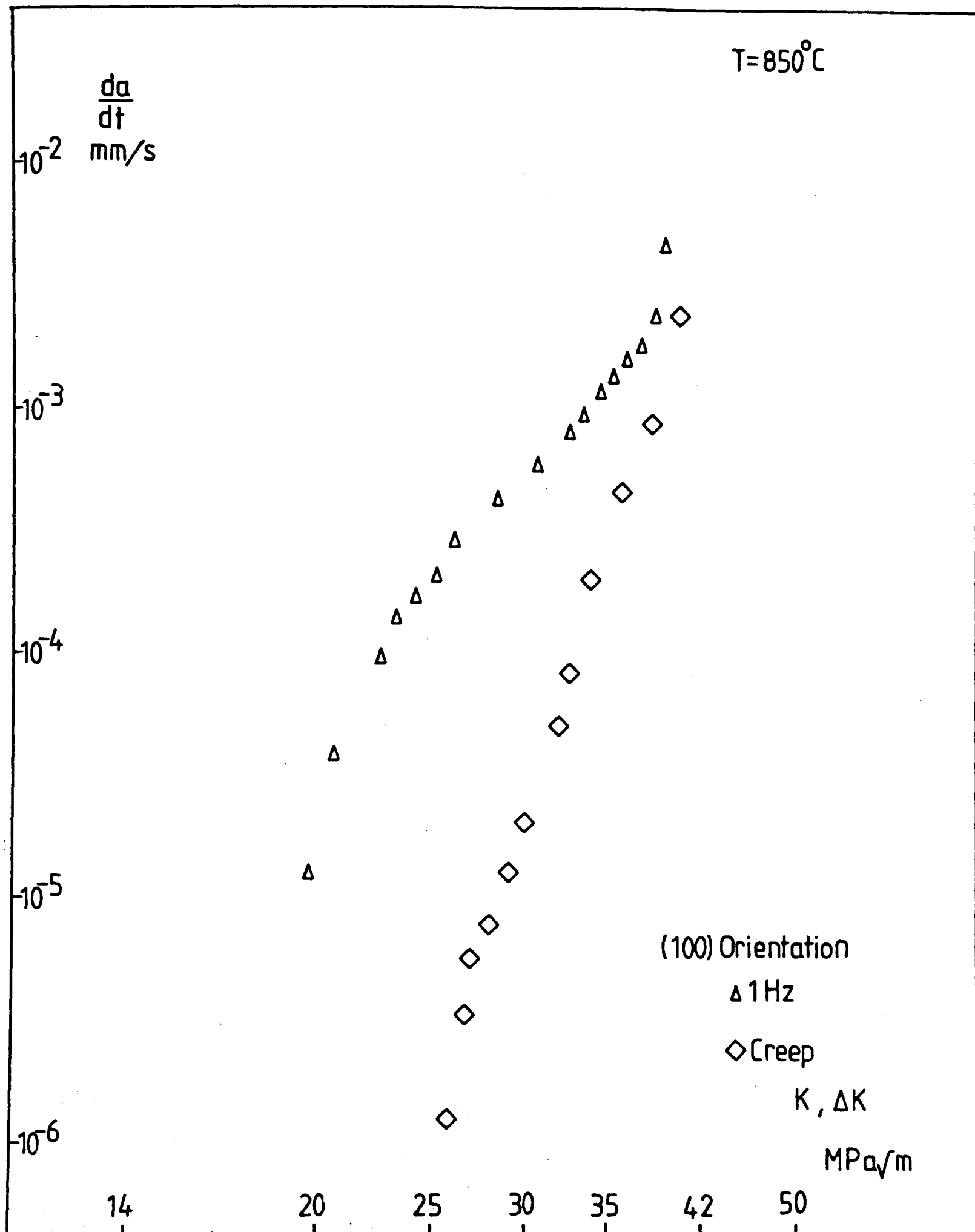


Figure 3.13

Variation of crack growth rate with frequency at constant ΔK and a temperature of 600°C .

a) $\Delta K = 16.0 \text{ MPa}\sqrt{\text{m}}$, $R=0.1$,

b) $\Delta K = 23.4 \text{ MPa}\sqrt{\text{m}}$, $R=0.1$,

c) $\Delta K = 30.0 \text{ MPa}\sqrt{\text{m}}$, $R=0.1$.

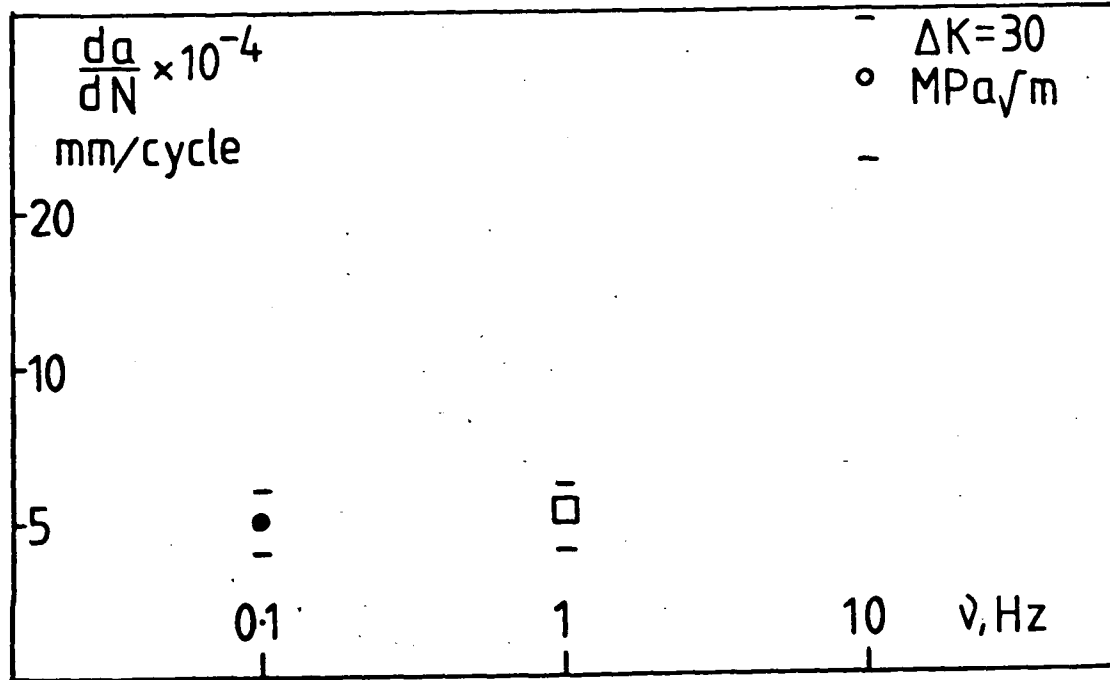
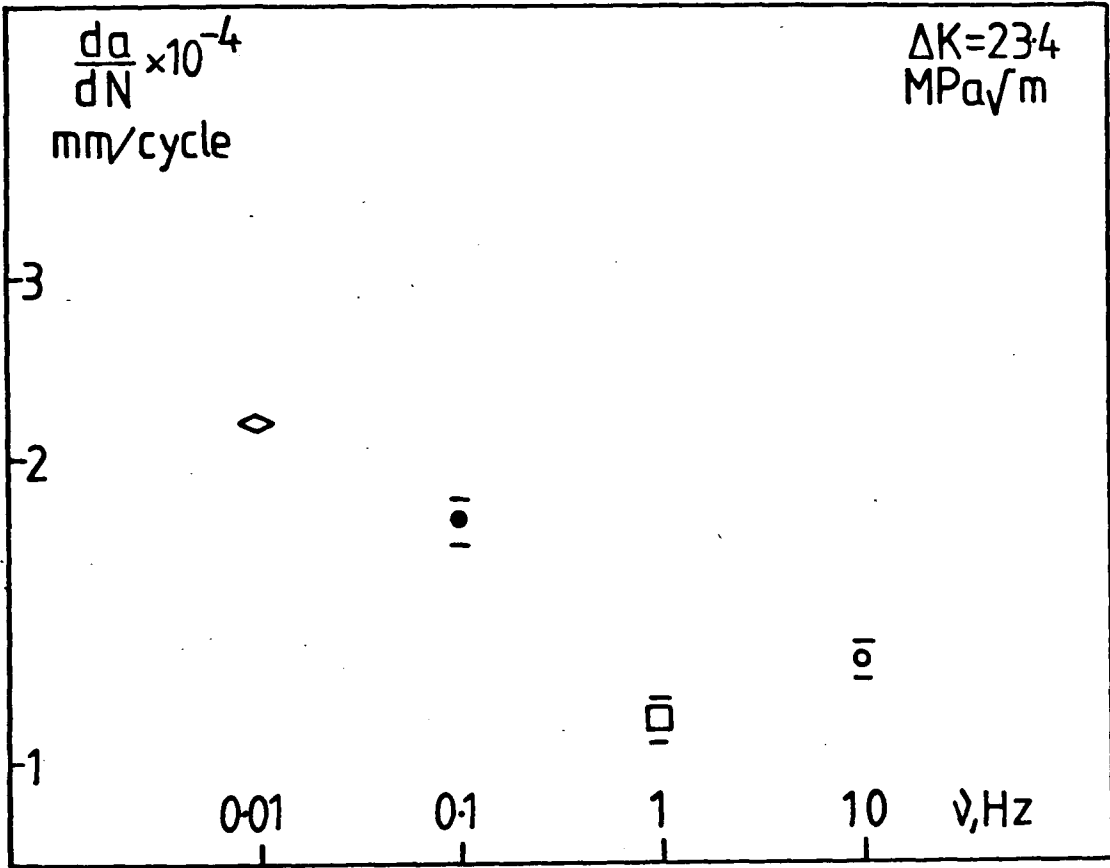
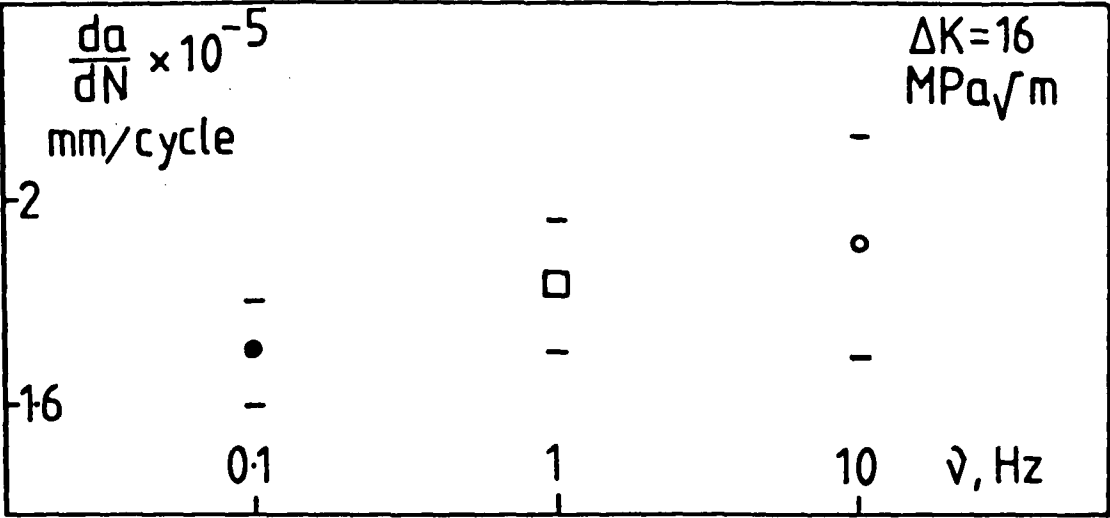


Figure 3.14

Crystallographic facet extending across the width
of a specimen after fracture at room temperature.



Figure 3.15

Fracture surface after fatigue at room temperature
in mid- ΔK regime.

- a) Fracture on both $\{111\}$ and apparent $\{100\}$,
- b) Crystallographic fracture on $\{111\}$.

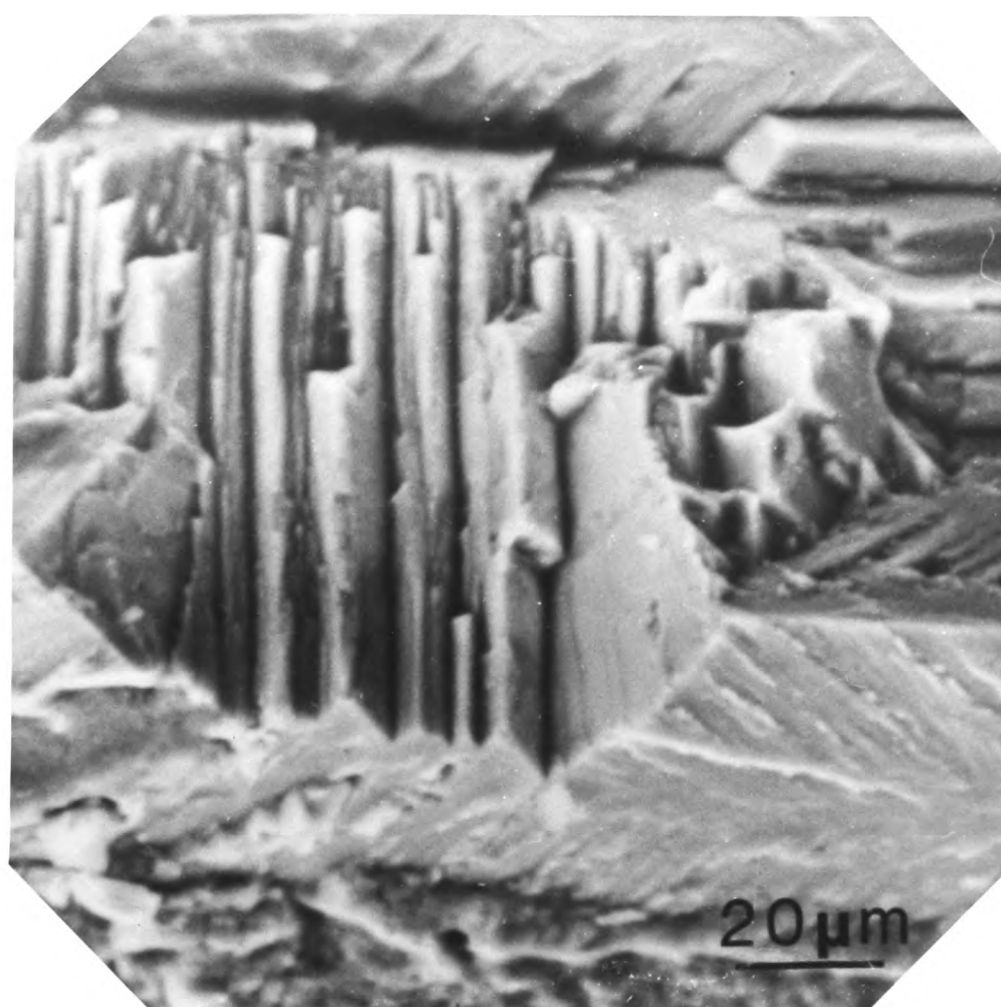
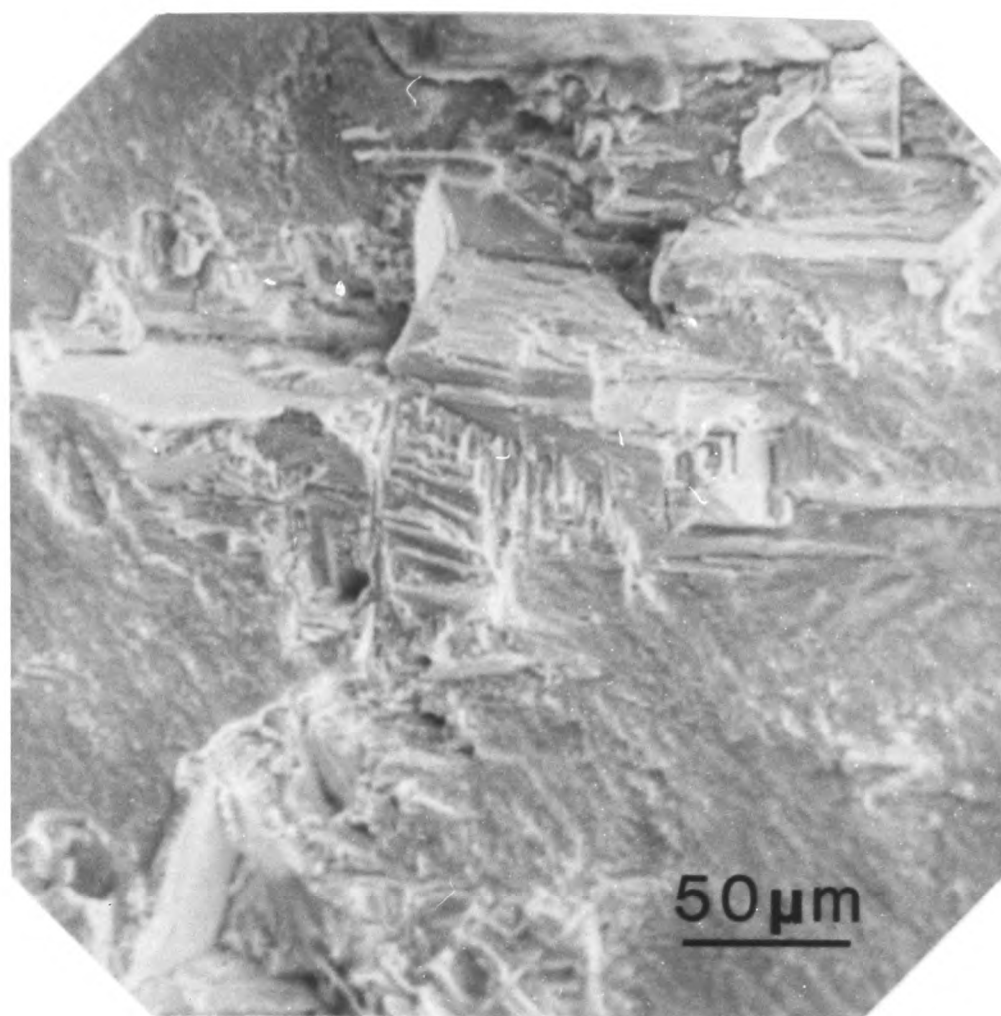


Figure 3.16

High magnification of {100} fracture at room temperature showing alternating fracture on {111}.

Figure 3.17

Room temperature fracture surface at high ΔK showing failure on {111} and fracture of the script carbides.

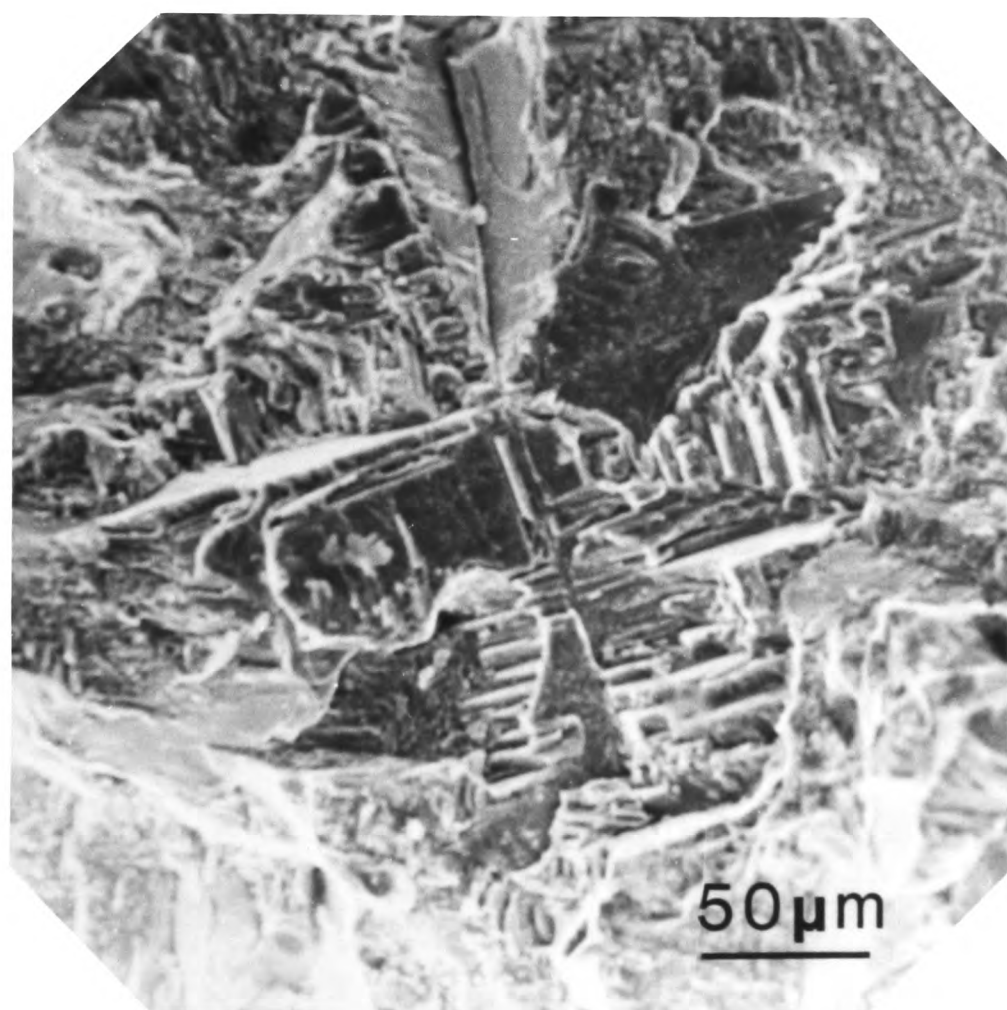
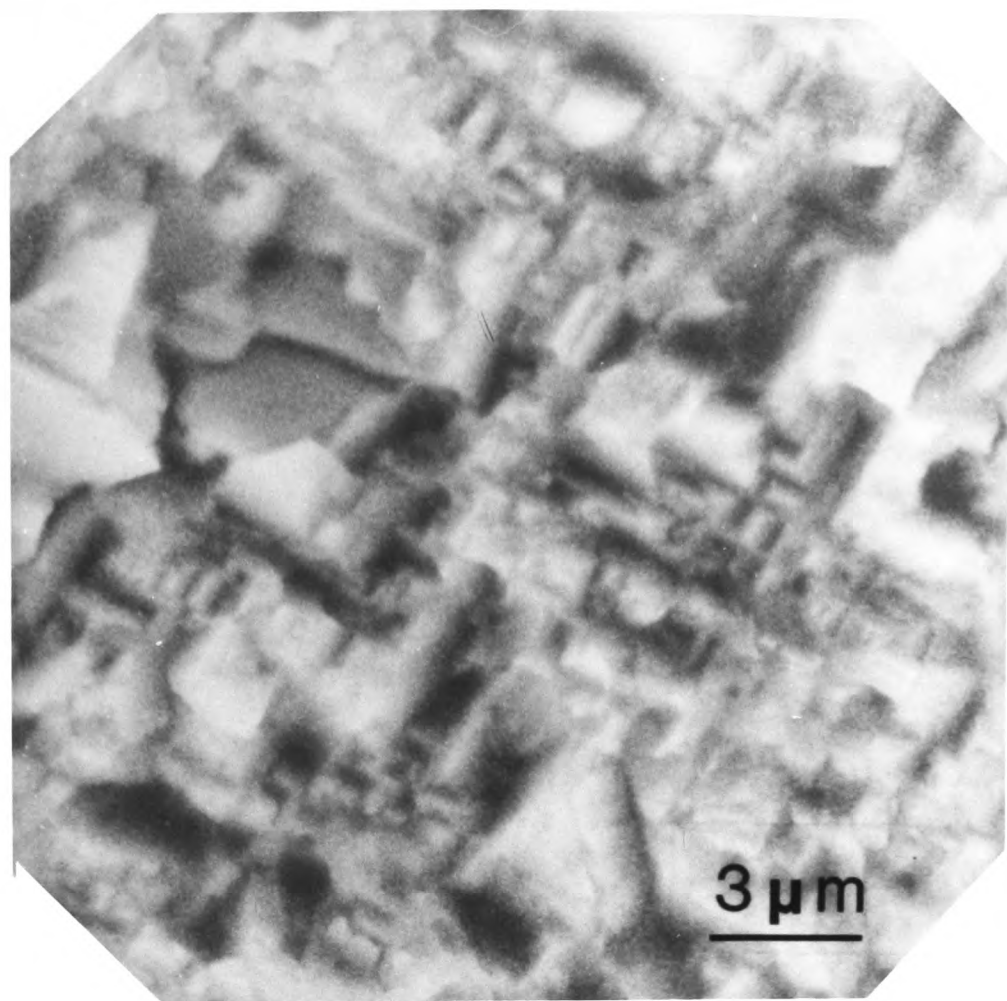


Figure 3.18

Fracture surfaces at 600°C in mid- ΔK regime.

a) $\nu = 10\text{Hz}$,

b) $\nu = 1\text{Hz}$,

c) $\nu = 0.1\text{Hz}$.

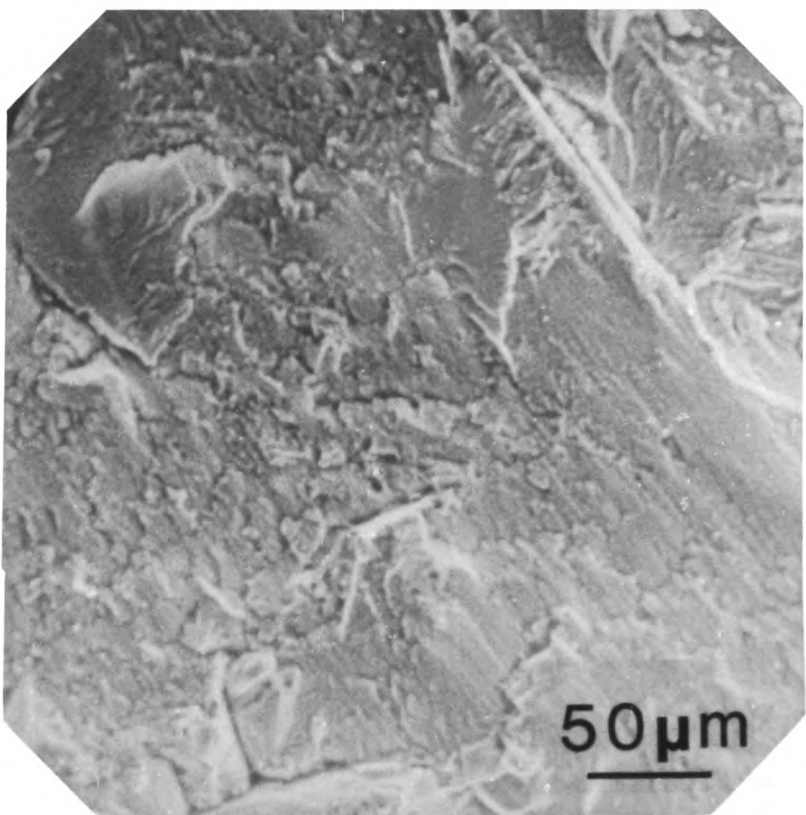
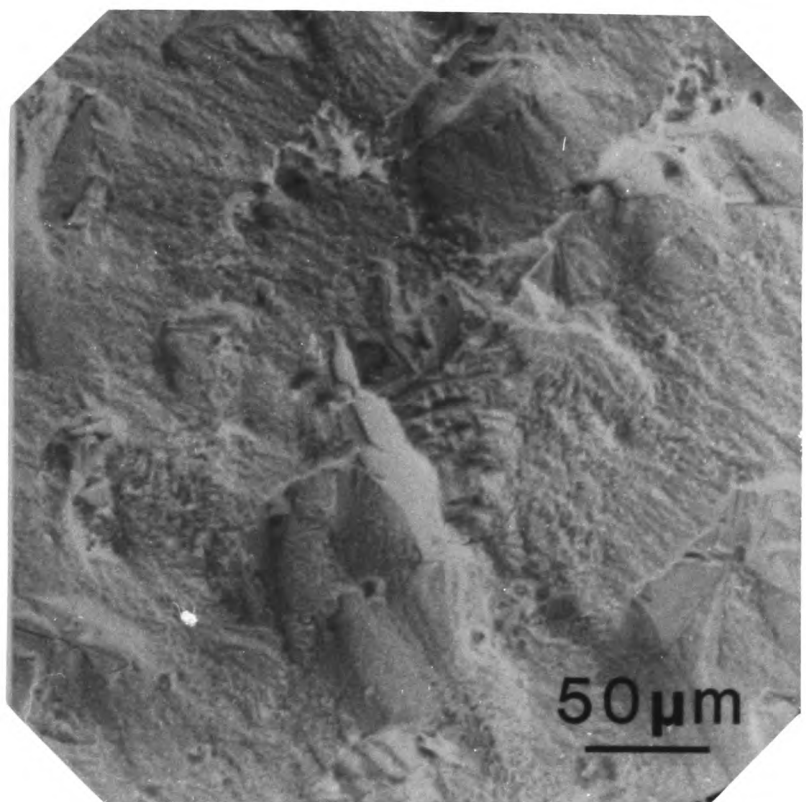
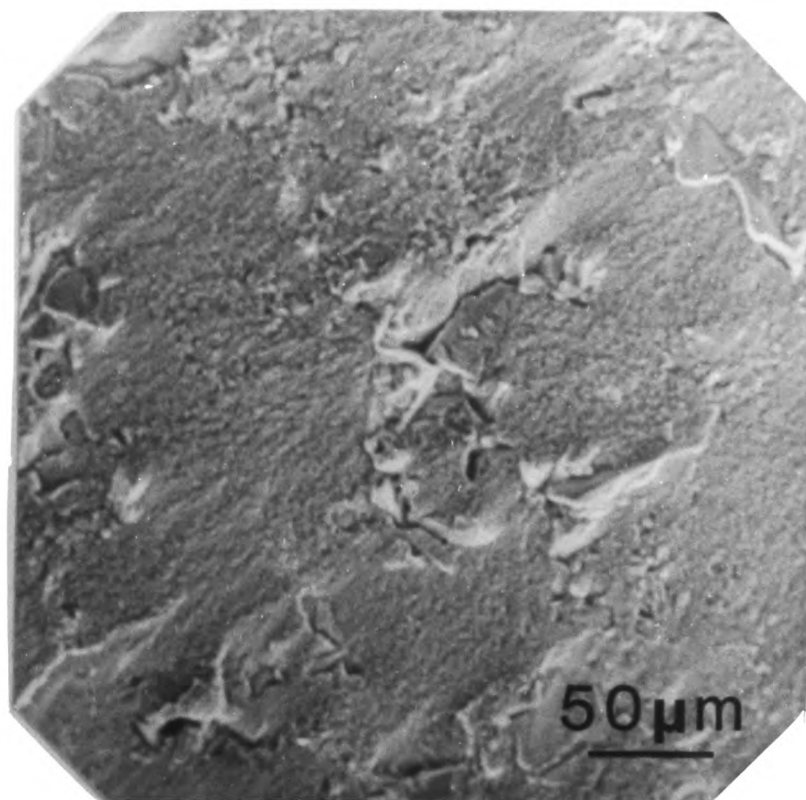


Figure 3.19

Carbide intersecting the fracture plane at 600°C and 10Hz with no evidence of further fracturing of the matrix.

