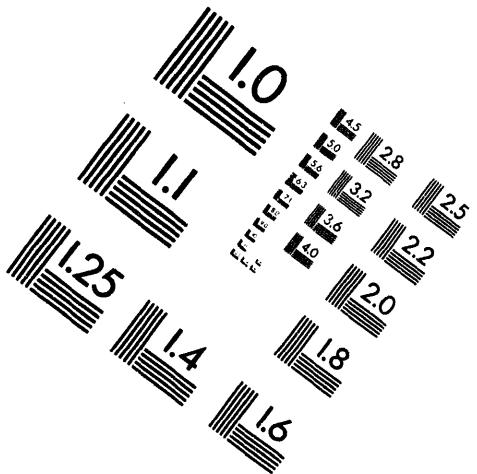
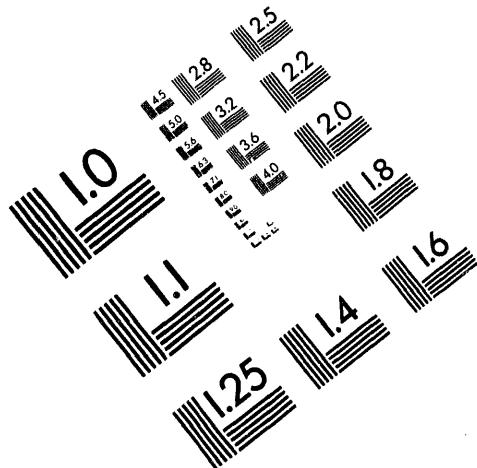




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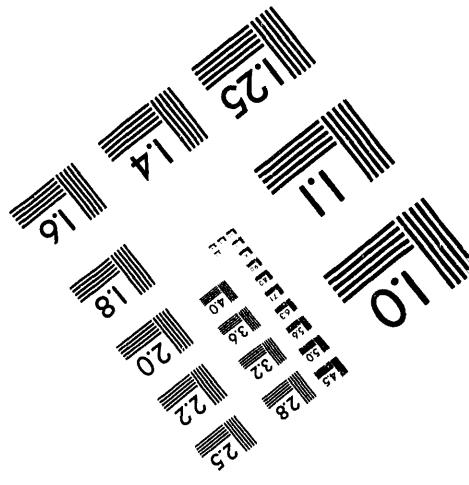
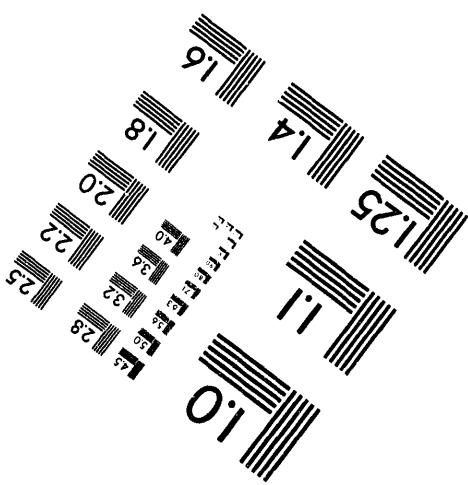
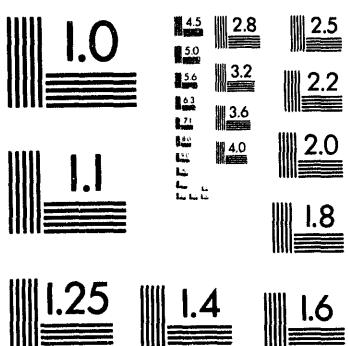
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# PROPERTIES OF WHISKER-REINFORCED CERAMICS

