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# Semi-Annual Progress Report for the Period Ending September 30, 1988

E. R. Fuller, Jr., R. F. Krause, Jr., M.D. Vaudin, and T. R. Palamides

U.S. DEPARTMENT OF COMMERCE National Institute of Standards and Technology (Formerly National Bureau of Standards) Ceramics Division Gaithersburg, MD 20899

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Prepared for U.S. Department of Energy Fossil Energy Advanced Research & Technology Development Fossil Energy Materials Program Oak Ridge National Laboratory Oak Ridge, TN

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Prepared for U.S. Department of Energy Fossil Energy Advanced Research & Technology Development Fossil Energy Materials Program Oak Ridge National Laboratory Oak Ridge, TN

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#### TOUGHENING MECHANISMS IN CERAMIC COMPOSITES

Semi-Annual Progress Report for the Period Ending September 30, 1988 \*

Prepared for U.S. Department of Energy, Fossil Energy Advanced Research & Technology Development Fossil Energy Materials Program Oak Ridge National Laboratory Oak Ridge, TN

Interagency Agreement DE-AI05-800R20679

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#### INTRODUCTION

The achievement of higher efficiency heat engines and heat recovery systems requires the availability of high-temperature, high-performance structural materials. Structural ceramics and, more recently, ceramic matrix composites have received particular attention for these applications due to their high strength and excellent resistance to corrosion, erosion and thermal shock. Even with these positive attributes, improved reliability and extended lifetime under service conditions are necessary for structural ceramics and ceramic composites to gain wide industrial acceptance. This reliability is only achieved with improved knowledge of in-service damage modes and failure mechanisms, and the processing knowledge to improve this performance by microstructural modifications. The inherent problems are mechanical and chemical in nature and are enhanced by the high temperatures, reactive environments, and extreme thermal gradients and thermal cycling, to which these materials are subjected.

With an objective of improved performance for heat engine/heat recovery applications, the NIST program addresses these problems through a determination and characterization of major toughening mechanisms in ceramic composites, examining both model crack-fiber systems and "real" composites. A key aspect of the program is a determination of the critical processing factors which influence microstructure and interfacial behavior in these materials, and which thereby influence these toughening mechanisms. The activities of the program are grouped under two major work areas, each designed to develop key data, associated test methods and companion predictive models. The status of these two work areas are detailed below.

\* Also in ORNL/FMP-88/2, Fossil Energy Materials Program, R. R. Judkins, Manager, Oak Ridge National Laboratory, Oak Ridge, TN, 1988.

#### DISCUSSION OF CURRENT ACTIVITIES

NIST-1.1. Crack-Fiber Interactions.

#### Novel Technique for Characterizing Fiber Bridging in Ceramic Composites

<u>Summary</u>. A fracture mechanics specimen known as the double-cleavage drilled-compression (DCDC) specimen has been used to study crack-fiber interactions and toughening increments in a model composite system of SiC monofilaments in a borosilicate glass matrix. The toughening increments were measured from changes in applied stress intensity factor as a function of crack length and number of monofilament fibers. Both the fiber-matrix debond strength and the interfacial frictional shear resistance, which influence these toughening increments, were measured independently by a single fiber pull-out test and were correlated with the toughness increases measured by the DCDC specimen.

Background. Fiber-matrix interfacial properties and the way in which a fiber debonds control the failure mode of ceramic matrix composites. Under tensile loading conditions, these materials generally fail by one of two mechanisms<sup>1,2</sup>: brittle fracture with no influence from the reinforcing fibers or fiber pull-out with either partial or general fiber bridging. When such a crack reaches a fiber, two possible situations can generally develop. The first is that the crack propagates directly through the fiber producing no modification in overall toughness. This results in a brittle failure, although there may be an increase in strength. The second is that upon impingement, the crack deflects along the fiber-matrix interface, bows around the fiber, and rejoins ahead of the fiber<sup>3</sup>. Subsequently, the fiber pulls out of the matrix, inhibiting the free separation of the crack surfaces. In some situations, this subsequent fiber bridging can be interrupted by fiber fracture, resulting in only partial bridging.

The principal physical properties that determine which behavior occurs are the debond strength and the frictional shear resistance of the fibermatrix interface. These properties are determined both by the components of the composite, the matrix and the fiber, and by the processing conditions that combined these components. In the present studies, these

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properties and the toughening increment that they impart to a crack are measured by a single fiber pull-out test and by a fracture mechanics tests, respectively. The fracture mechanics tests uses a specimen, known as the double-cleavage, drilled-compression specimen, into which SiC monofilaments have been introduced perpendicular to the prospective crack plane.

