DOE/ER/45141--T5

DE91 000987

Technical Report

Department of Energy
Division of Materials Science
Attention: Dr. Joseph Darby

# MICROMECHANISMS OF BRITTLE FRACTURE: ACOUSTIC EMISSIONS AND ELECTRON CHANNELING ANALYSES

Submitted by:

Professor William W. Gerberich

Department of Chemical Engineering and Materials Science
University of Minnesota
Minneapolis, MN 55455

June, 1990

# MICROMECHANISMS OF BRITTLE FRACTURE: ACOUSTIC EMISSIONS AND ELECTRON CHANNELING ANALYSES

## 1. Objective

The objectives of the work in this grant are to:

i) Evaluate the initial stages of cleavage nucleation in single and polycrystalline samples;

ii) Determine the controlling event(s) which lead(s) to unstable cleavage (is it an unstable "cluster" of microcracks or a crack-tip opening displacement criterion for an array of ligaments surrounding these microcracks?);

iii) Determine how the process zone, which depends upon microstructure and processing history, affects the controlling event(s);

iv) Use selected area channeling patterns (SACP's) to assist in an independent measure of the cleavage fracture stress of grains cleaved at or just outside the elastic-plastic boundary; also, use it to evaluate static and dynamic strain distributions;

v) Evaluate the effects of dislocation shielding and overload using combined methods of computational mechanics with discretized dislocation arrays and direct observations of dislocations using channeling, etch pit and birefringence methods.

## 2. Accomplishments of Prior Work

There have been four major accomplishments in the prior (and current to July 1, 1990) three-year effort. These deal with understanding hydrogen embrittlement thresholds, with computational studies which assist in understanding both thresholds and cleavage onset, with crack-tip dislocation emission and instability, and with process zone concepts in brittle fracture.

### 2.1. Hydrogen embrittlement

Since it was known from Vehoff and Rothe's studies[(1)] that hydrogen induced crack growth in Fe-3wt%Si single crystals was on the {100} cleavage plane, this was an ideal system for studying discontinuous cleavage. For the first time, a large scale program on large single crystals of iron was conducted where stress intensity levels, crack velocities and acoustic emission signals gave quantitative details on the micromechanistic process. From these investigations, the following can be summarized:

i) hydrogen-nucleated cleavage very near the root of a sharp crack is consistent with existing concepts of decohesion;

ii) embrittlement thresholds depend on both a critical hydrogen level being achieved in a local volume near the crack tip and a local stress which appears to approach the theoretical crystal strength;

iii) the threshold condition is <u>different</u> for plane stress and plane strain as verified by crack tunneling in a macroscopic $\langle 110 \rangle$ growth direction, which should maintain a straight crack front if there were no state of stress effect;

iv) there is evidence of anisotropic plasticity on the fracture surfaces strongly suggesting that dislocation shielding is an important contribution;

v) while thresholds as low as 16 MPa-m$^{1/2}$ have been observed, this may be slowly increased to $K_I$ values as large as 50 MPa-m$^{1/2}$ without failure;

vi) hydrogen cracking above threshold is discontinuous with 1 $\mu$m steps involved as verified by fractography and acoustic emission;

vii) the effect of test temperature on stage II growth kinetics is directly analogous to those observed in higher strength steels with either internal or external hydrogen environments.

This investigation has led to a major review (invited, to be published), a number of papers listed below and several others in various stages of preparation.

