DOE/ER/45051--3

DE91 001305

Fracture Toughness of Materials

Final Report

S.J. Burns
Materials Science Program
Department of Mechanical Engineering
University of Rochester
Rochester, NY 14627
716-275-4082

February 1984 - September 1988

Prepared for the U.S. Department of Energy
Under Grant No. DEFG02 - 84ER45051

DOE/84ER45051
Final Report

September 1990

ABSTRACT

Crack tip dislocation emission in bulk specimens have been measured in single crystal specimens and the measurements are well below the accepted theoretical values for dislocation emission. The image forces on a dislocation due to the presence of a semi-infinite crack are used to calculate the potential energy of the dislocation around the crack. Expressions for the radial and tangential forces and for slip and climb forces have been found.

Crack tip deformation in Mode I and Mode II fractures on both {100} and {110} planes have been observed in crystals of LiF. The deformation is shown to nearly completely shield {110} plane cracks and prevent their propagation while the deformation is less effective in shielding {100} plane cracks.

The fracture toughness of MgO-partially - stabilized ZrO_2 exhibiting transformation toughening been measured. The equations of linear elastic fracture mechanics have been self-consistantly formulated to include the residual displacement from the transformation wake. The initial toughness values may be as high as 25 MPa $\sqrt{m}$.

MgO single crystals were fatigued in plastic strain control at elevated temperatures. At high temperatures, dense bundles of dislocations were observed in transmission electron microscopy aligned perpendicular to the Burgers' vector directions. The deformation has some similarities with fatigue deformation FCC metals.

The isothermal Young's modulus of the polycrystalline superconductor Y-Ba-Cu-O was measured. This value is 238 GPa while Poisson's ratio is .285. These values are sensitative to porosity.

The thermodynamics of a superconducting second order phase transformation has been related to jumps in physical properties. A simple energy balance, without assuming an equation of state, is used to relate the rate of change of state variables to measurable physical properties. There are no preconceived assumptions about the superconducting mechanism.

INTRODUCTION

The sources of fracture toughness and the relationship of fracture toughness to microstructure in materials are not well understood. This grant concentrated on the dislocation emission from crack tips in single crystals. These dislocations provide the plasticity for ductile fracture toughness. The brittle-to-ductile transition in steels is the difference between a ductile crack and brittle crack. The ability of a crack to emit dislocations and the understanding of the emission process are thought to be related to the concept of the more practical brittle to ductile transition in steels. The attached papers provides direct evidence that experiments on dislocation emission from brittle cracks in bulk samples are not in agreement with present theories for dislocation crack emission. The present theories provide neither the correct temperature nor the correct rate dependence for fracture ductility.

This grant also studied the fracture behavior of partially-stabilized-zirconia oxide. The results are in three separate publications: The paper of fracture in MgO/ZrO_2, J. Amer. Ceramic Society, and the Ph.D. thesis by R.J. Seyler and G.C-S Chang. The major findings are discussed in the next sections and deal more with material properties rather than fracture behavior. The properties of PSZ-ZrO_2 is of course completely determined by the ability of this material to have a martensitic phase transformation from the metastable tetragonal phase, rich in the stabilizer, to the monoclinic low temperature phase.

Finally, this grant was in effect when the ceramic superconductors were first discovered and several papers were published in this area. The first paper was on measurements of polycrystalline elastic constants. The technique used was Hertz compression. This loading scheme is nearly completely in compression and should therefore minimize the effects of cracking and porosity. The phase transition for the superconductors from the superconductor to the normal state is the "classic" example of a second-order phase transition. Thermodynamic energy balances for pressure and temperature as independent state variables are well known in the thermodynamic literature. The DOE supported work however is broader in that a general thermodynamic system is considered with no assumption about the mechanism of the superconductivity but rather leaving measurable thermodynamic properties to specify the state variables that are important. This thermodynamic approach is unique in the literature. It is also the only systematic approach to specifying the thermodynamic variables that contribute to the onset of superconductivity.

The patent disclosure for combining both ceramic superconductor with a ductile metal phase is attached. DOE correspondence is also given. This process of using a cermet = ceramic plus metal for this new class of superconductors was first done with some (partial) DOE funds i.e. for the student support.