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## Abstract

The paper summarizes the findings of a series of studies of SiC whisker-reinforced alumina composites emphasizing the properties of these composites and the influence of composition and microstructure. First, both the steady-state toughness and the R-curve response increase with whisker content and diameter. The room temperature flexure strength of aluminas with different grain sizes also increases with whisker content. Furthermore, the resistance to slow crack growth under both monotonic and cyclic loading is substantially improved by whisker reinforcement in comparison to that of alumina and other ceramics. The thermal conductivity and electrical resistivity exhibit a substantial increase and decrease, respectively, with whisker loadings above 10 vol.%. The thermal expansion coefficients decrease in a systematic fashion with increase in whisker content. However, the expansion coefficients are greater in the direction parallel to the hot pressing axis as compared to the perpendicular direction due to internal stresses and the preferred orientation of whiskers. The changes in mechanical and thermal properties due to SiC whisker additions result in substantial improvement in thermal shock resistance for samples of various sizes. At elevated temperatures, tests in air show that improvements in strength and resistance to strength degradation are associated with SiC whisker reinforcement. The limiting factors in elevated temperature mechanical response of the composite at 1200°C and above are related to surface oxidation-reaction processes and creep. On the other hand, the creep resistance of the composite is far greater than alumina with a similar fine grain size at temperatures  $\geq$  1200°C. Furthermore by increasing the grain size of the alumina matrix, the creep resistance of the composite can be substantially increased. In essence, SiC whisker-

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*reinforced alumina composites provide an example of a system whose properties can be tailored through control of the microstructure and composition.*

## **Introduction**

The brittle nature and low fracture toughness found with most ceramics have led the ceramics community to seek different approaches to improving overall mechanical performance. The introduction of continuous fibers, whiskers, platelets or elongated grains offers one approach to increase the toughness of various ceramics. The toughening mechanisms involve formation of a zone behind the crack tip in which the reinforcing phase bridges the crack and is subsequently pulled out of the matrix. The manner (i.e., magnitude and rate) in which toughening effects develop as a small crack grows can influence the degree to which the resistances to fracture, thermal shock, impact damage, and slow crack growth are improved. In order to assess this, it is useful to look not only at the fracture toughness and R-curve (rising fracture resistance with crack extension) behavior, but also to determine a variety of mechanical properties for the same composite system.

Earlier studies showed that the incorporation of strong microscopic whiskers into a ceramic matrix can result in a three- to four-fold increase in steady-state (long,  $\geq 1000 \mu\text{m}$ , cracks) fracture toughness.<sup>1-3</sup> Certain whisker criteria can be used to optimize the toughening contribution. For instance, increases in the whisker diameter are beneficial in promoting frictional bridging and pullout processes. Similar effects are seen in silicon nitride ceramics where elongated  $\beta$ -phase grains can also bridge cracks<sup>4,5</sup> and increases in diameter of these larger elongated grains enhance the toughening effect.<sup>6,7</sup> Reinforcement morphology also plays a role. Smooth, straight whiskers are a more effective

reinforcement. In alumina containing 20 vol% SiC whiskers a steady-state toughness of  $\sim 8.5 \text{ MPa}\sqrt{\text{m}}$  was achieved with smooth  $\sim 0.7 \mu\text{m}$  diameter whiskers versus  $\sim 6.5 \text{ MPa}\sqrt{\text{m}}$  for  $\sim 1.5 \mu\text{m}$  diameter whiskers with a very rough, corrugated surface morphology.<sup>8</sup> This can be a result of increased whisker strength by minimizing stress concentrations that reduce whiskers' strength. A smooth interface associated with such whiskers also circumvents mechanical interlocking of the interface. As a result, both interfacial debonding and pullout should be enhanced.

### **Toughening Response: Whisker Reinforcement**

As we shall see, crack bridging processes evolve during crack growth. This leads to an increase in toughness with increase in crack length.<sup>9,10</sup> Immediately behind the crack tip, a bridging stress associated with elastic stretching of the whisker develops. Both the bridging stress and the corresponding interfacial shear stress increase rapidly with increasing distance from the crack tip. When the interfacial shear stress becomes sufficiently high, interfacial debonding occurs, and frictional bridging commences when the debonded whisker remains in contact with the matrix. Contact between the debonded whisker and matrix will be promoted when the matrix has the larger thermal expansion coefficient or when the whisker is inclined to the crack plane. The frictional bridging stress in the partially debonded whisker now increases at a somewhat slower rate (i.e., than that for an elastically bridging whisker) due to load transfer to the matrix. Thus, a frictional bridging whisker fractures further behind the crack tip and is accompanied by pullout when the point of fracture is not in the plane of the main crack.

