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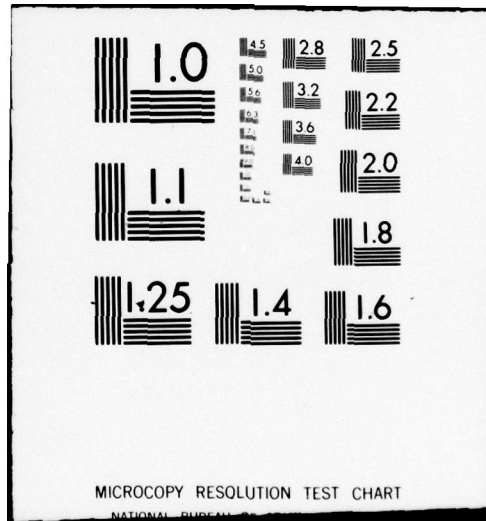
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9 Final Technical Report,
1 Oct 75 - 30 Sep 79

(period October 1, 1975 - September 30, 1979)

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Dr. A. H. Rosenstein

Air Force Office of Scientific Research

15 Grant No. AFOSR-76-2930

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6 FRACTURE AT ELEVATED TEMPERATURE IN
DISPERSION STRENGTHENED ALLOYS

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ABSTRACT

This report describes important new findings in the understanding of intergranular cavitation and fracture in alloys which derive their creep strength from the presence of hard second phase particles, such as the nickel base superalloys. The work is primarily theoretical but is supported in most instances, by simple experiments on model alloys. Criteria for the nucleation of cavities at high temperature are developed and it is shown that cavities are most likely to nucleate at particles in the grain boundaries. It is shown that the cavities can grow by diffusion, by power law creep or by a combination of the two. The validity of the diffusional mechanism has been confirmed through experiments with bicrystals. A semi-empirical equation for ductility when the cavities grow apparently by power law is obtained. The equation has wide applicability in commercial alloys, including the new nickel base alloys such as MERL 76. In this equation the ductility is related to the density of the particles in the grain boundary and to the strain rate sensitivity of the material. It has been deduced that the state stress can influence the relative importance of diffusional vs. power law creep mechanism of cavity growth. Some work on creep-fatigue interaction was initiated in the course of this program.

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BACKGROUND

The performance of a gas turbine engine depends heavily on the creep and fracture characteristics of the alloys from which it is constructed. Significant advances have been made in the development of highly creep resistant alloys by precipitation and solution strengthening techniques, e.g. the γ - γ' nickel base superalloys, but the creep fracture properties of these alloys are not adequately understood in terms of the microstructure. This study was begun with the intention of gaining an understanding of the fundamental mechanisms of intergranular cavitation from which these alloys suffer at and near the operating temperatures. It is the nucleation and growth of these cavities which is responsible for the low ductility of these materials at intermediate temperatures.

The design of the hot section of a gas turbine is based upon the stress rupture data which expresses the time to fracture as a function of the tensile load applied to the specimen. The temperature and the load are usually held constant during the experiment. Empirically, it is often observed that the time to fracture varies with stress in the same manner as the minimum or the secondary creep rate.⁽¹⁾ This has led to the philosophy among the alloy designers that decreasing the secondary creep rate is the way to extend stress-rupture life. This is a dangerous course to follow because the ductility of the material is likely to be as important as the rupture life at high temperatures. For example, the disk and the blade contain sites of stress concentrations e.g. notches, and the engine is subjected to a time dependent loading cycle during flight. Extrapolation of simple time to fracture data on smooth specimens under constant load to predict failure under much more complex

conditions is likely to be difficult if not impossible. Since any low ductility failure at high temperature whether in smooth specimens, at notches⁽²⁾, or by creep-fatigue, occurs by cavitation at grain boundaries, I feel that the results of the research program reported here are likely to be of a general importance.

The report follows the sequence in which the research was performed. Since almost all of the results have already been published in open literature, the discussion in the following sections will be kept brief. First a model for the nucleation of cavities at high temperatures will be described, followed by a model for fracture by the diffusional growth of cavities in the grain boundary. Experimental work in support of the diffusional growth will be presented. Thereafter the results of experiments on a model alloy in which the density of the second phase particles was controlled and varied will be reported. This study lead to the development of an empirical equation for the ductility which appears to be of quite general application. Strain rate has a strong influence on fracture at elevated temperature, there being at least three transitions in the mechanism of failure as the strain rate is increased. We have studied these transitions. We have proposed models for intergranular failure under fatigue conditions. These ideas and some experimental results are discussed toward the end of the report.

CAVITY NUCLEATION

A montage which summarizes the model which we have proposed and analyzed is shown in Fig. 1. It is assumed that cavities are produced under the influence of an applied tensile stress when vacancies form clusters which are of a critical size. As shown in the picture there are several sites where cavities may nucleate such as the grain matrix and various sites in the

grain boundaries including one where a second phase particle and the grain boundary form a triple point. Upon applying the concepts of the classical nucleation theory to this model it is found that the free energy barrier to nucleation can be described in terms of a critical radius, r_c , and a critical volume of the cavity-embryo $r_c^3 F_V$. The first depends only upon the normal stress:

$$r_c = \frac{2\gamma}{\sigma_n} \quad (1)$$

while the second depends on the site of nucleation. For example, it becomes quite clear from a visual inspection of the various type of nucleation sites in Fig. 1, that the site at the junction of the boundary and the particle has the smallest volume associated with it. The actual volume of the critical cavity will be determined by the three angles α , β and μ which depend upon the interfacial tensions γ , γ_I , γ_{IB} , and γ_B in the following manner:

$$\begin{aligned} \cos \alpha &= \gamma_B / 2\gamma \\ \cos \beta &= (\gamma_{IB} - \gamma_I) / \gamma \\ \cos \mu &= \gamma_B / 2\gamma_{IB} \end{aligned} \quad (2)$$

Here γ , γ_I , γ_B and γ_{IB} are the interface energies for the free surface of the matrix, free surface of the particle, the matrix-matrix interface and the particle-matrix interface, respectively. The function F_V is described by the equation:

$$\begin{aligned} F_V &= \frac{4\pi}{3} (2 - 3 \cos \theta + \cos^3 \theta) \\ \text{where, } \theta &= (\alpha + \beta - \mu) / 2 \end{aligned} \quad (3)$$

for the case of the void shown on the upper-right in Fig. 1. Note that increasing γ_B and γ_{IB} while decreasing γ and γ_I lowers the barrier for nucleation. The steady state rate of nucleation is then given by: (*1)[†]

$$\dot{I} = \frac{4\pi\gamma}{\sigma_n \Omega} \frac{\delta D_B}{\Omega^{1/3}} \rho_{\max} e^{\frac{-4\gamma^3 F_V}{\sigma_n^2 kT}} \quad (4)$$

[†] When an asterick preceeds the reference, then that reference is a publication due to this research. These references are listed separately on p. 21 .

where Ω is the atomic volume, $\rho_{\max}$ is the maximum number of nucleation sites, and δD_B the boundary thickness times the grain boundary self diffusion coefficient. The implication of Eq. (4) is that the nucleation rate changes exponentially with the applied stress which should lead to a threshold type of nucleation behavior as shown schematically in Fig. 1.

Even if the applied stress were greater than the threshold stress, it would be necessary for the vacancies to diffuse and gradually form a cluster of critical size. This would require time, and it leads to a condition for an incubation time for nucleation. This time can be estimated exactly only by extensive numerical calculations, but the lower bound for this time is simple to calculate and is given as follows: (*1)

$$t_i = \frac{r_c^3 F_V}{4\delta D_B} \quad (5)$$

As expected, the incubation time depends upon the number of vacancies required to collect a critical volume ($r_c^3 F_V$), and upon the rate at which the vacancies diffuse in the grain boundary (δD_B).

At present quantitative data to which our model can be compared is scarce but there is considerable metallographic and TEM evidence that the junction of the particle and the grain boundary is indeed the site where cavities are most frequently observed. (3,4,5)

According to this model, a cavity should form at each particle in the grain boundary which is aligned normal to the applied stress provided that the interface energies are the same for all particles. The density and the spacing of the cavities is therefore, likely to be the same as the spacing of the particles in the grain boundary. This is a simple but an important result since the growth of the cavities, by any mechanism, is likely to be very sensitive to the average spacing of the cavities. The ductility and the time-to-fracture, as we shall see later, are strong functions of the cavity spacing.

CAVITY GROWTH: CONTROLLED BY BOUNDARY DIFFUSION

If a tensile stress is applied normal to the plane of a grain boundary which contains an array of cavities, as shown in Fig. 2b, then the cavities will grow by the diffusion transport of atoms from the surface of the cavity to the grain boundary region adjacent to the cavity. If it is assumed that the cavities grow in a self-similar shape then there is a unique relationship between the rate of increase in the cross sectional area of the boundary which is occupied by the cavities and the rate of transport of matter. The latter can be calculated by resorting to the diffusion equations, equation of mechanical equilibrium and an equation which relates the excess chemical potential of the atoms in the interface across which a tensile stress has been applied relative to a stress free interface. The rate of increase of the volume of each of the cavities is then found to be given by:

$$\frac{dV}{dt} = 2\pi\Omega \frac{\delta D_B}{kT} \frac{(\sigma_\infty - P - 2\gamma) \left(1 - \frac{4r_b^2}{\lambda^2}\right)}{\ln \frac{\lambda}{2r_b} - \frac{3}{4} + \frac{4r_b^2}{\lambda^2} \left(\frac{1-r_b^2}{4\lambda^2}\right)} \quad (6)$$

where σ_∞ is the applied stress, P is the internal gas pressure in the cavities (normally equal to zero), λ is the average spacing between the cavities, and r_b is the radius of the circle projected by the cavities in the boundary plane. The volume of the cavity is related uniquely to r_b , and defining a damage parameter: $A = 4r_b^2/\lambda^2$, which is a measure of the reduction in the load bearing section of the boundary, Eq. (6) can be written in terms of dA/dt . The latter can be integrated to yield an equation for time to fracture: (*2)

$$t_f = 0.006 \frac{kT}{(\sigma_\infty + P)\Omega} \frac{\lambda^3}{\delta D_B} \quad (7)$$

It is interesting that according to Eq. (7) fracture is dependent on stress and temperature, and that the time-to-fracture is inversely proportional to the first power of stress. The activation energy in the rate equation for fracture is equal to the activation energy for grain boundary self diffusion. Note also the high sensitivity of the rupture life to the cavity spacing, λ .