Experimental Procedure. Double-cleavage, drilled-compression (DCDC) specimens were fabricated by sandwiching SiC monofilaments [ACVO SCS-6 SiC] between borosilicate glass plates [Pyrex glass, #7740], which were either 3.2 mm or 1.6 mm thick. Using a combination of glass thicknesses, various numbers of fibers could be incorporated into the matrix as simple arrays without a significant change in billet geometry. The specimens were diffusion-bonded by heating in an atmosphere of helium under a slightly positive gas pressure. To ensure proper fiber alignment and specimen reproducibility the glass plates were placed in a graphite mold which had guide grooves for the fibers. To aid the diffusion-bonding process, a dead weight load was applied to the composite billet to provide a hot-pressing pressure of 1.2 MPa.

The general heating cycle consisted of bringing the specimen slowly up to 660°C, holding for 3.5 h, heating to 705°C, holding for a specified time (as discussed below), and furnace cooling to room temperature. The maximum processing temperature of 705°C is above the annealing temperature of borosilicate glass (565°C), but not above the softening point (820°C). This temperature was selected to minimize sufficiently devitrification of the glass and to maintain billet dimensions and shape, while still achieving adequate bonding between the plates. The hold time at 705°C was varied according to the number of fibers which were incorporated into the billet: 135 min for one fiber; 120 min for two fibers; and 90 min for three fibers. This varied and complex heating schedule was required to suppress devitrification on the diffusion-bonded interface. Devitrification was more of an issue when the thinner (1.6 mm) glass plates were used, possibly due to the additional free surface area needed per billet or to a different surface texture on the thinner plates.

This fabrication sequence resulted in a small billet, which could be sectioned into three DCDC specimens. Following sectioning, a hole was drilled into the center of each specimen, and the specimens were polished.

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The hole was placed so that the monofilaments were located symmetrically about the hole with their axes perpendicular to that of the hole; thus, the plane of the mode I crack was perpendicular to the fiber axis.

The specimens were loaded in compression at a crosshead displacement rate of 0.05 mm/min, using a screw-driven mechanical testing machine with a 50 kN load cell. When properly loaded, two symmetric mode I cracks emanated from the top and bottom of the center hole. The cracks typically initiated at an applied stress of about 70 MPa and propagated unstably for a distance of approximately 1.25 to 2.5 hole radii. A unique feature of the DCDC specimen is that the stress intensity factor,  $K_I$ , decreases with crack length, c, following the initial crack pop-in. A consequence of this is that an increasing stress must be continually applied to advance the cracks. The crack speed is accordingly controlled by the stressing rate.

For a range of crack lengths, the applied compressive stress,  $\sigma_{app}$ , crack length, and stress intensity factor are related by:

$$(\sigma_{app}/r)/K_{I} = g(c/r) = \alpha + \beta \cdot (c/r), \qquad (1)$$

where is the r is the hole radius, g(c/r) is a dimensionless geometric function, and  $\alpha$  and  $\beta$  are geometric constants. In this range, a constant stressing rate extends the cracks at a constant speed. In addition, the stiffness of the DCDC specimen compared to that of typical load cells results in a constant stressing rate test when the crosshead is run at a constant displacement rate.

The dimensionless geometric function, g(c/r), was experimentally determined for the present testing configuration from specimens which contain no fibers. These "blank" specimens were fabricated in a similar manner to the composite specimens. The range of crack lengths over which Eqn. (1) was calibrated was approximately  $3 \cdot (c/r)$  to  $18 \cdot (c/r)$ . In this range the slope,  $\beta$ , was approximately the same for various specimens, but there was noticeable scatter in the intercept,  $\alpha$ . This may result from specimen misalignment or frictional effects at the ends of the specimen. This vertical shift in the calibration line is not of great concern since data for the toughening increment from various fiber arrays were normalized to the value of  $K_{IC}$  for glass measured on the same specimen. A linear

(4)

regression of the data for six calibration specimens gave a slope  $\beta$  of 0.17 and an average intercept value  $\alpha$  of 1.75.

During testing, the positions of the crack tips were monitored by standard photography using a cross-polarized illumination system with quarter wave enhancement plates to increase field brightness. The strain contours associated with these cracks were symmetric about the center hole. The position of the crack tip was taken as the nodal point of the strain contours. The double cracks were approximately equal in length. The technique of cross-polarized illumination gives a good value for the average crack-tip position because the strain contours are an average of the entire crack front, which is slightly curved for this type of specimen<sup>4</sup>.

