

# Grain Boundary Oxidation and Oxidation Accelerated Fatigue Crack Nucleation and Propagation

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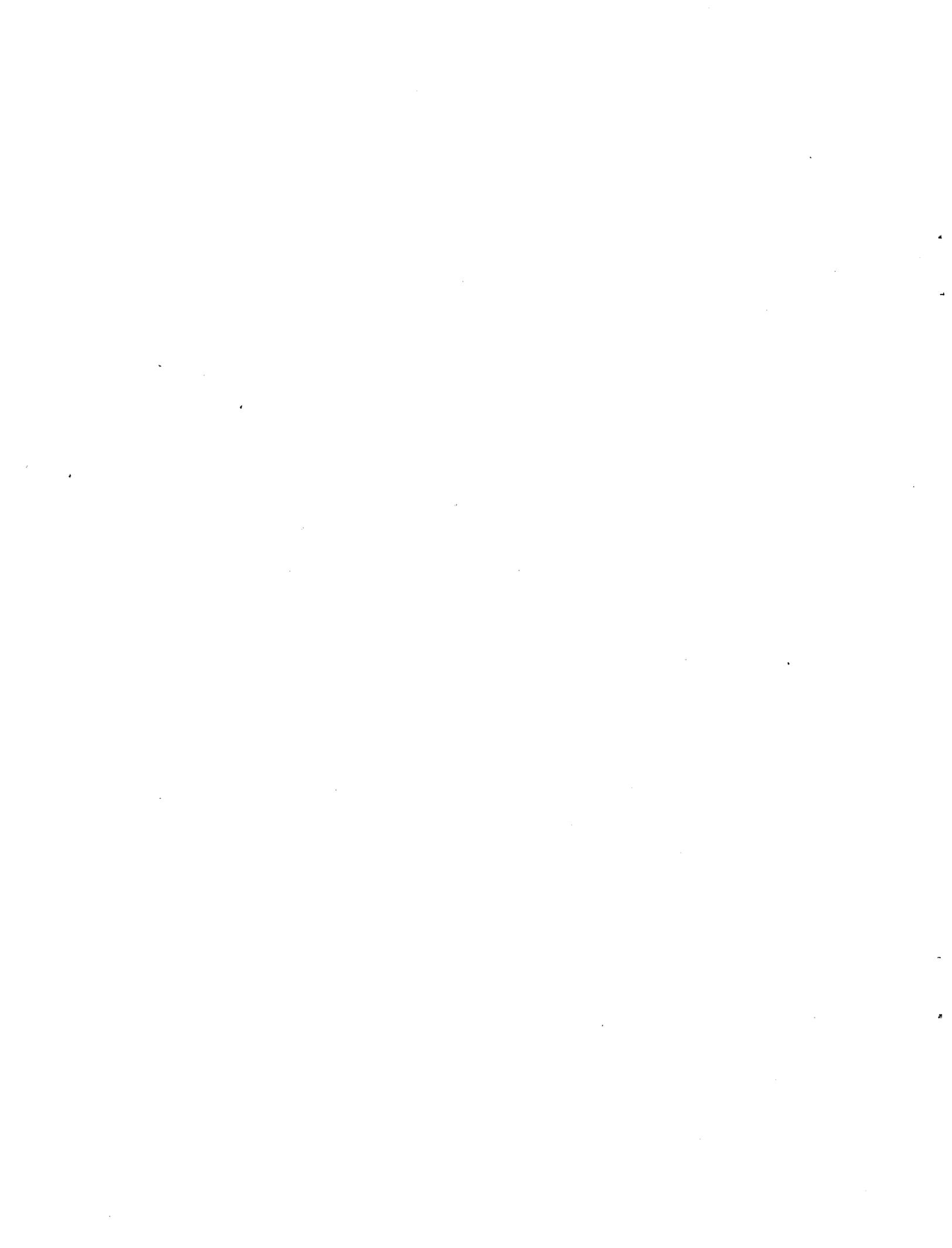
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## I. INTRODUCTION

Grain boundary oxidation may accelerate fatigue crack nucleation and propagation. McMahon and Coffin [1] studied oxide rupture as the process of fatigue crack nucleation in cast Udimet 500.

Antolovich et al. [2] have shown that, when an oxide reaches a critical size at an applied stress level, the oxide will fracture. The oxide crack will serve as a fatigue crack nucleus to grow by the subsequent cyclic load. Coffin [3], Solomon [4], and Solomon and Coffin [5] showed that fatigue crack growth in air has a strong frequency dependence, which is absent in vacuum. They attributed the accelerated fatigue crack growth rate and the frequency dependence in air to environmental effects.

Remy et al. [6] have shown that the oxidation of carbides accelerated fatigue crack growth in Mar-M509. Oshida and Liu [7] have studied the kinetics of grain boundary oxidation in a nickel-base superalloy, TAZ-8A. They treated the grain boundary oxide penetration as a precrack, and analyzed the effects of the precrack on the remaining fatigue life. Liu and McGowan [8], McGowan and Liu [9], Liu and Oshida [10] analyzed the effects of grain boundary oxidation on the accelerated fatigue crack growth. A model of intermittent microruptures of the grain boundary oxide was proposed and analyzed. In the low frequency region, the model shows that the fatigue crack growth rate is inversely proportional to cyclic frequency as observed in a number of materials.

