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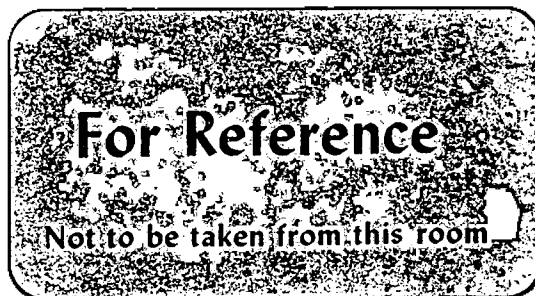
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MODELS OF CREEP CRACK GROWTH IN CERAMICS

M.D. Thouless  
(Ph.D. Thesis)

July 1984



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M. D. Thouless

(Ph. D. Thesis)

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and  
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July 1984

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## MODELS OF CREEP CRACK GROWTH IN CERAMICS

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(Ph. D. Thesis)

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## ABSTRACT

A model is developed to describe the velocity of a dominant crack in poly-crystalline ceramics at elevated temperatures. The crack propagates by the growth and coalescence of cavities in a damage zone at the crack tip. The growth of the cavities is assumed to be constrained by the linearly viscous matrix surrounding the damage zone. The resultant crack velocity is proportional to the applied mode I stress intensity factor ( $K$ ) and inversely proportional to the viscosity of the material ( $\eta$ ). It has a more complex dependence upon the grain size ( $\ell$ ), the cavity spacing ( $\lambda$ ), the damage zone size ( $z$ ) and the critical cavity size for grain facet failure ( $A_f$ ). The magnitude of the stress in the damage zone is very small, but it rises to a peak at the periphery. In the regime away from the influence of either the critical stress intensity factor or the threshold, the predicted velocities agree with published experimental results.

The effect of surface tension upon the crack velocity is shown to be negligible except in the region of the threshold. It is possible that  $A_f$  may be approximated by the condition for the cavity morphology to change from a lenticular to a crack-like shape. This transition occurs at lower values of  $A_f$  as  $(\ell/\lambda)$  decreases or  $K$  increases, consequently there is some resultant deviation from linearity with applied stress.

At elevated temperatures, a ceramic responds visco-elastically to stress; consequently at high crack velocities, the constraint may be dominated by the elastic characteristics of the matrix. An elastic solution is presented which tends to the unconstrained solution at large stresses. At lower stresses a threshold is predicted, but this is shown to be invalid because its existence negates the elastic assumption. In the vicinity of the critical stress intensity factor ( $K_c$ ) some of the grains fail by cleavage along the grain boundaries rather than by cavity growth, this is the basis for a very simple model designed to illustrate the asymptotic behavior of the crack velocity in the region of  $K_c$ .

Finally, some experimental and theoretical work is presented for crack growth in liquid phase sintered materials. The theoretical work is essentially an extension of the original model, adapted for the case where the material properties are dictated by the presence of a liquid phase along the grain boundaries. The experimental results are of only a very tentative nature, and provide the background for proposed future research.

Nomenclature List

|            |                                                                                                              |
|------------|--------------------------------------------------------------------------------------------------------------|
| A          | area fraction of the grain boundary that is cavitated                                                        |
| $A_f$      | critical value of A at which facet failure occurs                                                            |
| $A_{n,f}$  | value of A on the $n^{\text{th}}$ grain at the end of the time interval $\Delta t$                           |
| $A_{n,o}$  | value of A on the $n^{\text{th}}$ grain at the beginning of the time interval $\Delta t$                     |
| $\dot{a}$  | crack velocity                                                                                               |
| B          | $= 16\sqrt{\pi} \Omega D_b \delta_b \zeta F_b^{3/2} / 3kTF_v$                                                |
| b          | Burgers' vector                                                                                              |
| $b_j$      | Burgers' vector of the $j^{\text{th}}$ dislocation from the crack tip                                        |
| $C_s^*$    | rate equivalent of the path independent integral                                                             |
| $C_{m,n}$  | $= \frac{\sqrt{2n}}{2n-(2m-1)} - \frac{2\sqrt{2(n-1)}}{2(n-1)-(2m-1)} + \frac{\sqrt{2(n-2)}}{2(n-2)-(2m-1)}$ |
| $D_b$      | grain boundary diffusivity                                                                                   |
| $D_v$      | lattice diffusivity                                                                                          |
| E          | Young's modulus                                                                                              |
| $F_b$      | $= \sin^2 \psi$                                                                                              |
| $F_v$      | $= (2\pi/3) (2-3\cos\psi+\cos^3\psi)$                                                                        |
| $G^{gb}$   | strain energy release rate when a grain boundary fractures                                                   |
| $G_c^{gb}$ | critical strain energy release rate for grain boundary failure                                               |
| $g(A)$     | $= \frac{8}{5} A^{5/2} - \frac{2}{7} A^{7/2} - \frac{10}{9} A^{3/2} - \frac{4}{3} A^{3/2} \ln A$             |
| K          | Mode I stress intensity factor                                                                               |
| $K_c$      | critical stress intensity factor                                                                             |
| $K_{th}$   | threshold stress intensity factor                                                                            |
| k          | Boltzmann's constant                                                                                         |

x

|            |                                                                                                                            |
|------------|----------------------------------------------------------------------------------------------------------------------------|
| m          | an integer defining a grain position                                                                                       |
| n          | an integer defining a grain position                                                                                       |
| P          | void pressure                                                                                                              |
| R          | distance of the liquid film interface from the grain center                                                                |
| r          | radius of curvature of the void                                                                                            |
| $r_b$      | void radius in the plane of the grain boundary                                                                             |
| s          | ratio of the applied stress on a facet to the sintering stress<br>$= \sigma_a \lambda A^{1/2} / (1-A) 2\gamma_s F_b^{1/2}$ |
| T          | absolute temperature                                                                                                       |
| t          | time                                                                                                                       |
| u          | grain center displacement $= \lambda F_v A^{3/2} / \pi F_b^{3/2}$                                                          |
| $U_n$      | grain center displacement for the $n^{\text{th}}$ grain                                                                    |
| V          | cavity volume                                                                                                              |
| z          | number of grains in the damage zone                                                                                        |
| $\ell$     | grain size                                                                                                                 |
| $\gamma_s$ | surface tension                                                                                                            |
| $\Delta t$ | time for the crack to advance by one grain increment                                                                       |
| $\delta$   | thickness of liquid film in grain boundary                                                                                 |
| $\delta_0$ | initial thickness of liquid film in grain boundary                                                                         |
| $\delta_b$ | grain boundary thickness                                                                                                   |
| $\delta_c$ | critical thickness of intergranular liquid film for crack advance                                                          |
| $\zeta$    | $= 1 + 2D_v \lambda / \pi D_b \delta_b$                                                                                    |
| $\eta$     | viscosity                                                                                                                  |

|                |                                                                            |
|----------------|----------------------------------------------------------------------------|
| $\eta_b$       | bulk viscosity for a liquid phase sintered material                        |
| $\eta_l$       | viscosity of liquid phase in a liquid phase sintered material              |
| $\theta$       | angle a grain boundary makes with the crack                                |
| $\theta_c$     | value of $\theta$ below which grain boundaries fail by cleavage            |
| $\lambda$      | cavity spacing                                                             |
| $u$            | $= [EF_v / (\sqrt{2} \pi^2 F_b^{3/2} (1-\nu^2))] (\lambda/l) (1/\sigma_a)$ |
| $\nu$          | Poisson's ratio                                                            |
| $\xi$          | $= 1 + D_v/D_b \delta_b$                                                   |
| $\sigma$       | stress                                                                     |
| $\sigma_a$     | applied stress on a facet                                                  |
| $\sigma_c$     | compressive stress acting on a facet                                       |
| $\sigma_{a,m}$ | applied stress on the $m^{\text{th}}$ facet ahead of a crack               |
| $\sigma_{c,m}$ | compressive stress on the $m^{\text{th}}$ facet ahead of a crack           |
| $\psi$         | dihedral angle                                                             |
| $\Omega$       | effective atomic volume                                                    |
| $\Omega_s$     | normalized sintering stress = $1/s$                                        |

## I. INTRODUCTION

### 1.1 Background

Ceramics combine many useful properties that make them appealing for use in a broad range of applications. In particular, their high melting points confer upon them a resistance to creep far superior to that of metals. Consequently, there is much interest in the development of these materials for use as components in high temperature engines. In order to ensure that the optimum behavior can be obtained in such applications, it is important to understand how ceramics behave at elevated temperatures, and in particular to elucidate the failure mechanisms that operate.

At low temperatures, below about 1000°C, ceramics fail in a brittle manner by the catastrophic propagation of cracks. These originate at flaws which are inherent in the fabricated material, or are introduced during service. Brittle fracture is now fairly well understood. Of a more complex nature is the process of failure at higher temperatures known as creep rupture. Among the complexities associated with this behavior, is the fact that ceramics can undergo extensive permanent deformation by creep processes at high temperatures.

Recent experimental observations<sup>[1-8]</sup> suggest that at least two types of failure may be of importance in single phase ceramics. The first of these is of pertinence when a dominant crack exists in a homogeneous material. The crack can remain sharp and grow under the influence of an applied stress (Fig. 1). As the crack grows, the stress intensity factor associated with it can increase; when this

exceeds the critical value for the material, catastrophic failure results. At low values of the applied stress intensity factor, a threshold is observed below which the crack does not grow. Under these conditions, the crack opens and blunts in a fashion dictated by the bulk creep properties of the material. In conjunction with the blunting, intense damage is observed in shear bands inclined at about  $30^\circ$  to the crack axis at its tip (Fig. 2).<sup>[1]</sup>

The second type of failure is of importance in the absence of a major flaw. This type of failure is the subject of current experimental research.<sup>[8]</sup> Preliminary evidence suggests that very small cracks may be nucleated in regions of inhomogeneities (Fig. 3). These appear to grow rapidly even when the stress intensity factor is below the threshold observed for large cracks. When the cracks have grown sufficiently, so as to leave the region of influence of the inhomogeneity, they usually blunt, cease to grow and develop shear bands. The interaction of these shear bands can ultimately cause failure along a serrated fracture surface (Figs. 4,5). Although the models presented in this thesis concentrate upon the propagation of a dominant crack, possible implications for the other type of failure are discussed.

## 1.2 Previous Work on Crack Propagation

A variety of mechanisms have been proposed for the growth of cracks at high temperatures. These exist for both liquid phase sintered ceramics<sup>[9]</sup> and single phase materials.<sup>[10-18]</sup> They may be divided into direct crack extension models and damage zone models. A further

division is made into those mechanisms that assume the material at the crack tip behaves rigidly, and those that assume it behaves elastically.

#### 1.2.1 Direct Propagation of a Crack

The direct propagation of an isolated crack, growing along the grain boundary of a bi-crystal (Fig. 6), has been analyzed in Refs. 10 to 12. Under the influence of an applied stress, the atoms diffuse along the crack surface and into the grain boundary. There are two possible assumptions regarding the stress field ahead of the crack. The simplest pertains if the atoms from the crack surface are deposited as a uniform layer along the grain boundary. In this case, the grain boundary thickening is accommodated by a pure rigid body displacement with no deformation of the grains,<sup>[10,11]</sup> and the stress field is parabolic. In a more advanced model<sup>[11,12]</sup> the grains behave elastically, and the grain boundary thickening varies as a function of position ahead of the crack tip. The stress field, resulting from the elastic deformation of the grains, is superimposed upon that predicted from the applied stress acting on the crack. It is this modified stress field that dictates the crack growth (Fig. 6). The pertinence of this type of model is restricted, because observations of fracture surfaces<sup>[1]</sup> (Figs. 7,8) suggest that crack growth in ceramics occurs by a damage zone process. The two figures show creep fracture surfaces in a single phase alumina, and illustrate the intergranular cavities typically associated with a damage zone mechanism.

### 1.2.2 Damage Zone Models

In damage zone models it is assumed that cavities develop ahead of the main crack in a region known as the damage zone (Fig. 9). The cavities grow under the influence of the crack tip stress field until they coalesce with the crack tip. This causes incremental crack advance. The crack growth rate is then dictated by the characteristics of the cavity growth mechanisms, and by the magnitude of the crack tip stress field. A review of these mechanisms has been given by the present author<sup>[9]</sup> and elsewhere,<sup>[1]</sup> so only a very brief summary is presented here. Depending upon the material and stress level, cavity growth may occur in the damage zone by diffusion<sup>[20-28]</sup> and/or plastic flow.<sup>[29-32]</sup> In the diffusional regime, several modes of damage exist. It may consist of either equilibrium shaped<sup>[24-28]</sup> or crack-like cavities<sup>[11,12]</sup> (Fig. 10), growing in arrays, independently or cooperatively.<sup>[21-23]</sup>

The propagation of a crack into a damage zone has generally been analyzed by neglecting the influence of the damage on the stress field. Models of this type<sup>[13-16]</sup> have been generalized by Wilkinson and Vitek.<sup>[13]</sup> The growth rate of the cavities can be expressed as a function of the local stress at the cavity location (Fig. 9), the stress generally exhibits an inverse power law dependence on the distance from the crack tip (the exact form being determined by the deformation law for the material). These models yield a general expression for the crack velocity ( $\dot{a}$ );

$$\dot{a} \propto K^\beta$$

or

(1.1)

$$\dot{a} \propto C_S^{*\beta'}$$

where  $K$  is the stress intensity factor,  $C_S^*$  is the rate equivalent of the path independent integral, and the exponents  $\beta$  and  $\beta'$  depend on the cavity growth mechanism. Such models represent an upper bound for the crack velocity.

It was first recognized by Dyson<sup>[33]</sup> that the growth of cavities must be compatible with the deformation of the undamaged matrix, and constraining stresses result from any incompatibilities.<sup>[33-35]</sup> Similar effects occur in the present problem of a crack extending into a damage zone. The stress field ahead of the crack (Fig. 9), and consequently the crack velocity, are modified by the damage. This concept of constrained crack growth is of central importance to the development of the models in this thesis.

The modifications to the crack growth rate that occur in the presence of a stress redistribution, caused by the cavitation damage, have not been comprehensively addressed. Hutchinson<sup>[36]</sup> has analyzed the stress field due to a stationary crack with a cavitation zone surrounding it, but no attempt was made to derive a crack velocity. Raj and Baik<sup>[17]</sup> have developed a rigorous model for a crack with a co-planar damage zone advancing along the grain boundary of an elastic bi-crystal. Cocks and Ashby<sup>[18]</sup> have derived a simplified constrained crack growth model for a polycrystalline material subject to power law creep. This latter model invokes a generalized damage mechanism, and requires that the damage rate be dictated by the creep rate of the material

ahead of the crack tip, [37,38] as if there were no damage. This premise has been shown to result in an upper bound constraint and consequently a lower bound crack velocity. [19]

### 1.3 Work to be Addressed in this Dissertation

The primary emphasis of this work is focussed on the development of a comprehensive model for the propagation of a dominant crack in a polycrystalline ceramic. The crack is assumed to propagate into a damage zone which is subjected to a constraint from the surrounding matrix. The growth laws for the cavities are coupled with the stress field of the crack, as modified by the constraint, to yield analytical expressions for the crack velocity. The major advantage of this model over previous ones is that it incorporates the effects of a linear viscous matrix, as pertinent to most polycrystalline ceramics at creep rupture temperatures. [39-40] Initially, crack growth in single phase ceramics is analyzed using approximations which permit investigation of the effect of the damage zone size. To this end, the surface tension of the ceramic is ignored. The results show very encouraging agreement with experimental observations.

The next two chapters are modifications of the basic model. In Chapter III, the surface tension is included in the analysis for a damage zone of one grain length in size; the influence of a threshold stress intensity factor can consequently be calculated. Visco-elastic effects, which become important at high crack velocities, are analyzed in Chapter IV. Furthermore, a very simple and basic model is also presented to illustrate the effect of the critical stress intensity factor.

In Chapter V, the analysis is extended to the case where a glassy second phase exists at the grain boundaries. This is done in an attempt to elucidate the behavior of one type of micro-crack that may cause failure in the absence of a dominant crack. The analysis should be applicable to the growth of cracks in liquid phase sintered ceramics. Finally, in the last chapter, a brief discussion of the future work that should be conducted in order to make further advances in the field is presented.

## II. THE CRACK GROWTH MODEL

A model for crack growth in the post-threshold regime is presented. Damage in the form of grain boundary cavitation is assumed to occur ahead of the crack tip<sup>[1]</sup> (Figs. 7,8). In this section the analysis is performed for a linearly viscous, single phase ceramic; and the implications for the resulting crack velocity are then discussed.

### 2.1 General Considerations

The crack extension, although macroscopically Mode I, locally occurs on boundaries inclined to the crack surface (Fig. 11). Such boundaries are subject to both shear and normal stresses. The shear stresses cause grain boundary sliding and are considered to occupy a primary role in cavity initiation,<sup>[42,43]</sup> while the normal stresses dictate cavity growth and coalescence. The sliding induced nucleation is not given explicit consideration in the present model, but is implicitly involved in determining the damage zone size. The emphasis of the present analysis is upon the normal stresses, in the presence of freely sliding boundaries, and their influence on damage growth.

The problem may be approximated by assuming that the damage zone consists of an array of grains coplanar with the crack and embedded in a linearly viscous matrix (Fig. 12).<sup>\*</sup> Grain boundary sliding between grains in the damage zone is assumed to accommodate the damage gradient

---

<sup>\*</sup>The normal stresses on the grain boundaries are evidently overestimated in such a square grain model. However, for random grain boundary inclinations, the average normal stress is only about 12% less than that for square grains.

and hence, to equilibrate the stress on each grain. The cavities on a grain boundary are then expected to be of an approximately uniform size, in accord with observation (Figs. 7,8). A direct consequence of this premise is that the crack must advance by grain facet length increments, when the cavities on the grain boundary adjacent to the crack tip either reach a critical size or coalesce. This assumption is supported by observations<sup>[1]</sup> that the cracks always terminate at grain edges (Fig. 1).

The tensile stress in the damage zone immediately ahead of the crack is expected to be low,\* as reinforced by observations of equilibrium-shaped diffusional cavities on fracture surfaces<sup>[1]</sup> (Figs. 7,8). High stress mechanisms of cavity growth, such as plastic or independent diffusional growth, are considered unlikely. A suitable model considers grain boundaries containing regular arrays of equilibrium-shaped boundary cavities<sup>[25]</sup> (Fig. 13). Expressions relating the growth rate of equilibrium-shaped voids to the local stress on the grain boundaries ahead of the crack thereby provide the initial basis for the analysis. The role of potential crack-like transitions is examined in Chapter III.

The local stresses, as influenced by both the crack and the viscous constraint, are determined and used to obtain a set of expressions relating the cavity size, position ahead of the crack tip, and the crack velocity. Thereafter, the steady state crack velocity can

---

\*A low stress on the grain ahead of the crack is expected from the constraint on the damage zone (Section 2.4).

be deduced, provided that the damage zone size and the critical cavity size for crack advance are specified.

## 2.2 Cavity Growth

The rate of increase in the volume ( $V$ ) of a cavity within the damage zone, subject to a stress  $\sigma$ , may be obtained directly from Needleman and Rice<sup>[32]</sup> (this is the corrected form of an expression first given in Raj and Ashby<sup>[25]</sup>) as;

$$\dot{V} = \left( \frac{8\pi\Omega D_b \delta_b \zeta}{kT} \right) \left( \frac{\sigma - 2\gamma_s F_b^{1/2} (1-A)/\lambda A^{1/2} + P}{2\ln(1/A) - (1-A)(3-A)} \right) \quad (2.1)$$

where

$$\zeta = 1 + \frac{2D_v \lambda}{\pi D_b \delta_b}$$

and  $A$ , the area fraction of cavitated grain boundary, is related to the cavity spacing ( $\lambda$ ) and the void radius in the plane of the grain boundary ( $r_b$ ) by,<sup>[25]</sup>

$$A = (r_b/\lambda)^2 \quad (2.2)$$

$\Omega$  is the effective atomic volume,  $D_b$  and  $D_v$  are the grain boundary and lattice diffusivities,  $\delta_b$  is the grain boundary thickness,  $k$  is Boltzmann's constant,  $T$  is the absolute temperature,  $P$  is the internal pressure of the void, and  $F_b$  is a geometrical term involving the dihedral angle of the cavity. Cavity growth results in a grain center displacement,  $u$ , related to the volume of the cavities by:<sup>[17]</sup>

$$u = V/\pi\lambda^2 \quad (2.3)$$

Hence, invoking the geometric relations<sup>[17]</sup>  $V = r^3 F_v$ , and  $r_b^2 = r^2 F_b$ , where  $F_v = (2\pi/3) (2 - 3 \cos\psi + \cos^3\psi)$ ,  $F_b = \sin^2\psi$ , and  $\psi$  is the dihedral angle, the center displacement becomes,

$$u = \frac{\lambda F_v A^{3/2}}{\pi F_b^{3/2}} \quad (2.4)$$

giving a displacement rate

$$\dot{u} = \lambda \left( \frac{3F_v}{2\pi F_b^{3/2}} \right) A^{1/2} \dot{A} \quad (2.5)$$

Combining equations (2.1), (2.2) and (2.3), the rate of change of cavitated grain boundary area becomes,

$$\dot{A} = \left( \frac{8\Omega D_b \delta_b \zeta}{\lambda^3 kT} \right) \left( \frac{2\pi F_v^{3/2}}{3F_v} \right) \left( \frac{\sigma - 2\gamma_s F_b^{1/2}(1-A)/\lambda A^{1/2} + P}{A^{1/2}(2\ln(1/A) - (1-A)(3-A))} \right) \quad (2.6)$$

Equation (2.6) can be used to describe the growth of cavities on any grain ahead of the crack, provided that the stress on that grain is prescribed. In this context it is recognized that, for a slowly moving crack, a compressive stress ( $\sigma_c$ ) develops normal to cavitating grain boundaries, because of the constraint imposed by the viscous matrix. (For fast cracks an elastic or visco-elastic constraint would be valid.) Hence, the stress in equation (2.6) must be a resultant stress

$$\sigma = \sigma_a - \sigma_c \quad (2.7)$$

where  $\sigma_a$  is the stress associated with the unconstrained crack tip field.

The subsequent analysis of crack growth will be conducted, for purposes of analytic tractability, on the premise that  $\sigma$  appreciably exceeds the sintering stress ( $\sigma \gg 2(1-A)\lambda_s F_b^{1/2}/\lambda A^{1/2}$ ) and that the internal void pressure ( $P$ ) is negligible. The effect of the sintering stress is examined in the following chapter.

### 2.3 Crack Growth

The constraint in the crack tip damage zone can be modelled by introducing a result from dislocation mechanics. A series of edge dislocations are placed ahead and in the plane of the crack as in Fig. 14 to represent the displacements of each grain. The sum of the Burgers' vectors at each point must correspond to the displacement at that point,

$$u_n = \sum_{j=n}^z b_j \quad (2.8)$$

where  $u_n$  is the displacement of the  $n^{\text{th}}$  grain and  $b_j$  is the Burgers' vector of the  $j^{\text{th}}$  dislocation from the crack tip.

In this model, for a viscous material, the compressive stress at a distance  $x$  from the crack tip caused by the  $n^{\text{th}}$  dislocation is\*:

$$\sigma_c(x) = \frac{\eta b_n}{3\pi(n\ell-x)} \sqrt{\frac{n\ell}{x}} \quad (2.9)$$

The total compressive stress at this point is given by:

$$\sigma_c(x) = \sum_{n=1}^z \frac{\eta b_n}{3\pi(n\ell-x)} \sqrt{\frac{n\ell}{x}} \quad (2.10)$$

Taking the stress at the center of the  $m^{\text{th}}$  grain facet as a representative stress for that facet<sup>[44]</sup> then gives;

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\*The above expression is derived from the elastic solution<sup>[11,12]</sup> for the residual misfit stress  $\sigma(x)$  at an arbitrary distance  $x$  from the crack, introduced by an edge dislocation of Burgers' vector,  $b$ , located at  $x=x'$ :

$$\sigma(x) = \frac{Eb}{4\pi(1-\nu^2)(x-x')} \sqrt{\frac{x'}{x}}$$

$$\sigma_{c,m} = \frac{2\eta}{3\pi\ell} \sum_{n=1}^z \frac{\dot{b}_n}{2n-(2m-1)} \sqrt{\frac{2n}{(2m-1)}} \quad (2.11)$$

However, the misfit displacements are related by,

$$\dot{b}_n = \dot{u}_n - \dot{u}_{n+1} \quad (2.12)$$

and therefore,

$$\sigma_{c,m} = \frac{2\eta}{3\pi\ell} \sum_{n=1}^z \frac{\dot{u}_n - \dot{u}_{n+1}}{2n-(2m-1)} \sqrt{\frac{2n}{2m-1}} \quad (2.13)$$

Since  $\dot{u}_{z+1} = 0$ , it can be readily demonstrated that,

$$\sum_{n=1}^z \frac{\dot{u}_n - \dot{u}_{n+1}}{2n-(2m-1)} \sqrt{\frac{2n}{2m-1}} = \sum_{n=1}^z \frac{\dot{u}_n}{\sqrt{2m-1}} \left( \frac{\sqrt{2n}}{2n-(2m-1)} - \frac{\sqrt{2(n-1)}}{2(n-1)-(2m-1)} \right) \quad (2.14)$$

so that,

$$\sigma_{c,m} = \frac{2\eta}{3\pi\ell\sqrt{2m-1}} \sum_{n=1}^z \dot{u}_n \left( \frac{\sqrt{2n}}{2n-(2m-1)} - \frac{\sqrt{2(n-1)}}{2(n-1)-(2m-1)} \right) \quad (2.15)$$

Incorporating the displacement rate from equation (2.5) and noting that the viscosity is given by<sup>[45,46]</sup>

$$\eta = \frac{kT\ell^3}{12\pi\Omega D_b \delta_b \xi} \quad (2.16)$$

where  $\xi = (1 + D_v\ell/D_b\delta_b)$ , the compressive stress becomes

$$\sigma_{c,m} = \left( \frac{kT\ell^2}{18\pi^2\Omega D_b \delta_b \xi \sqrt{2m-1}} \right) \left( \frac{3F_v\lambda}{2\pi F_b^{3/2}} \right) \sum_{n=1}^z A_n^{1/2} \dot{A}_n \left( \frac{\sqrt{2n}}{2n-(2m-1)} - \frac{\sqrt{2(n-1)}}{2(n-1)-(2m-1)} \right) \quad (2.17)$$

The cavitated area pertinent to equation (2.17) can be obtained by combining equations (2.6) and (2.7), for the  $m^{\text{th}}$  grain, (and ignoring the  $\gamma_s$  and  $P$  terms for analytic tractability) as

$$\dot{A}_m = \left( \frac{16\pi\Omega D_b \delta_b \zeta F_b^{3/2}}{3kT\lambda^3 F_v} \right) \left( \frac{\sigma_{a,m} - \sigma_{c,m}}{A_m^{1/2} (2\ln(1/A_m) - (1-A_m) (3-A_m))} \right) \quad (2.18)$$

Then, by combining equations (2.17) and (2.18) to eliminate  $\sigma_{c,m}$ , the extent of cavitation on the grain boundaries within the damage zone can be represented by the expression,

$$\left\{ 2\ln(1/A_m) - (1-A_m) (3-A_m) \right\} A_m^{1/2} \dot{A}_m + \left( \frac{4\ell^2 \zeta}{9\pi^2 \lambda^2 \xi \sqrt{2m-1}} \right) \sum_{n=1}^z A_n^{1/2} \dot{A}_n \\ \times \left( \frac{\sqrt{2n}}{2n-(2m-1)} - \frac{\sqrt{2(n-1)}}{2(n-1)-(2m-1)} \right) = \left( \frac{16\pi\Omega D_b \delta_b \zeta F_b^{3/2}}{3kT\lambda^3 F_v} \right) \sigma_{a,m} \quad (2.19)$$

or

$$\int_{A_{m,0}}^{A_{m,f}} A_m^{1/2} \left\{ 2\ln(1/A_m) - (1-A_m) (3-A_m) \right\} dA_m + \left( \frac{4\ell^2 \zeta}{9\pi^2 \lambda^2 \xi \sqrt{2m-1}} \right) \\ \times \sum_{n=1}^z \int_{A_{n,0}}^{A_{n,f}} \left( \frac{\sqrt{2n}}{2n-(2m-1)} - \frac{\sqrt{2(n-1)}}{2(n-1)-(2m-1)} \right) A_n^{1/2} dA_n \\ = \left( \frac{16\pi\Omega D_b \delta_b \zeta F_b^{3/2}}{3kT\lambda^3 F_v} \right) \sigma_{a,m} \int_0^{\Delta t} dt \quad (2.20)$$

The limits of integration are obtained by assuming that the crack advances at a constant average velocity ( $\dot{a}$ ) in grain size increments,

such that

$$\dot{a} = \ell / \Delta t$$

where  $\Delta t$  is the time interval between crack advance increments. The cavity area on the  $n^{\text{th}}$  grain ahead of the crack is  $A_{n,0}$  at the beginning of the time interval ( $\Delta t$ ), and at the end of the interval it increases to  $A_{n,f}$ . The crack advances when the cavity area on the first grain reaches a critical value ( $A_{1,f}$ ) at which the cavities link by either brittle fracture of the ligament, or by rapid crack-like growth.

For steady-state growth of the crack (i.e. a damage zone size uniquely related to the velocity), the following conditions obtain

$$A_{n,0} = A_{n+1,f}$$

$$A_{z,0} = 0$$

$$\sigma_{a,m} = K / \sqrt{\pi(2m-1)\ell}^*$$

Utilizing these conditions, equation (2.20) can be integrated to obtain;

$$(\sqrt{2m-1}) \left\{ g(A_{m,f}) - g(A_{m+1,f}) \right\} + \frac{8\zeta(\ell/\lambda)^2}{27\pi^2\zeta} \sum_{n=1}^z C_{m,n} A_{n,f}^{3/2} = \frac{BK\Delta t}{\lambda^3\sqrt{\ell}} \quad (2.21)$$

where,

$$g(A) = \left(\frac{8}{5}\right)A^{5/2} - \left(\frac{2}{7}\right)A^{7/2} - \left(\frac{10}{9}\right)A^{3/2} - \left(\frac{4}{3}\right)A^{3/2} \ln A$$

$$C_{m,n} = \left( \frac{\sqrt{2n}}{2n-(2m-1)} - \frac{2\sqrt{2(n-1)}}{2(n-1)-(2m-1)} + \frac{\sqrt{2(n-2)}}{2(n-2)-(2m-1)} \right)$$

$$B = \frac{16\sqrt{\pi}\Omega D_b \delta_b \zeta F_b^{3/2}}{3kTF_v}$$

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\*It will be noted that the amplitude of the stress field at the crack tip has been assumed to be dictated by the elastic stress intensity factor, this can be done because the ceramic has been assumed to creep in a linear viscous fashion (see Appendix I).

Note that when  $n=1$ , the last term in the expression for  $C_{m,n}$  must be taken as zero. Equation (2.21) represents a set of  $z$  non-linear simultaneous equations ( $m=1, 2 \dots z$ ) with  $z$  unknowns ( $A_{2,f}, A_{3,f}, \dots, A_{z,f}, \Delta t$ ) which can be solved numerically for given values of  $A_{1,f}$ . The results are most conveniently expressed in terms of the following quantities:

$$\frac{\dot{a} k T \lambda^3}{\Omega_b \delta_b \zeta} = K \sqrt{\ell} (F_b^{3/2}/F_v) G [(\ell/\lambda) (\zeta/\xi)^{1/2}, z, A_{1,f}] \quad (2.22)$$

where  $G$  is a function. Alternatively, equation (2.22) may be expressed in terms of the viscosity (equation 2.16) to yield an expression more readily comparable with experimental measurements;

$$\dot{n} \dot{a} = K \sqrt{\ell} G^* [\ell/\lambda, z, A_{1,f}] \quad (2.23)$$

where, for simplicity of expression,  $\zeta/\xi$  has been set equal to unity and  $F_b^{3/2}/F_v$  has been taken as constant. This result has been plotted in Fig. 15 in the form of a dimensionless velocity ( $\dot{n} \dot{a} / K \sqrt{\ell}$ ) against the damage zone size for various values of the parameters  $\ell/\lambda$  and  $A_{1,f}$ .

It is directly evident that the crack velocity is proportional to the stress intensity factor and inversely proportional to the viscosity. However, its dependence upon the damage parameters is less apparent. Trends in the dimensionless velocity  $\dot{n} \dot{a} / K \sqrt{\ell}$  with the damage zone size  $z$  for various values of the relative cavity spacing ( $\ell/\lambda$ ) and the cavitation area at coalescence ( $A_{1,f}$ ) are depicted in Fig. 15. The corresponding unconstrained crack growth rates are also shown. These are given when the term  $\sigma_c$  in equation (2.7) is equal to zero, so that stress field is the unmodified linear field. It can be shown that the unconstrained crack velocity is given by,<sup>[41]</sup>

$$\dot{a} = \frac{BK \sqrt{\ell}}{\lambda^3 g(A_{1,f})} \sum_{m=1}^z \frac{1}{\sqrt{2m-1}} \quad (2.24)$$

It is observed that there is a marked difference in behavior between the constrained and unconstrained crack velocities for small damage zone sizes, but that the two behaviors become asymptotic at large  $z$  (such that  $\dot{a} \propto z^{1/2}$ ). The influence of the damage zone size on the crack velocity can be rationalized by recognizing that the damage zone is analogous to an extended crack and interacts with the surrounding matrix in a similar fashion. Thus, as the zone length enlarges, so the compliance of the specimen increases and the constraint decreases.

The effect of the constraint also decreases as either  $A_{1,f}$  or  $\ell/\lambda$  decrease, owing to a diminished constraining stress. The influence of  $A_{1,f}$  resides simply in the increased matter transport needed to induce crack advance. In contrast, the effect of  $\ell/\lambda$  appears to arise from the larger cavity growth rates that occur at large  $\ell/\lambda$ . This results in an increased constraint from the viscous matrix, despite the smaller volume of matter displaced prior to cavity coalescence. (Note that in Chapter IV an opposite trend in crack velocity with  $\ell/\lambda$  is obtained when conditions of elastic constraint are pertinent).

A comparison of the model with experimental results from Blumenthal<sup>[1]</sup> is presented in Fig. 16. Most of the requisite parameters have been obtained in the study with the exception of the damage zone size,  $z$ . Satisfactory agreement between the model and data occurs for a zone size of a few grains; this appears to be very reasonable and lends credence to the model.

## 2.4 The Stress Field

An expression for the compressive stress on the grains in the damage zone can be deduced from equations (2.15) and (2.16) as;

$$\sigma_{c,m} = \left( \frac{kT\ell^2}{18\pi^2\Omega D_b \delta_b \xi \sqrt{2m-1}} \right) \sum_{n=1}^z \dot{u}_n \left( \frac{\sqrt{2n}}{2n-(2m-1)} - \frac{\sqrt{2(n-1)}}{2(n-1)-(2m-1)} \right) \quad (2.25)$$

Then, noting that the displacement rate for all grains, including  $m = n$ , can be obtained from equations (2.5) and (2.18) as;

$$\dot{u}_m = \left( \frac{8\Omega D_b \delta_b \zeta}{\lambda^2 kT} \right) \left( \frac{\sigma_{a,m} - \sigma_{c,m}}{2\ell n(1/A_m) - (1-A_m)(3-A_m)} \right) \quad (2.26)$$

equations (2.25) and (2.26) can be combined with the linear viscous unconstrained crack tip stress field,

$$\sigma_{a,m} = \frac{K}{\sqrt{\pi(2m-1)}\ell}$$

to yield an explicit solution for the stress,

$$\sigma_{c,m} = \left( \frac{4}{9\pi^2} \right) \left( \frac{\ell}{\lambda} \right)^2 \left( \frac{\zeta}{\xi} \right) \frac{1}{\sqrt{2m-1}} \sum_{n=1}^z \left( \frac{K/\sqrt{\pi(2n-1)}\ell - \sigma_{c,n}}{2\ell n(1/A_n) - (1-A_n)(3-A_n)} \right) \times \left( \frac{\sqrt{2n}}{2n-(2m-1)} - \frac{\sqrt{2(n-1)}}{2(n-1)-(2m-1)} \right) \quad (2.27)$$

Equation (2.27) represents a set of  $z$  linear simultaneous equations for  $\sigma_{c,m}$  ( $1 \leq m \leq z$ ), plus an infinite set of linear equations for  $\sigma_{c,m}$  ( $z < m$ ). The results, for the beginning of the waiting interval,  $\Delta t$ , expressed in terms of the dimensionless stress ( $\sigma\sqrt{\ell}/K$ ), are plotted against the distance ahead of the crack for several values of  $\ell/\lambda$ ,  $z$  and  $A_{1,f}$  (Fig. 17). It is evident that the constraining stress decreases and hence the stress field ahead of the crack tends to the unconstrained

field as the damage zone size increases, and as  $\ell/\lambda$  and  $A_{1,f}$  decrease, for reasons discussed earlier. Note that, although the specific details of the stress distribution vary considerably inside the damage zone, a maximum is always observed at the damage zone perimeter.