To evaluate effects of processing on both bond strength and subsequent frictional shear resistance of the fiber-matrix interface, an independent single fiber pull-out test was developed; a schematic of the test configuration is shown in Fig. 1. With this experimental design an embedded fiber could be pulled from the matrix of the DCDC specimen directly without the need for further specimen preparation. This avoided any undesirable contamination of the fiber-matrix interface, which could result, for example, from the specimen polishing required for a fiber push-in test.

Once a DCDC specimen was fully cracked, it was physically pulled apart by hand. What remained was a 6.0 mm fiber, half of which was protruding from the glass matrix. A small metal capillary tube was filled with epoxy and bonded to the 3.0 mm protruding segment. Bonding to the glass matrix was avoided with a separating sheet of thin plastic. The remaining part of the test jig consisted of high-strength stainless steel wire and swivels, which minimized the potential for misalignment. The samples were tested in tension at a crosshead displacement rate of 0.10 cm/min on a screw-driven mechanical testing machine with a 50 N load cell.

<u>Results and Discussion</u>. The change in applied stress intensity factor,  $K_I$ , as a crack propagated pass a fiber array was monitored for linear fiber arrays of one, two, and three fibers. The results give the toughening increment,  $\delta(K_I)$ , for the bridging influence of that fiber array. Results for a single fiber are plotted in Fig. 2. The data for two specimens from the same billet were plotted in this figure. Although the data do not precisely superimpose, the reproducibility is reasonable.

(5)

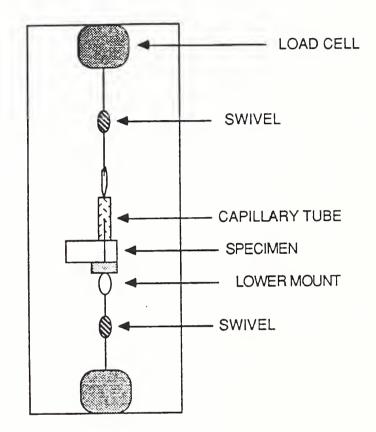
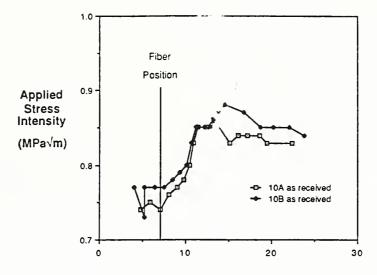


Fig. 1. Schematic illustration of the single fiber pull-out test used to measure the debond strength and frictional shear resistance of the fiber-matrix interface from fibers protruding from half of a double-cleavage, drilled-compression specimen.



Crack length/ Hole radius (c/r)

Fig. 2. Applied stress intensity factor, or fracture toughness, versus normalized crack length for two double-cleavage, drilledcompression specimens taken from the same billet. The toughness increase as the crack traverses the fiber is a measure of the incremental toughening from a single bridging fiber. The figure also illustrates the degree of reproducibility between specimens.

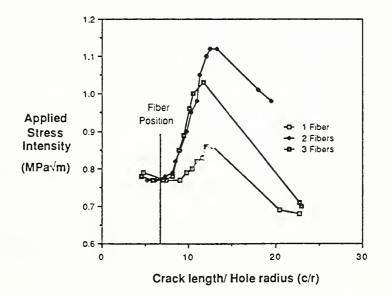


Fig. 3. Applied stress intensity factor versus normalized crack length for double-cleavage, drilled-compression specimens which contain a one-, two-, or three-fiber array at each end of the specimen. The reversal in magnitude of toughening increment for the twoand three-fiber specimens is consistent with the interfacial properties measured for these two specimens. See Table 1. Initially, the applied stress intensity factor increased as the crack passed the fiber. At a distance of about 8-10 fiber radii past the fiber the applied stress intensity factor decreases indicating that the fiber is being steadily pulled from the matrix.

Representative data for DCDC specimens with monofilament arrays of one, two and three fibers are plotted in Fig. 3. Similar trends to the data in Fig. 2 were observed. The major exception is the lack of data points for crack lengths between approximately  $12 \cdot (c/r)$  and  $18 \cdot (c/r)$ . In the figure the bounding data points have been connected by a straight line (linear decrease), which is probably not an exact representation of the toughening increment in this region. Due to the rapid acceleration of the crack, measurement of the stress and crack length in this region of these specimen was difficult. Most likely, the applied stress intensity factor dropped rapidly as the fiber(s) debonded or were pulled from the matrix.