The stress intensity factor associated with bridging processes along a line crack,  $K$ , is:<sup>11,12</sup>

$$K = K_0 + \left(\frac{2}{\pi}\right)^{0.5} \int_0^{D_{bz}} \frac{P dx}{x^{0.5}} \quad (1)$$

where  $K_0$  is the intrinsic toughness of the matrix,  $D_{bz}$  is the bridging zone length,  $x$  is the distance behind the crack tip, and  $P$  is the bridging stress profile. At this point, a simple, yet accurate, relationship between the crack opening at position  $x$  behind the crack tip:<sup>13</sup>

$$u = \frac{2(1 - v_c^2) K_0 [D_{bz}^2 - (D_{bz} - x)^2]^{0.5}}{(\pi D_{bz})^{0.5} E_c} \quad (2)$$

where  $E_c$  is the Young's modulus and  $v_c$  is the Poisson's ratio of the composite will be used in establishing the definitions for the bridging stress profiles and zone lengths for each bridging mechanisms. Here, the results for frictional and pullout processes are only summarized; a detailed description is given elsewhere.<sup>10</sup>

The frictional bridging stress as a function of the crack opening displacement is:

$$P_{fb} = 2f \left[ \frac{2\tau u E_w E_c}{d_w (1-f) E_m} \right]^{1/2} \quad (3)$$

and is dependent upon the whisker diameter ( $d_w$ ), Young's modulus ( $E_w$ ) and content ( $f$ ), the frictional shear resistance of the debonded interface ( $\tau$ ), and the Young's modulus of the composite,  $E_c$  and matrix,  $E_m$ . The interfacial shear stress is a product of the interfacial coefficient of friction and radial stress imposed on the interface due to mismatch in thermal expansion between matrix and whisker (compressive for SiC whiskers in alumina) and will decrease with increase in whisker content.<sup>14</sup>

With increase in the crack opening displacement, the pullout bridging stress for a pullout length of  $l_{po}$  decreases as:

$$P_{po} = 4 f \tau \frac{(l_{po} - 2u)}{d_w} \quad (4)$$

as the embedded whisker end is extracted. The total bridging stresses for each process obviously will increase with whisker content.

The overall toughening effect due to whisker bridging is contingent upon the determinations of the sizes of both frictional and pullout bridging zones. One can start by considering the mechanics for uniaxially-aligned whiskers normal to the crack surface and then account for the fact that whiskers are biaxially-aligned. When whiskers are not normal to the crack surface, their resistance to fracture is reduced (i.e., their "effective" strength is reduced) due to bending stresses at the crack surface. For biaxially-aligned whiskers, the average fracture strength is  $\sim 60\%$  of the uniaxial strength,  $\sigma_s$ .<sup>15</sup> The frictional bridging zone length,  $D_{fb}$ , is:

$$D_{fb} = 0.002 \pi \left[ \frac{d_w (1 - \lambda f) E_m \sigma_s^2}{2(1 - v_c^2) K_o \tau E_w} \right]^2 \quad (5)$$

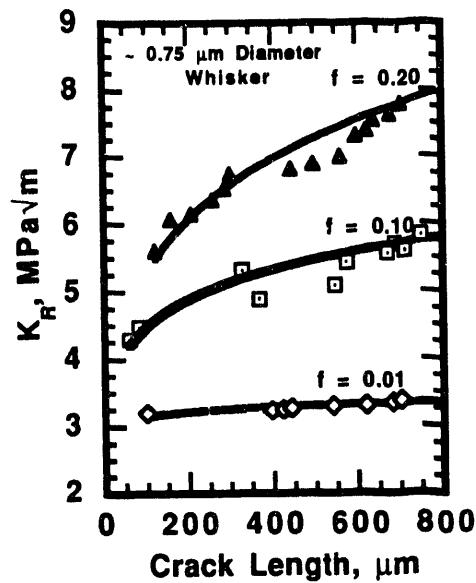
where  $\lambda$  represents the fraction of whiskers that form effective bridges. Then pullout length and crack opening displacement,  $2u$ , can be equated to determine the size of the pullout bridging zone,  $D_{po}$ :