TECHNICAL DETAILS

Crack Tip Deformation and Dislocation Emission:

Dislocation-free zones have been observed at crack tip regions for two deformation modes in LiF single crystals. These two deformation modes involve different dislocations and slip characteristics on the plane stress surface of a plane strain crack. This implies that the formation of dislocation-free zones exists for many types of dislocations and slip systems. The shapes of crack tip dislocation pile-ups for the jogging dislocations are similar to the shapes of the crack front. This implies that the crack tips are the dislocation sources.

The dislocation density distribution ahead of a crack tip is quite different from the predictions of BCS theory. The maximum density is not right at the crack tip but away from the crack tip. The shapes of crack tip dislocation pile-ups for the jogging dislocations are similar to the shapes of the crack front. This implies that the crack tip is again the dislocation source.

Since {100} planes of ionic crystals can be cleaved easily with a sharp knife and an impulsive force, it is commonly recognized that {100} planes of ionic crystals have the lowest fracture surface energy among its crystallographic planes. However {110} cracks are generated before {100} cracks during indentation testing. A dislocation pile-up mechanism for the initiation of {110} cracks may answer why {110} cracks are not initiated by the same dislocation pile-ups. The {110} crack tip is either completely shielded or partially shielded by the associated crack tip dislocations. In addition, the {110} crack tip is also blunted by dislocations whose Burgers' vectors have normal components to the crack plane. These two factors make the propagation of {110} crack very difficult, and the fracture reverts to a {100} cleavage plane.

Mechanical unloading also contributes to deformation in the crack tip region and propagates the crack front. This result, which may be useful in fatigue crack growth, shows additional deformation during unloading. It might be important in understanding unloading surface asperities and in fatigue fracture thresholds. A simple but easy-to-understand model which explains deformation during unloading has been proposed in our work.

The brittle to ductile fracture transition is an important material property that is poorly understood. Materials are often subject to service applications near the fracture transition temperature so this property has practical importance. In addition, chemical and temperature effects can give fracture transitions. This fracture transition is controlled, at least in part, by the ability of a crack tip to exhibit local ductility. Crack tip dislocation nucleation or generation of dislocations is a necessary condition for crack tip ductility. Engineering, fracture tough, materials must, of course, have extensive crack tip ductility. Our papers review some of the background concepts in dislocation crack tip nucleation and provides quantitative observations of etched dislocations in cracked single crystals of LiF. In general, however the analytic expressions for dislocation emission over estimate the stresses necessary for the onset of plasticity associated with fracture. The implication is that the crack tip stresses with atomic dimensions is not very well represented by linear elastic stress fields.

Fracture Toughness in Mg0-Partially-Stabilized ZrO_2

The fracture toughness of transformation toughening, ZrO_2 can be increased by an order of magnitude over ZrO_2 without tetragonal precipates. The mechanism of enhanced toughness have been proposed but are basically the work of Evans and McMeeking.. The work in ZrO_2 under this grant has concentrated on measuring the fracture toughness PSZ. The major result is that simple linear elastic fracture mechanics is not directly applicable to the fracture samples tested.

The equations of linear elastic fracture mechanics have been modified so the residual stresses introduced by the tetragonal to monoclinic transformation as observed on the load-displacement curve are accounted in a self-consistent way. The modification accounts for the residual load-point displacement offset that is observed in transformation-toughened zirconia alloys. The modification to linear elastic fracture mechanics is a crack growth dependent value, η, that can be determined experimentally by full or partial unloading during a crack growth experiment. The η values for extensive crack growth are about 20%; for initial crack growth the values are very large with 100% corrections not being uncommon.

The K_R or crack growth curve is totally changed by the η correction. The K_R curve in the samples tested shows that K decreases as the crack extends. This trend will have serious implications: first, it implies that wake effects are not as important as previously considered since the fracture toughness is largest when the crack just starts to propagate in the Mg-PSZ investigated. This particular fracture sample has been heat-treated so the martensitic start transformation temperature is just slightly below room temperature. The effects of transformation toughening are greatly exaggerated by having very large transformation zones and large fracture toughness at room temperature. With a decreasing K_R curve it seems reasonable to expect that crack tip blunting or local deformation may be source of some of the large toughness values reported here.

It should also be noted that toughness values reported for zirconia alloys might be very sensitive to the type and size of the fracture specimen used. The equations of LEFM are most frequently applied without correcting for residual load-point displacement offsets and without verifying that linear elastic fracture mechanics actually describes the specimen tested.