In Fig. 18 the stress field has been plotted for  $A_{1,f} = 0.7$ ,  $z = 4$  and  $\ell/\lambda = 10$ , at various times ( $\delta t$ ) during the crack advance interval ( $\delta t/\Delta t = 0, 0.1$  and  $1.0$ ). At the beginning of this interval ( $\delta t/\Delta t = 0$ ), cavitation exists only on the first three grain boundaries and the maximum stress occurs on the fourth grain. However, in this model, before one tenth of the time interval has elapsed cavitation occurs in the fourth grain and the peak stress translates to the following grain. The peak stress remains at this position until the corresponding point in the following interval when the crack has advanced by one additional grain.

## 2.5 The Effect of Grain Size

It has been shown that, at large damage zone sizes ( $z \gg \infty$ ), the constrained crack velocity tends to the unconstrained (i.e., upper bound) velocity (equation 2.24),

$$\dot{a} = \frac{Bk\sqrt{\ell}}{\lambda^3 g(A_{1,f})} \sum_{m=1}^z \frac{1}{\sqrt{2m-1}}$$

Hence, when  $\lambda$  is independent of  $\ell$ , the crack velocity is predicted to increase with grain size as  $\dot{a} \propto \ell^{1/2}$ .

The deviation between the constrained and unconstrained crack velocities increases as  $z$  decreases and reaches a maximum when  $z = 1$ . For conditions of maximum constraint ( $z = 1$ ), a closed solution for the constrained velocity can be obtained as

$$\dot{a} = \frac{BK \sqrt{\ell}}{\lambda^3 [g(A_{1,f}) + 8\sqrt{2}(\ell/\lambda)^2 A_{1,f}^{3/2} \epsilon/27\pi^2 \epsilon]} \quad (2.28)$$

If  $\lambda$  is grain size independent, the trends in velocity depend upon the magnitudes of  $\ell/\lambda$  and  $A_{1,f}$ . The maximum deviation from the unconstrained case occurs when the first term in the denominator is negligible, and  $\dot{a} \propto \ell^{-3/2}$ . It has been shown<sup>[19,41]</sup> that this lower bound velocity is equivalent to the solution of the crack growth problem when the damage is assumed to be strain controlled.

In conclusion, both direct and inverse relationships between the velocity and the grain size are predicted by this model. Unfortunately, no systematic experimental data that relate the creep crack velocity of ceramic polycrystals to grain size currently exist. It is interesting to note that the inverse relationship arises from the dependence of the diffusional creep rate on grain size.

## 2.6 Conclusion

In this chapter, a damage zone model has been developed for the growth of creep cracks. In the model, the presence of the damage zone is assumed to introduce a viscous constraint upon the crack growth. The crack velocity ( $\dot{a}$ ) is predicted to be of the form (equation 2.23)

$$\dot{a} = \frac{K \sqrt{\ell}}{\eta} G^* [\ell/\lambda, z, A_{1,f}] .$$

The velocity is directly proportional to the stress intensity factor ( $K$ ) and inversely proportional to the viscosity ( $\eta$ ), but its dependence upon the other parameters is more complex. It transpires that two asymptotic forms for the velocity exist, depending upon the importance of the constraint. One asymptote is realized as the ratio

of the grain size to cavity spacing ( $\ell/\lambda$ ) and the critical area fraction of cavities at coalescence ( $A_{1,f}$ ) are decreased, or as the damage zone size ( $z$ ) is increased. Under these conditions, the effect of the constraint becomes negligible, and the crack velocity tends to that determined for unconstrained crack growth. At the other extreme ( $\ell/\lambda$  large,  $A_{1,f} \gg 1$ ,  $z \gg 1$ ), the constraining stresses become very important and hence exert a substantial influence on the crack velocity.

One of the most notable differences between the constrained and unconstrained crack growth rates is the dependence of the velocity upon the grain size. Specifically, when the cavity spacing is independent of the grain size ( $\ell$ ), then  $\dot{a} \propto \ell^{1/2}$  in the absence of a constraint, and  $\dot{a} \propto \ell^{-3/2}$  when a large grain size dependent constraint develops.

### III. THE EFFECT OF THE SINTERING STRESS

#### 3.1 General Considerations

In the previous chapter a model was developed for crack growth in polycrystalline ceramics. In the interests of obtaining an analytical solution and investigating the effect of the damage zone size, the surface tension term was ignored. The limitations of this assumption may be examined by calculating the ratio ( $s$ ) of the applied stress on a facet ( $\sigma_a$ ) to the sintering stress. The sintering stress is the value of stress at which the cavities exhibit a transition between growth and shrinkage. The ratio ( $s$ ) is given by<sup>[20]</sup>

$$s = \sigma_a \lambda A^{1/2} / (1-A) 2\gamma_s F_b^{1/2}. \quad (3.1)$$

where  $\lambda$  is the cavity spacing,  $A$  is the area fraction of cavitated grain boundary,  $\gamma_s$  is the surface tension and  $F_b$  is a geometrical term involving the dihedral angle of the cavity. Obviously cavity growth, and hence crack growth, can only occur if  $s > 1$ , and the approximation of Chapter II is only valid if  $s \gg 1$ . A plot of the ratio of the applied to the sintering stress as a function of the cavity size ( $A^{1/2}$ ) is presented in Fig. 19, for assumed values of  $K$ ,  $\ell$  and  $\gamma_s$ .

The assumption that  $s \gg 1$  appears to be generally applicable for low values of  $\ell/\lambda$ , it is evidently not valid for  $\ell/\lambda \gtrsim 10$ . More generally, the validity of this assumption is dependent upon the material parameters and the stress intensity factor. A rigorous solution for the crack velocity must therefore take adequate account of the sintering stress, and it is the intent of this chapter to present suitable modifications to the basic model.

Of necessity, either the concept of a threshold stress intensity factor, or the effect of cavity nucleation rates must be introduced into any model that includes a sintering stress. The rate of nucleation of cavities in the damage zone decreases exponentially with the stress, resulting in an apparent critical stress.<sup>[25]</sup> This finite cavity nucleation stress provides a natural crack growth threshold in viscous solids, (an alternative threshold arises in elastic solids, as is demonstrated in the following chapter). Specifically, the threshold occurs when the stress on the first grain boundary ahead of the crack tip falls below the cavity nucleation stress.

In the first part of this chapter, a threshold is assumed in order to develop a description of post-threshold growth consistent with the presence of a cavity nucleation stress. Then, to illustrate a basis for the crack-growth threshold, specific consideration is given to the influence of the cavity nucleation rate on crack velocities. Finally the issue of a possible transition from equilibrium shaped cavities to those with a crack-like morphology is addressed.

### 3.2 Crack Growth Rate<sup>[47]</sup>

For reasons of mathematical tractability, the damage zone in this section is assumed to be confined to a single facet ahead of the crack, consistent with the observations of Section 2.3. As before, the viscous constraint on the crack tip damage zone affects the stress,  $\sigma$ , acting upon the damage zone. From equations (2.7), (2.11), (2.16) and (2.18) this can be shown to be given by

$$\sigma = \sigma_a - (\sqrt{2}/\pi)^2 (F_v/F_b^{3/2}) (\lambda/\ell)_n A^{1/2} \quad (3.2)$$

The stress induces a void growth rate within the damage zone,

$$\dot{A} = \left( \frac{4F_b^{3/2}}{9F_v} \right) \left( \frac{\ell}{\lambda} \right)^3 \left( \frac{\zeta}{\xi} \right) \left( \frac{\sigma_a}{n} \right) \left\{ \frac{1 - (\sqrt{2}/\pi^2) (F_v/F_b^{3/2}) (\lambda/\ell) n A^{1/2} \dot{A} - \Omega_s (1-A)/A^{1/2}}{A^{1/2} [2\ell n (1/A) - (1-A)(3-A)]} \right\} \quad (3.3)$$

where  $\Omega_s (1-A)/A^{1/2} = 1/s$  is the normalized sintering stress for the cavities in the damage zone. Rearranging equation (3.3) gives,

$$\left\{ \frac{A^{1/2} [2\ell n (1/A) - (1-A)(3-A)] + (4\sqrt{2}/9\pi^2) (\ell/\lambda)^2 (\zeta/\xi) A^{1/2}}{1 - \Omega_s (1-A)/A^{1/2}} \right\} \dot{A} = \left( \frac{4F_b^{3/2}}{9F_v} \right) \left( \frac{\ell}{\lambda} \right)^3 \left( \frac{\zeta}{\xi} \right) \frac{\sigma_a}{n} \quad (3.4)$$

Crack growth can only occur if  $\Omega_s (1-A)/A^{1/2} < 1$ . Hence, the left-hand side of equation (3.4) can be expanded by the binomial theorem. Making the substitution  $A^{1/2} = x$ , the expansion yields,

$$\sum_{n=0}^{\infty} \left\{ \frac{2\Omega_s^n x^2}{x^n} \sum_{r=0}^n \frac{(-1)^r n!}{r! (n-r)!} x^{2r} \right\} \left\{ -4\ell n x + 4x^2 - x^4 - 3 + \frac{4\sqrt{2}}{9\pi^2} \left( \frac{\ell}{\lambda} \right)^2 \left( \frac{\zeta}{\xi} \right) \right\} \dot{x} = \left( \frac{4F_b^{3/2}}{9F_v} \right) \left( \frac{\ell}{\lambda} \right)^3 \left( \frac{\zeta}{\xi} \right) \frac{\sigma_a}{n} \quad (3.5)$$

Equation (3.5) can now be integrated in the form,

$$\sum_{n=0}^{\infty} 2\Omega_s^n \sum_{r=0}^n \frac{(-1)^r n!}{r! (n-r)!} \int_{x_0}^{x_f} \left\{ x^{2+2r-n} (-4\ell n x - 3 + \frac{4\sqrt{2}}{9\pi^2} \left( \frac{\ell}{\lambda} \right)^2 \left( \frac{\zeta}{\xi} \right) + 4x^{4+2r-n} - x^{6+2r-n} \right\} dx = \left( \frac{4F_b^{3/2}}{9F_v} \right) \left( \frac{\ell}{\lambda} \right)^3 \left( \frac{\zeta}{\xi} \right) \frac{\sigma_a}{n} \quad (3.6)$$

to give

$$\sum_{n=0}^{\infty} 2 \Omega_s^n \sum_{r=0}^n \frac{(-1)^r n!}{r!(n-r)!} \{ F(X_f) - F(X_0) \} = \left( \frac{4F_b^{3/2}}{9F_v} \right) \left( \frac{\ell}{\lambda} \right)^3 \left( \frac{\zeta}{\xi} \right) \frac{\sigma_a \Delta t}{n} \quad (3.7)$$

where,\*

$$F(X) = - \frac{X^{3+2r-n}}{3+2r-n} \left\{ 4\ell nX - \frac{4}{3+2r-n} + 3 - \frac{4\sqrt{2}}{9\pi^2} \left( \frac{\ell}{\lambda} \right)^2 \left( \frac{\zeta}{\xi} \right) \right\} + \frac{4}{5+2r-n} X^{5+2r-n} - \frac{1}{7+2r-n} X^{7+2r-n}$$

The lower limit on the cavity size ( $X_0$ ) is dictated by the crack growth threshold criterion. The specific relation between  $X_0$  and the threshold stress intensity factor  $K_{th}$  is determined by equating the stress on the center of the facet within the damage zone to the sintering stress, whereupon,

$$\sigma_a \equiv K_{th} / \sqrt{\pi \ell} = \Omega_s (1 - X_0^2) / X_0 \quad (3.8)$$

For initial purposes,  $K_{th}$  is arbitrarily selected (e.g. to coincide with experimental observations) and assumed to be abrupt. A more detailed examination of the threshold, involving considerations of cavity nucleation rates within the damage zone is presented in the following section. These considerations provide a rationale for the existence of a relatively abrupt threshold, based on the inability of cavities to nucleate within a finite time, below the threshold. The upper limit  $X_f$  represents the cavity size at which facet failure

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\*If  $r=(n-3)/2$  the first term is replaced by  $\ell nX \{ 2\ell nX - 3 + (4\sqrt{2}/9\pi^2) (\ell/\lambda)^2 (\zeta/\xi) \}$ , if  $r=(n-5)/2$  the second term becomes  $+4\ell nX$ , and if  $r=(n-7)/2$  the last term is  $-\ell nX$ . Note that if  $n=0$ , the function  $F(X)$  is equivalent to the function  $g(A)$  in equation (2.21).

occurs quite abruptly. Herein  $\lambda_f$  is regarded as a variable. However, it may perhaps be specified a priori by the transition to a crack-like morphology, as discussed in Section 3.4. Upon substituting  $\Delta t = \ell/\dot{a}$  and  $\sigma_a = K/\sqrt{\pi\ell}$  the crack velocity can be derived from equation (3.7) as

$$\frac{\dot{a}}{K\sqrt{\ell}} = \left( \frac{4F_b^{3/2}}{9\sqrt{\pi}F_v} \right) \left( \frac{\ell}{\lambda} \right)^3 \left( \frac{\zeta}{\xi} \right) \left\{ \sum_{n=0}^{\infty} 2\Omega_s^n \sum_{r=0}^n \frac{(-1)^r n!}{r!(n-r)!} [F(\lambda_f) - F(\lambda_0)]^{-1} \right\} \quad (3.9)$$

The resultant relative stress,  $s$ , acting upon the damage zone is given by:

$$s = \frac{A^{1/2}}{\Omega_s(1-A)} \left\{ 1 - \frac{4\sqrt{2}}{9\pi^2} \left( \frac{\ell}{\lambda} \right)^2 \left( \frac{\zeta}{\xi} \right) \left[ 1 - \frac{\Omega_s(1-A)}{A^{1/2}} \right] \left[ 2\ln(1/A) - (1-A)(3-A) + \frac{4\sqrt{2}}{9\pi^2} \left( \frac{\ell}{\lambda} \right)^2 \left( \frac{\zeta}{\xi} \right) \right]^{-1} \right\} \quad (3.10)$$

The crack velocity predicted by equation (3.9) has been plotted in Fig. 20, and the resultant stresses acting upon the damage zone are shown in Fig. 21. The shape of these curves is easily rationalized. When the cavities are nucleated, the applied stress must just equal the stress caused by the surface tension, so that  $s = 1$ . A maximum value of  $s$  results because the magnitude of the applied stress is determined by two competing processes. As the ligaments between the cavities are reduced, the stress across the section must increase; but, at the same time, the growth rate of the cavities, and consequently the constraining stress, also increase with cavity size.

### 3.3 The Threshold Stress Intensity Factor<sup>[47]</sup>

In the preceding analysis, it was assumed that a sharp threshold stress exists below which it is impossible to nucleate cavities and hence to propagate a crack. A simple calculation is included in this section to demonstrate the basis for this assumption. The nucleation rate ( $\dot{\rho}$ ) for grain boundary cavities subject to a normal stress,  $\sigma_a$ , is given by<sup>[25]</sup>

$$\dot{\rho} = \frac{4\pi\gamma}{\Omega^{4/3}\sigma_a^3} D_b \delta_b (\rho_{\max} - \rho) \exp \left\{ -\frac{4\gamma^3 F_v}{\sigma_a^2 kT} + \frac{\sigma_a \Omega}{kT} \right\} \quad (3.11)$$

where  $\rho$  is the number of cavities per unit area,  $\rho_{\max}$  is the number of possible nucleation sites per unit area,  $\Omega$  is the atomic volume,  $k$  is Boltzmann's constant and  $T$  is the absolute temperature. This equation can be re-expressed in terms of the viscosity,  $\eta$ ,<sup>[45,46]</sup>

$$\dot{\rho} = \frac{\gamma k T \ell^3}{3\Omega^{7/3}\sigma_a \eta} (\rho_{\max} - \rho) \exp \left\{ \frac{-4\gamma^3 F_v}{\sigma_a^2 kT} + \frac{\sigma_a \Omega}{kT} \right\} \quad (3.12)$$

Near the threshold, a damage zone of a single grain is most plausible, so that  $\sigma_a = K/\sqrt{\pi\ell}$ . With this premise, equation (3.12) can be integrated to give

$$\ln \left[ \frac{\rho_{\max}}{\rho_{\max} - \rho_n} \right] = \frac{\gamma k T \ell^3 \sqrt{\pi\ell} t_n}{3\Omega^{7/3} K \eta} \exp \left\{ \frac{-4\pi\gamma^3 \ell F_v}{K^2 kT} + \frac{K\Omega}{\sqrt{\pi\ell} kT} \right\} \quad (3.13)$$

where  $t_n$  is the time required to nucleate a density of cavities  $\rho_n$ . Experimental observations indicate that a certain cavity density always accompanies crack propagation, suggesting that a minimum density  $\rho_n$  is needed to allow continuous cavity growth in the damage zone. Adopting this hypothesis,  $t_n$  becomes the minimum residence time

needed to observe crack propagation. Consequently, comparison of the residence time with the steady state crack velocity  $\dot{a}$  from equation (3.9) reveals a regime of nucleation limited crack growth, when  $t_n > \ell/\dot{a}_n$ . Nucleation limited crack growth occurs with the velocity,

$$\dot{a}_n = \ell/t_n \quad (3.14)$$

or,

$$\dot{n}\dot{a}_n = \frac{\sqrt{\pi}\gamma kT\ell^5}{3\Omega^{7/3}K\sqrt{\ell}\ln[\rho_{\max}/(\rho_{\max}-\rho_n)]} \exp\left\{\frac{-4\pi\gamma^3 F_v \ell^2}{(K\sqrt{\ell})^2 kT} + \frac{K\sqrt{\ell}\Omega}{\sqrt{\pi\ell} kT}\right\}$$

The resulting plot of  $\dot{n}\dot{a}$  and  $\dot{n}\dot{a}_n$  against  $K\sqrt{\ell}$  is shown in Fig. 22. The development of a very sharp threshold is noted, due to the nucleation limited regime. However, the magnitude of the threshold is several times greater than that observed experimentally.<sup>[1]</sup> The discrepancy probably arises because, (as commented upon in Section 2.1), additional stresses caused by grain boundary sliding transients aid the cavity nucleation process.<sup>[48]</sup>

### 3.4 Effect of Cavity Morphology

Previously, it has been assumed that surface diffusion is sufficiently rapid to maintain continuity of chemical potential over the surface of the cavities. Consequently, since the surface energy is assumed to be isotropic, the cavities exhibit a constant curvature and are referred to as being equilibrium shaped (Fig. 10). If the growth rate of the cavities becomes too rapid (either owing to a high stress, or boundary diffusivity) the surface diffusion cannot maintain a constant chemical potential, and a transition to a crack-like morphology

occurs<sup>[20,22]</sup> (Fig. 10). Since the volume increases less rapidly with growth rate for a crack-like cavity, the constraining stress will diminish, and the growth must accelerate. It is therefore conceivable that the bulk of the time for failure of a grain facet will be from nucleation to transition; the transition point may perhaps be taken as an approximation to the coalescence area  $A_f$ . A schematic of the failure of a facet by this process is shown in Fig. 23. At the transition point a "nose"<sup>[22]</sup> develops on the cavity, which then proceeds to grow in a crack-like fashion. When these protuberances meet, a dimpled surface is left, as is observed experimentally<sup>[1]</sup> (Figs. 7,8). Obviously, if the fracture surface is left at elevated temperatures for an extended period of time, the dimples will be removed by surface diffusion.

The relationship between the relative stress ( $s$ ) and the area ( $A$ ) at which the transition occurs is approximately:<sup>[20]</sup>

$$s = 1.5 + 3.2 \rho(A) \quad (3.15)$$

where,

$$\rho(A) = (1-A) Q(A) \Delta / A^{1/2}$$

$$Q(A) \equiv 3A^{1/2} [2\ln(1/A) - (3-A)(1-A)] / [4(1-A)^3]$$

and  $\Delta$  is the ratio of the surface to boundary diffusivities ( $D_s \delta_s / D_b \delta_b$ ). Equation (3.15) is plotted in Fig. 25.

From equations (3.10) and (3.15) it is possible to determine the conditions for transition and the resultant crack velocity, as illustrated in Figs. 25 and 26. Unfortunately, approximations in the analytical expression for the transition, given above, appear to induce spurious effects into the precise relationships. However, the general

trends are believed to be valid; in particular that the critical value of  $A$  at which the facet fails decreases as  $K$  increases or  $(\ell/\lambda)$  decreases. The observations of Blumenthal<sup>[1]</sup> do indeed suggest that  $A_f$  falls as  $K$  increases. This results in a deviation from linearity of the dependence of the crack velocity on stress (in the example illustrated in Fig. 26 the exponent on the stress becomes 2.5).

### 3.5 Conclusions

The preceding analysis reveals that the existence of a threshold determined by the sintering stress, does not appreciably reduce the post-threshold crack velocity. The calculations of the damage zone stresses are, however, affected by the sintering stress.

When the crack growth is limited by the cavity nucleation rate, then a very precipitous threshold is encountered. If only normal stresses on the grain boundary are considered, the resultant threshold is considerably higher than that observed experimentally. This lends substance to the belief that cavity nucleation and consequently the damage zone size is determined by grain boundary sliding, and not by normal stresses.

Finally, it is shown that a transition to a crack-like morphology may occur on the grain facets in the damage zone. The point of this transition may perhaps be taken as an approximation to  $A_f$ . The transition is suppressed by a high surface diffusivity and grain size to cavity spacing ratio, it is enhanced by high applied stresses.

#### IV. FURTHER CONSIDERATIONS

##### 4.1 Visco-elastic Constraint

In the model developed in the previous two chapters, the damage at the crack tip was assumed to be constrained by a linearly viscous medium. However, the constraint in real systems is expected to be visco-elastic in nature. An estimate of the range of applicability can be made by considering the relaxation time of the material  $\tau_R = G/\eta_s$ , where  $G$  is the shear modulus and  $\eta_s$  is the shear viscosity. Specifically, if the time for the crack to advance by one grain is significantly greater than the relaxation time, then the constraint is dominated by its viscous characteristics. Otherwise, the elastic properties become important. The visco-elastic problem is posed, but not solved, in Appendix II. The elastic solution is presented in this chapter and compared with the unconstrained and viscously constrained solutions.

##### 4.1.1 Crack Growth with Elastic Constraint

In the absence of a sintering stress, the growth rate of cavities in a crack tip damage zone is given by equation (2.1)

$$\dot{A} = \left( \frac{8\Omega D_b \delta_b \zeta}{\lambda^3 kT} \right) \left( \frac{2\pi F_b^{3/2}}{3F_v} \right) \left( \frac{\sigma}{A^{1/2} (2 \ln(1/A) - (1-A)(3-A))} \right)$$

where the stress ( $\sigma$ ) is related to the applied stress ( $\sigma_a$ ), by the relation (equation (2.7))

$$\sigma = \sigma_a - \sigma_c$$

and  $\sigma_c$  is the constraint on the damage zone, imposed by the elastic deformation of the surrounding material.

The constraint is modelled as in Chapter II; the cavitating grains are replaced by edge dislocations of variable Burgers' vectors corresponding to the displacements of the grains caused by cavity growth (Fig. 14). In this chapter, attention will be limited to a single cavitating facet. The stress at the mid-point of this grain is taken as a representative stress for the facet. Therefore, following Section 2.3, the constraining stress is given by

$$\sigma_c = Eb/[\sqrt{2}\pi(1-\nu^2)\ell] \quad (4.1)$$

where  $E$  is Young's modulus and  $\nu$  is Poisson's ratio. This is the elastic equivalent of equation (2.11).

The grain center displacement ( $u=b$ ) can be expressed in terms of  $A$  by equation (2.4)

$$u = \lambda F_v A^{3/2} / \pi F_b^{3/2}$$

Hence,

$$\sigma_c = [E F_v / (\sqrt{2}\pi^2 F_b^{3/2} (1-\nu^2))] (\lambda/\ell) A^{3/2} \quad (4.2)$$

Substituting equation (4.2) into (2.7) and (2.1), and re-arranging gives,

$$\frac{A^{1/2} [2\ell \ln(1/A) - (1-A)(3-A)] \dot{A}}{(1-\mu A^{3/2})} = \left( \frac{16\pi\Omega D_b \delta_b \zeta F_b^{3/2}}{3kT\lambda^3 F_v} \right) \sigma_a \quad (4.3)$$

where,

$$\mu = [E F_v / (\sqrt{2}\pi^2 F_b^{3/2} (1-\nu^2))] (\lambda/\ell) (1/\sigma_a).$$

Equation (4.3) can be integrated as;

$$\int_0^{A_f} \frac{A^{1/2} [2\ell \ln(1/A) - (1-A)(3-A)] dA}{(1-\mu A^{3/2})} = \frac{16\pi\Omega D_b \delta_b \zeta F_b^{3/2}}{3kT\lambda^3 F_v} \sigma_a \int_0^{\Delta t} dt \quad (4.4)$$

The limits of integration have been defined in Section 2.3. The left-hand side of equation (4.4) can be re-expressed in terms of the viscosity given by equation (2.16) to yield,

$$\int_0^{A_f} \frac{A^{1/2} [2\ell n(1/A) - (1-A)(3-A)] dA}{(1-\mu A^{3/2})} = \left( \frac{2F_b^{3/2}}{9F_v} \right) \left( \frac{\zeta}{\xi} \right) \left( \frac{\ell}{\lambda} \right)^3 \frac{\sigma_a}{\eta} \int_0^{\Delta t} dt \quad (4.5)$$

Cavity growth can only occur if the applied stress is larger than the constraint. This condition can be expressed mathematically as  $\mu A^{3/2} < 1$ . If the inequality is satisfied then, as in Section 3.2, the left-hand side of equation (4.5) can be expanded by the binomial theorem and integrated. The integration is facilitated by substituting  $x = A^{1/2}$  and noting that<sup>[49]</sup>

$$\int x^n \ell n x \, dx = \frac{x^{n+1} \ell n x}{n+1} - \frac{x^{n+1}}{(n+1)^2} \quad (4.6)$$

Furthermore, if the applied stress at the center of the cavitating grain is assumed to be representative for the entire facet, then it can be related to the stress intensity factor by  $\sigma = K/\sqrt{\pi \ell}$ . Hence performing the integration in equation (4.5) and re-arranging one obtains:

$$\dot{n}_a = \left( \frac{2F_b^{3/2}}{9\sqrt{\pi}F_v} \right) \left( \frac{\zeta}{\xi} \right) \left( \frac{\ell}{\lambda} \right)^3 K\sqrt{\ell} \left[ \sum_{n=0}^{\infty} \mu^n g_n(A_f) \right]^{-1} \quad (4.7)$$

where,

$$g_n(A_f) = A_f^{(3n+3)/2} \left[ \frac{2\ell n A_f}{3n+3} - \frac{5 + 9n}{(3n+3)^2} + \frac{4A_f}{(3n+5)} - \frac{A_f^2}{(3n+7)} \right]$$

and,

$$\mu = [F_V / (\pi \sqrt{2\pi} (1-\nu^2) F_b^{3/2})] (E\ell / K\sqrt{\ell}) (\lambda/\ell).$$

Equation (4.7) has been plotted in Figs. 27 and 28. The unconstrained solutions, obtained by putting  $n=0$  and dropping all other terms in the summation, and the viscous constraint solutions from Chapter II are also shown on these figures.

#### 4.1.2 Discussion

A threshold stress intensity factor is introduced by the elastic constraint. This threshold is predicted despite the absence of a cavity nucleation stress, and is given by the condition  $\mu A_f^{3/2} = 1$ , therefore,

$$K_{th}\sqrt{\ell} = [F_V / (\pi \sqrt{2\pi} (1-\nu^2) F_b^{3/2})] (\lambda/\ell) E\ell A_f^{3/2} \quad (4.8)$$

When the applied stress intensity factor is lower than the threshold, the predicted compressive stress exceeds the applied stress before the critical cavity size for crack advance can be reached. In practice this threshold is unimportant because the elastic model loses applicability as the velocity decreases, and in the absence of a critical nucleation stress, no threshold exists for the viscously constrained model.

An indication of the range of validity for the elastic model as opposed to the viscous model, can be estimated by comparing the time for the crack to advance by one grain with the relaxation time ( $\tau_R$ ) of the material. For the elastic constraint to be applicable

$$\tau_R = \eta_s / G \approx \eta / E \gg \ell / \dot{a} \quad (4.9a)$$

therefore,

$$\eta \dot{a} \gg E \ell \quad (4.9b)$$

Substituting the values for Young's modulus and the grain size used in Figs. 27 and 28, the condition becomes  $\eta \dot{a} \gg 3 \times 10^5 \text{ Pa m}^{-1}$ ; conversely if  $\eta \dot{a}$  is less than this value, the viscous solution is valid. It should be noted at this point that, at very high crack velocities, the elastically constrained solution tends to the unconstrained solution.

A notable difference between the model of this chapter and the one presented in Chapter II, is that in the present study the constraint becomes more significant as the grain size to cavity spacing ratio increases. Consequently, the crack velocity decreases as  $(\ell/\lambda)$  increases. This difference arises because, at a constant value of  $A$ , the rate of matter deposition into the grain boundary increases with  $(\ell/\lambda)$  but the amount decreases. It is expected that the inclusion of a sintering stress into the elastic model will magnify the effect of  $(\ell/\lambda)$  upon the crack velocity, because the trends are in the same direction.

#### 4.2 The Influence of the Fracture Toughness

A mixed mode of crack propagation, involving both cavity growth and intergranular cleavage, is believed to occur as the applied stress approaches the critical value. The incidence of cleavage increases as the applied stress is increased, and consequently the evidence of cavitation is observed to diminish.<sup>[1]</sup> The propensity for failure of a grain boundary by either mechanism depends upon the properties of that facet, and will vary in a probabilistic fashion. This concept yields the basis for the following simple model.

A crack extending across a cleaving facet is assumed to grow with infinite velocity. Its velocity across the other facets is given by equation (2.28)

$$\dot{a} = BK\sqrt{\lambda}\{\lambda^3[g(A_f) + 8\sqrt{2}(\lambda/\lambda)^2 A_f^{3/2}\zeta/(27\pi^2\xi)]\}^{-1}$$

for a co-planar damage zone of one grain, and in the presence of a viscous constraint. If the probability that a grain boundary will fail at a given stress intensity factor  $K$  is  $P_f(K)$ , so that

$$\int_0^{K_c} P_f(K) dK = 1, \quad (4.10)$$

then the average velocity ( $\dot{a}_{av}$ ) is given by

$$\dot{a}_{av} = \dot{a}/[1-P_f(K)] \quad (4.11)$$

Equations (4.11) and (2.28) can be combined to evaluate the crack velocity as a function of  $K$ , if  $P_f(K)$  is known. A realistic model would account for the statistical variation in the cleavage tendency due to the differences in the strengths of individual grain boundaries. The simple approach, presented here as a first approximation, assumes that the surface energy is constant for all facets, and the random orientation of the boundaries ahead of the crack is responsible for the distribution of  $P_f(K)$ . This model therefore invokes a concept ignored in the earlier portion of the thesis, namely that the grain boundaries are distributed at random angles to the crack. In the previous chapters, it was assumed that the facets were co-planar with the crack. This was done so as to enable the known result for the stress field of a dislocation co-planar with a crack to be used. It was only recog-

nized in passing that the unconstrained stresses normal to the grain boundaries vary as a function of the grain boundary angle.

If all the grain boundary energies are equal, then the boundaries inclined at the steepest angles to the crack will require a greater stress intensity factor to separate them, than those at shallower angles. It is this difference in separation tendency that produces an approximation for the function  $P_f(K)$  to be used in equation (4.11). The orientation of the boundaries will also alter the magnitude of the constraint term. Owing to the additional complexity of such a consideration, it will be ignored in order to present an analysis illustrating the basic principle. Inclusion of the accurate constraint would not fundamentally alter either the stress at which cleavage begins (except if cavity nucleation is inhibited by the constraint), or the important trends. It will simply affect small details of what is in any case a very approximate model.

A grain boundary immediately ahead of the crack is assumed to make an angle  $\theta$  with the crack (Fig. 29).  $\theta$  can take the values  $0 \leq |\theta| \leq \pi/3$  with equal probability. It is further assumed that if the strain energy release rate ( $G^{gb}$ ) for the grain boundary exceeds a critical value  $G_c^{gb}$ , then the facet will fracture in a brittle fashion with no cavity growth. If the crack is subjected to a mode I stress intensity factor ( $K$ ), then  $G^{gb}$  is given by<sup>[50]</sup>

$$EG^{gb} = k_1^2(1-\nu^2) + k_2^2(1-\nu^2) \quad (4.12)$$

where,

$$k_1 = \cos^3(\theta/2) K$$

and

$$k_2 = \sin(\theta/2) \cos^2(\theta/2) K$$

Therefore,

$$EG^{gb}/(1-\nu^2) = K^2 \cos^4(\theta/2) \quad (4.13)$$

Now it is assumed that the macroscopic value of  $K_c$  for the bulk material occurs when  $G^{gb} = G_c^{gb}$ , even on boundaries for which

$\theta = \pi/3$  (this condition ensures that all boundaries in the material fail by a brittle mode). Therefore, if  $\theta = \pi/3$ ,  $G^{gb} = G_c^{gb}$  and  $K = K_c$  then,

$$EG_c^{gb}/(1-\nu^2) = 9K_c^2/16 \quad (4.14)$$

Grain boundaries for which  $\theta = 0$  fail before all others, they fail if

$$K^2 \geq EG_c^{gb}/(1-\nu^2) \quad (4.15)$$

Hence from equations (4.14) and (4.15) it can be shown that at least some of the boundaries will fail in a brittle fashion if

$$K/K_c \geq 0.75 \quad (4.16)$$

It is observed from Fig. 16<sup>[1]</sup> that this is approximately the value of stress intensity factor at which the influence of  $K_c$  is observed.

Equation (2.28) can be modified for the case when all the grain boundaries are inclined at an angle  $\theta$  to the crack. If the variation in the constraining term with respect to the angle is ignored, then the crack velocity is given by,

$$\dot{a} = (K/K_c) BK_c \sqrt{\ell} \cos^3(\theta/2) \{ \lambda^3 [g(A_f) + 8\sqrt{2}(\ell/\lambda)^2 A_f^{3/2} \zeta / (27\pi^2 \xi)] \}^{-1} \quad (4.17)$$

Now consider the application of a stress intensity factor of such a magnitude that all grain boundaries for which  $|\theta| \leq \theta_c$  fail by cleavage. If the grain boundary angles are randomly distributed, the

probability of the facet immediately ahead of the crack failing in a brittle fashion is given by

$$P_f(K) = \theta_c / (\pi/3) = 3\theta_c / \pi \quad (4.18)$$

The remainder of the boundaries ( $\theta_c < |\theta| \leq \pi/3$ ) separate in a sub-critical manner with an average velocity of:

$$\dot{a} = (K/K_c) BK_c \sqrt{\lambda} \{ \lambda^3 [g(A_f) + 8\sqrt{2}(\lambda/\lambda)^2 A_f^{3/2} \zeta / (27\pi^2 \xi)] \}^{-1} \int_{\theta_c}^{\pi/3} \cos^3(\theta/2) d\theta / [\pi/3 - \theta_c] \quad (4.19)$$

Consequently, from equations (4.11), (4.18) and (4.19), the average velocity ( $\dot{a}_{av}$ ) over the entire range of grain boundary angles is given by:

$$\dot{a}_{av} = (K/K_c) BK_c \sqrt{\lambda} \{ \lambda^3 [g(A_f) + 8\sqrt{2}(\lambda/\lambda)^2 A_f^{3/2} \zeta / (27\pi^2 \xi)] \}^{-1} [3\pi / (\pi - 3\theta_c)^2] \int_{\theta_c}^{\pi/3} \cos^3(\theta/2) d\theta \quad (4.20)$$

This equation can be integrated to give the result;

$$\dot{a}_{av} = 2\pi(K/K_c) BK_c \sqrt{\lambda} \{ \lambda^3 [g(A_f) + 8\sqrt{2}(\lambda/\lambda)^2 A_f^{3/2} \zeta / (27\pi^2 \xi)] \}^{-1} \{ [3 - \sin^2(\pi/6)] \sin(\pi/6) - [3 - \sin^2(\theta_c/2)] \sin(\theta_c/2) \} (\pi - 3\theta_c)^{-2} \quad (4.21)$$

The value of  $\theta_c$  can be determined from equations (4.13) and (4.14)

$$3K_c/4 = K \cos^2(\theta_c/2) \quad (4.22)$$

Using a standard trigonometric formula, equation (4.22) can be re-expressed as

$$\theta_c = \cos^{-1}(3K_c/2K - 1) \quad (4.23)$$

for

$$K/K_c \geq 0.75$$

Equations (4.21) and (4.23) can be combined, and the crack velocity plotted as a function of the stress intensity factor, as in Fig. 30.

