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TOUGHENED CERAMICS LIFE PREDICTION

Final Technical Report
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Objective

To understand the room temperature and high temperature [$\leq 1370^{\circ}\text{C}$ (2500°F)] behavior of brittle materials such as *in situ* toughened ceramics, glasses and intermetallics as the basis for developing life prediction and test methodologies. A major objective is to understand the relationship between microstructure and mechanical behavior within the bounds of a limited number of materials. A second major objective is to determine the behavior as a function of time and temperature. Specifically, the room temperature and elevated strength and reliability, the fracture toughness, slow crack growth and the creep behavior. These results will provide input for parallel materials development and design methodology programs. Resultant design codes will be verified.

Summary

The following is a summary of accomplishments that occurred under the Toughened Ceramic Life Prediction Program:

I. Test Facility Development

A substantial facility for testing of brittle materials was developed. In most cases, the properties mentioned below can be measured from 25 to 1400°C :

Strength - flexural (ASTM C 1161)

- tensile (ASTM C 1273)
- biaxial flexure
- torsional

Fracture Toughness - precracked beam

- chevron notch
- indentation strength
- controlled flaw, etc....

Fatigue - static

- dynamic
- cyclic
- thermomechanical

Creep - flexural

- tensile
- compressive

Hardness - Vickers (C 1327)

- Knoop (C 1326)

Elastic Constants - Young's modulus (C 1259)

- Poisson's ratio

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II. *Materials Tested*

During the program a wide variety of materials were tested for various properties. Although the bulk of the testing was specifically for this project, some testing of mutual interest was performed for other programs and researchers

Silicon Nitrides

Silicon Carbides

Aluminas

Aluminum Nitride

Intermetallics

Glasses

Zirconia (Ce and Y stabilized)

Composites - SiC/TiB₂ particulate

- Si₃N₄/SiC whisker

- Ti/SiC MMC

- SiC/SiC CMC

III. *Cooperative work with government agencies and private industry*

General Electric Aircraft Engines (GEAE)

- development and verification of a reliability model for brittle , single crystal NiAl

Pratt and Whitney/NASA Marshall

- developed inspection reference specimens for the Space Shuttle Main Engine liquid oxygen alternate turbo pump bearings

Westinghouse Electric Corporation

- multiaxial strength data for verifying CARES for tungsten carbide bearing applications

MER Corporation

- Measurement of creep, strength and fracture toughness of developmental silicon nitrides

Garrett Ceramic Components

- measure creep, slow crack growth, oxidation strength and fracture toughness of developmental silicon nitrides reinforced with silicon carbide

Assistance to many others - Mann test labs

- Ceradyne, etc...

- Universities

IV. Round Robin Participation

Program members participated in ten round robin test programs sponsored by the International Energy Agreement (IEA), the American Society for Testing and Materials (ASTM), the Versailles Agreement on Material and Standards (VAMAS) or the National Institute for Standards and Testing (NIST), as listed below:

IEA - Flexural strength of ceramics
- Tensile strength of ceramics
- Effect of Machining Conditions on the Strength of Silicon Nitride

ASTM - Impulse excitation method for frequency determination

VAMAS - Fracture toughness of ceramics by the surface crack in flexure
- Fracture toughness of silicon nitride at high temperature
- Fractography of advanced ceramics
- Fracture toughness of ceramic matrix composites
- Fracture toughness of ceramics using the SEVNB method

NIST - Creep of silicon nitride

V. Development Of Test Standards

Personnel on the Toughened Ceramic Life Prediction Program participated significantly in the drafting and approval of two ASTM standard as listed below, and participated in task group meeting for other standards:

Determination of Slow Crack Growth Parameters of Advanced Ceramics by Constant Stress Rate Testing

Determination of Fracture Toughness of Advanced Ceramics

VI. Model Development

A numerical model describing the effects of preloading on fatigue properties of ceramics was developed and verified. A reliability model and failure criterion for brittle, anisotropic materials such as NiAl was developed and is being verified.

VII. Design Code Verification

Data bases for verification of the *CARES*, *CARES Life* and *CARES Creep* codes were/are being generated. The data sets included uniaxial and multiaxial strength, fatigue and creep testing of silicon carbide, silicon nitride and alumina ceramics. Also, a data set is being generated for a reliability model for design of single crystal turbine vanes and other anisotropic materials

VIII. Publications

The attached list chronologically details journal papers and technical memorandums published during the funding period. Also, bimonthly, semi annual and quarterly reports were published as required.