1. W.W. Gerberich, C.E. Hartbower and X. Chen, "Internal Carbon Embrittlement," in *Environmental Degradation of Engineering Materials, III*, M.R. Louthan, Jr., R.P. McNitt and R.D. Sisson, Jr., eds., The Pennsylvania State University, University Park, PA (1987) pp. 105–119.
2. W.W. Gerberich, T. Livne, M. Kaczorowski and X.-F. Chen, "Crack Growth from Internal Hydrogen — Temperature and Microstructural Effects in 4340 Steel," *Met. Trans. A*, May (1988) pp. 1319–1334.
3. W.W. Gerberich, X. Chen and R. Caretta, "Carbon Segregation Induced Grain Boundary Embrittlement," MRS Symposium Vol. 122 (1988), pp. 399–404.
4. W.W. Gerberich, "The Micromechanics and Kinetics of Environmentally-Induced Fractures," ASM Materials Science Seminar, S. Nair, ed., ASM, Metals Park, OH (in press) 1989.

2.2. Computational studies

These were primarily aimed at obtaining a better description of the local stress field near a crack tip since there was a concern that continuum models did not necessarily give the local maximum. For example, it was difficult to understand bond breaking (cleavage) by stresses which were 1/100 of the shear modulus. Atomistic studies had not been developed sufficiently and it was felt that the continuum mechanics of small-scale yielding solutions were both too far field and too smooth to represent microstructural discontinuities. For these reasons, an intermediate scale discretized dislocation model after Atkinson and Clements[2] was utilized to simulate the stresses. This was chosen since it allows an anisotropic elastic solution, with dislocation point forces, appropriate to iron. It was also of interest since it allowed the stress tensor to be evaluated for this model which is similar to those of Rice and Thomson,[3] Burns,[4] Li,[5] Argon,[6] Brede and Haasen[7] and others. Although a large number of papers have been written on this subject, few had attempted to calculate the stress tensor associated with the crack and dislocations at equilibrium. Thus, a major accomplishment of this study was utilizing the power of the supercomputer to analyze the local stress distribution as a function of slip character. Some of the following findings were quite surprising to us:

i) in a single-slip system, the number of dislocations in a localized slip band and the length of the band required for equilibrium increased with increasing stress intensity;

ii) with two slip systems, the local slip band length was controlled by the macroscopic slip angle. This length increased by a factor of six as the slip angle deviated from the crack plane to 90 degrees to the crack plane. Correspondingly, the maximum normal stress decreased by a factor of six;

iii) for applied stress intensities as low as 2 MPa-m$^{1/2}$, near theoretical stresses (20 GPa) were reached at the crack tip. These stresses only existed over a narrow 1 nm region;

iv) the critical region over which this stress was achieved increased as to the square of the applied stress intensity.

v) at hydrogen embrittlement thresholds, the simulation for $K_{I_{th}}$ = 16 MPa-m$^{1/2}$ provided a stress maximum occurring at 20 nm below the crack-tip, well within the 1 $\mu$m discontinuous crack advance observed;

vi) a simulation of prior overloads and the attendant far-field residual plasticity provided a rationale for the experimental observations in Figure 1.

Such crack/dislocation equilibrium simulations of stress distributions provided background for several analytical descriptions of brittle instabilities as described in several overview papers in preparation and the following:

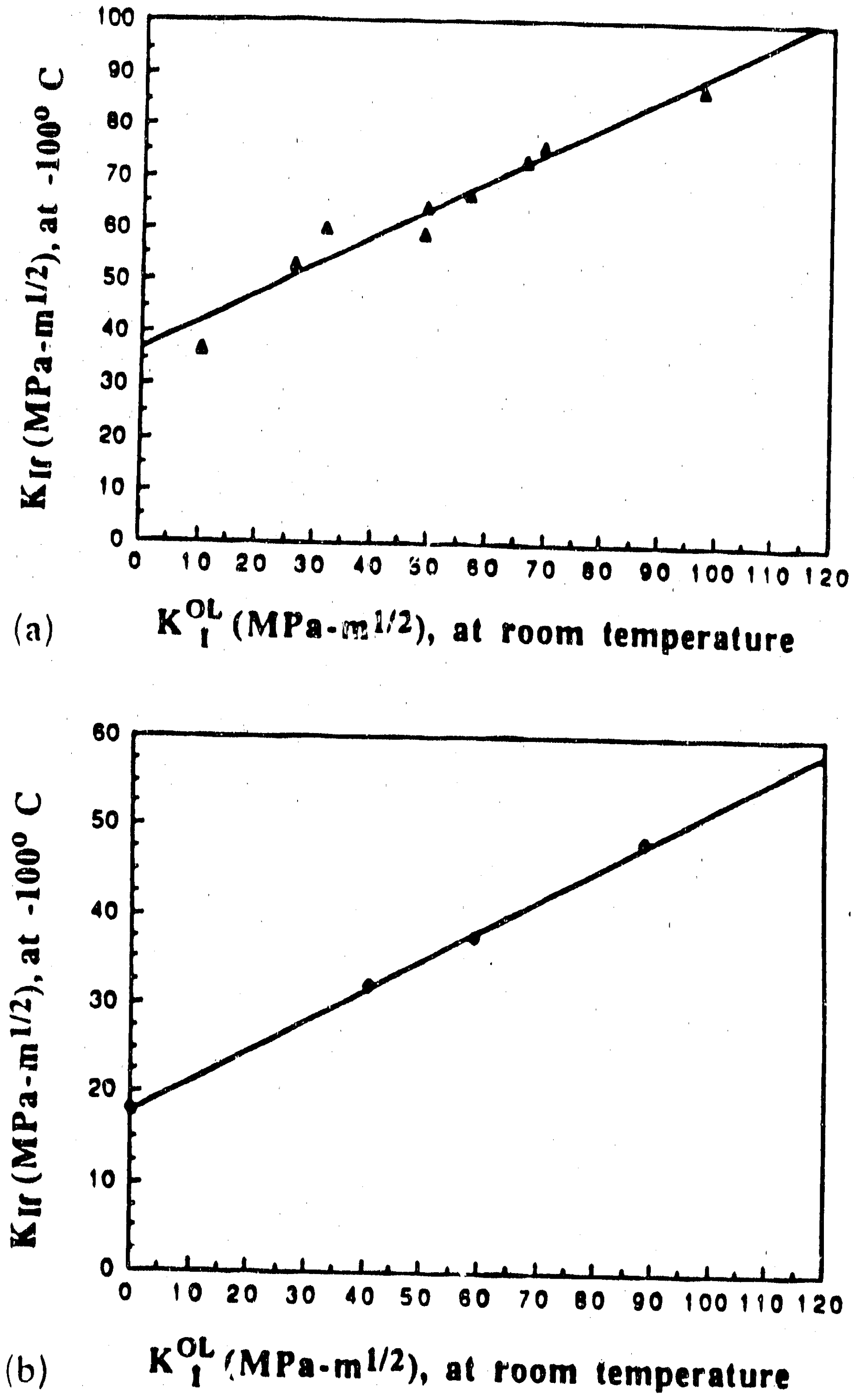


Figure 1. The effects of prestaining $K_{If}$ in low temperature tests on (a) HSLA steel and (b) Fe-3wt%Si single crystals.

5. M. Lii, T. Foecke, X. Chen, W. Zielinski and W.W. Gerberich, "The Effect of Low Energy Dislocation Structures on Crack Growth Onset in Brittle Crystals," *Mat. Sci. Engng.* **A113** (1989), pp. 327–338.

6. X. Chen, T. Foecke, M. Lii, Y. Katz and W.W. Gerberich, "The Role of Stress State on Hydrogen Cracking in Fe-Si Single Crystals," *Engng. Fract. Mech.* **35**, No. 6 (1990), pp. 997–1017.

7. W.W. Gerberich, T.J. Foecke and M. Lii, in Proc. 19th Canadian Fracture Conference on *Constitutive Laws of Plastic Deformation and Fracture* (1989), to be published.

8. M.J. Lii, X.-F. Chen, Y. Katz and W.W. Gerberich, "Dislocation Modeling and Acoustic Emission Observations of Alternating Ductile/Brittle Events in Fe-3wt%Si Crystals," accepted for publication, *Acta Met.* (1989).