In this paper, the earlier studies on grain boundary oxidation kinetics [7], the effects of grain boundary oxidation on fatigue life [11], and the model of intermittent microruptures of grain boundary oxide [10] will be reviewed and synthesized.

## II. GRAIN BOUNDARY OXIDATION KINETICS

Figure 1a shows a cross-section of an unoxidized coupon of the cast nickel-base superalloy, TAZ-8A. The microstructure etched by a mixed solution of HCl and  $H_2O_2$  (by 10:1 volume ratio) shows the  $\gamma/\gamma'$  matrix and the needle like prominent Chinese-script MC carbides, primary MC particles (white particles) and  $M_{23}C_6$  type carbide colonies (dark particles). Only the  $M_{23}C_6$  carbides were found along grain boundaries. It is noted that very few of these carbides are found in the thin surface layer of the test coupon.

Figure 1b shows the picture of a cross-section of an oxidized cylindrical coupon. The surface is covered by a thin layer of surface oxide. The grain boundary oxide penetrated much deeper into the specimen than the thickness of the surface oxide. When the applied stress is high enough the grain boundary oxide may crack, and the cracked grain boundary oxide may serve as a nucleus to grow by the subsequent fatigue cycles. If the grain boundary oxide crack is formed quickly, the fatigue crack nucleation period as well as the overall fatigue life will be shortened.

Oshida and Liu [11] measured the grain boundary oxide penetration depth,  $a$ , as a function of the oxidation temperature  $T$  and the exposure time  $t$ . At a given oxidation temperature and exposure time, the grain boundary penetration depth varies widely from one grain boundary to another. The statistical distribution of the grain boundary oxide penetration depth was studied, and its effect on the statistical scatter of the fatigue life will be analyzed.

Cylindrical test coupons were oxidized in a laboratory air at temperatures of 600, 800, and 1000°C for exposure times from 100 to 1000 hours. The oxidized disk coupons were sectioned, and the deepest grain boundary oxide penetration depth,  $a_{mi}$ , of the  $i$ th sectioned surface was measured. After the measurement, a thin layer of the coupon approximately 80  $\mu m$  thick was ground off and another  $a_{mi}$  of the new section was measured. This process was repeated twelve times for each oxidized coupon to collect twelve data points for the statistical analysis.

Altogether twelve sets of measurements at twelve combinations of  $T$  and  $t$  were collected. It is assumed that the relation between  $a_{mi}$ ,  $t$ , and  $T$  has the

form

$$a_{mi} = \alpha_i t^n \exp\left(-\frac{Q}{RT}\right) \quad (1)$$

The linear regression analysis of the data gives the empirical relation

$$a_m = 1.34 \times 10^{-3} t^{0.25} \exp(-4.26/RT) \quad (2)$$

$a_m$  in cm,  $t$  in seconds, the activation energy in Kcal/mol, and  $T$  in  $^{\circ}\text{K}$ . The coefficient of correlation is 0.96. The plot of  $\ln(a_{mi}/t^{0.25})$  versus  $(1/T)$  is shown in Figure 2. A sizeable scatter is shown in the figure. Couling and Smoluchowski [12] and Turnbull and Hoffman [13] have shown that grain boundary diffusion is a function of the angle of the mis-orientation of the two neighboring grains. Therefore it is reasonable to expect that a statistical scatter of  $a_{mi}$  exists.

Equation (2) is an empirical equation obtained by the regression analysis. The deviation of each individual measurement from the empirical relation can be expressed in terms of the variation  $\alpha_i$

$$\alpha_i = a_{mi} t^{-n} \exp\left(\frac{Q}{RT}\right) \quad (3)$$

With the measured  $a_{mi}$  at temperature  $T$  and exposure time  $t$  known,  $n = 0.25$ , and  $Q = 4.26$  Kcal/mol., the value of  $\alpha_i$  can be calculated from Equation (3). The values of  $\alpha_i$ 's for the 144 measurements are ranked in the increasing order. The probability of finding a value less than  $\alpha_i$  on a sectioned surface is

$P(\alpha_i)_j = M_i / (1 + N)$ .  $M_i$  is the ranking number of  $\alpha_i$  and  $N$  is the total number of the measurements.

The probability of finding an  $\alpha$ -value on a sectioned surface equal to or more than  $\alpha$  is  $[1 - P(\alpha)_j]$ . The Weibull plot of  $\ln \ln[1/(1 - P(\alpha)_j)]$  versus  $\alpha$  of all of the 144 data points is shown in Figure 3.

The Weibull distribution function is

$$[1 - P(\alpha)_j] = \exp\left[-\frac{(\alpha - \alpha_u)^b}{\alpha_o}\right] \quad (4)$$

For the data,  $b = 1.85$  is the shape parameter or Weibull modulus;  $\alpha_u = 0.53 \times 10^{-3}$ , the location parameter; and  $\alpha_o = 4.8 \times 10^{-6}$ , the scale parameter.

The maximum value of  $\alpha$  along the periphery of a sectioned surface can be considered as the maximum penetration of an exposed area of  $\pi Dd$  of the test coupon.  $D$  is the coupon diameter and  $d$  is the grain size. Another sectioned surface at a distance one grain diameter away contains an entirely different set of grain boundaries and it is another independent sample of the total exposed area of the test coupon.