IX. Recommendations

The CARES creep code is still under revision and data is being generated for verification. Continued work along this vein is useful, especially considering Solar Turbines efforts in field testing of ceramic turbine blades and in the rig testing of blades for 1000 hours. A second area of continuing research is the reliability of brittle single crystal materials such as silicon, germanium and nickel aluminide. A model has been developed and continued work to verify it might benefit the electronics community and GEAE.

Publications

"Crack Growth Resistance of Texture Alumina," J. A. Salem, J. L. Shannon, Jr. and R.C. Bradt, *Journal of the American Ceramic Society*, 72, [1], 20-27, 1989.

"Ultrasonic Imaging of Textured Alumina," D.B. Stang J. A. Salem and E.R. Generazio, NASA Technical Memorandum 101478, May, 1989.

"Fracture Resistance of a TiB_2 Particle/SiC Matrix Composite at Elevated Temperature," M.J. Jenkins, J. A. Salem and S.G. Seshardri, *Journal of Composite Materials*, Vol. 23, pages 77-91, 1989.

"Strength and Toughness of Monolithic and Composite Silicon Nitrides," J. A. Salem, NASA Technical Memorandum 102423, January, 1990.

"Numerical, Micro-Mechanical Prediction of Crack Growth Resistance in a Fibre-Reinforced/Brittle Matrix Composite," M.G. Jenkins, A. Ghosh and J.A. Salem, in the *Proceedings of the Society for Experimental Mechanics Spring Conference*, Albuquerque, New Mexico, June 4-6, 1990.

"Processing and Mechanical Behavior of Whisker-Toughened Silicon Nitride," H. Yeh, E. Solidum, J. Schienle, K. Karasek and J. Salem, *proceedings of the 28th Annual SAE Automotive Technology Development Contractors Coordination Meeting*, Dearborn, Michigan, October 22-25th, 1990, P243, pages 155-259.

"Strength and Fracture Toughness of Monolithic and SiC Whisker-Reinforced Silicon Nitrides," S.R. Choi and J.A. Salem, in the *Proceedings of the American Ceramic Society Symposium on Composites*, Orlando, Florida, November, 1990.

"Effects of Pre-cracking method on Fracture Properties of Alumina," J.A. Salem, M.G. Jenkins, M.K. Ferber, and J.L. Shannon, Jr., pp. 762-769 in the *Proceedings of Society of Experimental Mechanics Conference on Experimental Mechanics*, Milwaukee, Wisconsin, June 10-13, 1991.

"Comparison of Dynamic Fatigue Behavior Between SiC Whisker -Reinforced Composite and Monolithic Silicon Nitrides," with S.R. Choi, NASA TM 103707, February, 1991.

"Strength, Toughness and R-Curve Behavior of SiC Whisker Reinforced Composite Si_3N_4 with Reference to Monolithic Si_3N_4 ," S. R. Choi and J. A. Salem, *J. Mater. Sci.*, 27 1491-98 1991.

"Dynamic Fatigue Property of Silicon Carbide Whisker-Reinforced Silicon Nitride," S. R. Choi, J. A. Salem and J. P. Gyekenyesi, *Ceram. Eng. Sci. Proc.*, 12[7-8] 1524-1536 1991.

"Elevated Temperature Mechanical Behavior of Monolithic and SiC Whisker-Reinforced Silicon Nitrides," J. A. Salem, S. R. Choi, W.A. Sanders and D. S. Fox, NASA TM 105245, Lewis Research Center, National Aeronautics and Space Administration, Cleveland, OH (1991).

"Reliability Analysis of a Structural Ceramic Combustion Chamber, J.A. Salem, J. Manderscheid, M. Freedman and J. Gyekenyesi, *proceedings of ASME International Gas Turbines and Aeroengine Conference*, 91-GT-155, Orlando, Florida, June 3-6, 1991.