In spite of the simplicity of Eq. (7) there was no direct experimental data in support of this mechanism. We reasoned that since the model strictly applies to a flat planar boundary extending across the entire cross section of the specimen (see Fig. 2), the suitable experiment will be to test bicrystal specimens. This was done and good agreement between theory and experiment was obtained, for the first time, for this mechanism as shown in summary form in Fig. 2. Note in Fig. 2 that the applicability of Eq. (7) is limited to a certain range of temperature (and stress^(*3)); beyond it a non-linear mechanism of fracture (in stress) is manifested which may be an indication of power law creep rather than a diffusional mechanism of cavity growth.

EXPERIMENTS WITH POLYCRYSTALS HAVING CONTROLLED PARTICLE DENSITIES

Since the theory predicts the particle spacing in the grain boundaries to be an important parameter in the creep-rupture behavior of polycrystalline materials, experiments were carried out on specimens in which the particle densities were carefully controlled and systematically varied. Copper, dispersion strengthened with particles of fused silica produced by the internal oxidation technique, was chosen for this purpose. The results are reported in detail in Metallurgical Transaction.^(*4) The important conclusions are summarized here.

Modes of Fracture

It was found that there were three principal modes of fracture: a low strain rate, an intermediate strain rate and a high strain rate mode.

Optical micrographs are shown in Fig. 3. At low strain rates cavities form the entire cross section of the specimen and appeared to form preferentially in those boundaries which were aligned normal to the tensile axis. This is the so called 'r' type of cavitation. We did not find any clear evidence that grain boundary sliding had been instrumental in the growth of 'r' type cavities. At intermediate strain rates there was a transition to a more shear type of failure where intergranular cracks initiated at the surface of the specimens and failure occurred when their number and their size exceeded some limit. In this instance grain boundary sliding (and perhaps environment since the argon may have contained some oxygen as an impurity) obviously played an important role in the initiation of cavities and cracks. At high strain rates the specimens failed not by cavitation but by the growth of a neck in the specimen cross-section. In this instance copious recrystallization was observed in the neck region which was taken as evidence of dynamic recrystallization. We feel that recrystallization relieved stress concentration at triple grain junctions and hence suppressed wedge type of intergranular cracking.

The 'r' type of cavitation was studied in detail. For all particle densities it was found that the stress dependence for the time to fracture followed a power law rather than a linear law as would have been expected if the growth of cavities were diffusion controlled (Eq. 7). In fact we found that, invariably, the power law stress exponent for time to fracture was always the same as the stress exponent for steady state creep rate. We carried out interrupted tests to determine at which stage the cavities were fully nucleated and found that nucleation was essentially complete before the onset of steady state creep. We concluded, therefore, that the similarity of the stress exponent in the rate equation for fracture and in the rate equation for secondary creep, implied that the cavities were growing by the power law creep rather than by the diffusional transport mechanism.

If the cavities were indeed growing by matrix creep, then one would expect the rate of growth of the cavities to be affected by the spacing between the cavities and by the strain rate sensitivity of the material. The data were analyzed in terms of these two parameters and the following equation was found to be an excellent representation of the data: (*4)

$$\epsilon_f = 0.23 \frac{\lambda}{d} e^{\frac{4}{n-1}} \quad (8)$$

Here ϵ_f is the strain to fracture, λ is the cavity spacing, d is the grain size and n is the power law stress exponent in the rate equation of steady state creep. The equation has a rigorous theoretical lower bound for $n \rightarrow \infty$. The creep rupture data for many materials was found to be in good agreement with the above equation. In particular the agreement between it and stress rupture data on new powder metallurgy nickel base alloys^{(6)†} is shown in Fig. 4.

In summary, the basic result of these studies was that the growth and linkage of an array of cavities in the grain boundaries of a polycrystal is strain controlled. The strain is localized in a band of material surrounding the grain boundary which contains the cavities.

A MODEL FOR SLIDING INDUCED CRACKING OR 'WEDGE CRACKING'

We learnt from our experiments on polycrystals that strain rate had a considerable influence on the mechanism of fracture. In particular sliding appeared to play an important role only at the higher strain rates, when wedge shaped cracks appear to be forming. The model which we proposed for wedge cracking was similar to the model for 'r' type cracking except that the localized displacement for the growth and coalescence was provided not by the average strain in the specimen but instead by the localized displacement due

[†] These are the results obtained by Pratt & Whitney Aircraft Company under the sponsorship of an AFOSR program with M.J. Blackburn and C.C. Law.

to grain boundary sliding as shown in Fig. 5. This idea then lead to the prediction that if the strain rates applied to the specimen were so fast that the boundaries did not have enough time to slide then wedge cracking would not occur. This critical strain rate, above which wedge cracking would not be possible under any circumstances would then be a true upper bound strain rate for wedge cracking. This strain rate depends upon the resistance of the grain boundary to sliding and is given by: (*5)

$$\dot{\epsilon}_u = \frac{\sigma_y}{\eta d} \quad (9)$$

where η is the sliding resistance defined as:

$$\eta = \frac{\sigma_s}{\dot{U}} \quad (10)$$

where d is the grain size, $\dot{U}$ is the rate of sliding, and σ_s is the applied shear stress (assumed to be equal to the yield stress in shear in Eq. 9). The sliding resistance would depend upon the microstructure of the grain boundary. If the boundary contains particles then an appropriate equation for η would be: (*5)

$$\eta_p = \frac{kT}{8\Omega} \frac{f_b p^2}{\delta D_B} \quad (11)$$

where p is size of the particles and f_b is the area fraction of the particles in the grain boundary. In case the boundary is free of particles then the sliding rate would depend upon the ledge structure of the boundary and the following equation would be more appropriate: (*5)

$$\eta_l = \frac{kT}{8\Omega} \frac{h^2}{\delta D_B} \quad (12)$$

where h is the height of the ledges in the grain boundary. It is expected that h would be of atomic dimensions. If Eqs. (11) and (12) cannot be used for the lack of microstructural or diffusion data, then the sliding resistance can be measured directly by internal friction damping experiments such as those used by Ke^(7,8) and Mosher and Raj⁽⁹⁾.

According to the above model wedge cracking would be possible if the strain rate is less than the upper bound strain rate given by Eq. 9. However, when the strain rates are very slow then there is a transition from wedge cracking to 'r' type of cavitation behavior. We believe that this transition is related to the relaxation of the stress concentration produced by sliding at the triple junctions by diffusion and other creep mechanisms. We do not as yet have a quantitative model to describe this second transition although we expect that it would be sensitive to the grain size, and to the ratio of the relative resistance of the grain boundary and the grain matrix to shear deformation.

STRAIN RATE EFFECTS IN FRACTURE AT HIGH TEMPERATURE

From the modeling work which has been described in the previous section it was predicted that intergranular failure by wedge cracking is likely to be strain rate dependent. Experiments, therefore, were carried out on polycrystalline copper to study the transition from transgranular ductile fracture at high strain rates to wedge cracking fracture at intermediate strain rates. A further decrease in the strain rate, it was anticipated, would lead to a transition from wedge cracking to 'r' type intergranular fracture. Experiments were carried out in simple tension, as well as biaxial tension in order to study the influence of the stress state on fracture. In the material which we studied the uniaxial and the multiaxial tests results were comparable, but this is by no means a general result; in other materials e.g. nickel base superalloys quite a different result may have been obtained.

This investigation has lead to two significant advances. First we have shown that the mode of fracture changes in a systematic manner as the strain rate is changed, and second, we have developed a technique which can be used to study fracture at elevated temperatures under the application of biaxial stress.

The results from the tensile tests are shown in Fig. 6^(*6). At low temperatures and high strain rates the fracture mode is ductile transgranular, at intermediate strain rates and intermediate temperatures fracture mode is intergranular-wedge type, at low strain rates fracture is intergranular 'r' type, and at high strain rates and high temperatures the ductility is essentially one hundred percent because of the incidence of dynamic recrystallization. As we shall see later, it is important to distinguish between fracture by wedge and 'r' cavitation because the models which will be proposed to explain the influence of cycle shape on fatigue life in the regime of creep-fatigue-interaction will depend upon which type of cavities are present.

Mechanical testing under biaxial loading was carried out by driving a punch with a hemispherical end into the specimen which was in the shape of a sheet and which had been secured along its circular edge. The strain rates were controlled, approximately, by changing the rate of insertion of the punch into the sheet. The strain to fracture was measured by measuring the distortion of the small circles which had been etched into the surface of the sheet specimens by optical lithography. Those circles which were close to the final rupture were selected for measurement of the fracture strain. The scale and the geometry of the circles is shown in Fig. 7^(*6). The deformation of the circles for a ductile mode of fracture at room temperature is shown in Fig. 8a while the case for wedge cracking failure at high temperature is shown in Fig. 8b. The geometrical model which was developed for correlating the punching speed to the strain rate is given in the Appendix.^(*6) This leads to the experimental points drawn as bars in Fig. 6. There was agreement between the tensile and the biaxial tests.