Let  $P(\alpha)$  be the probability to find the maximum value less than  $\alpha$  on a unit surface area of a test coupon,  $P(\alpha)_S$  the probability of finding a value less than  $\alpha$  on a surface area  $S$ , and  $P(\alpha)_j$  the probability for a sectioned surface.

$$[1 - P(\alpha)_S] = [1 - P(\alpha)]^S \quad (5)$$

For an area of  $\pi Dd$ ,

$$[1 - P(\alpha)_j] = [1 - P(\alpha)]^{\pi Dd} \quad (6)$$

$$\ln[1 - P(\alpha)_S] = (S/\pi Dd) \ln[1 - P(\alpha)_j] \quad (7)$$

Combining (4) and (7),

$$P(\alpha)_S = 1 - \exp(-R) \quad (8a)$$

$$R = (S/\pi Dd) [(\alpha - \alpha_u)^b / \alpha_o] \quad (8b)$$

The value of  $P(\alpha)_S$  can be taken as the value of  $P(a)_S$ , the probability to find a grain boundary penetration depth "a" on an exposed surface area  $S$ .

It has been known that fatigue lives of gas turbine engine components have very wide statistical scatter. It is also known that the statistical scatter of fatigue crack propagation is rather narrow. Therefore grain boundary oxidation and its effect on fatigue crack nucleation could be one of the contributing factors to the wide variation in the fatigue lives of gas turbine engine components.

### III. GRAIN BOUNDARY OXIDATION AND LOW CYCLE FATIGUE LIFE

In the intermediate  $\Delta J$  region, fatigue crack growth rate is related to  $\Delta J$  by a power relation,

$$\frac{da}{dN} = A\Delta J^m \quad (9)$$

Shih and Hutchinson have shown that for a small crack in a Ramberg-Osgood elastic-plastic solid in general yielding [14,15]

$$\frac{J}{\sigma_Y \epsilon_Y a} = \eta_1 \frac{W_D}{\sigma_Y \epsilon_Y} + \eta_2 \quad (10)$$

where  $\eta_1$  and  $\eta_2$  are functions of strain hardening exponent "n". For a given material, they are constants. The value of  $\eta_2$  is usually much smaller than the first term.  $W_D$  is the deformation work density

$$W_D = \int_0^\epsilon \sigma \, d\epsilon \quad (11)$$

$\sigma$  and  $\epsilon$  are the applied stress and strain away from the cracked region. Zheng and Liu [16] have studied the crack tip fields for small cracks in plates in tension for piecewise power hardening materials.  $\sigma = E\epsilon$  for  $\sigma \leq \sigma_Y$  and  $(\sigma/\sigma_Y) = (\epsilon/\epsilon_Y)^n$  for  $\sigma > \sigma_Y$ . Small single edge cracks ( $a/w = 0.1$ ) in both plane stress and plane strain were studied. They found that Equation (10) is also applicable to the piecewise power hardening materials.

For cyclic loading, Equation (10) is modified to the form

$$\Delta J = (\eta_1 \Delta W_D + \eta_2 \sigma_{YC} \epsilon_{YC}) a \quad (12)$$

Crack tip stresses and strains will cause a crack to grow, only when the crack tip is open. Take the simple assumption that under a completely reversed cyclic axial loading, a crack tip is open when the applied stress becomes tensile during the loading half of the cycle (see Figure 4).

Therefore

$$\Delta W_D = \int_0^{\Delta \epsilon_p} \sigma d\epsilon + \frac{\Delta \epsilon_e}{2} \sigma d\epsilon \quad (13)$$

$\sigma$  and  $\epsilon$  are the applied stress and strain. For a piecewise power hardening material,  $\Delta W_D$  is the cross-hatched area in Figure 4, which can be approximated by

$$\Delta W_D = \frac{\Delta \sigma^2}{8E} + \frac{1}{2} (\sigma_{Yc} + \frac{\Delta \sigma}{2}) \Delta \epsilon_p \quad (14)$$

Substituting (12) into (9) and integrating,

$$(\Delta W_D)^m (N_f - N_o) = \frac{1}{A n_1^m (1 - m)} (a_f^{(1 - m)} - a_o^{(1 - m)}) \quad (15)$$

The remaining fatigue life after a crack reaches the size  $a_o$  is  $(N_f - N_o)$ .  $a_f$  is more or less a constant. Therefore the fatigue life with a precrack,  $N_{fa_o}$ , is a function of the precrack size,  $a_o$ .

$$N_{fa_o} = f(a_o) \quad (16)$$

The grain boundary oxide crack can be considered a precrack. If the relation (16) is deterministic, the probability to have a fatigue life of  $N_{fa_o}$  is also the probability to have an oxide crack size  $a_o$  on a specimen surface. If the oxidation process is fast, the fatigue life could be reduced considerably. It is well known that the fatigue life of an engine component has a wide statistical scatter. Perhaps grain boundary oxidation and grain boundary oxide cracking contribute to a significant part of the scatter.

The reduction in fatigue life is caused by the shortened "crack nucleation" period. The accelerated fatigue crack growth due to oxidation will be discussed in the next section.