2.3. Dislocation emission

Our early studies dealt with dislocation emission from grain boundaries and how this might influence fatigue substructure and fatigue life. This has more recently gravitated toward fundamental issues of dislocation emission at crack tips. Specifically, what type of character does dislocation emission have in model materials such as NaCl and how does this influence cleavage stability in single crystals? Much of this work is in progress this fiscal year. Some of the important accomplishments thus far are given below:

i) fatigue structure evolution can be followed at a surface with selected area channeling patterns (SACP);

ii) surface and bulk substructures are different in magnitude and morphology during fatigue;

iii) SACP observations assist in theoretical modeling of cyclic work-hardening behavior;

iv) anisotropic slip across interfaces at cracks or grain boundaries can be analyzed by electron channeling;

v) theoretical models of the stress intensity for dislocation emission $K_{Ie}$ predict values a factor of three higher than the extensive observations in {100} NaCl which demonstrate $K_{Ie}$ to be $0.067^{+0.003}_{-0.007}$ MPa-m$^{1/2}$;

vi) birefringence can be used for probing internal slip band emission from crack tips;

vii) as many as eight separate slip bands were found to emit from the tip of a crack in NaCl. These dislocations, being internal would not form surface etch pits, making some of the previous calculations by Narita, *et al.*[8] in this material questionable.

These and other observations have been published in the following:

9. W.W. Gerberich, J.K. Sheth and M. Kaczorowski, "Fatigue-Induced Nanoscale Patterns and Microstructures," in NATO Adv. Workshop on Pattern Defects and Microstructures in Nonequilibrium Systems, D. Walgraef, ed., NATO ASI Series E, No. 121, Martinus Nijhoff, The Netherlands (1987) pp. 237–256.

10. M. Kaczorowski and W.W. Gerberich, "The Degradation of Selected Area Channeling Patterns as a Function of Glide Anisotropy," *J. Mat. Sci.* **22** (1987) pp. 3227–3230.

11. J.K. Sheth and W.W. Gerberich, "The Effect of Test Frequency and Geometric Asperities on Crack Closure Mechanisms," in ASTM STP 982, J.C. Newman and W. Elber, eds., Philadelphia (1988) pp. 112–120.

12. T.J. Foecke and W.W. Gerberich, "Birefringence Observations of Dislocation Emission at Crack Tips in NaCl Single Crystals," *Scripta Metall.* **24** (1990), pp. 553–558.

### 2.4. Process zones

While our process zone efforts date back more than 10 years ago, recent direction has been toward the brittle fracture problem.(9) This approach was prompted by early acoustic emission events(10 in rising load tests which implied that microcracking was discontinuous when initiated from sharp cracks. This type of process was observed in the range of 130 and 193 K suggesting that weakest link approaches did not apply in the temperature range associated with the lower shelf.(10) This has been recently supported by Rogers and Clayton(11) on slightly different plain carbon steels. Although these test specimens were very thin, stable microcracking at 77 K did proceed prior to total instability. This and other evidence by Irwin(12) suggests that more detailed measurements of cleavage onset in many polycrystalline microstructures are needed. Some of that has been accomplished in this program with the following findings:

i) Acoustic emission detection of discontinuous microcrack events initiate at $K_I$ values as low as 15 MPa-m$^{1/2}$ at 193 K, even though $K_{Ic} \simeq 45$ MPa-m$^{1/2}$;

ii) process zone models are useful in modeling the microstructural details of a discontinuous brittle fracture process in a polycrystalline solid;

iii) based on the temperature dependence of yield stress, $\sigma_{ys(T,\dot{\epsilon})}$ and a proposed grain size dependence of fracture stress, $\sigma^*_{f(d)}$, the $K_{Ic}$ dependence on these variables are predicted. However, this was based upon one *ad hoc* assumption and three adjustable parameters;

iv) the geometrical arrangement and size distribution of grains was suggested to be the controlling process in HSLA steels rather than weakest link considerations;

v) discontinuous cleavage patches were identified and related to the magnitude of the stress intensity over and above that necessary to nucleate a coplanar crack.