$$D_{po} = 0.0056 \pi \left[ \frac{d_w \beta (1 - \lambda f) E_m \sigma_s}{2(1 - v_c^2) \tau K_o} \right]^2 \quad (6)$$

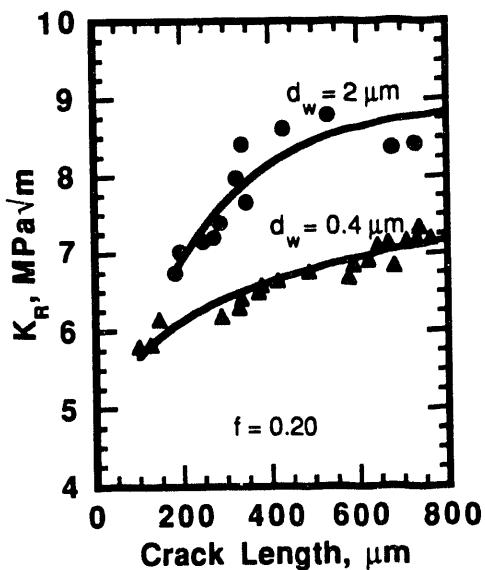
where  $\beta$  is the fraction of the debonded whisker length that is actually pulled out. It is immediately apparent that whisker diameter and strength,

as well as the thermal expansion mismatch with the matrix that controls  $\tau$ , can be used to vary the size of the pullout zone. Furthermore, whisker lengths (and distribution of lengths) will be a factor; this condition is modified as whisker fracture can occur at any point along the debonded length.

Based on the above discussions, it should be possible to tailor the R-curve response of a specific matrix reinforced with whiskers of a given compound, e.g., SiC, by manipulating whisker properties, characteristics, and whisker content. First, frictional bridging will lead to a rapid increase in fracture resistance,  $K_R$ , for small crack extensions. The corresponding rate of increase in  $K_R$  should also rise substantially with whisker content. Secondly, for large crack extensions where pullout dominates, the R-curve rises more slowly with crack extension, but will be shifted upwards with increase in whisker content. Finally, increases in whisker diameter should shift the R-curve upward while extending the frictional bridging zone size. As a result of frictional bridging, the composite with the larger diameter whisker will exhibit a stronger R-curve effect when the crack extends up to 200  $\mu\text{m}$  in length. However, increases in whisker diameter have negligible effects on the rate of increase in fracture toughness where pullout dominates. In fact, the measured R-curves for SiC whisker-reinforced fine grained aluminas are consistent with these predictions, Figure 1. These observations suggest a number of factors (e.g., whisker content, strength, diameter and length, thermal expansion mismatch stress, or interfacial debonding stress) that will modify both how quickly the fracture toughness will increase as a crack extends and the magnitude of increased toughness. One should not, then, expect that all combinations of whiskers and matrix materials will provide the same effects. At the same time, there is some latitude in selecting compositions that might prove useful in tailoring other properties (e.g., thermal properties, hardness).



a



b

Figure 1. The fracture toughness/resistance,  $K_R$ , of SiC whisker-reinforced fine grained alumina increases with crack extension due to the evolution of crack bridging processes. This R-curve response increases with increase in whisker content (a) and with whisker diameter (b).