#### IV. GRAIN BOUNDARY OXIDATION AND FATIGUE CRACK PROPAGATION

Fatigue crack growth at elevated temperatures is sensitive to cyclic frequency. Figure 5 shows the frequency effect on fatigue crack growth rate at a constant  $\Delta K$  level and at a constant temperature  $T$ . The cyclic wave shapes of these tests are also shown. Several of the tests had hold time at  $K_{\max}$ . The others had sawtooth wave forms. The crack growth rates of Inconel 718, Astroloy, and Waspaloy, in the low frequency region, are inversely proportional to cyclic frequency,  $\nu$ , and are linearly proportional to the time duration at the  $K_{\max}$ . The time rate of fatigue crack growth,  $da/dt = \alpha(da/dN)(1/\nu)$  is a constant.  $\alpha$  is a constant, its value depends on the wave shape of the fatigue cycle. In the low frequency region, fatigue crack growth is intergranular.

For a sustained load test, i.e., constant-K test,  $da/dt$  is constant and crack growth is intergranular. Crack growth under a sustained constant-K test is commonly known as creep crack growth. The fatigue crack growths in the low frequency region have these two growth characteristics. Thus the fatigue crack growth in the low frequency region is often referred to as creep crack growth, even though fatigue crack growth rates at elevated temperatures in vacuum and in air are quite different, and the difference cannot be explained in terms of creep damage.

The grain boundary oxide penetration of TAZ-8A is given by Equation (1). Perhaps, the oxide penetration depth at a crack tip can be written in the form

$$a = \beta(D_{gb}t)^m \quad (17)$$

where  $D_{gb}$  is the grain boundary diffusion coefficient and  $m$  is an empirical constant.  $\beta$  could be a function of the crack tip field and the oxygen concentration in the ambient.

Antolovich has found that, at an applied stress, the oxide in a smooth specimen will rupture, when it reaches a critical size [2]. The critical crack tip oxide size at rupture must be related to  $K$ .

These tests had a hold time at  $K_{max}$ . At  $K_{max}$ , the crack tip oxide will rupture when the oxide reaches a critical size,  $\delta a$ , and the crack will grow by the same amount. Once the crack tip grows to its new position, this process of grain boundary diffusion, grain boundary oxidation, grain boundary oxide rupturing, and the incremental crack growth will repeat again. This process can be repeated many times during the hold time at  $K_{max}$ .

At a given  $K$ -level, the time increment,  $\delta t_i$ , necessary for the oxide to reach the critical size  $\delta a_i$  are related directly to  $a_i$  by a relation such as

$$\delta a_i = \beta (D_{gb} \delta t_i)^m \quad (18)$$

The number of microruptures,  $n$ , during the hold time,  $\Delta t$ , is  $n = \Delta t / \delta t = \Delta a / \delta a$ .  $\Delta a$  is the crack growth during the hold time of one fatigue cycle. " $n$ " is proportional to  $\Delta t$  but inversely proportional to frequency,  $\nu$ . Fatigue crack growth per cycle is the sum of the intermittent microruptures per cycle.

$$\frac{da}{dN} = n \delta a = \frac{1}{\nu \delta t} \delta a$$

or

$$\left(\frac{da}{dN}\right)_\nu = \frac{\nu_0}{\nu} \times \left(\frac{da}{dN}\right)_{\nu_0} \quad (19)$$

$\left(\frac{da}{dN}\right)_{\nu_0}$  is the crack growth rate at a reference frequency  $\nu_0$ .  $da/dN$  is inversely proportional to  $\nu$ . This agrees with the empirical data for Inconel 718, Astroloy, and Waspaloy in Figure 5 in the low frequency region. The same conclusion that  $da/dN$  is inversely proportional to cyclic frequency, can be derived for other wave forms if the frequency does not affect crack tip field [10].

The microruptures, which take place along oxidized grain boundary, result in intergranular crack growth. Therefore both the constant time rate of fatigue crack growth and the intergranular fracture are not necessarily caused by creep damage. They can be the result of grain boundary oxidation. Creep cracking growth has a connotation of crack growth by creep deformation, grain boundary void formation, grain boundary cavitation, and/or the growth of such voids and cavities. At least for some materials, to call fatigue crack growth as creep cracking in the low frequency region is a misnomer.

## V. SUMMARY AND CONCLUSIONS

1. Grain boundary may accelerate fatigue crack nucleation and propagation.
2. Grain boundary kinetics of a nickel-base superalloy, TAZ-8A was studied. The grain boundary oxide penetration depth can be expressed in a modified form of the Arrhenius relation.
3. Grain boundary oxide penetration depth varied widely from one boundary to another. The Weibull's distribution function of grain boundary oxide penetration was studied. The variation in oxide penetration may contribute to the wide statistical scatter of the fatigue lives of gas turbine engine components.
4. The grain boundary oxidation kinetics are essential for the development of a quantitative life prediction methodology based on a mechanistic model for those materials susceptible to fatigue life impairment by oxidation.
5. The effects of grain boundary oxide crack on fatigue life are analyzed and discussed in terms of the reduced fatigue crack "nucleation" period.
6. An intermittent grain boundary oxide microrupture model is proposed for the fatigue crack growth in the low frequency region. The observed inverse relation between  $da/dN$  and cyclic frequency,  $\nu$ , and the observed intergranular fatigue crack growth are consistent with the proposed model.
7. To call fatigue crack growth in the low frequency region "creep cracking" because of constant time rate of crack growth and intergranular fracture, is a misnomer for those materials susceptible to fatigue life impairment by the oxidation process.

