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BRITTLE TO DUCTILE TRANSITION IN CLEAVAGE FRACTURE

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NOTICE

I. INTRODUCTION AND REVIEW OF PREVIOUS RESEARCH

Many engineering materials with attractive potential for high temperature structural applications, including the b.c.c. transition metals, many intermetallic compounds, and covalent solids such as nitrides, carbides, etc. have more or less severe intrinsic problems of brittleness at low temperature manifested in cleavage fracture. While this imposes important restrictions on their application that must be considered in any design, what is particularly troublesome is the abrupt transition in fracture behavior from relatively ductile to very brittle, over a narrow temperature range (or strain rate range). The problem becomes more troublesome still, because the transition temperature can shift significantly up or down depending on many extrinsic effects such as deformation rate, triaxiality of stress state and the presence or absence of microstructural sources of perturbation, initiating the brittle behavior. Coping with these considerations successfully requires an improved level of basic understanding of the actual mechanism of the fracture transition rather than relying entirely on empirical approaches of suppressing the transition to lower temperatures by microstructure manipulations or compositional alterations.

The problem of fracture transition from brittle to ductile or vice-versa has been under consideration for a very long time and its interpretation has been mired in much detail that has obscured its fundamental aspects. The principal difficulties toward a better understanding have come from considering the transition as a ductile to brittle (D-B) transition in which, indeed, the basic mechanistic aspects of the phenomenon are masked in much microstructural detail. While this detail is often of technological importance since it permits operational suppression of the transition temperature, more success in fundamental understanding of the phenomenon can be had by considering it as a brittle to ductile (B-D) transition. This latter point of view was pioneered by Rice and Thomson (1974)(R&T) who have considered the fundamental process of the fracture transition as a crack tip initiated plasticity response where brittleness (or cleavability) in a crystalline material is the result of an energy barrier to the nucleation of dislocations from crack tips at impending motion, i.e., where the crack is subjected to a condition of $K_I = K_{Ic}$. In their first order linear analysis they have succeeded in differentiating intrinsically brittle (cleavable) solids from intrinsically ductile (non-cleavable) solids. Several key experimental studies on initially dislocation-free large Si crystals performed by St. John (1975), Brede and Haasen (1988) and Hirsch and co-workers (1989) have established an alternative rate controlling possibility as the rate of motion away from the crack tip of the nucleated dislocations, in solids where the dislocation mobility is particularly low.

Whether the process of crack tip initiated plastic response is actually controlled by the rate of nucleation of dislocations away from the crack tip, or the mobility of the created dislocations away from the crack-tip, the fracture transition is considered to be governed by the potential brittle propagation response of a sharp crack that fundamentally resides in the intrinsic brittle behavior, or the "cleavability" of the solid. To initiate the brittle response a sharp crack is essential. Thus, e.g. while α-Fe is an intrinsically brittle solid and will readily undergo cleavage fracture when surface imperfections or micro-cracks are present, α-Fe single crystals with smooth surfaces can be plastically deformed at 4.2°K as was demonstrated by Allen (1959). On the other hand intrinsically ductile (non-cleavable) solids containing sharp cracks can not be coaxed to exhibit cleavage-like brittle behavior under any physical condition of deformation - barring strong stress corrosion cracking agents. This is not to say that under certain conditions ductile behavior in intrinsically ductile solids can not be significantly curtailed. As was shown by the dynamic crack analysis of Freund and Hutchinson (1985), a propagating sharp crack with a dynamic plastic zone around it (not dwelling on the basic inconsistency of the assumption) will undergo a very significant reduction in the size of this plastic zone when the crack velocity reaches a significant fraction of the terminal velocity (0.1-0.3) of a brittle crack. This results when dislocation mobility in the plastic zone is in the phonon drag range and its external effect is a mere linear dependence of the plastic shear rate on shear stress. This, however, can not initiate a bifurcation of response if the solid is not cleavable and since cracks lack inertial properties (Eshelby, 1969), the effect is benign, and reversible, upon the reduction of crack velocity into the sub-sonic range.

