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MICROMECHANISMS OF BRITTLE FRACTURE: STM, TEM AND ELECTRON CHANNELING ANALYSIS

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TABLE OF CONTENTS

Pages

- I. INTRODUCTION
- II. FUNDING HISTORY AND REVIEW OF ACCOMPLISHMENTS
- III. RECENT WORK
- IV. APPENDICES
- Quasi-Equilibrium Modeling of the Toughness Transition During Semi-Brittle Cleavage
 - Cleavage

 | UNIV.2. Fracture of Semi-Brittle U-Ti Polycrystals Shielding Model Extension to a

 Lower Crystal Symmetry
 - YOUNIV. 3. Fracture Toughness of Intermetallics Using a Micro-Mechanical Probe
 - Continuous Microscratch Measurements of the Practical and True Works of Adhesion for Metal/Ceramic Systems
- WWW.5. A Micro-Mechanical Toughness Test Applied to NiAl

I. INTRODUCTION

The original thrust of this grant was to apply several newly developed techniques in scanning tunneling and transmission electron microscopy to elucidate the mechanism of brittle fracture. This grant spun-off several new directions in that some of the findings on bulk structural materials could be utilized on thin films or intermetallic single crystals. For example, utilizing electron channeling to evaluate the strain gradients around crack tips later became useful to evaluate a substrate contribution to nonlinear energy dissipation about a scratch track for thin-film delamination.

Modeling and material evaluation efforts in this grant are represented in Figure 1. Out of this grant evolved the field we have designated as Contact Fracture Mechanics. By appropriate modeling of stress and strain distribution fields around normal indentations or scratch tracks, various measures of thin film fracture or decohesion and brittle fracture of low ductility intermetallics is possible. These measures of fracture resistance in small volumes are still evolving and as such no standard technique or analysis has been uniformly accepted. For brittle ceramics and ceramic films, there are a number of acceptable analyses such as those published by Lawn, Evans and Hutchinson. For more dissipative systems involving metallic or polymeric films and/or substrates, there is still much to be accomplished as can be surmised from some of the findings in the present grant.

In Section II we review the funding history and accomplishments associated mostly with bulk brittle fracture. This is followed by Section III which covers more recent work on using novel techniques to evaluate fracture in low ductility single crystals or thin films using micromechanical probes. Basically Section III outlines how the recent work fits in with the goals of defining contact fracture mechanics and gives an overview of how the several examples in Section IV (the Appendices) fit into this framework.

II. FUNDING HISTORY AND REVIEW OF ACCOMPLISHMENTS

II.1. Funding History (Bulk Fracture)

The origins of this work, initiated with funding in 1984, were predominantly associated with fatigue studies where it was noted that a combination of low temperature and cyclic loading produced cyclic cleavage in BCC iron-base systems. To gain a fundamental understanding of these processes, it was decided that it was necessary to probe both the dislocation dynamics and quasi-statics of crack growth processes. In the first three years, this led us to investigations of both quasi-static and dynamic cleavage of poly- and single crystals of iron and titanium base systems. Approximately 15 papers were published, the two most significant being:

W.W. Gerberich and E. Kurman, "New Contributions to the Effective Surface Energy of Cleavage," *Scripta Metall.* 19 (1985) p. 295.

W.W. Gerberich, T. Livne and X. Chen, "A Transient Model for Sub-Critical Cracking in BCC Alloys," in *Modeling Environmental Effects on Crack Growth Processes*, R.H. Jones and W.W. Gerberich, eds., TMS-AIME, Warrendale, PA (1986) pp. 243–258.

The first of these led us to a concentrated effort in studying ligament effects in process zones, particularly associated with the ductile-to-brittle transition (DBTT) in steels. The

Modeling Materials Atomistic Models of Thin Film Interfaces and Decohesion Contacts CONTACT Dislocation Thin Film FRACTURE Quasi-Statics Brittle MECHANICS and Fracture Kinetics AFM Continuum Bulk STM Mechanics of SEM Crystal Singularities SACP Brittle in TEM Fracture Elastic-Plastic Nanoindentation i Media Continuous Microscratch

Figure 1: Schematic of a field of study called contact fracture mechanics.

second of these, as a consequence of introducing sharp cracks into Fe-3wt%Si single crystal by hydrogen-assisted fatigue, led us to the discovery that both sustained-load cracking and very definitive crack arrest markings (CAM) could also be produced. This inaugurated a concentrated 6-year effort in quantifying both the sub-microstructural and macroscopic aspects of hydrogen embrittlement by decohesion along {100}(011) crack planes and localized crack growth directions. These two areas are summarized below. As discussed subsequently, a new area dealing with the mechanical behavior of thin films was initiated in 1990.

II.2. Accomplishments '87-'95

Over forty publications resulted from this research time frame, some of which are listed below. A number of major accomplishments dealt 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.