Figure 3.20

Striation markings on the fracture surface at 600°C and 10Hz.

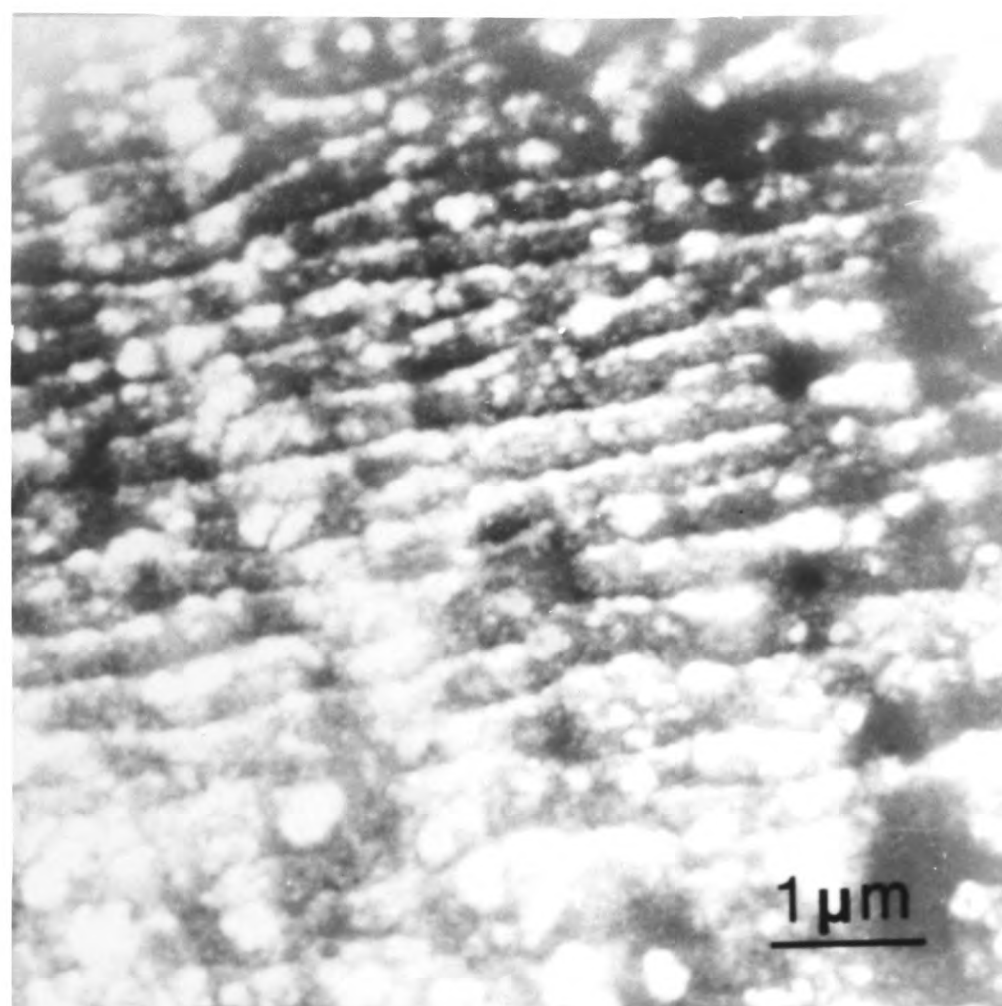
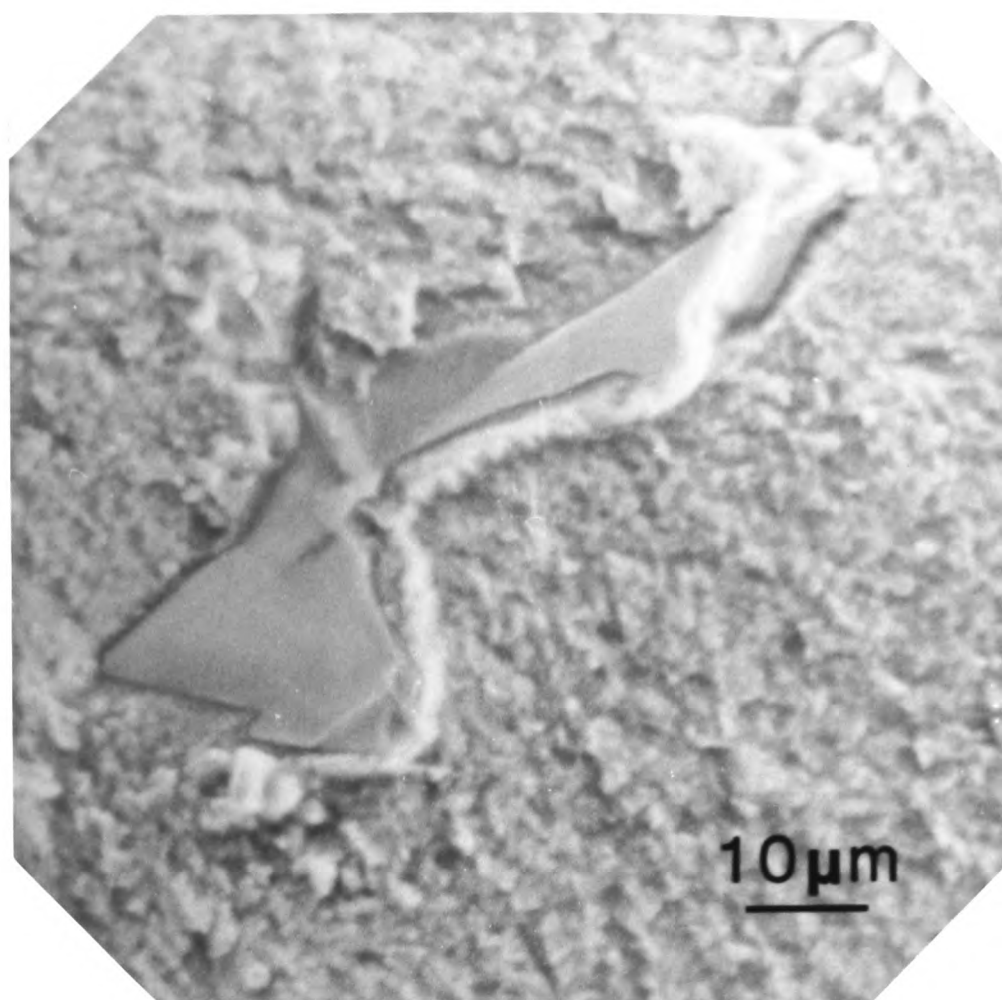


Figure 3.21

Fracture surfaces for high $\Delta K'$ s, approaching K_c for the testing conditions, at 600°C showing fracture of the carbide phases at

- a) 10Hz,
- b) 1Hz,
- c) 0.1Hz.

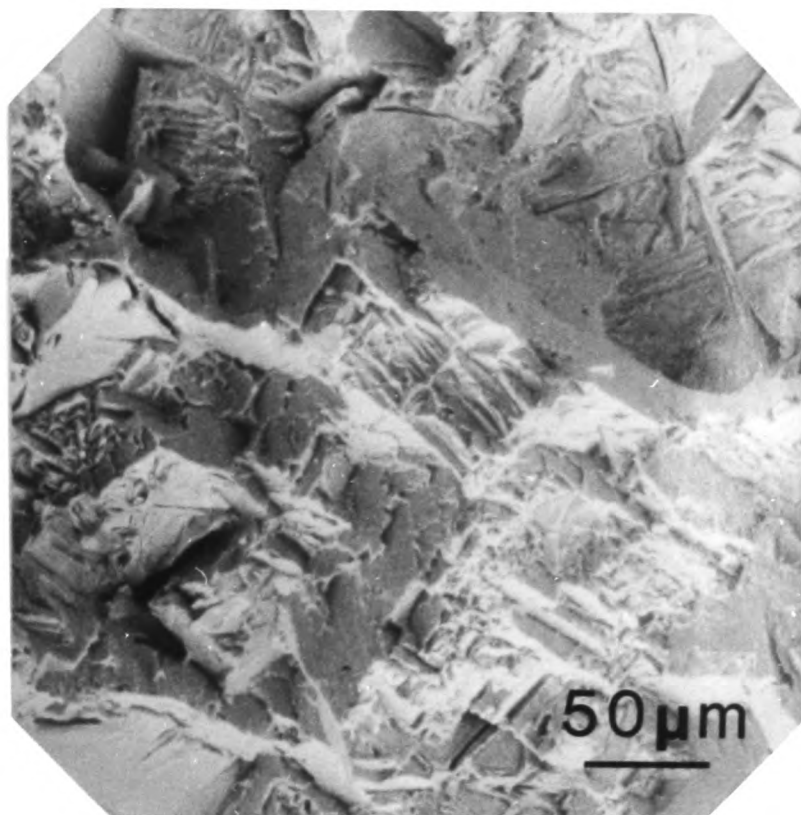


Figure 3.22

Fracture surfaces at 850°C in mid- ΔK regime for

a) 1Hz,

b) 0.1Hz.

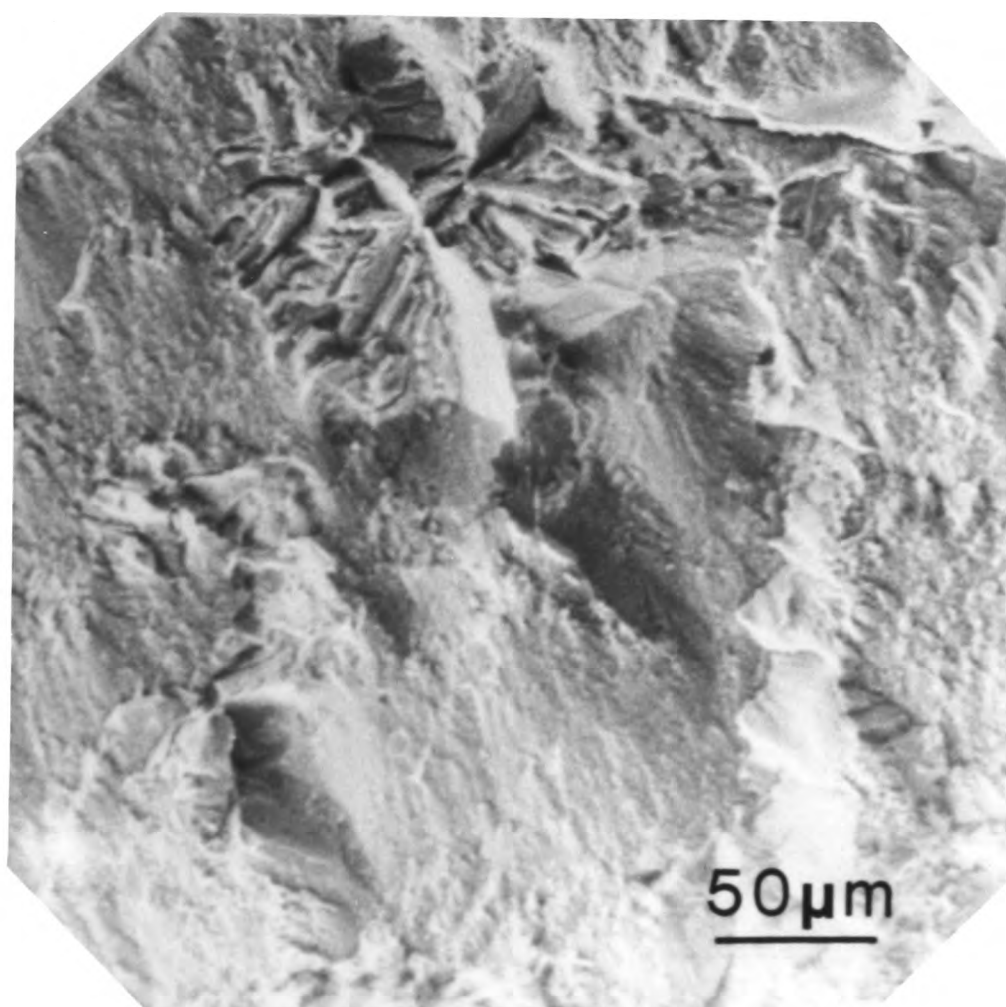
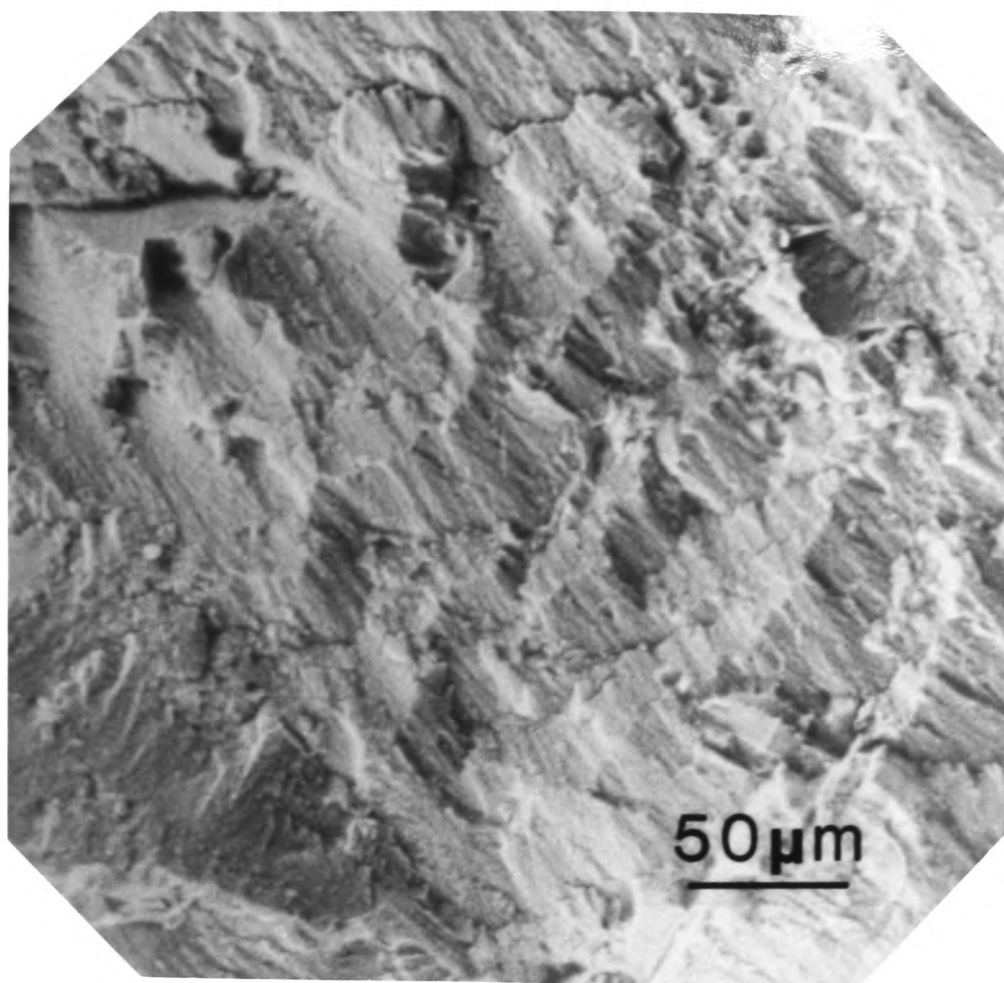


Figure 3.23

Fracture surfaces at 850°C and high $\Delta K'$ s,
approaching K_c for the testing conditions, showing
fracture of the carbides at

- a) 1Hz,
- b) 0.1Hz.

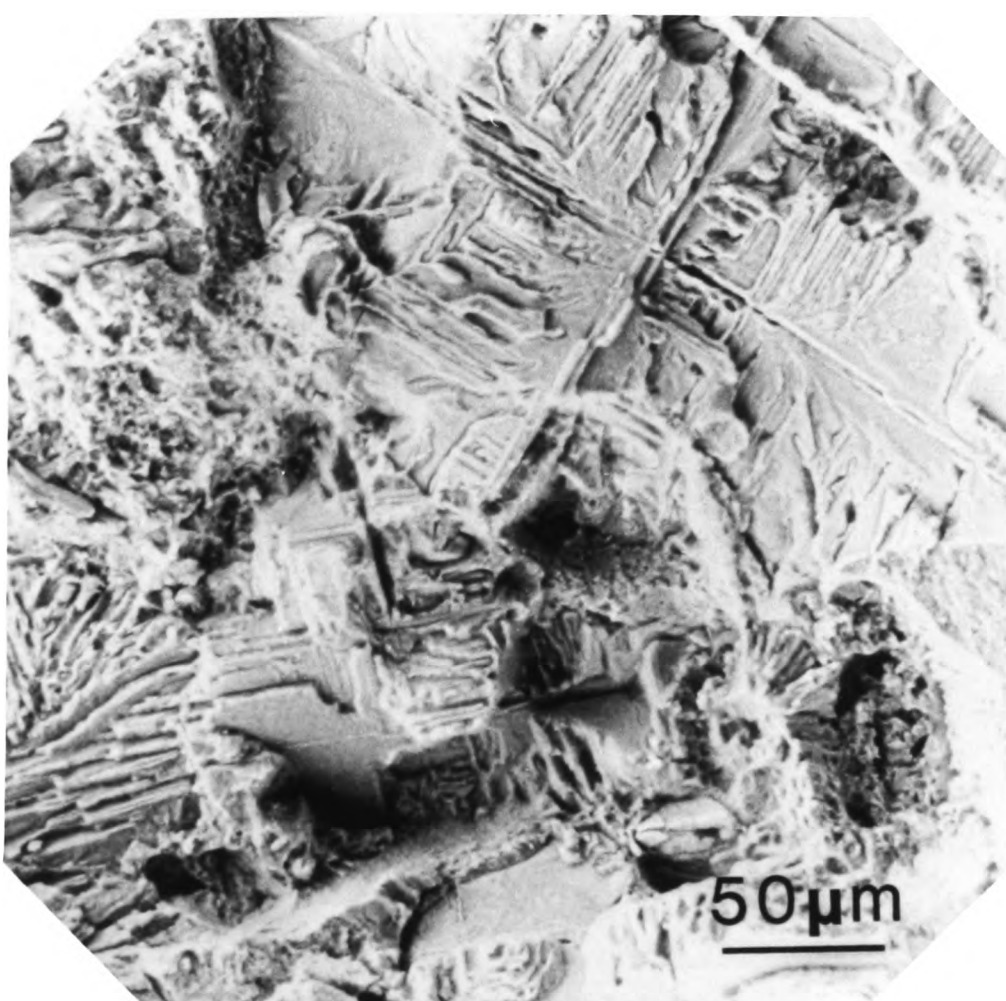


Figure 3.24

Fracture surfaces after creep crack growth at

- a) mid value of K ,
- b) high value of K approaching K_c .

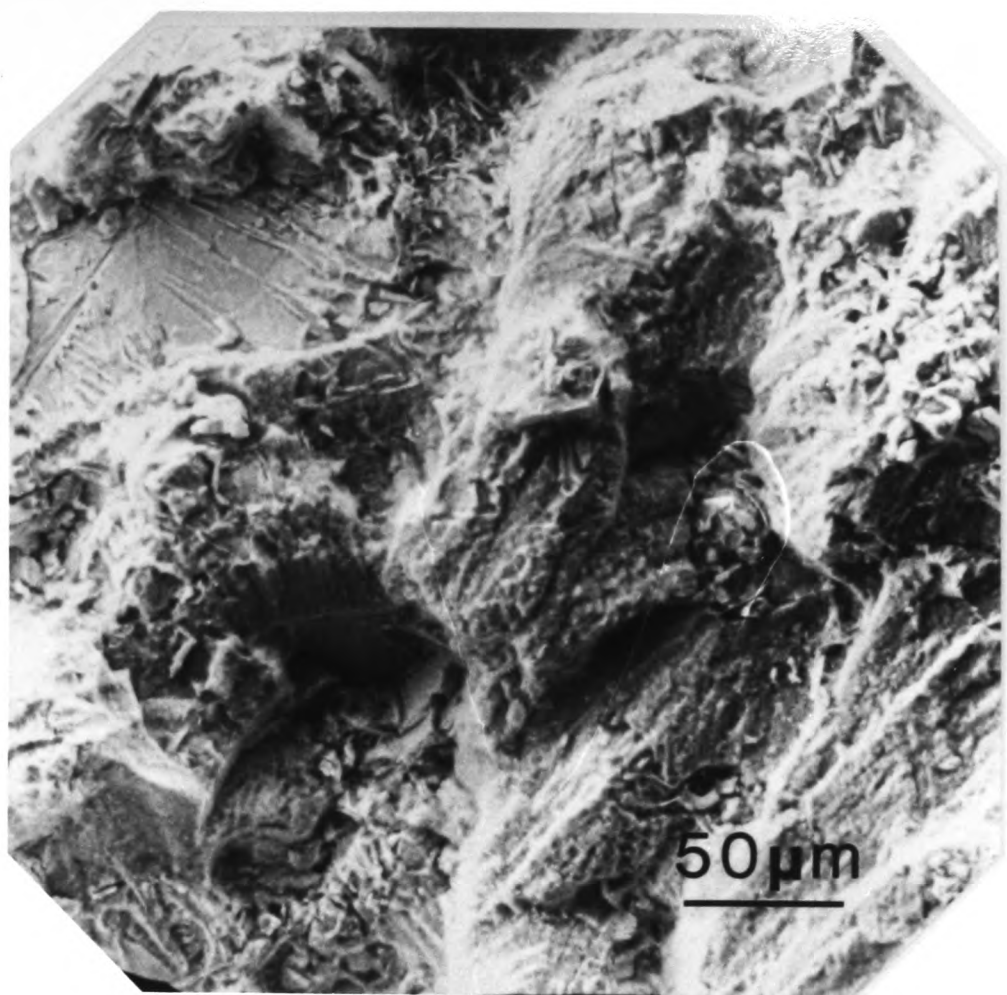


Figure 3.25

Effect of deformation on {100} selected area
channelling patterns with $\theta=\pm 10^\circ$, differentiated
backscattered electron signal.

- a) Unstrained material,
- b) True strain = 2.9%.

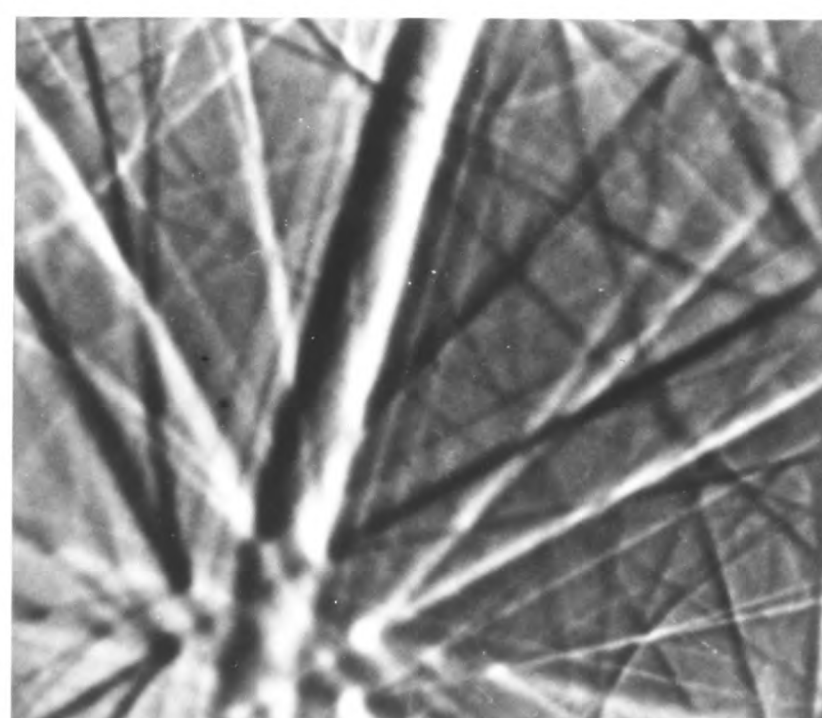
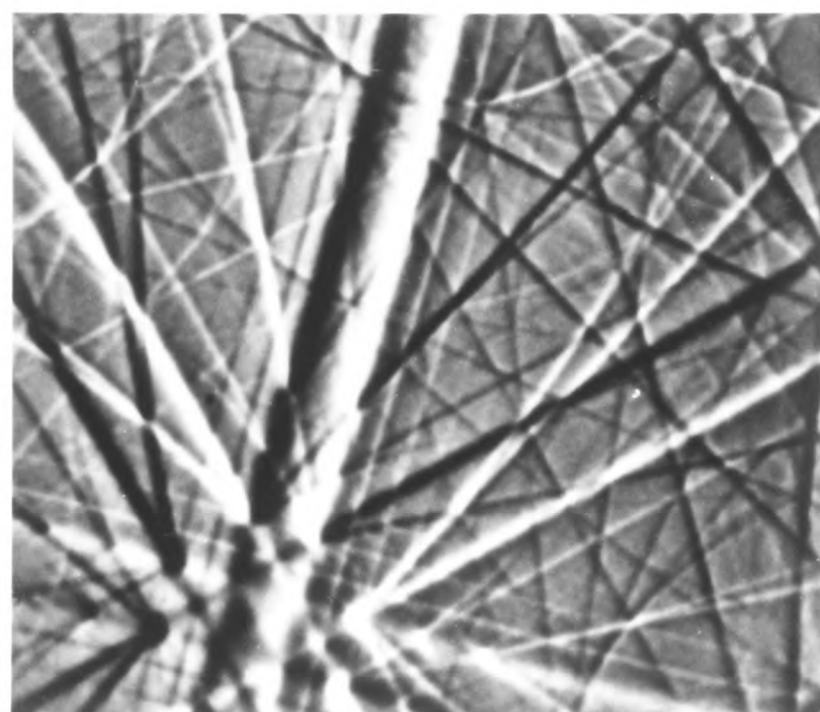


Figure 3.26

Variation of angular resolution of {200} and {220}
S.A.C.P. lines with strain for Mar M 002 single
crystals.