While intrinsically ductile solids can not be coaxed into truly brittle behavior (barring the presence of strong stress corrosion cracking agents, or making them extensively microporous by particle irradiation, or other measures (Sieredski, 1989)), intrinsically brittle solids can be coaxed into ductile behavior, only to revert to brittle cleavage behavior upon the appropriate perturbation. The above mentioned ductile response of nearly perfect α-Fe single crystals at 4.2°K or similar behavior of W crystals at cryogenic temperatures in orientations not favoring twinning and twin intersections (Argon and Maloof 1966) are examples, as is the entire phenomenology of D-B transitions in low carbon steels (Hahn et al. 1959). As the extensive experiments of Hahn et al. on E-steel have demonstrated, coarse grain polycrystalline samples will exhibit ductile behavior even in the presence of mild notches if the local plastic strain rates are not too high. As the temperature is decreased, or the local plastic strain rate is increased by increasing the severity of notches, the ductile response can be terminated by brittle cleavage. The experiments of the above investigators have demonstrated that this bifurcation requires local perturbations in the form of microcrack production by one of the many means of inhomogeneous plastic flow such as by dislocation pile-ups, intersection of deformation twins, cracking of carbides or pearlite colonies. When these suddenly "injected" cleavage micro-cracks can not be arrested by the crack tip initiated plasticity processes discussed above, they will continue to propagate in a cleavage mode and result in brittle behavior. The conditions of successful brittle crack injection by sudden microcrack production has recently been studied experimentally by Lin, Evans and Ritchie (1987) in steels and modelled by Jokl et al. (1989). As discussed by Argon (1987), all the known phenomenology of the D-B transition, including, grain size effects, previous cold work, temperature, strain rate, etc. can be accounted for by the point of view that the fundamental ingredient of the bifurcation in response is the crack tip initiated processes of plasticity and their associated kinetics. Thus, it would appear that fundamental understanding of the transition in terms of intrinsic processes of crack tip initiated ductile response including the brittle to ductile transition temperature T_{BD} is governed by dislocation emission at the crack tip controlled either by dislocation nucleation (in solids with high dislocation mobility) or by dislocation mobility itself (in solids with very sluggish mobility). Therefore, in a large class of materials including cleavable metals (αFe, Cr, W, Mo, Ir, etc.) and many ionic solids and intermetallic compounds (NaCl, LiF, MgO, NiAl, etc.) where dislocation mobility is high the basic analysis of R&T (1974) should provide the fundamental framework for theoretical understanding of the transition. A large number of experimental studies summarized by Ohr (1985) and more recently by Argon (1987) have demonstrated that the initial linear analysis of R&T, while fully successful in differentiating cleavable solids from non-cleavable ones, gives unacceptably high values for the actual energy barriers for dislocation emission, making kinetically based estimates for the T_{BD} through its use somewhat meaningless. This discrepancy has been under scrutiny by a number of investigators. The important new break-throughs in the modeling of the B to D transition of cleavage fracture have come recently through a general recognition initiated by Argon (1987) that: (a) the saddle point configuration of the activated state of a dislocation loop at the crack tip is likely to have two important activation coordinates which are: the average radius, and the Burgers displacement, indicating that the saddle point loop is an imperfect dislocation (Argon, 1987, Rice, 1992, Rice et al 1992); (b) the nucleation of the loop occurs in nearly decohering material in which the elastic properties are very substantially reduced (Argon, 1987; Cheung et al, 1991, Rice et al, 1992); (c) as temperature increases, elastic properties, and hence, barrier energies are decreased further (Argon, 1987; Cheung et al, 1991); (d) work in forming the dislocation "loop" is extracted from a crack tip environment where the down portion of atomic interactions are dominant and where the emission of the "loop" results in substantial modification of the crack tip stress environment (Argon, 1988 unpublished). These considerations have been implemented in a large degree independently by Rice and co-workers (see Rice, 1992, Rice et al, 1992) and by Schoeck and Puschl (1991). They indicate that indeed the saddle point configuration consists of a dislocation "loop" in which the relative interatomic translation consists of a half core configuration of a Peierls dislocation.

On the experimental side, Argon (1987) has proposed that the definitive probe of the basic mechanism, whether it be of the R&T type or the mobility type, is to conduct experiments in which cleavage cracks in single crystals, propagated at various controlled velocities up a temperature gradient become arrested at a position where the local temperature equals the brittle-to-ductile transition temperature appropriate to that velocity. The special attraction of the experiment is its particularly simple interpretation and its direct relation to the fundamental mechanism that can be theoretically simulated by either direct atomistic approaches (Cheung and Yip (1991)) or a combination of atomistic approaches and dislocation mechanics (Cheung, Argon and Yip (1991), Schoeck and Puschl (1991)).