CAVITATION AT PARTICLES DURING VERY HIGH STRAIN RATES

In high temperature forming operations such as hot rolling of sheet, and superplastic forming of alloys, the strain rates can be large, ranging

from 10^{-3} to $1.0s^{-1}$ while the temperatures are near half the melting point. At these high strain rates cavities can initiate at hard second phase particles, which if allowed to grow will limit ductility and hence the formability of the materials. We have developed a model by which we have been able to calculate the influence of strain rate on the initiation of the cavities. The basis of the model is that if the applied strain rate is slow enough the stress concentration at particles will be relaxed at the same rate or faster than it is produced because of the concentration of flow near the particles. The model leads to an expression for a critical strain rate below which the cavities will not form. This calculation has the potential for application in forming operations because it defines an upper-bound in strain rate below which the ductility of the material should not suffer from the presence of second phase particles. The expression for the critical strain rate is as follows: (*7)

$$\dot{\epsilon}_{crit} = \frac{118 (1-\nu)(1-2\nu+\frac{2}{3})}{(\frac{5}{6} - \nu)^2} \cdot \frac{G\nu}{kT} \cdot f_v \frac{\delta D_b}{p} \quad (13)$$

the idea being that in order to avoid fracture the applied strain rate should be less than the above value. Here p is the size of the particles, f_v is the volume fraction of the second phase particles, G & ν are the elastic constants of the matrix, and δD_b is the boundary thickness times the diffusion coefficient along the matrix-particle interface. Note that the fracture mechanism considered here to cavitation at all particles, not just the particles in the grain boundaries.

The correlation between this model and results on aluminum⁽¹⁰⁾ from published literature are shown in Fig. 9.

HIGH TEMPERATURE FATIGUE: MODELS AND EXPERIMENTS

We have proposed two basic models by which intergranular cavitation can affect failure under cyclic loading. (*8) These are shown schematically in

Fig. 10. In the first model shown in Fig. 10a 'r' type cavities form in the grain boundaries. When a fatigue crack grows into a material in which such cavities have been initiated then, provided the criterion for the growth of cavities is satisfied within the constraints of the strains and stresses in the region of the crack tip, the crack growth rate will be accelerated and the fatigue life would decrease. Since there are several mechanisms by which cavities can grow, the model warrants a much more detailed study of the interactions between fatigue cracks and cavities. But in order to establish the basic validity of this model in general, we carried out the following experiment. Fatigue crack growth experiments were carried out in austenitic stainless steel (stainless steel was chosen for these exploratory experiments because of the need for a material which was relatively insensitive to environmental effects and a material which could be tested for crack propagation in fully yielded condition as this minimizes the problems of reproducibility in mechanical testing^(*9).) Crack growth was measured at a constant plastic strain amplitude but under a variety of loading cycles. In the first instance the crack growth rate was measured for continuous cycling with a triangular wave shape. Crack growth was transgranular and the curve represented by open data points in Fig. 11 was obtained.^(*10, 11) Then, through theoretical reasoning,^(*12) a long hold cycle was imposed on the specimen before starting the crack growth measurement as this was expected to introduce cavities of the 'r' type in the grain boundaries. When this hold cycle was followed by crack measurements using a continuous cycle, then beyond a certain crack length, a transition to faster crack growth rate, which was found to be mixed intergranular and transgranular, was observed. When a long compression hold was imposed on the specimen in order to remove the cavities by sintering,^(*10) then the crack growth rate reverted to the slower rate and the mode of fracture again became transgranular. This result clearly indicates the role of cavities on fatigue crack propagation. Much more work is needed to develop the

model for application to other materials, and a detailed understanding of the criterion for the interaction between the crack and the cavities is needed. Since the cavities can grow by power law creep and by diffusion, and since the stress and strain field near the crack tip is multiaxial, the interaction will be quite complex.

The second model for creep fatigue interaction is shown schematically in Fig. 10b. The supposition is that during cyclic loading wedge cracks initiate and grow. When their number exceeds a critical value they begin to coalesce and soon thereafter failure ensues. Our present thinking is that wedge cracking is more likely to lead to the accumulation of damage in the specimen in general rather than to local damage near a crack tip. The reason being that one wedge crack is of the size of approximately a grain facet. In order for several of these cracks to accumulate the plastic zone size near a crack tip will have to be several times the grain size which is unlikely unless the grain size is very small (a few microns). However, it is possible that wedge cracking will have some influence on the rate of fatigue crack propagation. In order to study this experimentally the following experiment was performed:

We recall from the discussion in the preceeding section that wedge cracking is likely to be sensitive to the strain rate. In particular, the concept of an upper bound in strain rate was introduced which separates the region of wedge cracking (slow strain rates) from the region where wedge cracking would not occur (fast strain rates). Crack growth experiments were performed for sawtooth shaped, unsymmetrical strain rate cycles. The cycles were of two types: slow/fast and fast/slow i.e. in the slow/fast cycle for example, the tension going strain rate was slower than the compression going strain rate. The reasoning was that since wedge cracks are more likely to form in tension the slow/fast cycle should produce more damage

than the fast/slow cycle. There was one more aspect to the experiments. It was argued that if the temperature is increased for the same slow/fast cycle then a maximum damage should occur when the slow strain rate is below the upper bound but the fast strain rate is above it. At the lower temperatures both strain rates will be above the upper bound so there would be no sliding and hence no wedge cracking damage would be produced, and at the higher temperatures sliding would occur in tension and in compression so that the damage produced is likely to be at least partly restored during compression; therefore, the rate of accumulation of wedge cracking damage would be less than at the intermediate temperatures. As shown in Fig. 12^(*13) our results do bear out the prediction of our simple model. More significantly the location of the upper bound strain rate as calculated from Eqs. 9 and 11 is pretty much in agreement with the position of peak in Fig. 12.

HIGH LIGHTS

I will summarize here those achievements which are likely to have a long range impact in the area of high temperature intergranular fracture.

- (1) The kinetic model for the nucleation of cavities is an important contribution. It explains rigorously and in a self-consistent manner for the first time, why cavities nucleate at second phase particles in the grain boundaries. It explains the role of interface energies and the grain boundary diffusion coefficient on nucleation, and it provides a time-temperature condition for nucleation.
- (2) For the first time clear experimental evidence has been presented that fracture can occur by diffusional growth of cavities in the grain boundaries. These tests were done in bicrystals, and reasons were put forward as to why, in a uniaxial test, diffusional growth of cavities

is not found to be the rate controlling factor in fracture of polycrystals. It was argued that the diffusional mechanism could operate in polycrystal if a triaxial stress is applied to it. This deduction may be of critical importance when considering the growth of cavities in regions of constrained deformation such as crack tips and notch-roots.

- (3) The expression which provides a semi-empirical description of the ductility in polycrystals (when tested in uniform uniaxial stress) in terms of the grain size, the density of the second phase particles in the grain boundary, and the strain rate sensitivity of the material (see Eq. 8) will, I believe, continue to prove to be useful.
- (4) The concept of differentiating between the 'r' type and the wedge type of cavities on the basis of grain boundary sliding is new and is still not appreciated in the literature. We have proposed that both types of cracking are similar in the sense that both require the growth of cavities which nucleate at second phase particles. But the difference is that in the case of wedge cracks the displacement required for cavity growth is driven (and accommodated) by grain boundary sliding whereas sliding plays no obvious role in the growth of 'r' cavities (Fig. 5). The concept of an upper bound strain rate for wedge cracking, will, I believe be useful in studies of tensile testing and in studies of high temperature fatigue.

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Figures

1. A schematic illustration of the kinetic model for nucleation of cavities proposed by the author. The possible nucleation sites are shown on the top-left. The most likely site shown on the top-right is the junction of the grain boundary and the second phase particle. The equation for steady state rate of nucleation predicts a threshold type of nucleation behavior as shown on the bottom right.
2. Theory and experiment for the growth of cavities in the grain boundary by the diffusional mechanism. According to the model the cavities grow by transport of atoms from the cavity surface to the adjacent grain boundary regions. An equation for time to fracture containing no adjustable parameters is obtained; and it agrees well with the experimental data from tests on bicrystals.
3. Various modes of fracture in polycrystals. At low strain rates fracture occurs by 'r' type of cavities which form preferentially at the grain boundaries aligned normal to the tensile axis. As the strain rate is increased there is a transition to wedge type of intergranular cracking while at the very high strain rates dynamic recrystallization in the neck region leads to nearly one hundred percent ductility.
4. A comparison of theory and experiment for stress rupture data from nickel base superalloys prepared by powder metallurgy.
5. The similarities and the differences in the mechanisms of 'r' type and 'wedge' type of intergranular cracking. In both, separation occurs by the growth of cavities which form at the second phase particles. However, in 'r' cavitation the growth of the cavities is accommodated by creep in the matrix in general, while in 'wedge' cracks the displacement for cavity growth is provided by grain boundary sliding.
6. Change in the mode of fracture in tensile tests on copper in which the strain rate was controlled and varied. The experimental points with the error bars were obtained from biaxial instead of uniaxial testing.
7. The geometry and the scale of the circles which were etched on to the surface of sheet specimens for biaxial testing. Optical lithography was used.
8. The appearance of the fracture surface after fracture in a sheet specimen at room temperature (a) and at high temperature (b). Note the difference in the ductility.