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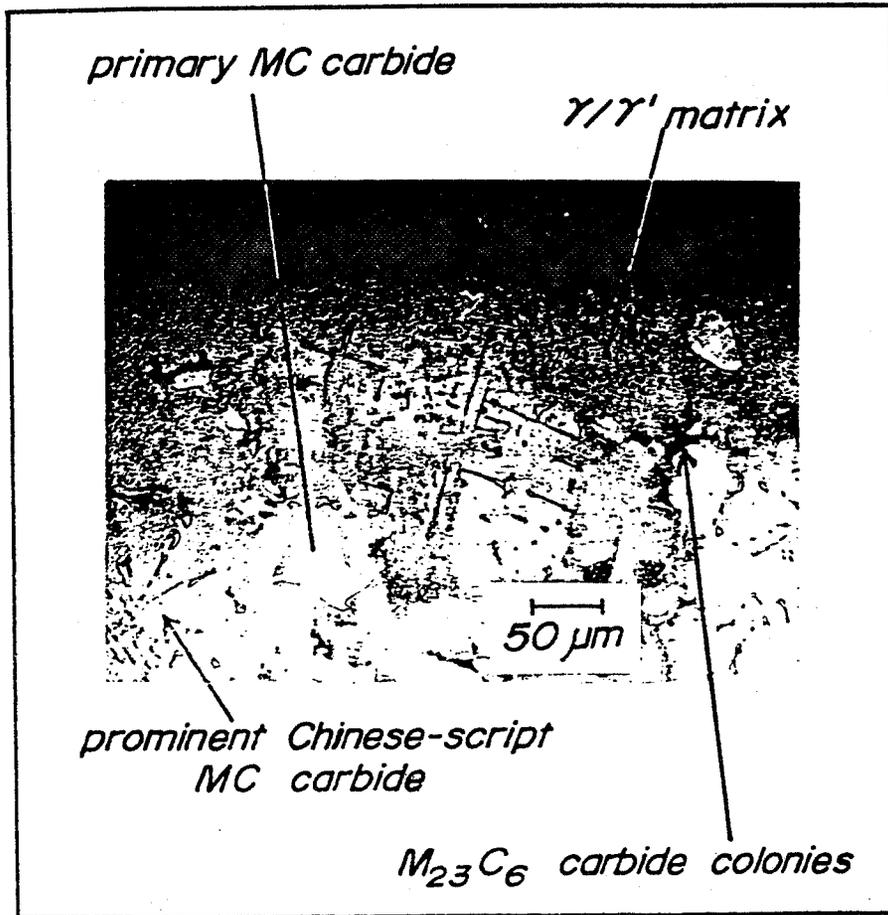