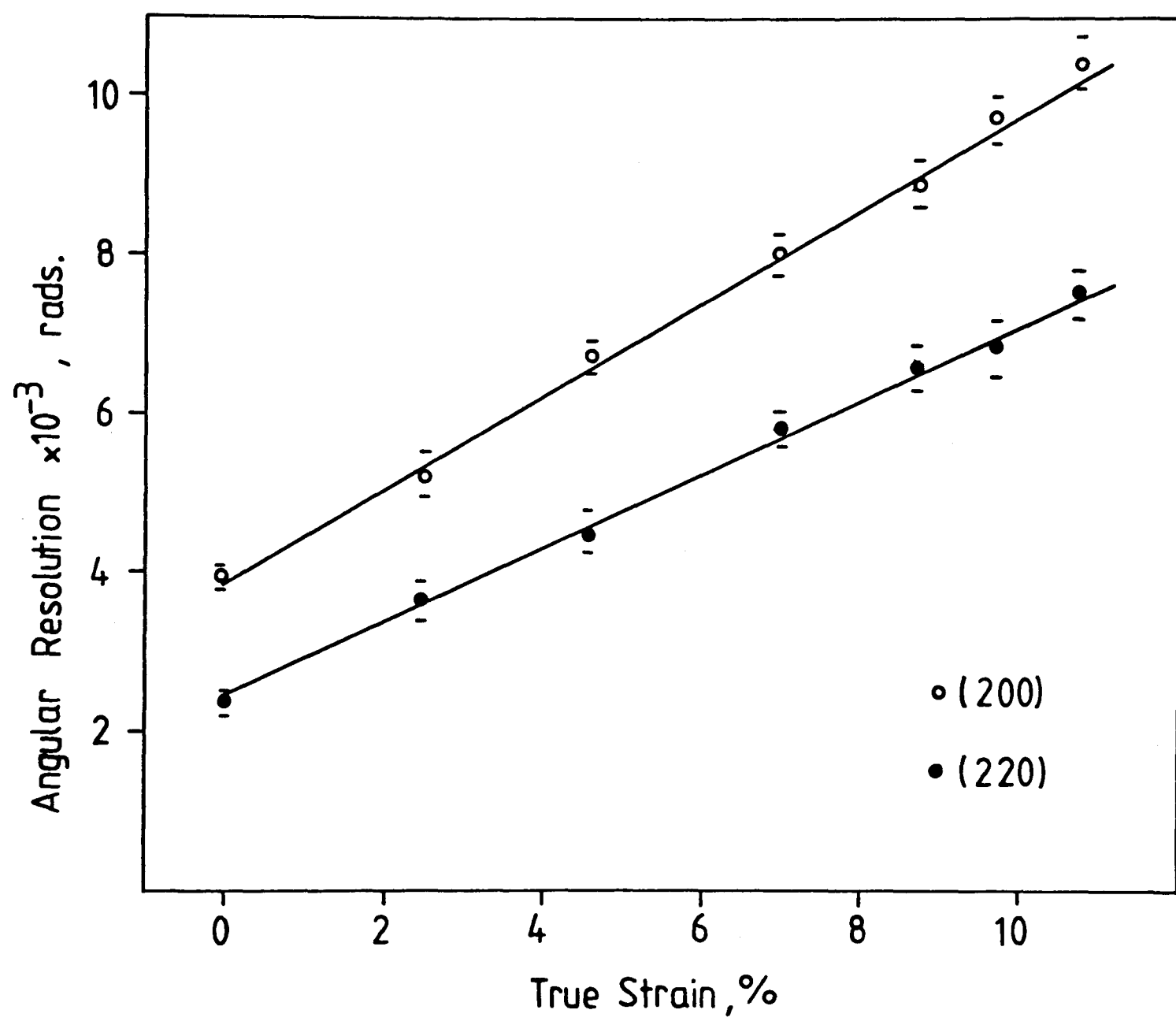


Figure 3.27

Plastic zone size at room temperature, 10Hz, $K_{\max}$
= 24MPa√m and R=0.1.

Figure 3.28

Plastic zone size at 600°C, 10Hz, $K_{\max}$ = 26MPa√m
and R=0.1.

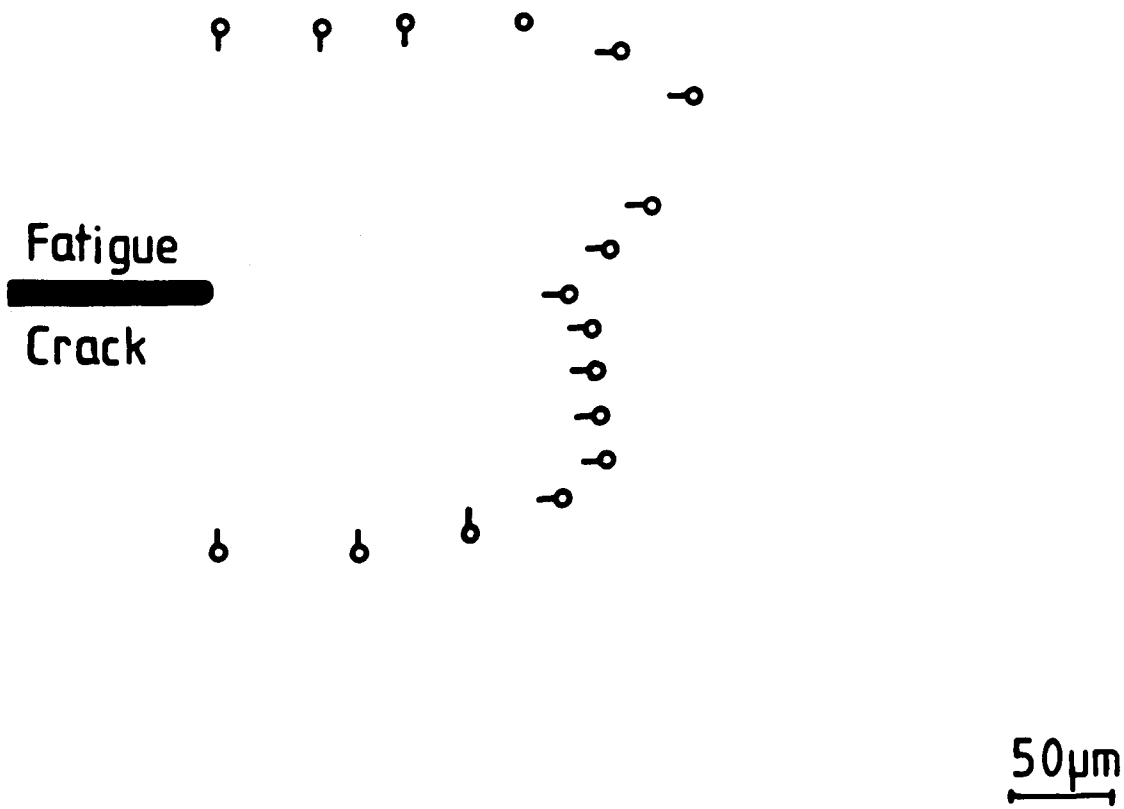
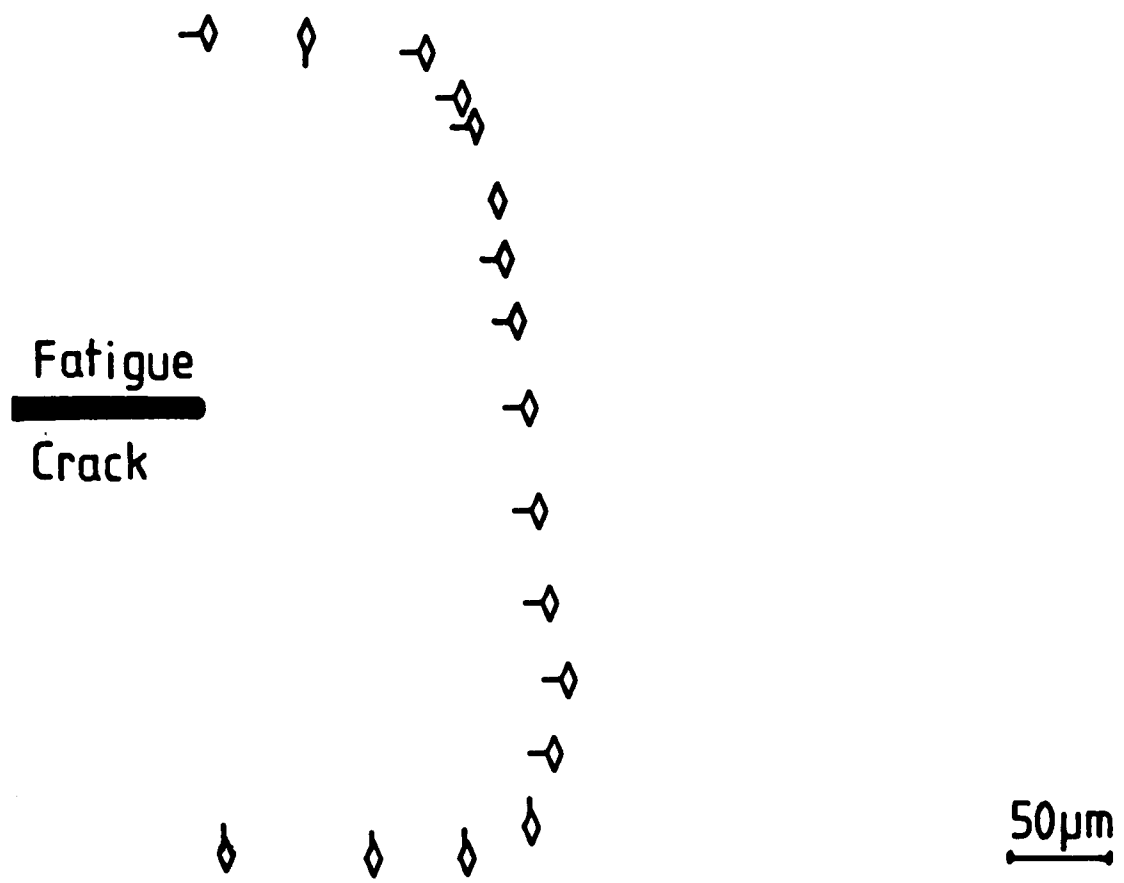


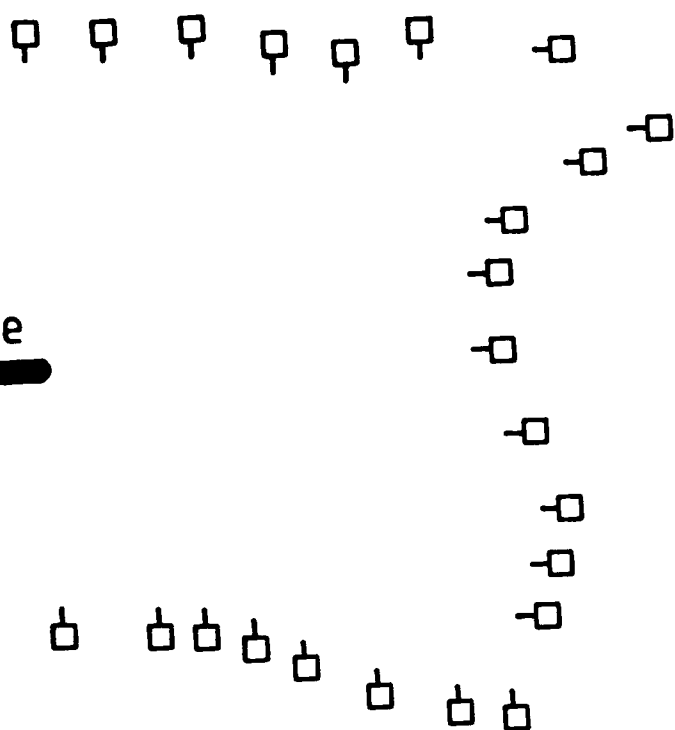
Figure 3.29

Plastic zone size at 600°C, 1Hz, $K_{\max} = 26\text{MPa}\sqrt{\text{m}}$
and $R=0.1$.

Figure 3.30

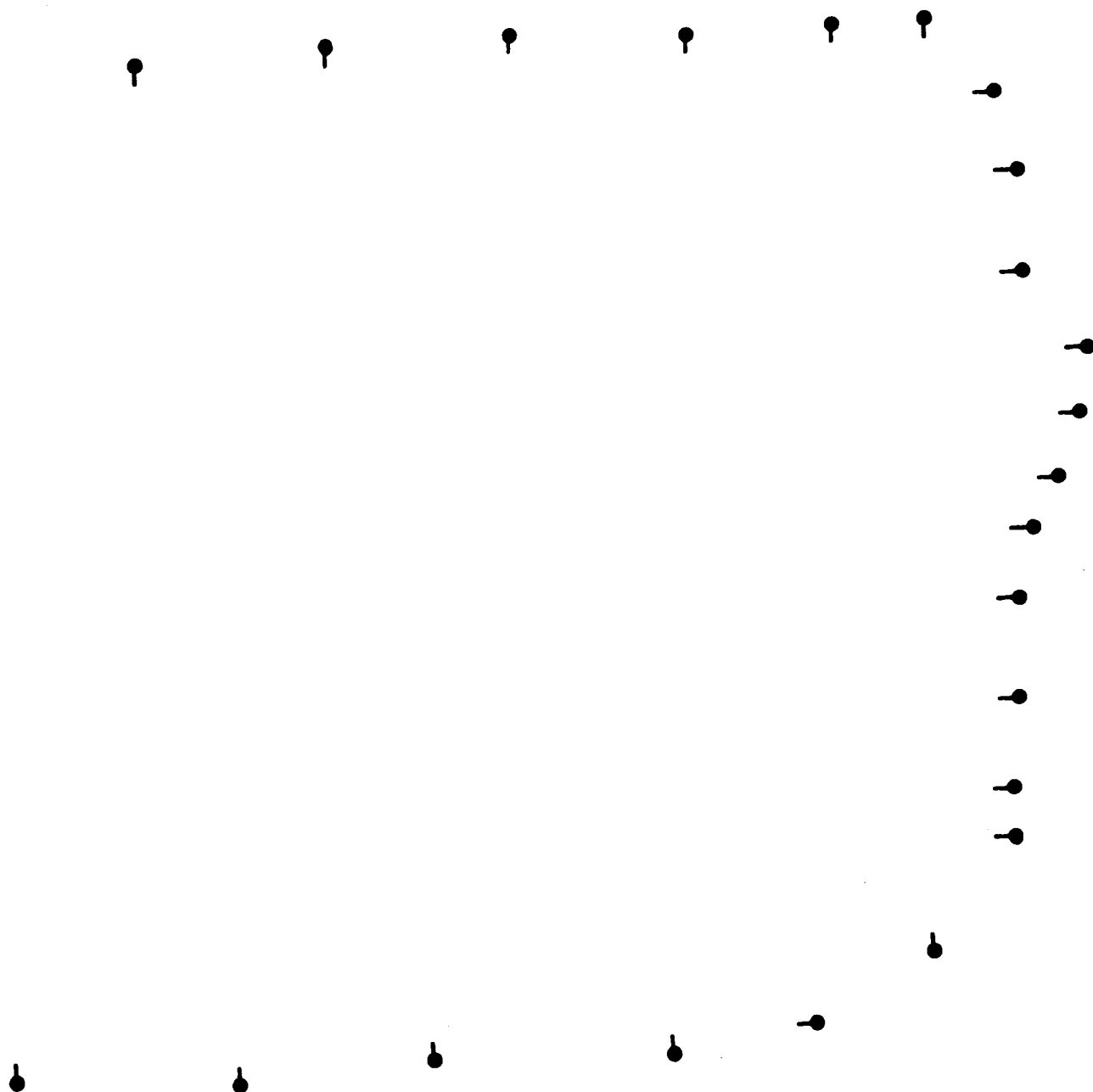
Plastic zone size at 600°C, 0.1Hz, $K_{\max} = 26\text{MPa}\sqrt{\text{m}}$
and $R=0.1$.

Fatigue
Crack



50µm

Fatigue
Crack



50µm

Figure 3.31

Plastic zone size at 600°C, 0.1Hz, $K_{\max} = 50\text{MPa}\sqrt{\text{m}}$
and $R=0.1$.

Fatigue
Crack

100 μm

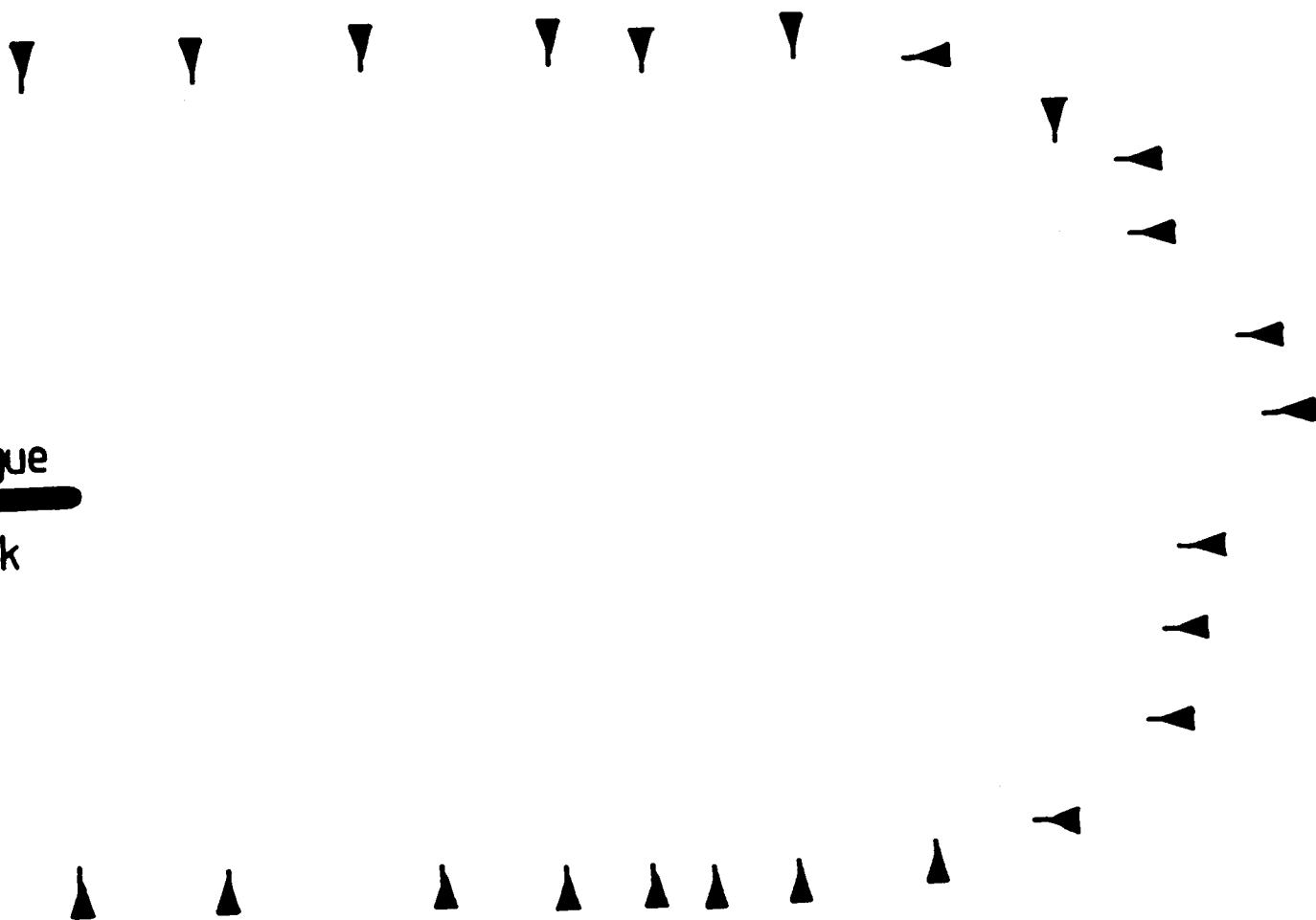


Figure 3.32

Plastic zone sizes at 850°C, $K_{\max} = 34\text{MPa}\sqrt{\text{m}}$, $R=0.1$
and $\nu = 1\text{Hz}$ and 0.1Hz .

Fatigue

Crack

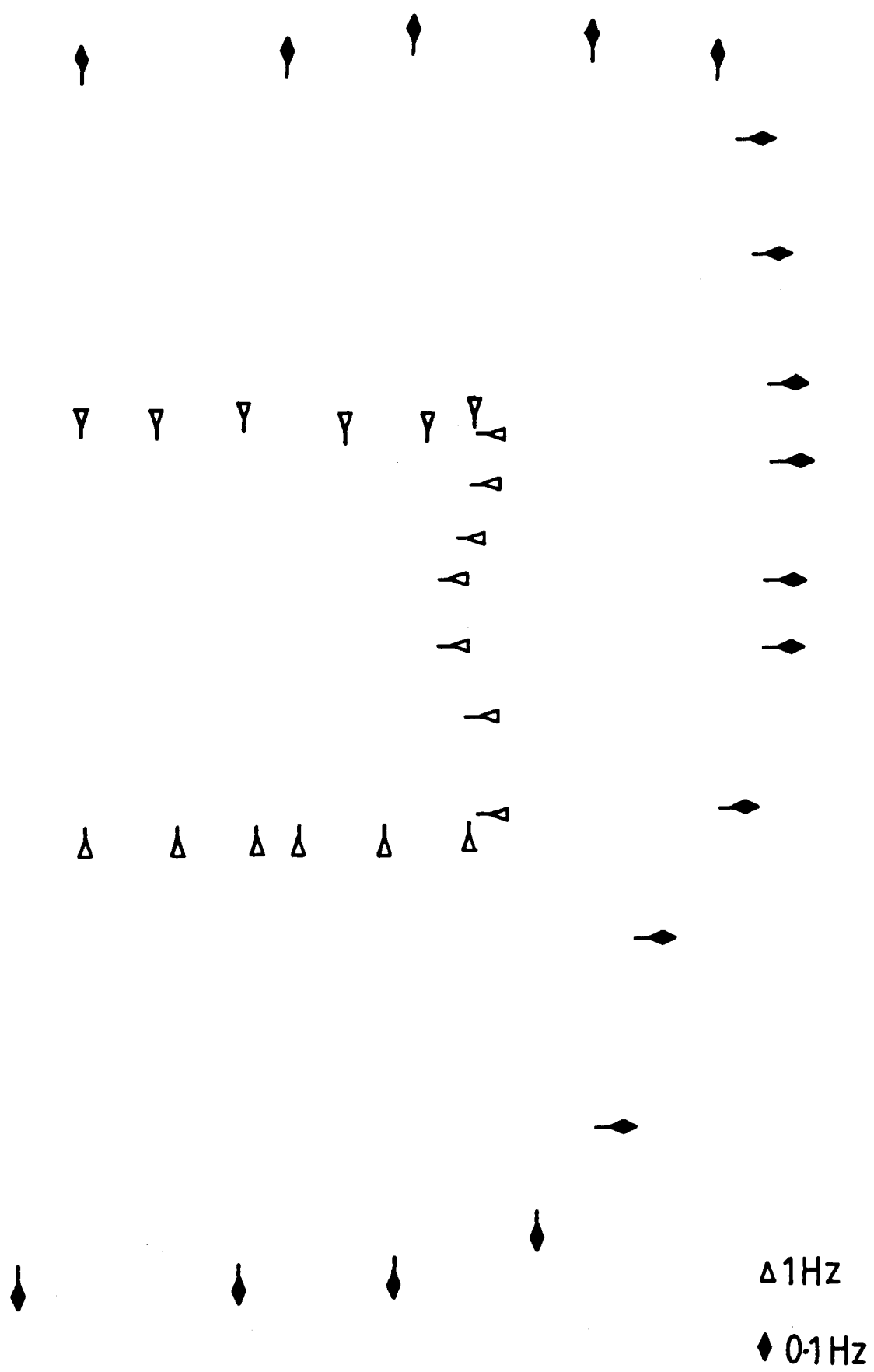
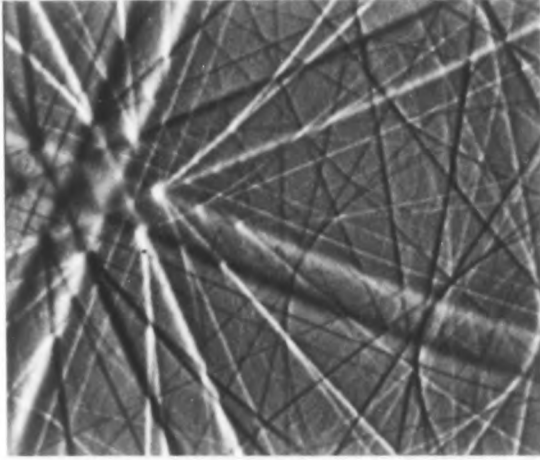
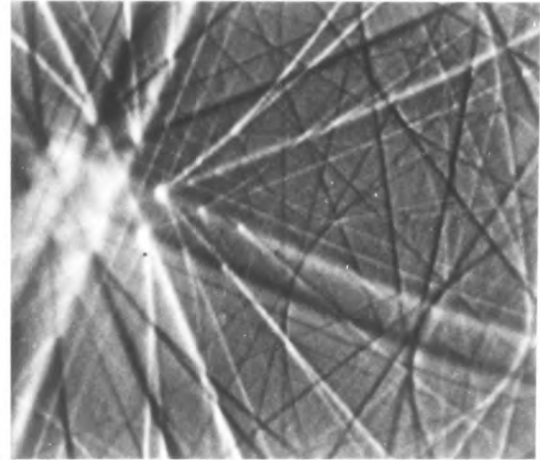


Figure 3.33

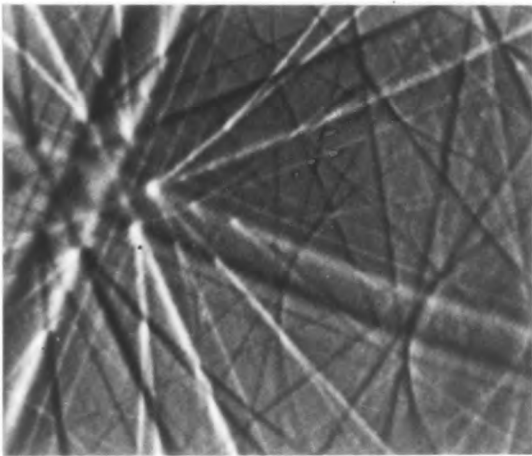
Selected area channelling patterns at various distances from the crack tip, in the plastic zone developed at 600°C and 1Hz. Measured plastic zone size = 223 μ m.



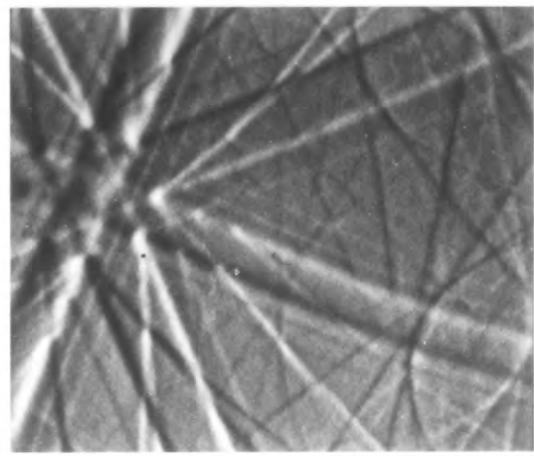
$d_{pz} = 297 \mu\text{m}$



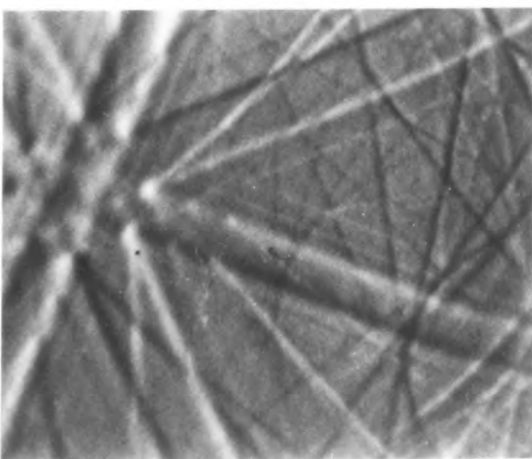
$227 \mu\text{m}$



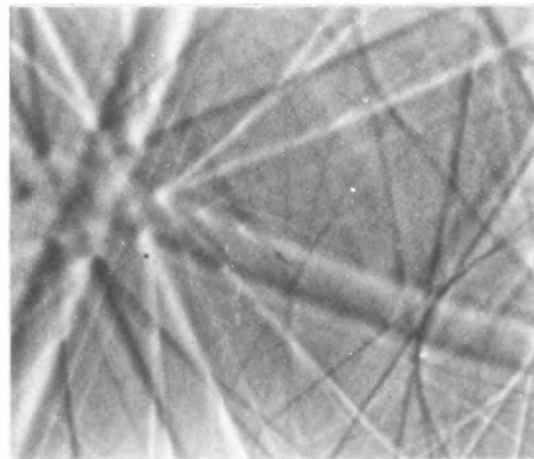
$207 \mu\text{m}$



$187 \mu\text{m}$



$147 \mu\text{m}$



$87 \mu\text{m}$

Figure 3.34

Variation of strain within the plastic zone ahead of the crack tip at room temperature and three frequencies at 600°C.