The specimen which showed the largest increase in toughness was the specimen with two-fiber array. This observation was not expected because increasing the number of fibers in a given specimen cross-section is equivalent to increasing the fiber volume fraction and the toughening increment should increase with a larger volume fraction. Examining the fracture surfaces for the two and three fiber specimens, one sees a smooth planar fracture, indicative of mode I cleavage. There was some evidence of slight devitrification at the diffusion-bonded layer between the glass plates, but close observations of the interfacial region next to the fiber indicated such devitrification did not greatly influence the fiber-matrix interface.

Representative scanning electron micrographs of the fiber-matrix interface region are shown in Figs. 4a & 4b for DCDC specimens which contained two and three fiber arrays, respectively. The interface appears well bonded except for a small tapered void along the glass plate interface. The void for the two fiber specimen appears to be smaller than that for the three fiber specimen. This may indicate that the fibers in this specimen were more securely bonded than in the three fiber specimen, or that the greater fiber-matrix contact area resulted in a higher frictional pull-out resistance. The specimens were processed for different times at the maximum temperature to suppress devitrification along the interface, so that different interfacial properties are entirely possible.

(8)

(a)

(b)

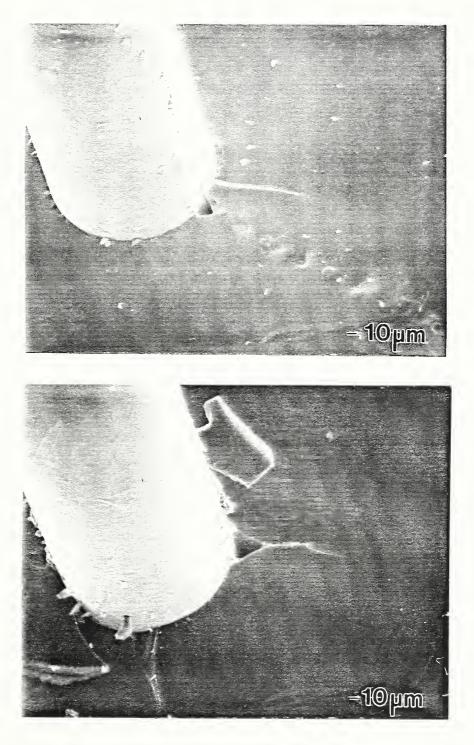


Fig. 4. Scanning electron micrographs of representative monofilaments from double-cleavage, drilled-compression specimens which contain a (a) two - and (b) three-fiber array at each end of the specimen. Such a speculation was supported by the single fiber pull-out tests. Measured values of the frictional shear resistance (debond strength) for the one fiber specimens ranged from 3.3 to 4.1 MPa (3.7 to 5.3 MPa), those for the two fiber specimen ranged from 3.7 to 4.9 MPa (5.3 to 6.4 MPa), and those for the three fiber specimens ranged from 3.2 to 3.7 MPa (5.4 to 8.2 MPa). Average data are shown in Table 1. Thus, the interfacial properties, specifically frictional shear resistance, of fibers in the one and three fiber specimens appear were similar, while those for the two fiber specimens were significantly greater. This increased frictional resistance resulted in an enhanced toughening increment. The final column of Table 1 confirms this. The percentage change in the toughening increment normalized by the concentration of fibers indicates a 22% increase for the two fiber specimen over that of an unreinforced matrix, while the one and three fiber specimens exhibit only an 11% enhancement.

<u>Conclusions</u>. The fiber-reinforced DCDC (double-cleavage, drilledcompression) specimen is a simple and versatile testing configuration for obtaining useful information about the interaction of cracks with reinforcing fibers in ceramic matrix composites. The single fiber pull-out test is a complementary technique for independently measuring the fiber-matrix interfacial properties. The combined testing procedure provides a systematic means for investigating relationships between processing, fiber-matrix interfacial properties and enhancements in material toughness.

Number of Fibers	Time at 705°C (min)	Debond Strength <sup>a</sup> (MPa)	Frictional Shear <sup>a</sup> Resistance (MPa)	$\delta(K_I)/K_{IC}$ per fiber
1 Fiber	135	$4.2 \pm 0.7$	3.7 ± 0.3	11%
2 Fiber	120	$6.0 \pm 0.5$	$4.5 \pm 0.6$	22%
3 Fiber	90	6.6 ± 1.4	3.4 ± 0.3	11%

Table 1. Processing conditions, average interfacial properties, and normalized toughening increment for one, two, and three fiber specimens.