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Personnel

B. K. Min	Post-doctoral associate
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F. Abbo	Graduate student (M.S.)
S. Parchuri	Graduate student (Ph.D.)
W. Pavinich	Graduate student (M.S.)

Coupling

Visits of much mutual benefit have been exchanged with Dr. C.C. Law and Dr. M.J. Blackburn of Pratt & Whitney Aircraft, VT., Middletown, CT., Professor R. Pelloux of MIT and J. Nicholas of AFML-WPAFB. The interaction with Professor Julia Weertman of North Western University has been useful.

Publications

- *1. "Nucleation of Cavities at Second Phase Particles in Grain Boundaries," R. Raj, Acta Metallurgica, 1978, Vol. 26, pp. 995-1006.
- *2. "Correction to: Intergranular Fracture at Elevated Temperature," R. Raj, H. M. Shih and H. H. Johnson, Scripta Metallurgica, 1977, Vol. 11, pp. 839-842.
- *3. "Intergranular Fracture in Bicrystals," R. Raj, Acta Metallurgica, 1978, Vol. 26, pp. 341-349.
- *4. "Fracture at Elevated Temperature," W. Pavinich and R. Raj, Metallurgical Transactions A, 1977, Vol. 8, pp. 1917-1933.
- *5. "The Importance of Wedge Cracking in Creep-Fatigue," B. K. Min and R. Raj, Canadian Metallurgical Quarterly, 1979, Vol. 18, pp. 171-176.
- *6. F. Abbo, "The Effect of Strain Rate and Biaxial Loading on High Temperature Fracture," M.S. Thesis, Cornell University, 1979.

- *7. "Diffusional Relaxation of Stress Concentration at Second Phase Particles," R.C. Koeller and R. Raj, Acta Metallurgica, 1978, Vol. 26, pp. 1551-1558.
- *8. "Mechanisms of Intergranular Fracture in High Temperature Fatigue", Cornell Materials Science Center Report #2873, July 1977. Prepared at the request of NMAB, National Academy of Sciences.
- *9. "A New Fixture for Fatigue Testing," B. K. Min and R. Raj, Journal of Testing and Evaluation, 1979, March issue, in press.
- *10. "Hold Time Effects in High Temperature Fatigue," B. K. Min and R. Raj, Acta Metallurgica, 1978, Vol. 26, pp. 1007-1022.
- *11. "The Effect of Cycle Shape on Creep Fatigue Interaction in Austenitic Stainless Steels," R. Raj and B.K. Min, Mechanical Engineering, 1978, Vol. 100, November, p. 122. Paper No.: 78-PVP-89, presented at the ASME/CSME Pressure Vessel and Piping Conference.
- *12. "Time Dependent Effects in Creep Fatigue," R. Raj, ASME MPC-3 Symposium on Creep Fatigue Interactions, New York, Dec. 1976, pp. 337-346.
- *13. "A Mechanism of Intergranular Fracture During High Temperature Fatigue," B. K. Min and R. Raj, Fatigue Mechanisms, ASTM-STP, 1979, in press.

Theses

M.S.

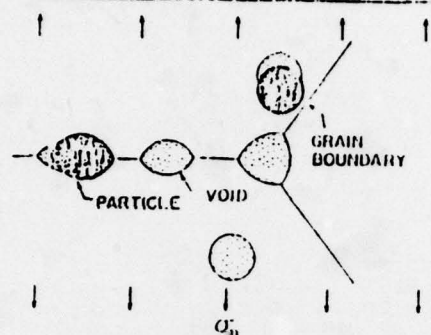
- W. Pavinich, "Fracture at Elevated Temperature," Cornell University, May 1977.
- F. Abbo, "Strain Rate Dependence of Intergranular Fracture," August 1979.

Ph.D. (in progress)

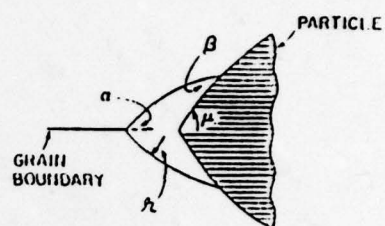
- S. Baik, "High Temperature Fatigue".
- S. Parchuri, "Effect of Environmental and Alloy Chemistry on High Temperature Fracture".

MODEL FOR MICROVOID FORMATION DURING CREEP-FATIGUE DEFORMATION

POSSIBLE SITES FOR VOID FORMATION



GEOMETRY OF VOID FORMATION (CONTROLLED BY SURFACE ENERGIES)



ρ : RADIUS OF CURVATURE OF VOID SURFACE
 α, β, μ : ANGLES BETWEEN BOUNDARY, VOID, AND PARTICLE

KINETICS OF VOID FORMATION

$$\text{NUCLEATION RATE} = \frac{4\pi\gamma}{\sigma_n \Omega} \frac{\delta D_0}{\Omega^{1/3}} \rho_{\max} / e^{\frac{4\gamma^3 f_v}{\sigma_n^2 kT}}$$

FRACTURE DURING CREEP AND FATIGUE IS DETERMINED BY THE NUCLEATION AND GROWTH OF VOIDS.

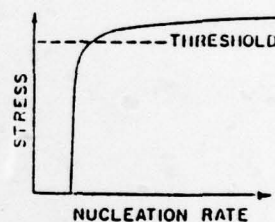


Fig. 1. A schematic illustration of the kinetic model for nucleation of cavities proposed by the author. The possible nucleation sites are shown on the top-left. The most likely site shown on the top-right is the junction of the grain boundary and the second phase particle. The equation for steady state rate of nucleation predicts a threshold type of nucleation behavior as shown on the bottom right.

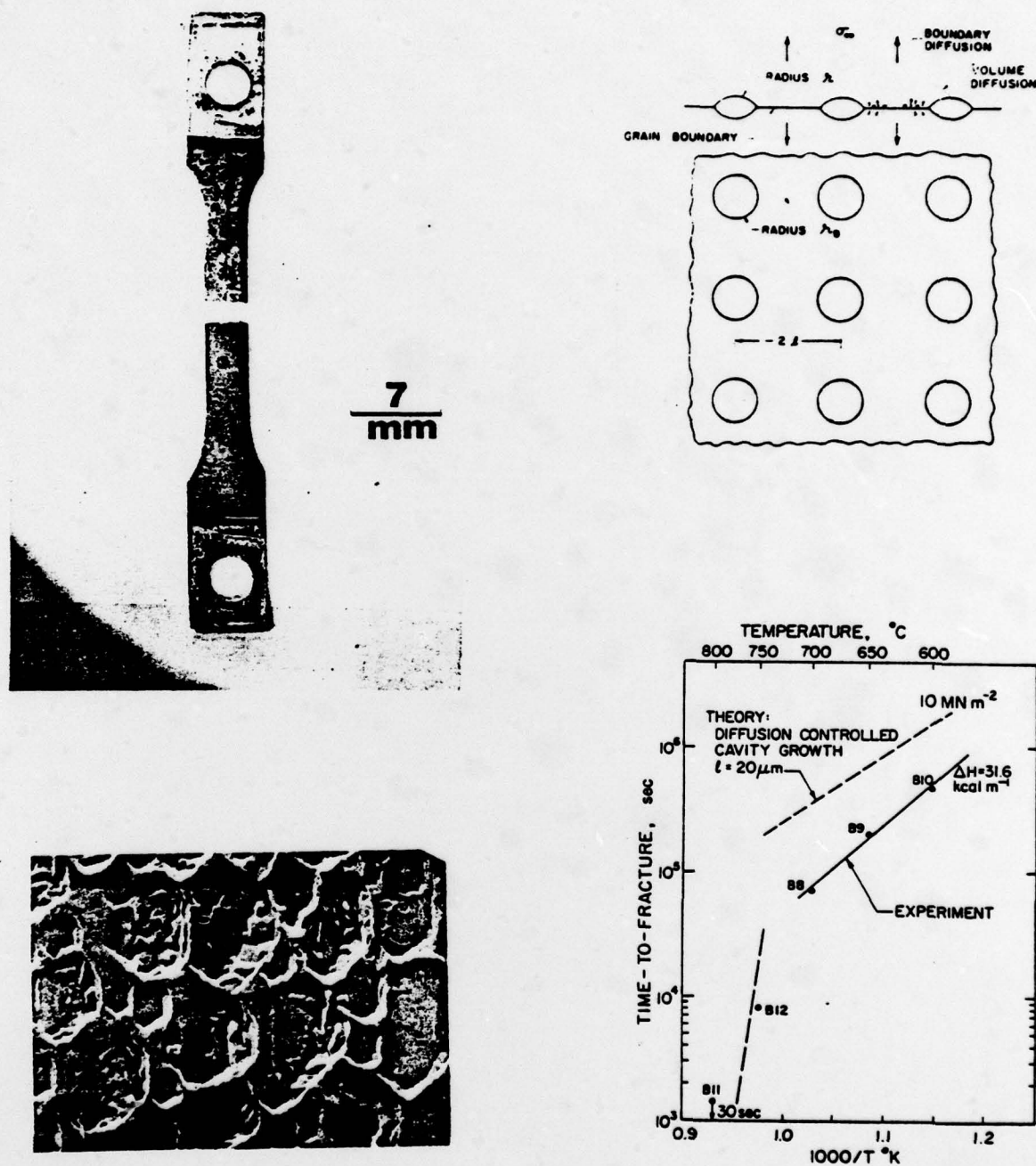
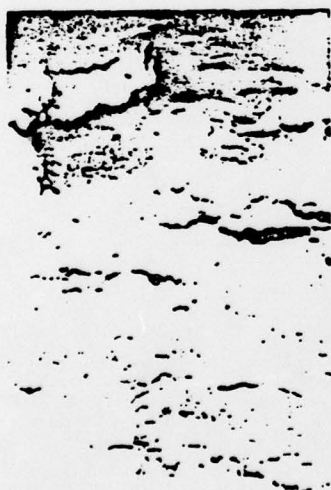
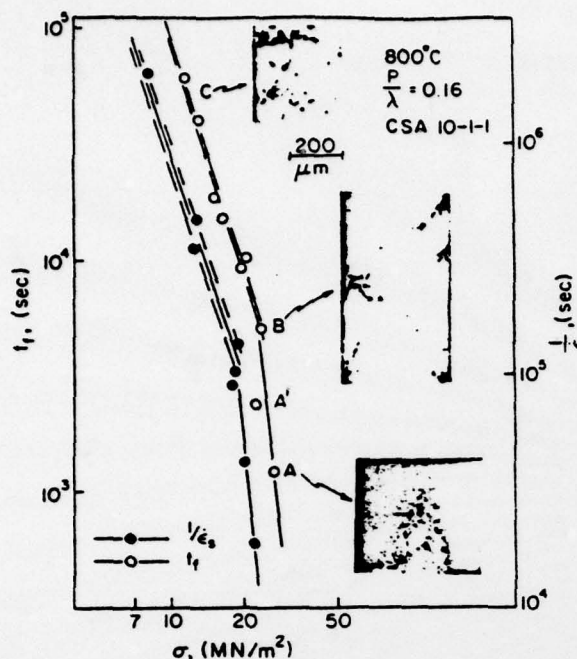
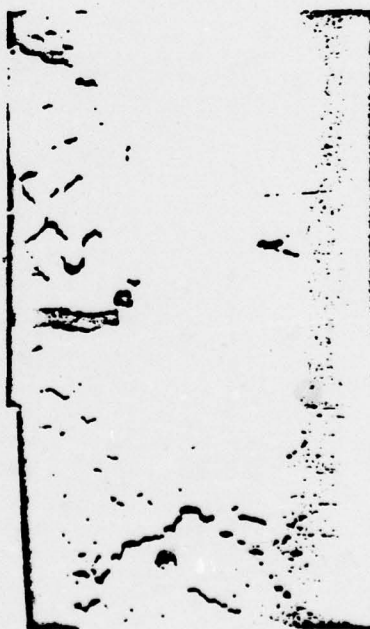