### 4.3 Conclusions

In this chapter the effect of a visco-elastic constraint on crack growth at elevated temperatures has been investigated. To approximate this behavior, the viscous solution is considered to be valid if (equation 4.9b);

$$\dot{n}a \gg El$$

Otherwise, a model incorporating an elastic constraint is a suitable approximation.

An elastic constraint gives rise to a threshold, but this is of no significance, because in the region of a threshold, the condition for using the elastic model is invalidated. A major difference between the viscous and elastic solutions is, that for a fixed  $A_f$  the dependence upon the grain size to cavity spacing is inverted between the two.

As the applied stress intensity factor is increased towards the critical value for catastrophic failure, the number of cavitating facets decreases and the number that fail in a brittle fashion correspondingly increases. A simple model to illustrate the trends of this effect, shows the expected strong deviation from linearity in the velocity versus stress plot. This occurs when the stress intensity factor is above about 75% of its critical value, and is in accord with experimental observations.<sup>[1]</sup>

## V. HIGH TEMPERATURE CRACK PROPAGATION AND FAILURE IN A CERAMIC CONTAINING A LIQUID PHASE

Amorphous second phases are often present in limited quantities along two grain interfaces and at three grain junctions within polycrystalline ceramics. The amorphous material often derives from sintering additives, or it may be present as an impurity. In both cases, the amorphous regions exert a very important influence on the mechanical behavior at high temperatures. In liquid phase sintered materials, cracks initiate and propagate entirely within the second phase.<sup>[9,51]</sup> Furthermore, even in nominally single phase materials, isolated zones containing glassy phases may be responsible for the nucleation of cracks and ultimate failure.<sup>[1]</sup>

Spherical or oblate cavities<sup>[51,52]</sup> have been observed to nucleate in the liquid film. The nucleation has been attributed to stress concentrations caused by grain boundary sliding transients.<sup>[9,52]</sup> The cavities can then grow, resulting in the failure of individual facets.<sup>[51]</sup> This process occurs under both general loading, and within the damage zone ahead of a crack tip. The latter process is responsible for sub-critical crack propagation.<sup>[9]</sup> A number of preliminary studies have been made of the nucleation of cavities,<sup>[9,51-55]</sup> but the process is not yet satisfactorily understood. The crack propagation stage of failure has not received significant attention.<sup>[9]</sup> In this chapter some preliminary observations of crack growth in a model two phase material ( $\text{Al}_2\text{O}_3/\text{SiO}_2$ ) are presented. Then, based on these and related observations, a damage zone model of crack growth is derived and discussed.

## 5.1 Experimental

### 5.1.1 Procedures

The alloy selected for study ( $\sim 90\% \text{Al}_2\text{O}_3/10\% \text{SiO}_2$ ) contained grains  $\sim 4\mu\text{m}$  in diameter and a continuous amorphous phase. The material was thus chemically stable in the air environment used for testing, and had a grain size similar to the single phase alumina investigated in prior research. A comparison of results should therefore reflect the direct influence of the amorphous phase on the creep rupture process.

Beam specimens ( $25 \times 3 \times 3 \text{ mm}$ ) were tested in four point flexure at constant displacement rates of  $\sim 8.5 \times 10^{-5} \text{ m s}^{-1}$ , at  $900^\circ\text{C}$  and  $1100^\circ\text{C}$ . Some tests were conducted to failure, others were interrupted and the surface examined in the SEM to assess the mode of damage evolution. In the latter tests, the load was retained until the specimens had been cooled to room temperature. In some instances, the specimens were then fractured at room temperature, in order to examine the fracture morphology associated with the creep damage. A few tests were performed on specimens containing indentation cracks, introduced as described elsewhere.<sup>[1]</sup> The indentation load of 20 N was selected so that the cracks were approximately semi-circular and about  $100\mu\text{m}$  in radius.

### 5.1.2 Measurements

All constant strain-rate tests were characterized by the appearance of a peak load, determined by the primary and tertiary modes of creep (Fig. 31). Steady-state conditions were never observed. Characteristic schemes for rupture based on the steady-state creep rate (e.g. the Monkman-Grant relation) cannot, therefore, be applied to the

present material. This contrasts with single phase alumina in which extensive steady state flow precedes rupture,<sup>[1,56]</sup> and the bulk viscosity dictates the crack growth rate.

Some creep data were obtained by compression tests performed on columns (3 x 3 x 8 mm) at a strain rate of  $\sim 8.5 \times 10^{-5} \text{ m s}^{-1}$ . The data obtained are shown in Fig. 32. The associated activation energy, of  $480 \pm 50 \text{ kJ mol}^{-1}$ , is similar to the value for single phase aluminas,<sup>[57]</sup> but is very different from the activation energy for the viscosity of the glassy phase in silicon nitride ( $680 \text{ kJ mol}^{-1}$  in Ref. 58).

### 5.1.3. Observations

The creep damage induced on the tensile surface of the specimens was examined in a scanning electron microscope. A multiplicity of small cracks was observed to nucleate on the tensile side of the specimen above a threshold stress of 50 to 100 MPa (at 1100°C). Certain cracks could be readily associated with an inhomogeneity such as a large pore (Fig. 33) or the edge of the specimen, but other cracks were nucleated in apparently homogeneous material (Fig. 34). The individual cracks then propagated across the tensile surface. Such cracks generally formed and grew more rapidly than indentation cracks placed on the same specimen, so that failure surfaces of indented samples rarely included the indentation. For this reason, attempts at specific measurements of the growth rates of small cracks, using indentation techniques,<sup>[1]</sup> have not been successful at this juncture.

Observations of the crack tip region on the tensile surface, indicated a relatively thick layer in a small zone near the tip (Fig. 35). This layer contained a series of holes, separated by ligaments, and is presumed to consist of amorphous material.

The incidence of crack blunting in this material was investigated by repeatedly reducing the stress on a nucleated crack, and re-examining the region around the crack tip. No significant morphological changes were detected at the crack tip for the loads (0.5 to 0.95 of the growth load) and times (up to 5 hours) used in these tests. Crack blunting effects thus appear to be less dominant than in single phase materials. Consequently, crack propagation times are deemed to contribute more importantly to the overall creep rupture process in materials containing an amorphous phase.

A substantial morphological difference was observed between the creep damaged and brittle fracture regions. This was particularly apparent in the specimens that were fractured at room temperature (Figs. 36 and 37). The crack front can be identified in this figure by a transition in the appearance of the fracture surface. The creep damaged regions were invariably intergranular (Fig. 36) and revealed the presence of amorphous islands. By contrast, the brittle fracture regions were relatively planar and consisted of an appreciable proportion of transgranular failure (Fig. 38). The transgranular nature of the brittle failure was made more evident by etching using a dilute HF solution (Fig. 39). At very high strain rates (and crack velocities), the cracks grew between agglomerates (Fig. 40).

#### 5.1.4 Implications for Crack Propagation

The preceding measurements and observations suggest that creep rupture in materials containing a continuous amorphous phase is strongly influenced by the propagation of intrinsically nucleated cracks. The propagation involves the presence of co-planar damage near the crack tip, manifested as holes in the liquid phase. A co-planar damage zone description of creep crack growth is thus deemed as pertinent to this class of microstructure. Furthermore, the fracture surface observations (Figs. 35 and 36) suggest that the holes nucleate at the three grain channels and grow across the two grain interfaces. A hole spacing dictated by the grain size is thus implied.

The size of the damage zone and its dependence on crack velocity or microstructure cannot be assessed from the present study. Similarly, the crack surface displacement required for the failure of the liquid phase in the damage zone has not been unequivocally deduced. Further work will be needed to assess both of these important characteristics.

### 5.2 Crack Propagation Model

#### 5.2.1 General Considerations

The experimental observations of the preceding section provide the basis for a preliminary crack propagation model. The characteristics of the model are similar to that developed for crack propagation in single phase materials. Specifically, the crack grows into a damage zone contained within a creeping medium. In this instance, the damage consists of holes contained within the amorphous phase. The holes grow by viscous flow of the second phase and

eventually coalesce to allow crack advance. Simultaneously, the medium is subject to creep by a solution/re-precipitation process, dictated by the grain size and the diffusive properties of the amorphous phase.

For preliminary analysis, the damage zone is considered to consist of a single grain; an assumption that allows a straight-forward analytic solution for the crack growth rate. Trends in the crack growth rate with the damage zone size are considered in a following section. The failure of the liquid phase is considered to occur at a critical strain in the liquid or, equivalently, a critical crack surface displacement,  $\delta_c$ . This premise is tantamount to assuming that failure proceeds when the holes reach a critical volume, independent of the crack velocity. A suggestion that liquid phase failure coincides with the onset of a finger-like instability is discussed in Appendix III.

#### 5.2.2 The Crack Growth Rate for a Unit Damage Zone Size

In this section, the cavitation of the liquid in a region, one facet size in length, ahead of the crack is considered. The holes grow from the three grain channel at the extremity of the facet and extend across the intervening two grain interface (Fig. 41). Sliding of the grains is assumed to occur readily, so that the grain facet remains essentially parallel as the holes grow towards their critical coalescence volume.

For a completely wetting liquid, as required for a liquid phase sintered material, the governing equation for the flow of the liquid is given by<sup>[51,59]</sup>

$$\frac{d^2 \sigma(x)}{dx^2} = - \frac{12 \eta_l \dot{\delta}}{\delta^3} \quad (5.1)$$

where  $\sigma$  is the stress,  $\eta_l$  is the viscosity of the liquid phase,  $\delta$  is the liquid thickness,  $\dot{\delta}$  is the rate of change of the thickness, and  $x$  is measured from the grain center. Using the boundary conditions  $d\sigma/dx = 0$  at  $x = 0$ , and  $\sigma = 2\gamma/\delta$  at  $x = \pm R$  (where the liquid interface is at  $x = R$  and  $\gamma$  is the surface tension of the liquid) an equation for the stress can be obtained as

$$\sigma(x) = \frac{6\eta_l \dot{\delta}}{\delta^3} (R^2 - x^2) + \frac{2\gamma}{\delta} \quad (5.2)$$

Volume conservation of the liquid yields a relationship between  $R$  and  $\delta$  given by,

$$\frac{R}{\delta} = \frac{1}{2} \left( \frac{\ell}{\delta_0} - \frac{\pi}{4} \right) \left( \frac{\delta_0}{\delta} \right)^2 + \left( \frac{\pi - 4}{8} \right) \quad (5.3)$$

where  $\delta_0$  is the initial thickness of the liquid phase and  $\ell$  is the grain facet length.

The stress obtained from equation (5.2) must equal the applied stress minus the constraining stress, when averaged over the first grain (cf. equation 2.7). All values are taken at the mid-point of the first grain. Therefore,

$$\frac{2}{\ell} \int_0^R \sigma(x) dx = \frac{K}{\sqrt{\pi \ell}} - \frac{2\sqrt{2}}{3\pi \ell} \eta_b \dot{\delta} \quad (5.4)$$

where  $K$  is the applied stress intensity factor and  $\eta_b$  is the effective viscosity of the polycrystal (due to solution/re-precipitation controlled creep). Substitution of equation (5.1) into (5.4), followed

by integration yields;

$$\left\{ 8\sqrt{\pi} \eta_{\ell} \left( \frac{R}{\delta} \right)^3 + \frac{2\sqrt{2}}{3\sqrt{\pi}} \eta_b \right\} \dot{\delta} = K\sqrt{\ell} - 4\sqrt{\pi} \gamma \left( \frac{R}{\delta} \right) \quad (5.5)$$

Noting that  $R$  is related to  $\delta$  (equation 5.3), a differential equation in  $\delta$  and time ( $t$ ) can be obtained, as,

$$\left\{ 8\sqrt{\pi} \eta_{\ell} \left[ \frac{1}{8} \left( \frac{\ell}{\delta_0} - \frac{\pi}{4} \right)^3 \left( \frac{\delta_0}{\delta} \right)^6 + \frac{3}{4} \left( \frac{\ell}{\delta_0} - \frac{\pi}{4} \right)^2 \left( \frac{\delta_0}{\delta} \right)^4 \left( \frac{\pi - 4}{8} \right) + \frac{3}{2} \left( \frac{\ell}{\delta_0} - \frac{\pi}{4} \right) \left( \frac{\delta_0}{\delta} \right)^2 \left( \frac{\pi - 4}{8} \right)^2 + \left( \frac{\pi - 4}{8} \right)^3 \right] + \frac{2\sqrt{2}}{3\sqrt{\pi}} \eta_b \right\} \dot{\delta} = K\sqrt{\ell} - 4\sqrt{\pi} \gamma \left[ \frac{1}{2} \left( \frac{\ell}{\delta_0} - \frac{\pi}{4} \right) \left( \frac{\delta_0}{\delta} \right)^2 + \left( \frac{\pi - 4}{8} \right) \right] \quad (5.6)$$

It will be noted that the right-hand side of equation (5.6) must be greater than zero if the cavities are to grow. This condition gives rise to a natural threshold stress intensity factor.

Failure of the facet is deemed to occur when the liquid thickness reaches a critical value  $\delta_c$ , at a time  $t = \Delta t$ . Upon facet failure the crack advances by a facet length, so the crack velocity is given by  $\dot{a} = \ell / \Delta t$ . Subject to these conditions, equation (5.6) can be expressed as the integral,

$$\int_{\delta_0}^{\delta_c} \left[ C_1 (\delta_0 / \delta)^6 + C_2 (\delta_0 / \delta)^4 + C_3 (\delta_0 / \delta)^2 + C_4 \right] \left[ 1 - C_6 (\delta_0 / \delta)^2 \right]^{-1} d\delta = \frac{C_5}{\eta_b} \int_0^{\Delta t} dt \quad (5.7)$$

where

$$\begin{aligned}
C_1 &= \sqrt{\pi}(\ell/\delta_0 - \pi/4)^3 (\eta_\ell/\eta_b) \\
C_2 &= 3\sqrt{\pi}(\ell/\delta_0 - \pi/4)^2 [(\pi-4)/4] (\eta_\ell/\eta_b) \\
C_3 &= 3\sqrt{\pi}(\ell/\delta_0 - \pi/4) [(\pi-4)/4]^2 (\eta_\ell/\eta_b) \\
C_4 &= \sqrt{\pi} [(\pi-4)/4]^3 (\eta_\ell/\eta_b) + (2\sqrt{2}/3\sqrt{\pi}) \\
C_5 &= K \sqrt{\ell} - 2\sqrt{\pi}\gamma [(\pi-4)/4] \\
C_6 &= 2\sqrt{\pi}\gamma (\ell/\delta_0 - \pi/4)/C_5
\end{aligned}$$

The denominator on the left-hand side can be expanded by the binomial theorem because of the growth condition  $(C_6(\delta_0/\delta)^2 < 1)$ . Such an expansion enables the integration to be conducted analytically, to give an expression for the crack velocity,

$$\begin{aligned}
\eta_b \dot{a} = C_5(\ell/\delta_0) & \left\{ \sum_{n=0}^{\infty} C_6^n \left[ \frac{C_1}{2^{n+5}} \left[ 1 - \left( \frac{\delta_0}{\delta_c} \right)^{2n+3} \right] + \frac{C_2}{2^{n+3}} \left[ 1 - \left( \frac{\delta_0}{\delta_c} \right)^{2n+3} \right] \right. \right. \\
& \left. \left. + \frac{C_3}{2^{n+1}} \left[ 1 - \left( \frac{\delta_0}{\delta_c} \right)^{2n+1} \right] + \frac{C_4}{2^{n-1}} \left[ 1 - \left( \frac{\delta_0}{\delta_c} \right)^{2n-1} \right] \right] \right\}^{-1} \quad (5.8)
\end{aligned}$$

Consequently, it is possible to calculate a crack velocity if the material properties and the critical liquid thickness are known. Typical trends are plotted in Figs. 42 to 44, for various values of  $\eta_b/\eta_\ell$  and  $\delta_c/\delta_0$ . The specific choices,  $\delta_c/\delta_0 = 21.5$  and  $\delta_c/\delta_0 = 68.2$  have been made, because these values coincide with the upper bound (which occurs when the two menisci come into contact) for  $\ell/\delta_0 = 100$  and 1000 respectively.

The crack velocity is evidently dependent upon a large number of parameters. However, it is apparent from Figs. 43 and 44 that two distinct regimes of behavior exist above the threshold. When the ratio of the bulk viscosity to the viscosity of the intergranular phase

$(\eta_b/\eta_\ell)$  is very large, then  $\eta_b \dot{a}$  is independent of the viscosity ratio. The crack velocity is therefore inversely proportional to  $\eta_b$  and independent of  $\eta_\ell$ . Conversely, at lower values of the viscosity ratio, the velocity is inversely proportional to the viscosity of the intergranular phase and independent of the bulk viscosity. Furthermore, appreciably above the threshold, equation (5.8) can be expressed in these two regimes as:

$$\eta_b \dot{a} \approx 5K\sqrt{\ell}(\eta_b/\eta_\ell) [\sqrt{\pi}(\ell/\delta_o)^2]^{-1} \quad (5.9a)$$

or

$$\eta_b \dot{a} \approx 3\sqrt{\pi}K\sqrt{\ell}(\ell/\delta_o) [2\sqrt{2}(\delta_c/\delta_o - 1)]^{-1} \quad (5.9b)$$

if  $\eta_b/\eta_\ell$  is very large.

An analysis is presented in Appendix IV which reveals that these two cases correspond to the two limits of zero and maximum constraint, respectively.

### 5.2.3 Crack Propagation in an Extended Damage Zone

The analysis of the preceding section has been developed for a damage zone of a single grain. It is possible to elucidate the trends in crack velocity with an increasing damage zone by adapting the analysis of Chapter II. Two limiting regimes of behavior have been identified corresponding to the degree of constraint, as shown in Appendix IV. In this section, the effect of the damage zone size is considered for each limit separately, to give an indication of the trends.

In the absence of a constraint, the growth of the film thickness ( $\delta_m$ ) on the  $m^{\text{th}}$  facet ahead of a crack is given by a modified version of equation (A3.2)

$$\sqrt{\pi} \eta_{\ell} (\ell/\delta_0)^3 (\delta_0/\delta_m)^6 \dot{\delta}_m = K\sqrt{\ell}/(\sqrt{2m-1}) \quad (5.10)$$

Upon integration this yields,

$$(\sqrt{\pi} \eta_{\ell}/5) (\ell/\delta_0)^2 [(\delta_0/\delta_{i,m})^5 - (\delta_0/\delta_{f,m})^5] = K\sqrt{\ell}/(\dot{a}\sqrt{2m-1}) \quad (5.11)$$

where  $\delta_{i,m}$  and  $\delta_{f,m}$  are the film thicknesses on the  $m^{\text{th}}$  facet at the beginning and end of the time interval,  $\Delta t$ .

If there are  $z$  grains in the damage zone then, using the previous notation,  $\delta_{i,z} = \delta_{f,m}$ , and  $\delta_{f,1} = \delta_c$ ; also at steady-state  $\delta_{i,m-1} = \delta_{f,m}$ . Equation (5.11) therefore represents a set of  $z$  equations in  $z$  unknowns. By adding these equations, the crack velocity

$$\dot{a} = [5K\sqrt{\ell}/\sqrt{\pi}(\ell/\delta_0)^2] [1-(\delta_0/\delta_c)^5]^{-1} (1/\eta_{\ell}) \sum_{n=1}^z 1/\sqrt{2n-1} \quad (5.12a)$$

is obtained. For large values of  $z$ , this reduces to the approximation

$$\dot{a} \approx [5K\sqrt{\ell}/\sqrt{\pi}(\ell/\delta_0)^2] [1-(\delta_0/\delta_c)^5]^{-1} (1/\eta_{\ell}) (\sqrt{2z}) \quad (5.12a)$$

The unconstrained crack velocity is therefore proportional to the square root of the grain size, this is the same result as for single phase materials. [19,41]

For the maximum constraint limit, equation (A4.4) becomes,

$$\frac{2\eta_b}{3\sqrt{\pi}\sqrt{2m-1}} \sum_{n=1}^z \dot{\delta}_n \left( \frac{\sqrt{2n}}{2n-(2m-1)} - \frac{\sqrt{2(n-1)}}{2(n-1)-(2m-1)} \right) = \frac{K\sqrt{\ell}}{\sqrt{2m-1}} \quad (5.13)$$

Remembering that  $\delta_{i,m-1} = \delta_{f,m}$ , integration and re-arrangement yields

$$\sum_{n=1}^z C_{m,n} (\delta_{f,n}/\delta_0) = (3\sqrt{\pi}/2) (\ell/\delta_0) (K\sqrt{\ell}/\eta_b \dot{a}) \quad (5.14)$$

where (as in Chapter 1)

$$C_{m,n} = \frac{\sqrt{2n}}{2n-(2m-1)} - \frac{2\sqrt{2(n-1)}}{2(n-1) - (2m-1)} + \frac{\sqrt{2(n-2)}}{2(n-2) - (2m-1)}$$

(for  $n = 1$ , the last term in the expression for  $C_{m,n}$  is taken as 0).

Once again, a set of  $z$  linear equations has been obtained which can be readily solved. In this case there is a very strong dependence of the crack velocity upon the damage zone size; the exponent on  $z$  varies between approximately 1.5 (if  $z$  is small) and infinity.

The normalized crack velocity is plotted in Figs. 45 and 46 as a function of damage zone size, for a number of different values of the important parameters in the two regimes. It will be appreciated that the lines are asymptotes. The detailed solution is dictated by the asymptote which yields the lowest velocity for a given set of parameters. Consequently, as in Chapter II, the maximum constraint is more likely to be applicable for small damage zones, whereas the unconstrained solution is more important for larger damage zones (because the zone behaves as an extended crack).

### 5.3 Discussion

The high temperature crack propagation data in liquid phase materials suggest that the crack velocity is proportional to a relatively high power ( $\geq 6$ ) of the stress intensity.<sup>[9,60-62]</sup> The model apparently indicates an exponent of unity. The difference may be associated with mechanism changes, as argued for single phase materials. There are, however, two parameters whose variation with  $K$  is not specified, namely  $\delta_c$  and  $z$ . Both of these have a substantial influence upon the crack velocity and need further study. Intuition suggests that  $\delta_c$  may

well decrease with an increasing strain rate and hence stress, this would raise the exponent on  $K$ . There is a greater latitude in the effect of the damage zone size; but again, the relation between the applied stress and the damage zone size is not known. A simple assumption, made in prior studies, is that the damage zone is taken to be proportional to the square of the applied stress intensity factor, [9,17] however it is felt that such a simple relationship may not be appropriate.

The threshold condition is given when the stress on the first grain is less than the back stress due to the surface tension of the interface. The threshold stress intensity factor is therefore given by

$$K_{th}\sqrt{l} = 2\sqrt{\pi}\gamma (l/\delta_0) \quad (5.15)$$

This may have an important bearing upon the growth of cracks in liquid phase regions contained in single phase matrices. In areas with thick intergranular films the threshold can be reduced to very low levels indeed, and cracks may grow in such regions even when the applied stress intensity factor is much lower than the observed threshold for the bulk ceramic.

#### 5.4 Conclusions

In this chapter, a model has been presented for the propagation of cracks in materials with a glassy phase along their grain boundaries. The model is an adaptation of that developed in earlier chapters for a single phase material. The magnitude of the resultant velocity is very dependent on a number of parameters. There are two limiting cases for the velocity that can be expressed analytically. When the bulk vis-

cosity is very much larger than the viscosity of the intergranular phase, then the crack velocity becomes

$$\eta_b \dot{a}/K\sqrt{\ell} = (3\sqrt{\pi}/2) (\ell/\delta_0) \left\{ \sum_{n=1}^z C_{m,n} (\delta_{f,n}/\delta_0) \right\}^{-1}$$

Alternatively it is,

$$\eta_b \dot{a}/K\sqrt{\ell} = (5/\sqrt{\pi}) (\delta_0/\ell)^2 \left[ 1 - (\delta_0/\delta_c)^5 \right]^{-1} (\eta_b/\eta_\ell) \sum_{n=1}^z 1/\sqrt{2m-1}$$

Experimental data suggest that the stress dependence should be much higher than that predicted by the model. This discrepancy could arise because the velocity is very dependent upon  $\delta_c$  and  $z$ , and the relationship between these parameters and the stress is not yet understood. Further work is required to address this issue.

Some preliminary experimental results for liquid phase sintered materials have been described. Among the more interesting and significant results were the observed fracture mechanism changes. At low temperatures, transgranular fracture accompanied catastrophic failure. At higher temperatures, intergranular sub-critical crack growth occurred; at very high crack velocities this appeared to become inter-agglomerate in nature.

## VI. FUTURE WORK

### 6.1 Future Research for Single Phase Materials

The theory associated with the growth of dominant cracks in single phase ceramics at high temperatures is believed to be satisfactory within the limits of determining the undefined parameters (Fig. 47). This was confirmed by the experiments of Blumenthal.<sup>[1]</sup> However, one aspect that arose in the model, but has not yet been subject to experimental investigation, is the dependence of the crack velocity upon the grain size. It would be informative to conduct Blumenthal's experiments with a series of aluminas of differing grain size.

Two very important parameters have been retained in the theory as variables that must be determined experimentally; these are the damage zone size and the cavity spacing. Both are connected with the nucleation stage of the process, and are not well understood. Substantial additional work remains to be done in this area to enable it to be fully comprehended.

For reasons described in detail in the introduction, far more work must be applied to study the mechanisms of failure in the absence of a dominant crack. In particular, the influence and development of the shear band process is of prime importance. Experimental investigations in this field are being currently conducted,<sup>[8]</sup> but a parallel theoretical study would also be of value.

### 6.2 Future Research for Liquid Phase Sintered Materials

A major area in which considerable effort could be expended profitably, is in the study of crack growth in liquid phase sintered materials. Some elementary work of a theoretical nature and parallel

preliminary experimental studies have been presented in the previous chapter. It would be very instructive to repeat Blumenthal's experiments, measuring crack velocities as a function of the stress intensity factor, temperature, grain size and amount of liquid phase. The results of Chapter 5 suggest that an investigation into the influence of agglomerates upon the crack growth in commercially available materials would also be of interest.

The effect of strain rate and temperature upon the strengths of materials with a glassy phase have been reported.<sup>[9,63-65]</sup> The strengths generally follow the form shown schematically in Fig. 48. The peak appears to be peculiar to liquid phase sintered materials, it moves to higher temperatures with an increased strain rate. Currently, no completely satisfactory explanation of this phenomenon exists in the literature.

An investigation into the variation of fracture toughness with temperature would be of paramount interest, since a great deal of confusion exists in the literature. There is evidence that the fracture toughnesses at very high strain rates do not in fact increase with temperature,<sup>[66]</sup> but these strain rates are far in excess of those used for observing the peak in fracture strength.

It is possible to obtain unambiguous results for the fracture toughness in the regime where transgranular fracture accompanies catastrophic failure. The critical flaw size can be readily determined from Scanning Electron Microscope pictures, and the critical load can, of course, be measured. This type of experiment should be performed over as wide a range of strain rates and temperatures as is possible.

Trends in the variation of  $K_c$  with temperature should be elucidated, and an attempt made to correlate it with the variation of strengths.

Where  $K_c$  cannot be measured, the effect of inter-agglomerate crack growth could be investigated. The basis of the model developed in the previous section would be unchanged, but the effective grain size would have to be replaced by the agglomerate size. This, coupled with the larger amount of liquid phase present, may profoundly affect the velocity. The simplifying assumption of using the average stress on the first grain ahead of the crack would be invalid for agglomerates because of their size. The model would therefore have to be adapted without this assumption. It is of importance to note that the viscosity is dependent only upon the grain size, and consequently the constraint should be essentially independent of the agglomerate size.

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## APPENDIX I

## THE STRESS FIELD AT A CRACK TIP IN A LINEARLY VISCOUS MATERIAL

A discussion of the stress field at a crack tip in materials subject to elastic deformation and primary or secondary creep is given in a number of Refs. [1-3]. A brief summary of the result for a material undergoing pure secondary creep, and in particular linearly viscous flow, is presented here.

A constitutive law for creep may be of the form:

$$(\dot{\epsilon}_e/\dot{\epsilon}_0) = (\sigma_e/\sigma_0)^n \quad (A1.1)$$

where  $\dot{\epsilon}_e$  is the equivalent strain rate,  $\sigma_e$  is the equivalent stress,  $\dot{\epsilon}_0$  and  $\sigma_0$  are normalizing parameters, and  $n$  is the power law creep exponent. If equation (A1.1) is obeyed then, for plane stress, the stress at a position  $r$  and angle  $\theta$  ahead of a crack is given by; [3-5]

$$\sigma_e = (1/I_n)^{1/(n+1)} (\sigma_0^n/\dot{\epsilon}_0)^{1/(n+1)} C_s^* (1/(n+1)) r^{-1/(n+1)} \tilde{\sigma}_e(\theta) \quad (A1.2)$$

where  $I_n$  is a numerical coefficient given by, [5]  $C_s^*$  is the time dependent equivalent of the J integral (formally  $\dot{J} = dJ/dt$ ), and  $\tilde{\sigma}_e(\theta)$  is a function of  $\theta$ .

In the limit of  $n = 1$ ,  $I_n = 2\pi$ ,  $\dot{\epsilon}_e/\sigma_0^n$  is equal to the viscosity  $\eta$ , and  $K_I^2 = \eta C_s^*$ , so that when  $\theta = 0$ ,

$$\sigma = \frac{1}{\sqrt{2\pi r}} (\eta C_s^*)^{1/2} = \frac{K_I}{\sqrt{2\pi r}} \quad (A1.3)$$

where  $K_I$  is the mode I stress intensity factor. Since it is well known that in plane stress,  $EG = K_I^2$  (where  $G$  is the strain energy release rate, and  $E$  is Young's modulus), it follows that a sufficient proof that  $\eta C_s^* = K_I^2$  if  $n = 1$  is to show that  $EG = \eta C_s^*$ .

If  $n = 1$ , then  $J$  is synonymous with the strain energy release rate  $G$ , and  $C_s^*$  becomes equal to the time dependent equivalent of  $G = (\dot{G})$ . For a crack of length  $2a$  in an infinite elastic plate of thickness  $b$ , the well known Griffith analysis<sup>[6]</sup> shows that the strain energy ( $U$ ) stored in the region of the crack is given by:

$$U = \pi a^2 b \sigma^2 / E. \quad (A1.4)$$

So that the strain energy release rate (equals the rate of change of strain energy with respect to the crack area) is given by;

$$G = \sigma^2 \pi a / E. \quad (A1.5)$$

A similar approach shows that in a linearly viscous plate, the rate of change of strain energy with respect to time is given by;

$$\dot{U} = \pi a^2 b \dot{\sigma}^2 / \eta. \quad (A1.6)$$

So that the time dependent equivalent of  $G$  is,

$$\dot{G} = \sigma^2 \pi a / \eta. \quad (A1.7)$$

Hence  $EG = \eta G$ , and  $\eta C_s^* = K_I^2$  as required.

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## APPENDIX II

## THE CONSTRAINT IN A VISCO-ELASTIC MATERIAL

The constraint for a visco-elastic body can be calculated by the method described in Ref. 1. The Fourier transform of the misfit stress ( $\bar{\sigma}_c$ ) at an arbitrary distance  $x$  from a crack in a Maxwell solid, introduced by an edge dislocation of Burgers' vector,  $b$ , located at  $x = x'$ , is given by;

$$\bar{\sigma}_c = \frac{\bar{Q}(3K\bar{P} + \bar{Q})b}{\pi\bar{P}(3K\bar{P} + 4\bar{Q})(x-x')} \sqrt{\frac{x'}{x}} \quad (A2.1)$$

where a bar signifies the Fourier transform of a variable,  $K$  is the bulk modulus,  $P = (1/\tau_R + d/dt)$ ,  $Q = G d/dt$ ,  $G$  is the shear modulus,  $\eta_s$  is the shear viscosity, and  $\tau_R = G/\eta_s$ . Since  $G/K = 3(1-2\nu)/2(1+\nu)$ , the inverse Fourier Transform leads to the constraint for a Maxwell type material,

$$\sigma_c(x) = \frac{Eb}{8\pi(1+\nu)(x-x')} \sqrt{\frac{x'}{x}} \left\{ e^{-t/\tau_R} + \frac{(1+\nu)}{(1-\nu)} e^{-(1+\nu)t/(3\tau_R(1-\nu))} \right\} \quad (A2.2)$$

Therefore if  $x' = \ell$ ,  $x = \ell/2$  and  $u = \lambda F_v A^{3/2} / \pi F_b^{3/2}$  as in the text, then

$$\sigma_c = \frac{EF_v}{2\sqrt{2}\pi F_b^{3/2}(1+\nu)} \left(\frac{\lambda}{\ell}\right) A^{3/2} \left\{ e^{-t/\tau_R} + \frac{(1+\nu)}{(1-\nu)} e^{-(1+\nu)t/(3\tau_R(1-\nu))} \right\} \quad (A2.3)$$

Note that if  $t = 0$ , the elastic solution (equation 4.2) is generated.

The solution of the crack growth problem with this form of constraint is complicated. The description of the approximate behavior given in the text is sufficient to illustrate the important trends.

## APPENDIX II REFERENCE

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### APPENDIX III CONSIDERATION OF MENISCUS INSTABILITIES

The only ill-defined parameter in the crack growth relations derived in Chapter V, is the critical thickness of the intergranular film at which the facet fails ( $\delta_c$ ). One possible criterion pertinent to determining  $\delta_c$  is the meniscus instability. This is analogous to the idea developed in Chapter III, in which a transition of cavity morphology was suggested. This approach is outlined below, but unfortunately is not viable for reasons that became apparent during the analysis.

The conditions under which the meniscus of a film of liquid between two parallel plates becomes unstable and develops "fingers" is given by Fields and Ashby,<sup>[1]</sup>

$$\lambda_{crit} = \pi \sqrt{\frac{\gamma \sigma^2}{\eta_l V_R}} \quad (A3.1)$$

where  $\lambda_{crit}$  is the critical wave-length of the disturbance that grows most rapidly, and  $V_R$  is the velocity of the interface at  $x = R$ , given by<sup>[1]</sup>

$$V_R = - \frac{\sigma^2}{12\eta_l} \left( \frac{d\sigma}{dx} \right)_R \quad (A3.2)$$

This can be readily demonstrated to reduce to

$$V_R = \dot{\delta}(R/\delta) \quad (A3.3)$$

Combining equations (A3.3), (5.6) and (5.3) yields the condition;

$$(\lambda_{crit}/\ell)^2 = \gamma \pi^2 (\delta_0/\ell)^2 (\delta_c/\delta_0) \left[ \frac{1}{2} (\ell/\delta_0 - \pi/4) (\delta_0/\delta_c)^2 + \left( \frac{\pi-4}{8} \right) \right]^{-1} \frac{\eta_b}{\eta_l C_5} \quad (A3.4)$$

$$\left[ C_1 (\delta_0/\delta_c)^6 + C_2 (\delta_0/\delta_c)^4 + C_3 (\delta_0/\delta_c)^2 + C_4 \right] \left[ 1 - C_6 (\delta_0/\delta_c)^2 \right]^{-1}.$$

Hence,  $\delta_c$  can be obtained once  $(\lambda_{crit}/\ell)$  for meniscus instability has been prescribed. For unconstrained growth, the critical value of the wave-length for finger-like growth decreases as the film thickness increases. This leads to the expected result that, after a period of stable meniscus propagation, the critical wave-length becomes sufficiently small (e.g. less than the grain size) that instability necessarily occurs. However, constraint appears to reverse the trend, and the critical wave-length increases as growth proceeds, because of the depression of  $\delta_c$  induced by the constraint. Consequently, the condition of instability can only be satisfied at the beginning of growth. Such behavior may occur and lead to finger-like cavities in some instances. However, a more generalized criterion for  $\delta_c$  is evidently needed to provide a complete description of crack growth rates.