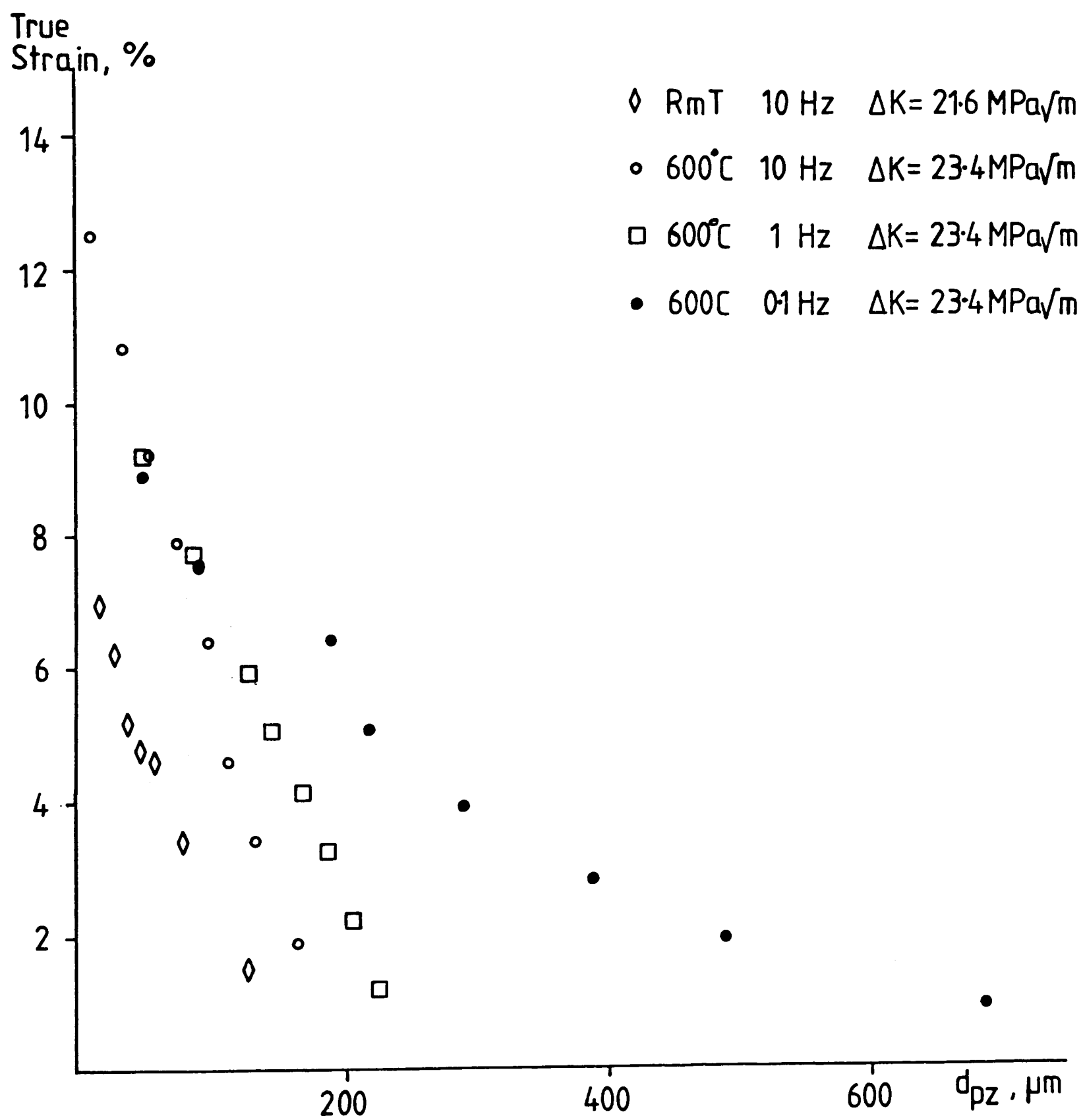
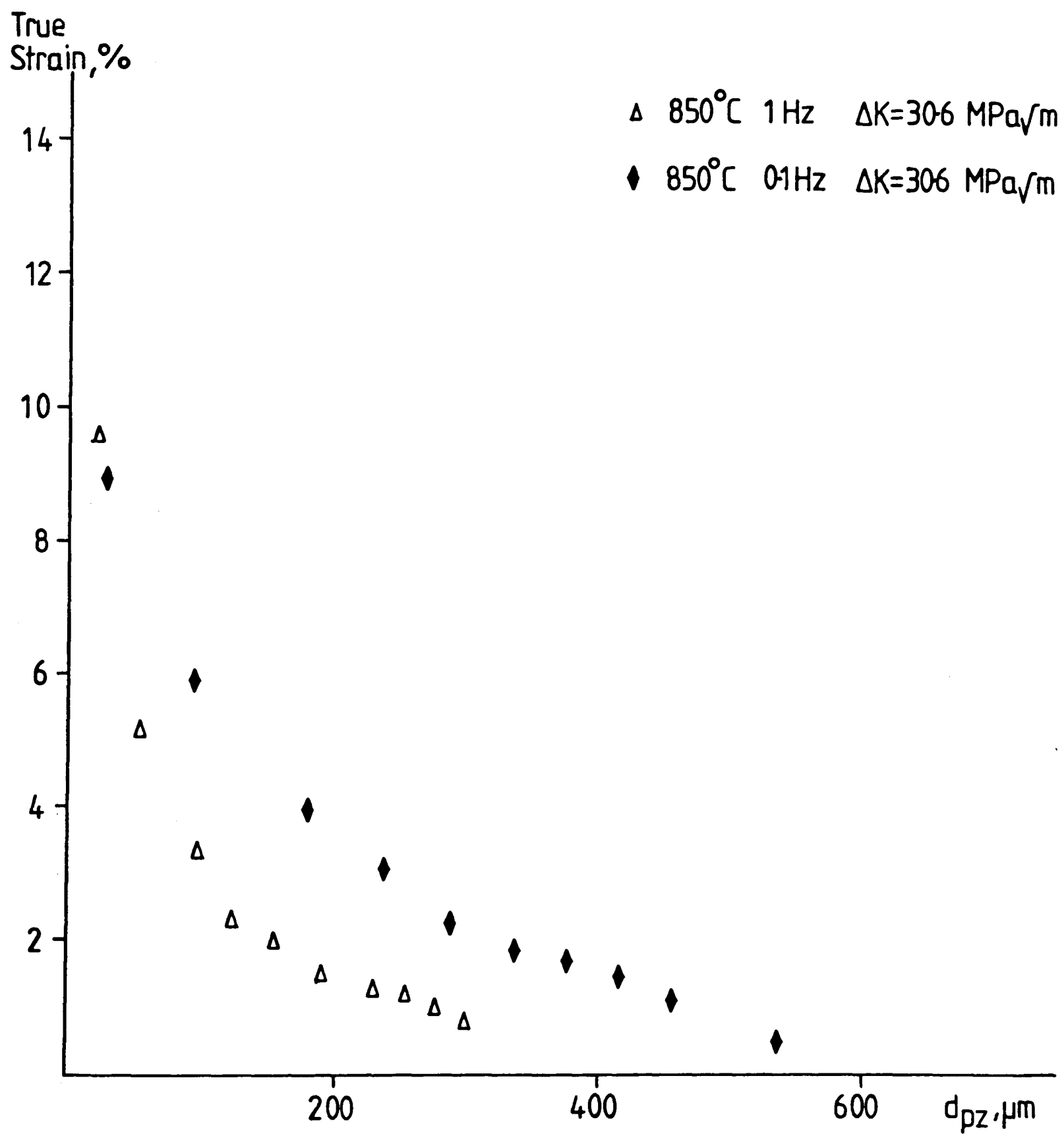


Figure 3.35

Variation of strain within the plastic zone ahead of the crack tip at 850°C and frequencies of 1Hz and 0.1Hz.



Chapter 4

Discussion

The initial discussion will consider the effects of temperature and frequency on the observed plastic zone size associated with a propagating crack, the crack growth behaviour will then be discussed. It is convenient to discuss the latter in terms of the three regimes of behaviour commonly observed; first the low growth rate regime, where threshold behaviour may be encountered. Secondly, high crack growth rates, where the fracture toughness is found to depend on the temperature and frequency and finally the intermediate crack growth regime is discussed and models for the fracture behaviour and frequency dependence of the crack growth rate are presented.

4.1 Plastic Zone Size

The measurement of the plastic zone sizes (figs. 3.27 to 3.32) and the summary in Table 3.3 indicate that the measured plastic zone size is a complex function of the temperature, frequency and applied stress. Linear elastic fracture mechanics considerations have shown that the monotonic plastic zone size, r_p , may be related to the applied stress intensity by equation (3.1). Use of the temperature dependent yield stress in conjunction with the constant of proportionality evaluated from the room temperature plastic zone size, allows the plastic zone sizes at elevated temperature to be calculated, these values are included in Table 3.3.

At 600°C and 10Hz the measured and calculated values are close, at lower frequencies and higher temperatures departures from this behaviour occur. It is pertinent to consider the development of the plastic zone during loading, and initially this will be carried out for material deforming in an elastic/plastic manner.

The response of the material to cyclic loading may be estimated using a superposition argument (Rice, 1967). On loading a monotonic plastic zone will develop when the crack tip stresses exceed the material's yield stress (fig. 4.1a). With continued loading the size of the plastic zone increases until the maximum load is reached and the maximum monotonic plastic zone size, r_2 , is obtained (fig. 4.1b). The size of the plastic zone will therefore be effectively determined by the monotonic yield stress.

On unloading (fig. 4.1c) a cyclic plastic zone of size r_3 will be developed together with the monotonic plastic zone. At the minimum load the cyclic plastic zone reaches its maximum size r_4 (fig. 4.1d). On reloading the sequence is reversed at a rate determined by the frequency of cycling. Therefore, for an elastic/plastic material regions exist in which tensile and reversed loading occur, the latter being restricted to areas close to the crack tip.

At low homologous temperature plastic deformation occurs by dislocation glide. However, as the temperature increases additional deformation mechanisms become possible, for example dislocation climb may occur at rates determined by such parameters as the temperature, stress and local diffusion rates. Under these conditions material flow is temperature and time dependent and the steady state strain rate, $\dot{\epsilon}$, may be described by the power-law relation

$$\dot{\epsilon} = B\sigma^n \quad (4.1)$$

where B is a temperature dependent constant, σ the applied stress and n the power-law creep exponent. Deformation of material at the crack tip by time dependent processes may lead to modifications in the development of a plastic zone.

On initial loading the crack tip elastic stress distribution may be relaxed by creep deformation. Initially creep strains develop rapidly near the crack tip and with increasing time creep redistributes the stresses across the specimen. As creep proceeds strain accumulates and a plastic zone may be described which increases with time. An estimate of the plastic zone size may be made by considering relaxation of the crack tip elastic stress distribution by creep occurring according to equation (4.1).

The elastic normal stress distribution ahead of a crack has been given by linear elastic fracture mechanics (section 1.2.1) as

$$\sigma = \frac{K \cdot f(\theta)}{(2\pi r)^{1/2}} \quad (4.2)$$

where σ is the local elastic stress at a distance r from the crack tip at a stress intensity K and $f(\theta)$ is an angular function. The elastic strain distribution is therefore

$$\epsilon_{el} = \frac{K \cdot f(\theta)}{E(2\pi r)^{1/2}} \quad (4.3)$$

where E is Young's modulus.

If it is assumed that creep relaxes the crack tip stresses at a rate given by equation (4.1), the creep strain ahead of the crack tip,

ϵ_{cr} , after a time t may be obtained from equations (4.1) and (4.2) as

$$\epsilon_{cr} = \int_0^t \frac{BK^n}{(2\pi r)^{n/2}} dt \cdot f(\theta) \quad (4.4)$$

Thus giving

$$\epsilon_{cr} = \frac{BK^n}{(2\pi r)^{n/2}} t \cdot f(\theta) \quad (4.5)$$

A plastic zone in which the strain accumulated by creep exceeds the elastic strain may then be defined. At the boundary of the plastic zone the elastic strain is equal to the creep strain, by equating equations (4.3) and (4.5) the creep zone boundary, r_{cr} , may thus be obtained as

$$r_{cr} = \frac{K^2}{2\pi} (EBt)^{2/(n-1)} \cdot f(\theta) \quad (4.6)$$

A comparison of the quantitative predictions of equation (4.6) with measured plastic zone sizes is difficult in view of the complex stress distribution associated with a crack. The stresses attendant at a crack tip are dependent on the distance ahead of the crack tip and also the state of stress, that is plane stress or plane strain. Consequently the creep behaviour and rate parameters in equation (4.1) may vary from those associated with uniaxial tensile creep tests. In addition, precise solutions require knowledge of the rate at which creep takes place and would therefore have to consider both primary and secondary creep behaviour. However, a qualitative interpretation of equation (4.6) can be made. The plastic zone size will be dependent

on the applied stress (through K), the time for which the stress acts and the temperature (through the constant B and the creep exponent n of equation (4.1)). An increase in any of these parameters will give rise to an increase in the plastic zone size.

If we consider cyclic loading of the material it can be appreciated from fig. 4.1 that during loading the possibility of relaxation of the elastic stress distribution by creep arises. In this situation the frequency of cycling will be important as it determines the time for which the stress is applied. The time of loading in equation (4.6) then becomes related to the period of the loading waveform.

Therefore, at a constant temperature and stress intensity the application of high cycling frequencies will give rise to rapid reversal of the crack tip stresses and the period of the loading waveform is short. Consequently the effect of creep deformation is limited since the time in equation (4.6) is small, the plastic zone size is then determined by the monotonic yield stress. If the frequency of cycling decreases, the period of the waveform increases and the time in equation (4.6) may become significant. Creep relaxation may then occur and departures from the plastic zone size based on the monotonic yield stress arise. With continuing decreases in the frequency creep may become more significant and increased plastic zone sizes result.

This behaviour is clearly shown in the measured plastic zone sizes after elevated temperature cycling. At 600°C and 10Hz creep deformation is limited because of the high frequency and the plastic zone size is effectively that determined by the monotonic yield stress. With decreasing frequency at 600°C, increasing effects of

creep relaxation occur and an increased plastic zone size results.

Similar behaviour is observed at 850°C where decreases in the frequency from 1Hz to 0.1Hz give rise to larger plastic zone sizes. However, comparison of the plastic zone sizes at 850°C with those at 600°C shows that at 0.1Hz a decrease in the plastic zone size accompanies the increase in temperature despite the higher applied stress intensity.

The higher creep rates obtained at 850°C may, in addition to affecting the plastic zone size, influence the crack tip geometry. If creep relaxation during loading leads to blunting of the crack tip the effective stress concentration of the crack will be reduced and the stress distribution affected (Rice and Johnson, 1970). In the extreme the stress distribution may approach that associated with a notch having a finite radius of curvature.

Kim et al. (1979) have considered the effect of a finite crack tip radius of curvature on the plastic zone size. They have shown that the actual plastic zone size of a blunted crack, r_p , may be given by the plastic zone of an imaginary sharp crack, r_k , minus the geometric effect of blunting, this leads to

$$r_p = r_k - r_\rho \quad (4.7)$$

This effect is shown schematically in fig. (4.2). At a constant stress intensity, r_k remains the same but, as the radius of curvature of the crack tip r_ρ increases the effective plastic zone size r_p decreases. Therefore at 850°C blunting of the crack tip by increased creep relaxation may limit the spread of plasticity. This is clearly manifested by the observation that the plastic zone sizes at 850°C are not simply related to those at 600°C and by the restricted increase in

size on decreasing the frequency at 850°C.

The effect of the stress intensity factor on the plastic zone sizes at 600°C and 0.1Hz may be seen by comparing the values at stress intensities of 26MPa/m and 50MPa/m. The observed value at the higher stress intensity is not simply related to the value at the lower stress intensity by equation (3.1). At $K_{\max}=50\text{MPa}/\text{m}$ the observed plastic zone size is approximately half that expected from the value observed at $K_{\max}=26\text{MPa}/\text{m}$. At the higher stress intensity the material's fracture toughness is approached and it is likely that different mechanisms of failure occur within the plastic zone. If, for example, material separation ahead of the crack tip occurs substantial stress relaxation may accompany the process (Raj and Baik, 1980). This would alter the crack tip stress distribution thus reducing the size of the crack tip plastic zone.

At elevated temperatures it is therefore suggested that crack tip relaxation processes occur which may have differing results. At intermediate levels of stress intensity where fracture is limited to regions close to the crack tip these may be

- (i) relaxation of crack tip stresses giving an increased plastic zone size with decreasing frequency at a constant temperature and stress intensity.
- (ii) As creep rates increase, for example by increasing the temperature, blunting of the crack tip may result. This will affect the crack tip stress concentration and thereby limit the extent of the crack tip plastic zone.

At values of stress intensity approaching the fracture toughness, failure of material within the plastic zone may give considerable stress relaxation. The plastic zone size will then be lower than the

values extrapolated from the measured plastic zone sizes at lower stress intensities would suggest.

4.2 Crack Propagation at Low Growth Rates

The behaviour of the material at low growth rates for different temperatures, frequencies and stress ratios is shown in fig. 4.3. The crack growth rates measured here are not threshold values but changes in the gradient of the da/dN versus ΔK curves may be observed at lower values of ΔK .

At room temperature (fig. 4.3a) the value of R is observed to have little effect and no significant change in the gradient of the curve is obtained at low values of ΔK . The latter observation is similar to that reported by Purushothamen and Tien (1978) for single crystals of Mar M 200, but contrasts with the rapid decrease in da/dN observed on decreasing ΔK in polycrystalline specimens.

At elevated temperature (figs. 4.3b and c) changes in the gradient at low growth rates may be seen. At a constant temperature, increases in the frequency and stress ratio lead to retardation of growth at lower values of ΔK . At a constant frequency, higher growth rates are associated with higher values of R and lower temperatures.

The mechanisms by which crack growth occurs at elevated temperature should consider the effects of material failure at the crack tip and possible environmental influences. In general at low growth rates environmental interactions may lead to embrittlement of the material, blunting of the crack tip or closure of the crack during part of the loading cycle.

We will first consider the possible effects of embrittlement as

proposed by McMahon and Coffin (1970). Embrittlement of material ahead of the crack tip is effectively determined by the rate at which diffusion of the embrittling species can take place. Since diffusion occurs more rapidly at higher temperatures increased embrittlement may be expected. Similarly, the diffusion distance is dependent on time so that if the time of application of the loading cycle is increased, that is the frequency is decreased, an increased diffusion distance and therefore increased embrittlement may be expected. This may be particularly noticeable at low growth rates where the rate of advance of the crack tip is slow so that if critical concentrations of species are required they are more likely to be attained. In either case the increased embrittlement would be expected to lead to

- a) lower values of threshold ΔK for lower frequencies at a constant temperature, and
- b) lower values of threshold ΔK for higher temperatures at a constant frequency.

Clearly this is not observed in the present work where the trend in behaviour is the direct opposite of that expected from embrittlement arguments. Further, the levels of strain (figs. 3.34 and 3.35) within the plastic zone show no evidence of decreasing as they approach the crack tip. Instead, they continue to rise towards the crack tip.

In a similar manner, Scarlin (1978) has considered that the increased diffusion at elevated temperature may give rise to the formation of a precipitate-denuded zone close to the crack tip. Although no evidence of denuded zones at the crack tip was observed in the present work, even the small γ' precipitates were observed to be present close to the fracture plane, it is interesting to consider

what the effect of such a region might be.

Clearly the mechanical properties of the material at the crack tip will be altered by removal of the strengthening precipitate, and yet if the denuded region is extensive it may only lead to blunting of the crack tip. Alternatively, if the required reactions can occur rapidly enough to determine the crack growth rate and thus give rise to lower crack tip failure strains, lower thresholds would again be associated with increased temperatures and decreased frequencies.

Blunting of the crack tip may result from deformation at low frequencies and high temperatures and would give rise to the trend of threshold behaviour observed here. However, as we have seen the plastic zone size at 600°C and 10Hz may be adequately predicted from the room temperature data. This implies that the distribution of crack tip stresses is similar in the two cases and that therefore the crack tip geometries are similar. If blunting of the crack tip was responsible for the threshold behaviour similar thresholds may then be expected at room temperature and at 600°C and 10Hz. However, the 600°C 10Hz data reveal deviations from the room temperature data at low values of ΔK . Similarly, increasing the R ratio may be expected to increase crack tip blunting so that the behaviour would then be the opposite of the observed effect of the R ratio.

Alternative models for growth in the threshold regime consider that microstructurally sensitive closure reduces the effective cyclic stress intensity, ΔK_{eff} , according to

$$\Delta K_{eff} = K_{max} - K_{op} \quad (4.8)$$

where K_{op} is the lowest stress intensity at which the crack is open.

Crack closure has been considered to arise from crack tip

plasticity (Elber, 1970), from fracture surface roughness (Ritchie, 1982) or from oxidation products on the crack surface (Haigh, 1975; Ritchie et al., 1980). Plasticity-induced closure arises from the effects of plastic flow in the wake of the crack tip. During cyclic loading both a cyclic and a monotonic plastic zone are developed (Rice, 1967) and since the monotonic plastic zone is larger than the cyclic plastic zone it has been considered to determine the behaviour of material in the wake of the crack tip. Monotonic loading produces deformation perpendicular to the crack plane thus, on unloading, material behind the crack tip may touch and closure of the crack tip results. Material having a larger plastic zone will generally show more closure since a larger volume of material is deformed leading to larger offsets at the crack face.

Whilst plasticity-induced closure may provide an explanation in terms of the increased thresholds observed on decreasing the frequency, it would require that under conditions of similar plastic zone size comparable threshold values may be obtained. However, the plastic zone sizes at 850°C and 600°C at 1Hz and 0.1Hz are similar yet significantly higher thresholds are observed at 850°C. Also the room temperature and 600°C 10Hz crack growth data are different and yet may be expected to have similar plastic zone sizes (section 4.1).

Haigh (1975) suggested that the increased volume associated with oxidation products behind the crack tip may result in crack tip closure. More recently, Suresh et al. (1981) have observed that at low crack tip displacements the oxide thicknesses on fracture surfaces may be of the order of the cyclic crack tip opening displacement. The increased oxide thickness arises from the repeated breaking and compacting of the surface under these conditions.

At elevated temperature oxidation will be more rapid and, for a constant temperature, increased oxide thicknesses may be obtained on decreasing the frequency; at a constant frequency increases in temperature will similarly increase the oxide thickness. To a first approximation closure of the crack will result when the oxide thickness is greater than the minimum crack tip opening displacement. On either increasing the temperature or decreasing the frequency crack tip closure will then occur at higher values of K_{Op} , the value of ΔK_{eff} is therefore lower and crack retardation occurs at higher values of the applied ΔK . At a constant temperature, decreasing the frequency will therefore increase the observed threshold ΔK , whereas at a constant frequency higher thresholds will result at higher temperatures.

Increasing the value of R has the effect of increasing the proportion of the cycle over which the crack is open. Consequently closure is limited and growth continues to lower values of the applied ΔK . The features outlined above are shown in the results of the present work. It is therefore suggested that the major effect at low growth rates arises from oxidation induced crack tip closure retarding the crack growth and giving rise to higher threshold values at higher temperatures and lower frequencies.

4.3 Crack Growth at High Growth Rates

At high growth rates the material's response is observed to be dependent on the frequency of cycling and the temperature (figs. 3.7 to 3.11). The growth rates are seen to be higher at higher frequencies and, at 600°C, the estimated K_c is observed to be frequency dependent

(Table 3.3).

This result may, initially, be explained in terms of the increased plastic zone size with decreasing frequency. For a brittle material, fracture at the crack tip will occur when the elastic energy released on crack extension is sufficient to provide the surface energy of the fractured material (Griffiths, 1920). Crack extension will then be characterised by a critical potential energy release rate, G_c , which is equal to the surface energy of the fractured material produced by crack extension, 2γ . However, if plastic flow occurs the energy required will be increased by an amount representing the plastic work at the crack tip, γ_p (Orowan, 1945). Failure will then occur when

$$G_c = 2\gamma + \gamma_p \quad (4.9)$$

The value of G can be related to the stress intensity factor, K , by

$$G = \frac{\alpha K^2}{E} \quad (4.10)$$

where $\alpha=1$ for plane stress and $(1-\nu^2)$ for plane strain (Knott, 1973) so that equation (4.9) gives

$$K_c = \sqrt{\frac{E (2\gamma + \gamma_p)}{\alpha}} \quad (4.11)$$

The dissipation of energy at the crack tip will occur mainly in the form of the strain energy of dislocations in the plastic zone. Increasing the plastic zone size, for example by decreasing the frequency at elevated temperature, gives larger values of γ_p and correspondingly higher values of K_c .

Whilst this explains the observed behaviour in the present case, it is not consistent with the microstructural fracture characteristics. At high values of ΔK the fracture surfaces show extensive fracture of the brittle carbides (figs. 3.21 and 3.23). In addition, the plastic zone at 600°C, 0.1Hz and $K_{\max}=50\text{MPa}/\text{m}$ suggests that alternative stress relaxation mechanisms, such as material separation, are occurring. Examination of the etched longitudinal section of the crack tip at high ΔK (fig. 4.4) shows that fracture of large carbides occurs ahead of the crack tip under these conditions. This process will be accompanied by considerable stress relaxation due to the material separation and explains the reduced plastic zone size noted in section 4.1. At high values of ΔK it would therefore appear that fracture of the carbides directly ahead of the crack tip followed by subsequent rupture of the intervening ligament is the determining fracture process.

If we now consider this behaviour in terms of the crack tip stresses, the fracture of carbides will be observed when the tensile stress ahead of the crack, σ_{yy} (see fig. 1.5), attains the fracture stress of the carbide σ_f . This will cause localised unstable crack advance and the observed value of K_c is given when this condition is exceeded.

The level of the local stress attained ahead of the crack tip, for a given applied stress, will be affected by the constraint imposed on the plastic flow of material. This will be indicated by the stress intensification Q ,

$$Q = \frac{\sigma_{11}(\text{max})}{\sigma_y} \quad (4.12)$$

where σ_y is the monotonic yield stress. Therefore high constraint (high values of Q) implies that high stresses occur at the crack tip. If the plastic zone at the crack tip is small, yielding in the thickness direction cannot take place due to the constraint of the surrounding elastic material and Q is high. If the plastic zone size is large, the through thickness stress σ_{zz} (see fig. 1.5) may be more easily relaxed, the constraint is lower and Q falls.

As we have seen (section 4.1), at a constant temperature, larger plastic zone sizes are obtained on decreasing the frequency and this behaviour may be explained in terms of the time-dependent relaxation of crack tip stresses. Therefore at 600°C and 10Hz, a higher degree of constraint is imposed than at the same temperature but lower frequencies. Thus, with decreasing frequency the constraint also decreases. Consequently at 600°C and 10Hz the criterion for fracture of the carbide, that is $\sigma_{yy} = \sigma_f$, will be achieved at lower applied stresses, and therefore lower stress intensities, than at 600°C and 1Hz. Similarly, the increased relaxation at 600°C and 0.1Hz will give rise to higher applied stresses to attain K_c than at 600°C and 1Hz.

At high stress intensities crystallographic fracture of material is also observed to take place in regions adjacent to the carbides. When fracture of the carbide takes place it is likely to occur rapidly and result in high strain rates at the tip of the fracturing carbide. Under these circumstances dislocation motion is most likely to be concentrated into inhomogeneous planar arrays on the $\{111\}$ slip planes (Gell and Leverant, 1973) resulting in crystallographic fracture. Anton (1983) has recently observed a similar phenomenon using impact loading at high temperatures and Jablonski and Sargent (1981) reported

final failure on the slip planes after crack propagation along {100}.

4.4 Crack Growth at Intermediate Growth Rates

At intermediate levels of ΔK the crack propagation rate shows both a temperature and frequency dependence. This is closely associated with a change in the fracture behaviour from crystallographic failure (figs. 3.14 to 3.16) at room temperature to stable crack growth along (001) at elevated temperatures (figs. 3.17 and 3.22). This is confirmed by the observation that the crack tip strains found after elevated temperature deformation are approximately twice those found at room temperature (figs. 3.34 and 3.35) and are independent of frequency.

4.4.1 Room Temperature Crack Growth

Some authors have considered the failure on {111} planes to be the result of a brittle cleavage mode of failure (Sadananda and Shahinian, 1979). The present work, however, indicates that the failure is associated with crack tip plasticity and in this respect is in accord with the recent results of Anton (1983) who noted the blurring of Laue spots from crystallographic facets.

The mechanism of crack growth is influenced by the crystallography at the crack tip (fig. 4.5). In the present system cracks were arranged to propagate primarily along [100](001) with a vertical stress axis of [001]. Under these conditions it can be seen that the {111} planes will be at 54.7° to the stress axis and subject to a high resolved shear stress. Dislocation glide will take place on {111} and shear the coherent γ' precipitate. A shear of $a/2 \langle 110 \rangle$

$\{111\}$ in the ordered γ' produces a high energy antiphase domain boundary (A.P.B.) in which like atoms are adjacent. Passage of a second $a/2 \langle 110 \rangle \{111\}$ dislocation removes the A.P.B. and restores order to the γ' . Hence dislocations in ordered phases are often observed to move in pairs separated by A.P.B. and are termed superlattice dislocations (Flinn, 1960).

Differences in the stacking fault energies of the precipitate and matrix may result in variations in this behaviour. The various possibilities by which shear of the γ' may then occur have been considered by Kear et al. (1970) and essentially involve shear of the precipitate by $a/6 \langle 211 \rangle \{111\}$ dislocations. This may necessitate the generation of interfacial dislocation loops and the partial dislocations may then be separated by mixtures of A.P.B. and stacking fault.