Average and standard deviation of four tests on 1- and 2-fiber specimens and of three tests on the 3-fiber specimens. NIST-1.2. Real Ceramic Composite Systems.

Creep Rupture Behavior of a SiC Whisker-Reinforced Alumina Composite

<u>Summary</u>. A ceramic composite material that has received much attention because of its increased toughness<sup>5</sup> and creep resistance<sup>6</sup> compared with alumina is aluminum oxide reinforced with silicon carbide whiskers. In this study, the creep and creep rupture behavior of a 25 wt% SiC whisker-reinforced alumina ceramic with 4.9% porosity were measured at temperatures between 1100°C and 1300°C and at applied stresses between 55 and 306 MPa, although the applied stresses at each temperature varied over a much narrower range. Creep strains were determined from loading-point displacement measurements in four-point flexure.

In the current reporting period, several long term tests were completed. Creep and creep rupture data were measured over a wide range of creep strains (0.05% to 2.0%) and failure times (0.3 to 1600 h). Failure times occur in two apparently different creep regimes: one characterized by a creep-hardening type behavior (higher applied stresses) and another in which this creep-hardening regime was followed by a long steady state regime (lower applied stresses). The transition in behavior from that at high stresses to that at low stresses is marked by a large change in failure time with only a small change in applied stress, for example, at 1300°C the failure time increases by a factor of 35 for a 16% decrease in stress. Transmission electron microscopy studies have shown that ruptured specimens from the low and high stress regimes do not display markedly different microstructural characteristics. The microstructure of the material has been modeled as an arrangement of elastic and viscous elements, and the experimental data was found to behave approximately as predicted for such a model.

Experimental Procedure and Results. The material studied was a 25 wt% (29 vol%) SiC whisker-reinforced alumina ceramic with 4.9% porosity. The material was provided as a billet through the courtesy of Dr. J. F. Rhodes of the Advanced Composite Materials Corporation. The techniques of creep specimen preparation and creep data collection have been described in the previous semiannual report<sup>7</sup>.

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Three types of samples were prepared for transmission electron microscopy (TEM) studies: 1) material from the as-received billet; 2) material from the tensile region of specimens ruptured at  $1300^{\circ}C$ ; and 3) material from the approximately 50  $\mu$ m thick scale of the specimen tested at  $1300^{\circ}C$ and 72 MPa. The samples were cut as thin slices with a diamond saw. The samples from the ruptured specimen were cut parallel to the tensile surface. Disks 3 mm in diameter were ultrasonically cut from the slices and the disks were ground, dimpled, and ion-milled to perforation. TEM observations were made using conventional TEM, high resolution imaging, and selected area electron diffraction, operating at 120 and 300 kV.

The creep experiments, in progress at the time of the previous report, have either been completed or terminated. Some experiments lasted up to 1600 h (9.5 weeks). Table 2 gives a summary of the results. Two apparently different regimes of creep behavior were observed, as reported in the previous semiannual report<sup>7</sup>. Examples of both types of behavior are illustrated in the log(strain) versus log(time) data plotted in Fig. 5 for creep at 1300°C. For high stresses, the graph of log(strain) versus log(time) is approximately linear right up to the time of rupture. Such a creep response is typified by a creep-hardening behavior, which can be represented by:

$$\epsilon = Bt^{a}, \qquad (2)$$

where a and B are constants. Both time-to-failure and strain-to-failure in this regime <u>increased</u> with <u>decreasing</u> applied stress. This type of behavior was observed by Porter<sup>8</sup> at much higher temperatures ( $1500^{\circ}C$ ).

For low stresses, failure occurred during a long period of steadystate creep behavior following the creep-hardening regime. Steady-state strain rates in this regime were extremely small (as low as  $10^{-11}$ /s, which is the limit of the present measurement resolution). Several experiments at the smallest creep rates were terminated before failure; accordingly, there is a sparsity of data in the low stress regime. Although more data is needed, the strain at failure appears to <u>decrease</u> and the failure time to <u>increase</u> with <u>decreasing</u> applied stress.

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Table 2. Creep rupture behavior of a 25 wt% SiC whisker-reinforced alumina composite with 4.9% porosity. Failure times and strains with a ">" symbol indicate the interrupted value for specimens which had not yet ruptured.