#### APPENDIX III REFERENCE

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## APPENDIX IV

APPROXIMATE RELATIONSHIPS FOR THE CRACK VELOCITY IN A  
LIQUID PHASE SINTERED MATERIAL

The unconstrained crack velocity may be derived by ignoring the constraint term in equation (5.5) and assuming that if  $K \gg K_{th}$ , the surface tension term may also be ignored so that,

$$8\sqrt{\pi} \eta_l (R/\delta)^3 \dot{\delta} = K\sqrt{\ell} \quad (A4.1)$$

but from equation (5.3),

$$(R/\delta) \approx (1/2) (\ell/\delta_0) (\delta_0/\delta)^2$$

so that,

$$\sqrt{\pi} \eta_l (\ell/\delta_0)^3 (\delta_0/\delta)^6 \dot{\delta} = K\sqrt{\ell} \quad (A4.2)$$

which upon integration between the standard limits as in equation (5.7) yields

$$\dot{a} = [5K\sqrt{\ell}/\sqrt{\pi} (\ell/\delta_0)^2] [1 - (\delta_0/\delta_c)^5]^{-1} (1/\eta_l) \quad (A4.3)$$

which compares with equation (5.9a).

The opposite limit obtains when the constraint is so large that it equals the applied stress, and the cavity grows under a zero net stress (which can only occur, of course, if the surface tension term is ignored, as is valid when  $K \gg K_{th}$ ). In this case the governing equation reduces to

$$2\sqrt{2} \eta_b \dot{\delta}/3\sqrt{\pi} = K\sqrt{\ell} \quad (A4.4)$$

which upon integration yields equation (5.9b)

$$\dot{a} = [3\sqrt{\pi} K\sqrt{\ell} (\ell/\delta_0)/2\sqrt{2} (\delta_c/\delta_0 - 1)] (1/\eta_b).$$

The existence of these two limits has an interesting parallel with the model developed in Chapter II for crack growth in a single phase material. For a damage zone of one grain, the crack velocity is given by equation (2.28)

$$\dot{a} = BK\sqrt{\ell}/\{\lambda^3[g(A_f) + 8\sqrt{2}(\ell/\lambda)^2 A_f/27\pi^2]\}$$

The unconstrained solution is obtained when the first term in the denominator dominates, and the maximum constraint solution pertains when the second term dominates.<sup>[41]</sup> It was shown that this latter case is applicable when the damage is strain controlled.<sup>[19]</sup>

FIGURE CAPTIONS

- Fig. 1 Scanning electron micrographs of a crack tip in a single phase alumina after loading at 1300°C. Arrows denote successive crack tip positions. (Courtesy W. R. Blumenthal)
- Fig. 2 S.E.M. micrographs of a crack in alumina near the crack-growth threshold. Intense damage is located in shear bands at the crack tip. (Courtesy W. R. Blumenthal)
- Fig. 3 Crack nucleation in large grained regions and in an area contaminated by glass. (Courtesy W. R. Blumenthal)
- Fig. 4 Four stages in the failure of alumina, (a) crack nucleation, (b) crack propagation, (c) crack blunting, (d) crack coalescence of shear bands. (Courtesy W. R. Blumenthal)
- Fig. 5 An alumina bar after failure by creep rupture. The interaction of shear bands results in a serrated fracture surface. (Courtesy B. J. Dalgleish)
- Fig. 6 An isolated crack growing along the grain boundary of an elastic bi-crystal. A flux of atoms flows from the surface of the crack ( $j_s$ ) into the grain boundary ( $j_b$ ). If the diffusion along the boundary is slow compared to surface diffusion, then the atoms are deposited non-uniformly along the boundary and the stress field is modified.
- Fig. 7 S.E.M. micrograph of a creep fracture surface. The crack propagated downwards. (Courtesy W. R. Blumenthal)

- Fig. 8 S.E.M. micrographs of the cavitated region on a creep fracture surface, (a) near the original crack front, (b) in the central region, (c) at the end of the damage zone. (Courtesy W. R. Blumenthal)
- Fig. 9 A damage zone model of crack growth, the presence of cavities modifies the stress field ahead of the crack.
- Fig. 10 Equilibrium and crack-like cavities with dihedral angles  $\psi$ .
- Fig. 11 Cavitation occurs on the boundaries ahead of the crack tip which are subject to both shear and normal stresses.
- Fig. 12 The theoretical situation considered in this thesis. The damage zone consists of an array of grains co-planar with the crack and embedded in a linearly viscous matrix.
- Fig. 13 The arrangement of equilibrium cavities on a grain boundary in the Raj and Ashby model for intergranular failure at elevated temperatures.
- Fig. 14 An array of dislocations ahead of a crack, used to model the constraint.
- Fig. 15 Plots of normalized crack velocity against the number of grains in the damage zone, for  $\psi = 75^\circ$  and  $\zeta/\xi = 1$ .
- Fig. 16 Predictions of constrained crack growth (for a damage zone of a single grain) as a function of the stress intensity factor compared to experimental values from Ref. 1. The spread in the theoretical lines represents the uncertainty in the values of viscosity.

- Fig. 17 Plots of normalized stress against the ratio of the distance ahead of the crack to the grain size ( $n$ ), immediately after an increment of crack advance.  $\zeta/\xi = 1$ . (The stress has been drawn as a continuous curve for clarity, although to be strictly in accordance with the model it should have been plotted as discrete values for each grain.)
- Fig. 18 A plot of the normalized stress against the ratio of the distance ahead of the crack to the grain size, at various times between increments of crack advance ( $\delta t/\Delta t$ ).  $\zeta/\xi = 1$ . (See also the note on the caption of Fig. 17).
- Fig. 19 A plot of the relative stress on the damage zone (assumed to consist of a single grain) as a function of the cavity size,  $\gamma = 1 \text{ Jm}^{-1}$ ,  $K = 3 \text{ MPa}\sqrt{\text{m}}$ ,  $\ell = 2 \times 10^{-6} \text{ m}$ ,  $\psi = 75^\circ$ . The effect of the sintering stress has been ignored.
- Fig. 20 A plot of the product of the crack velocity (constrained) and viscosity against  $K\sqrt{\ell}$  illustrating the effect of the inclusion of a sintering stress in the model.  $K_{th}\sqrt{\ell} = 2 \times 10^3 \text{ Nm}^{-1}$ .
- Fig. 21 A plot of the relative stress on a damage zone of a single grain as a function of the cavity size,  $\gamma = 1 \text{ Jm}^{-1}$ ,  $K\sqrt{\ell} = 4 \times 10^3 \text{ Pa m}$ ,  $K_{th}\sqrt{\ell} = 2 \times 10^3 \text{ Pa m}$ .
- Fig. 22 A plot of  $\dot{n}a$  against  $K\sqrt{\ell}$ , illustrating how a threshold may arise if crack propagation is limited by cavity nucleation.
- Fig. 23 The separation of ligaments by a transition to crack-like cavity growth.

Fig. 24 The transition curves for equilibrium to crack-like growth as a function of the ratio of the applied stress to sintering stress ( $s$ ) and the cavity size, for various values of the boundary to surface diffusivity ratios ( $\Delta$ ).

Fig. 25 The variation in the resultant stress on the damage zone changes the cavity growth from an equilibrium to a crack-like mechanism.

Fig. 26 A plot of the resultant crack velocity against applied stress intensity factor, assuming that the critical cavity size for coalescence is given by the size at transition.  $\ell/\lambda = 5$ ,  $K\sqrt{\ell} = 4 \times 10^3 \text{ Nm}^{-1}$ ,  $K_{th}\sqrt{\ell} = 2 \times 10^3 \text{ Nm}^{-1}$ .

Fig. 27 A plot of the crack velocity against applied stress intensity factor, showing the relationship between the unconstrained, viscously constrained and elastically constrained solutions. The values of the parameters used are  $A_f = 0.2$ ,  $\ell/\lambda = 5$ ,  $E = 300 \text{ GPa}$ , and  $\ell = 10^{-6} \text{ m}$ .

Fig. 28 A plot of the crack velocity against applied stress intensity factor showing the relationship between the unconstrained, viscously constrained and elastically constrained solutions. The values of the parameters used are  $A_f = 0.2$ ,  $\ell/\lambda = 10$ ,  $E = 300 \text{ GPa}$  and  $\ell = 10^{-6} \text{ m}$ .

Fig. 29 A schematic illustrating the configuration considered in Section 4.2. A grain boundary extends at an angle  $\theta$  to the axis of the crack, the magnitude of  $\theta$  can take any value between 0 and  $\pi/3$ .

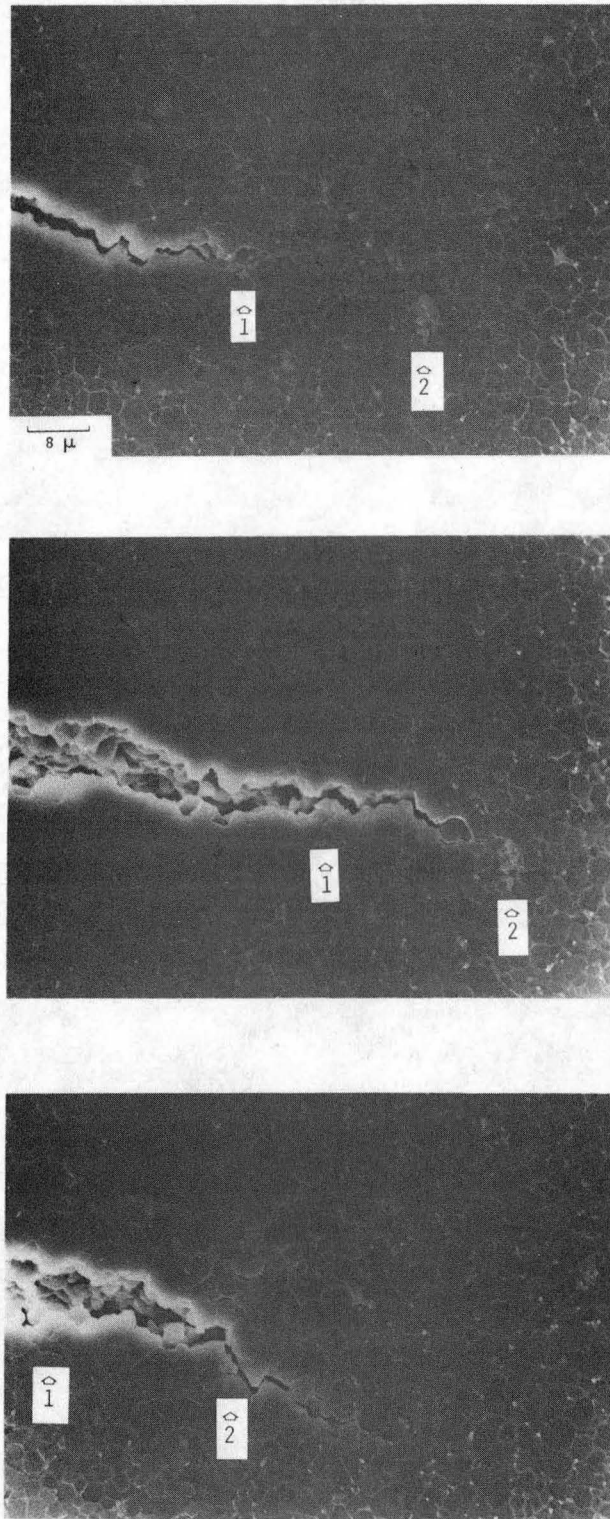
- Fig. 30 A plot of normalized crack velocity against  $K/K_c$ , showing the influence of the critical stress intensity factor.  $A_f = 0.2$  and  $\ell/\lambda = 5$ .
- Fig. 31 A schematic illustrating a typical load/displacement plot obtained for liquid phase sintered alumina in four point bending tests.
- Fig. 32 An Arrhenius plot for the viscosity of the liquid phase sintered alumina.
- Fig. 33 S.E.M. micrograph showing the association of some micro-cracks with porous regions. The white spheres have been identified as gold from the coating process.
- Fig. 34 S.E.M. micrograph showing the nucleation of a crack in an apparently homogeneous region.
- Fig. 35 S.E.M. micrograph of crack tips, illustrating the zone presumed to contain amorphous material.
- Fig. 36 S.E.M. micrograph of the fracture surface of a liquid phase sintered alumina. The specimen was held at load while being cooled to room temperature, it was then fractured. The picture shows a region immediately behind the crack front, illustrating the ligaments and islands of amorphous material.
- Fig. 37 S.E.M. micrograph of the fracture surface of a liquid phase sintered alumina after an interrupted test. The crack front can be identified by the transition in the appearance of the surface. The creep damaged region is intergranular, whereas the region fractured at room temperature is transgranular.

- Fig. 38 S.E.M. micrograph of a room temperature fracture surface of a liquid phase sintered alumina.
- Fig. 39 S.E.M. micrograph of a room temperature fracture surface of a liquid phase sintered alumina after an etch in 5% HF acid for 20 minutes. The glassy phase has been removed to clearly reveal the transgranular nature of the fracture surface.
- Fig. 40 S.E.M. micrograph of an inter-agglomerate failure surface obtained at high strain rates.
- Fig. 41 The theoretical configuration considered in Section 5.1. Ahead of the crack there exists a grain boundary of length  $\ell$ , filled with a liquid phase of thickness  $\delta$ . Cavities are initiated in the triple points at the end of the grain and grow towards the center.
- Fig. 42 A theoretical plot of  $\eta_b \dot{a}$  against  $K\sqrt{\ell}$  for a liquid phase sintered material. The ratio of the critical to initial film thickness has been taken to be 1.5. (---) represents the plot for  $\ell/\delta_0 = 100$ , and (—) represents the plot for  $\ell/\delta_0 = 1000$ .
- Fig. 43 A theoretical plot of  $\eta_b \dot{a}$  against the ratio of the bulk to liquid phase viscosities.  $K\sqrt{\ell} = 2 \times 10^3 \text{ Nm}^{-1}$ .
- Fig. 44 A theoretical plot of  $\eta_b \dot{a}$  against the ratio of the bulk to liquid phase viscosities.  $K\sqrt{\ell} = 10 \times 10^3 \text{ Nm}^{-1}$ .
- Fig. 45 Theoretical plots of the normalized velocity against the number of grains in the damage zone size ( $z$ ) for a liquid phase sintered material. The difference between the unconstrained and constrained solutions is illustrated.  $\ell/\delta_0 = 100$ .

Fig. 46 Theoretical plots of the normalized velocity against the number of grains in the damage zone ( $z$ ) for a liquid phase sintered material.  $\ell/\delta_0 = 1000$ .

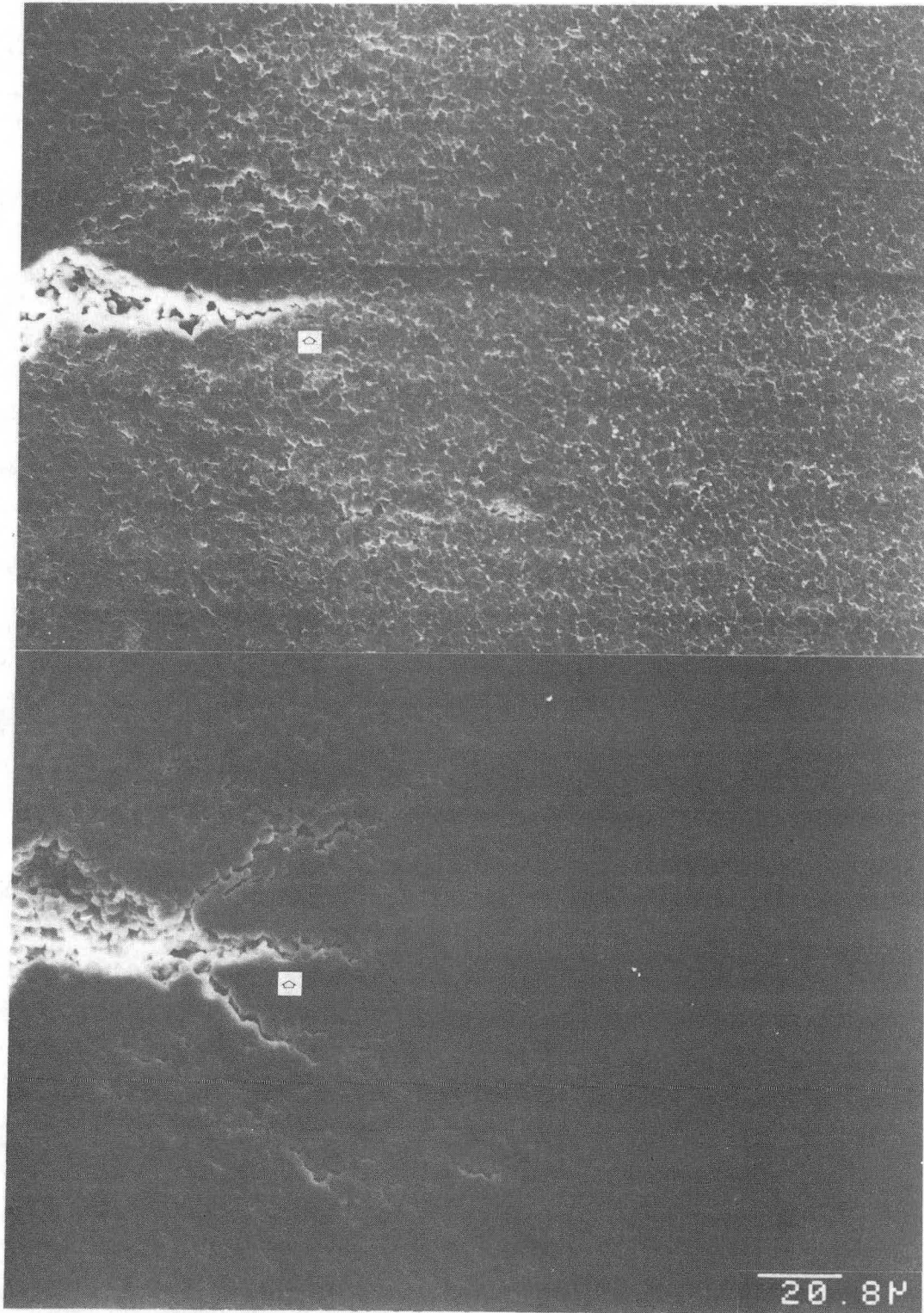
Fig. 47 A plot comparing the theory presented in this thesis with the experimental results of Blumenthal.<sup>[1]</sup> The theoretical curve includes the effect of a threshold and the critical stress intensity factors.

Fig. 48 A schematic showing the variation of strength with temperature and strain rate for liquid phase sintered ceramics.



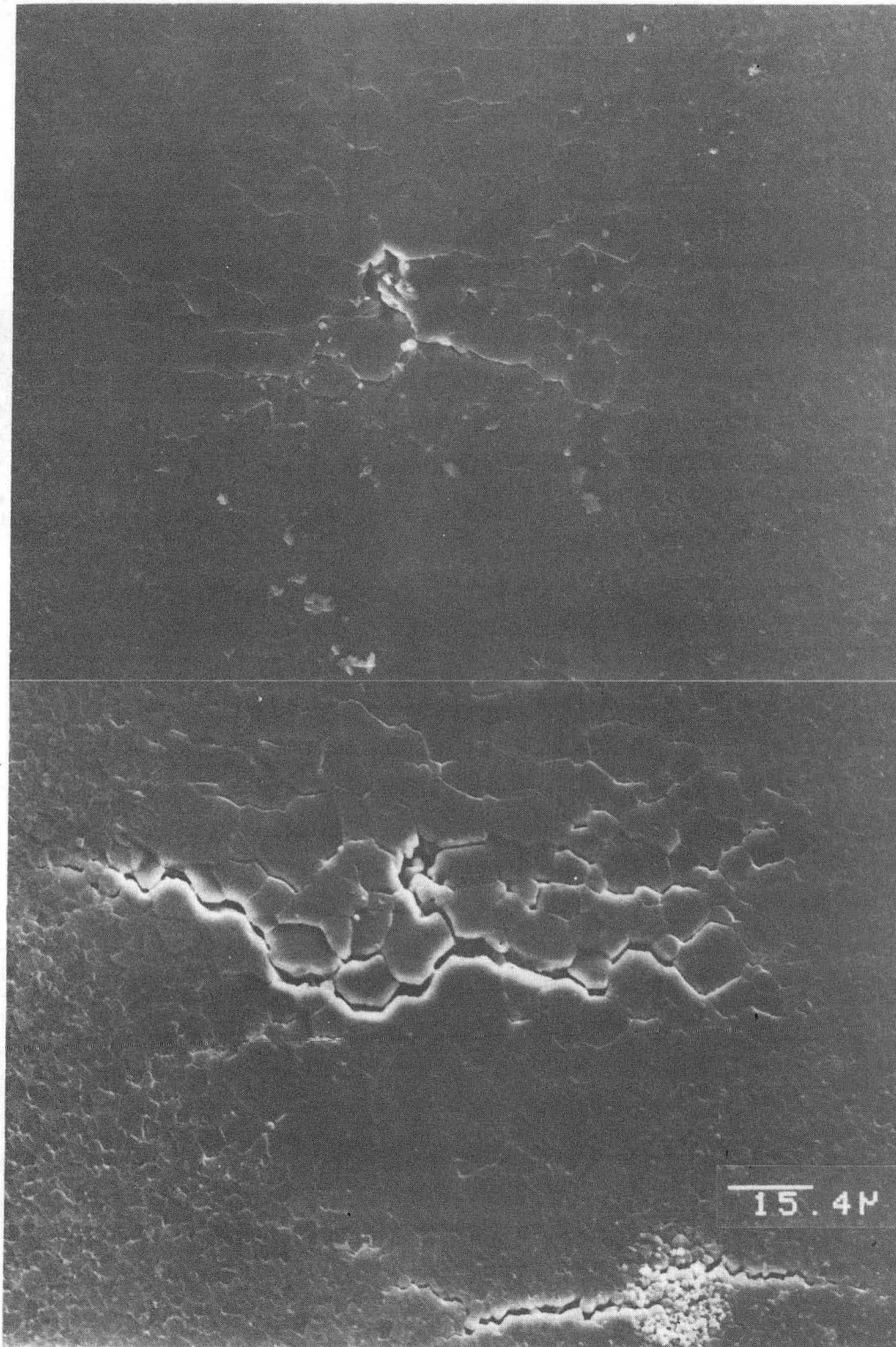
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Fig. 1. Scanning electron micrographs of a crack tip in a single phase alumina after loading at 1300°C. Arrows denote successive crack tip positions (Courtesy W. R. Blumenthal).



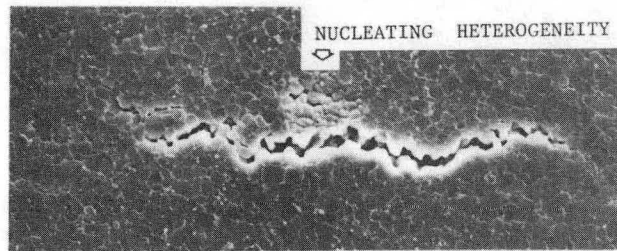
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Fig. 2. S.E.M. micrographs of a crack in alumina near the crack-growth threshold. Intense damage is located in shear bands at the crack tip (Courtesy of W.R. Blumenthal).

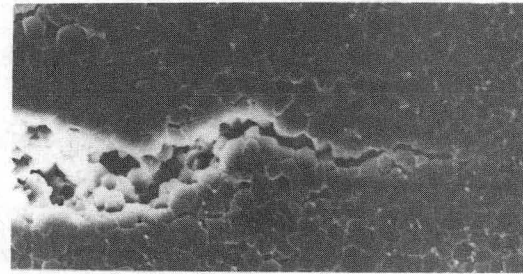


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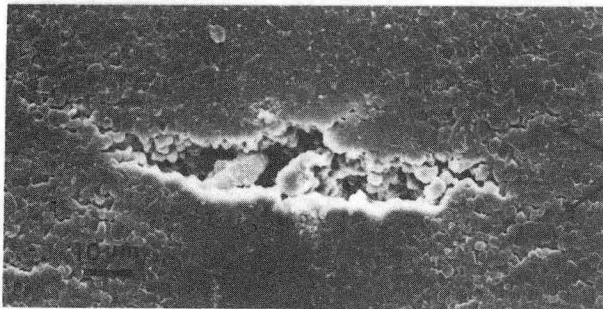
Fig. 3. Crack nucleation in large grained regions and in an area contaminated by glass (Courtesy of W.R. Blumenthal).



A) CRACK NUCLEATION

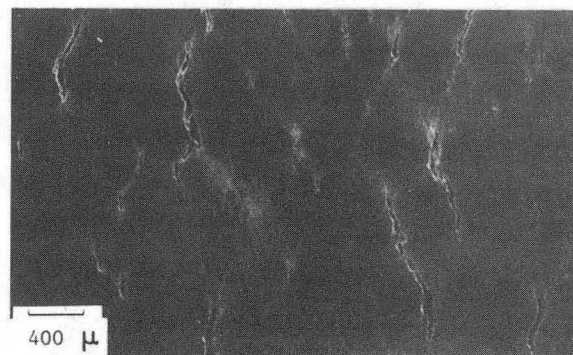


B) CRACK PROPAGATION



C) CRACK BLUNTING

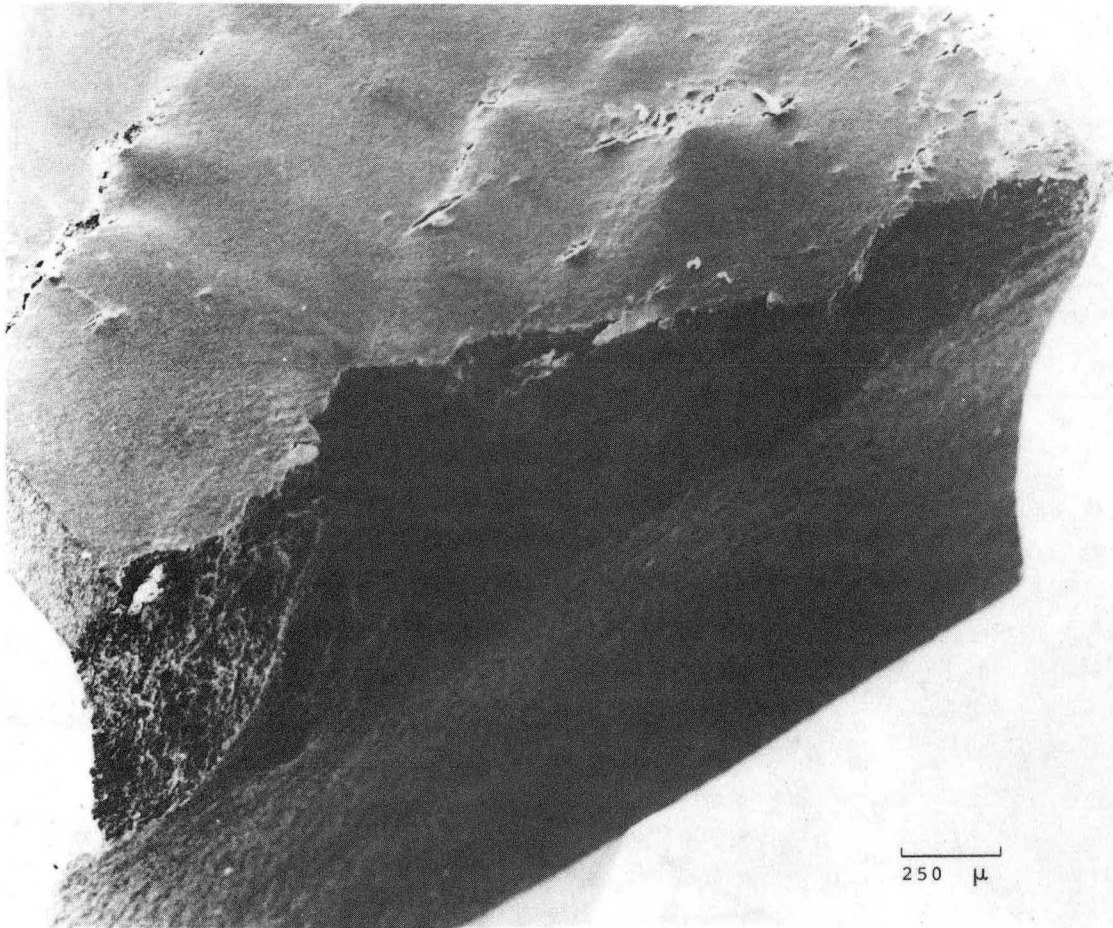
DAMAGE ZONES



D) CRACK COALESCENCE AT SHEAR BANDS

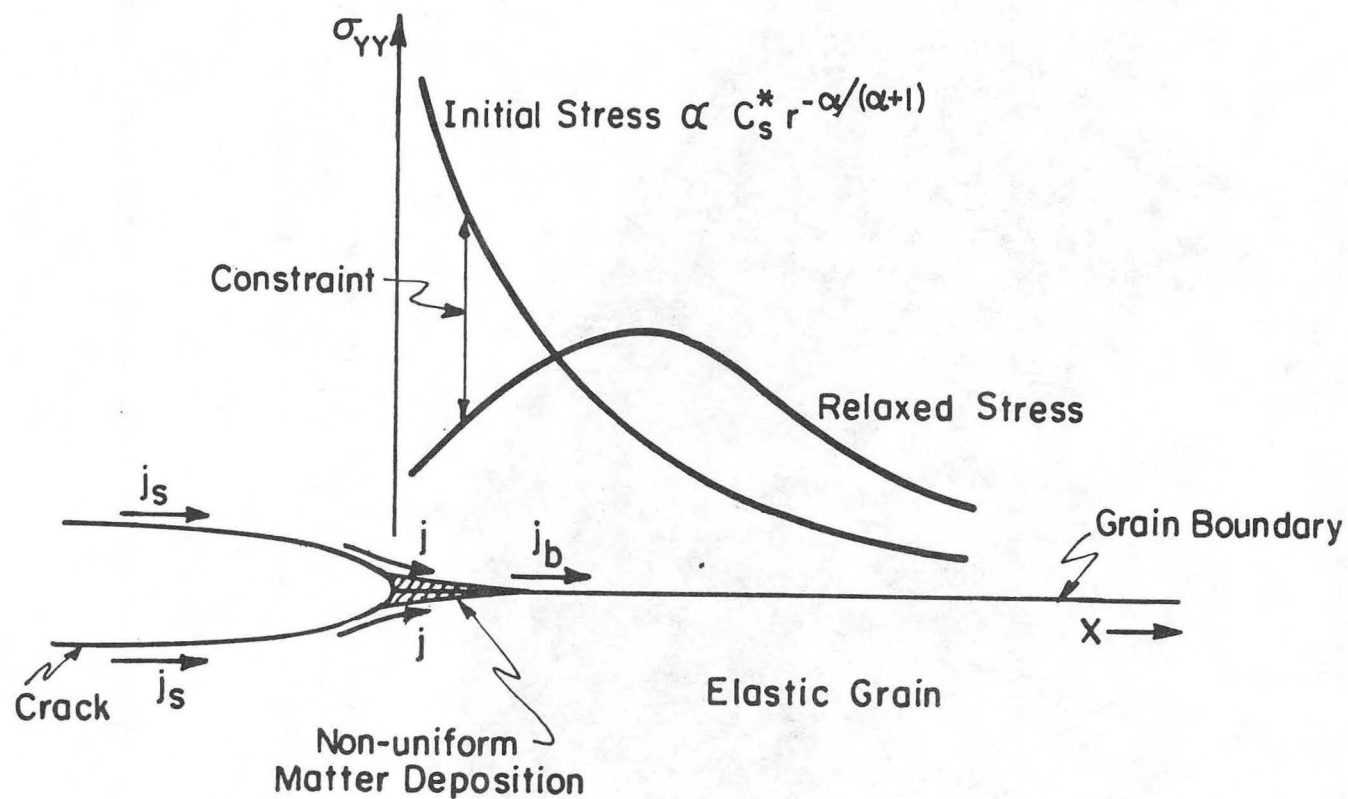
XBB 836-5001A

Fig. 4. Four stages in the failure of alumina. (a) crack nucleation, (b) crack propagation, (c) crack blunting, (d) crack coalescence of shear bands (Courtesy of W.R. Blumenthal).



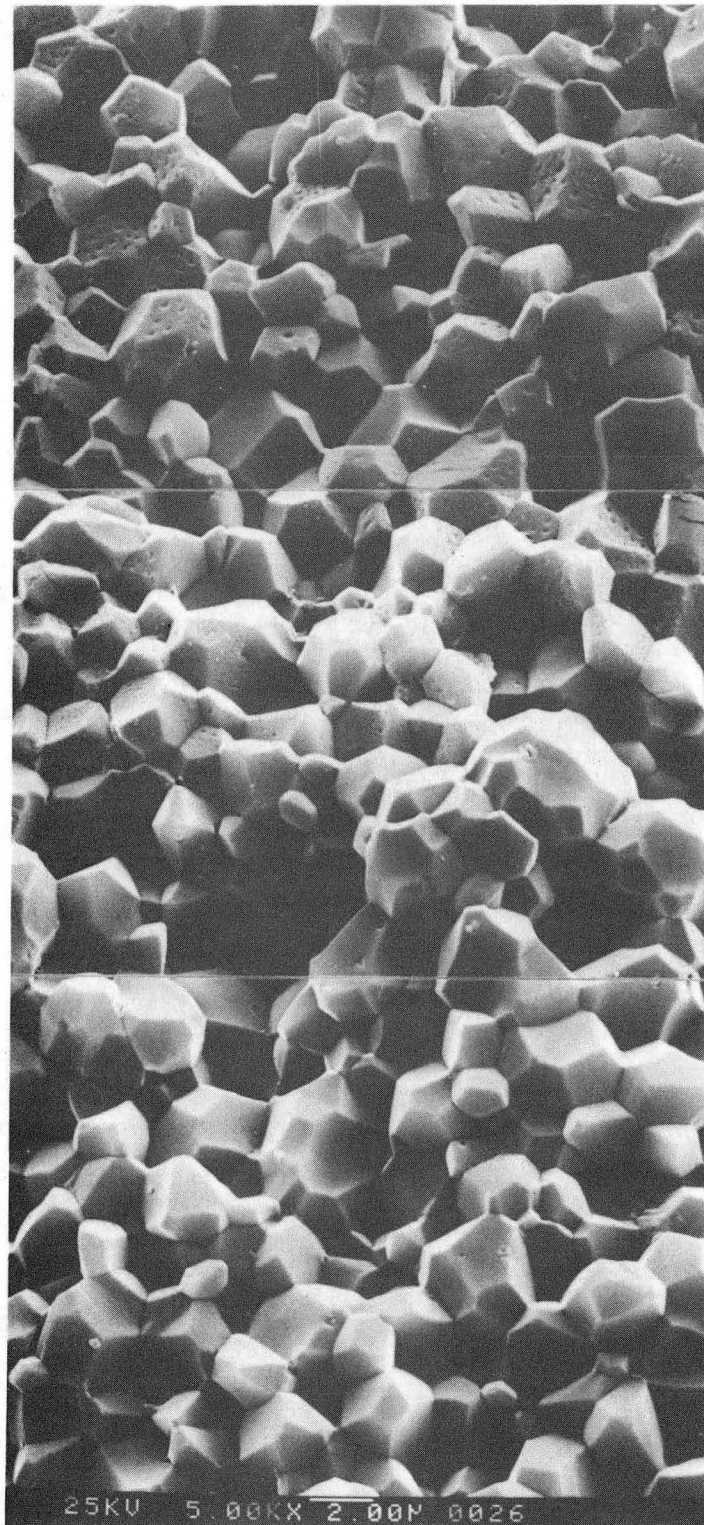
XBB 834-3482

Fig. 5. An alumina bar after failure by creep rupture. The interaction of shear bands results in a serrated fracture surface (Courtesy of B.J. Dalgleish).