The shearing of the γ' will result in local work softening of the slip plane (Hornbogen and Zum Gahr, 1975) and the stress required to glide a second superlattice dislocation along the slip plane will be reduced. Further dislocation motion therefore becomes concentrated along specific slip planes and the resulting inhomogeneous deformation has been observed to produce large slip offsets at the specimen surface (Gell et al., 1973).

Examination of an etched section taken from a test interrupted prior to failure (fig. 4.6) shows the local crack propagation along $\langle 110 \rangle \{111\}$. The local crack extension on $\{111\}$ will not be sensitive to changes in the chosen macroscopic crack propagation direction. For example, if a macroscopic crack were oriented to propagate along $[110]$ (001), the $\{111\}$ slip planes remain subjected to high shear stresses determined by the vertical stress axis. Thus although the macroscopic

crack growth may be along the $[110](001)$, it may still occur microscopically on $\{111\}$ planes and does not therefore affect the crack propagation rate. Changing the macroscopic crack propagation direction will, however, simply lead to a change in the morphology of the fracture surface. For crack extension on $[110](001)$ the crystallographic facets will lie at 90° to the crack growth direction and at 54.7° to the crack growth plane. For $[100](001)$ growth the trace of the $\{111\}$ facets will be at 45° to the crack growth direction and the $\{111\}$ facets will again be at 54.7° to the crack growth plane.

The mechanism of failure on $\{111\}$ has been proposed (Gell and Leverant, 1968a) to be associated with the reversed dislocation motion at the crack tip. This gives rise to weakening of the slip plane and material separation occurs under the crack tip stresses. Alternatively, small cracks may nucleate on the slip plane and link with the main crack (Sadananda and Shahinian, 1981a). In both of these situations increasing the applied stress may be expected to increase the crack tip stress thus giving rise to increased material failure and correspondingly higher crack growth rates. During crack growth this situation may be attained by increasing the value of R which, for a constant ΔK , effectively increases K_{max} . However, the effect of R on the crack growth rate at room temperature is found to be negligible.

An alternative mechanism by which crystallographic failure may occur is if the slip offsets at the crack tip give rise to crack advance (fig. 4.7), a situation that has been considered by numerous authors, for example Neumann (1974) and Hornbogen and Zum Gahr (1976). On loading, a number of dislocations, n_o , will give rise to a slip step at the crack tip (fig. 4.7), on unloading, a number of dislocations, n_r , will reverse their motion and not contribute to

crack advance. In general, slip will not be completely reversible and $n_r < n_o$, thus a resultant slip step will be obtained and an increment of crack extension, da , result with

$$da = c(n_o - n_r) b \quad (4.13)$$

where c is a constant dependent on the orientation of the slip plane and b is the Burger's vector of the dislocation.

Therefore it can be seen that crack extension which is independent of the maximum applied stress can be obtained and little effect of the R ratio will be observed, these features are exhibited at room temperature.

In addition, the fracture surface features may also be explained in terms of this concept. The size of the crystallographic slip step produced on loading will be determined by the number of dislocations which produce the slip step. In general the crack growth may be considered to be made up of a large number of slip steps giving many small $\{111\}$ facets on the fracture surface. However, when inhomogeneous slip is obtained dislocation motion may be concentrated on specific planes and larger crystallographic facets result. Thus both large crystallographic facets and a number of smaller facets may be expected to arise from crack propagation at room temperature.

4.4.2 Elevated Temperature Crack Growth

The observation that crack propagation at elevated temperature occurs on (001) is similar to those reported by Scarlin (1976) in D.S. IN 738 LC, Jablonski and Sargent (1981) in a single crystal superalloy and Sadananda and Shahinian (1981) in polycrystalline Udimet 700.

Facetted growth on planes other than the slip plane is not restricted to superalloys at high temperature. In many cases the plane of crack growth may be determined by the symmetry of the slip systems in relation to the crack plane. For example, two intersecting $\{111\}$ slip planes may lead to a $\{100\}$ crack growth plane, this has been observed by Neumann (1974) in copper and by Garrett and Knott (1975) in aluminium. On the other hand, systems in which slip is severely restricted such as titanium alloys may exhibit facetted growth along, or close to, the cleavage plane (Ward-Close and Beevers, 1980).

Sadananda and Shahinian (1981d) have proposed that $\{100\}$ growth in superalloys represents failure by cleavage along $\{100\}$ (section 1.6.2). Such a mode of failure would therefore represent one in which limited plasticity occurred before fracture. However, the present work indicates that the mechanism of fracture is associated with high levels of strain at the crack tip (figs. 3.34 and 3.35). In addition, the failure is often accompanied by striation formation (fig. 3.20) also indicating failure due to plasticity at the crack tip. Scarlin (1976) has similarly reported the blurring of Laue spots taken from the crack growth plane and attributed this to plastic deformation. The mechanism by which crack growth on $\{100\}$ occurs is therefore closely connected to the deformation mechanisms of superalloys at elevated temperature.

With increasing temperature, superalloys are observed to undergo an anomalous increase in the yield stress attaining a peak value at approximately 700°C before falling again. This behaviour is paralleled by the response of the single phase γ' (Davies and Stoloff, 1965) and has consequently been attributed to the properties of the ordered phase. Although various mechanisms have been suggested,

for example strain ageing (Guard and Westbrook, 1959) and lattice friction (Johnston et al., 1966), the most detailed mechanisms for the temperature dependence of the yield stress are based on the observed changes in the slip behaviour of ordered alloys with increasing temperature (Thornton et al., 1970).

At room temperature slip occurs predominantly on {111} but with increasing temperature there is an increasing tendency for slip on {100} because of the favourable reduction in A.P.B. energy that occurs (Kear and Wilsdorf, 1962). At intermediate temperatures the increase in the flow stress may then be attributed to an interaction between the two slip systems causing dislocation debris. At higher temperatures this debris may anneal out and slip on {111} or {100} is no longer restricted and the flow stress falls.

Similar observations have been made on the creep strength of ordered alloys (Martin et al., 1957). Below the critical temperature for ordering of β -brass higher creep rates are observed than in the ordered state. This may be due to the restricted cross slip associated with the paired dislocations in the ordered alloy.

In alloys which contain discrete precipitates of an ordered phase and where slip at elevated temperatures is more favourable on {100}, the passage of an $a/2 \langle 110 \rangle$ {111} matrix dislocation into the precipitate may require the production of misfit dislocations with large Burger's vectors. If the original matrix dislocation is dissociated into partial dislocations, this process will also necessitate constriction of the partials. Further, if dislocation motion in the γ' is paired, cross slip of one half of the superlattice dislocation onto the more energetically favourable {100} may lead to a sessile dislocation being produced, see fig. 4.8 (Kear, 1966) which

would hinder further dislocation motion.

In contrast, increases in the temperature will lead to decreases in the flow stress of the surrounding γ matrix as dislocation climb around obstacles on the slip plane becomes more favourable. Therefore with increasing temperature the microstructure of the alloy in the present work will be one in which the precipitates increase in yield stress whilst the matrix decreases in yield stress, effectively giving a soft matrix containing a hard precipitate.

Gell and Leverant (1973) have noted a more homogeneous dislocation distribution with increasing temperature and decreasing strain rate, a result supported by the observations of Beardmore et al., (1969). Similarly Leverant and Kear (1970) have observed shearing of the γ' precipitate during primary creep and yet the dislocation structure obtained during secondary creep was more cellular with no clear indication of shearing of the γ' . More recently, Jablonski and Sargent (1981) found no evidence of shearing of the γ' precipitate during high temperature fatigue, and dislocations remained in the γ/γ' interface.

To investigate the dislocation structure obtained in the present work thin foils were prepared from the crack tip plastic zone of a specimen fatigued at 600°C and 10Hz. Thinning was carried out using an Edwards ion beam thinner operating at 5kV, a beam current of 60-90 μ A and an incidence angle of 5°. The foil was examined in the high voltage transmission electron microscope operating at 1000kV. The dislocation structures are shown in figs. 4.9a and b. At the lower magnification (fig. 4.9a) relatively few dislocations can be seen within the γ' phase, an observation more clearly established in fig. 4.9b where the dislocations in the matrix surrounding the γ'

precipitates may be clearly seen. Therefore in the present work deformation may be contained within the matrix due to the restrictions on dislocations either entering or shearing the γ' precipitate.

The examination of etched longitudinal sections from interrupted tests show that crack propagation occurs parallel to (001) (fig. 4.10). This growth is also seen to be accompanied by a series of irregular perturbations, the separation of which is, however, much greater than the crack growth per cycle.

At higher magnifications (fig. 4.11) the irregular perturbations may be seen to be associated with some limited shear of the γ' precipitate and crack propagation occurs primarily within the γ matrix. This is confirmed in fig. 4.12 where the crack propagation within the matrix reveals a series of more regular perturbation whose spacing is comparable with the observed growth rate per cycle.

The crack propagation sequence will therefore be similar to that illustrated in fig. 4.13. In fig. 4.13a crack propagation takes place in the γ matrix and the failure may be accompanied by striation type growth. Propagation within the γ' is limited because of the higher flow stress of the surrounding precipitate and its resistance to the passage of dislocations either through or into it. Since the γ' precipitates are cuboidal their faces lie on {100} thus constraining the growth of the crack to the γ matrix lying, macroscopically, on {100}. However, as the γ' precipitates are not regularly aligned on {100} a situation may arise where a γ' precipitate intersects the crack propagation plane (fig. 4.13b). To propagate further the crack must either grow around the precipitate (fig. 4.13c) or shear the precipitate and grow through it (fig. 4.13d). The shear of the γ' precipitate which blocks propagation will leave a perturbation on the

crack plane whose spacing has little relation to the crack growth rate. Once this precipitate has been passed growth continues in the matrix along {100} as before.

Therefore it can be seen that the crack growth sequence can be modelled in terms of plasticity controlled failure at the crack tip to give growth along {100}. The crack propagates preferentially in the γ matrix and is constrained by the surrounding γ' precipitates to grow along {100}. Such a mode of growth would be expected to be found in alloys containing high volume fractions of γ' since the precipitates then form cubes aligned on {100}. Crack growth on {100} has been observed in the high volume fraction γ' alloys D.S. IN 738 LC (Scarlin, 1976), a single crystal superalloy (Jablonski and Sargent, 1981) and polycrystalline Udimet 700 (Sadananda and Shahinian, 1981).

4.4.3 Frequency Dependent Crack Growth

In the mid ΔK regime the high temperature crack growth rates have been observed to increase with decreasing frequency (figs. 3.8 to 3.11). In particular, decreases in the frequency below 1Hz give rise to significant increases in the crack growth rate per cycle. The previous discussion has shown that the mechanism of crack advance is dependent on crack tip plasticity. Further, the increased crack growth rate is not associated with a change in the mechanism of crack growth, since the strain at the crack tip is independent of the frequency, and no significant change in the fracture surface occurs.

Although crack growth occurs under the application of an alternating stress plastic flow leading to crack extension will occur during tensile loading. On unloading resharpenering of the crack tip

will occur so that the extent of the failure is limited and a degree of stable crack propagation per cycle is obtained.

Many authors have considered that crack growth may be determined by a displacement at the crack tip. However, for crack advance to occur, this displacement must be accompanied by some material failure. For example, growth by striation mechanisms requires that plastic flow of material effects an increase in the surface area of the crack and a corresponding increase in the crack length. Thus, although a displacement criterion may result in plastic flow it is not a sufficient fracture criterion since it does not state in what size of region the fracture displacements are concentrated. McClintock and Irwin (1964) therefore suggested that ductile fracture may occur when a critical strain, is attained over a certain distance, r_c (fig. 4.14). Gerberich (1970) has suggested that this criterion is in fact analogous to that of a critical displacement since the displacement may be related to a microstructural strain and microstructural size parameter.

Consider, initially, the problem of whether or not a crack will advance by an amount da under a monotonic load when the strain distribution is that given in fig. 4.14. For this situation, extension of the crack tip from a to $a+da$ would give rise to a strain at the critical distance r_c ahead of the new crack tip position which is lower than the necessary fracture strain ϵ_f . Therefore, to effect crack extension the strain at the point $a+da$ must be increased by an amount $d\epsilon$ until the fracture strain is reached. When this occurs the crack tip may then extend from a to $a+da$.

In practice the required increment $d\epsilon$ may arise from two components. First, when the crack extends an increment of plastic

strain, $d\epsilon_g$, will be produced at the point $a+da$ due to the growth of the crack, this situation has been more rigorously analysed by McClintock and Irwin (1964). If this strain increment is less than the required increase $d\epsilon$, the crack will not extend. In this case the applied stress must be raised until an additional strain of $d\epsilon_s$ ($=d\epsilon-d\epsilon_g$) is obtained (fig. 4.14). If the applied stress is insufficient to produce the strain $d\epsilon_s$ no crack extension will occur. However, once the strain increment is attained the crack will extend by da . With continued loading the fracture strain is reached over greater distances and larger increments of crack growth occur.

Under cyclic loading a similar process may occur. However, in this situation it is convenient to envisage that the strain increment due to the applied stress, $d\epsilon_s$, is constant for a given applied stress. For a crack loaded at a constant value of ΔK a given strain increment is available to cause fracture, the higher the value of ΔK , the higher $d\epsilon_s$. The magnitude of $d\epsilon_s$ will then determine the distance over which the crack advances (see fig. 4.15). For low values of ΔK , $d\epsilon_s$ is sufficient only to satisfy the fracture criterion over a small distance da_1 (fig. 4.15a). For larger values of $d\epsilon_s$ (higher values of K) the distance over which the fracture criterion is satisfied increases, da_2 (fig. 4.15b). Therefore, if da_1 and da_2 represent the crack extensions per cycle it can be seen that higher growth rates will be given for higher values of applied ΔK . Let us now consider the present case in terms of the observed strain distributions ahead of the crack tip and the measured growth rates for differing frequencies of testing.

Analysis of the strain distributions ahead of the crack tip has shown that at elevated temperature the crack tip strain is independent

of the frequency of cycling (figs. 3.34 and 3.35). With decreasing frequency there is an increase in the plastic zone size and a decrease in the strain gradient. The situation may then be approximated to that shown schematically in fig. 4.16.

At high temperatures the mechanism of fracture at low frequency is not observed to differ from that obtained at higher frequencies. Crack propagation is observed to continue preferentially in the γ matrix and no additional modes of failure are observed in the mid ΔK regime. Therefore it is likely that the microstructural distance relating to the fracture criterion is not affected by changes in the testing conditions and remains a microstructurally constant parameter.

For a given distance ahead of the crack tip it can be seen that at 600°C the strain increment required to attain fracture is largest when the frequency of cycling is high, that is

$$d\epsilon_{10} > d\epsilon_1 > d\epsilon_{0.1}$$

As previously stated this increment may be composed of a component due to crack extension and a component due to the applied stress, it is likely that the component due to crack extension is similar in the cases considered. There is observed to be little difference between the strain gradients for 10Hz and 1Hz so that the strain increments required for crack extension will be similar, that is $d\epsilon_{10} \approx d\epsilon_1$. At a constant ΔK and R ratio, therefore, the available strain increment may lead to fracture over similar distances. Thus the crack extension will be similar and similar growth rates result.

However, at 0.1Hz $d\epsilon_{0.1}$ is significantly less than the required strain increment at either 10Hz or 1Hz. Therefore, at a constant ΔK and stress ratio, fracture will be obtained over a larger distance

(fig. 4.17) and higher crack growth rates result. If the frequency of cycling is reduced further, for example to 0.01Hz, the increase in the crack growth rate will continue because of the resulting change in the strain gradient.

At 850°C, the situation is less clear since crack tip stress relaxation affects not only the strain distribution but also the crack geometry. However, it can be seen that even under these conditions small increases in crack growth may be obtained. Further reductions in the frequency may lead to increased geometrical changes of the crack tip so that the crack growth rates may then begin to decrease when the blunting effect of the crack tip dominates the deformation behaviour.

The model presented for the frequency dependence of crack growth may be used to provide an explanation of the increased effect of frequency at higher values of R (fig. 3.9) and is illustrated in fig. 4.18. At $R=0.1$ the strain distributions will be those shown in fig. 4.18a and increased growth rates will occur at the lower frequency. At a constant value of ΔK the effect of increasing the value of R to 0.25 is to increase the values of both $K_{\max}$ and $K_{\min}$. In view of the higher value of $K_{\max}$ larger plastic zone sizes will be obtained at $R=0.25$ compared to those at the same ΔK but with $R=0.1$. In addition, the higher $K_{\max}$ will give rise to an increased amount of relaxation at 0.1Hz and little, if any, will be observed at 10Hz. The crack tip strain gradient at 0.1Hz will therefore be less than that at the same frequency but $R=0.1$ (fig. 4.18b) and the 10Hz strain gradient will be close to that obtained at $R=0.1$.

Therefore, by increasing the R ratio at a constant ΔK the strain increment provided by the applied stress will increase and, at a constant frequency, increases in the growth rate will be observed due

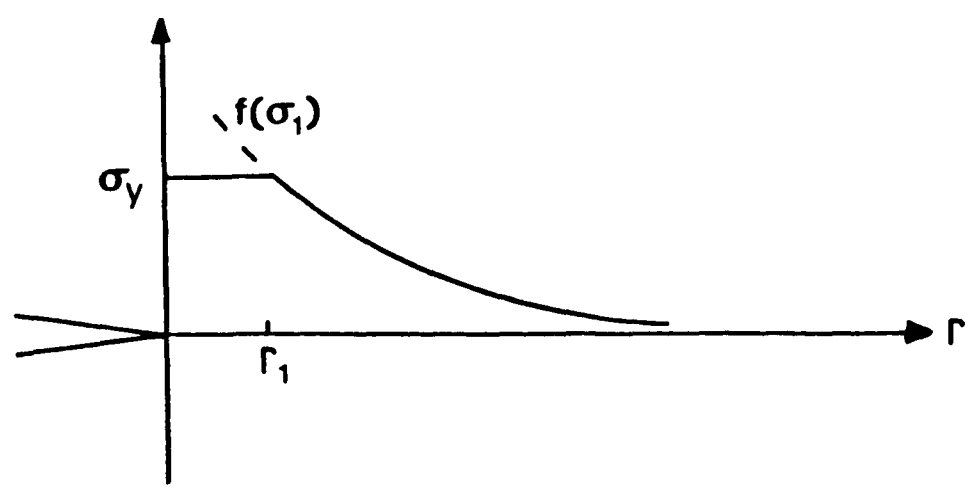
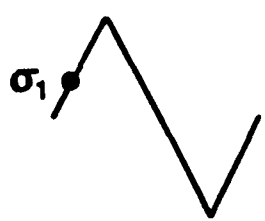
to the change in strain gradient. In addition, due to the decreased strain gradient associated with cycling at 0.1Hz larger increases in the growth rate will occur on decreasing the frequency from 10Hz to 0.1Hz at $R=0.25$ than are observed when $R=0.1$ (see fig. 4.18).

The preceding discussion has shown that the crack growth rate at elevated temperature may be modelled in terms of fracture occurring when a critical strain is achieved over a characteristic microstructural distance. Using this criterion, together with the measured strain gradients ahead of the crack tip, the frequency dependence of the crack growth rate may be rationalised. Extension of the model to consider the effect of the stress ratio correctly predicts the effect of increasing the stress ratio and also the increased effect of frequency observed at the higher stress ratio.

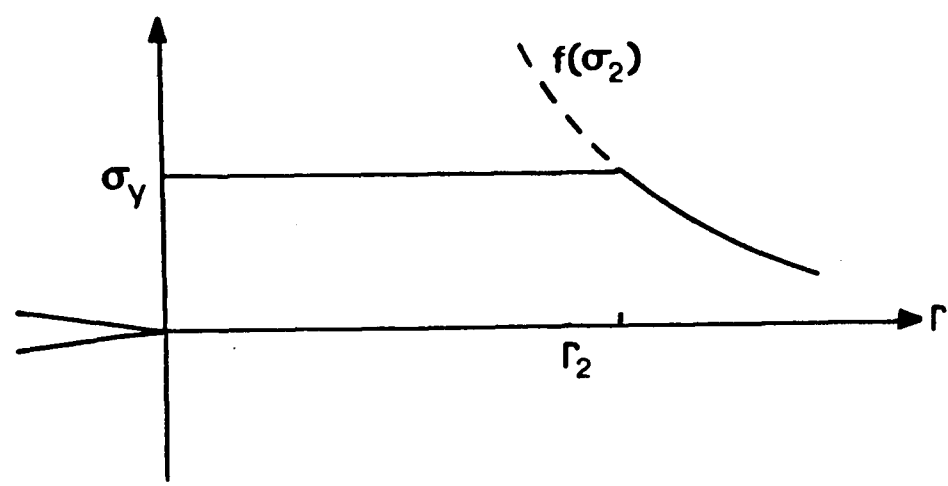
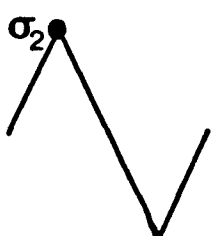
Figure 4.1

Schematic representation of the crack tip stress distribution during cyclic loading, solid lines represent the resultant stress distribution at various times during cycling.

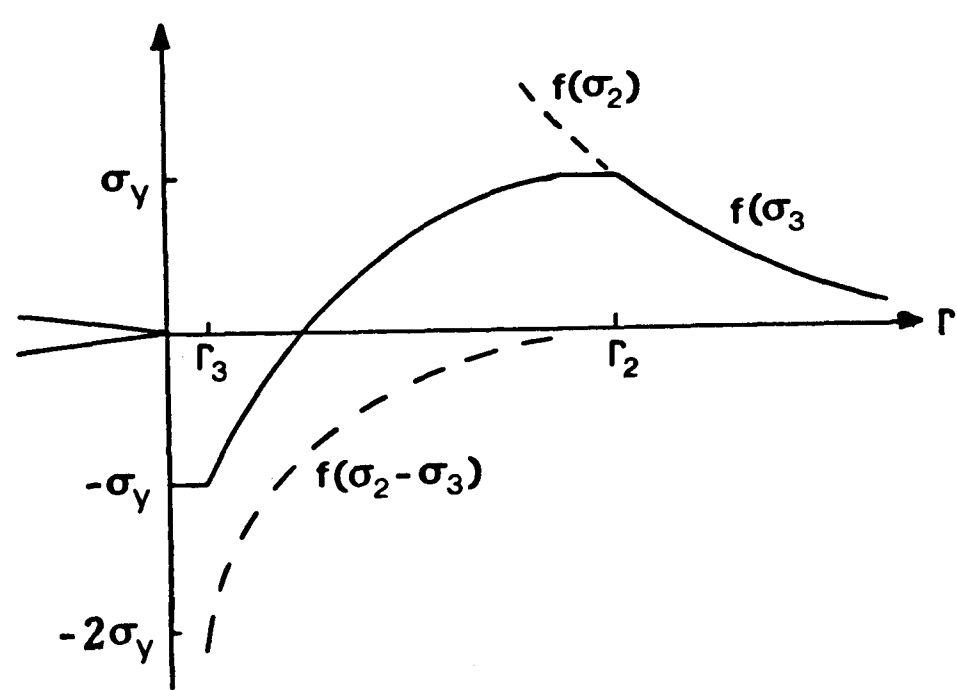
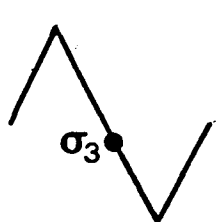
a)



b)



c)



d)

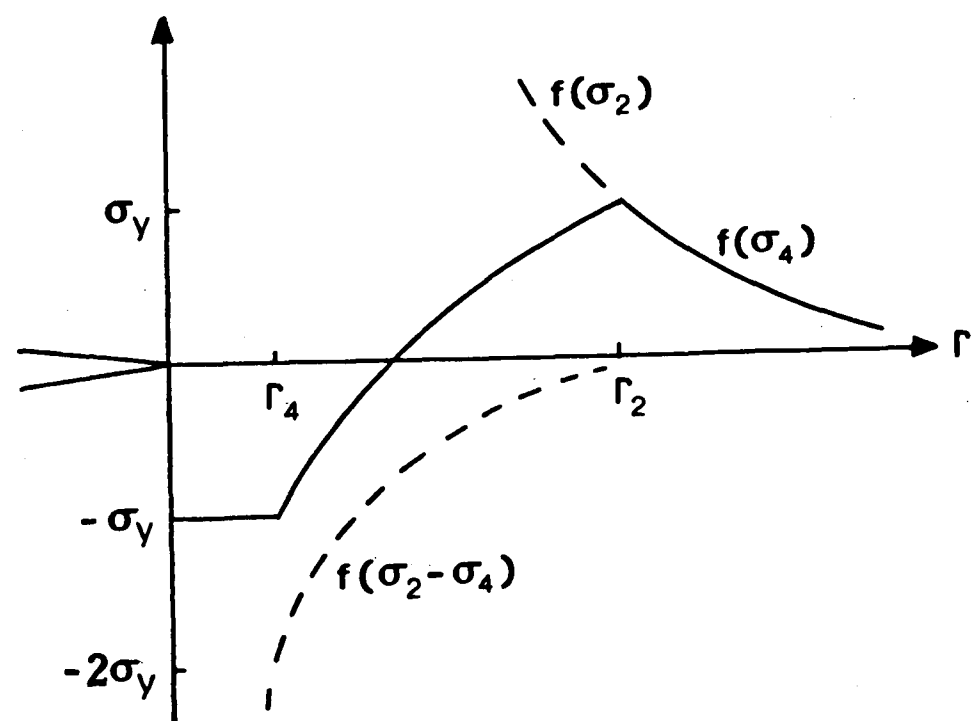
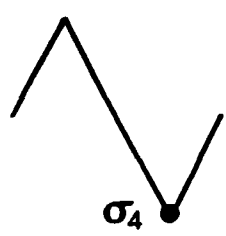
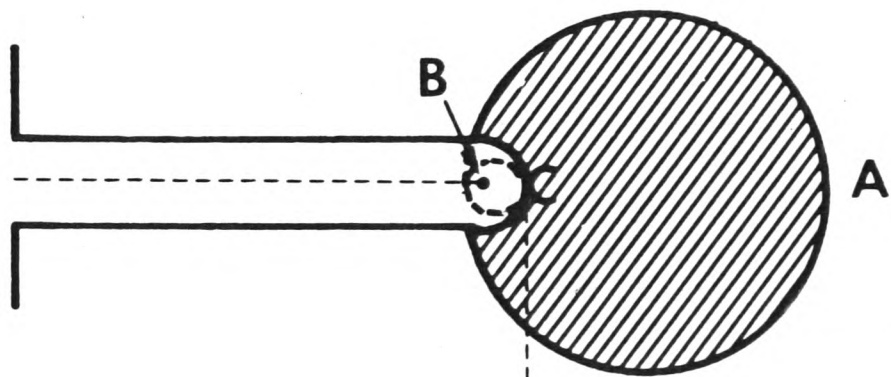


Figure 4.2

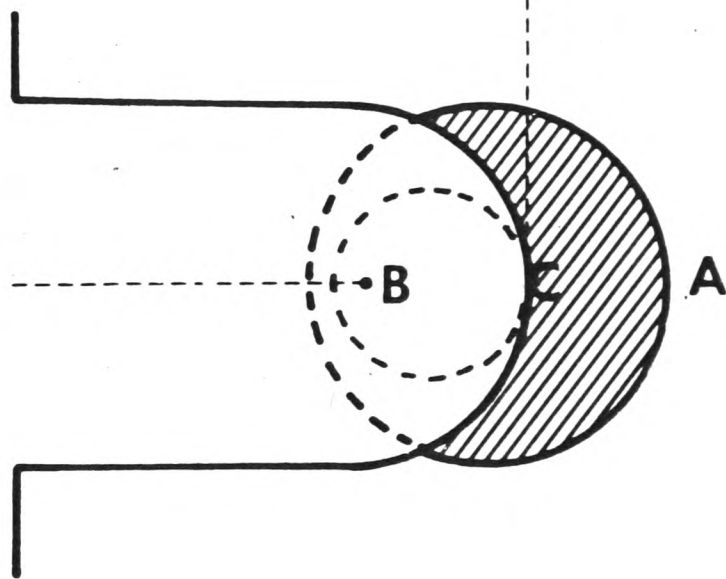
Schematic representation of the relationship between the plastic zone size and the crack tip geometry at the same applied stress intensity. Increasing the radius of curvature results in a decrease in the plastic zone size.