Temperature °C	Stress <sup>a</sup> (MPa)	Failure Time (hours)	Failure Strain <sup>a</sup> (percent)
1000	202	>359	>0.04
	301 <sup>b</sup>	>1229	>0.23
1000	249	>359	>0.04
	374 <sup>b</sup>	87	0.01
1100	231	139	0.18
	306	25.1	0.17
1116	200	865	0.53
	250	149	0.36
1124	126	>1588	>0.32
	149	>1588	>0.52
1145	175	262.4	0.62
	200	64.1	0.39
	224	35.6	0.39
	248	24.9	0.34
1200	123	>1170	>0.89
	124	>290	>0.45
	132	>52	>0.68
	147	60.8	1.18
	149	61	1.04
	174	19.9	0.83
	174	27.2	0.87
	198	12.3	0.70
	200	7.5	0.73
	249	0.32	0.15
1245	82	>756	>0.82
	98	161.7	1.14
	122	27.0	1.86
	147	7.5	1.17
1300	55	>1012	>0.31
	62	1001	1.07
	72	27.6	1.85
	98	5.1	1.96
	98	14	1.61
	124	16	0.87

<sup>a</sup> Value calculated assuming a power-law-creep, stress exponent of unity.

<sup>b</sup> Creep test continued at the higher applied stress from previous specimen.

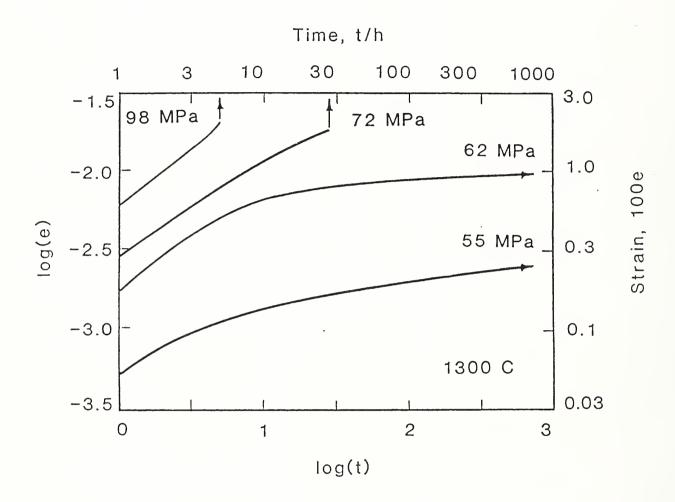


Fig. 5. Logarithmic creep data at 1300°C [logarithm of creep strain versus logarithm of time] for flexural specimens of a 25 wt% SiC whisker-reinforced alumina composite with 4.9% porosity. Note the change in behavior between applied stresses of 62 and 72 MPa.

Optical microscopy of the as-received composite showed that the material consisted of regions 20  $\mu$ m to 80  $\mu$ m in diameter containing a relatively high density of whiskers separated by narrow bands about 5  $\mu$ m wide with a lower whisker fraction. TEM examination of the as-received material revealed intragranular pores in the Al<sub>2</sub>O<sub>3</sub> and cavities at a few of the junctions between whiskers and the Al<sub>2</sub>O<sub>3</sub> grain boundaries, accounting for the 4.9% porosity. The whiskers are single crystals of  $\beta$ -SiC aligned along the <111> direction and faceted on {112} planes, as originally reported by Nutt<sup>9</sup>, and the cavities were frequently found to be triangular prisms in shape, bounded by a (112) SiC plane and two low-index Al<sub>2</sub>O<sub>3</sub> planes, for example, a (0001) plane. The cavitation between adjacent whiskers in the material was often more pronounced than next to isolated whiskers.

TEM observations were made on the microstructure of specimens tested at 62 MPa and 1300°C, and at 72 MPa and 1300°C with failure times/strains of 1001 h/1.07% and 28 h/1.8%, respectively. These specimens come from low and high stress regimes, respectively, and their failure times differ by a factor of 35, although the applied stresses differ by only 16%. Despite the large difference in time-to-failure between these two specimens, there was little difference between the observed microstructures. Figs. 6a and 6b are taken from the tensile regions of the specimens and both show considerably increased cavitation at the whisker/grain boundary triple junctions compared with the as-received material. The cavities were frequently lined with a thin layer of glass as seen clearly in Fig. 6a taken from the lower stress specimen; the glass is of the order of 50 nm thick and is adhering to both phases. The Al<sub>2</sub>O<sub>3</sub> triple junctions displayed a much lower level of cavitation and glass decoration. Cavitation was the principal evidence of deformation observed in these specimens. Little dislocation activity was observed in the specimens beyond small angle grain boundaries in a few of the larger  $Al_2O_3$  grains. The specimens were not removed from the furnace immediately after failure; in some cases the specimens were held at test temperature for several hours after failure, so it is possible that any dislocations generated during creep were annealed out of the  $Al_2O_3$  grains before the dislocation structure was frozen in.