These and other observations have been published in the following:

13. W.W. Gerberich, "Novel Techniques as Applied to Fracture Process Zone Theory," in NATO-Adv. Workshop on Chemistry and Physics of Fractures, R.M. Latanision and R.H. Jones, eds., NATO ASI Series E, No. 130, Martinus Nijhoff, The Netherlands (1987) pp. 419–436.

14. W.W. Gerberich, "Substructural and Microstructural Influence on Crack Closure," in *Fatigue '87*, R.O. Ritchie and E.A. Starke, Jr., Eds., E MAS, U.K. (1987).

15. W.W. Gerberich, S.-H. Chen, C.-S. Lee and T. Livne, "Brittle Fracture: Weakest Link or Process Zone Control?," *Met. Trans. A* **18A** (1987) pp. 1861–1875.

16. W.W. Gerberich, R.H. Jones, M.A. Friesel and A. Nozue, "Acoustic Emission Monitoring of Stress Corrosion Cracking," *Mat. Sci. Engng.* **A103** (1988) pp. 185–191.

17. R.H. Jones, M.A. Friesel and W.W. Gerberich, "Acoustic Emission from Intergranular Subcritical Crack Growth," *Metall. Trans. A* **20A** (1989) pp. 637-648.

18. W.W. Gerberich, "Metallurgical Aspects of Crack-Tip Failure Processes," ASTM STP 945, D.T. Read and R.P. Reed, eds., Am. Soc. Test. Mat'ls., Philadelphia (1988) pp. 5–18.

2.5. Proposed application of previous work

Initial models utilizing process zone concepts have been previously derived.[9,10] These are more macroscopic in nature since the local effects of stress and slip distribution are not considered. In these, there were *ad hoc* assumptions on cleavage fracture, no fundamental scale parameters, and no connectivity between single and polycrystalline behavior. More recently, we have made some progress in making that connection.[13] Applying such approaches to the DBTT phenomena, consider the cleavage process to evolve as follows:

- a grain of most favorable size and orientation cleaves at $K_{In}$, as related to some single crystal value (and appropriate constraints);
- at higher $K_I$ values, more grains cleave as the process zone, $\Delta$, grows in size;
- when the stress intensity associated with the sample resistance ($K_I^R$) is exceeded by the applied stress intensity, an instability occurs. That is, brittle fracture occurs when $dK_I/d\Delta = 0$.

Although this is not prepared for publication yet, it is illustrative to consider the original process zone concept as given by[14]

$$K_I \left(\frac{\pi}{c}\right)^{1/2} - 2\left\{\sigma_{ys} - \sigma_{sc}\right\} \cos^{-1}\left(\frac{c+\Delta}{c+\Delta+R_p}\right) - 2\sigma_{sc} \cos^{-1}\left(\frac{c}{c+\Delta+R_p}\right) = 0 \quad (1)$$

where the process zone strength, $\sigma_{sc}$, is given by $(1 - f_v^c)\sigma_{ys}$ for an elastic, perfectly plastic material with $f_v^c$ the microcrack volume fraction. The half-crack, $c$, is assumed to be stationary and $R_p$ is the plastic zone size. First, from previous studies,[9,10] the microcrack volume fraction is related to the ratio of applied stress intensity to the nucleation value for a coplanar cleavage plane, giving

$$f_v^c \simeq \frac{3}{\pi} \sec^{-1}\left(\frac{K_I}{K_{In}}\right)^{1/2} \quad ; \quad K_I \leq K_{In} < 4K_I \quad (2)$$