(After Kim et al., 1979)

①



②



$$\overline{AB}_1 = \overline{AB}_2, \overline{BC}_1 < \overline{BC}_2$$

$$\therefore \overline{AC}_1 > \overline{AC}_2$$

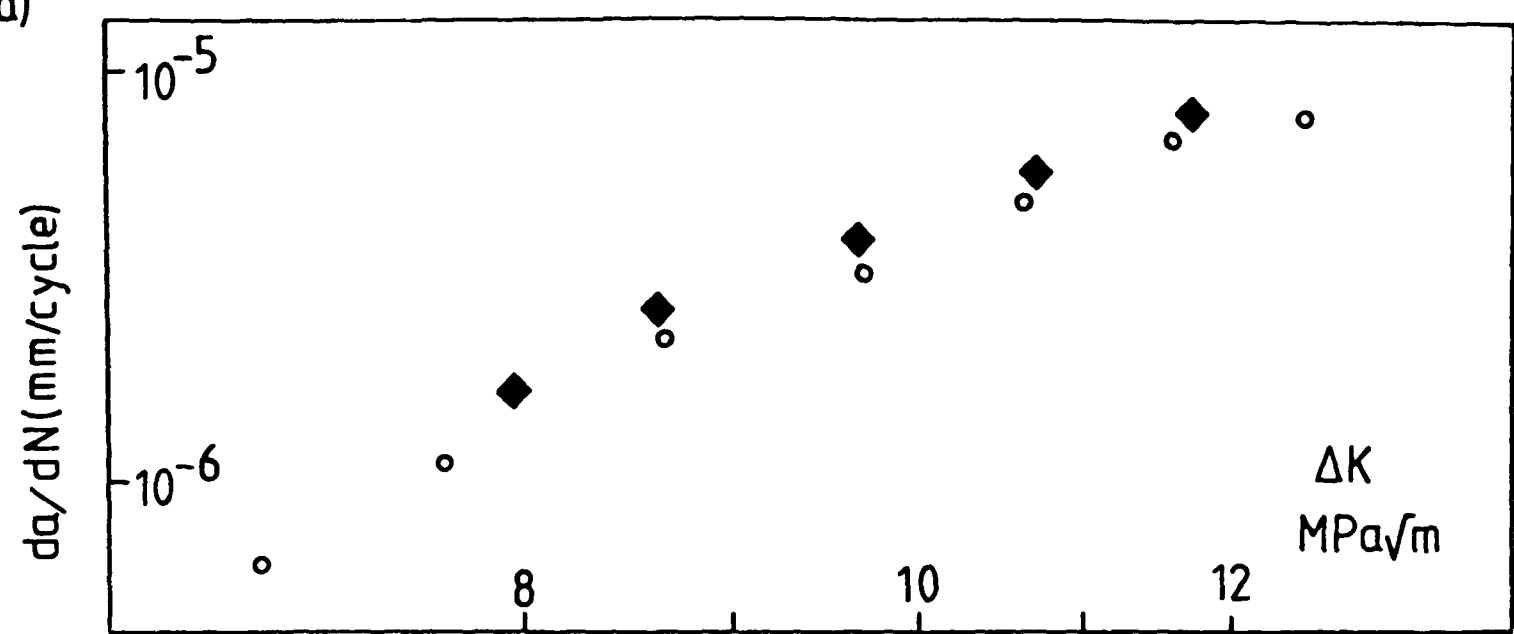
Figure 4.3

Low crack growth rates at different temperatures,
frequencies and stress ratios.

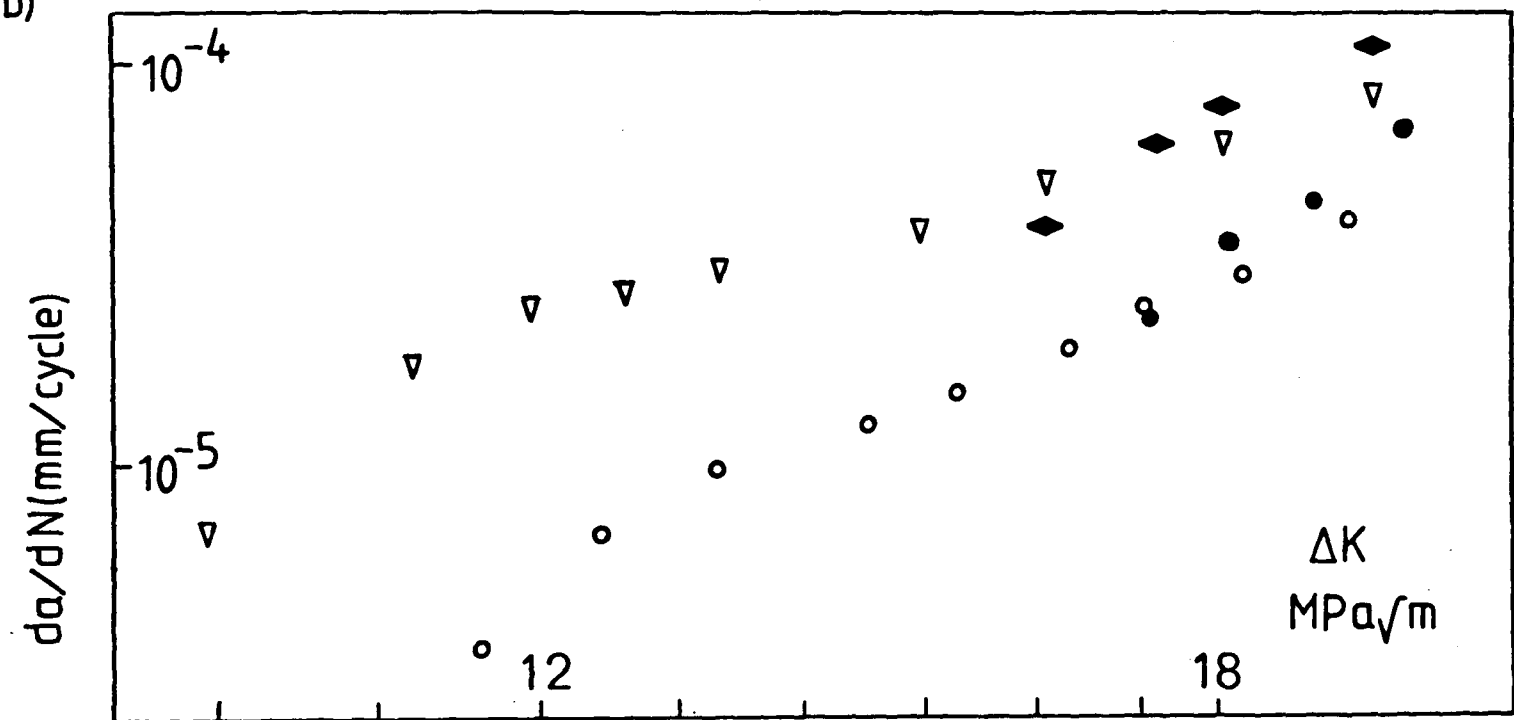
- a) Room Temperature,
- b) 600°C,
- c) 850°C.

Temp.	ν , Hz	R=0.25	R=0.1
R.T.	10	◆	○
600	10	▽	○
	0.1	◀	●
850	1	▲	△
	0.1	◇	◆

a)



b)



c)

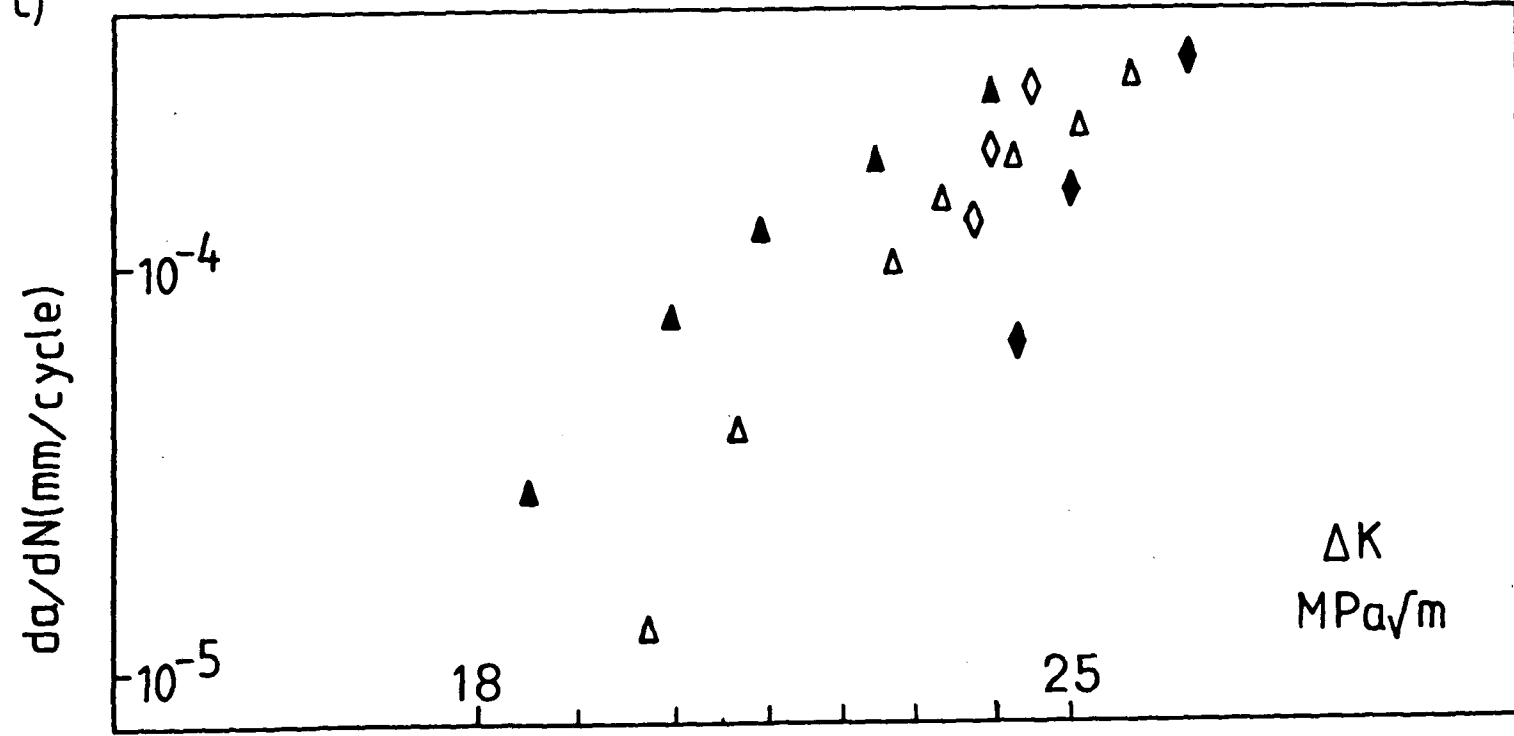


Figure 4.4

Crack tip region showing extensive carbide
cracking ahead of the main crack tip with
 $K_{\max}=50\text{MPa}\sqrt{\text{m}}$ at 600°C and 0.1Hz .

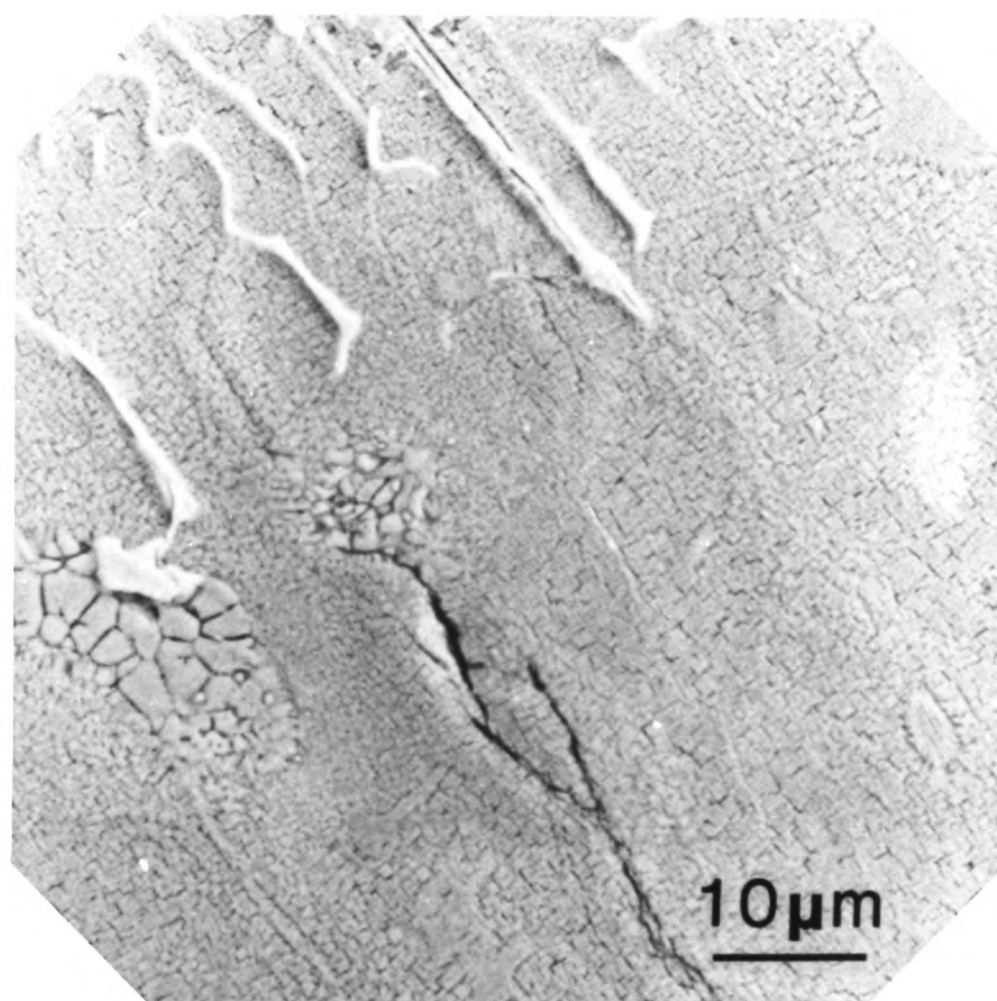
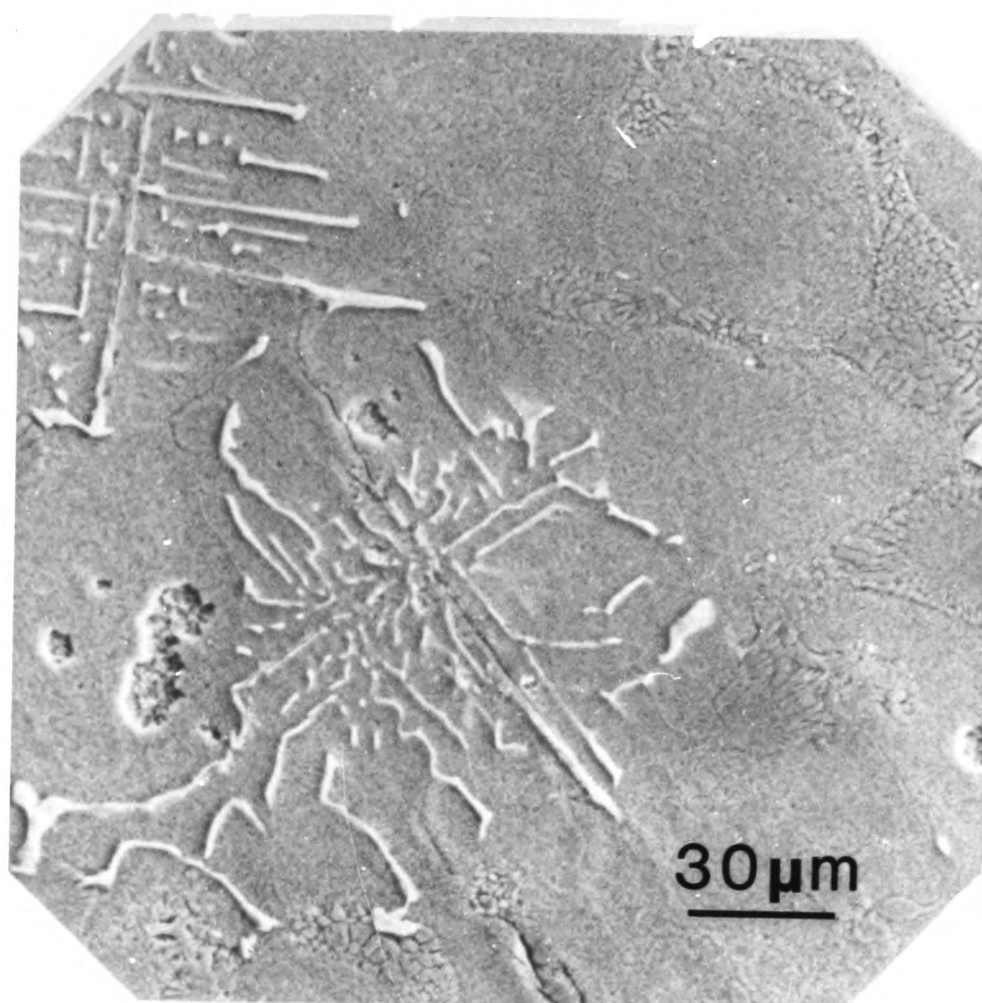
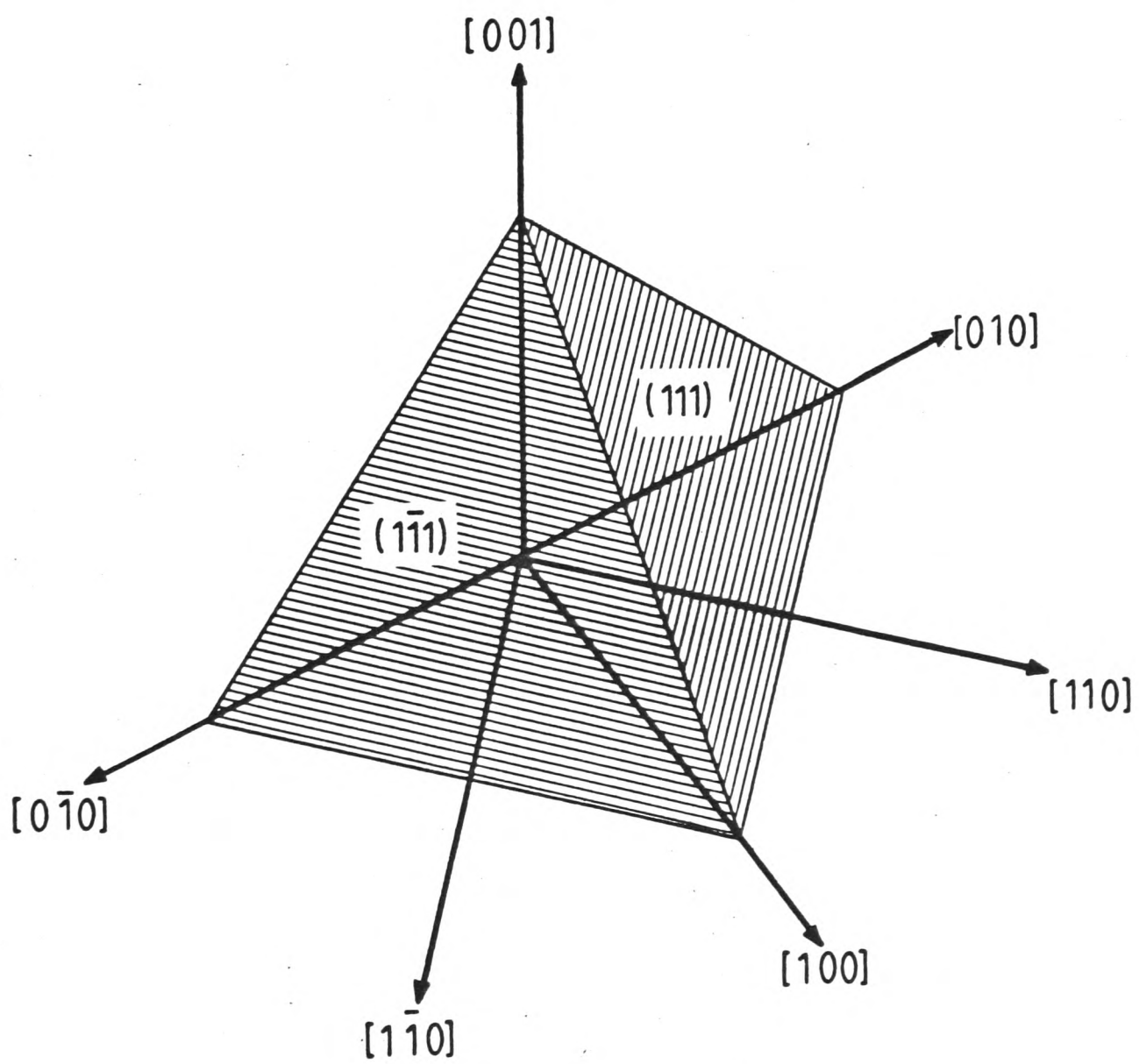
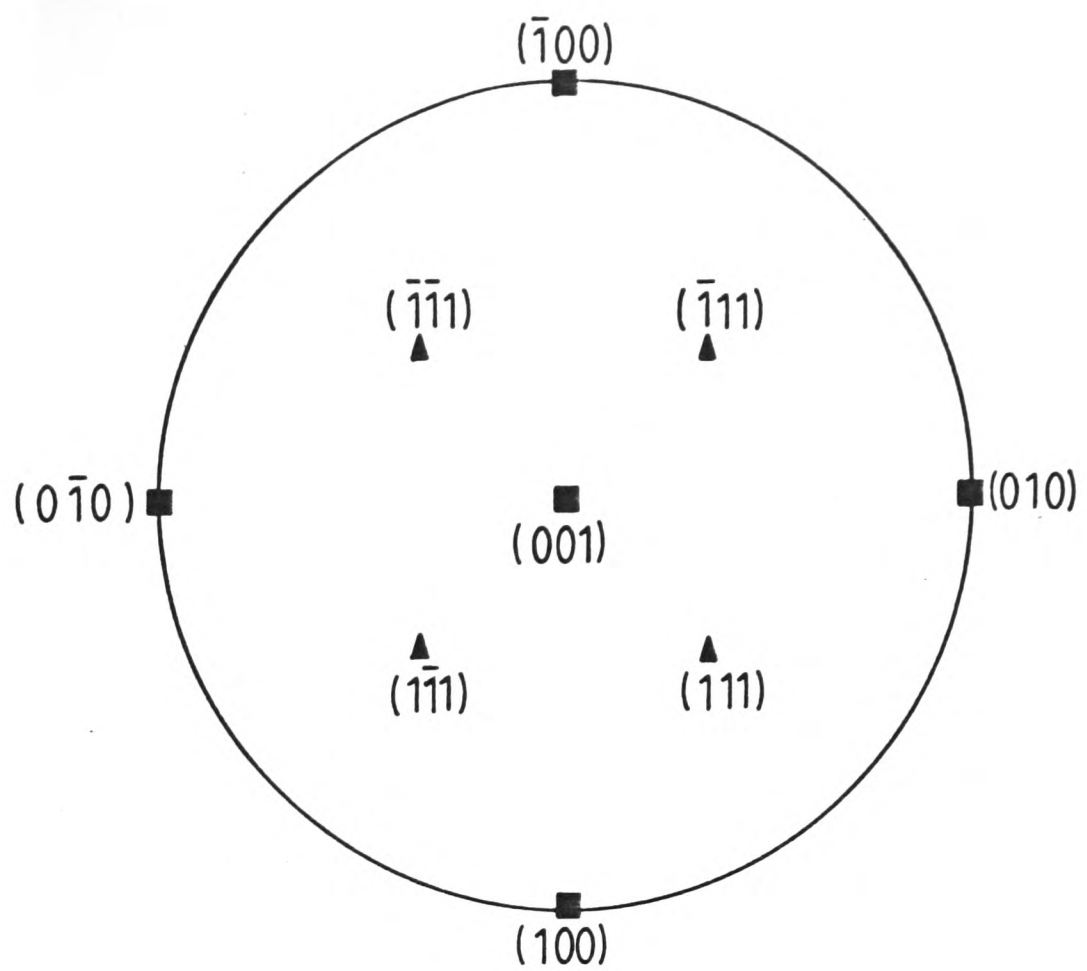


Figure 4.5

Crack tip crystallography with tensile loading on
[001] and crack propagation on (001).



(001) Crack growth plane

Figure 4.6

Etched section showing crack propagation by
fracture on $\langle 110 \rangle$ $\{111\}$ at room temperature.

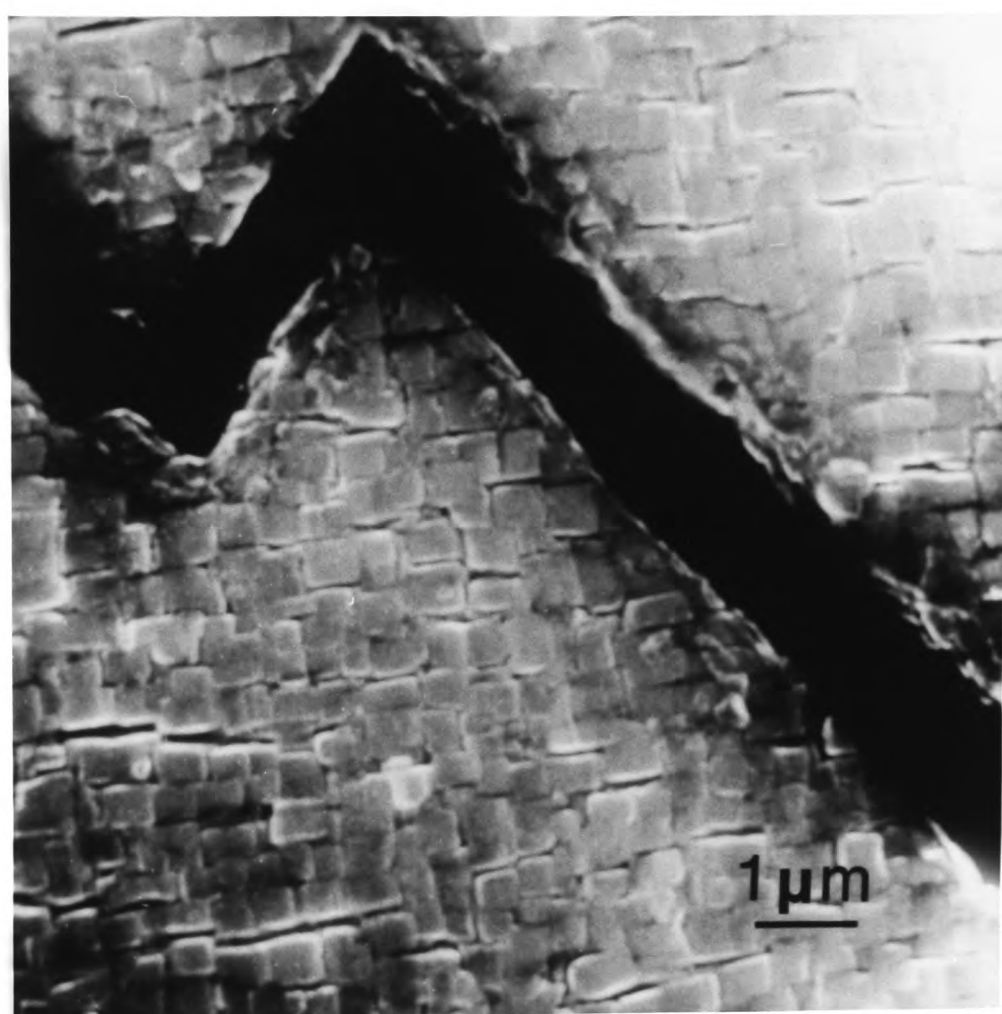


Figure 4.7

Schematic representation of crack propagation during one cycle resulting from slip offsets at the crack tip.

(After Hornbogen and Zum Gahr, 1976)

Figure 4.8

Schematic representation of the production of a sessile superlattice dislocation by cross slip.