Since the specimens were tested in air, reaction layers (or scales) formed at their surfaces; previously polished surfaces had their finishes

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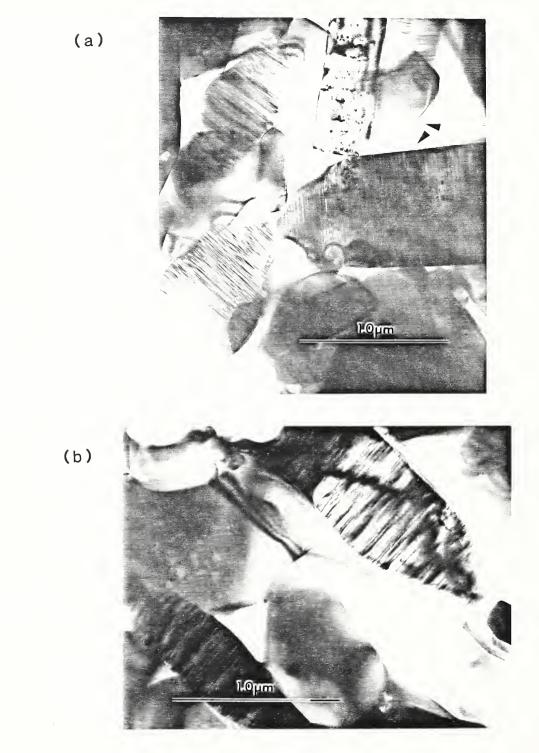


Fig. 6. Transmission electron micrographs of creep specimens tested to failure at 1300°C and applied stresses of (a) 62 MPa and (b) 72 MPa. The time-to-failure was 1,001 h at 62 MPa, but only 28 h at 72 MPa. Both microstructures show a high degree of cavitation at whisker/grain-boundary triple junctions. dulled. Numerous pits were formed on the surface, which were on the order of 20 to 40  $\mu$ m in diameter. The pits obscured microindents which had been placed on the side of the creep specimen to monitor any shifts in the neutral strain axis during creep. The microstructure of the scale close to the scale/composite interface where the specimen was taken consists of grains of alumina dispersed in a siliceous glass containing some Al and Ca, with occasional colonies of thin tabular mullite grains,  $3(Al_2O_3) \cdot 2(SiO_2)$ .

<u>Discussion</u>. The high-stress creep data and the primary portion of the low-stress creep data are in general well described by Eqn. (2) as can be seen from the practically linear plots of Fig. 5. A linear least-squares fit of the data to the logarithm of Eqn. (2) showed that the time exponent, a, lies in the range 0.35 to 0.75 and appears to remain fairly constant over a range of applied stresses at a given temperature. In general B increases with increasing stress at a given temperature.

An increase in cavitation was observed by TEM to be the principal deformation mechanism, within the limitations on dislocation observation noted above. There was little difference in microstructure between the two specimens observed despite their considerable differences in deformation behavior with time. Both specimens were studied after failure, in contrast to the observations of Lipetzky et al.<sup>10</sup> who interrupted their creep experiments to make TEM observations once steady state had been reached. They found a change from glass pocket formation at grain boundary-interface junctions in a specimen from the low stress regime (35 MPa at 1300°C) to cavitation in a specimen in the high stress regime (100 MPa at 1300°C). Cavitation was observed in the present study in both stress regimes and also in the as-received material. Comparison of the two sets of microstructural observations is difficult because our specimens had failed whereas those of Lipetzky et al. had not, the starting materials had different porosities and the applied stresses differed considerably between the two studies. More systematic studies (preferably in tension) of cavitation as a function of strain are needed to determine the reasons for the apparent change in creep behavior between the low and high stress regimes. In addition, since the cavities observed were large enough to be visible in a scanning electron microscope, SEM studies of polished sections

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taken from creep specimens would provide quantitative information (unobtainable in TEM) on the distribution of cavities in the material.

Porter<sup>8</sup> has suggested that these composite materials, in their response to an applied stress, can be modeled as an elastic element (the network of SiC whiskers) in parallel with a viscous element (the alumina matrix); this Kelvin-Voigt compound model is in series with another viscous element representing the non-recoverable deformation of the composite which is caused by the incomplete nature of the whisker network and by broken whiskers. The strain-time response,  $\epsilon(t)$ , of such a model under load is:

$$\epsilon = k_1 \cdot [1 - \exp(-t/\tau)] + k_2 t, \tag{3}$$

where  $k_1$ ,  $k_2$  and  $\tau$  are constants related to the applied stress and the elastic and viscous constants of the model. This equation has been fitted to the data with partial success, although Eqn. (2) fits the primary creep data better.