Figure 1a Cross-section microstructure of unoxidized test coupon, TAZ-8A

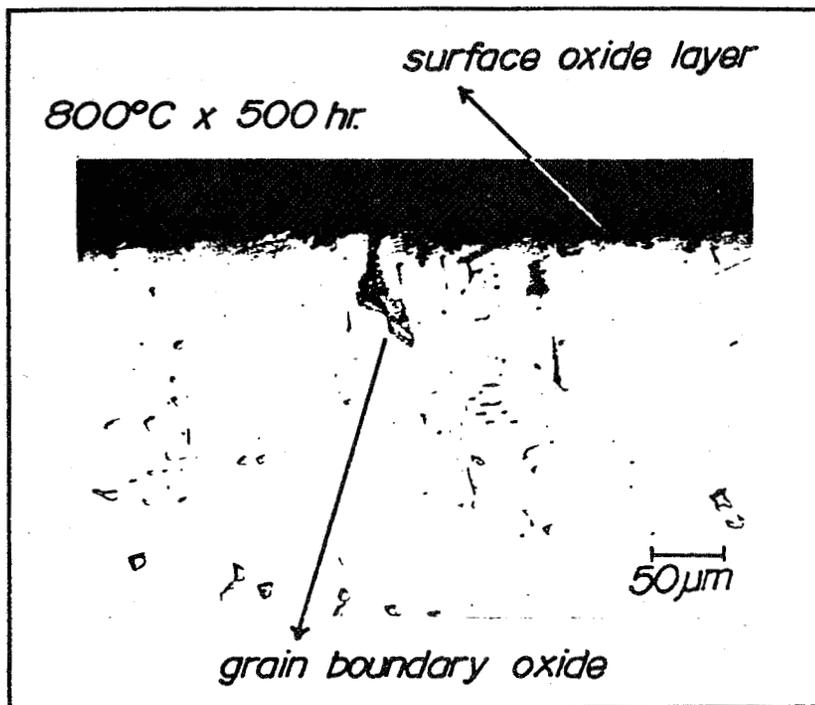


Figure 1b Cross-section of oxidized test coupon

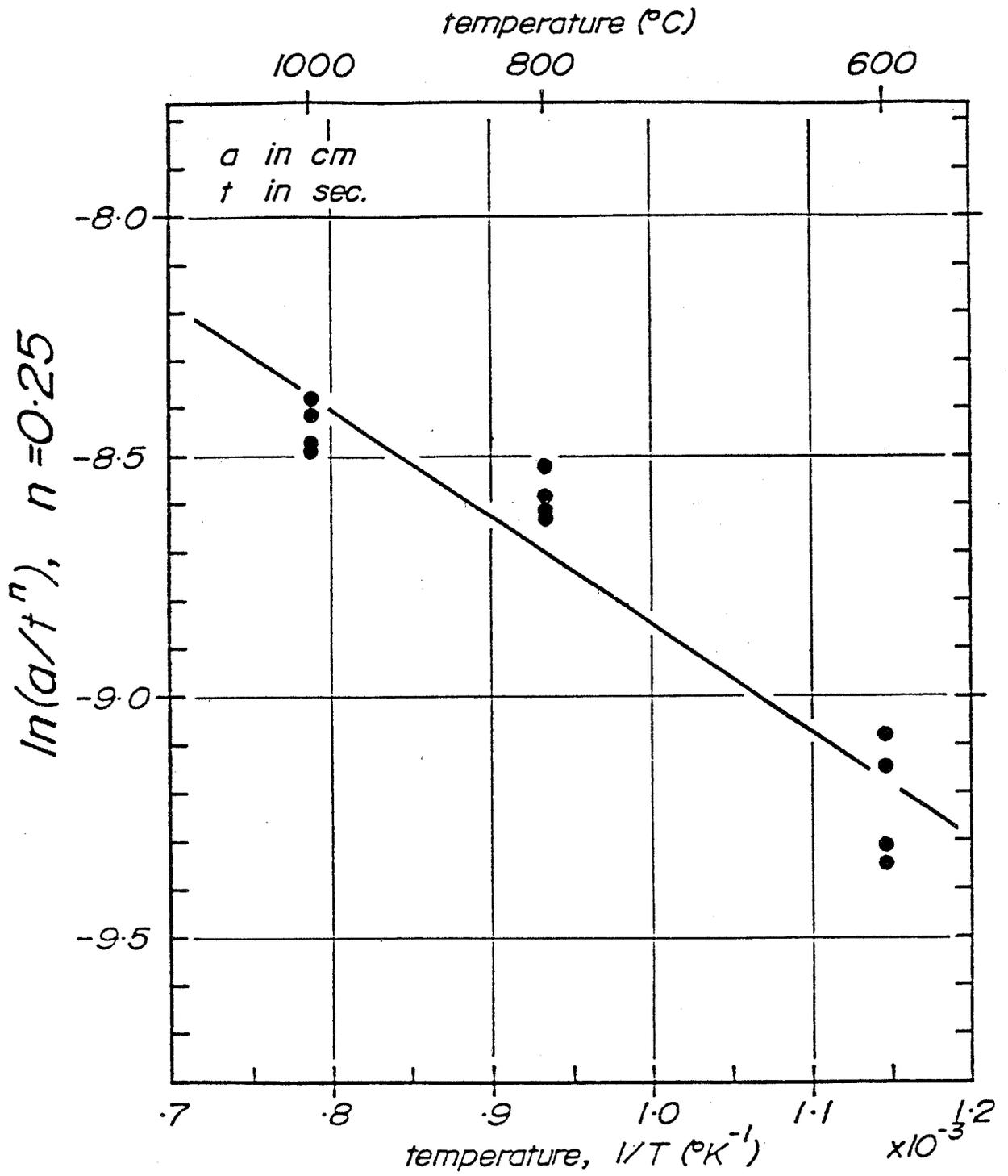


Figure 2 Arrehius plot of  $a_{mi}$ , based on Equation (2) in the text

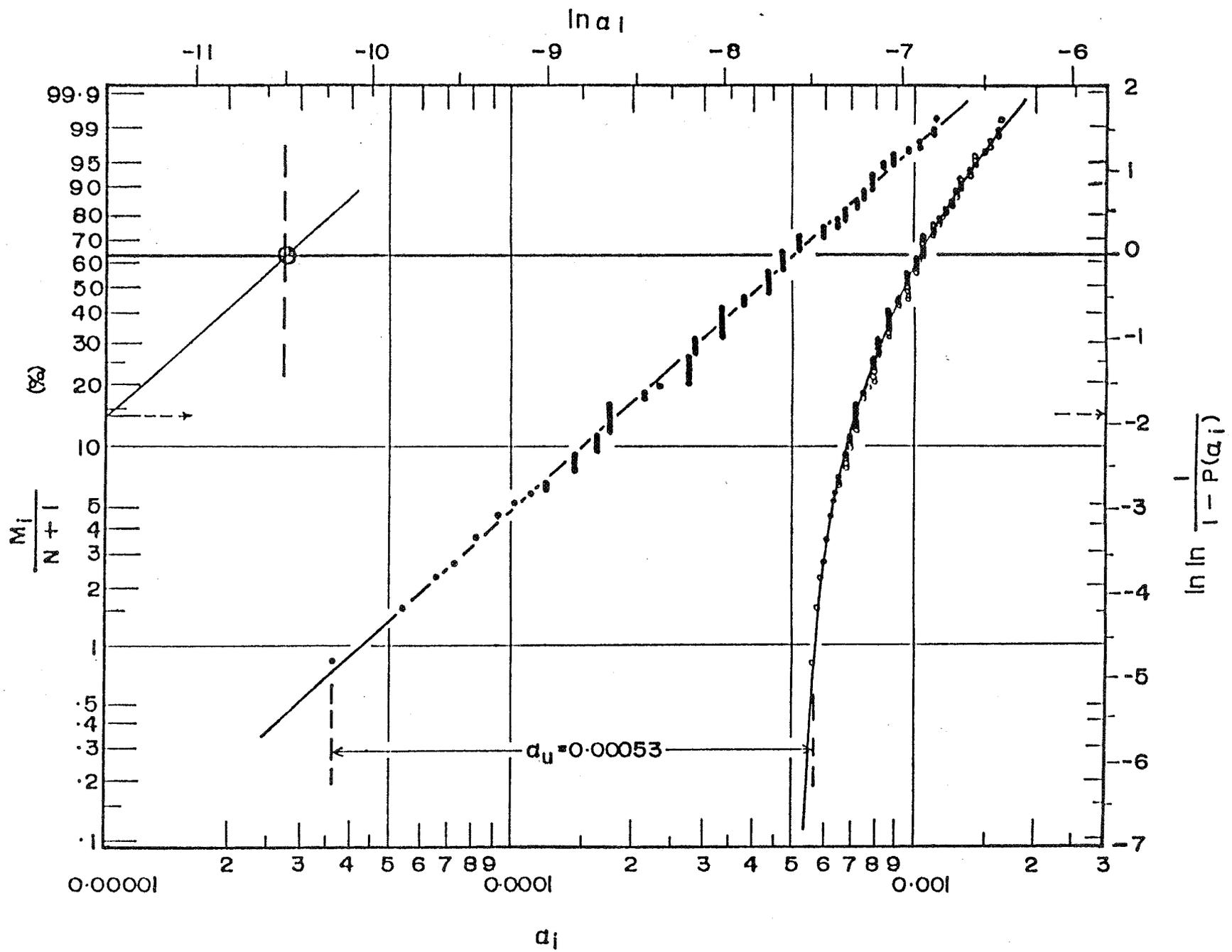


Figure 3 Weibull plot of  $\alpha$ , based on Equation (3) in the text

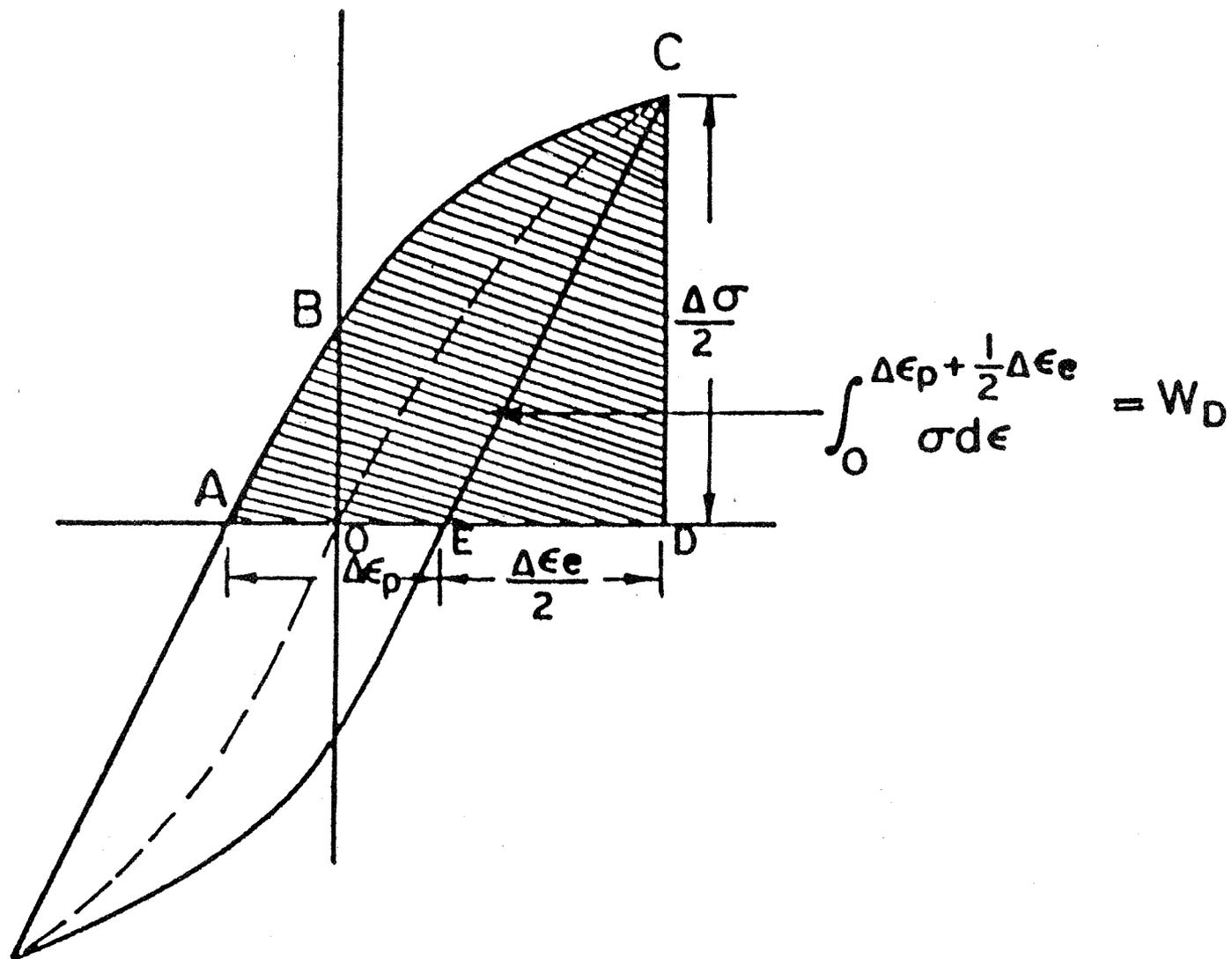


Figure 4 Cyclic stress-strain loop and the deformation work density

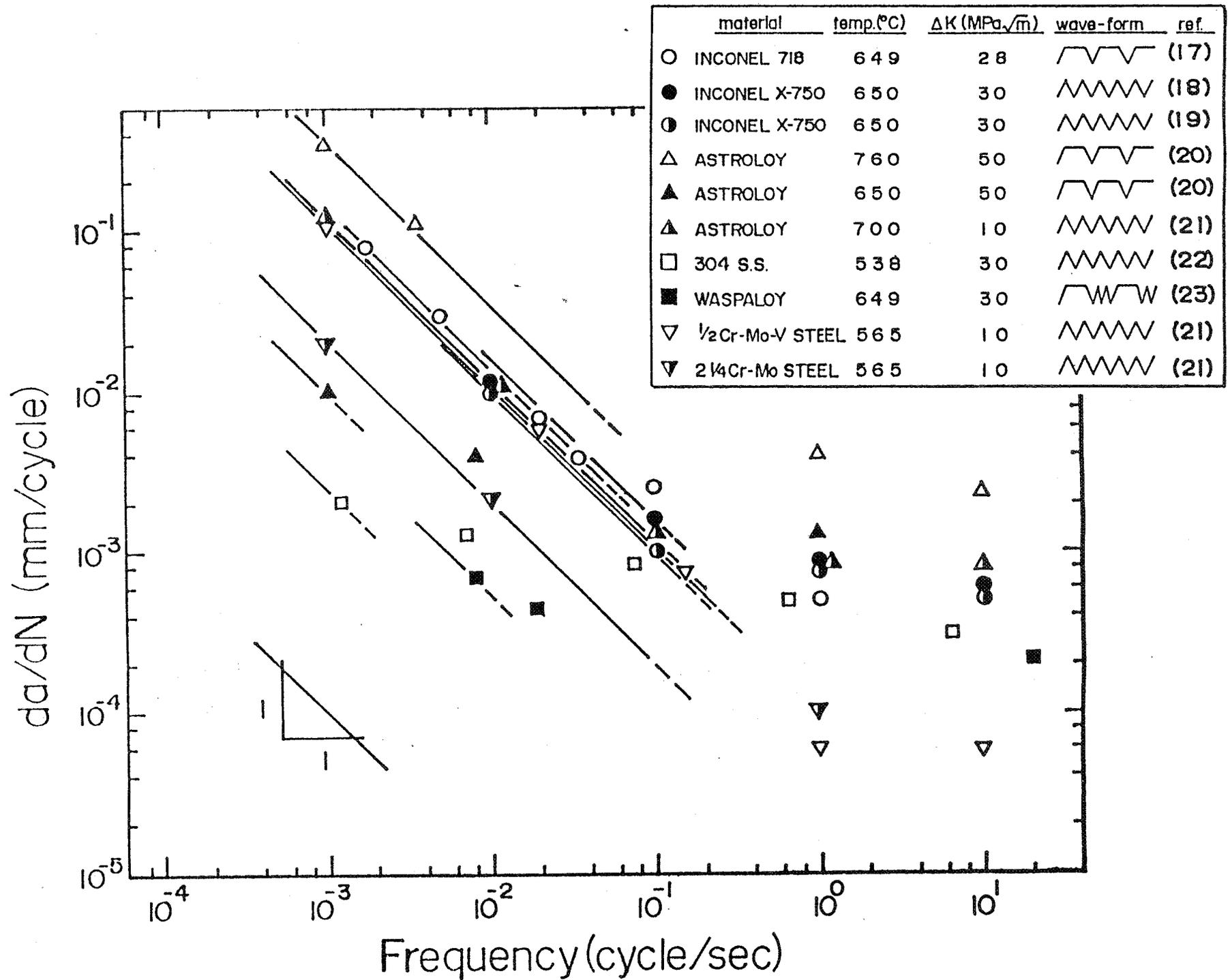


Figure 5 Frequency dependencies of high temperature low cycle fatigue crack growth of various materials

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16. Abstract <b>Fatigue life at elevated temperatures is often shortened by oxidation. Grain boundary oxidation penetrates deeper than the surface oxidation. Therefore, grain boundary oxide penetration could be the primary cause of accelerated fatigue crack nucleation and propagation, and the shortened fatigue life at elevated temperatures. Grain boundary oxidation kinetics was studied and its statistical scatter was analyzed by the Weibull's distribution function. The effects of grain boundary oxidation on shortened fatigue life were analyzed and discussed. A model of intermittent microruptures of the grain boundary oxide was proposed for the fatigue crack growth in the low frequency region. The proposed model is consistent with the observations that fatigue crack growth rate in the low frequency region with hold time at <math>K_{max}</math> is inversely proportional to cyclic frequency and that crack growth is intergranular.</b>					
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