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6. AUTHOR(S) Professor K. W. White				
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) University of Houston Department of Mechanical Engineering Houston, TX 77204-4792			8. PERFORMING ORGANIZATION REPORT NUMBER	
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13. ABSTRACT (Maximum 200 words) <p>Post fracture tensile (PFT) experiments on two microstructures elucidated the crystallographic and microstructural contributions to the pullout resistance. In alumina, the average residual stresses arising from TEA diminish with increasing temperature, causing two effects evidenced by a general downward shift of the characteristic wake stress-displacement curves. Similar tests conducted on the cubic spinel exhibited no change in bridging efficiency. Similar experiments at the higher temperatures identify topographic changes of the fracture surface as the critical influence on the observed increased toughening behavior.</p> <p>Also, using the post fracture tensile (PFT) technique, the isolated wake region was tested in direct tension to determine the wake stiffness, offering further insight to those featured of the microstructure that control the bridging efficiency. From these studies, we found that alternate, more compliant micro-mechanisms must be included in the modeling effort to faithfully represent the relatively low measured wake stiffnesses.</p>				
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ASSERT Technical Report: ASSERT Linked to:**Fracture Process Zone Studies of High Temperature Structural Ceramics****Introduction**

The wake studies conducted over the last 3.5 years were directed at achieving an understanding of the wake role under various loading conditions. The complete stress-displacement relationship describes both elastic behavior, prior to the maximum stress, and a strain softening behavior beyond critical conditions. Our wake studies may, therefore, be loosely categorized into these two regimes. The first two years targeted the strain softening mechanisms, and the last 1.5 years examined the details of the initial (macroscopically elastic) portions of the stress-displacement curve.

In consideration of the strain-softening regime, two microstructures elucidated the crystallographic and microstructural contributions to the pullout resistance. TEA contributions were studied at temperatures below 600°C for both the alumina and spinel to avoid the effects of the softened grain boundary phases expected at higher temperatures. In alumina, the average residual stresses arising from TEA diminish with increasing temperature, causing two effects evidenced by a general downward shift of the characteristic stress-displacement curves. Similar tests conducted on the cubic spinel exhibited no change in bridging efficiency over the same temperature range.

A behavioral change, characterized by an increase in the limiting COD, appears in the stress-displacement results of both alumina materials at temperatures greater than 600°C, and coincides with the expected softening point of a similar grain boundary phase. Based upon studies of strain rate and time dependence, no evidence for viscous behavior was observed. Instead, topographic changes of the fracture surface near this temperature explain the increase in toughening behavior at high temperatures. This was confirmed through the PFT test which eliminated the surface roughness variable.

From this previous data the connection between the energy dissipative mechanisms described by the strain softening behavior were related to characteristic properties of the microstructure. In the last year, focus has shifted to the region of the wake behavior prior to critical conditions. First, using the post fracture tensile (PFT) technique, the wake region was isolated and tested in direct tensile tests. The stiffnesses measured through this test implicate the various bridging mechanisms in bridging through a simple Hooke's law-based model. The stiffness of wake ligaments provides some insight to those features of

the microstructure that affect the bridging efficiency. Compliant mechanisms, such as ligament bending, asperity loading, and grain rotation, predict the relatively low measured stiffness, as compared with the conceptual frictional grain pullout model. Secondly, we obtained preliminary results for the load cycling characteristics of the wake.

Current Studies

The post-fracture tensile (PFT) technique provides the grain-bridging stiffness as a function of crack opening displacement (COD) in the crack wake. A rather simple analysis based on Hooke's law incorporates the PFT stiffnesses into a model which provides insight to the nature of the active bridging mechanism. The conceptual model, based on prismatic grains which are extracted from sockets in the mating fracture face, is not consistent with the low measured wake stiffnesses. This, in addition to fractographic evidence, suggest the strong influence of other, more compliant, mechanisms such as grain rotation, asperity loading and ligament bending.

