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FRACTURE BEHAVIOR OF SURFACE-MODIFIED CERAMICS

PROGRESS REPORT FOR PERIOD
JUNE 1ST, 1992 TO MAY 31ST, 1993

DOE/ER/45252--6

DE93 013707

GRANT NO: DE-FG02-86ER45252

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April 1993

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1. RESEARCH PROGRESS

The overall objective of the grant is to understand crack growth on surfaces that are being acted on by macroscopic residual stresses. Under the current grant research has been performed in 3 areas; 1) fracture behavior of ion-exchanged glasses, 2) fracture of SiC-coated graphite and 3) sub-critical crack growth in ion-exchanged glasses. The work in the first two areas has been completed and is the subject of two Ph. D. theses. The third project was initiated in September 1990 and is expected to be completed by December 1993.

1.1. Indentation of Ion-Exchanged Glasses

The work in this project has concentrated in 4 areas; A) development of a simple technique to measure residual stress profiles, B) in-situ observation of indentation crack formation on ion-exchanged surfaces, C) strength variability behavior of brittle materials under stabilizing and destabilizing fracture fields and D) crack stability and crack growth resistance curves due to macroscopic residual compressive stress profiles.

1.1.1. Residual Stress Profile Determination Using Strain Gage Measurements

The use of a strain gage/polishing technique to determine residual stress profiles in materials was investigated. For the work under the current DOE grant, it was important to develop a simple technique for characterization of the residual stress profiles. The chosen technique comprises of mounting a strain gage on one side of a beam shaped sample, and polishing the other side of the specimen. The removal of material causes the sample to bend, and the strain recorded on the strain gage can be used to obtain the prior stress profile. The details of the approach have been published. The analytical procedure involves solving an integral equation (which arises from the elasticity solutions of the beam bending problem), fitting the strain data obtained from the strain gage during polishing to a polynomial, and substituting the coefficients of this polynomial into the solution of the integral equation. The result is the prior residual stress profile in the sample. Numerical simulations, using different stress profiles and introducing errors in the strain data (to simulate experimental errors in strain determination), were performed. The results of these tests indicate that the procedure developed is numerically robust, and that it can be used to assess stress profiles which do not vary steeply. Polishing experiments on tempered glass were conducted and the stress profiles were derived employing the suggested procedure.

The results were in good agreement with independent determination of the stress at the surface and with stress profiles associated with the tempering process.

1.1.2. Indentation Fracture Sequences for Glasses under Residual Surface Compression

The indentation sequence for ion-exchanged soda-lime silica and alumino-silicate glasses were investigated and the data were compared with observations on the stress-free surface.[§] This work uncovered some previously unreported phenomena, e.g., the observation of cone cracking under sharp indenters on glasses considered to be "normal" (alumino-silicate) and the complete absence of median cracking on ion-exchanged soda-lime silica glass. The analysis of the in-situ observations have revealed the initiation of cracks from the indentation site can be qualitatively explained using simple stress superposition arguments. The overall effect of the residual compressive stress is to alter the flaw morphology formed during the indentation process, from median to radial, i.e. restricting the indentation flaw depth. The formation of lateral cracks is retarded by the ion-exchange compressive stress. Further details regarding these experiments have been reported in a published paper. Similar experiments on tempered glasses have yielded identical results, i.e., a complete suppression of median cracking and a radial crack morphology. This is an important result indicating for surface modified materials, the damage due to contact events not only initiates at higher loads but will also be surface localized. In view of the altered indentation crack morphology, previous analyses which assumed that the flaw shape was semi-circular must be modified. The growth of indentation cracks on ion-exchanged glass surfaces were studied and the results are available in the Ph. D thesis of R. Tandon

1.1.3. Strength Variability in Brittle Materials Under Stabilizing and Destabilizing Fracture Fields

It has been shown in this project that macroscopic surface residual stresses can have a pronounced effect on strength variability. In order to understand this variability in more detail, materials with flaws which experience various stabilizing and destabilizing fields during their extension to failure were examined theoretically. The results of the study have been accepted for publication (Appendix 1).

[§] Using an indentation device at the T. J. Watson Research Center, IBM, Yorktown Heights, NY, which permits in-situ observations during the indentation cycle.