Superconducting Phase Transformations in High T_c Ceramics

Thermodynamics has formed the basis for understanding conventional low temperature superconductors. The transition to a superconducting state from a normal state contains information on all physical process that contribute to the superconductivity. Thermodynamics has contributed to the physical understanding of superconductivity in the new high-T_c superconductors. In a superconducting state, charge carrier pairs transport supercurrents. These paired carriers are ordered, and therefore reduce the entropy of the superconducting state, as compared to that of the normal state extended across the normal-superconducting phase boundary. The number of charge carrier pairs at the superconducting transition temperature goes to zero, hence the entropy across the phase boundary is continuous, and the phase transformation is necessarily second-order. Physically, these paired charge carriers can only be formed as a result of some mediating interaction within the superconducting crystal. In the metallic superconductors this interaction is provided by the lattice, as described by the BCS theory. In the ceramic or high-T_c superconductors there are many proposals for microscopic interaction mechanisms.

In the paper: Thermodynamics of the Superconducting Phase Transformation in High Tc Ceramics with Magneto-electric effects, I have assumed a second order phase transformation. A second order phase transformation in a dielectric ceramic superconductor is then shown to relate jumps in physical properties to the ratio of state variables, i.e., to the phase boundary, near the critical point. The major jumps in specific heat, compressibility, permittivity, permeability, and spinodal composition curvature, between the normal and superconducting phases, are found from the continuity of the entropy, volume, electromagnetic fields (with zero fields), and chemical equilibrium. The thermal expansion, pyroelectric effects, and the piezoelectromagnetic effects are important differences between ceramic and metallic superconductors. The most important conclusions from experimental measurements are that the lattice plays a minor role in the superconductor transformation while the magnetoelectric jump may be related to the jump in permeability, i.e., the Meissner effect, and the jump in permittivity. The oxygen miscibility gap which controls order / disorder transformations thermodynamically mandate that only metastable compositions are obtained in the metal oxide, so absolute stability of the system may never be achieved. An explicit criterion to suggest other superconductor systems is given from magnetoelectric materials, i.e., some of the pervoskities.

PATENT DISCLOSURE

The powder metallurgy processing of the ceramic superconductors is one method to make a tough, non-brittle ceramic, superconducting wire. DOE through the support of GCS Chang supported some of the original work in this area. It was demonstrated that cermets could be made (Cerment = Ceramic plus Metal) by using powder metal processing. We processed using liquid phase sintering and conventional powder metallurgy methods for the cermets and demonstrated that we had made superconductors with a wide selection of processing routes. The following copies of correspondence shows the disposition of this patent disclosure.

pc. A. Hammersla

Department of Energy
Chicago Operations Office
9800 South Cass Avenue
Argonne, Illinois 60439

May 10, 1988

Ms. Margit Iwanowicz
Research Administrator
Office of Research & Project
  Administration
518 Hylan Building
University of Rochester
Rochester, NY 14627

Dear Ms. Iwanowicz:

SUBJECT: DOE CASE NO. S-67,981 - CONTRACT NO. DE-FG02-84ER45051 (Burns)
"SUPERCONDUCTING CERMETS FABRICATED BY POWDERED-
MATERIALS TECHNIQUE"

This will acknowledge and thank you for the invention disclosure which was sent to us with your letter of disclosure dated April 28, 1988. We have assigned the above case number to this item. Please refer to the above number in all future correspondence. Our telephone number is 312-972-2165.

Very truly yours,

Arthur A. Churm

Arthur A. Churm, Chief
Office of Patent Counsel

UNIVERSITY OF
ROCHESTER

OFFICE OF RESEARCH & PROJECT ADMINISTRATION

November 22, 1989

Mr. Paul Gottlieb, Chief
Office of Patent Counsel
U. S. Department of Energy
Chicago Operations Office
9800 South Cass Avenue
Argonne, IL 60439

RE: Grant No. DE-FG02-84ER45051
Your Case No. 267981

Dear Mr. Gottlieb:

On April 25, 1988 we disclosed an invention made under the referenced grant. This letter is to advise you that it is not the University's intention to seek patent protection. It is our understanding that both publication and other issued patents preclude such protection.

Please feel free to contact me should you have any questions on this matter.

Sincerely,

Jay

Jane A. Youngers
Director

pw

518 Hylan Building
University of Rochester
Rochester, New York 14627
(716) 275-5373
Fax: (716) 275-9492

ORAL PRESENTATIONS

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Ph.D THESIS

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1990 C-S Chang, Thesis title: Micromechanism of Transformation and Toughening in Magnesia Partially Stabilized Zirconia".

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