Such experiments have been successfully performed in the past (in the mid 50's) in the study of B-D transition of fracture in large steel plates where, however, the interpretations remained controversial because of the level of understanding of the phenomenon at that time. This experiment, as a specially instrumented tapered double cantilever beam (TDCB), has now been perfected under the expired DOE grant as a definitive tool in the study of the intrinsic mechanism in single crystalline samples and is described in the following section.

II. SUMMARY OF ACCOMPLISHMENTS

2.1 The Instrumented Tapered Double Cantilever Beam (TDCB)

Since brittle cleavage cracks have no significant inertial properties (Eshelby (1969)), the arrest of cleavage cracks running at constant velocity under quasi-static equilibrium conditions up a temperature gradient provides ideal conditions for the exploration of the mechanism of brittle to ductile (B-D) transition. At the point of arrest the time of residence of the high stress environment of the cleavage crack tip at any material point that it samples becomes equal to the time constant of the critical process of fracture transition, governed by the local temperature. Higher velocity cracks, subjecting material points to the same high stress for shorter periods of time will be arrested at higher temperatures where the time constant of the critical process is correspondingly shorter. The simple theoretical model of Argon (1987) gives the dependence of the B-D transition temperature T_{BD} on crack velocity v as

$$T_{BD} = \frac{\Delta G}{k}\frac{1}{ln(c/v)} \tag{1}$$

where c is the velocity of a shear wave and ΔG, the slope of the relationship, gives directly the activation free energy of the critical process of arrest in the traveling crack tip environment.

In the experiment performed in a system illustrated in Figure 1 the TDCB specimen having a constant K factor shape with a temperature gradient is pried open quasi-statically by the actuator of the testing machine. Under ideal conditions over the constant K_I range of the sample, if machine perturbations are eliminated, and material perturbations are damped, the crack velocity is directly proportional to the actuator velocity of the testing machine. In the experiment a real image of the bright edges of the crack is formed on the screen of a position sensitive detector, by a well-collimated laser optical system. The changing intensity of the bright image on a dark background is recorded to give information on the crack front velocity. In the experiments performed on TDCB shaped Si single crystals, oriented for cleavage on either the {111} or the {110} planes, a number of troubling features of jerky crack extension were encountered.

Through analysis of both the quasi-static and dynamic response of the entire loading system, including specimen, loading elements, and the servo-hydraulic components of the machine it was established that the extrinsic perturbations can be nearly completely eliminated by careful tuning of the equipment and scrupulous attention to detail on specimen shape to achieve a true constant K_I condition. Material perturbations, however, have presented special challenges. It was established that the observed jerky extensions in the form of crack accelerations to a few percent of the sound velocity followed by equally sudden