- 1. W.W. Gerberich, "The Micromechanics and Kinetics of Environmentally Induced Failures," ASM Materials Science Seminar, 1987, S.V. Nair, J.K. Tien, R.C. Bates and O. Buck, eds., ASM International, Metals Park, OH (1989) pp. 201–228.
- 2. 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.
- 3. 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.
- 4. 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.
- 5. 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.
- T.J. Foecke and W.W. Gerberich, "Birefringence Observations of Dislocation Emissions at Crack Tips in NaCl Single Crystals," Scripta Met. et Mat. 24 (1990) pp. 553-558.
- 7. M.-J. Lii, X.-F. Chen, Y. Katz and W.W. Gerberich, "Dislocation Modeling and Acoustic Emission Observation of Alternating Ductile/Brittle Events in Fe-3wt%Si Crystals," *Acta Metall.* 38 (1990) pp. 2435–2452.
- 8. S.-H. Chen, Y. Katz and W.W. Gerberich, "Crack Tip Strain Fields and Fracture Microplasticity in Hydrogen Induced Cracking of Fe-3wt%Si Single Crystals," *Philos. Mag. A* 63, No. 1 (1990) pp. 131–155.
- 9. X. Chen and W.W. Gerberich, "The Kinetics and Micromechanics of Hydrogen Assisted Cracking in Fe-3wt% Single Crystals," *Metall. Trans. A* **22A** (1991) pp. 59-70.
- W.W. Gerberich, H. Huang, W. Zielinski and P.G. Marsh, "A Dislocation Shielding Prediction of the Toughness Transition during Cleavage of Semibrittle Crystals," Metall. Trans. 24A (1993) pp. 535-543.

- W.W. Gerberich, P. Marsh, J. Hoehn, S. Venkataraman and H. Huang, "Hydrogen/Plasticity Interactions in Stress Corrosion Cracking," in Corrosion-Deformation Interactions, CD1 '92, T. Magnin, ed., Les Editions de Physique Les Ulis, France (1993) pp. 325-343.
- 12. J.W. Hoehn, S.K. Venkataraman, H. Huang and W.W. Gerberich, "A Micro-Mechanical Toughness Test Applied to NiAl," *Mat'ls Sci. and Engng.* A192/193 (1995) pp. 301-308.
- 13. W.W. Gerberich, P.G. Marsh and J.W. Hoehn, "Hydrogen Induced Cracking Mechanisms Are There Critical Experiments," in 5th Intern. Conf. on the Effects of Hydrogen on Material Behavior, N.R. Moody and A.W. Thompson, eds., TMS-AIME (1995).

From these investigations, the following can be summarized:

- i) hydrogen cracking above threshold is discontinuous with 1 μ m steps involved as verified by fractography and acoustic emission;
- ii) 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;
- iii) 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;
- iv) for applied stress intensities as low as 2 MPa-m^{1/2}, near theoretical stresses (20 GPa) were reached at the crack tip;
- v) theoretical models of the stress intensity for dislocation emission K_{Ie} predicted values a factor of three higher than the extensive observations in {100} NaCl which demonstrated K_{Ie} to be $0.067^{+0.003}_{-0.007}$ MPa-m^{1/2};
- vi) Acoustic emission detection of discontinuous microcrack events in HSLA steel 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};
- vii) process zone models are useful in modeling the microstructural details of a discontinuous brittle fracture process in a polycrystalline solid;
- viii) a strong plane-stress versus plane-strain effect existed for hydrogen-induced slow crack growth in Fe-3wt%Si as reflected by both crack tunneling and the local plastic strain distributions;
- ix) crack velocity observations as a function of test temperature were consistent with a kinetic model based upon hydrogen transport, collection and decohesion.
- x) a discretized dislocation simulation allowed a local stress analysis which could be used to understand initiation, arrest and reinitiation events on the {100} cleavage plane in local (011) directions of Fe-3wt%Si single crystals;
- xi) a simplified dislocation shielding model can represent the low temperature fracture toughness of mild steel as a function of grain size and test temperature in the lower shelf regime;