"Mechanical Behavior and Failure Phenomenon of an *In Situ* Toughened Silicon Nitride," J.A. Salem, S.R. Choi, M. Freeman and M.G. Jenkins, *Journal of Materials Science*, Vol 26, 1992. (NASA TM 103741)

"Analysis of Precracking Parameters for Ceramic Single-Edge-Precracked-Beam Specimens," S. R. Choi, A. Chulya and J. A. Salem, pp. 73-88 in *Fracture Mechanics of Ceramics*, Vol 10, Edited by R. C. Bradt et al., Plenum Press, NY (1992). (NASA TM 105568)

"Fracture Toughness of Advanced Ceramics at Room Temperature," G.D. Quinn, J. A. Salem, I. Bar-on, K. Cho, M. Foley and H. Fang, *J. Res. Nat. Inst. Stds. and Tech.*, Vol 97, No. 5, 579-607 (1992).

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"Indentation Flaw Formation and Strength Response of Silicon Nitride Ceramics at Low Indentation Loads," S. R. Choi and J. A. Salem, *J. Mater. Sci. Lett.*, 11 1398-1400 (1992).

"Some Observations in Fracture Toughness Testing with Chevron-Notched Specimens," J. A. Salem, J.L. Shannon, Jr., and M. G. Jenkins in *Chevron -Notch Test Experience: Metals and Non-Metals*, ASTM STP 1172, K. R. Brown and F. I. Baratta, Eds., ASTM Philadelphia, 1992, pp. 9-25.

"Chevron-Notch, Flexure Tests For Measuring the Elevated Temperature Fracture Resistance's of Structure Ceramics," M.G. Jenkins, M.K. Faber, A. Ghost, and J. Peas and J. A. Salem in *Chevron -Notch Test Experience: Metals and Non-Metals*, ASTM STP 1172, K. R. Brown and F. I. Baratta, Eds., ASTM Philadelphia, 1992, pp. 159 - 177.

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"Evaluation of the Fracture Toughness of Nb-40-8Cr-1W-0.05B Intermetallic Material by Indentation Techniques," S.R. Choi, J.A. Salem and M.G. Hebsur, *J. Matl. Sci.*, 40, 155-160 (1993).

"Effects of Environment on Fracture Toughness and Strength of Alumina," S. R. Choi, V. Tikare and J.A. Salem, *Scripta Metallurgica et Materialia*, 29 189-192 (1993).

"Creep Behavior of Flexure Beam Specimens of SiC Whisker-Reinforced Composite and Monolithic Si₃N₄'s," S. R. Choi, J. A. Salem, and W. A. Sanders, *Ceram. Eng. Sci. Proc.*, 14[7-8] 113-121 (1993).

"Failure Prediction Using the Ring-On-Ring Test and the CARES/LIFE Integrated Design Program," L. M. Powers, J. A. Salem, and S. R. Choi, in *Reliability, Stress Analysis and Failure Prevention*, ASME DE-Vol. 55, pp. 55-63 (1993).

"Thermal and Elastic Anisotropy in Single Crystal Al₂O₃ and SiC Whiskers," with Z. Lee and R.C. Bradt, NASA TM 106516, 1994.

"Production of Small Flaws in Ceramic Bearings for the Verification of Nondestructive Inspection Capabilities," Session VIII, *Conference on Advance Earth-to-Orbit Propulsion Technology*, Marshall Space Flight Center, AL, May 17-19, 1994

"Measurement and Modeling of Grinding Damage in Ceramics," in *Proceedings of the Conference on Design for Manufacturability*, Indianapolis, Indiana, April 25-27, 1994.

"Test Method Effects in Creep and Crack Growth Testing of Ceramics," in *proceedings of the Society for Experimental Mechanic Spring Conference on Experimental Mechanics*, Baltimore, MD, June 6-8, 1994.

"Crack Growth Resistance of In Situ-Toughened Silicon Nitride," S. R. Choi and J.A. Salem, *J. Am. Ceram. Soc.*, 77[4] 1042-46 (1994).

"Comparison of Tension and Flexure to Determine Fatigue Life Prediction Parameters at Elevated Temperatures," S. R. Choi, J. A. Salem, and J. L. Palko, pp. 98-111 in Life Prediction Methodologies and Data for Ceramic Materials, C. R. Brinkman and S. F. Duffy, editors, STP 1201, ASTM (1994), Philadelphia, PA.

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"Dynamic, Static, and Cyclic Fatigue of Alumina with Indentation-Induced Flaws," S. R. Choi and J. A. Salem, *J. Mater. Sci. Lett.*, 14 1286-1288 (1995).