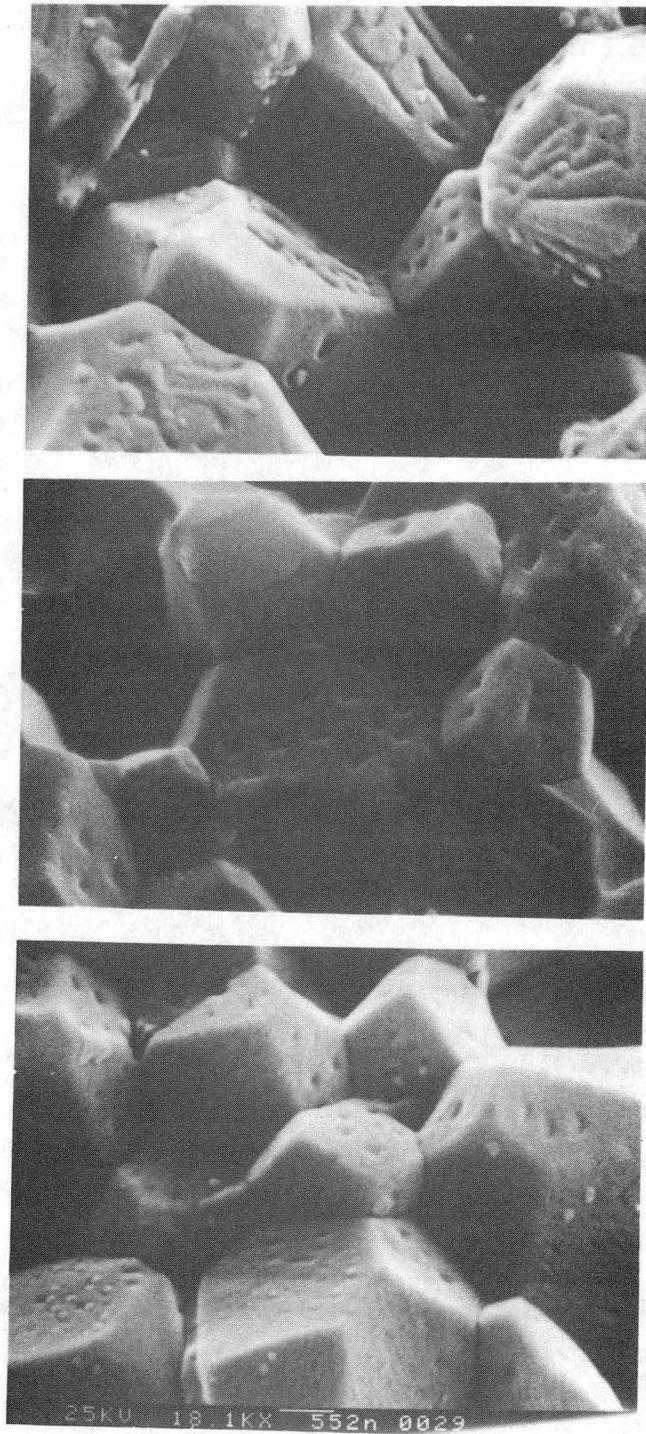
XBL 832-5231

Fig. 6. An isolated crack growing along the grain boundary of an elastic bicrystal. A flux of atoms flows from the surface of the crack ( $j_s$ ) into the grain boundary ( $j_b$ ). If the diffusion along the boundary is slow compared to surface diffusion, then the atoms are deposited non-uniformly along the boundary and the stress field is modified.



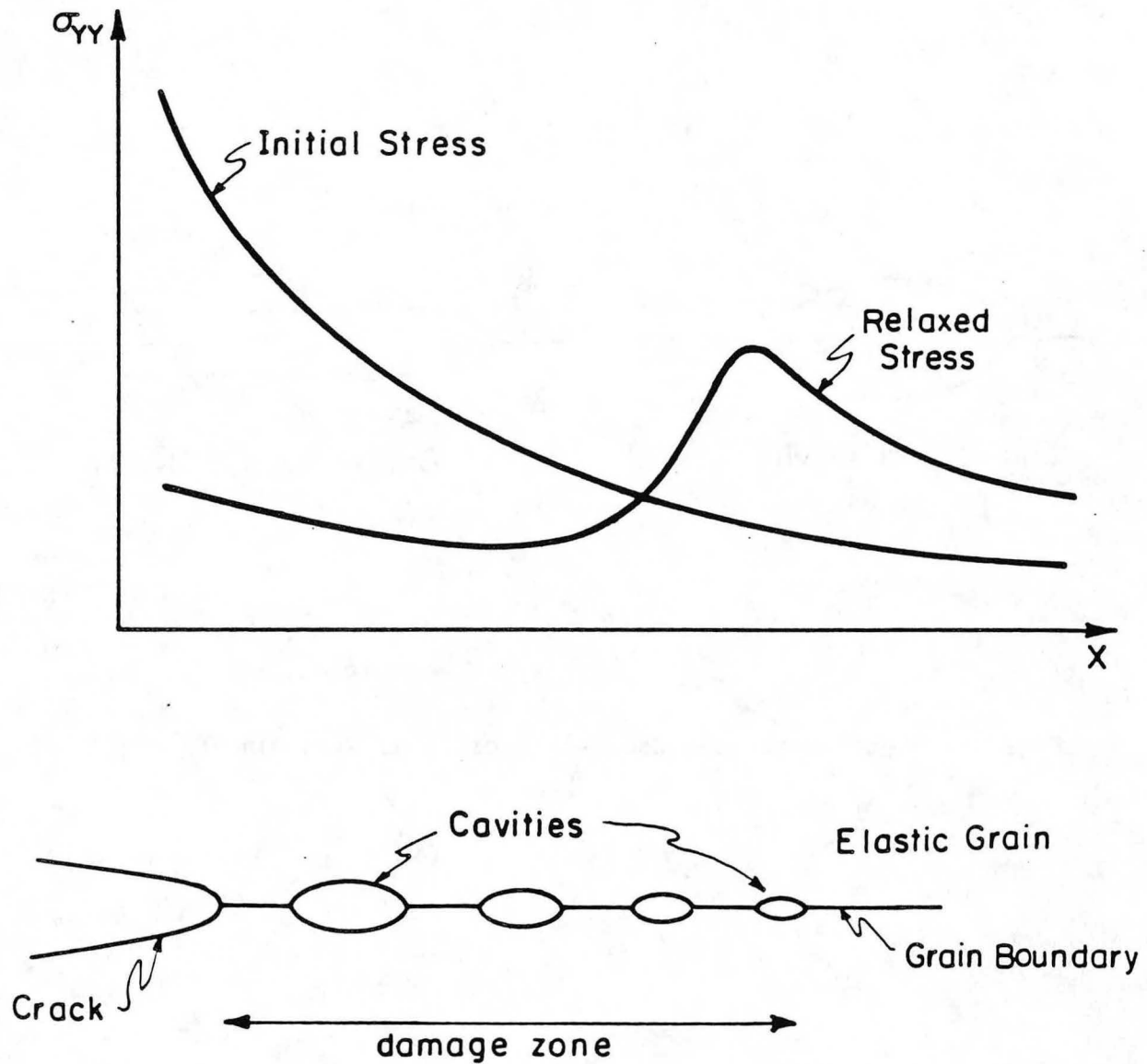
XBB 834-3479

Fig. 7. SEM micrograph of a creep fracture surface. The crack propagated downwards (Courtesy of W.R. Blumenthal).



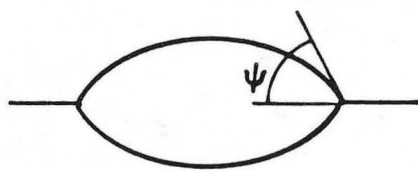
XBB 834-3477

Fig. 8. S.E.M. micrographs of the cavitated region on a creep fracture surface, (a) near the original crack front, (b) in the central region, (c) at the end of the damage zone (Courtesy of W.R. Blumenthal).

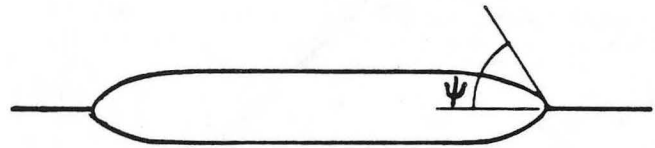


XBL 832-5230

Fig. 9. A damage zone model of crack growth, the presence of cavities modifies the stress field ahead of the crack.



Equilibrium Cavity



Crack-like Cavity

XBL 82 9-6513A

Fig. 10. Equilibrium and crack-like cavities with dihedral angles  $\psi$ .

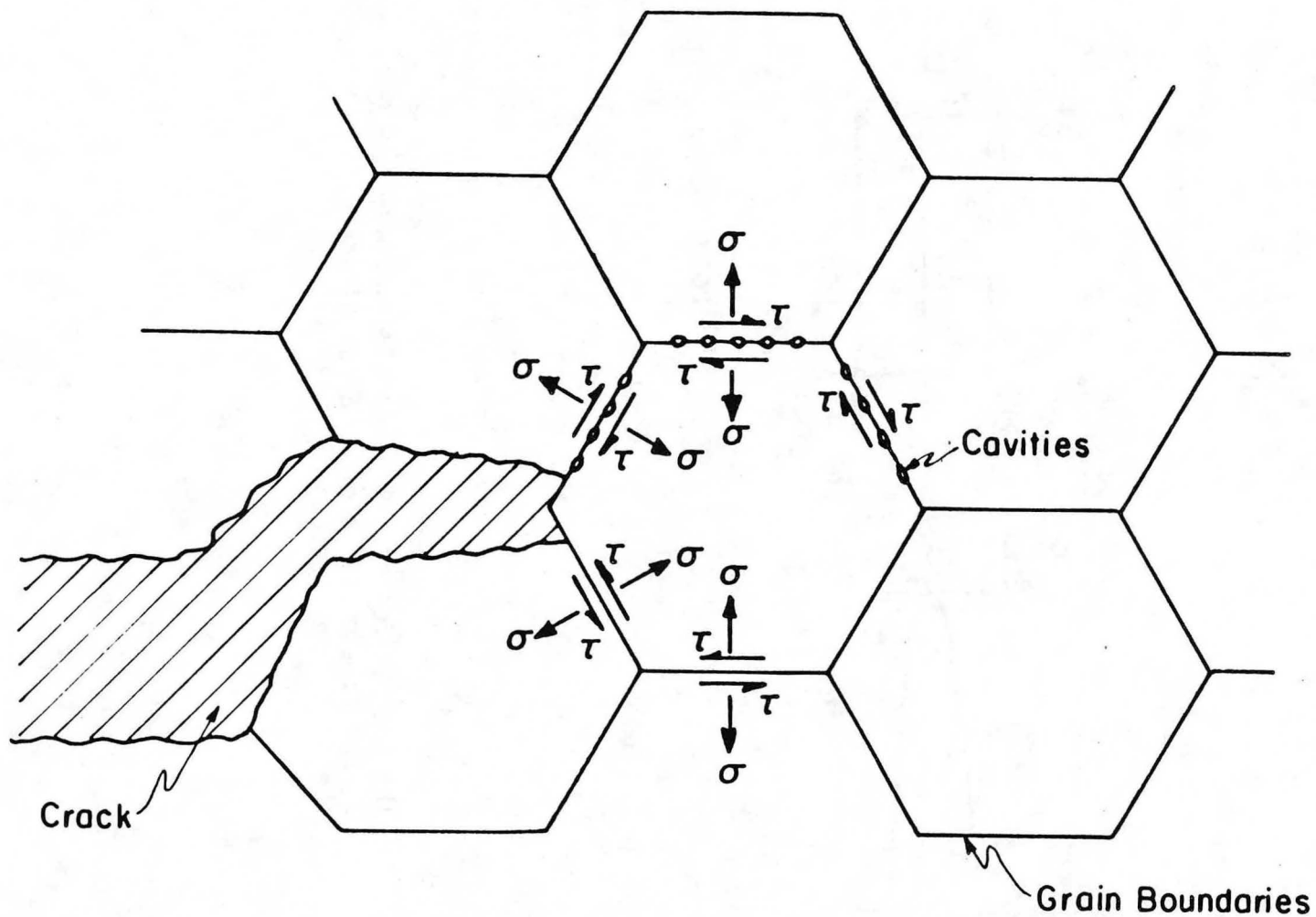
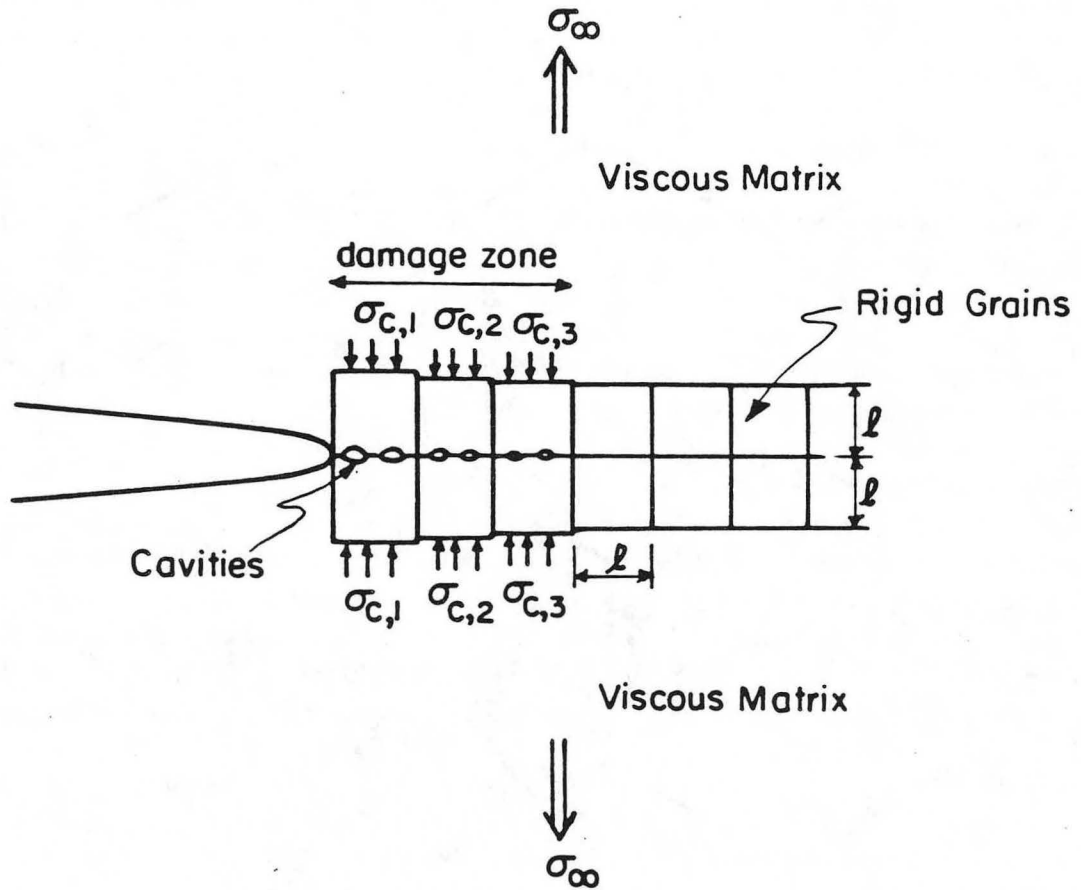


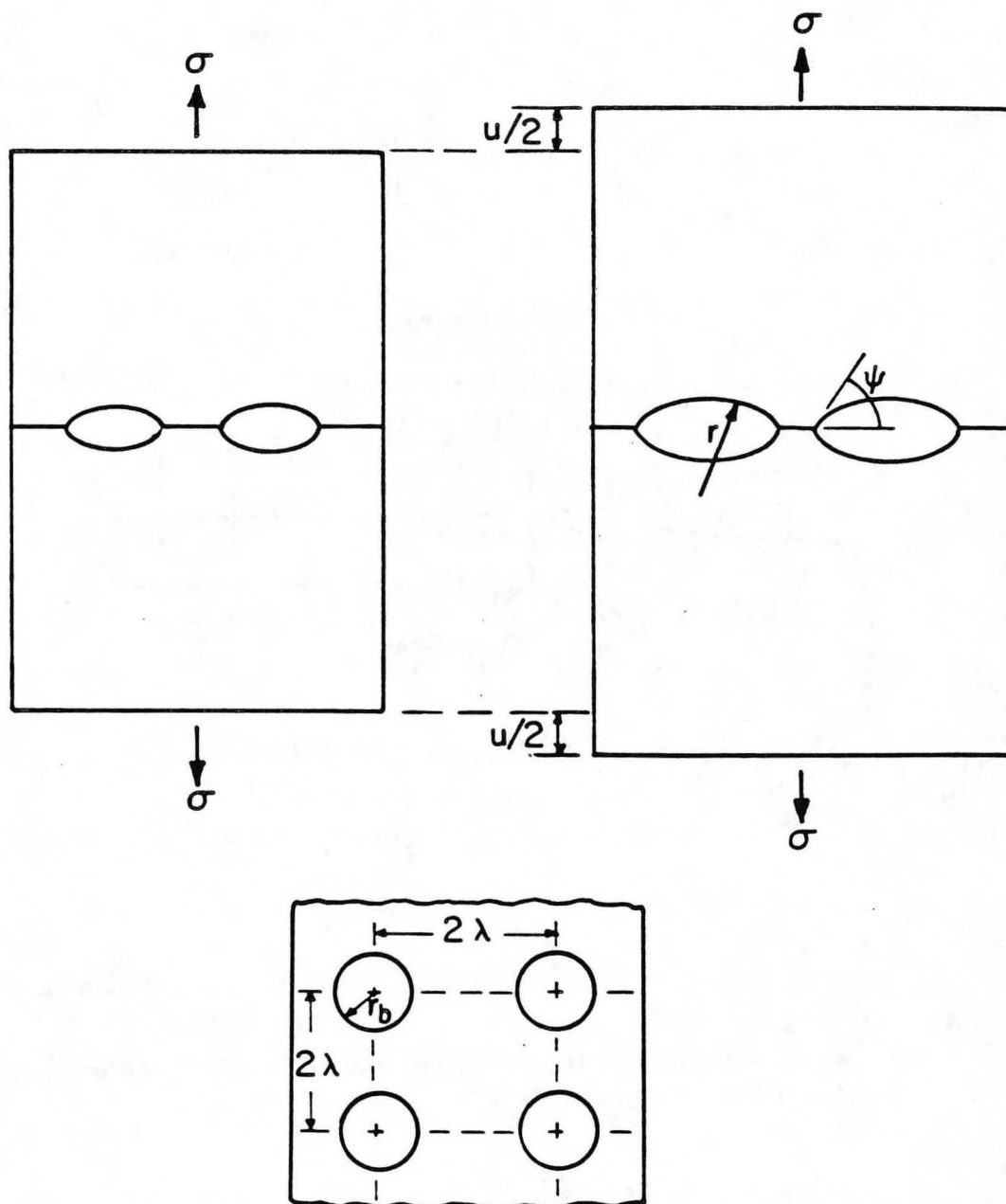
Fig. 11. Cavitation occurs on the boundaries ahead of the crack tip which are subject to both shear and normal stresses.

XBL 832-5229



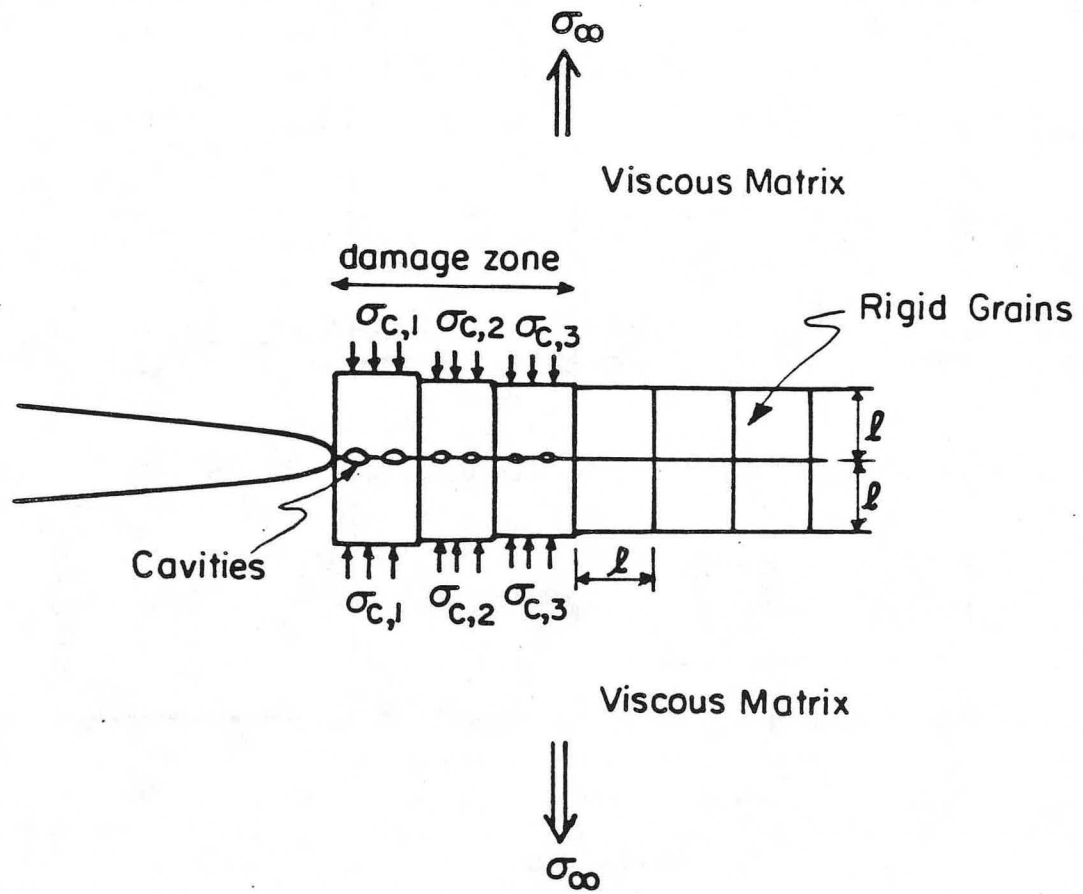
XBL 832-5228

Fig. 12. The theoretical situation considered in this thesis. The damage zone consists of an array of grains co-planar with the crack and embedded in a linearly viscous matrix.



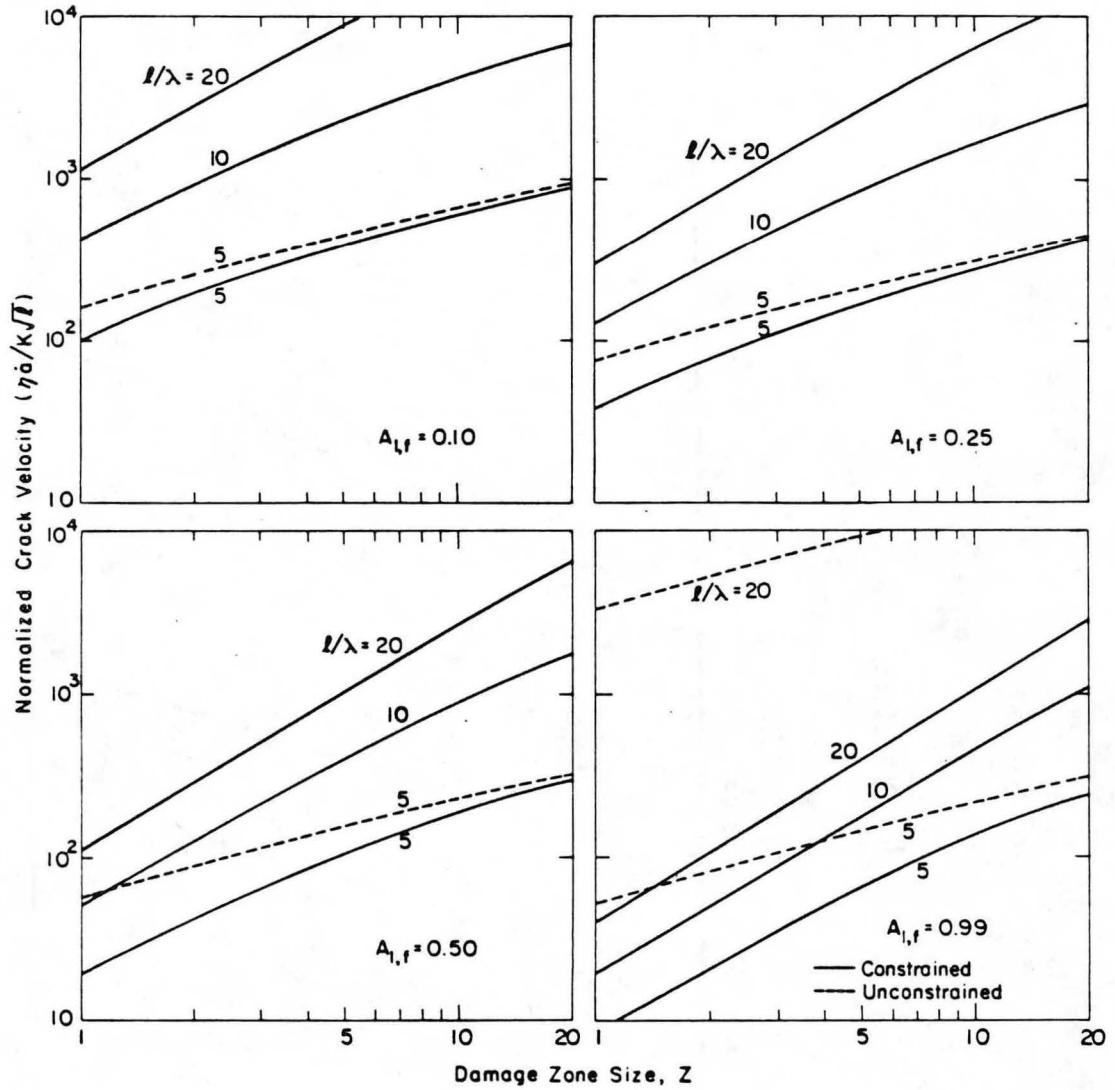
XBL829-6516A

Fig. 13. The arrangement of equilibrium-cavities on a grain boundary in the Raj and Ashby model for intergranular failure at elevated temperature.



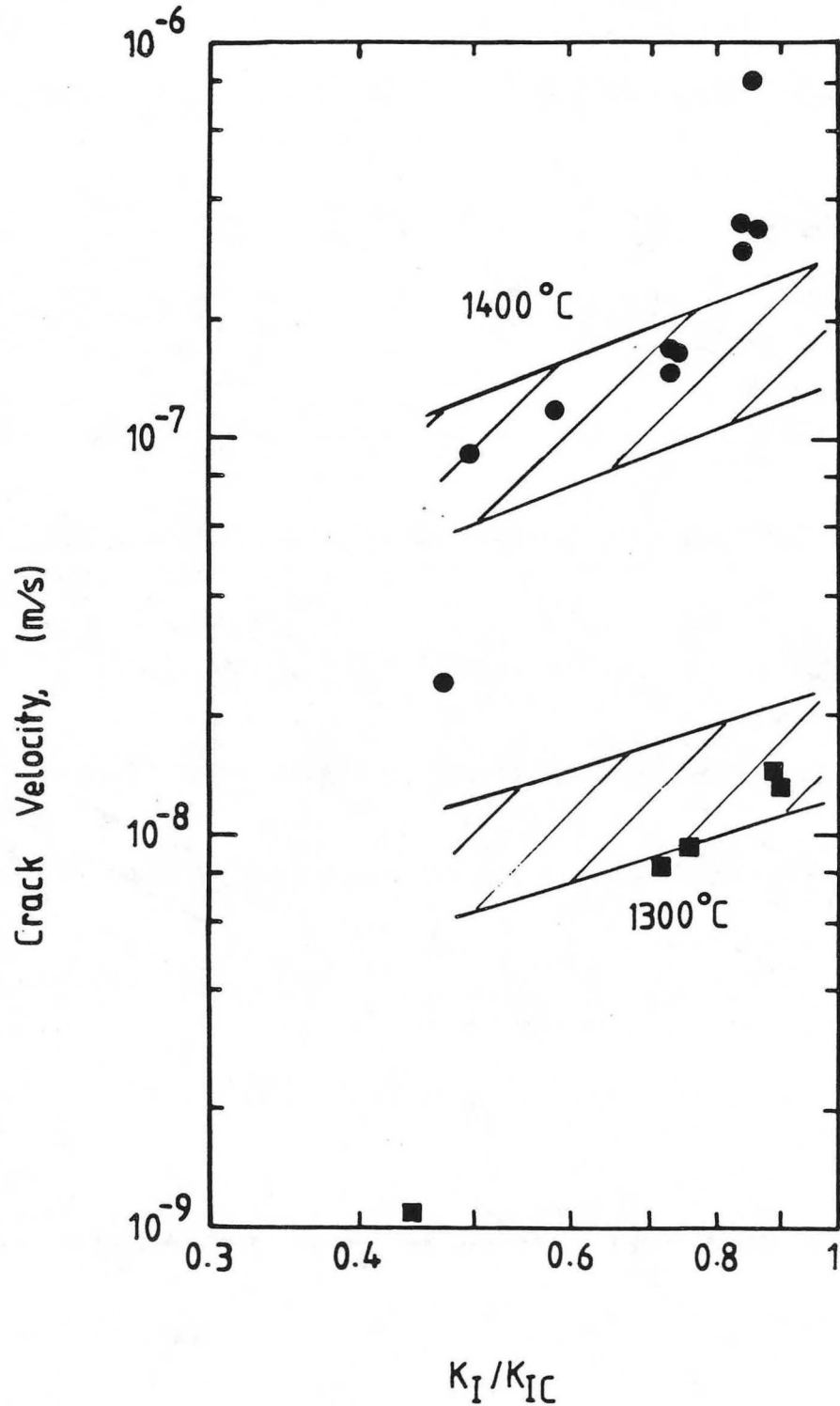
XBL 832-5228

Fig. 14. An array of dislocations ahead of a crack, used to model the constraint.



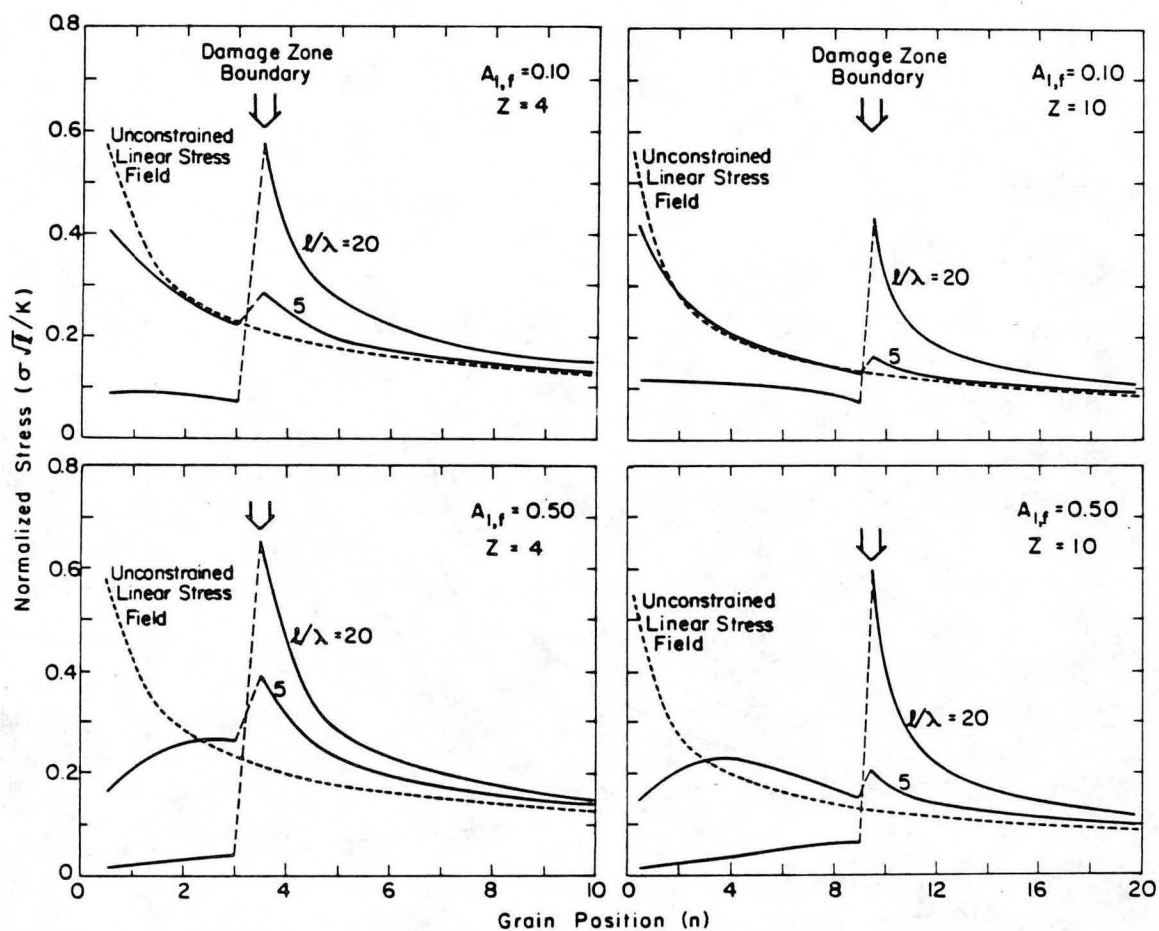
NL 832-5237

Fig. 15. Plots of normalized crack velocity against the number of grains in the damage zone, for  $\psi = 75^\circ$  and  $\zeta/\xi = 1$ .



XBL 8410-4150

Fig. 16. Predictions of constrained crack growth (for a damage zone of a single grain) as a function of the stress intensity factor compared to experimental values from Ref. 1. The spread in the theoretical lines represents the uncertainty in the values of viscosity.



JEL 32-5236

Fig. 17. Plots of normalized stress against the ratio of the distance ahead of the crack to the grain size  $(n)$ , immediately after an increment of crack advance  $\zeta/\xi = 1$ . (The stress has been drawn as a continuous curve for clarity, although to be strictly in accordance with the model it should have been plotted as discrete values for each grain.)