(After Kear, 1966)

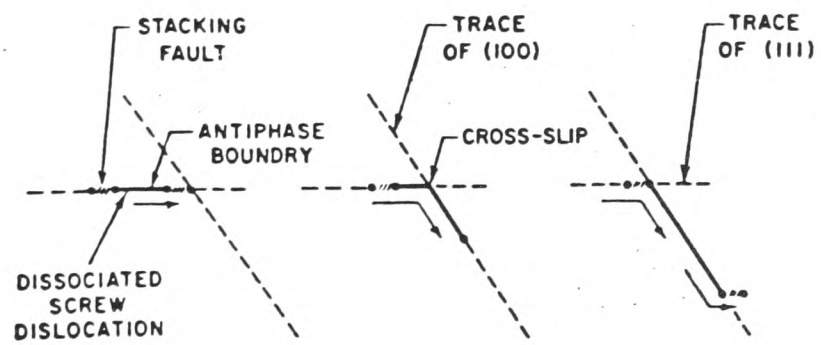
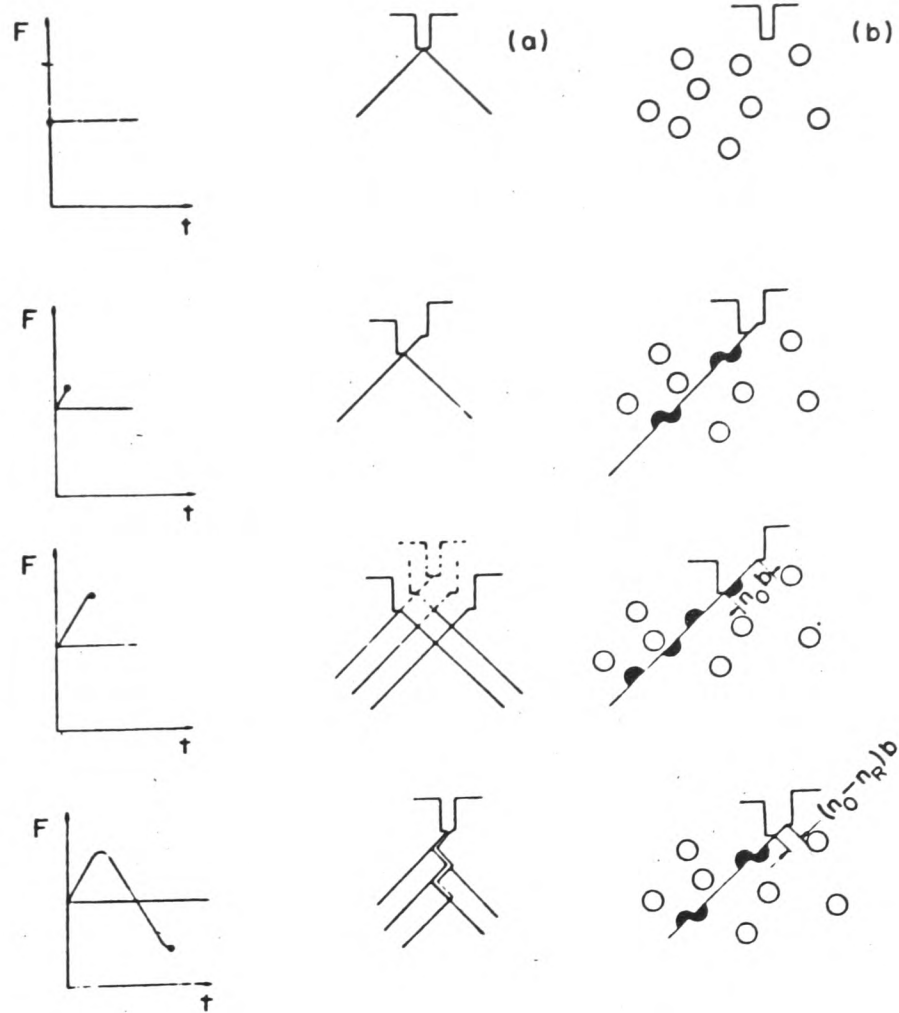


Figure 4.9

Dislocation structure in the crack tip plastic zone formed during fatigue at 600°C, 10Hz and $K_{\max} = 26\text{MPa}\sqrt{\text{m}}$.

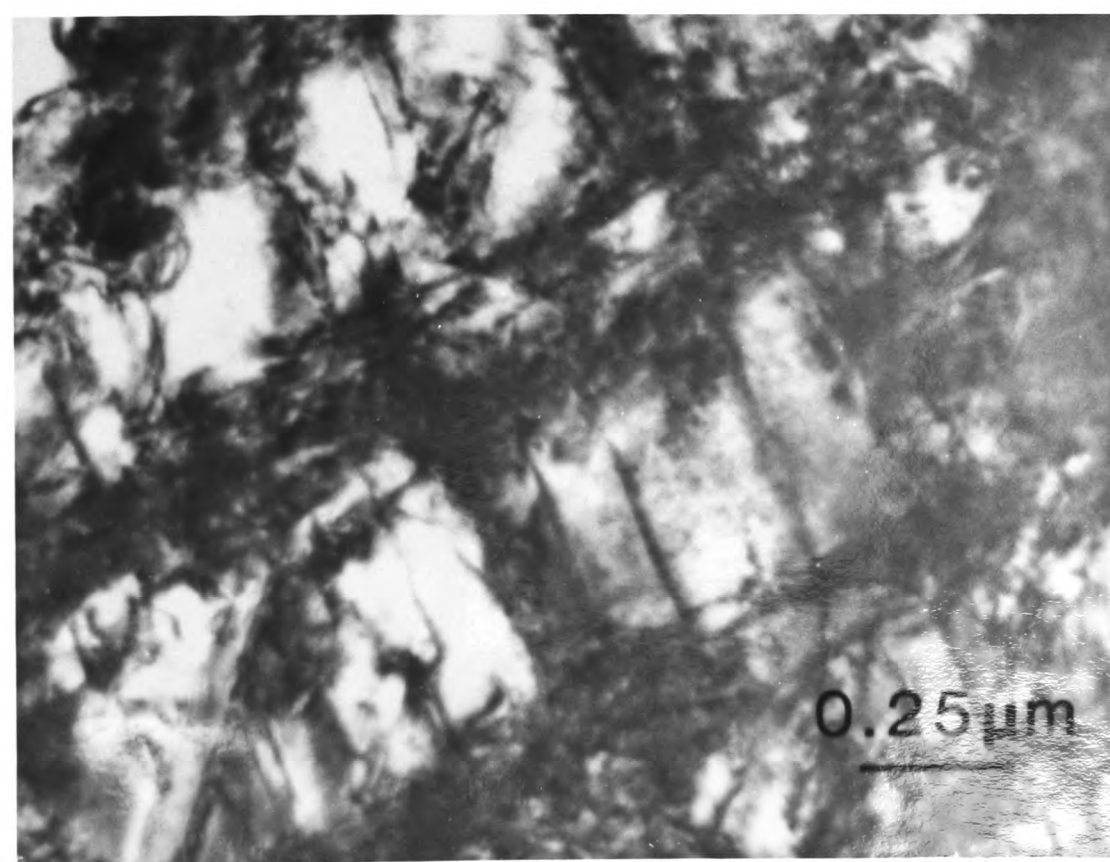
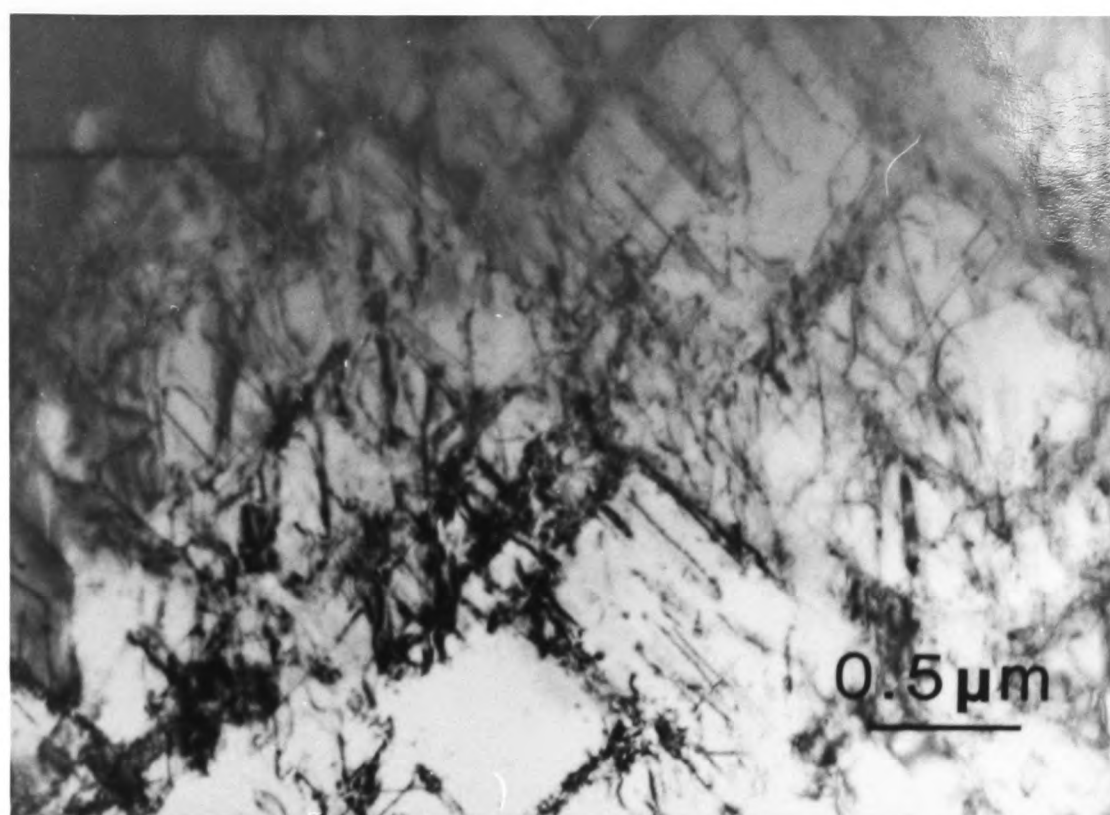


Figure 4.10

Microstructural features of crack propagation on
(001) at 600°C and 10Hz.

Figure 4.11

Crack propagation on (001) taking place in the
 γ matrix at 600°C and 10Hz.

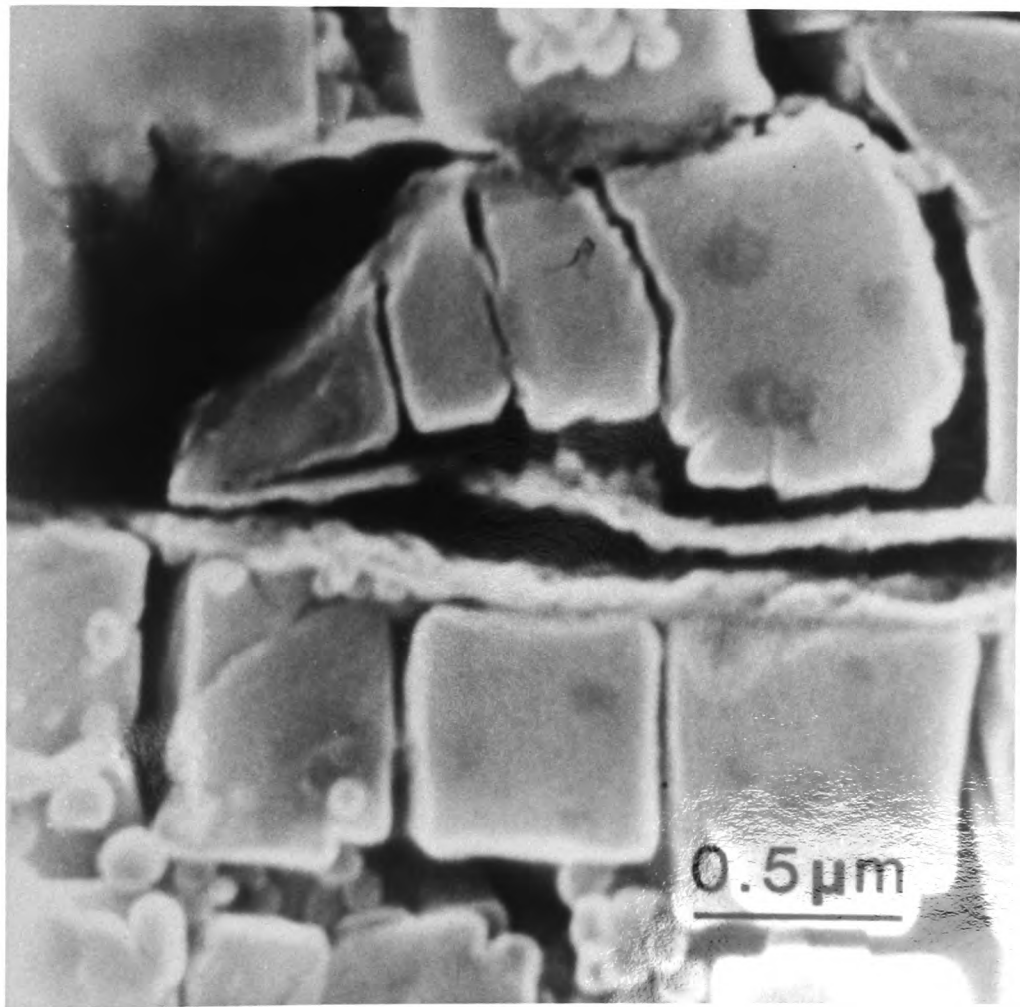
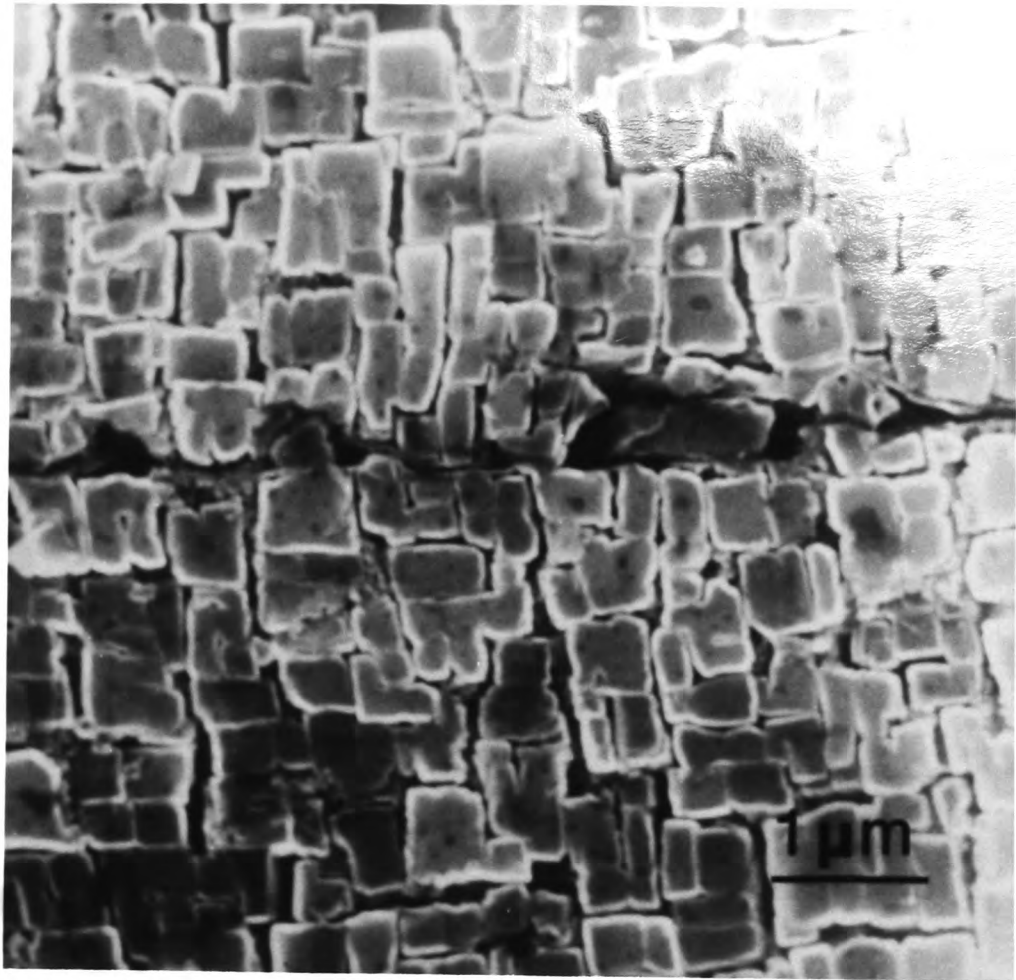


Figure 4.12

Crack propagation in the γ matrix at 600°C and
10Hz.

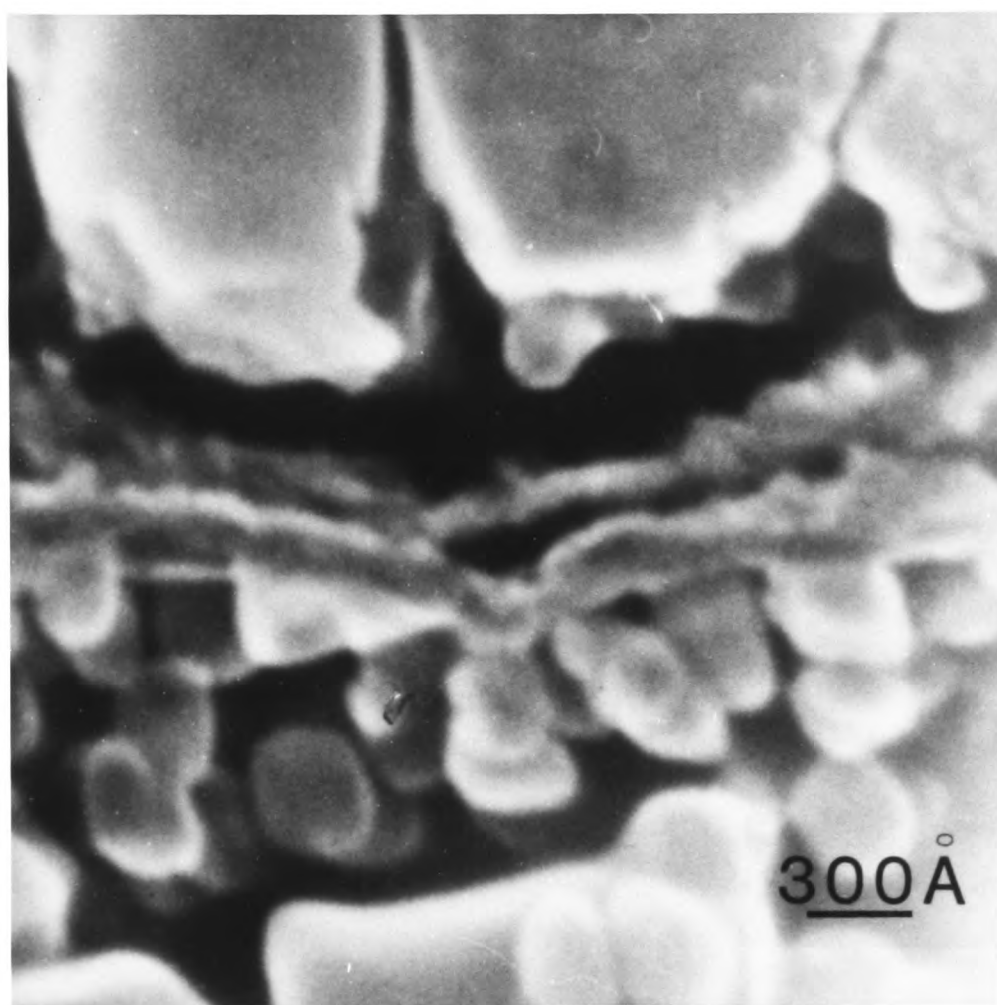
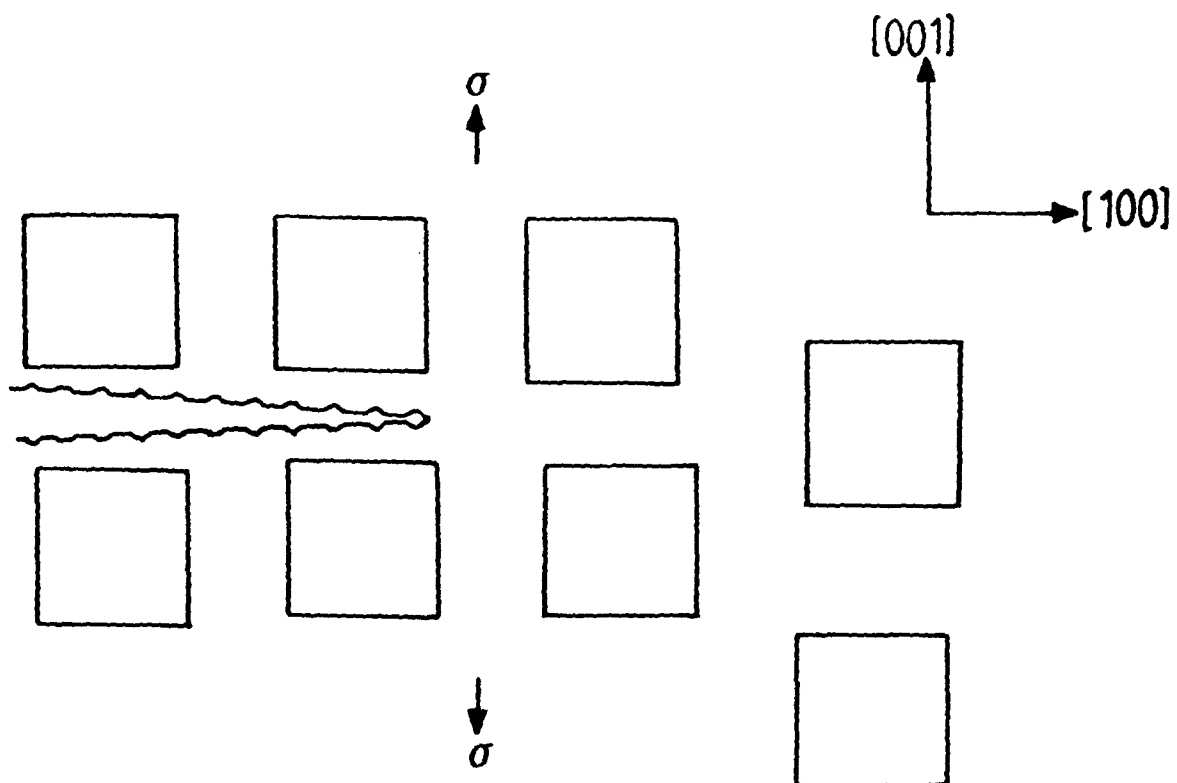


Figure 4.13

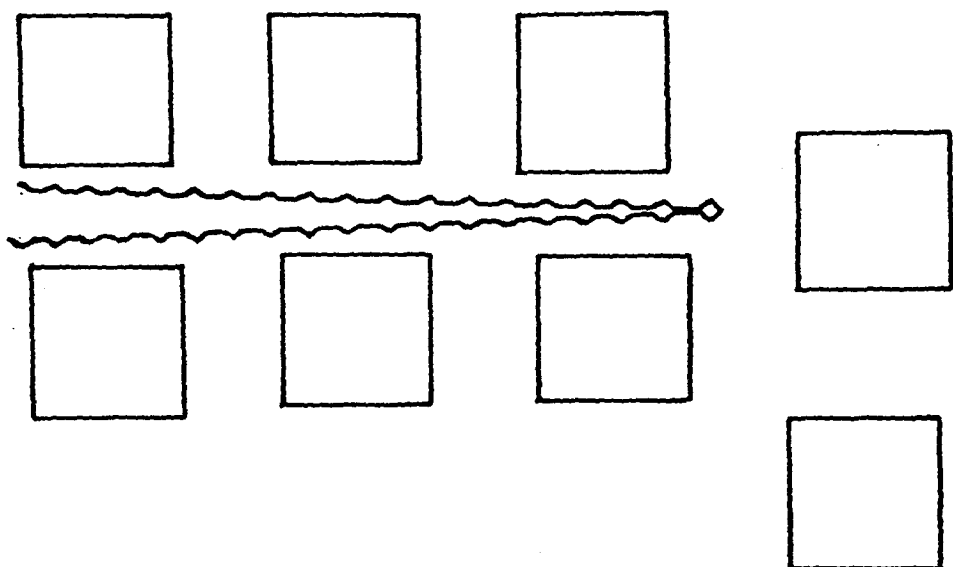
Schematic representation of the proposed crack propagation sequence

- a) crack advance in the γ matrix on (001) due to the constraints of the surrounding cuboidal γ' .
- b) Intersection of the crack plane with a γ' precipitate giving rise to further crack extension by either
- c) by-passing of the precipitate or
- d) shear of the precipitate.

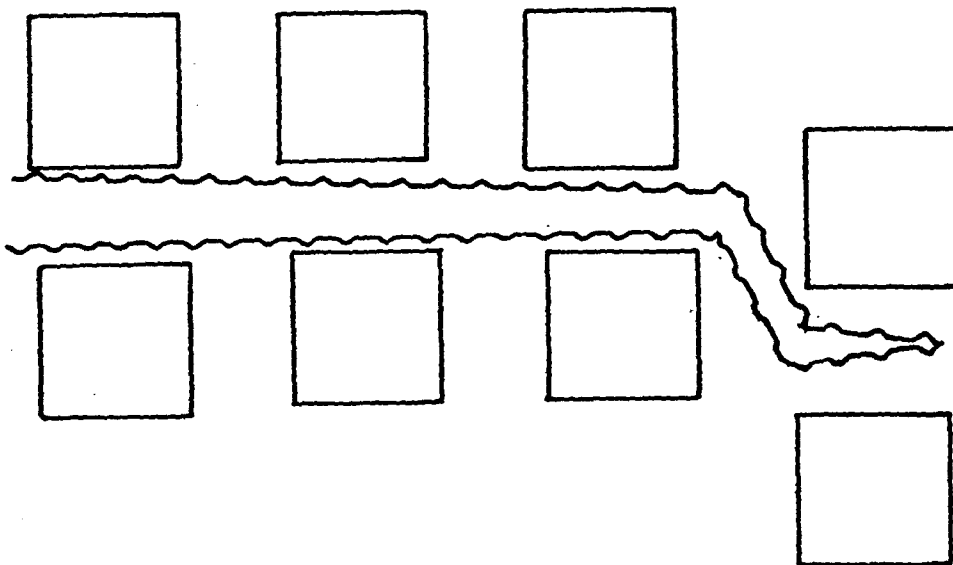
a)



b)



c)



d)

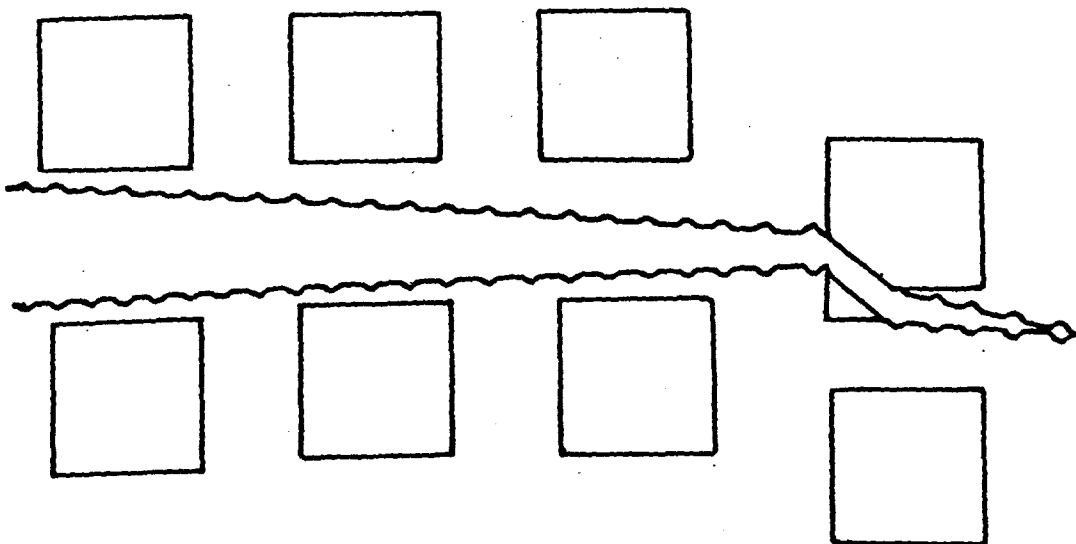


Figure 4.14

Crack advance by fracture when a critical strain ϵ_f is attained over a critical microstructural distance r_c . For the crack to extend by da additional strain $d\epsilon$ must be attained at the point $(a+da)$. The increment may arise due to crack growth ($d\epsilon_g$) and the applied stress ($d\epsilon_s$).