Finally, it was noted earlier that one can use additional approaches in combination with whisker-reinforcement to alter the toughness of the alumina-based composites. For instance, the alumina-matrix grain size can be a factor in the level of toughness achieved.<sup>16-18</sup> This grain size effect is well documented in alumina and other noncubic ceramics, where anisotropy introduces local residual stresses and microcracking, and is attributed to crack bridging by matrix grains. Subsequently, it was shown that toughness of SiC whisker-reinforced aluminas can be raised an additional 20 to 60% by increasing alumina grain size.<sup>1c</sup>

### **Mechanical and Other Properties of Whisker-Reinforced Aluminas**

Whisker-reinforced aluminas can also exhibit substantial improvements in fracture strength<sup>19</sup>, strength distributions (higher Weibull modulus),<sup>20</sup> and resistance to thermal shock<sup>21</sup> and slow crack growth under static,<sup>19,22</sup> and cyclic<sup>23,24</sup> loading conditions as compared to unreinforced aluminas. Some of these improvements in mechanical properties are shown in Table I.

#### Room Temperature Fracture Strength

The room temperature fracture strengths of hot pressed whisker-reinforced aluminas with fairly coarse surface finishes (i.e., 180 grit diamond resinoid bonded grinding wheel) are quite impressive. In fine grained alumina composites, strengths of ~ 700 to nearly 900 MPa are obtained for whisker loadings of  $\geq$  20 vol. %, Figure 2. A very interesting feature is the strengthening effects achieved in large grained alumina composites where strengths of up to 600 MPa have been attained. Recall that in unreinforced aluminas large grain sizes are used to gain increases in steady-state toughness, but, as shown here, this results in greatly reduced strengths. As noted earlier, when whisker-reinforced, these large grained aluminas exhibit greater toughness than the composites with finer grained alumina.

Table I. Mechanical Performance of Reinforced and Unreinforced Alumina  
 Hot Pressed Alumina (G=1.5  $\mu\text{m}$ ) -20 Vol % SiC Whiskers

Temperature	22°C	1000°C	1200°C
Flexure Strength, MPa <sup>a</sup>	800	775	550
Fracture Toughness, MPa $\sqrt{\text{m}}$	8.3	8.1	10
Hot Pressed Alumina (G = 1.5 $\mu\text{m}$ )			
Temperature	22°C		
Flexure Strength, MPa <sup>a</sup>	550		
Fracture Toughness, MPa $\sqrt{\text{m}}$	2.8		
Cyclic Fatigue Response (Tension-Tension with $\sigma_{\text{min}} = 0.1\sigma_{\text{max}}$ ) <sup>b</sup>			
Hot Pressed Alumina (Grain size = 1.5 $\mu\text{m}$ ) -20 Vol% SiC Whiskers			
Maximum Stress, MPa	435	330	+367 <sup>c</sup>
% of Fracture Strength	100	75	84
Cycles	1	1.1x10 <sup>5</sup>	+4x10 <sup>4</sup>
	Failed	No Failure	NF
			NF
			Failed
Sintered Alumina (Grain size = 5 $\mu\text{m}$ )			
Maximum Stress, MPa	318	249	221
% of Fracture Strength	100	78	69
Cycles to failure	1	500	4x10 <sup>3</sup>
			3x10 <sup>4</sup>
			7x10 <sup>5</sup>

#### Slow Crack Growth Under Static and Cyclic Loading

The retention of strength as a result of damage/crack growth is another important consideration. The slow growth of cracks that eventually leads required for fast fracture (i.e., see times to failure for cyclic loading of alumina Table 1); often applied stresses of only > 50% of the fast fracture

<sup>a</sup> Surfaces prepared with 180 grit diamond resinoid bonded surface grinding wheel with the grinding direction parallel to the length of the four point flexure bar.

<sup>b</sup> K. C. Liu, ref. 25; tension-tension testing at room temperature of alumina-25 wt. % SiC whisker composite fabricated by Cercom, Vista, CA.

<sup>c</sup> Stress cycled under these conditions for number of cycles shown; no failure. Testing continued after changing the maximum stress.