Two cases were considered: (a) when the flaws are subjected to a residual surface stress and (b) when the resistance to crack extension increases with crack length (R- or T-curve behavior). In the analysis, the critical crack size distribution was treated as a continuous function. By invoking the equilibrium and instability criteria for fracture, and by describing the physical stabilizing and destabilizing fields in terms of the appropriate stress intensity factors, the forms of the strength distributions were obtained. General forms for the crack size distribution were assumed and fundamental statistical descriptions, i.e., mean and standard deviation, were used to characterize the strength and variability. The results of the analysis were insensitive to the crack size distribution assumed and indicated in the cases studied that the variability did not necessarily depend on the critical microstructural parameters in the same fashion. This indicates optimum microstructures must exist in which increases in strength must be balanced against increases in strength variability. Directions for altering the microstructure of materials in order to obtain this balance were discussed.

1.1.4. Crack Stability and T-Curves Due to Macroscopic Residual Compressive Stress Profiles

Stabilization of the fracture process and resistance to strength degradation have been observed for materials with increasing T-Curves. In our studies, the possibility of using residual compressive stresses to induce crack stabilization was examined theoretically and the results have been published in three papers. Reprints of these papers are attached as Appendix 2, 3 and 4. The approach outlined in these papers represents a new way to improve the strength of materials using surface compression, but in a way in which the material shows increased strength, flaw tolerance and substantial reductions in strength variability.

In the analysis, forms for the residual compressive stress profiles, in which the maximum compression lay ahead of the crack tip, were considered. The stress intensity factors for linear through-the-thickness cracks subjected to these profiles were derived and then used to construct the T-Curves for the stress profiles considered. It was demonstrated that the presence of these T-Curves can lead to crack stability under the action of applied tensile stresses, and to strength insensitivity to the initial flaw size. The effects of additional localized stress fields (similar to those produced by indentation) on crack growth in these materials was also considered. In this case, the strength was found to be relatively insensitive to the magnitude of the localized loading. The presence of the T-Curves due to residual compressive stresses was also shown to reduce the strength variability. It was therefore concluded that

residual stresses can be used to improve mechanical reliability in ways which are usually associated with microstructural toughening mechanisms.

1.2. Fracture Behavior of SiC-Coated Graphite

The aim of this research is to investigate the fracture behavior of CVD (chemical vapor deposition) SiC coatings on graphite substrates. The coatings were deposited at various temperatures (range:1200-1300°C) and times (range:1-4 h). These materials were cut into a sandwich configuration (SiC/graphite/SiC). The samples had coating thicknesses ranging from 10 μm to 150 μm and substrate thicknesses of 3.24 mm. X-ray diffraction studies indicated that the coatings were beta-SiC (cubic). Extensive preferred orientation was observed, with the [111] texture most dominant in the growth direction

The primary areas of interest in the mechanical characterization were Young's modulus and residual stress measurements of the coating, and fracture behavior of the layered structure. The average Young's moduli of polished coatings were measured (in situ) using strain gage and dynamic resonance techniques and were determined to be 448 and 417 GPa, respectively. In both techniques, the determination of a representative coating thickness is critical. The major source of experimental error was due to coating thickness measurement which prevented distinguishing any fine changes in Young's modulus due to the deposition conditions. These moduli were also found to be in good agreement with literature values and consistent with the observed preferred orientation. When large coating thickness variations are present, the strain gage technique should be better than the free ends resonance because the thickness variation underneath the strain gage should be less than across the entire sample. The results of this work are given in Appendix 5 and have been accepted for publication.

As indicated above, the residual strains in the coatings were measured through the use of a strain gage/polishing technique. The samples bowed upon polishing, indicating that the residual strains in the coatings are compressive. The average residual stresses ranged from -80 to -380 MPa and were highly dependent on coating thickness. These values were compared to the residual stresses calculated from thermal expansion data. The calculated stresses were approximately half the measured values, indicating growth stresses are significant in this system.