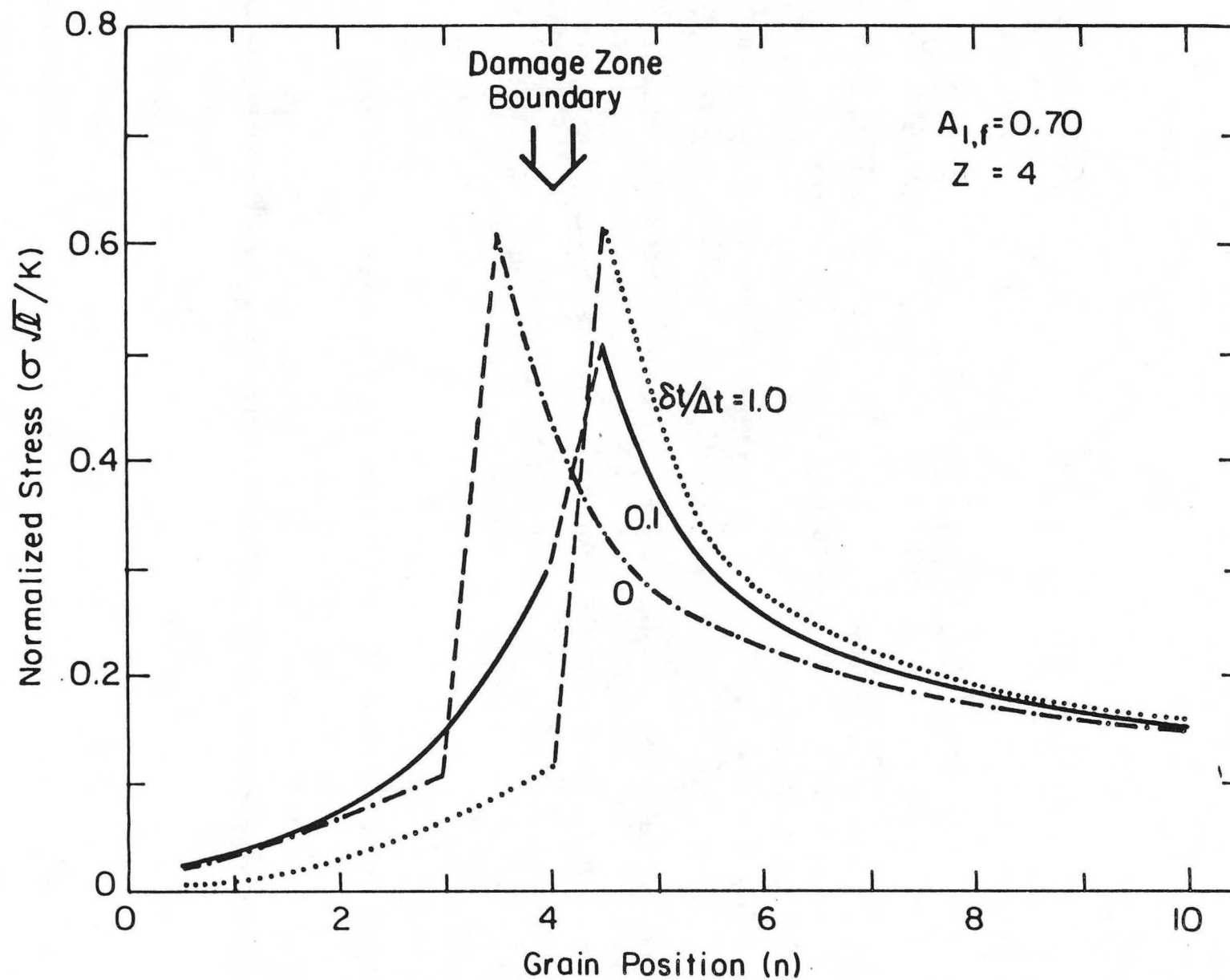
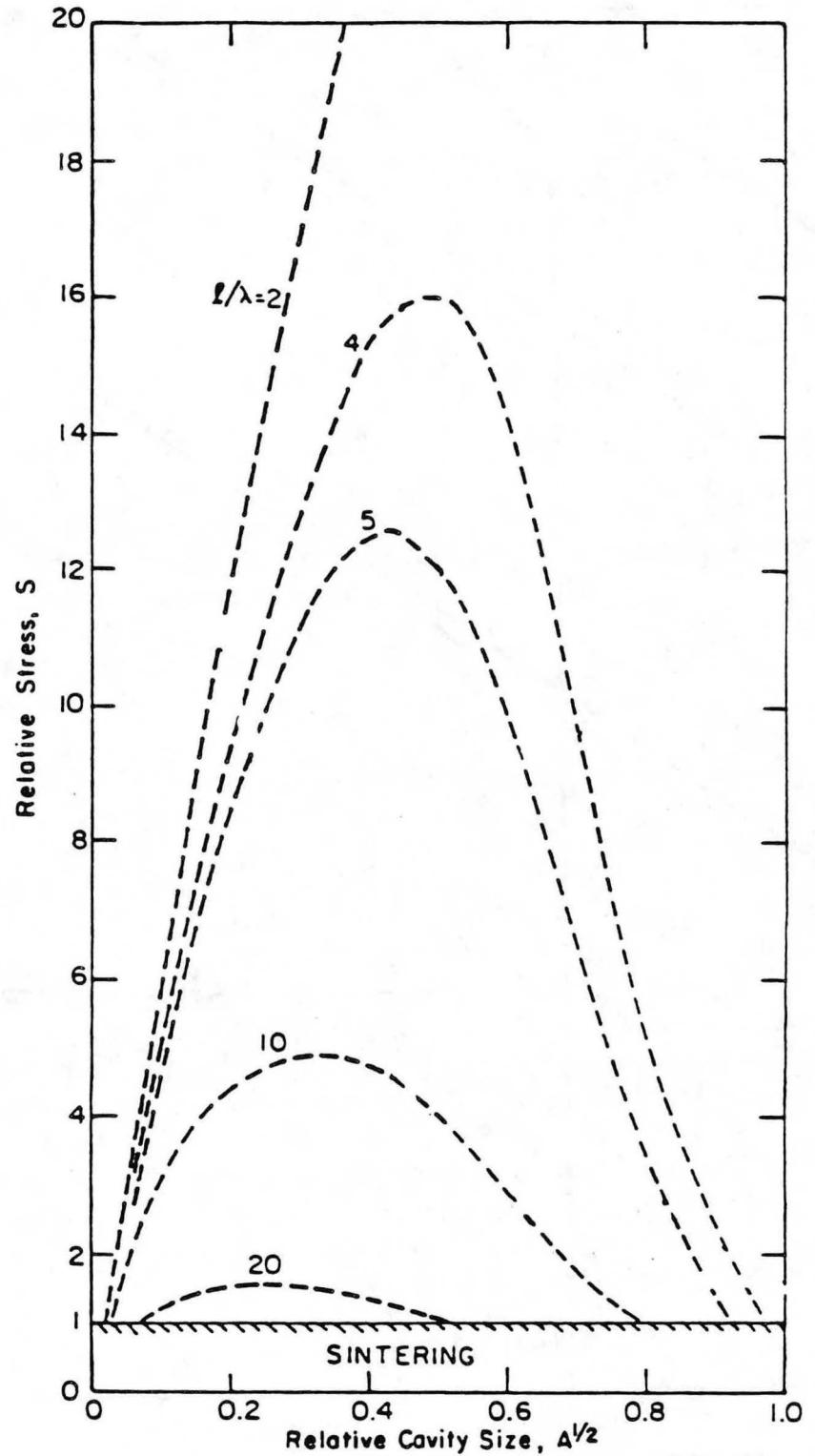


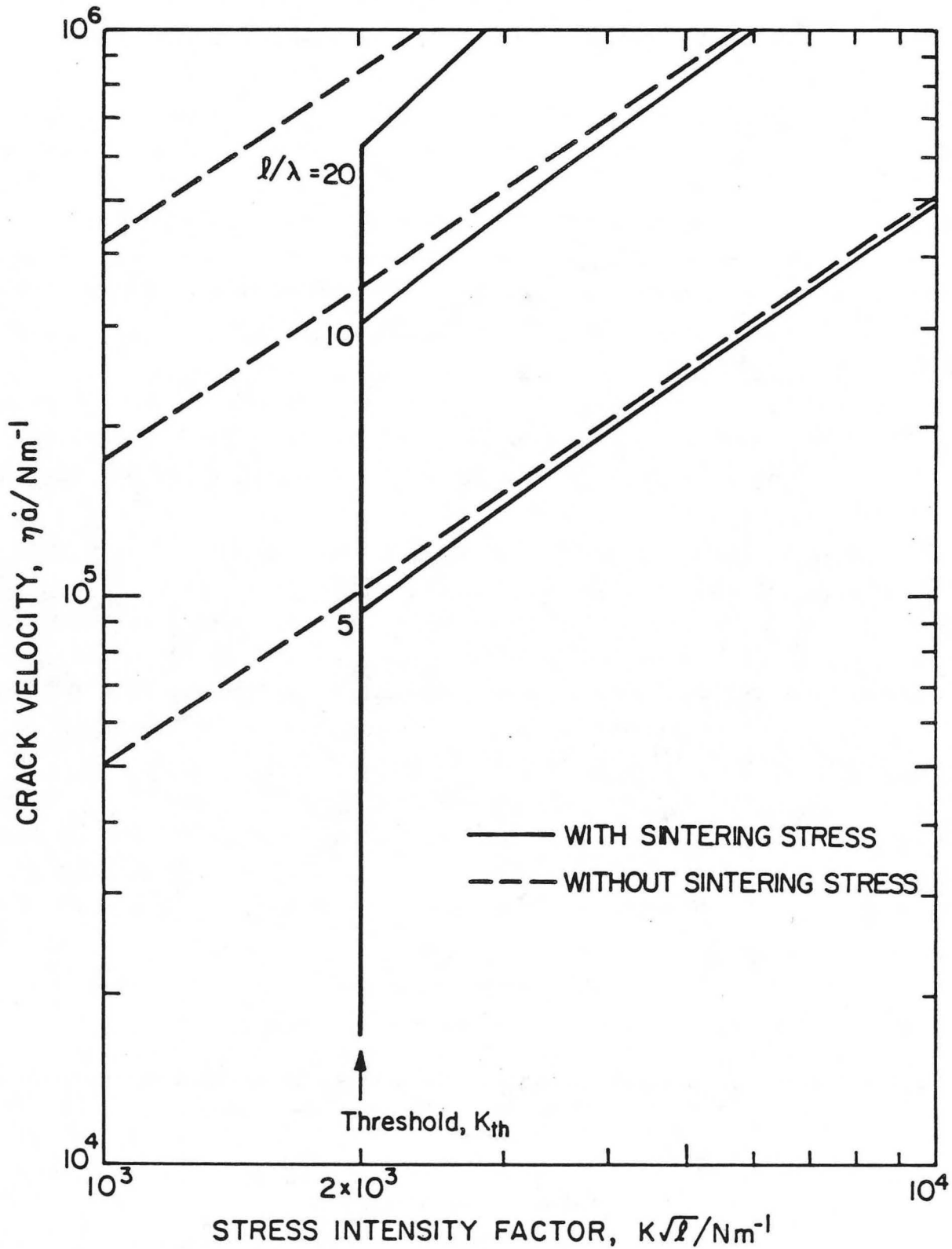
Fig. 18. A plot of the normalized stress against the ratio of the distance ahead of the crack to the grain size, at various times between increments of crack advance ( $\delta t/\Delta t$ ).  $\zeta/\xi = 1$ . (See also the note on the caption of Fig. 17).

XBL832-5235



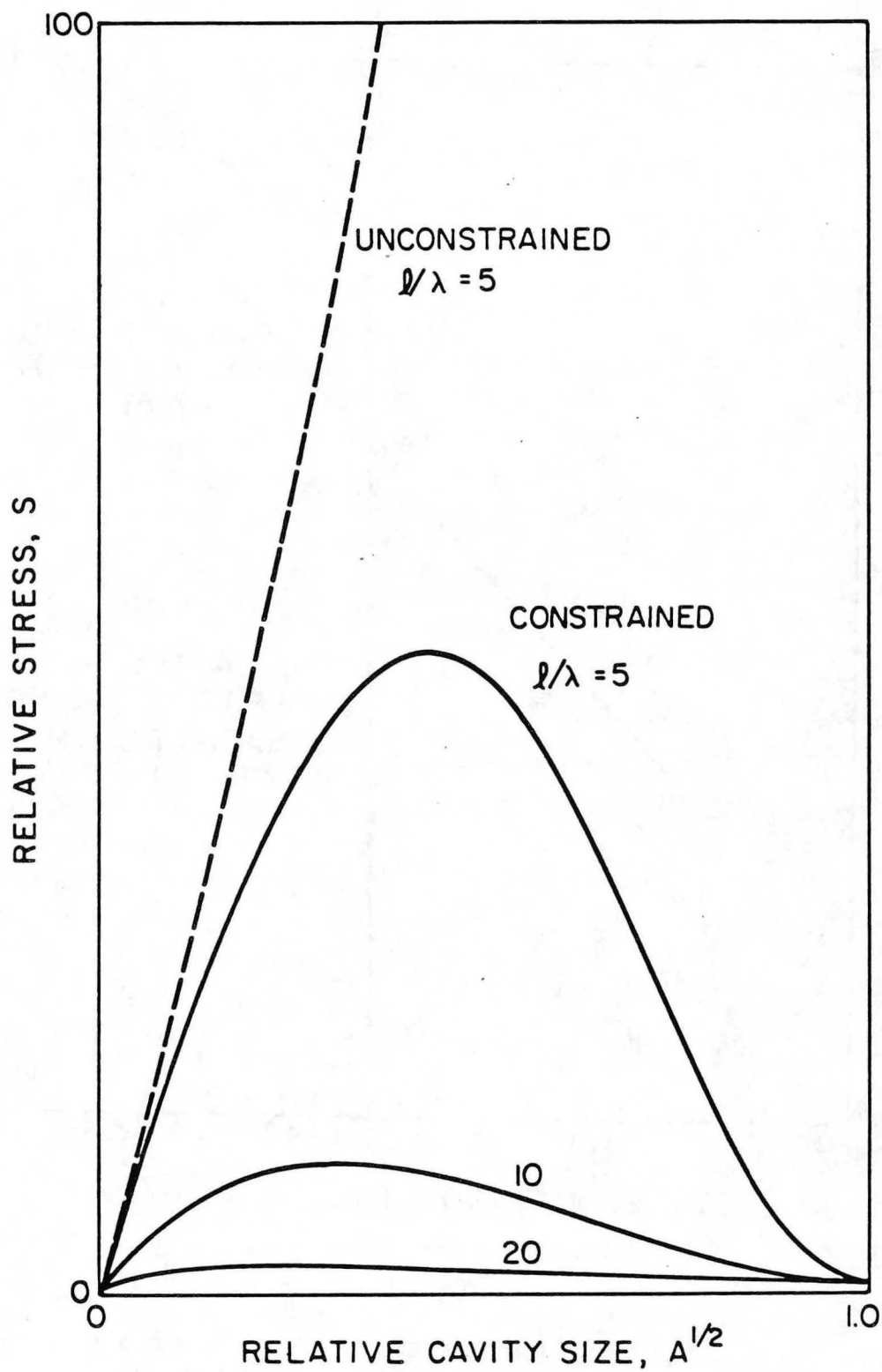
XBL 832-5234A

Fig. 19. A plot of the relative stress on the damage zone (assumed to consist of a single grain) as a function of the cavity size,  $\gamma = 1 \text{ Jm}^{-1}$ ,  $K = 3 \text{ MPa}\sqrt{\text{m}}$ ,  $l = 2 \times 10^{-6} \text{ m}$ ,  $\psi = 75^\circ$ . The effect of the sintering stress has been ignored.



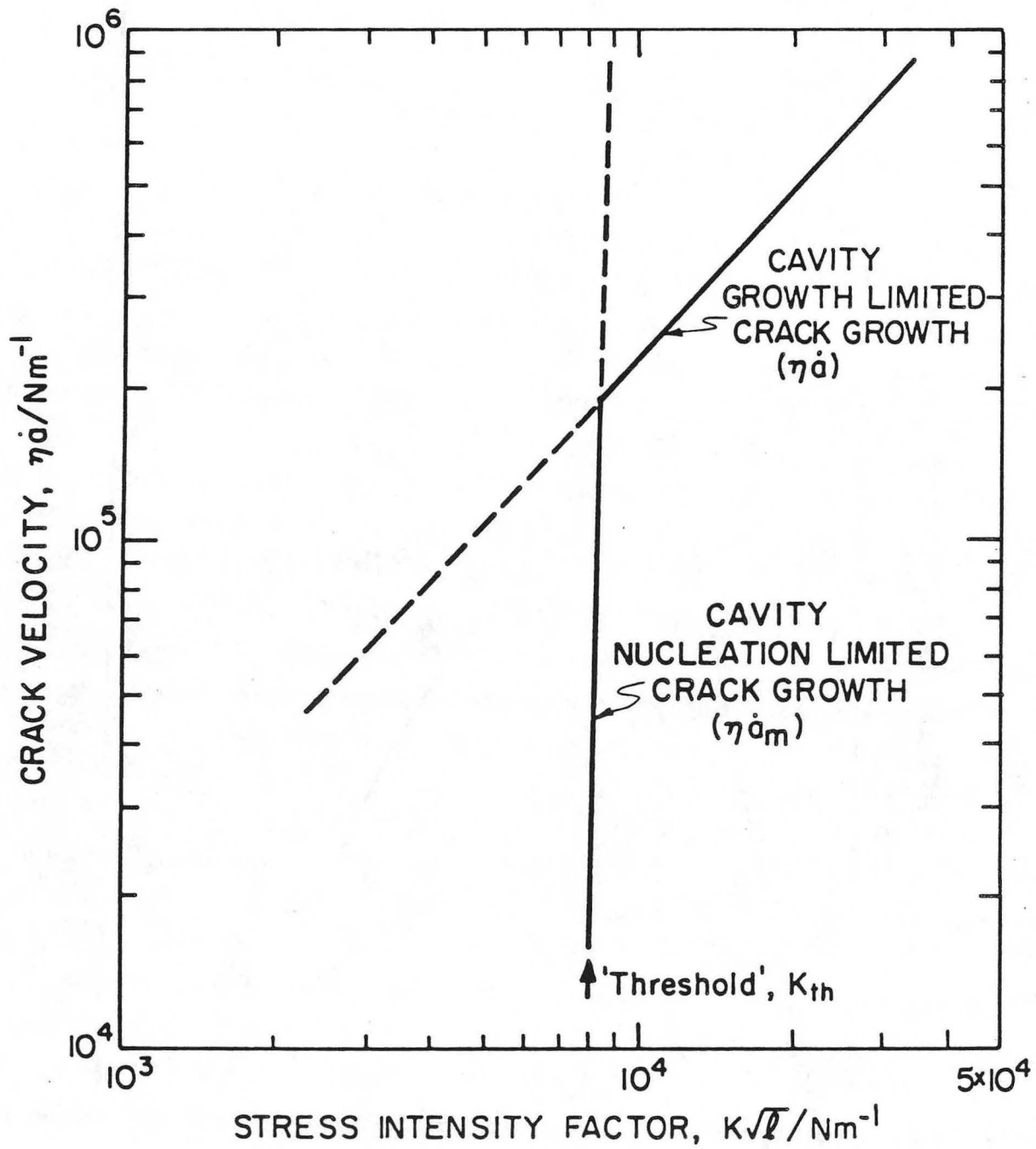
XBL847-7146

Fig. 20. A plot of the product of the crack valocity (constrained) and viscosity against  $K\sqrt{l}$  illustrating the effect of the inclusion of a sintering stress in the model.  
 $K_{th}\sqrt{l} = 2 \times 10^3 \text{ Nm}^{-1/2}$ .



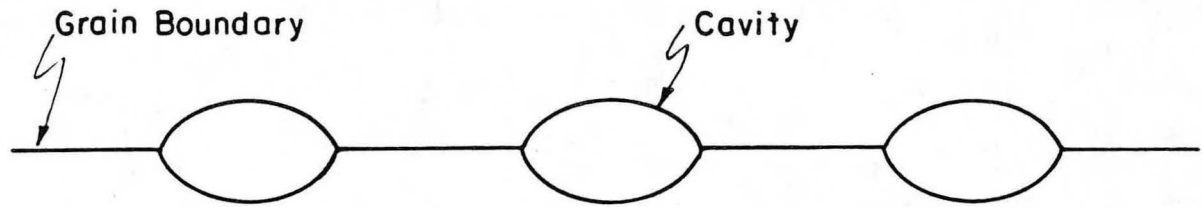
XBL847-7147

Fig. 21. A plot of the relative stress on a damage zone of a single grain as a function of the cavity size,  $\gamma = 1 \text{ Jm}^{-1}$ ,  $K\sqrt{\ell} = 4 \times 10^3 \text{ Pa m}$ ,  $K_{th}\sqrt{\ell} = 2 \times 10^3 \text{ Pa m}$ .



XBL 845-6946 A

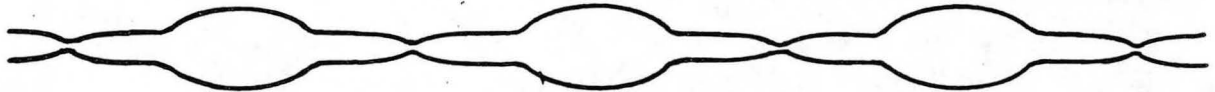
Fig. 22. A plot of  $\eta \dot{a}$  against  $K\sqrt{l}$ , illustrating how a threshold may arise if crack propagation is limited by cavity nucleation.



(a) EQUILIBRIUM CAVITIES ON GRAIN BOUNDARY



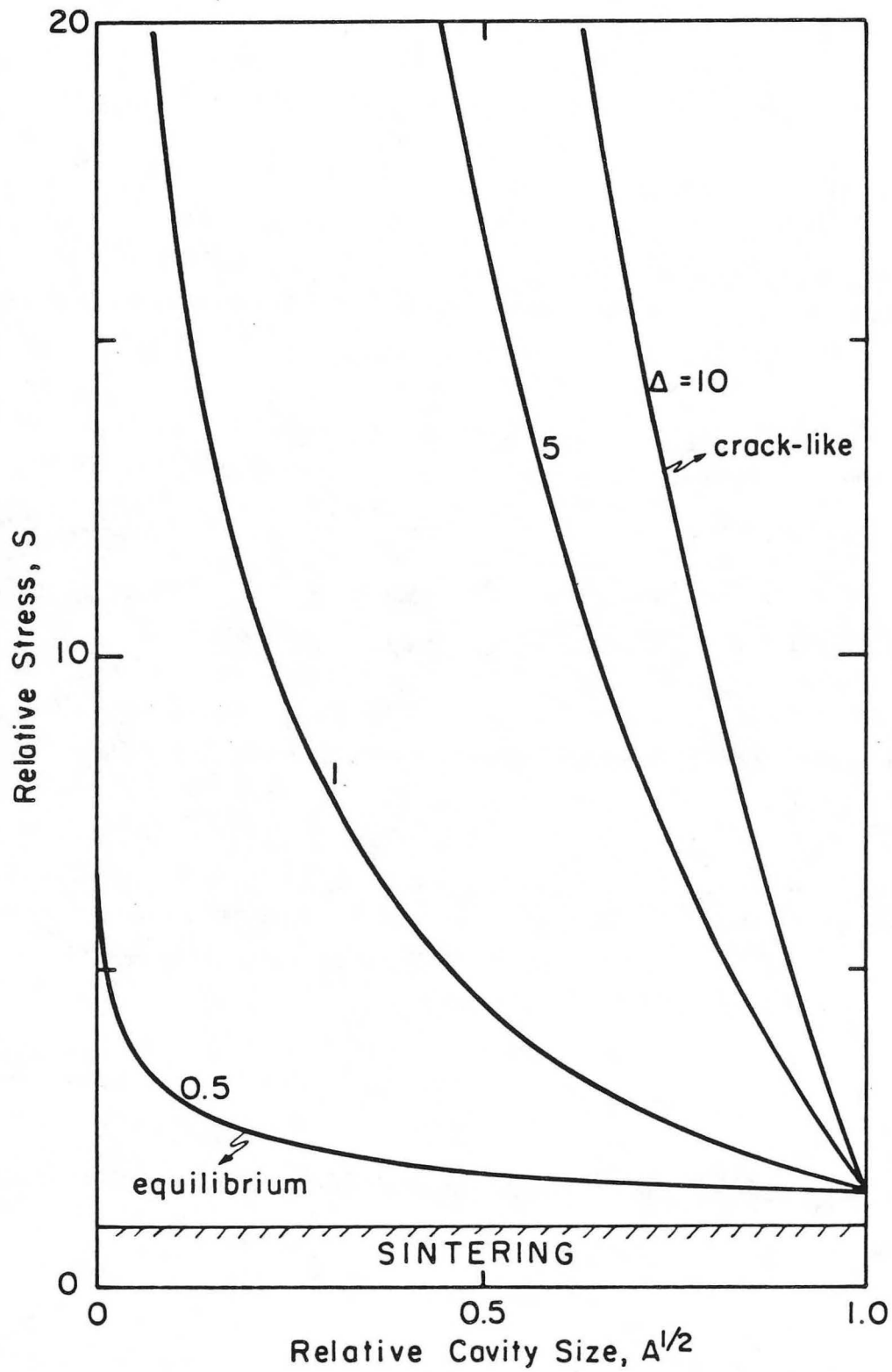
(b) TRANSITION TO CRACK-LIKE CAVITIES



(c) FAILURE OF GRAIN BOUNDARY

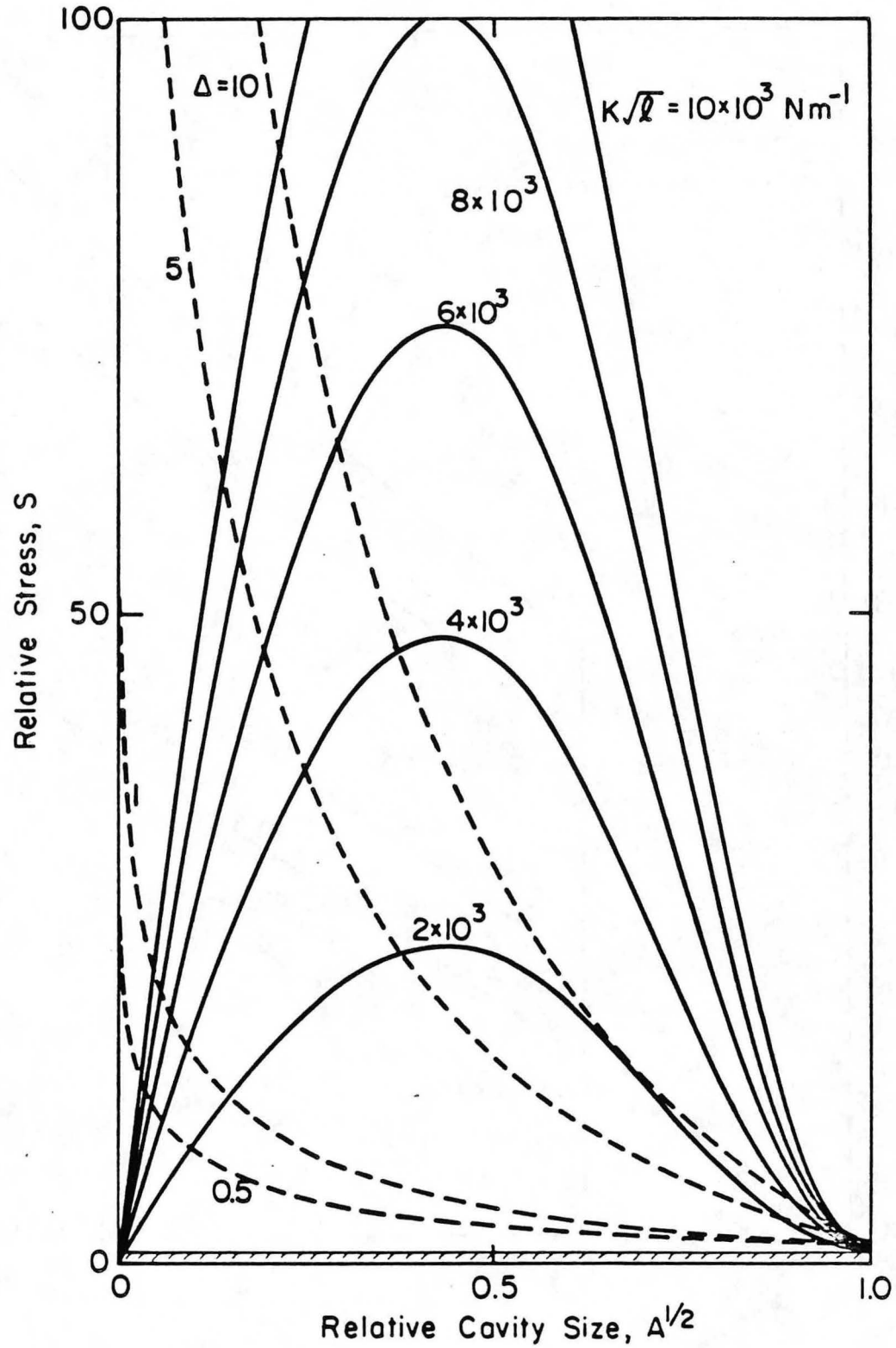
XBL 832-5233

Fig. 23. The separation of ligaments by a transition to crack-like cavity growth.



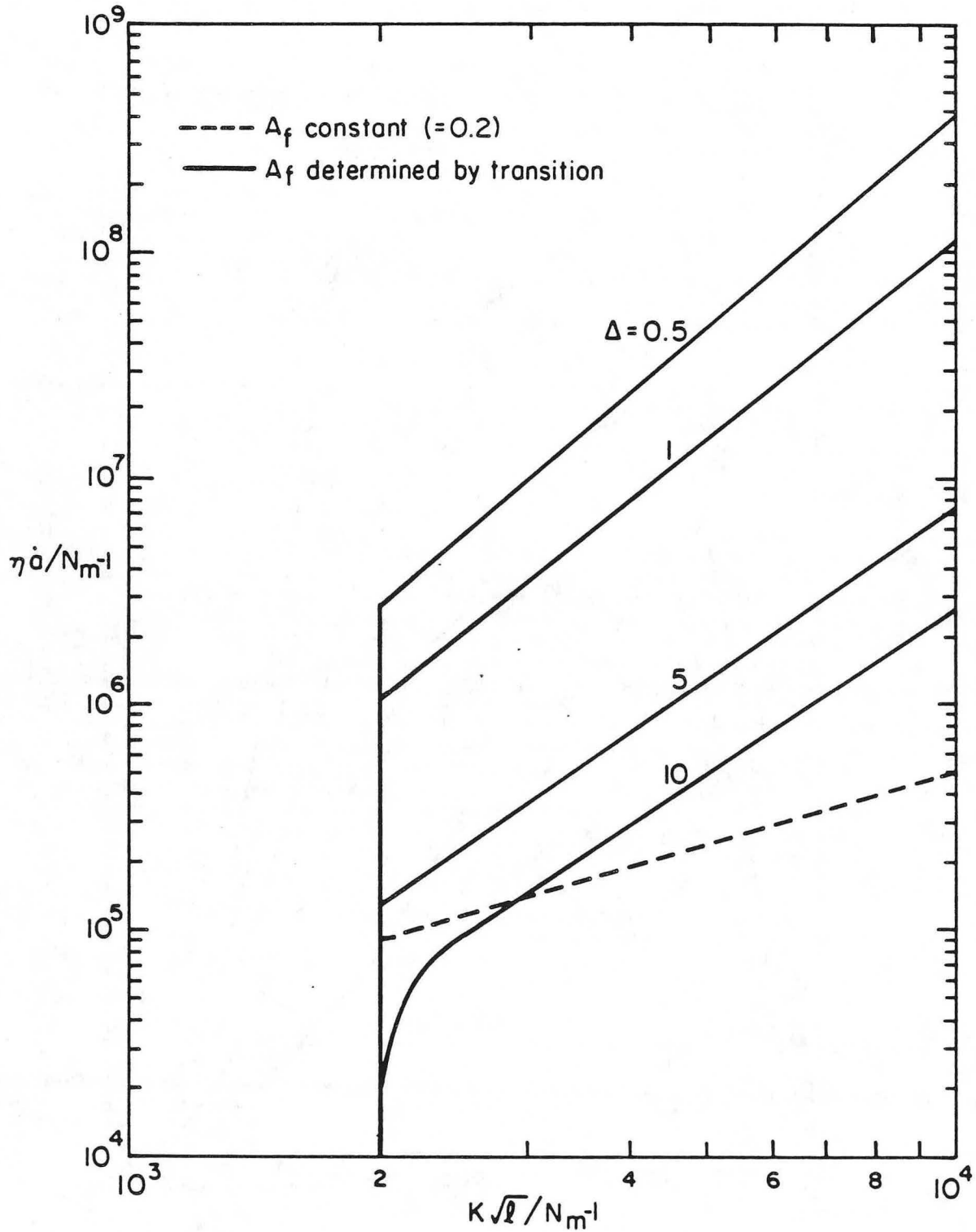
XBL 8410-7428

Fig. 24. The transition curves for equilibrium to crack-like growth as a function of the ratio of the applied stress to sintering stress ( $s$ ) and the cavity size, for various values of the boundary to surface diffusivity ratios ( $\Delta$ ).



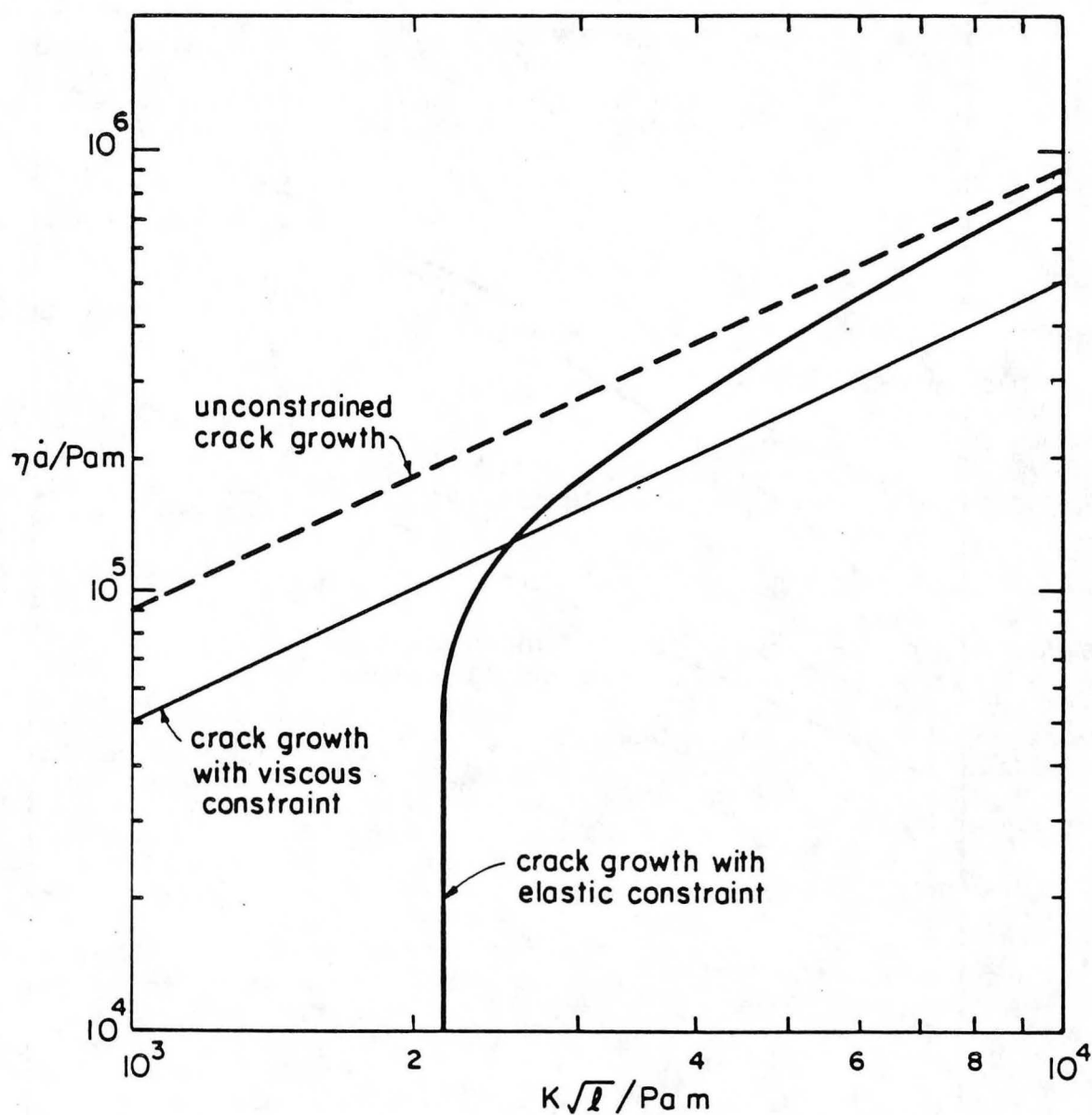
XBL 8410-74 29

Fig. 25. The variation in the resultant stress on the damage zone changes the cavity growth from an equilibrium to a crack-like mechanism.



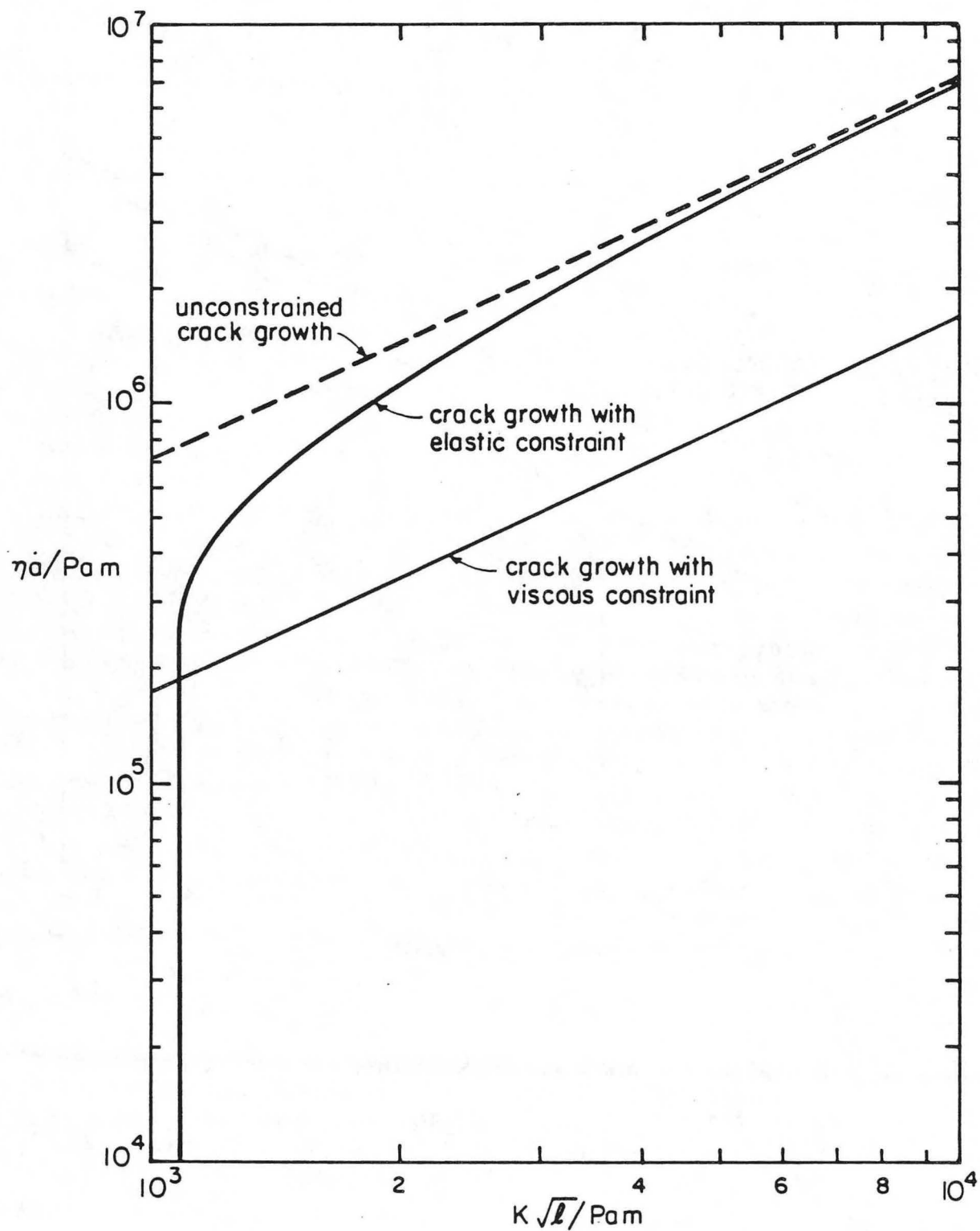
XBL 8410-7420

Fig. 26. A plot of the resultant crack velocity against applied stress intensity factor, assuming that the critical cavity size for coalescence is given by the size at transition.  $\ell/\lambda = 5$ ,  $K\sqrt{\ell} = 4 \times 10^3 \text{ Nm}^{-1}$ ,  $K_{th}\sqrt{\ell} = 2 \times 10^3 \text{ Nm}^{-1}$ .