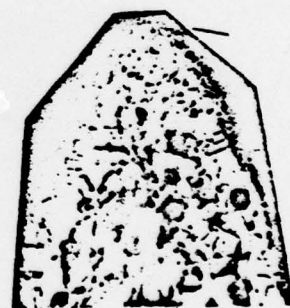
Fig. 2. Theory and experiment for the growth of cavities in the grain boundary by the diffusional mechanism. According to the model the cavities grow by transport of atoms from the cavity surface to the adjacent grain boundary regions. An equation for time to fracture containing no adjustable parameters is obtained; and it agrees well with the experimental data from tests on bi-crystals.



(a)



(b)



(c)

Fig. 3. Various modes of fracture in polycrystals. At low strain rates fracture occurs by 'r' type of cavities which form preferentially at the grain boundaries aligned normal to the tensile axis. As the strain rate is increased there is a transition to wedge type of intergranular cracking while at the very high strain rates dynamic recrystallization in the neck region leads to nearly one hundred percent ductility.

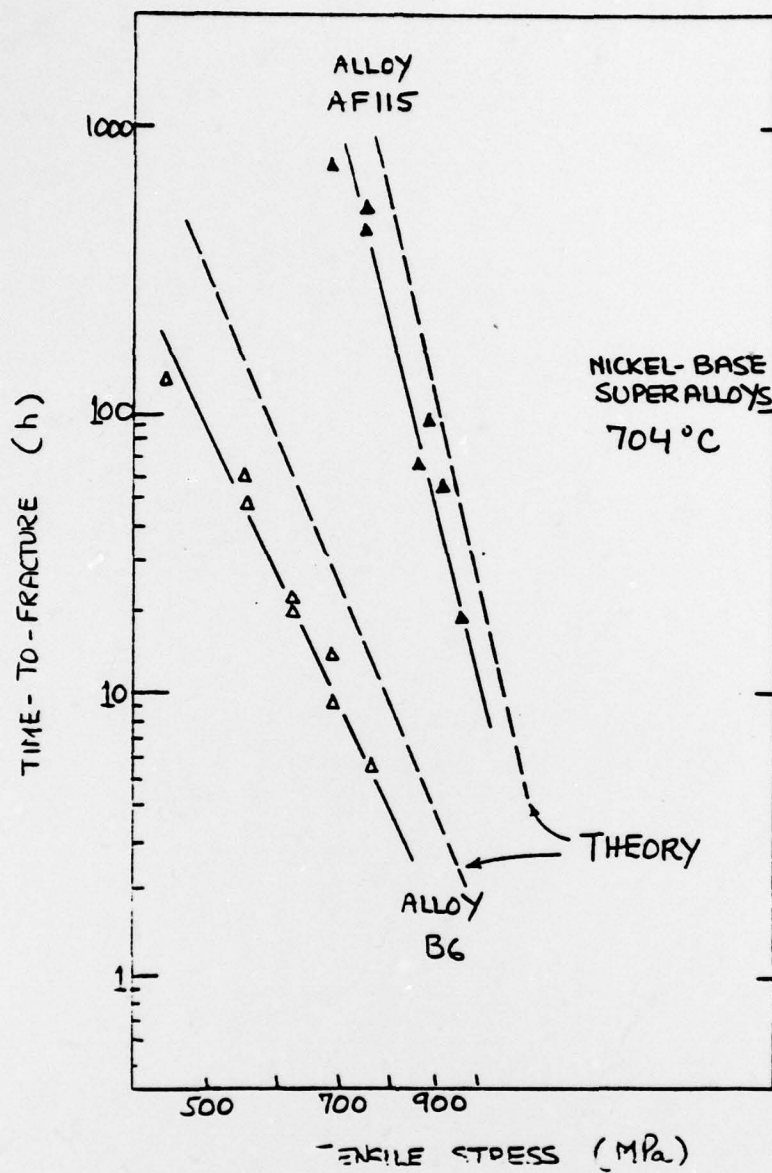


Fig. 4. A comparison of theory and experiment for stress rupture data from nickel base superalloys prepared by powder metallurgy.

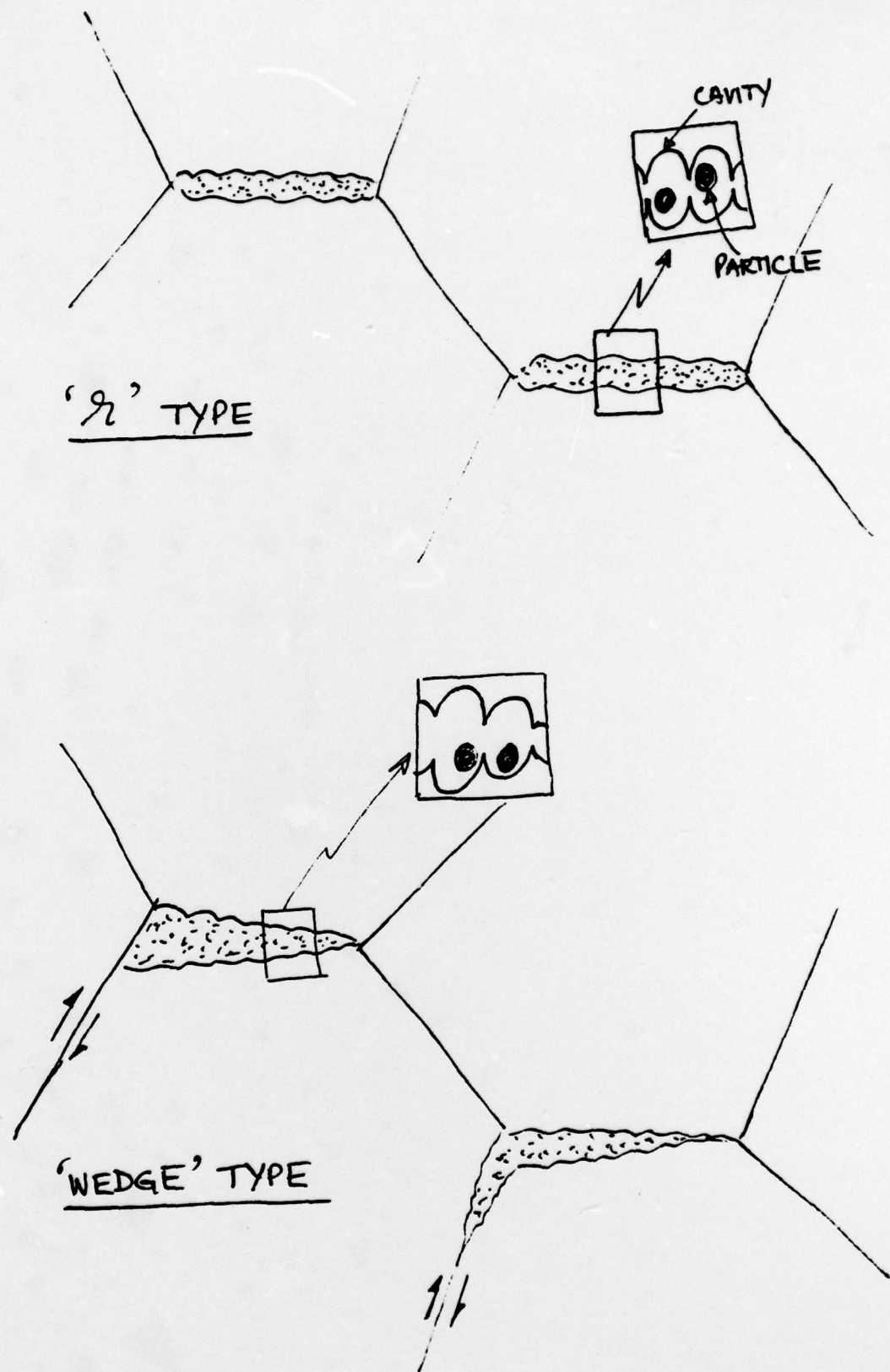


Fig. 5. The similarities and the differences in the mechanisms of 'r' type and 'wedge' type of intergranular cracking. In both, separation occurs by the growth of cavities which form at the second phase particles. However, in 'r' cavitation the growth of the cavities is accommodated by creep in the matrix in general, while in 'wedge' cracks the displacement for cavity growth is provided by grain boundary sliding.