- xii) dislocation shielding models are also applicable to intermetallics like NiAl as can be shown from bulk fracture toughness and in situ TEM experiments.
- II.3. Previous Students and Colleagues
- X.-F. Chen (PhD 1989) First PhD student to graduate on this grant dealt with the kinetic aspects of hydrogen-induced sustained-load cracking in Fe-3wt%Si single crystals. Is currently at Singapore Institute of Standards and Industrial Research.
- T. Livne (1985–86) Visiting researcher dealt with macroscopic fracture mechanics and acoustic emission of discontinuous cleavage. She is currently at RAFAEL in Haifa, Israel.
- C.-S. Lee (1986-87) Post-doctoral associate investigated crystallographic aspects of the process zone. He is currently a Professor at Postfach in South Korea organizing a research program on the mechanical behavior of titanium alloys.
- J.K. Sheth (MS 1986) Investigated cyclic-cleavage fatigue crack growth in Fe-3wt%Si single crystals. Returned to Bombay to manage his family business.
- B. Wilfahrt (MS 1990) Analyzed liquid metal embrittlement of brazed joints. Brian did this fracture mechanics study in conjunction with industry (salary support). Part of this led to ideas on how stacking fault energy may play a role in several embrittlement mechanisms.
- S.-H. Chen (PhD 1990) Did the first analysis of state of stress effects on the plastic strain gradient very near crack tips using selected area channeling methods. She is currently in charge of failure analysis on a number of projects at INTEL Corporation in Chandler, Arizona.
- T.J. Foecke (PhD 1991) Analyzed cracks in silicon with scanning tunneling microscopy and dislocation emission in alkali halides. Tim won an NRC Fellowship and was two years with Robb Thomson and Dave Lashmore at NIST performing in situ TEM experiments on multilayer structures. He is currently a staff member in charge of a program on the mechanical properties of multilayers.
- W. Zielinski (MS 1991) Transmission electron microscopy of crack-tip dislocation arrangements returned to Warsaw in November, 1992 and is working on a PhD.
- Y. Katz (1988-89) Visiting professor from Ben Gurion University and the Nuclear Research Establishment, Beer Sheva, Israel. Was responsible for many of the ideas associated with the dynamics of crack initiation and arrest in semi-brittle fracture.
- J.W. Hoehn (PhD 1996) Responsible for examining corrosion/deformation interactions under both fatigue and fracture loadings. Developed in situ TEM and SEM techniques for following dislocation arrays and crack growth rate in NiAl and Fe-3wt%Si single crystals.

A. Wright (1994-95) Post-doctoral associate analyzed nonlinear deformation under a bimaterial interface utilizing finite element analysis (ABAQUS).

III. RECENT WORK

More recently, the computational work has emphasized the arrangements of shielding dislocations at crack tip emphasized in a published paper[†] "Quasi-Equilibrium Modeling of the Toughness Transition During Semi-Brittle Cleavage" as reproduced in Appendix IV.1. This addresses the box designated as dislocation quasi-statics and kinetics in Figure 1. This essentially demonstrates that you could predict the fracture toughness of a broad group of aluminides from knowing the true Griffith energy and the flow strength of the crystals. This was further demonstrated by applying the dislocation model to a series of semi-brittle crystals of lower crystal symmetry evaluated over a temperature range of 80 to 400° K and a strain rate regime ranging from 10^{-3} to 10^{-1} s⁻¹. This is shown in Appendix IV.2 where K_{Ic} , plane strain fracture toughness was predicted within about 10 percent.

Carry this approach over to the micromechanical probing of small volumes; a contact fracture mechanics study of {100}(100)NiAl is presented in Appendix IV.3. The same formulation where K_{Ic} is proportional to an exponential term containing the calculated Griffith energy divided by the yield strength was shown to predict the microscratch toughness of bulk NiAl. This was also accomplished for Al₃Sc as a function of scratch rate. The brittleness transition as a function of stress intensity rate was verified by compact tension fracture toughness tests for NiAl as seen in Figure 4 of Appendix IV.3. This study was the first to demonstrate that microscratch evaluations could be applied to semi-brittle intermetallics. Such a micromechanical probing method was concurrently developed for analyzing metallic thin films deposited on semiconductors or ceramics. As a paradigm for the microelectronics and magnetic recording industries, microscratch evaluations of nine different film/substrate combinations are listed in Table 1 of a paper "Continuous Microscratch Measurements of the Practical Work of Adhesion for Metal/Ceramic Systems. †† This is reproduced in Appendix IV.4. While the first two chapters in the Appendix deal with the brittle fracture modeling, the latter two address the experimentally-based micromechanical probing. Finally, we return to the toughness evaluation of NiAl using a micromechanical scratch technique.

[†] H. Huang and W.W. Gerberich, Acta Metall. Mater. 42, No. 3 (1994) pp. 639-647.

[#] S. Venkataraman, D.L. Kohlstedt and W.W. Gerberich, *J. Mater. Res.* 11, No. 12 (1996) pp. 3133-3145.

APPENDIX IV.1.

QUASI-EQUILIBRIUM MODELING OF THE TOUGHNESS TRANSITION DURING SEMI-BRITTLE CLEAVAGE

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APPENDIX IV.3.

FRACTURE TOUGHNESS OF INTERMETALLICS USING A MICRO-MECHANICAL PROBE

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APPENDIX IV.4.

CONTINUOUS MICROSCRATCH MEASUREMENTS OF THE PRACTICAL AND TRUE WORKS OF ADHESION FOR METAL/CERAMIC SYSTEMS

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