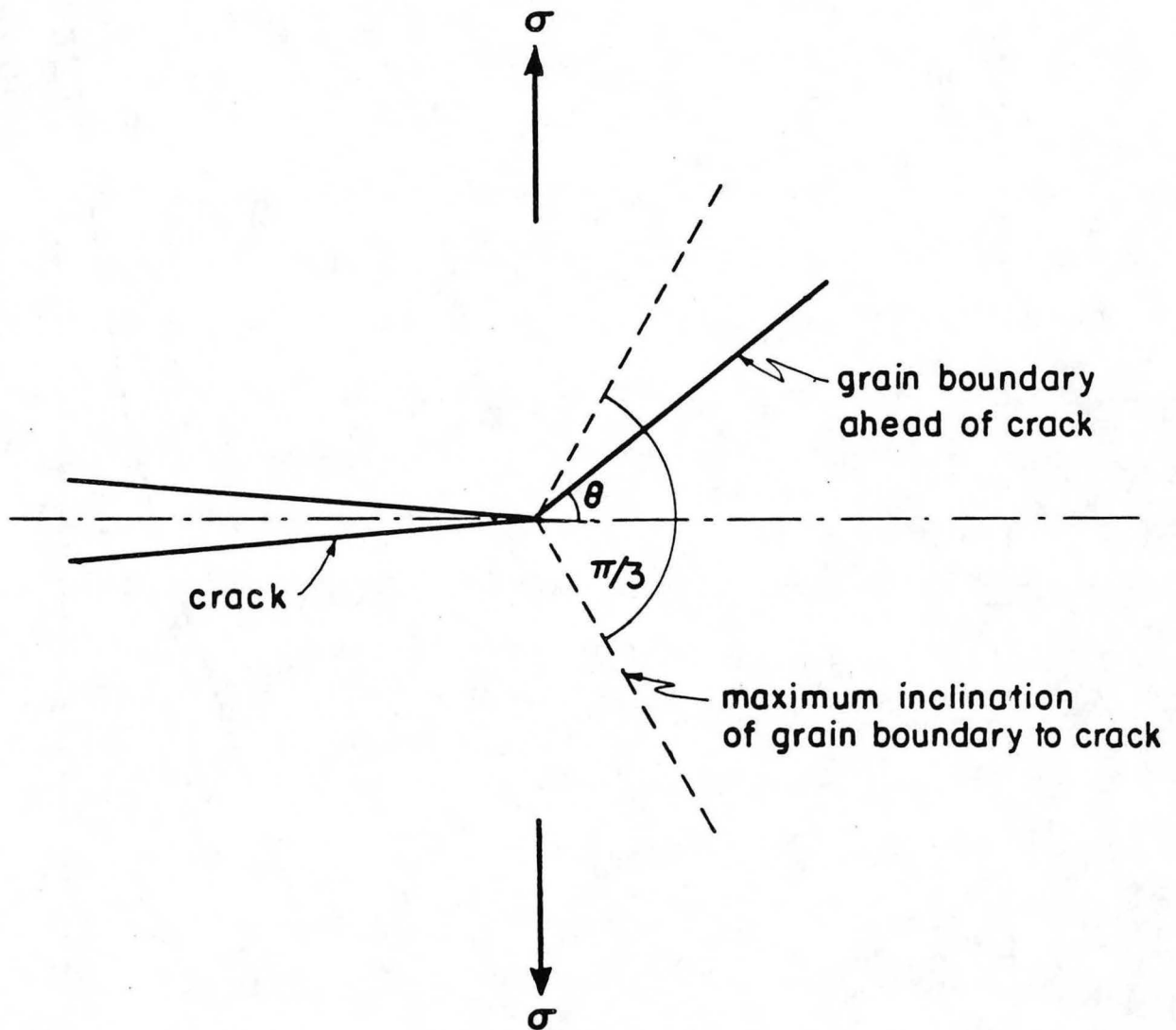
XBL 8410-7417

Fig. 27. A plot of the crack velocity against applied stress intensity factor, showing the relationship between the unconstrained, viscously constrained and elastically constrained solutions. The values of the parameters used are  $A_f = 0.2$ ,  $l/\lambda = 5$ ,  $E = 300 \text{ GPa}$ , and  $l = 10^{-6} \text{ m}$ .



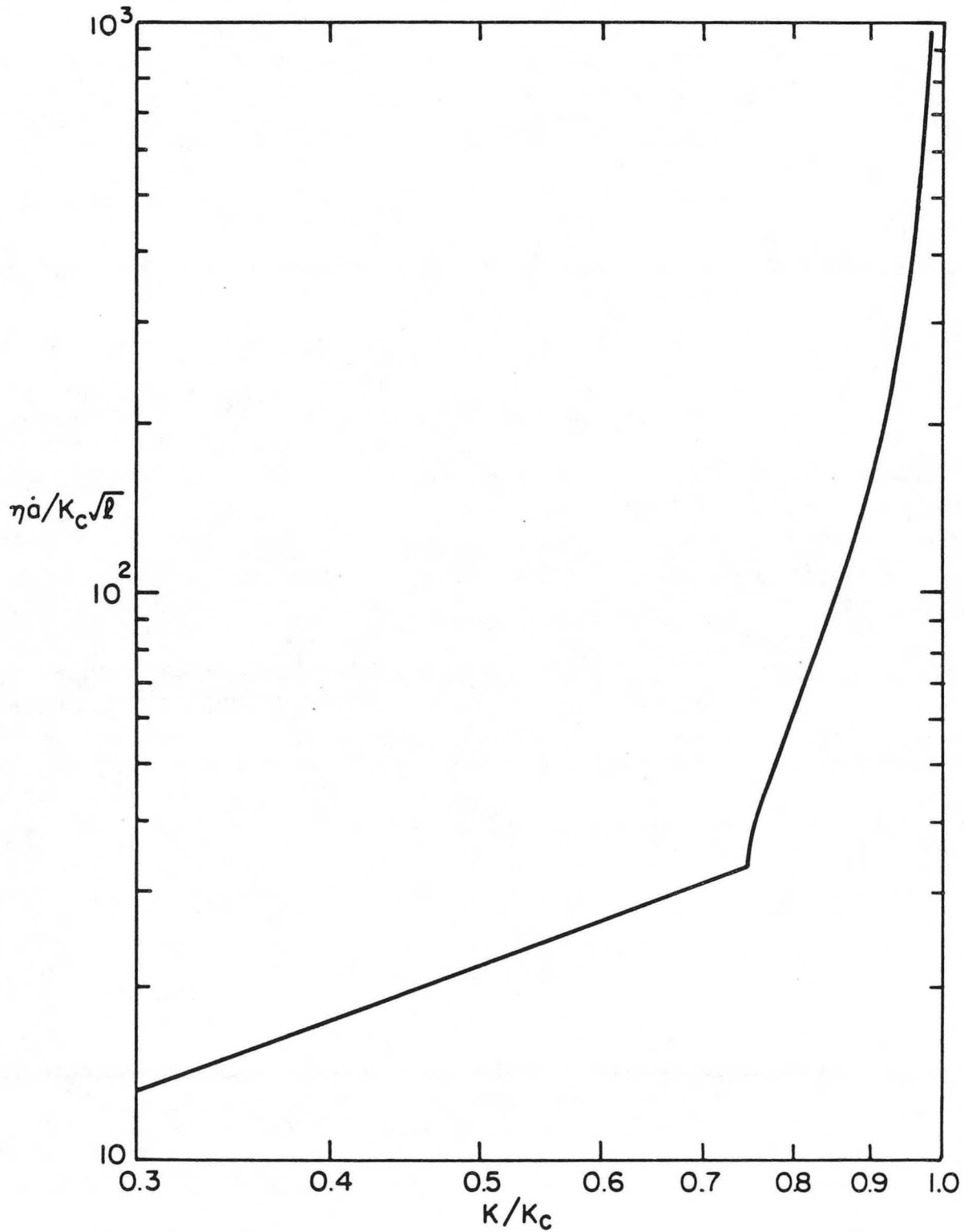
XBL8410-7421

Fig. 28. A plot of the crack velocity against applied stress intensity factor showing the relationship between the unconstrained, viscously constrained and elastically constrained solutions. The values of the parameters used are  $A_f = 0.2$ ,  $l/\lambda = 10$ ,  $E = 300 \text{ GPa}$  and  $l = 10^{-6} \text{ m}$ .



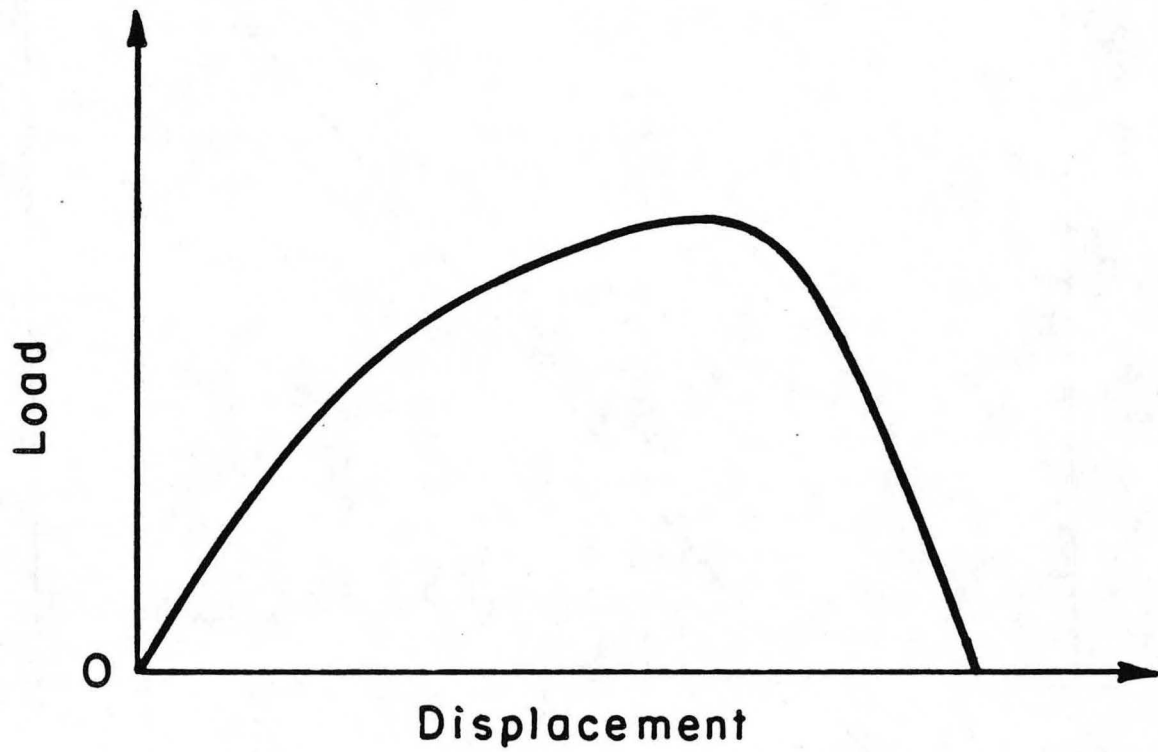
XBL 8410-7422

Fig. 29. A schematic illustrating the configuration considered in Section 4.2. A grain boundary extends at an angle  $\theta$  to the axis of the crack, the magnitude of  $\theta$  can take any value between 0 and  $\pi/3$ .



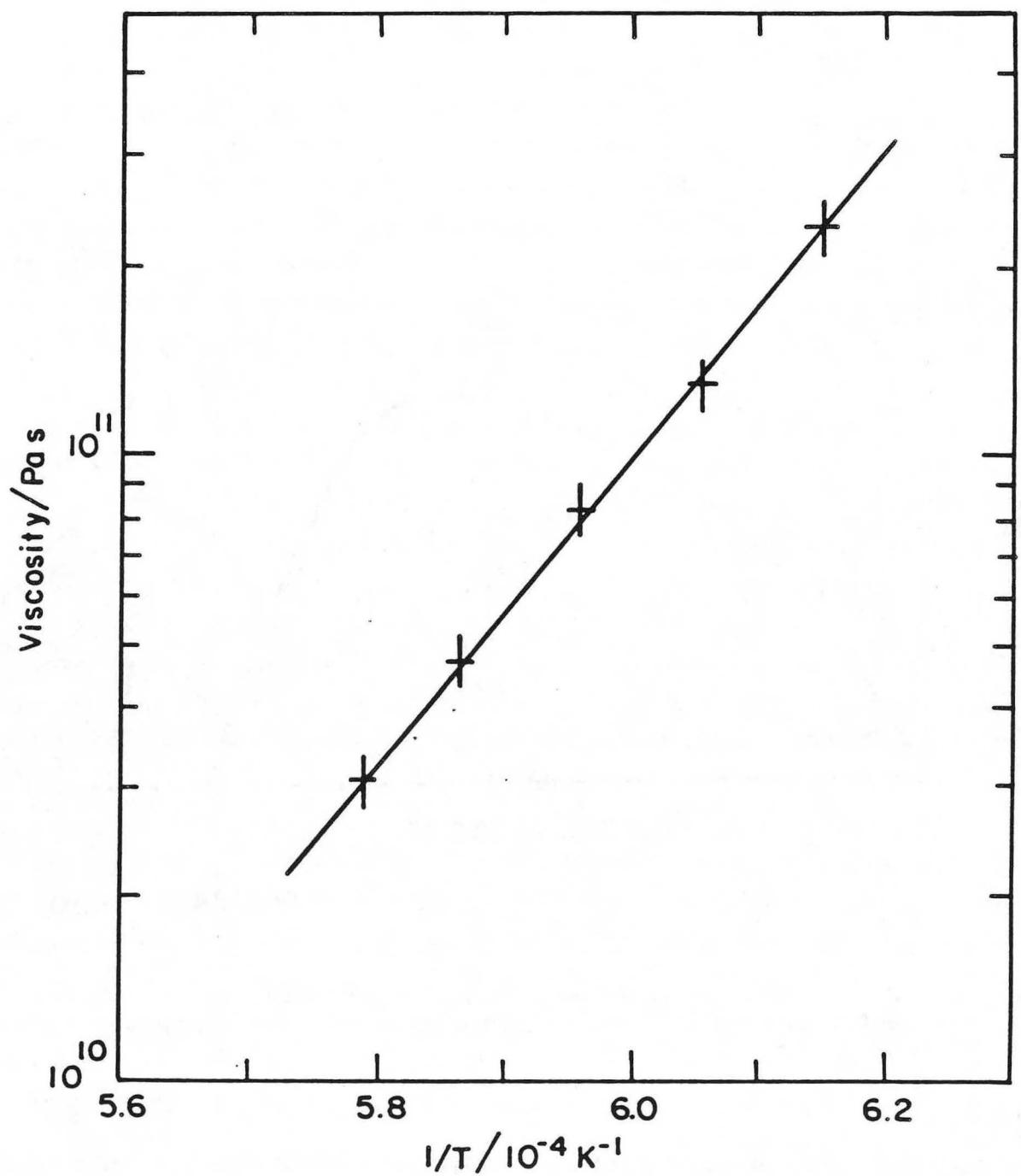
XBL 8410-7419

Fig. 30. A plot of normalized crack velocity against  $K/K_c$ , showing the influence of the critical stress intensity factor.  $A_f = 0.2$  and  $l/\lambda = 5$ .



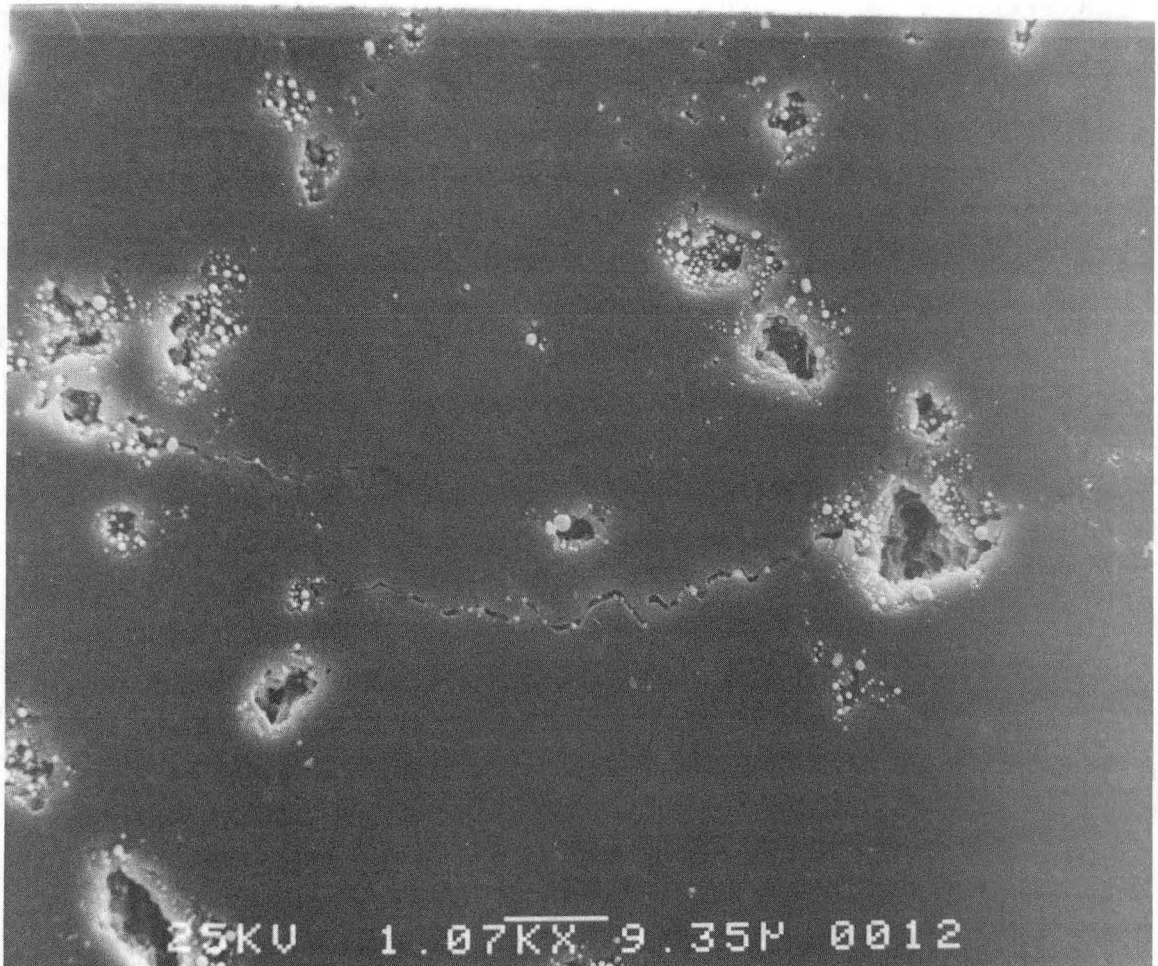
XBL 8410-7430

Fig. 31. A schematic illustrating a typical load/displacement plot obtained for liquid phase sintered alumina in four point bending tests.



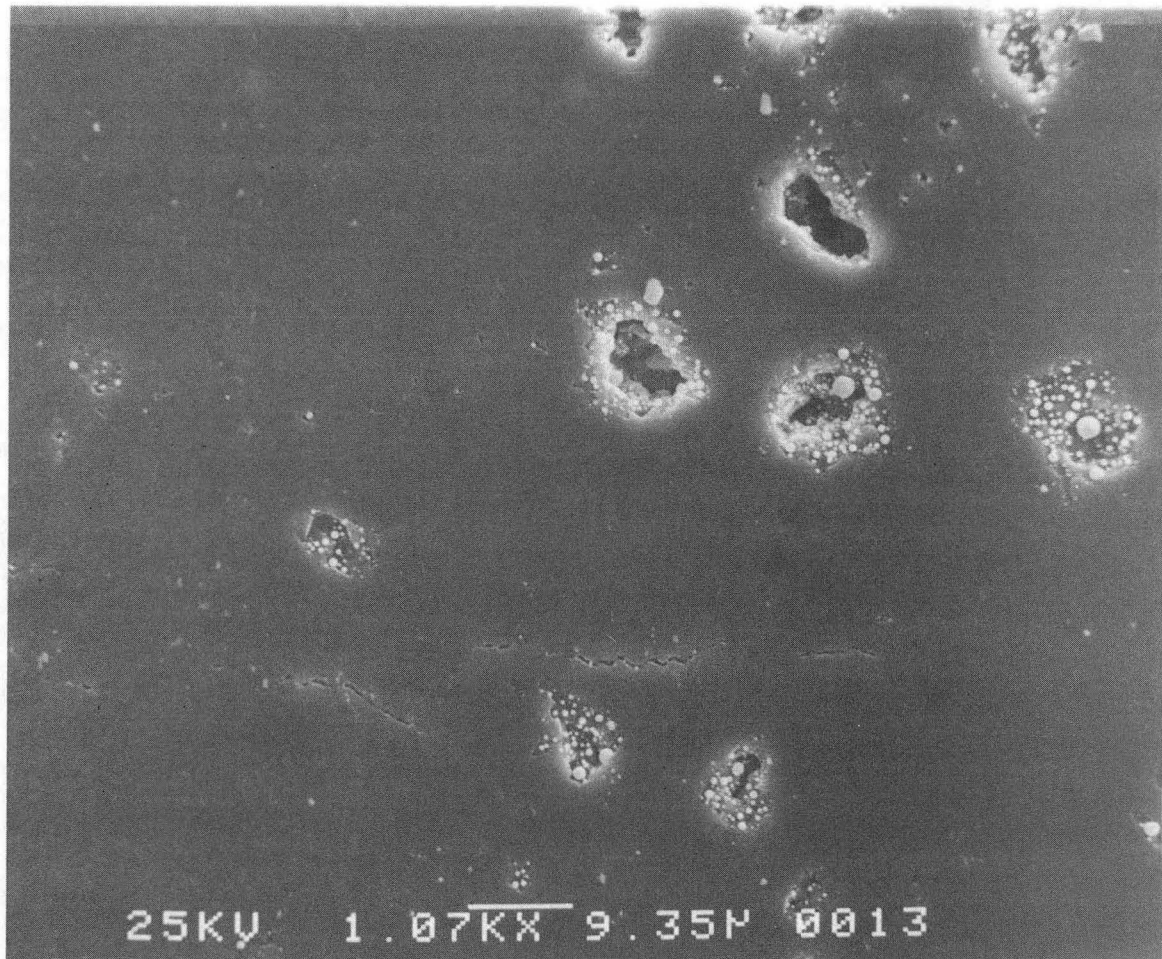
XBL 8410-7425

Fig. 32. An Arrhenius plot for the viscosity of the liquid phase sintered alumina.



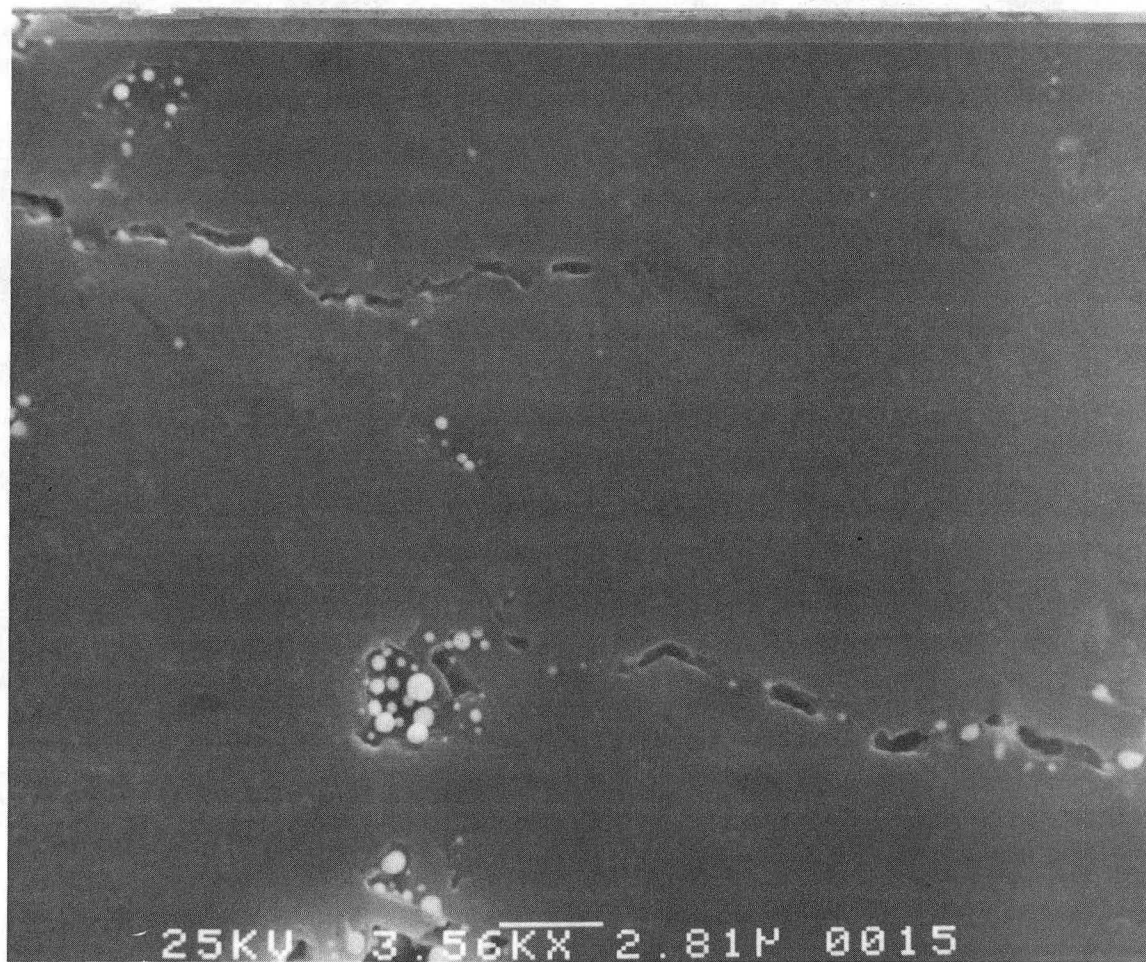
XBB 830-10872

Fig. 33. S.E.M. micrograph showing the association of some microcracks with porous regions. The white spheres have been identified as gold from the coating process.



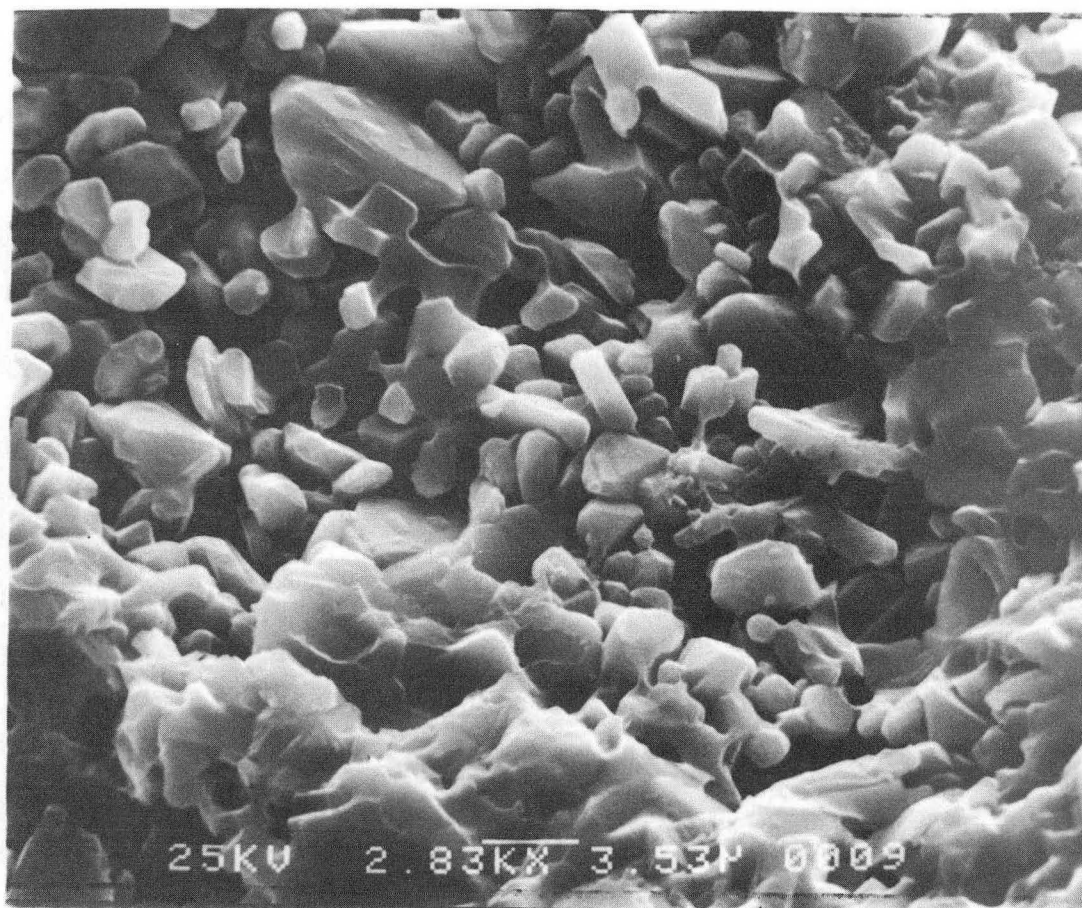
XBB 830-10873

Fig. 34. S.E.M. micrograph showing the nucleation of a crack in an apparently homogeneous region.



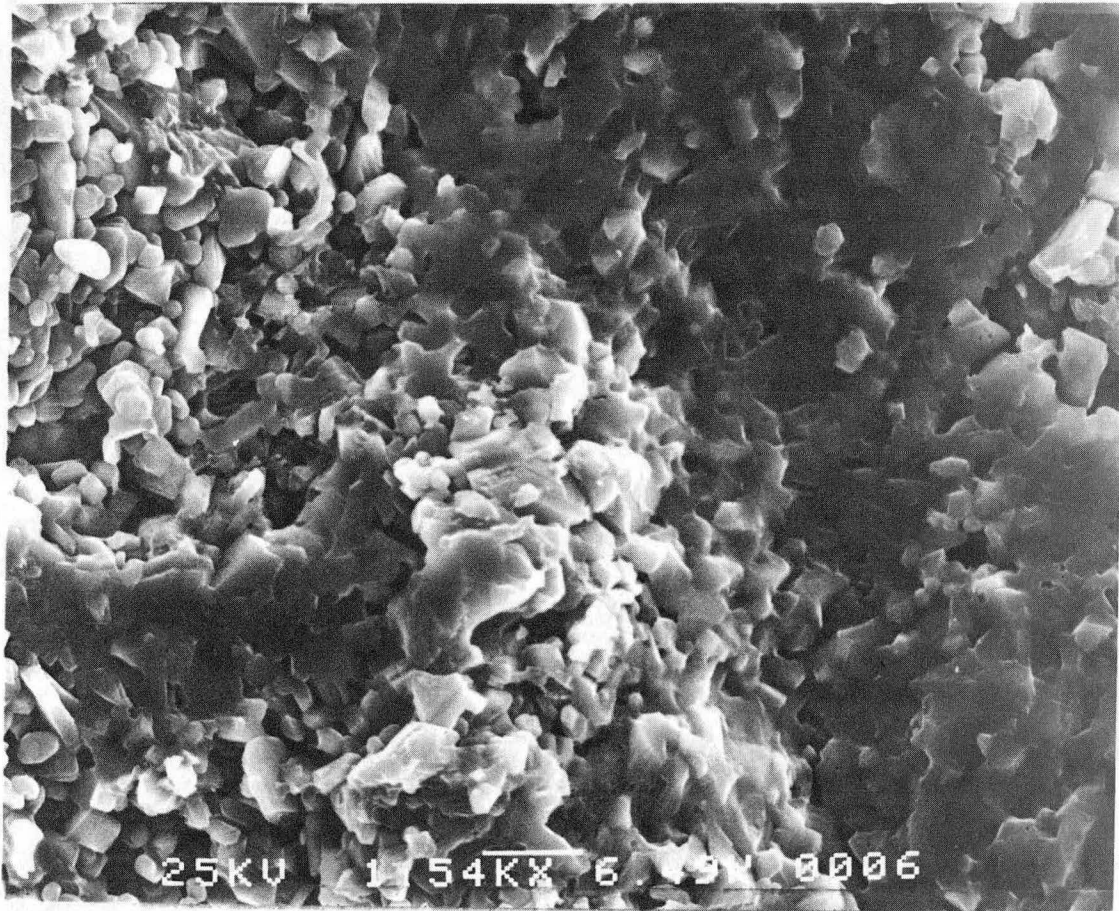
XBB 830-10870

Fig. 35. S.E.M. micrograph of crack tips, illustrating the zone presumed to contain amorphous material.



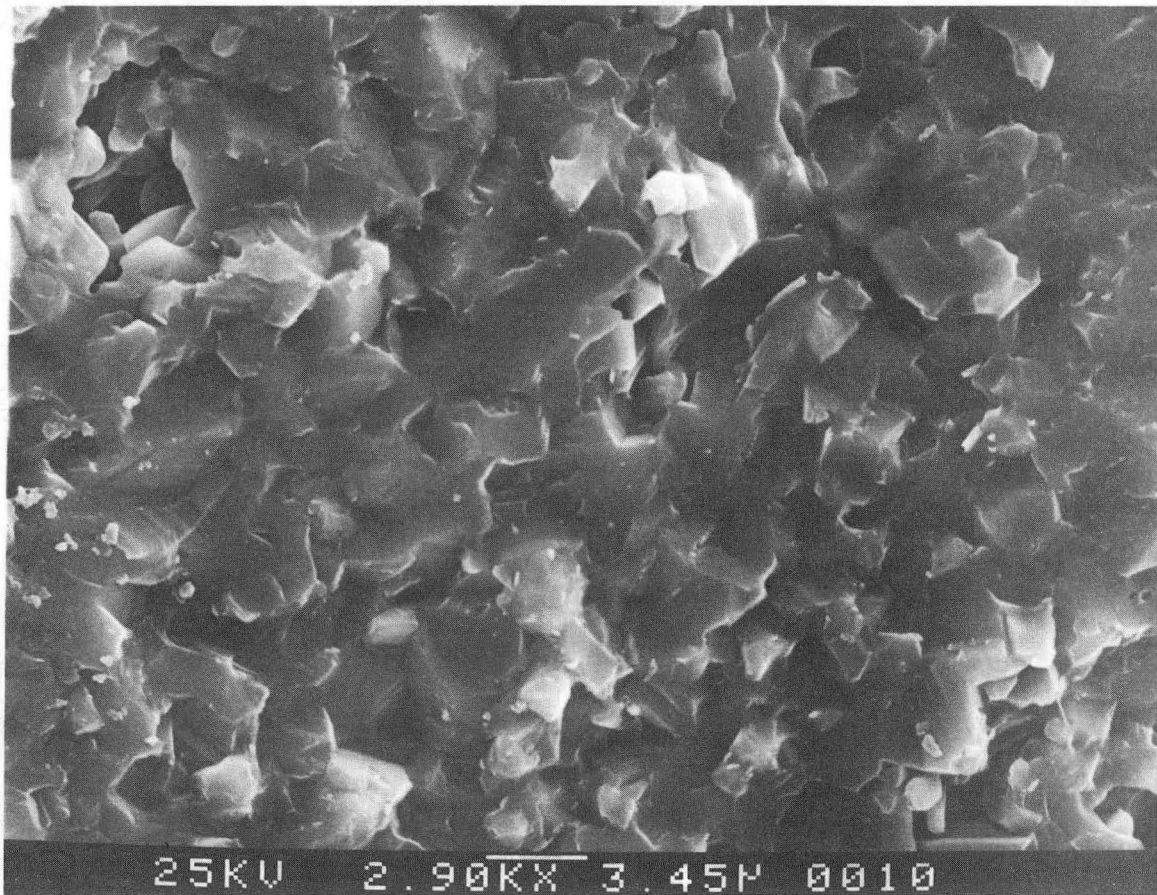
XBB 830-10725

Fig. 36. S.E.M. micrograph of the fracture surface of a liquid phase sintered alumina. The specimen was held at load while being cooled to room temperature, it was then fractured. The picture shows a region immediately behind the crack front, illustrating the ligaments and islands of amorphous material.