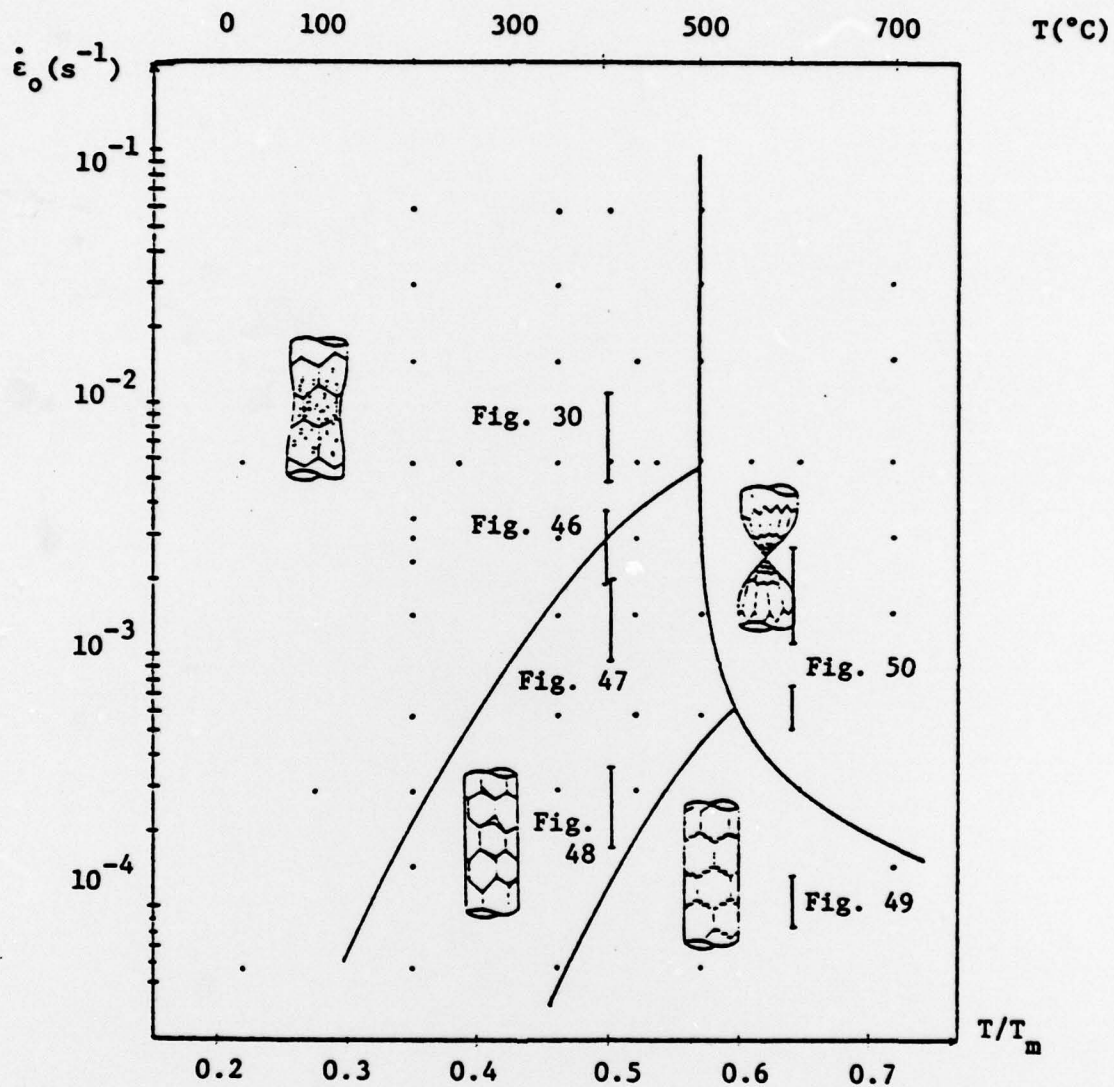


Fig. 6. Change in the mode of fracture in tensile tests on copper in which the strain rate was controlled and varied. The experimental points with the error bars were obtained from biaxial instead of uniaxial testing.

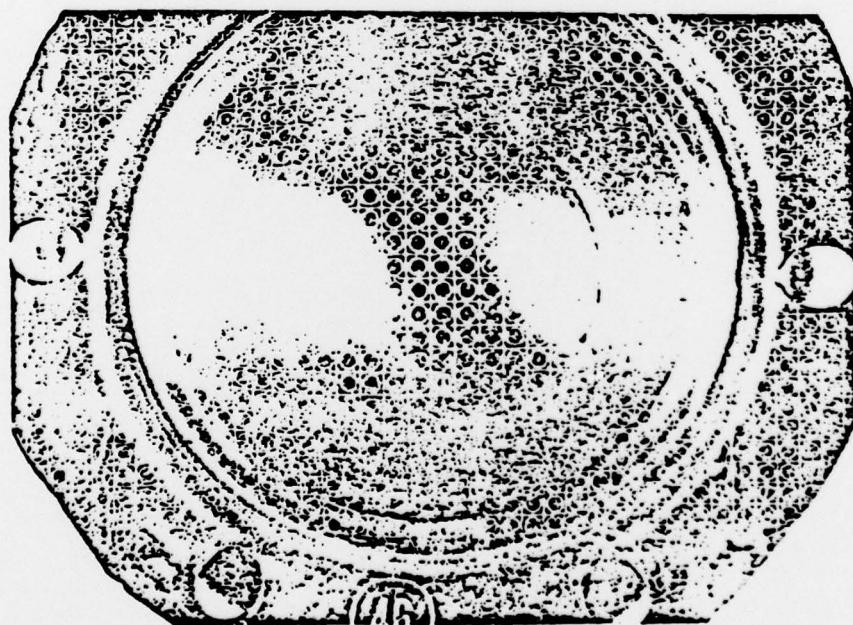
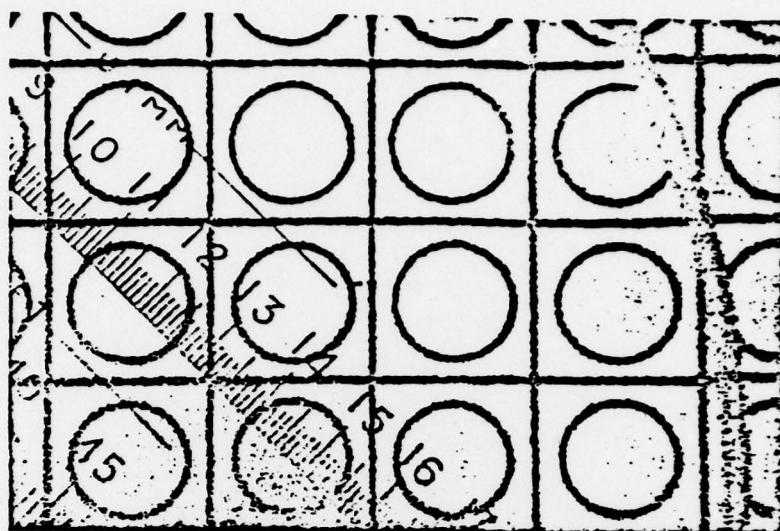