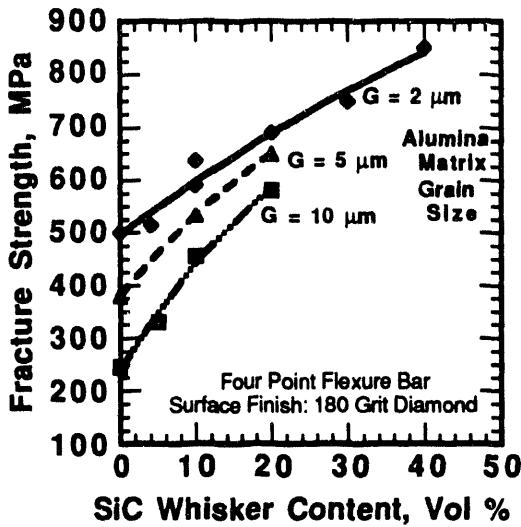


Figure 2. Flexure strengths increase with whisker content even in larger grain sized aluminas.

strength are sufficient to result in time-dependent failures. The chemical activity of reactive species (e.g., water) and temperature at the crack tip are controlling factors. Therefore increases in relative humidity at room temperature would promote crack growth at even lower stress levels. When the starting flaw size is the same, this is equivalent to stating that slow crack growth will be initiated at applied stress intensities more than one-half the fracture toughness value. For an alumina ceramic with a fracture toughness of  $4.5 \text{ MPa}\sqrt{\text{m}}$ , crack growth should occur at applied stress intensities  $\geq 2.5 \text{ MPa}\sqrt{\text{m}}$ , Figure 3. One of the beneficial effects of SiC whisker reinforcement of alumina is the fact that stress intensity to initiate slow crack growth ( $\sim 7 \text{ MPa}\sqrt{\text{m}}$ ) is a factor of two to three times greater than that required in unreinforced alumina, Figure 3.<sup>19,23</sup> Not only will whisker-reinforced aluminas withstand higher stresses and avoid fast fracture, but time-dependent failures will be avoided even

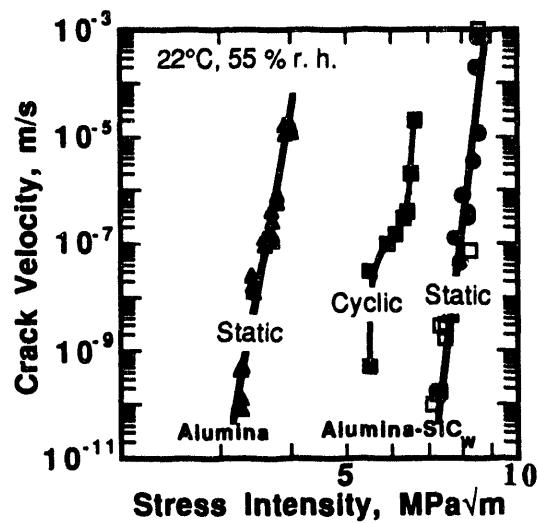


Figure 3. Whisker-reinforcement raises resistance to slow crack growth, under both static and cyclic loading. For composites, closed symbols-28 vol% whiskers; open symbols-20 vol% whiskers.

when exposed to quite high stress levels. This is reflected in Table I by the absence of failures in the composite even when subjected to peak cycle stress < 90% of the fast fracture strength. This is quite impressive for any class of materials. However, alternating opening and closing of the crack under cyclic loading can degrade the whisker and reduce the effectiveness of the crack bridging processes. The results for tension-tension cyclic fatigue do reveal that cyclic loading promotes slow crack growth to a degree as compared to static loading conditions, Figure 3. However, even under cyclic loading conditions, the reinforced alumina is far less susceptible to slow crack growth than the unreinforced alumina subjected only to static loading conditions.

#### Thermal and Electrical Properties

The thermal expansion behavior of composites can be a critical issue in design of components where one seeks to minimize expansion-contraction mismatch or maintain specific clearances between

components subjected to temperature variations. In addition, tailoring the thermal expansion coefficients may be an important consideration in optimizing resistance to stresses generated during heating-cooling cycles or imposed by other thermal gradients. In composites, the expansion coefficients will be a product of the expansion coefficient of each phase and the volume fraction of each phase present (e.g., rule-of-mixtures).<sup>26</sup> The response is compounded when longitudinal axes of the reinforcing fibers or whiskers are oriented within the body.