The fracture behavior of the β -SiC coated graphite substrates was examined in 4 point flexure. When the coating thickness exceeded a "critical thickness" of $\sim 50 \mu\text{m}$, catastrophic failure of the sample was observed. However, when the coating thickness was less than $\sim 50 \mu\text{m}$, crack arrest/multiple cracking was observed in the coating being subjected to applied tension, despite the fact that the graphite has a

lower toughness than SiC. The ability to arrest cracks in this fashion in a brittle material would be a very useful attribute in structural applications. Observation of the crack arrest/multiple cracking phenomenon in the samples was accomplished using a fluorescent dye penetrant. In addition, arrested cracks were observed in the SEM using a loading fixture which was placed in the microscope specimen chamber. All of the observed, arrested crack tips were located in the graphite. The results of the study on the fracture behavior are presented as Appendix 6 and a manuscript has been accepted for publication.

In order to understand the conditions for the arrest of the initial crack, the process was modelled using fracture mechanics and it was found the critical thickness should depend on the coating strength, substrate toughness, relative Young's moduli, and residual stresses. For example, an increase in the relative Young's moduli ($E_{SiC}/E_{Graphite}$) or substrate toughness, increases the likelihood that the first crack to propagate would also arrest. Similarly, a decrease in the coating strength or residual stress (compressive), produces the same effect. Subsequently, experimental data was applied to the model to test its validity and to predict a critical coating thickness. The predicted critical thicknesses were ~20% less than the observed values. The results of the above analysis are important for the fracture behavior of brittle laminates of all types, indicating that crack stability and arrest can be designed into these brittle composite materials. A copy of the paper is attached as Appendix 7 and it has been accepted for publication.

1.3. Sub-Critical Crack Growth in Ion-Exchanged Glasses

The aim of this research is to investigate the influence of surface residual stresses on sub-critical crack growth behavior of glasses. A loading jig was designed and constructed to be placed on a microscope for the in-situ studies of crack growth using indentation to introduce the surface cracks. Crack velocity/stress intensity data will be obtained for annealed and ion-exchanged glasses. The jig is operational and preliminary experiments will be started before the end of the current contract year.

Dynamic fatigue tests were conducted on soda lime silica glass in air to quantify sub-critical crack growth behavior for one of the baseline materials. The strength of annealed specimens was measured in four-point flexure as a function of stress rate (0.04 MPa/s to 425 MPa/s). The fatigue susceptibility exponent (N) was determined to be 20.7 which is in good agreement with the literature values. Samples of the same glass were ion-exchanged at 450 C for 1 hr. in a potassium nitrate bath which resulted in a strength increase of ~ 200 MPa. Dynamic fatigue tests were performed on

annealed and ion-exchanged specimens. The pre-existing natural flaws caused a large scatter in the strength data of ion-exchanged specimens, which made the determination of an exact N value difficult. Nevertheless, statistical calculations of confidence intervals on N value obtained showed a definite decrease in the slope of the dynamic fatigue strength data, once the glasses are ion-exchanged. The change in the slope of the dynamic fatigue data may just be a reflection that subcritical crack growth does occur when the surface is in compression. If the surface compressive stress in the ion-exchanged specimens is assumed to be equal to the increase in strength (~ 200 MPa) and subtracted from the strength of ion-exchanged specimens, an N value of ~ 20 was determined, essentially the same as the stress-free glass. This would imply for fairly deep layers of surface compression compared to the critical flaw size, the mechanism of sub-critical crack growth is not affected by the surface stress, i.e., the sub-critical crack growth is related to the actual stress intensity factor (K) not the applied K . Unfortunately the scatter associated with the data makes a definite conclusion difficult.

In order to reduce the scatter in the data, the above experiments were conducted on indented samples. The N values were found to be 20.1 and 20.7 for 'annealed-indented' and 'annealed-exchanged-indented' ($P = 9.8$ N) specimens, respectively. Tests were also performed on annealed, ion-exchanged glass with an indentation force of 3.9 N; the N value in this case was 17.1. For the indented case, residual stresses due to the indentation process were taken into account in the analysis. Interestingly, in these specimens, in which the crack size is much larger than the depth of surface compression, the surface is on average, still in compression throughout the failure process. It has been shown previously, however, that crack growth in these cases occurs primarily below the surface, so that even though the surface is essentially in compression, there is no strength increase. A similar effect may be going on in these cases, in which the ion exchange does not change the actual K that is controlling the sub-critical crack growth when the surface compression layer is thin.