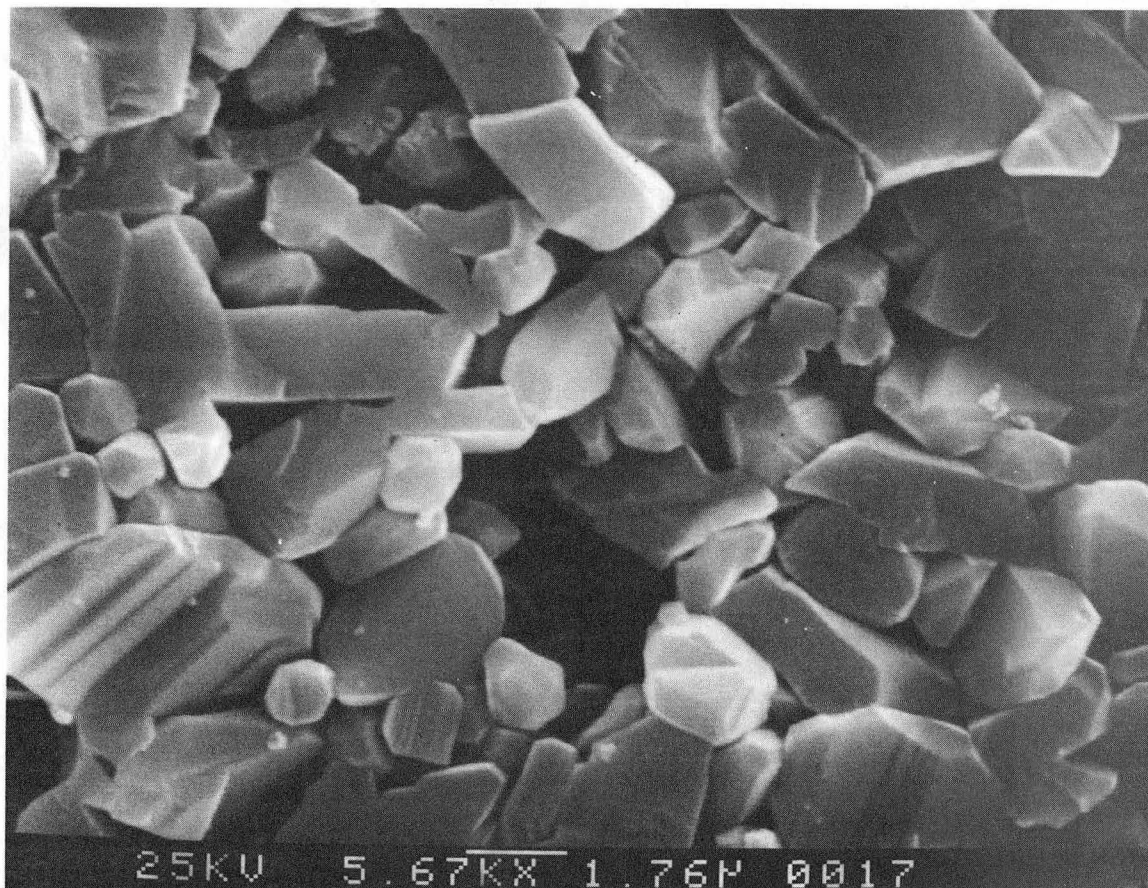
XBB 830-10727

Fig. 37. S.E.M. micrograph of the fracture surface of a liquid phase sintered alumina after an interrupted test. The crack front can be identified by the transition in the appearance of the surface. The creep damaged region is intergranular, whereas the region fractured at room temperature is transgranular.



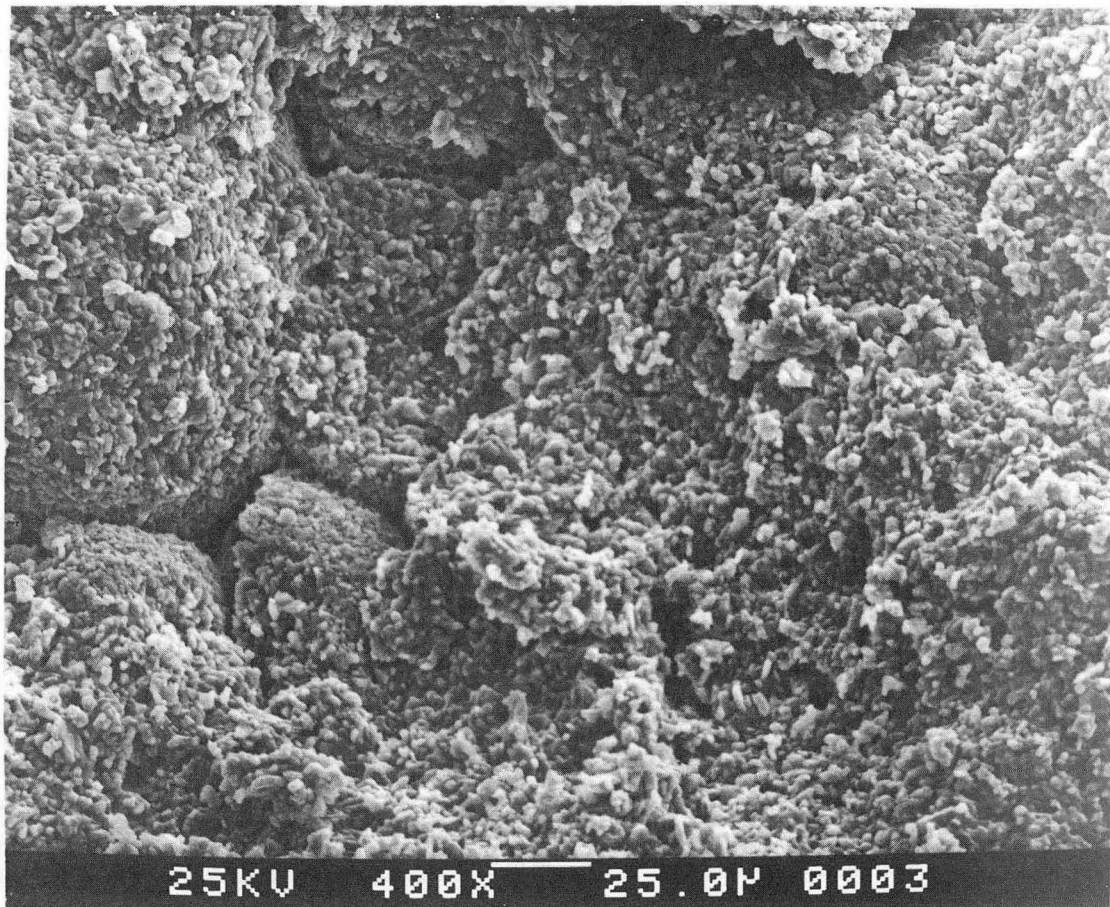
XBB 848-6572

Fig. 38. S.E.M. micrograph of a room temperature fracture surface of a liquid phase sintered alumina.



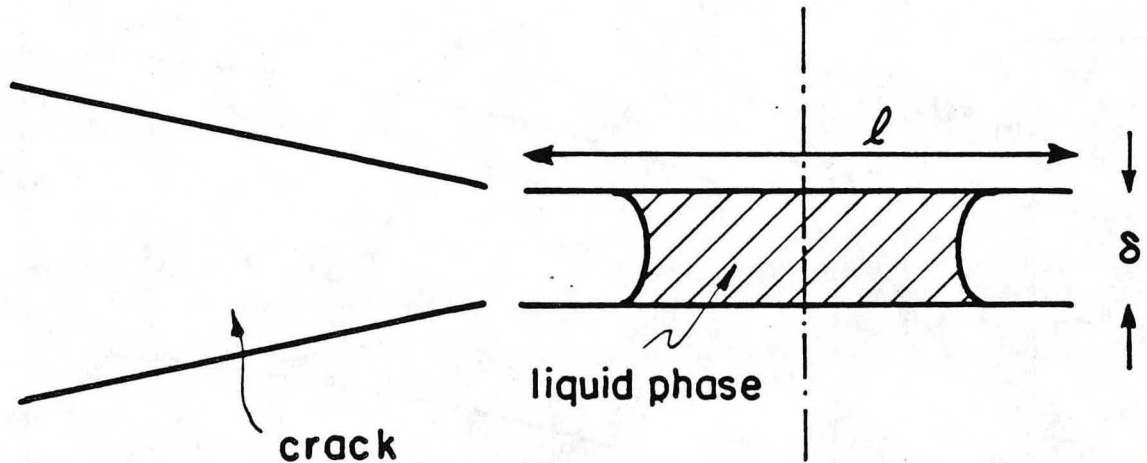
XBB 848-6573

Fig. 39. S.E.M. micrograph of a room temperature fracture surface of a liquid phase sintered alumina after an etch in 5% HF acid for 20 min. The glassy phase has been removed to clearly reveal the transgranular nature of the fracture surface.



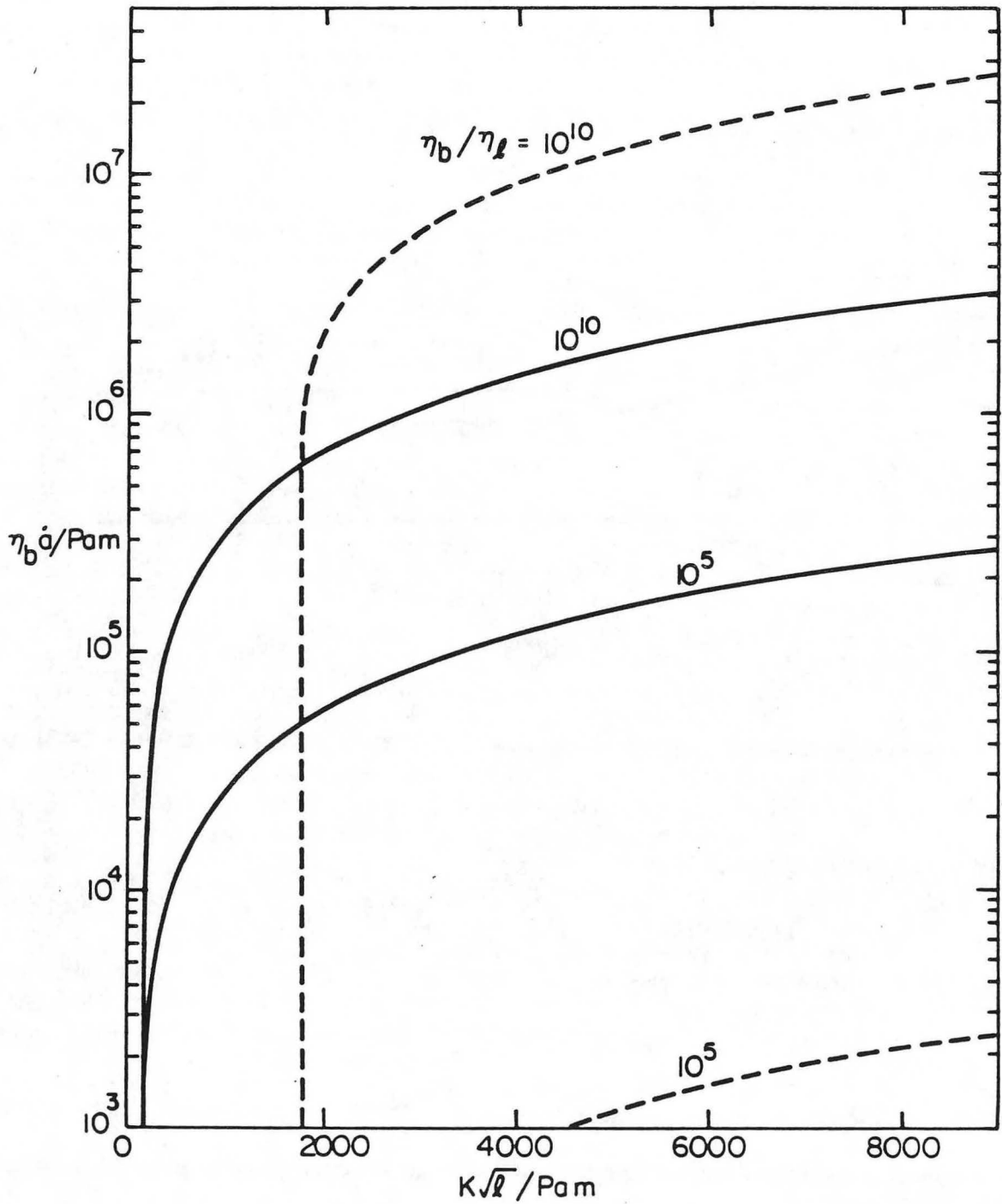
XBB 830-10722

Fig. 40. S.E.M. micrograph of an inter-agglomerate failure surface obtained at high strain rates.



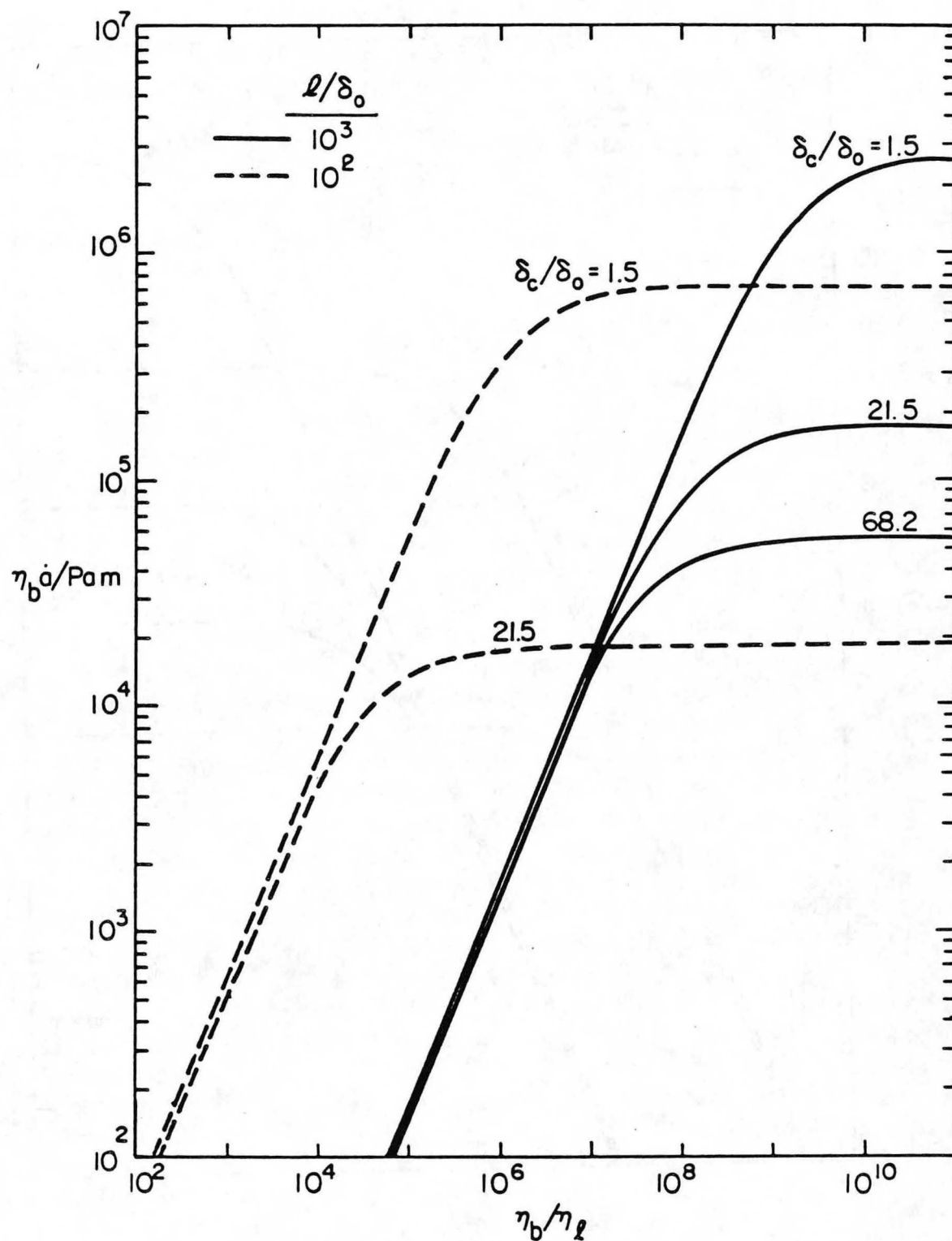
XBL 8410-7431

Fig. 41. The theoretical configuration considered in Section 5.1. Ahead of the crack there exists a grain boundary of length  $l$ , filled with a liquid phase of thickness  $\delta$ . Cavities are initiated in the triple points at the end of the grain and grow towards the center.



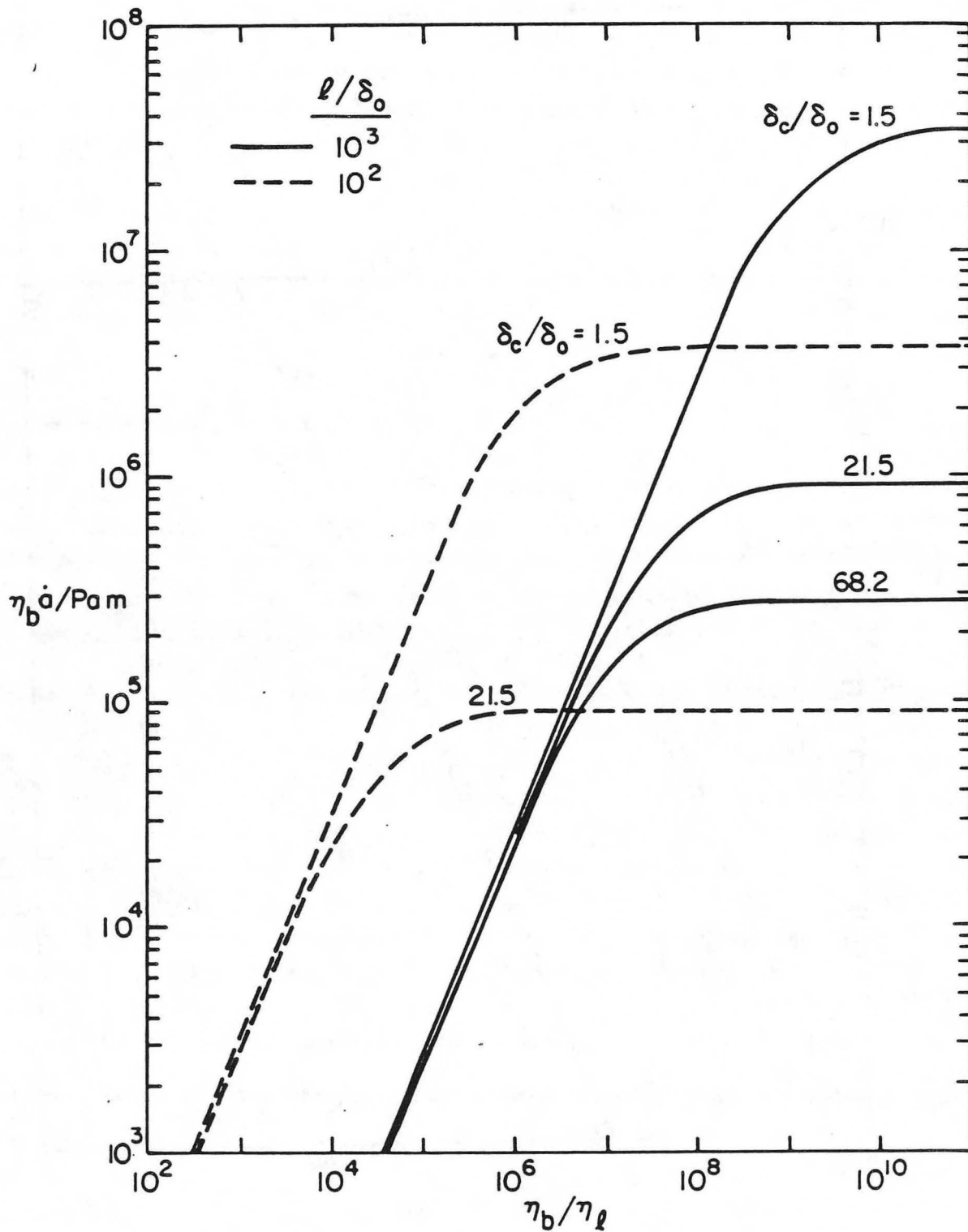
XBL 8410-7418

Fig. 42. A theoretical plot of  $\eta_b \dot{a}$  against  $K\sqrt{\ell}$  for a liquid phase sintered material. The ratio of the critical to initial film thickness has been taken to be 1.5. (---) represents the plot for  $\ell/\delta_0 = 100$ , and (—) represents the plot for  $\ell/\delta_0 = 1000$ .



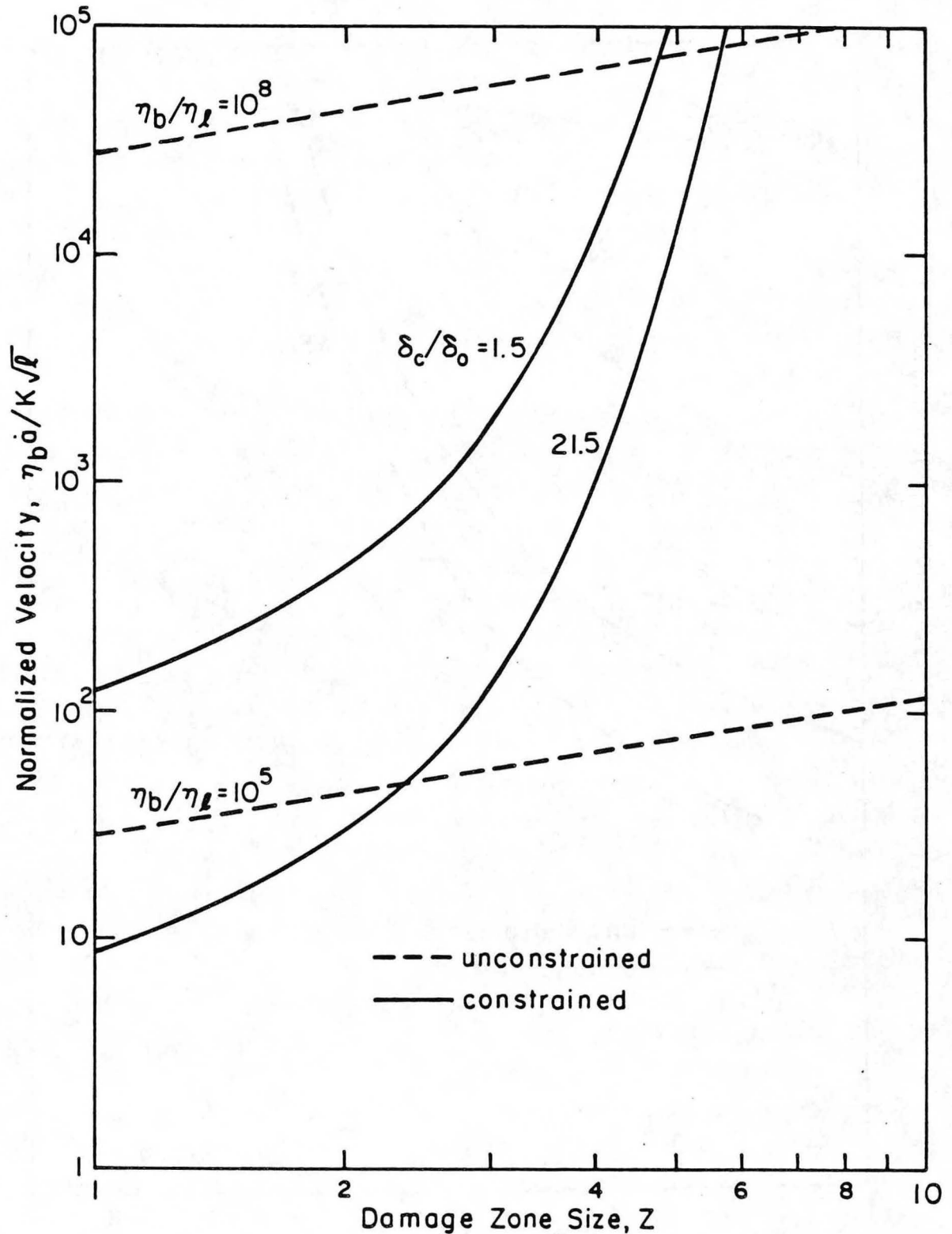
XBL 8410-7423

Fig. 43. A theoretical plot of  $\eta_b \dot{a}$  against the ratio of the bulk to liquid phase viscosities.  $K\sqrt{l} = 2 \times 10^3 \text{ Nm}^{-1}$ .



XBL 8410-7424

Fig. 44. A theoretical plot of  $\eta_b \cdot a$  against the ratio of the bulk to liquid phase viscosities.  $K\sqrt{l} = 10 \times 10^3 \text{ Nm}^{-1}$ .



XBL 8410-7427

Fig. 45. Theoretical plots of the normalized velocity against the number of grains in the damage zone size (z) for a liquid phase sintered material. The difference between the unconstrained and constrained solutions is illustrated.  $\ell/\delta_0 = 100$ .

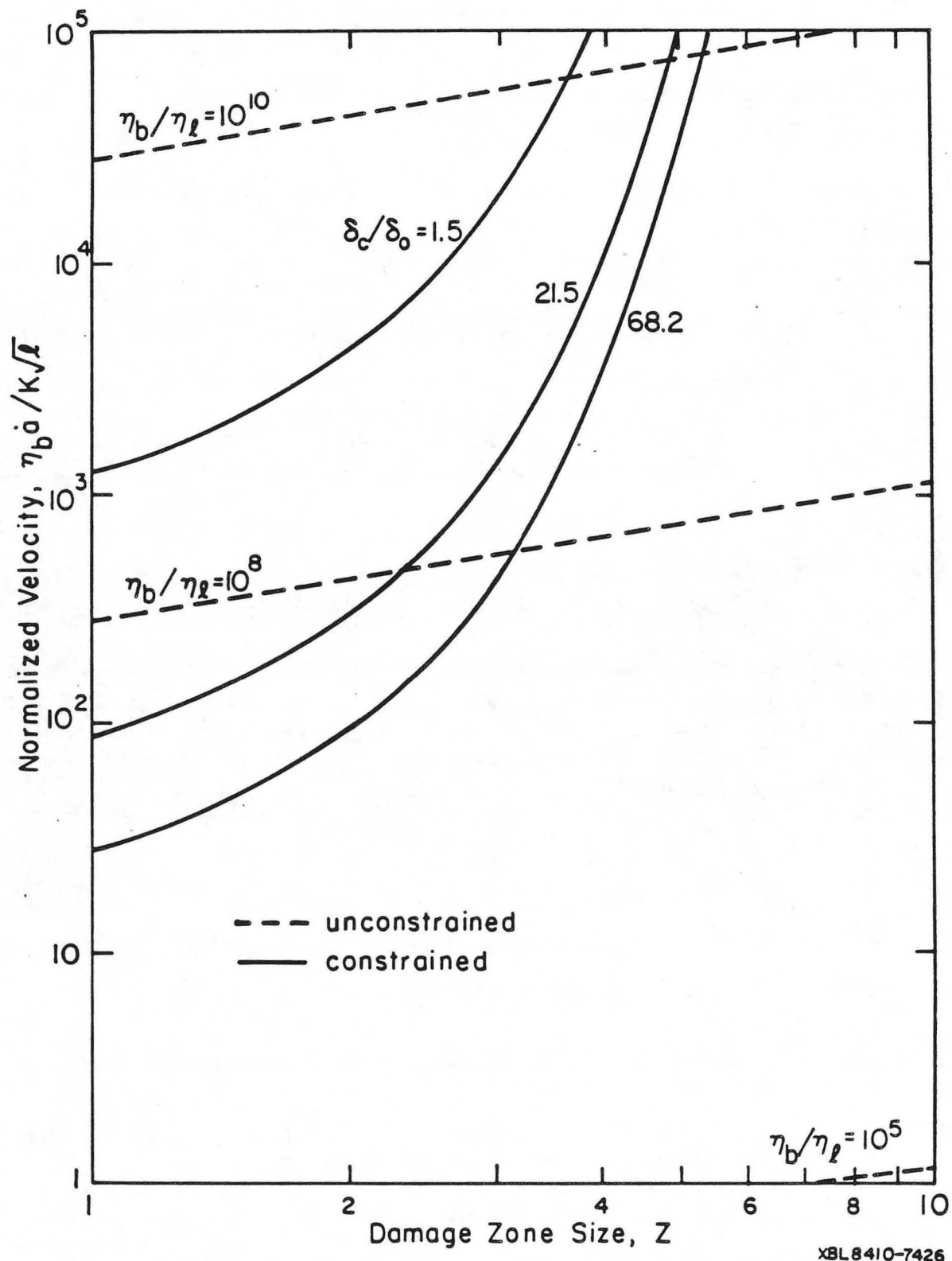
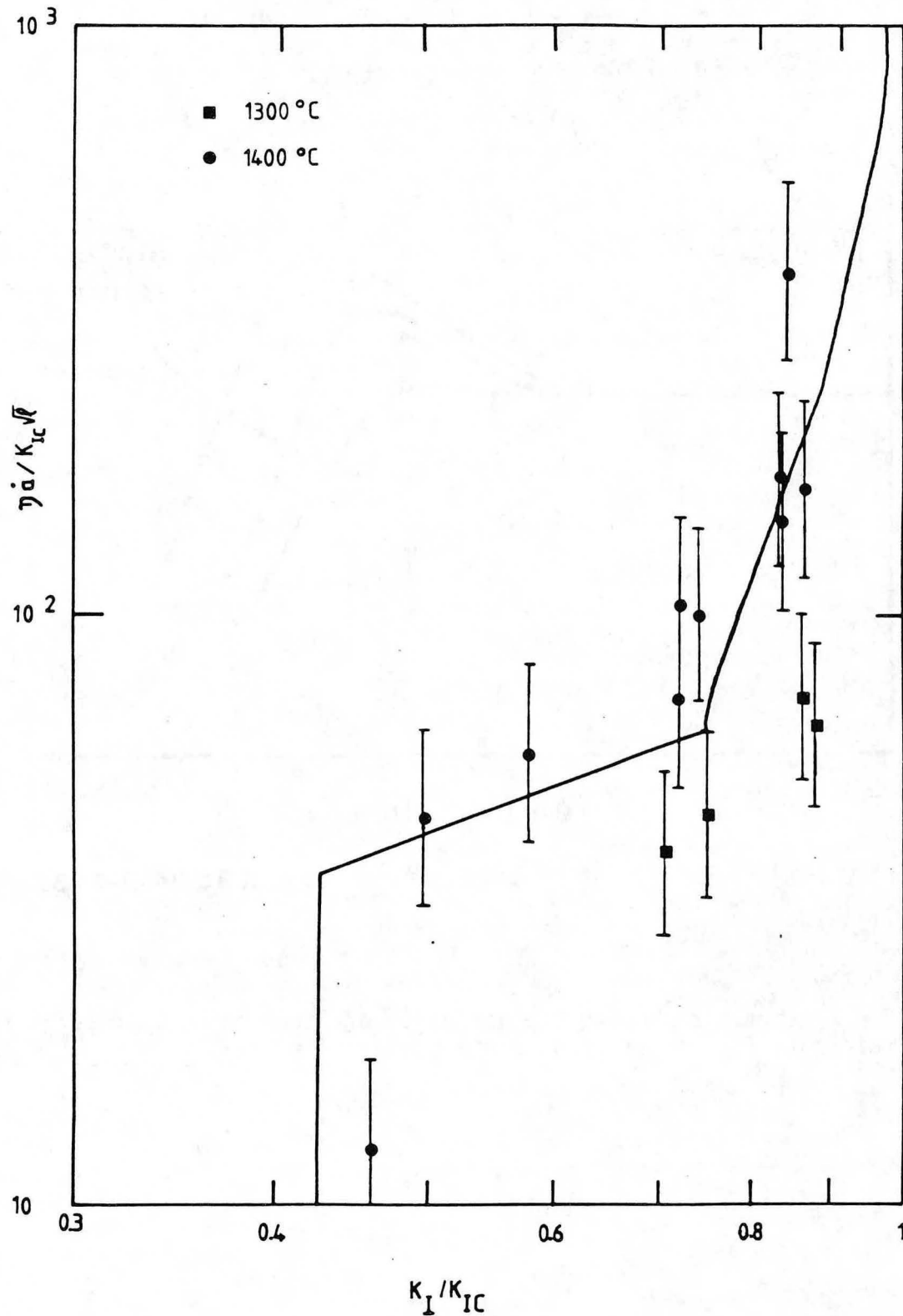
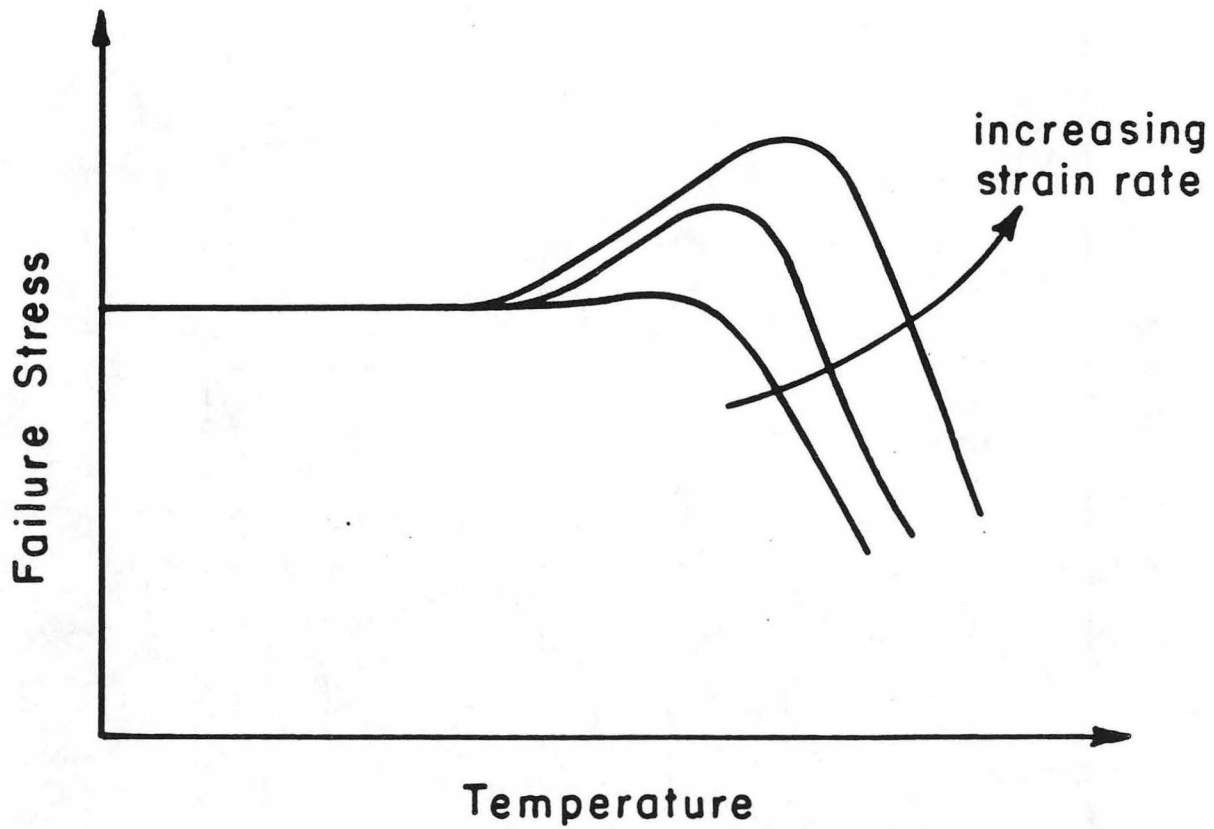


Fig. 46. Theoretical plots of the normalized velocity against the number of grains in the damage zone ( $z$ ) for a liquid phase sintered material.  $\ell / \delta_0 = 1000$ .



XBL 8410-4149

Fig. 47. A plot comparing the theory presented in this thesis with the experimental results of Blumenthal.<sup>[1]</sup> The theoretical curve includes the effect of a threshold and the critical stress intensity factors.



XBL 8410-7432

Fig. 48. A schematic showing the variation of strength with temperature and strain rate for liquid phase sintered ceramics.

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