Models of linear thermal expansion coefficients ( $\alpha$ ) of composites containing uni-axially aligned fibers indicate that  $\alpha$  values will depend upon orientation with respect to fiber axes.<sup>26-28</sup> This is due to internal stresses brought on by mismatch in linear thermal expansion coefficients and elastic properties between fiber and matrix. These internal stresses are a function of (1) reinforcement content<sup>26,28-30</sup>, (2) aspect ratio of reinforcements<sup>27,31</sup>, and (3) degree of preferred orientation of the reinforcement axis.<sup>31</sup> The elastic strains, associated with internal stresses couple with the expansion/contractions generated during heating and cooling of the composite. These combined strains dictate the expansion coefficients of the composites. Furthermore, the axial internal stress in the whisker (and as a result, in the matrix) is greater than that normal to the whisker axis as a result of the axi-symmetric geometry of the whisker. This leads to an anisotropy in the expansion coefficients of composites with aligned fibers.<sup>28</sup>

The thermal expansion behavior suggested by analyses based on the above discussion is, in fact, observed in the SiC whisker-reinforced aluminas. First, the lower thermal expansion coefficient of SiC, as compared to alumina, leads to a decrease in the linear thermal expansion coefficients of the composites with increase in SiC whisker content, Figure 4. The internal stresses, due to the low thermal expansion coefficient of SiC versus alumina, introduced in the post densification cooling cycle place the whiskers in compression and the

matrix in tension. As a result of this and the preferred orientation of the whiskers, the expansion coefficient of the composite in the direction parallel to the hot pressing axis (HPA) should be greater than that perpendicular to HPA for each composition due to the fact that the longitudinal axis of the whiskers lies, more or less, in a plane normal to HPA. This is, indeed, observed in the SiC whisker-reinforced alumina, Figure 4.

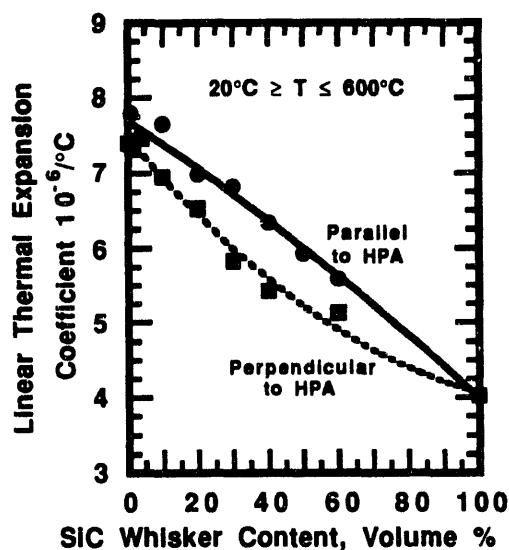


Figure 4. The thermal expansion coefficients ( $\alpha$ ) decrease with whisker content. Alignment of the whiskers during hot pressing results in an orientation dependence of  $\alpha$ .

As a result of its much greater thermal and electrical conductivity as compared to alumina, the addition of SiC whiskers will also raise thermal conductivity and lower electrical resistivity of alumina-based composites. However, neither property will exhibit a simple rule-of-mixtures law dependent on whisker content. When the conducting phase (e.g. SiC) content exceeds the percolation limit (i.e., in the range of 10 to 20 vol% of this phase), much more substantial changes in these properties can be expected.<sup>32</sup> Above this whisker loading, interconnected highly

conductive whisker paths are established. Such behavior is observed in the measured electrical resistivity<sup>20</sup> and thermal conductivities<sup>33</sup> of the SiC whisker-reinforced aluminas where rapid changes in electrical resistivity and thermal conductivity are observed as the whisker content is decreased below 20 vol.%, Figure 5. Enhancing the degree of preferred orientation of whiskers could result in establishing a difference in the whisker content to exceed the percolation limit for measurements parallel versus perpendicular to the pressing axis. This might allow one to select a composition that leads to a much higher electrical and thermal conductivity in the plane normal to the hot pressing axis versus in the direction of the HPA.