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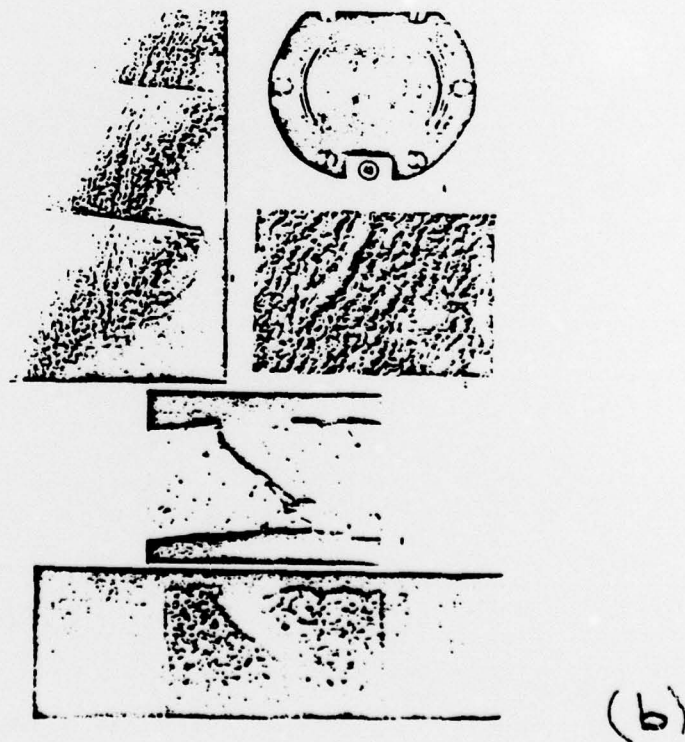
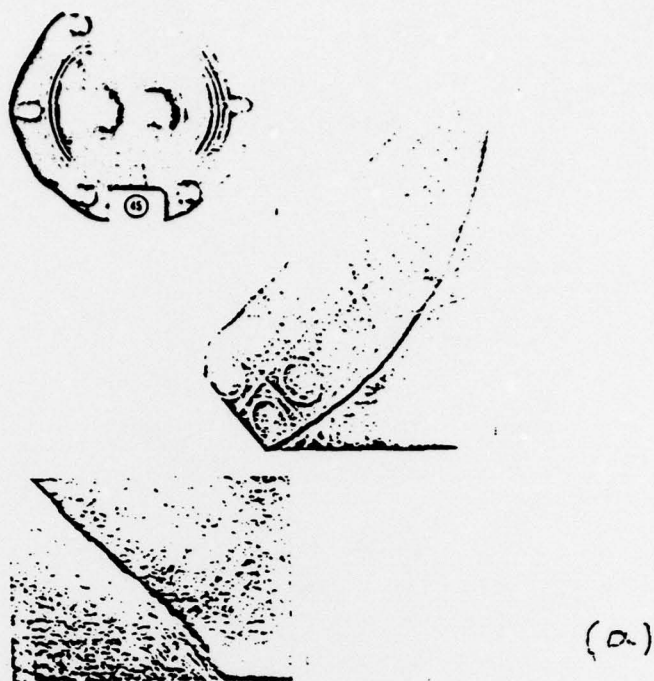


Fig. 8. The appearance of the fracture surface after fracture in a sheet specimen at room temperature (a) and at high temperature (b). Note the difference in the ductility.

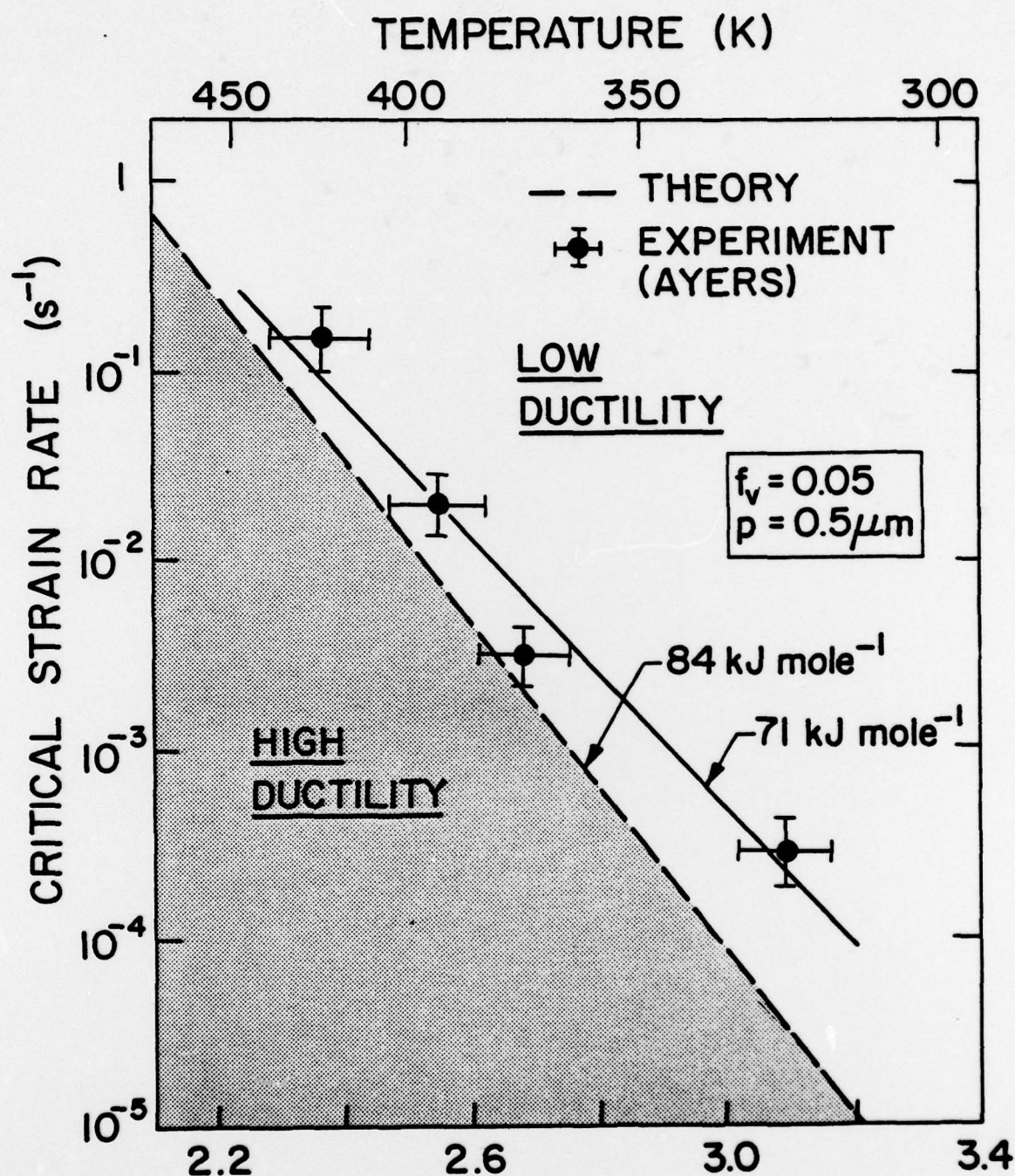
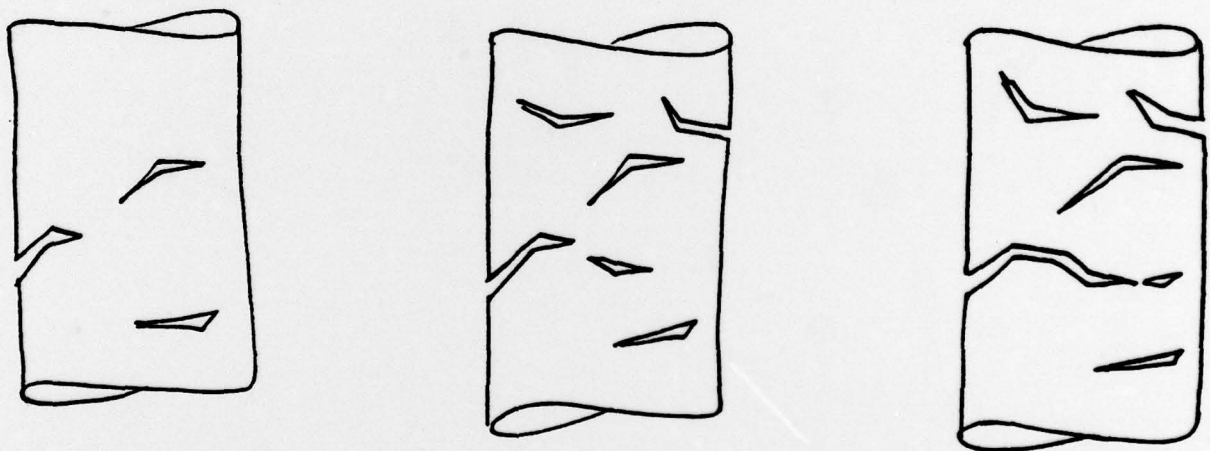
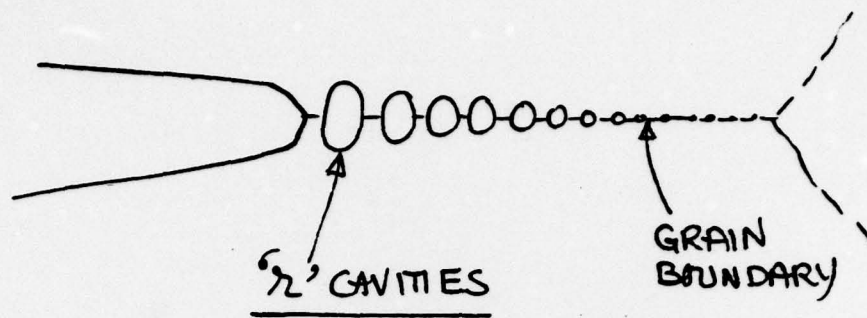


Fig. 9. The derivation of a critical strain rate below which cavitation at second phase particles should not occur, as applied to aluminum. Note reasonably good agreement with theory. Such diagrams have a potential use in the design of high temperature forming operations.



"WEDGE" CRACKING

Fig. 10. Two models for cavitation and fracture under conditions of high temperature fatigue. The 'r' type of cavities are expected to increase the rate of crack propagation when the criteria for cavity growth are satisfied in the local region near the crack tip. It is expected that the dominant effect of wedge cracking would be to accumulate cracks in a large cross section of the specimen; when their number and size exceeds a certain value then fracture would occur rapidly.

