Final Technical Report

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Thermomechanical Response of Layered Materials

Submitted by:

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Summary of Technical Accomplishments

Several major advances have been made during the final phase of this program during the past three years. These research findings have been published in four journal articles, which are appended herewith, and presented in numerous invited, keynote and plenary lectures at major international conferences and technical meetings.

Key research accomplishments are summarized below.

1. A new experimental technique involving instrumented indentation was demonstrated for the specific purpose of calibrating certain variations in elastic properties as a function of position in thermally sprayed and sintered materials. It was shown that the characteristics of indentation response, as predicted by the theory of Giannakopoulos and Suresh (International Journal of Solids and Structures, May 1997), consistent with experimental observations in several different graded microstructures. Details of this investigation are reported in reference #1.
2. This work experimentally demonstrated for the first time that controlled gradients in elastic properties alone can lead to the suppression of damage and cracking during contact loading. Hertzian (spherical) indentation experiments were conducted in a graded alumina-glass composite whose Young's modulus increased with depth beneath the indented surface. An in situ processing method involving impregnation of a dense, fine-grained alumina by an aluminosilicate glass was employed to fabricate such a composite. With this technique, a monotonic, unidirectional variation in Young's modulus of as much as 50% was introduced over a distance of approximately 2 mm, while keeping the coefficient of thermal expansion and the Poisson ratio for the glass and the alumina nearly the same. The macroscopically graded, elastic composite so produced with nearly full density has essentially no macroscopic, long-range residual stresses following processing. The unidirectional variation in Young's modulus under the indenter is shown to fully suppress the formation of Hertzian cone cracks. Without these elastic-modulus gradients, cone-crack formation was observed in bulk glass and alumina. Finite element analyses of spherical indentation on elastically graded substrates were also performed to develop a quantitative understanding of the experimental trends. It was reasoned that the present innovations, involving functionally graded surfaces and their in situ processing, provide new possibilities for enhancing certain contact damage resistance characteristics in various ceramic materials for a broad range of engineering applications. Furthermore, this contact-damage-resistance phenomenon in functionally graded ceramics is elastic in nature, and is, therefore, likely to be immune to mechanical fatigue within the elastic limit. The results of this study are published in the journal paper, reference #2.
3. New methods were developed in this work for the estimation of mechanical properties of ductile alloys and brittle ceramics by recourse to continuous measurements of load-penetration curves with spherical microindenters. Elastic and plastic properties of metals and Young's modulus of ceramics were determined in the microindentation regime by continuous measurements of load versus depth of penetration with spherical indenters. Calibration procedures, usually applied in nanoindentation experiments, were not needed in the microregime where spherical indenters (rather than sharp indenters with microscopica spherical tips) can be manufactured. As indenters of larger diameters are used, the elastic response of the specimen can be probed during

the loading stage of the indentation tests (and not only during unloading, as is the case with nanoindenters). Hence an accurate determination of Young's modulus can be achieved without a prior knowledge of possible "piling up" or "sinking in" which may occur at the perimenter of the contact area. The contact response of materials was shown to undergo four distinct regions: (i) pre-Hertzian regime, (ii) Hertzian regime, (iii) small-scale plasticity, and (iv) large-scale plasticity. A general methodology for estimation of yield strength and hardening exponent of metals was also proposed for the last regime. Details of this work are reported in the journal paper, reference #3.

4. New experimental results and computational simulations were carried out to demonstrate that diffusion bonding of a metal to a ceramic can lead to gradients in grain morphology, texture and microhardness as a function of location from the interface. The evolution of grain morphology, crystallographic texture and microhardness in Al bonded to alumina was studied. Specimens of bilayer Al-alumina and symmetric trilayer alumina-Al-alumina were produced by solid-state diffusion bonding. Metallographic examination revealed the size and shape of grains in the Al layer, and the X-ray diffraction technique was used to measure the crystallographic texture at various through-thickness and in-plane locations. The results showed the existence of gradients in grain size, grain shape, texture, and microhardness through the thickness of the Al. Away from the interface, the aluminum grains were equiaxed, with a sharp cube recrystallization texture. Near the interface, elongated and slanted grains, with a rotated cube texture, were observed. The microhardness was seen to correlate with the distribution of grain size. Finite element analyses employing crystal plasticity models were carried out to simulate polygranular flow of Al during diffusion bonding. The interface constraint imposed by the alumina layer was found responsible for the evolution of the observed gradients in microstructure in the Al layer.