(After McClintock and Irwin, 1960)

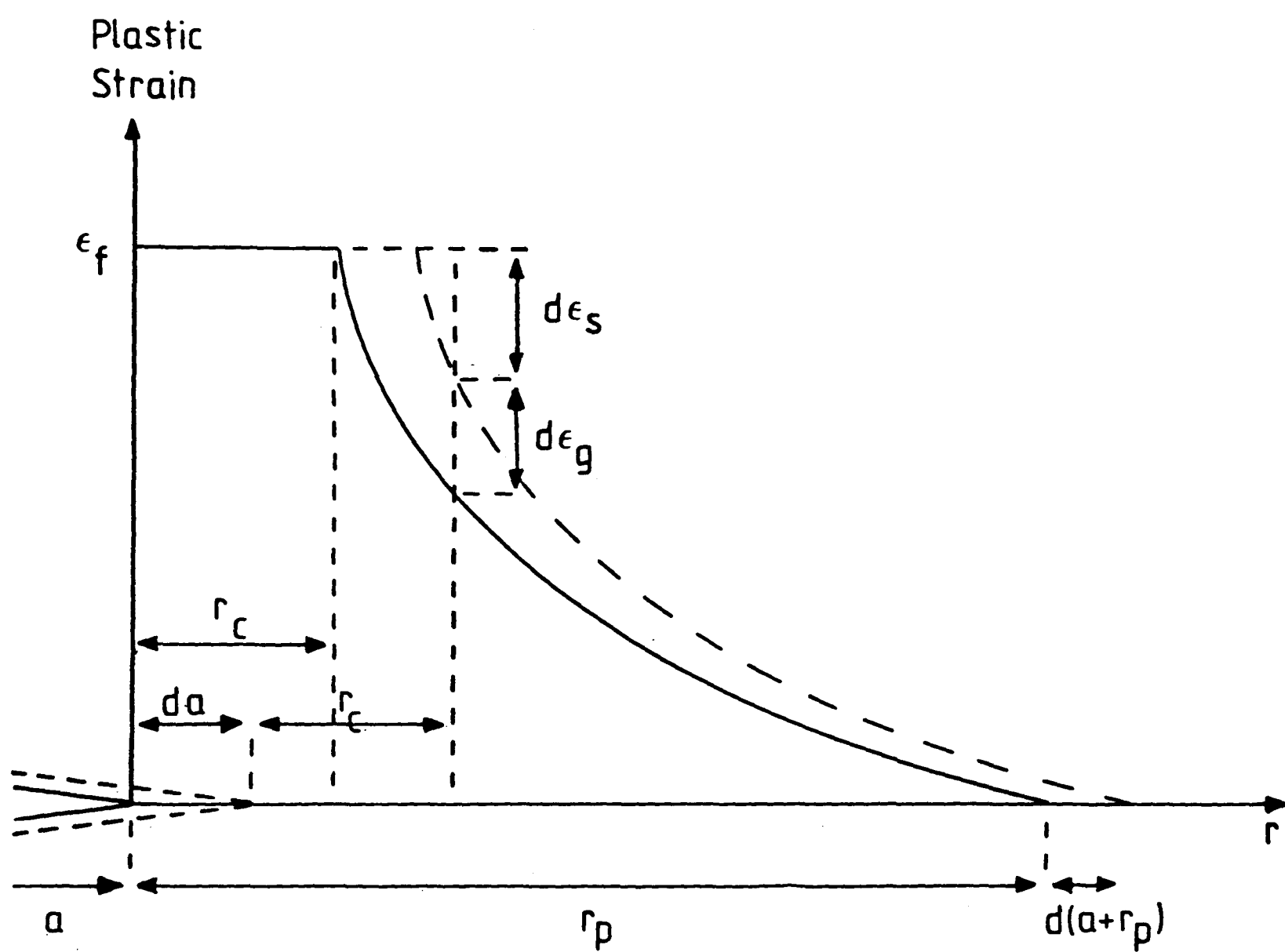


Figure 4.15

Extension of McClintock and Irwin's fracture criterion to cyclic loading,

- a) low ΔK , small plastic zone r_1 , the strain increment $d\epsilon_{s1}$ due to the applied stress is small and crack extension, da_1 , is small.
- b) Higher ΔK , a larger plastic zone, r_2 , is now obtained and an increased strain increment $d\epsilon_{s2}$ results in crack extension by da_2 ,
 $da_2 > da_1$.

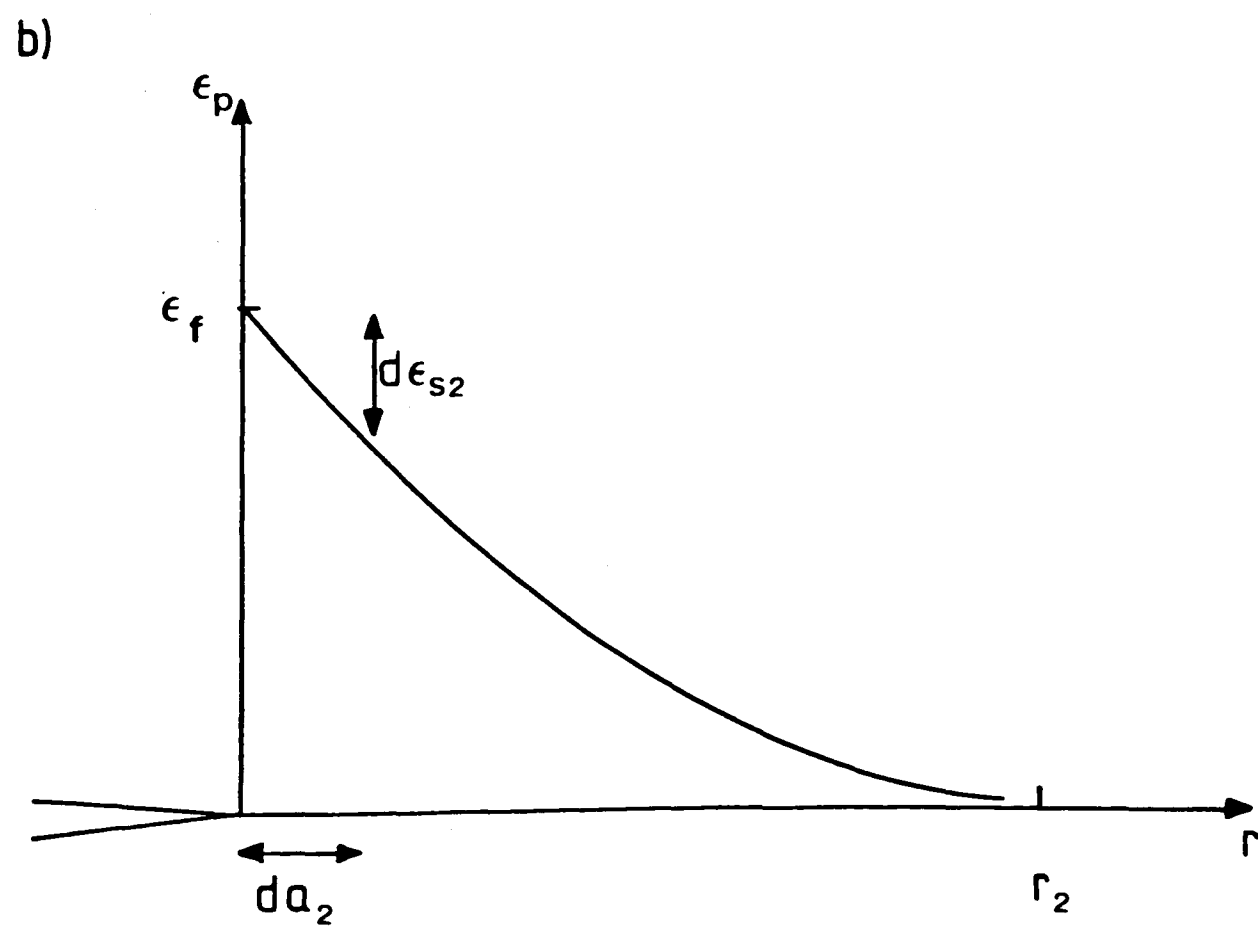
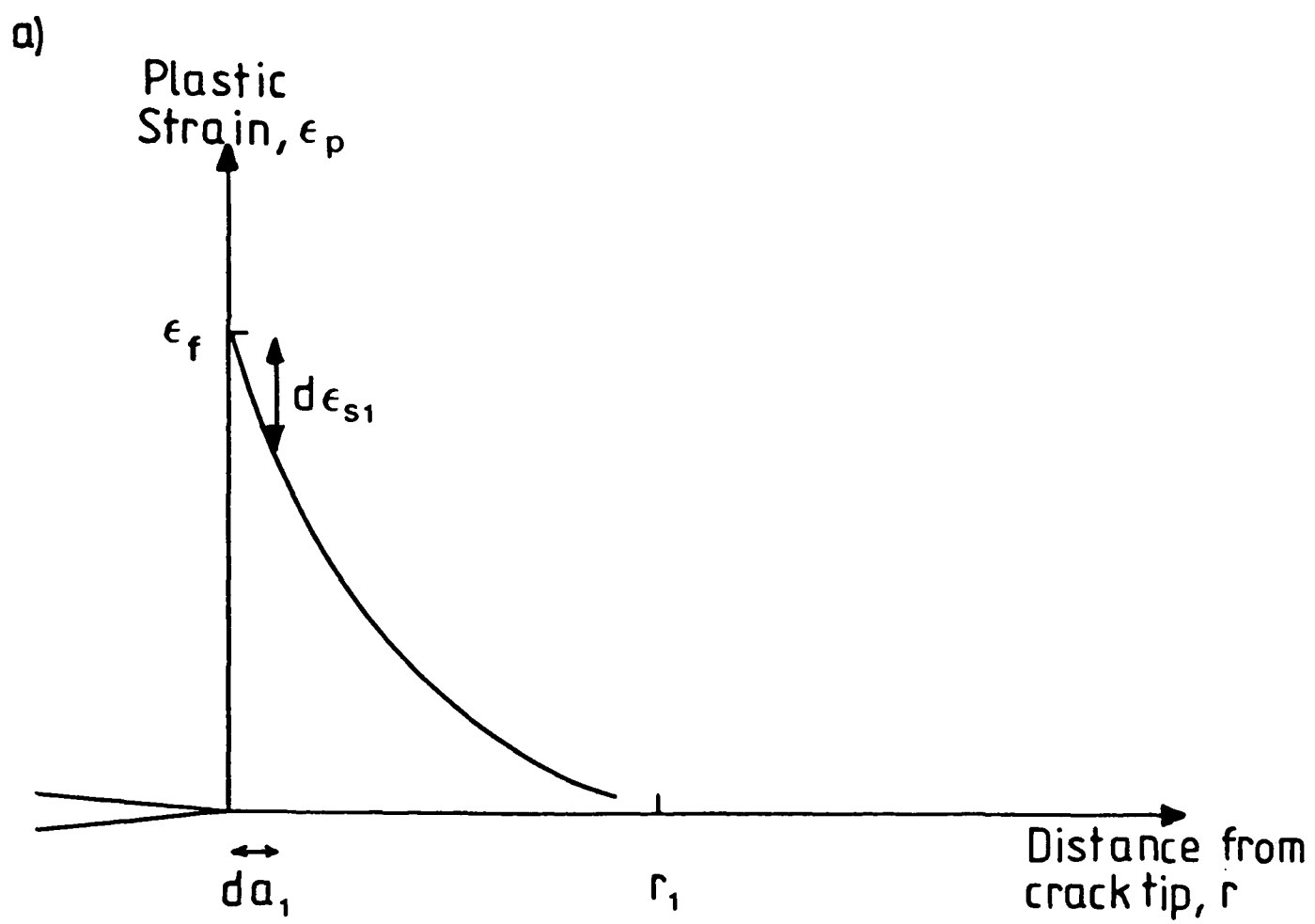


Figure 4.16

Schematic representation of the strain distributions in the present work at 600°C and frequencies of 10Hz, 1Hz and 0.1Hz. At a given microstructural distance the strain increments required to achieve failure are shown with $d\epsilon_{10} > d\epsilon_1 > d\epsilon_{0.1}$.

Figure 4.17

Effect of decreasing the frequency on the crack extension at a constant K_{max} . The applied strain increment now gives rise to increased crack extension at lower frequencies,

$$da_{10} \approx da_1 < da_{0.1} < da_{0.01}.$$

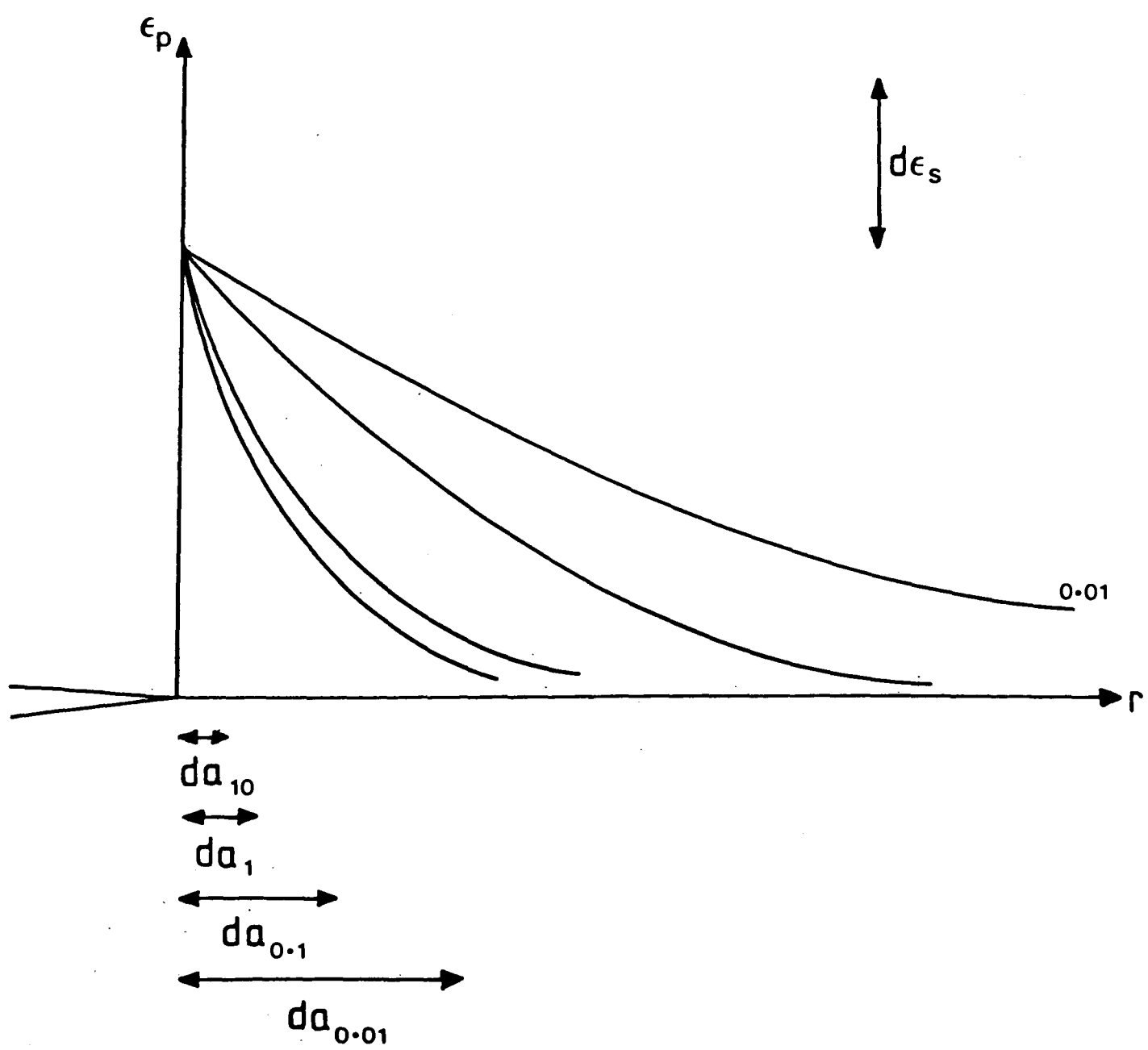
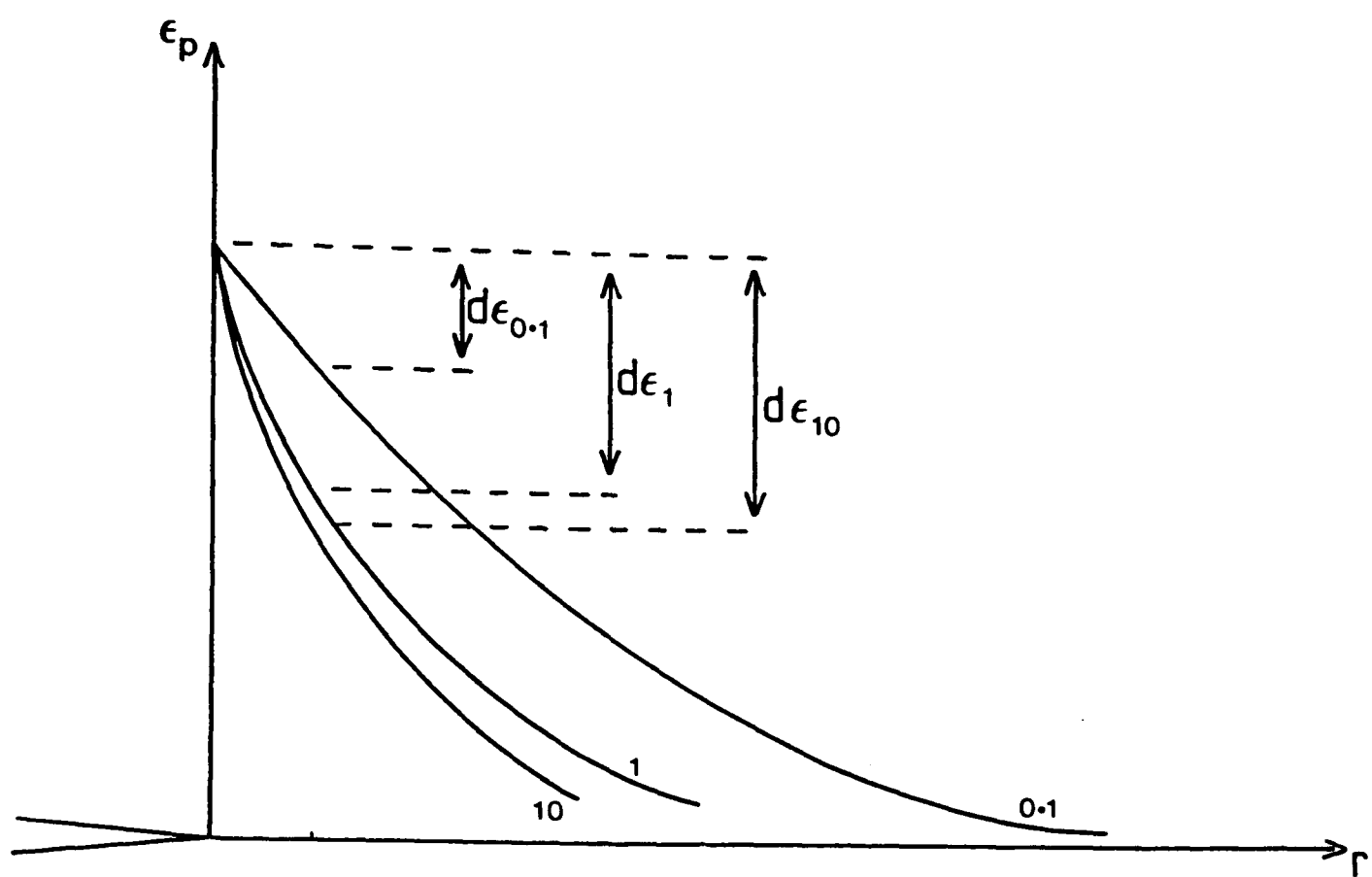


Figure 4.18

Effect of the stress ratio, R , on the frequency dependence of the crack extension per cycle at a constant ΔK .

a) Low R , $da_{10} < da_{0.1}$.

b) High R , the larger plastic zone sizes and larger strain increments for crack growth at a constant ΔK now give,

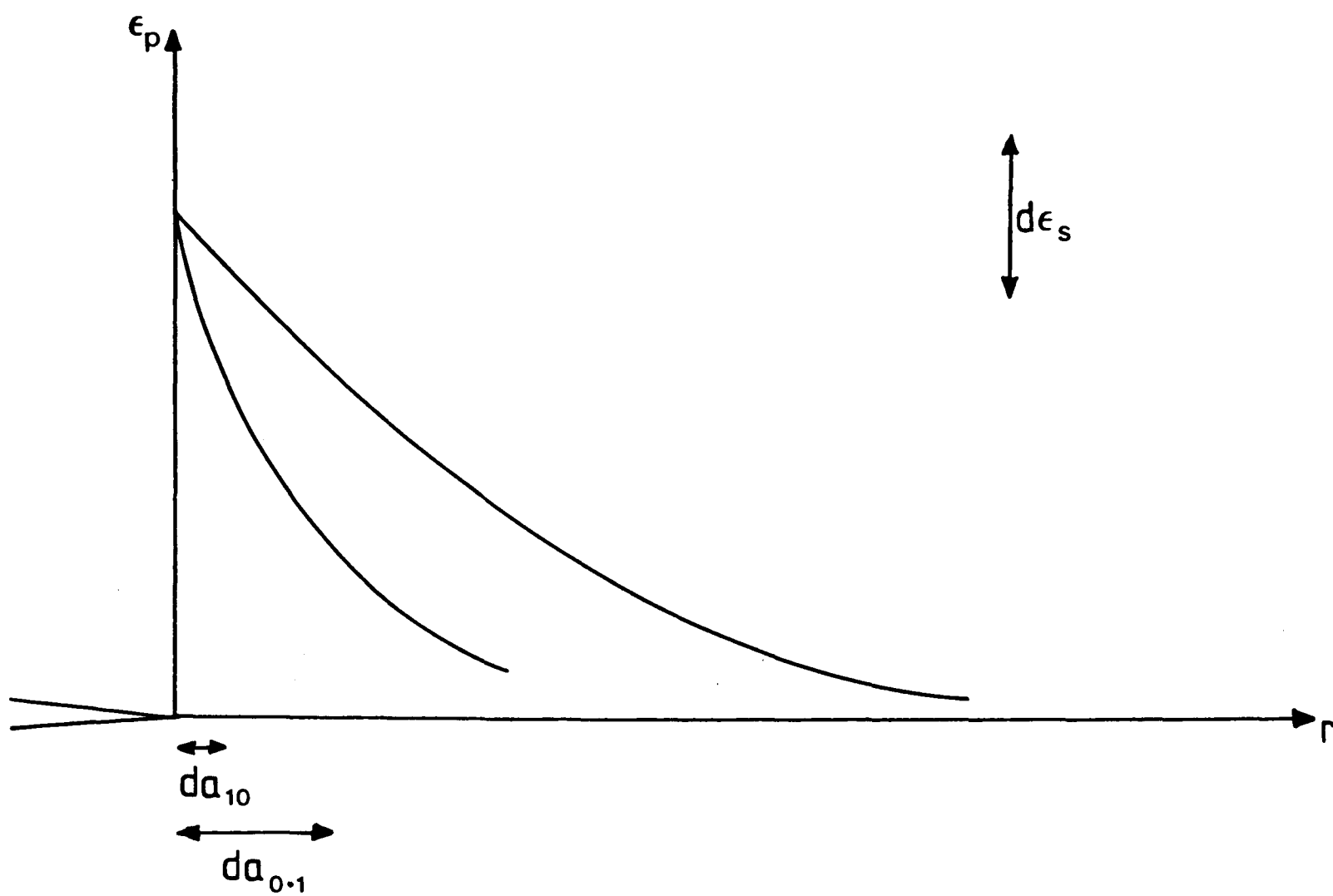
$$da_{10} (\text{low } R) < da_{10} (\text{high } R),$$

$$da_{0.1} (\text{low } R) < da_{0.1} (\text{high } R),$$

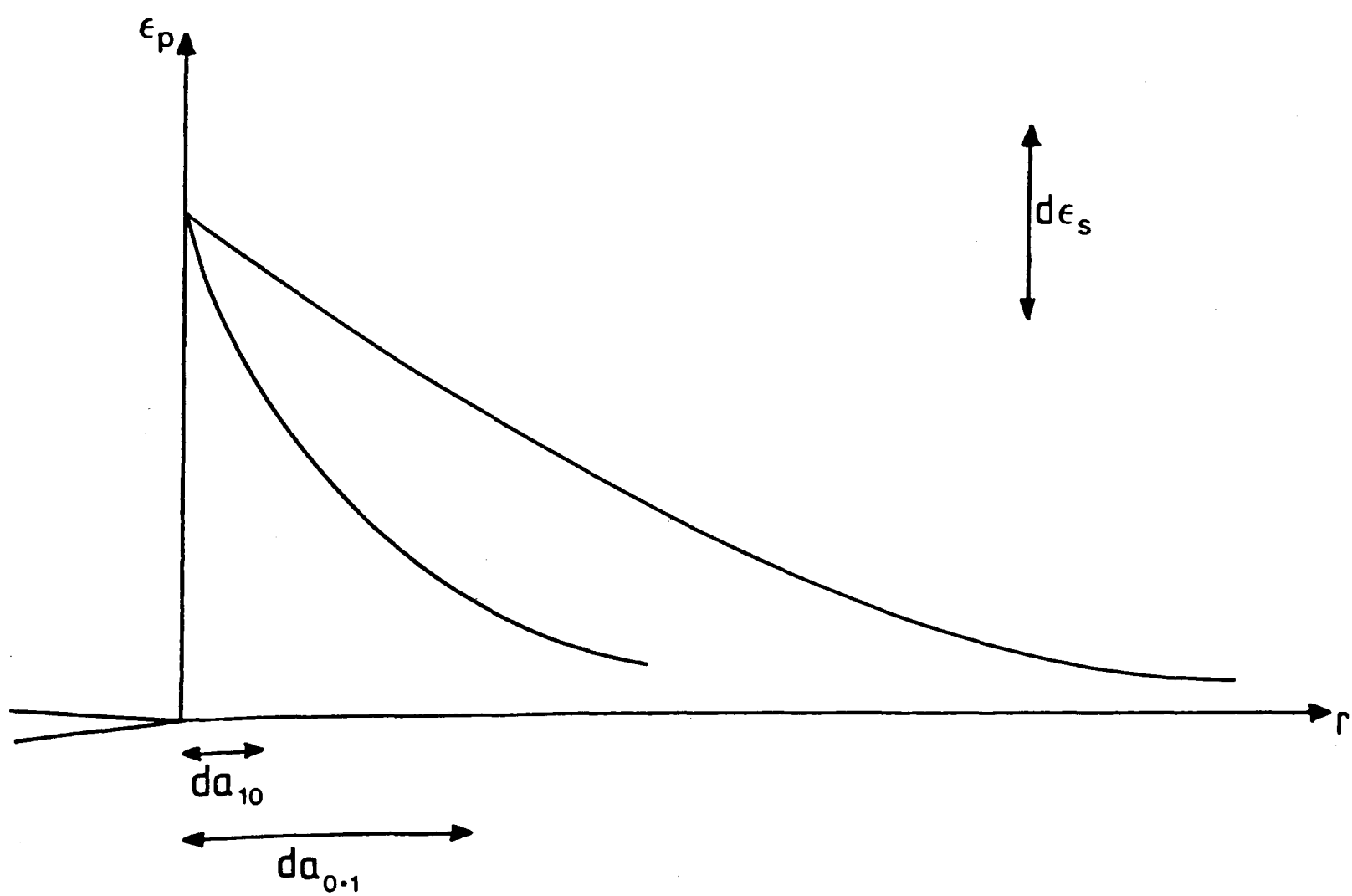
$$\text{and } da_{10} (\text{high } R) \ll da_{0.1} (\text{high } R),$$

therefore giving an increased effect of frequency at higher R ratios.

a)



b)



Chapter 5

Summary

This research has investigated the fatigue behaviour of a single crystal superalloy as a function of the temperature and frequency. The main conclusions obtained from the work are given below.

5.1 Conclusions

1. The plastic zone size associated with a propagating fatigue crack is a function of the stress intensity, temperature and frequency. At elevated temperature the plastic zone size is observed to increase with decreasing frequency at a constant stress intensity. This increase may be modified at higher temperatures where crack tip geometry changes may influence the behaviour.
2. Measurement of the strain gradients within the plastic zone show that the crack growth mechanism is associated with crack tip plasticity at both ambient and elevated temperature. The crack tip strain at ambient temperature is considerably lower than that at elevated temperature where it is approximately independent of the frequency.
3. At elevated temperature lower fatigue thresholds are obtained at higher cycling frequencies, behaviour which it is suggested is associated with oxide induced closure of the crack tip. Similarly, as the frequency decreases an increase in the value

of K_c is observed, this behaviour is consistent with the observed relaxation of the crack tip stresses.

4. At room temperature failure occurs on $\langle 110 \rangle \{111\}$ and crack propagation takes place by the emergence of slip steps at the crack tip.
5. At elevated temperature crack growth occurs on $\{100\}$. The dislocation structure at the tip of a propagating crack is restricted to the γ matrix and crack propagation occurs principally within the γ matrix. This behaviour may be rationalised in terms of the increased strength of the γ' with increasing temperature compared to the decreased strength of the γ matrix resulting from an increase in the temperature. The crack is then constrained to propagate along $\{100\}$ by the presence of the large cuboidal γ' precipitates.
6. At elevated temperature the intermediate crack growth rates are observed to increase with decreasing frequency. In view of the measured strain gradients within the plastic zone, it is proposed that this behaviour is reasonable if failure is considered to take place when a critical strain is achieved at a critical distance ahead of the crack tip.

5.2 Suggestions for Further Work

It has been proposed that the crack tip propagation characteristics of superalloys at high temperature may be influenced by the presence of the γ' precipitates. More specifically, it is suggested that the deformation is concentrated within the γ matrix and that crack growth is then constrained by the surrounding γ'

precipitates. Thus, an investigation of the elevated temperature crack propagation characteristics of a series of microstructures containing varying dispersions of γ' precipitate could specifically examine this hypothesis.

The present work has shown that crack tip stress relaxation at elevated temperature may occur rapidly under the application of symmetrical waveforms. Similar effects may be expected under conditions of a varying rise time and hold time. The rate at which this occurred could be systematically studied using the techniques described in this work. The various interactions of rise time and hold time signals to the crack tip deformation and crack growth rate could therefore be examined.

The extent of stress relaxation is also likely to affect the crack propagation rate and several authors have suggested that this may be due to the influence of cavity nucleation and growth. The introduction of discrete particles into the matrix and the grain boundaries would allow the effects of the observed crack tip stress relaxation, cavity nucleation and growth and crack propagation rates to be explored.

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