Preliminary data from the last year also examines the microstructural role of crack-face bridging ligaments subjected to small applied displacements. This work reveals the more subtle role of subgrain-size features responsible for degradation of the R-curve by load-cycling in the small displacement regime. Based on the current results, fatigue-related damage depends upon damage to features on the order of $0.1 \mu\text{m}$, or 0.5% of the mean grain size. Applied displacements beyond the $0.1 \mu\text{m}$ critical displacement for this alumina resulted in nonrecoverable displacements.

Displacement Measurements

For accurate, noncontact measurement of crack face separations in the submicron range, we used a laser interferometric displacement gage (LIDG). Details were outlined in the last report.

Post Fracture Tensile Stiffnesses

This work was reported in the last reporting period (Sept. 1996), and has since been accepted for publication in the ACS Journal.

Cyclic Loading Results

Some of this work has been accepted for publication in *Acta Met. et Materialia*. since the work continues, I provide a revised summary. The post-fracture tensile (PFT) technique isolates the grain-bridging elements which are responsible for the rising R-curve behavior in many ceramic systems and characterizes the load capacity as a function of crack-face separation. In the past, the PFT technique provided the characteristic stress-displacement relationship of the cohesive zone under large-scale monotonic-loading conditions, but the same technique is now advanced as a technique to study the initial response of the grain bridging mechanism to small applied loads under cyclic loading conditions. A closed-loop controller forces the specimen to follow a prescribed load path, using a piezoelectric actuator to supply the necessary displacements. The actual specimen displacements at the crack faces are measured independently using the same laser interferometric displacement gage (LIDG) described above. Since the displacements of interest fall below $0.1\ \mu\text{m}$, the LIDG provides the best method for crack-face displacement measurements. Sample data obtained by the current technique elucidates interesting aspects of the bridging mechanism at early stages of pullout, which escape detection by previous techniques in the literature.

The stress-displacement curve for one load-unload cycle using the current setup appears in Fig. 2. For this particular test, the load was applied sinusoidally such that the stress varied from 0.15 MPa to 1.25 MPa over a period of roughly 400 seconds. For low numbers of cycles, the load-unload cycle can be described by three linear regions. The first, starting from 0.15 MPa and extending to 0.98 MPa, describes the initial loading response. At first glance, the displacements in Fig. 5 conflict with those in Fig. 2, but in actuality the behaviors agree quite well. Note that in Fig. 2 we do not have data below roughly 3 MPa and that portion of the curve was approximated by a dashed line extrapolating linearly to the origin. It is interesting to note in Fig. 5 that at the transition point (0.98 MPa, $0.1\ \mu\text{m}$) the stiffness decreases abruptly to a behavior characteristic of the macroscopic loading stiffness in Fig. 2. Therefore, if the second regime in Figure 5 was extrapolated to 3 MPa, the displacement would be in the $1\ \mu\text{m}$ to $2\ \mu\text{m}$ range.

The unloading stiffness appears to evidence a value similar to the initial loading compliance. If the frictional model presented by Guiu et al. (1992) accurately describes the grain-bridging mechanism then we would expect a transition point during the unload cycle, similar to the one noted during the loading portion of the cycle. When frictional mechanisms prevail, the presence of a lower transition point characterizes an abrupt decrease in stiffness again and the displacements return to zero, closing the loop. Instead, Fig. 5 indicates a permanent residual displacement. For this particular load-unload cycle

the specimen experiences an increase of approximately $0.11 \mu\text{m}$ in total crack-face separation.

A paper in press at *Acta Met. et. Mater.*, acknowledging AFOSR Support (Hay and White, 1997), presents the effect of starting damage on the transition point and stiffness, but that work may be summarized by stating that with repeated load cycling, the critical load and displacement defining the transition point increase slowly, evidencing some sort of strengthening mechanism. Therefore, under controlled load conditions the contribution to total residual displacement decreases with each cycle. That work also confirms the LIDG technique for measuring crack-face separations by measuring the COD before and after 12 load cycles in the SEM. Both methods indicate an increase in total crack-face separation of roughly 400 nm after 12 cycles.