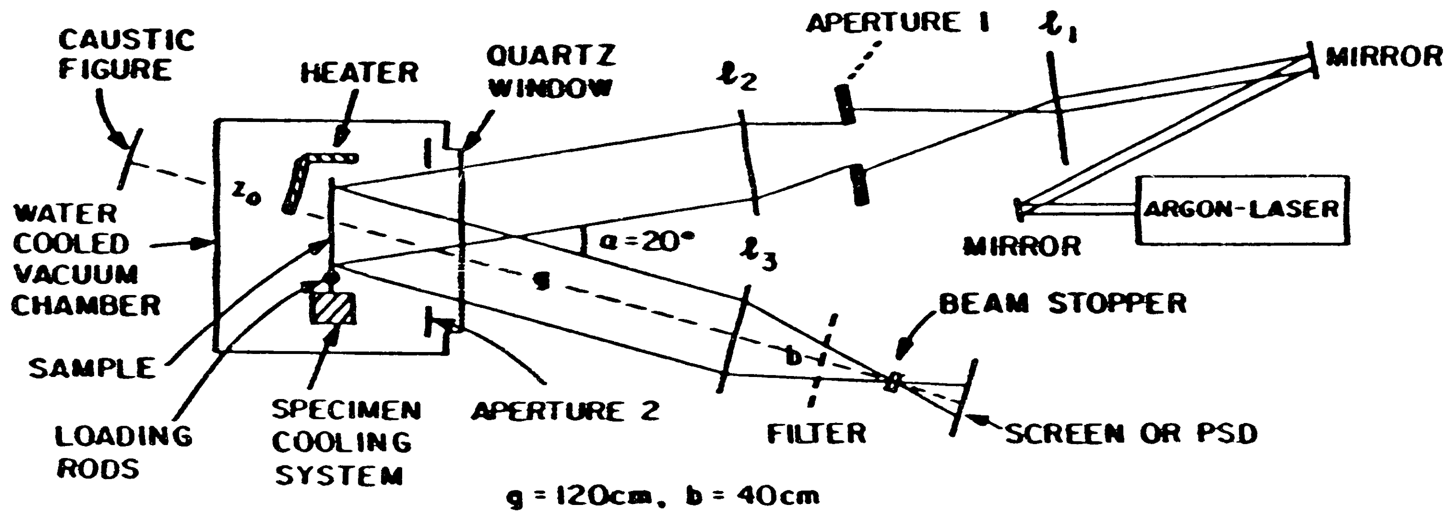

Figure 1 Experimental set-up for the TDCB experiment.

decreases to zero velocity, repeating quasi-periodically, are triggered by small changes in cleavage fracture work along the length of the extending crack. The major perturbation of this type occurs in starting up the crack propagation from a starter crack which usually has a rougher surface requiring a slight excess in K_I than what is required for further propagation. These troublesome perturbations have been contained by applying to the two side surfaces of the specimen micron thick ductile platinum coatings by sputter deposition. These coatings which remain reflective at elevated temperature were found to impart enough parallel energy dissipation to the propagating crack to substantially eliminate the troubling quasi-periodic accelerations and decelerations without affecting the eventual fracture transition which is a bulk feature.

A new generation of experiments were in progress on Si single crystals at the time of the funding cut-off, exploring the critical crack arrest processes on both the $\{111\}$ and $\{110\}$ cleavage planes in the $< 110 >$ direction. In the latter geometry, ideal conditions exist to nucleate or propagate dislocations on slip planes that are oblique with respect to the crack front on which slip activity is favored. In addition cleavage cracks can also be propagated on the $\{110\}$ planes in the $< 001 >$ direction where plasticity on the $\{111\}$ planes in the blunting mode can be initiated. Here, however, care is required in designing the specimen shape to avoid breaking off the cantilever arms by cleavage.

In Si the evidence suggests that nucleation of dislocation loops from the crack tip is easier than moving these dislocations away from the crack tip. Therefore, it is expected that examination of the fracture surfaces by x-ray topographic imaging techniques as well as by etch pitting should show evidence of initiation of such loops at lower temperatures than the temperature where the eventual arrest occurs. Preliminary etch pit studies in Si support this possibility. To obtain additional verification and to actually map out the 3-D arrangement of these zones of emitted dislocations, Berg-Barrett x-ray topographic studies had been begun at the termination of the program, utilizing a newly acquired Rigaku topographic camera.

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13. G. Schoeck and W. Puschl, *Phil. Mag.*, **64**, 931 (1991).

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2.2 Publications

Publications that have resulted since the beginning of the research program are:

1. A. S. Argon, *Acta Metall.*, **35**, 185 (1987).
2. M. Brede, K. J. Hsia and A. S. Argon, *J. Appl. Phys.*, **70**, 758, (1991).
3. K. S. Cheung, A. S. Argon and S. Yip, *J. Appl. Physics*, **69** 2088 (1991).
4. K.J. Hsia and A.S. Argon, "Experimental Study of Micromechanisms of Brittle to Ductile Transition in Si Single Crystals," to be presented at the 8th International Fracture Conference in Kiev, in April 1993.

Copies of these publications are attached as Appendix I.

2.3 Oral Presentations

1. "Brittle to Ductile Transitions in Cleavage Fracture", TMS-AIME Spring Meeting in Denver, Colorado, February, 1987.

2. "Crack Tip Initiated Plasticity in Cleavage Fracture", Seminar in Materials Science, U. Pennsylvania, Philadelphia, PA, April 1987.

3. "Brittle to Ductile Transitions in Fracture", Seminar in Engineering Mechanics, Brown University, Providence, R. I., April 1987.

4. "Mechanism of Brittle to Ductile Transition of Cleavage Fracture", Center for Materials Science Seminar, Los Alamos National Laboratory, Los Alamos, NM, August 1987.

2.4 Personnel Associated with the Program

1. Dr. Markus Brede, post doctoral associate, April 1987 to July 1989.
2. Dr. Kuen J. Hsia, post doctoral associate, August 1989 to July 1991.

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