Then, solving for $dK_I/d\Delta = 0$ within two limits of $\Delta \ll c$ and $\Delta \sim c$ but with $R_p \ll c$, a first order estimate gives

$$\frac{K_{Ic}^2}{3\pi\sigma_{ys}^2} \simeq \frac{c\left[1 - \frac{3}{\pi} \sec^{-1}(K_{Ic}/K_{In})^{1/2}\right]}{\frac{3}{\pi} \sec^{-1}(K_{Ic}/K_{In})^{1/2}} \quad (3)$$

There is only one unknown here, $K_{In}$. However, this has a complicated dependence on temperature, grain size, etc. To make this connective to the single crystal observations, we use the same form as Eq. (1) to model some recent but very limited single crystal data in Figure 2. Here $A$ = 3.5 MPa-m$^{1/2}$ and $B$ = 1.8 × 10$^{-4}$ MPa-m$^{1/2}$/K. To fit polycrystalline data from Stonesifer and Armstrong,[15] an *ad hoc* assumption that the value for nucleating a cleavage microcrack in a polycrystalline array is related to the single crystal value is made. This is given by

$$K_{In_{(poly)}} \sim K_{In_{(single)}} \left(\frac{\ell s}{d}\right)^{1/4} \quad ; \quad d \leq \ell s \quad (4)$$

where $d$ is the grain size and $\ell s$ is a length scaling parameter taken as 450 $\mu$m. With Eqs. (2), (3) and (4), $\ell s$ is the adjustable parameter for the prediction of $K_{Ic}$ from Eq. (3). Using yield strength data as a function of temperature and grain size, the prediction of this data in Figure 3 is fair but underpredicts the temperature effect and overpredicts the grain size effect. Since the single crystal data were for Fe-3wt%Si and the polycrystalline data were A 533 B steel, the degree of fit is not surprising. It is encouraging but not definitive. In the remaining contractual period, a more definitive connectivity will be attempted with evaluations of Fe-Si polycrystals presently on hand.

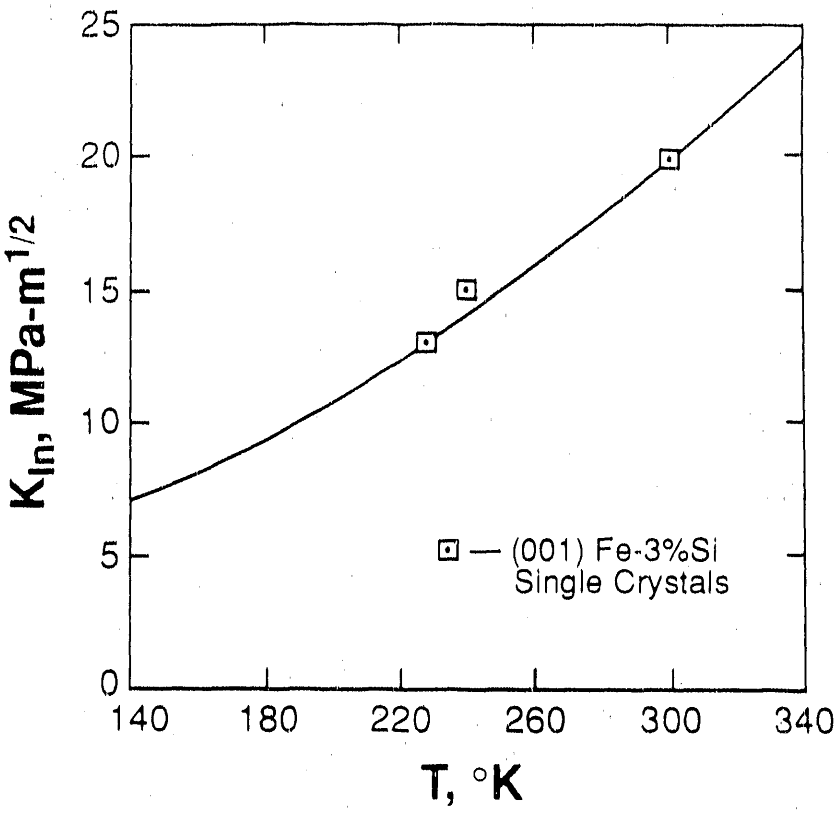


Figure 2. Fit of Eq. (1) to limited single crystal data. Note that the constants $A$ and $B$ differ from those for Figure 1.