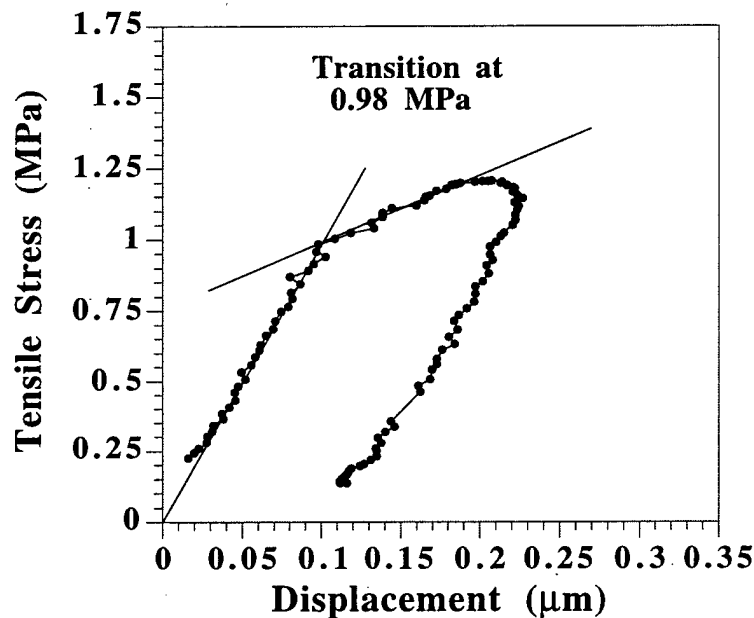


Fig. 2 A typical load-unload cycle for PFT#2.

Modeling Effort

The micromechanical modeling effort, focused largely at the University of Washington, has produced a paper accepted by *International Journal of Fracture*. The first generation model has been developed to the point of requiring the high temperature capability for further development. Last summer, we replaced the Moire method with a speckle interferometry method, using reflections directly from the specimen surface, without the need for any attached surfaces. The student working on this project, D. Tran,

has been developing this system over the last 8 months, and is meeting with some unforeseen difficulties, largely in the purchased software. We expect to resolve these issues by May of 97.

The entire effort during the past six month was expended in setting up a phase-shifted, electronic speckle pattern interferometry (ESPI) system for characterizing the fracture process zone of a ceramic fracture specimen at high temperature. ESPI, which does not require a grating, was chosen over the proposed moire interferometry due difficulty in developing a grating which can withstand temperature in excess of 1000 C and remain attached to the specimen. The system consists of a 5-watt Coherent Innova 308 laser which produces a speckle image of the ceramic fracture specimen. This speckle image is recorded by a CCD camera and stored in a frame grabber.

The recorded digitized speckle patterns, prior to and after loading the fracture specimen, are processed by a commercially available software to provide the displacement field. Sensitivity of the in-plane displacement measurement is increased by a dual beam illumination and phase shifting one of the beams. Considerable time was expended in debugging the unproven commercial software. The digital speckle pattern interferometry (DSPI) has finally become operational now.

During the coming six months, a series of experiments will be conducted with high density alumina, wedge-loaded double cantilever (WL-DCB) specimens at 1000 C. The recorded displacement fields associated with stable crack growth in the WL-DCB specimens will be used to drive a finite element model of the specimen in its generation mode to extract the bridging stress along the crack.

The micromechanical modeling effort is being continued by a postdoc. at U of W, where the first generation model will be advanced to include further details of the wake mechanism.

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Hay, J.C., White, K.W. "The Stiffness of Grain Bridging Elements in a Monolithic Alumina," *J. Amer. Ceram. Soc.*, Accepted (1997).

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