Dynamic fatigue tests have been started on a second glass (sodium aluminosilicate) because deeper ion-exchange layers can be obtained in this glass. This will allow the influence of thicker compressive layers on the fatigue susceptibility to be studied. In addition to the dynamic fatigue data, in-situ observations of the sub-critical crack growth process will be made on the two glasses under study. It is felt that the in-situ observations are the key feature of this project and will lead to a much better understanding of the sub-critical crack growth than attempting to deduce the behavior

from strength data. The data for the in-situ observations will be obtained using indentation cracks introduced before and after ion exchange and will be compared with that obtained on stress-free surfaces. The loading apparatus will also allow measurement of the stress intensity factor and the crack velocity. The initial set of experiments will consider the influence of water on the crack growth as this has been studied extensively for (surface) stress-free glasses. Apart from in-situ and dynamic fatigue tests, experiments will be performed to determine surface residual stresses using the strain gage technique, described earlier.

2. PERSONNEL SUPPORTED BY GRANT

D. J. Green, Principal Investigator

T. R. Watkins, Graduate Assistant

Ph. D. Thesis: Fracture Behavior of SiC-Graphite Composites.

Graduation Date: May 1992

R. Tandon, Graduate Assistant

Ph. D. Thesis: Nucleation and Formation on Indentation Cracks on Compressively-Stressed Surfaces

Graduation Date: August 1992

P. Jain, Graduate Assistant

Ph. D. Thesis: Sub-critical Crack Growth in Ion-Exchanged Glasses.

Expected Graduation Date: December 1993

Reprints & Preprints removed

3. PUBLICATIONS AND PRESENTATIONS

3.1 Publications (1992)

1. R. Tandon and D. J. Green., "The Effect of Crack Growth Stability Induced by Residual Compressive Stresses on Strength Variability," J. Mater. Res. 7[3] (1992).
2. R. Tandon and D. J. Green, "Crack Stability on Residually-Stressed Surfaces," The Physics of Non-Crystalline Solids, edited by L. D. Pye, W. C. LaCourse and H. J. Stevens, pp. 659-63, Taylor & Francis, London, 1992.
3. R. Tandon, D. J. Green and R. F. Cook, "Strength Variability and T Curve Effects," Acta Metall. 41[2] 399-408 (1993).
4. T. R. Watkins, E. Ryba and D. J. Green, "Measurement of In-Plane Elastic Modulus of CVD SiC Coatings", J. Am. Ceram. Soc. to be published.
5. T. R. Watkins and D. J. Green, "Fracture Behavior of CVD SiC-Coated Graphite: I. Experimental Results", J. Am. Ceram. Soc. to be published.
6. T. R. Watkins and D. J. Green, "Fracture Behavior of CVD SiC-Coated Graphite: II. Theoretical Model", J. Am. Ceram. Soc. to be published.
7. T. R. Watkins, "Fracture Behavior of SiC-Graphite Composites," Ph.D. Thesis, The Pennsylvania State University, 1992
8. R. Tandon, "Influence of Residual Stress-Induced R-Curves on the Fracture Behavior of Brittle Materials", Ph.D. Thesis, The Pennsylvania State University, 1992.

3.2 Presentations (1992)

1. D. J. Green, "Crack Arrest in Layered Structures," Annual Meeting of Canadian Ceramics Society, Toronto, February 1992.
2. T. R. Watkins, "Fracture Behavior of CVD SiC/Graphite Composites: Experimental Results" Annual Meeting of American Ceramic Society, Minneapolis, April 1992.
3. T. R. Watkins, "Fracture Behavior of CVD SiC/Graphite Composites: Stress Intensity Model for Crack Arrest" *ibid.*
4. R. Tandon, "Cone Crack Initiation under Vickers Indentation in Silicate Glasses" Annual Meeting of American Ceramic Society, Minneapolis, April 1992.
5. P. Jain, "Dynamic Fatigue Behavior of Residually-Stressed Glasses" Annual Meeting of American Ceramic Society, Minneapolis, April 1992.
6. D. J. Green, "Crack Arrest in Graded and Layered Structures," Invited Paper, Pacific Coast Meeting of American Ceramic Society, San Francisco, 1992.

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