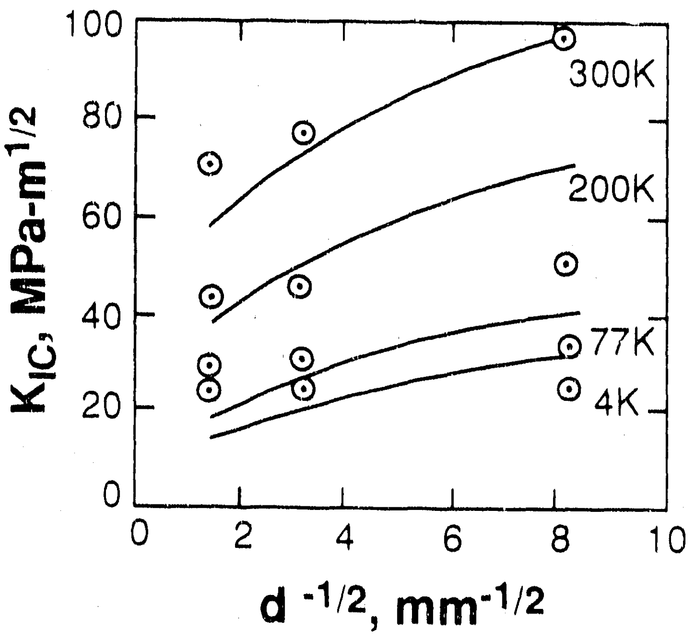


Figure 3. Zeroth order prediction (solid curves) of $K_{Ic}$ data[34] for polycrystals from single crystal cleavage nucleation data.

## 12. REFERENCES


1. H. Vehoff and W. Rothe, *Acta Metall.* **31** (1983) p. 1781.
2. C. Atkinson and D.L. Clements, *Acta Metall.* **21** (1973) p. 55.
3. J.R. Rice and R. Thomson, *Phil. Mag.* **29** (1974) p. 73.
4. B. Majumdar and S. Burns, *Int. J. Fract.* **21** (1983) p. 229.
5. V. Lakshmanah and J.C.M. Li, *Mater. Sci. and Engng.* **A104** (1988) p. 95.
6. A.S. Argon, *Acta Metall.* **35** (1987) p. 185.
7. M. Brede and P. Haasen, *Acta Metall.* **36** (1988) p. 2003.
8. N. Narita, K. Higashida and S. Kitano, *Scripta Metall.* **21** (1987) p. 1273.
9. W.W. Gerberich, ASTM STP 945, D.T. Read and D.P. Reed, eds., Philadelphia (1988) p. 5.
10. W.W. Gerberich, S.H. Chen, C.S. Lee and T. Livne, *Metall. Trans A* **18A** (1987) p. 1861.
11. C.B. Rogers and J.Q. Clayton, *Scripta Metall.* **23** (1989) p. 1785.
12. G.R. Irwin, private communication (1987).
13. M. Lii, T. Foecke, X. Chen, W. Zielinski and W.W. Gerberich, *Mater. Sci. and Engng.* **A113** (1989) p. 327.
14. W.W. Gerberich and N.R. Moody, ASTM STP 675, Philadelphia (1979) p. 292.
15. F.R. Stonesifer and R.W. Armstrong in *Fracture 1977* **2A**, D.M.R. Taplin, ed., Pergamon Press (1977) p. 1.

# -END-

# DATE FILMED

11 / 06 / 90