Journal Publications:

1. Suresh, S., Giannakopoulos, A.E. and Alcala, J. "Spherical Indentation of Compositionally Graded Metal-Ceramic Composites: Theory and Experiments", *Acta Materialia*, 45, 1307-1321, 1997.
2. Jitcharoen, J., Padture, N.P., Giannakopoulos, A.E. and Suresh, S., "Hertzian-Crack Suppression in Ceramics with Elastic-Modulus-Graded Surfaces", *Journal of the American Ceramic Society*, 81, 2301-2308, 1998.
3. Alcala, J., Giannakopoulos, A.E. and Suresh, S., "Continuous Measurements of Load-Penetration Curves with Spherical Micro-Indenters and the Estimation of Mechanical Properties", *Journal of Materials Research*, 13(5), 1390-1400, 1998.
4. Lin, C.-T., Shen, Y.-L., Becker, R. and Suresh, S., "Grain Morphology, Texture, and Microhardness Gradients in Aluminum Diffusion-Bonded to Aluminum Oxide", *Acta Materialia*, 42(2), 501-515, 1999.

Invited/Keynote/Plenary Lectures in International Conferences and Technical Meetings:

September 1996, Opening Keynote Lecture, "Fracture at Interfaces", 11th European Conference on Fracture, Poitiers, France.

October 1996, Keynote Lecture, "Plasticity Aspects of Fracture in Multi-Layered Materials", ASM Symposium on Metal-Ceramic Interfaces, Cincinnati, Ohio.

April 1997, Overview Lecture, "Mechanical-Nonmechanical Interactions in Layered and Graded Materials in Integrated Systems", Acta/Scripta Workshop on Coupled Effects in Integrated Systems, Monterey, CA.

June 1997, Keynote Lecture, "Control of Residual Stresses and Cracking in Layered and Graded Materials", 5th International Conference on Residual Stresses, Linköping, Sweden.

July 1997, Keynote Lecture, "Surface Engineering for Damage Tolerance", Symposium on Measurement of Residual Stresses, Los Alamos National Laboratory, Los Alamos, New Mexico.

November 1997, Keynote Lecture, "Indentation of Graded Materials", Special Symposium on Contact Mechanics, University of Barcelona, Spain.

November 1997, Keynote Lecture, "Contact-Damage Resistance of Graded Coatings", International Symposium on Functional and Smart Materials and Sensors, ASME International Congress, Dallas, TX.

December 1997, Keynote Lecture, "Design of Graded Surfaces for Contact-Damage Resistance", International Symposium on Functionally Graded Materials, MRS Meeting, Boston, MA.

June 1998, Invited Lecture, "Gradient Materials for Surface Damage Protection", Max-Planck Institute Symposium on Small Volume Structures, Ringberg Castle, Tegernsee, Germany.

June 1998, Keynote Lecture, "The Engineering of Surfaces with Gradient Microstructures", Gordon Conference, New Hampshire.

October 1998, Opening Plenary Lecture, "Thermomechanical Properties of Functionally Graded Materials", Fifth International Conference on Functionally Graded Materials, FGM 98, Dresden, Germany.

October 1998, Invited Lecture, "Indentation of Graded Materials", Acta Materialia Conference on Interfaces, La Jolla, CA.

December 1998, Invited Lecture, "The Engineering of Surface Microstructures for Enhanced Damage Tolerance", Symposium on Modelling of Metallic Materials, Materials Research Society, Boston, MA.

June 1999, Keynote Lecture, "Probing Residual Stresses in Thin Films and Small Volume Structures", Engineering Foundation Conference on Thin Films, Il Coccio, Italy.

September 1999, Keynote Lecture, "Microstructural Design at Surfaces for Tribological Protection", Risoe Materials Conference, Risoe, Roskilde, Denmark.

December 1999, Keynote Lecture, "Contact Deformation at Surfaces and Thin Films", First Annual Conference, Netherlands Institute for Metals Research, The Netherlands.