Other models can be proposed. For instance the elastic element in the above model could be replaced with a perfectly plastic element which is rigid below a critical stress and flows at that stress; this element would attempt to represent the behavior of a  $SiC/Al_2O_3$  interface with a glass coating. Alternatively, another elastic element could be inserted into the Kelvin-Voigt element in series with the dashpot to represent the elastic response of the alumina. Whatever mechanistic model is used to analyze the data should successfully match the complete creep behavior, including the primary creep, which is so well described rheologically by Eqn. (2). The fact that the initial regions of the plots in Fig. 5 are linear with slopes between 0.35 and 0.75 as discussed above suggests a modification to Eqn. (3) in which the argument of the exponent is raised to some power, a, as determined by fitting Eqn. (2) to the data.

One possibility is that the flexural test itself modifies the creep response. Collaborative research has been initiated with S. M. Wiederhorn of NIST to obtain tensile creep data on this material and with T.-J. Chuang of NIST to analyze the flexural data based upon tensile and compressive rheological models, such as Eqn. (3).

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<u>Conclusions</u>. The creep data presented here for SiC whisker reinforced  $Al_2O_3$  show two apparently different regimes of response to a range of applied stresses at a given temperature. Transmission electron microscopy of ruptured specimens in the two regimes i dicates no major microstructural differences between them. The creep data have been fitted to a rheological equation that is based on a reasonable viscoelastic model of the composite material with partial success.

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FORM NB5-114A (REV.11-84 1. PUBLICATION OR U.S. DEPT. OF COMM. 2. Performing Organ. Report No. 3. Publication Date REPORT NO. **BIBLIOGRAPHIC DATA** NISTIR 88-4018 SHEET (See instructions) February 1989 4. TITLE AND SUBTITLE Toughening Mechanisms In Ceramic Composites 5. AUTHOR(S) E. R. Fuller, Jr., R. F. Krause, Jr., M. D. Vaudin, and T. R. Palamides 6. PERFORMING ORGANIZATION (If joint or other than NBS, see instructions) 7. Contract/Grant No. NATIONAL BUREAU OF STANDARDS 8. Type of Report & Period Covered **U.S. DEPARTMENT OF COMMERCE** GAITHERSBURG, MD 20899 Interm Report 9. SPONSORING ORGANIZATION NAME AND COMPLETE ADDRESS (Street, City, State, ZIP) U. S. Department of Energy Fossil Energy Materials Program Oak Ridge National Laboratory Oak Ridge, Tennessee 10. SUPPLEMENTARY NOTES Document describes a computer program; SF-185, FIPS Software Summary, is attached. 11. ABSTRACT (A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here) A fracture mechanics specimen known as the double-cleavage drilled-compression (DCDC) specimen has been used to study crack-fiber interactions and toughening increments in a model composite system of SiC monofilaments in a borosilicate glass matrix. The toughening increments were measured from changes in applied stress intensity factor as a function of crack length and number of monofilament fibers. Both the fiber-matrix debond strength and the interfacial frictional shear resistance, which influence these toughening increments, were measured independently by a single fiber pull-out test and were correlated with the toughness increases measured by the DCDC specimen. A ceramic\_composite material that has received much attention because of its increased toughness<sup>5</sup> and creep resistance<sup>6</sup> compared with alumina is aluminum oxide reinforced with silicon carbide whiskers. In this study, the creep and creep rupture behavior of a 25 wt% SiC whisker-reinforced alumina ceramic with 4.9% porosity were measured at temperature between 100°C and 1300°C and at applied stresses between 55 and 306 MPa, although the applied stresses at each temperature varied over a much narrower range. Creep strains were determined from loading-point displacement measurements in fourpoint flexure. 12. KEY WORDS (Six to twelve entries; alphabetical order; capitalize only proper names; and separate key words by semicolons) bridging; ceramics; composites; cracks; creep; creep rupture; fibers; fracture toughness; interfaces; toughening; whiskers 13. AVAILABILITY 14. NO. OF PRINTED PAGES X Unlimited For Official Distribution. Do Not Release to NTIS 23 Order From Superintendent of Documents, U.S. Government Printing Office, Washington, DC 15. Price 20402. X Order From National Technical Information Service (NTIS), Springfield, VA 22161 \$9.95

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