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"Reliability Analysis of Uniaxially Ground Brittle Materials," J. A. Salem, N. N. Nemeth, L. M. Powers and S. R. Choi, *Journal of Eng. Gas Turbines and Power*, Trans. of the ASME, 118 863-871 (1996).

"Preloading Technique in Dynamic Fatigue Testing of Ceramics: Effect of Preloading on Strength Variation," S. R. Choi and J. A. Salem, *J. Mater. Sci. Lett.* 15[22] 1963-65 (1996).

"'Inert' Strengths of Silicon Nitride Ceramics at Elevated Temperatures," S. R. Choi and J. A. Salem, *Ceram. Eng. Sci. Proc.*, 17[3] 454-461 (1996).

"Free-Roller versus Fixed-Roller Fixtures in Flexural Testing of Advanced Ceramic Materials," S. R. Choi and J. A. Salem, *Ceram. Eng. Sci. Proc.*, 17[3] 69-77 (1996).

"Error Identified as Hysteresis in Flexure Testing of Advanced Ceramics," S. R. Choi and J. A. Salem, *Scripta Metallurgica et Materialia*, 35[4] 491-194 (1996).

"High-Temperature Slow Crack Growth of a Silicon Carbide Determined by Constant Stress-Rate and Constant Stress Testing", S. R. Choi and J. A. Salem, in press *J. Mater. Sci.* (1996).

"Fatigue Strength as a Function of Preloading in Dynamic Fatigue Testing of Glass and Ceramics," S. R. Choi J. P. Gyekenyesi, the *Trans. of the ASME, J. Eng. Gas Turbines & Power*, 119 [3] 493-499 (1997).

"'Ultra'-Fast Fracture Strength of Advanced Ceramics at Elevated Temperatures," S. R. Choi and J. A. Salem, in press, *Material Sci. & Eng. A.* (1997)

"Estimation of Slow Crack Growth Parameters for Constant Stress-Rate Test Data of Advanced Ceramics and Glass by the Individual Data and Arithmetic Mean Methods," S. R. Choi, J. A. Salem, and F. A. Holland, *NASA TM 107369*, Lewis Research Center, National Aeronautics and Space Administration, Cleveland, OH (1997).

"Error in Flexure Testing of Advanced Ceramics under Cyclic Loading," *Ceram. Eng. Sci. Proc.*, 18[3] 495-502 (1997).

"A Strain Gage Technique to Measure Stable Crack Extension in Ceramics," J. A. Salem, L. J. Ghosn, S. R. Choi, and M. G. Jenkins, *Proceedings of the Society for Experimental Mechanics Spring Conference*, June 2-4, 1997, Bellevue, WA.

"Crack Branching and Fracture Mirror Data for Glasses and Advanced Ceramics," S. R. Choi and J. A. Salem, in press for *NASA TM* (1997).

"'Ultra'-Fast Strength Behavior of Advanced Ceramics at Elevated Temperatures," S. R. Choi, J. A. Salem, and J. P. Gyekenyesi, presented at the 6th International Symposium on Ceramic Materials & Components for Engines, October 19-24, 1997, Arita, Japan. To be published in the Proceedings.

"Back-Face Strain As A Method For Monitoring Stable Crack Extension In Ceramics," J. A. Salem, L. J. Ghosn, S. R. Choi, , To be published in the *proceeding of the 22nd Annual Cocoa Beach Conference*, January 20 - 24, 1998.

"Reliability Modeling Of Brittle Anisotropic Materials," J. A. Salem, R. Noebe and J. Manderscheid, To be published in the *proceeding of the 22nd Annual Cocoa Beach Conference*, January 20 - 24, 1998.

Professional Activities

Co-Chairperson "High Temperature Deformation" Session at the Fracture Mechanics of Ceramics Conference, Nagoya, Japan, July 15-17, 1991

Chairperson "Progress on International Standards for Ceramics" session at the 38th ASME International Gas Turbine and Aeroengine Congress, Cincinnati, Ohio, May 24-27, 1993.

Chairperson "Design for Manufacturability" session III at the American Ceramic Society Annual Basic Sciences Meeting, Indianapolis, Indiana, April 25-27, 1994.