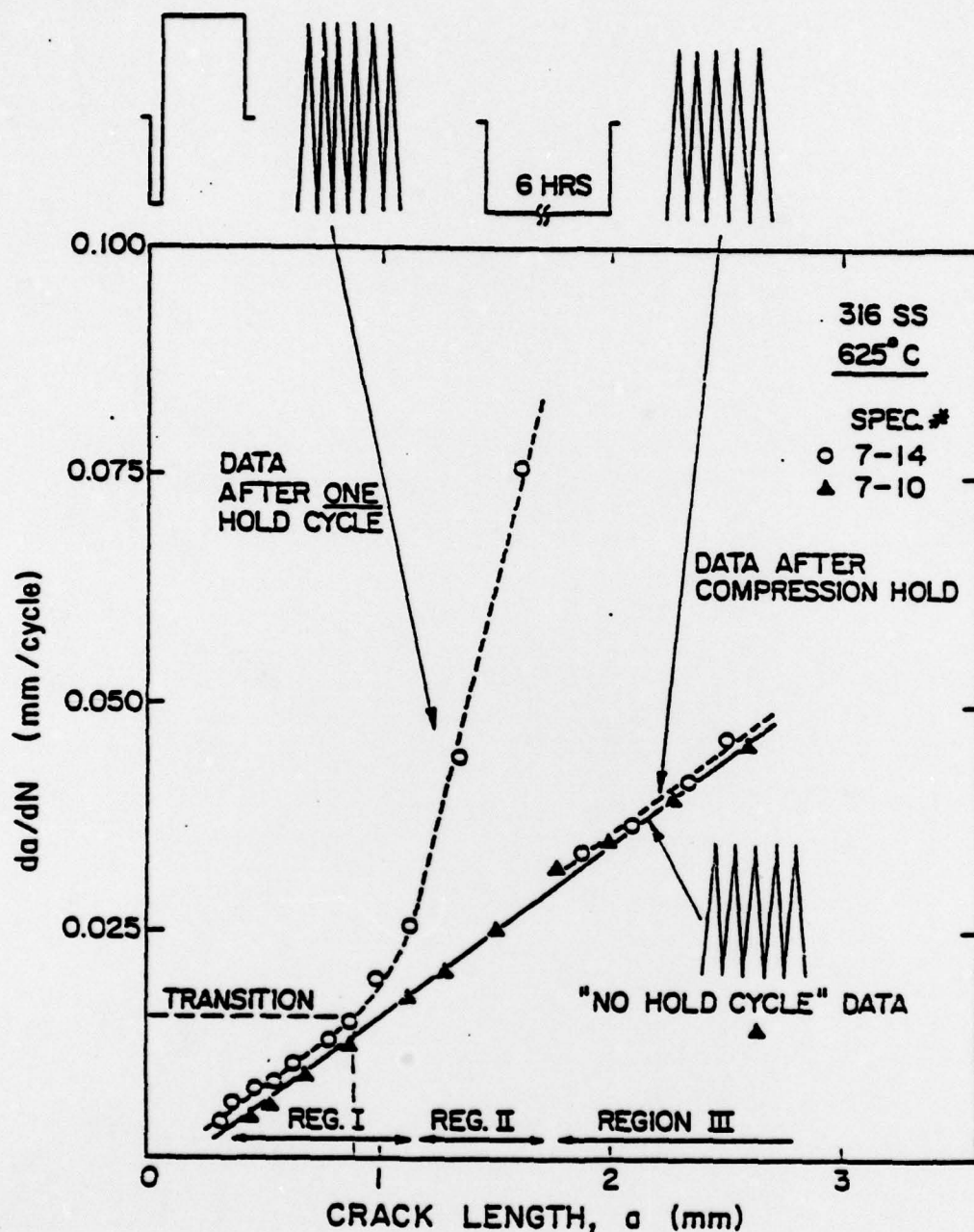
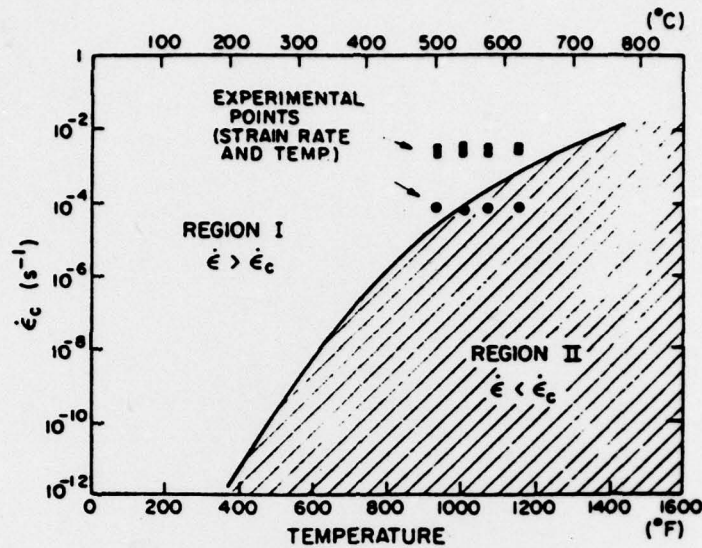
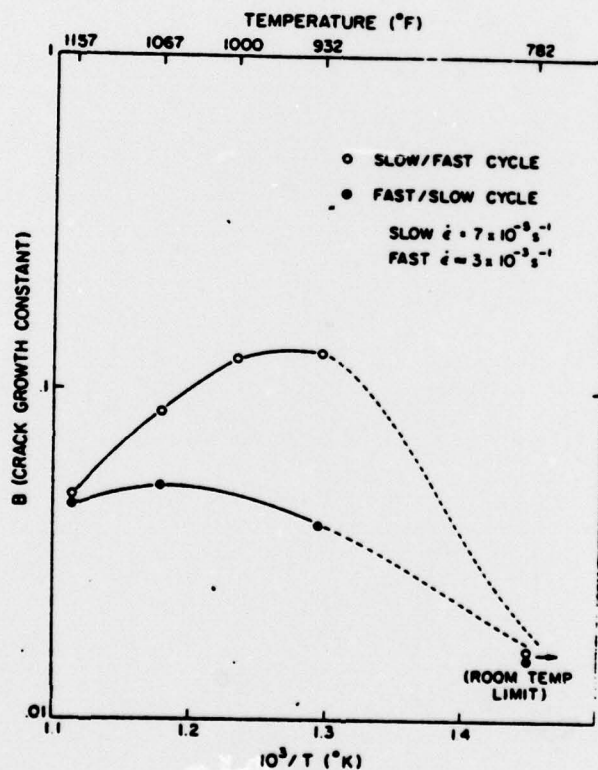


Fig. 11. The results of a 'model' experiment to study the influence of 'r' cavitation on fatigue cracks propagation at a fixed plastic strain amplitude. When a hold cycle is imposed to initiate the 'r' cavities then the crack growth rate accelerates after the crack tip opening displacement exceeds a critical value. However, if a compression hold is applied then cavities are removed by sintering and the crack growth rate decreases to the rate which was obtained without any cavities (open points).



(a)



(b)

Fig. 12. The influence of a slow/fast and fast/slow saw tooth shaped strain rate cycles on fatigue crack growth. It is postulated that the maximum in the rate of fatigue crack propagation is observed because at the intermediate temperature sliding occurs only in the tension going direction (since the slow rate is below the upper bound while the fast strain rate is above the upper bound), thus providing the optimum conditions for the accumulation of wedge cracks.

REPORT DOCUMENTATION PAGE		ADDITIONAL INSTRUCTIONS BEFORE COMPLETING FORM	
1. REPORT NUMBER AFOSR-TR- 80-0043		3. RECIPIENT'S CATALOG NUMBER	
4. TITLE (and Subtitle) Fracture at Elevated Temperature in Dispersion Strengthened Alloys		5. TYPE OF REPORT & PERIOD COVERED Final 10/1/75-9/30/79	
7. AUTHOR(s) Rishi Raj		8. CONTRACT OR GRANT NUMBER(s) AFOSR-76-2930	
9. PERFORMING ORGANIZATION NAME AND ADDRESS Department of Materials Science and Engineering Cornell University Ithaca, NY 14853		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS 2306/A1 61102F	
11. CONTROLLING OFFICE NAME AND ADDRESS AFOSR/NE, Bldg. 410 Bolling Air Force Base, Washington, DC 20332		12. REPORT DATE December 1979	
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)		13. NUMBER OF PAGES 36	
		15. SECURITY CLASS. (of this report) UNCLASSIFIED	
		15a. DECLASSIFICATION/DOWNGRADING SCHEDULE	
16. DISTRIBUTION STATEMENT (of this Report) Approved for public release; distribution unlimited.			
17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)			
18. SUPPLEMENTARY NOTES			
19. KEY WORDS (Continue on reverse side if necessary and identify by block number) Fracture, intergranular fracture, high temperature fracture, creep-fatigue interaction, second phase particles			
20. ABSTRACT (Continue on reverse side if necessary and identify by block number) This report describes important new findings in the understanding of intergranular cavitation and fracture in alloys which derive their creep strength from the presence of hard second phase particles, such as the nickel base superalloys. The work is primarily theoretical but is supported in most instances, by simple experiments on model alloys. Criteria for the nucleation of cavities at high temperature are developed and it is shown that cavities are most likely to nucleate at particles in the grain boundaries			

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20. It is shown that the cavities can grow by diffusion, by power law creep or by a combination of the two. The validity of the diffusional mechanism has been confirmed through experiments with bicrystals. A semi-empirical equation for ductility when the cavities grow apparently by power law is obtained. The equation has wide applicability in commercial alloys, including the new nickel base alloys such as MERL 76. In this equation the ductility is related to the density of the particles in the grain boundary and to the strain rate sensitivity of the material. It has been deduced that the state stress can influence the relative importance of diffusional vs. power law creep mechanism of cavity growth. Some work on creep-fatigue interaction was initiated in the course of this program.

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