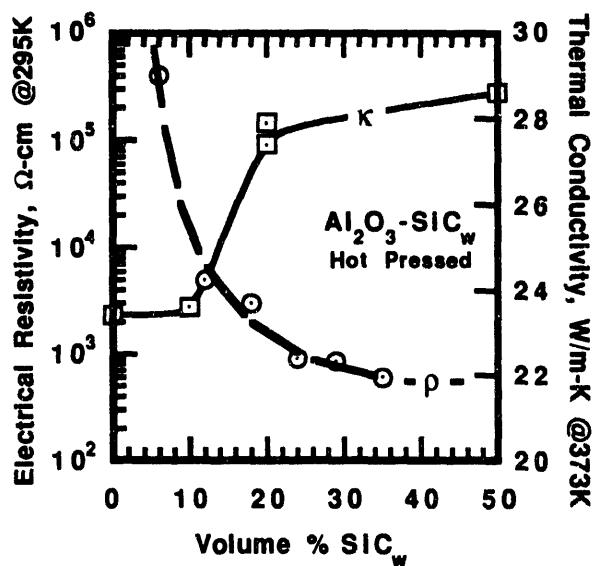


Figure 5. Both the electrical ( $\rho$ ) and thermal ( $\kappa$ ) conduction change significantly with increase in whisker contents above 10 vol.% related to the formation of interconnected SiC whisker paths.

#### Thermal Shock Resistance

Crack growth due to stresses generated by either fixed thermal gradients or rapid heating or cooling can lead to either a loss in strength or time dependent failures just as with mechanical stresses.<sup>34</sup> One of the more

popular test techniques to evaluate thermal stress/shock resistance is the down-quench test involving measurement of strength retained after the sample is quenched from an elevated temperature,  $T$ , into a bath held at a fixed, but lower, temperature,  $T_B$ . Loss of strength after quenching is assumed to be a result of crack growth initiated when the thermal tensile stress,  $\sigma_T$ , equals the material's unshocked fracture strength  $\sigma_o$ .<sup>34c-e</sup> The magnitude of the thermal stress is dependent not only on the magnitude of the temperature drop (i.e.,  $\Delta T = T - T_B$ ) but also on various material properties (e.g.,  $\alpha$ , the linear thermal expansion coefficient;  $E$ , the Young's modulus;  $k$ , the thermal conductivity;  $\nu$ , the Poisson's ratio), the characteristic (or shortest) heat transfer length  $a$  of the specimen (i.e., herein equal to the thickness  $t$  of the flexure bar), as well as the surface heat transfer coefficient  $h$  of the specific quenching media.

The critical temperature change,  $\Delta T_c$ , that produces a tensile stress resulting in a loss in fracture strength after quenching is defined as:<sup>34c-e</sup>

$$\Delta T_c = [ \sigma_o (1-\nu) / \alpha E ] f (ah/k) \quad (7)$$

where the last term,  $f (ah/k)$ , reflects the role of heat transfer in establishing the temperature gradient in the sample. For a circular rod, this term can be approximated as:<sup>34b,35</sup>

$$f (k/t h) \sim 1.5 + 4.67(k/th) - 0.5 \exp(-51k/th) \quad (8)$$

where  $t$  is the diameter of the rod. Note that when the  $k/t$  term is small,  $\Delta T_c$  becomes independent sample size (e.g., Eq'n 8 reduces to a constant (~ 1.5)). However, for large  $k/t$  values,  $\Delta T_c$  exhibits an inverse dependence on the minimum sample dimension (e.g., the thickness of the test sample here). For a given material, this implies that the critical  $\Delta T$  value should first decrease and then transition to become more or less independent of size as the thickness of the test specimen increases. Both the  $\Delta T_c$  values and the critical sample thickness above which  $\Delta T_c$  is

independent of thickness will be functions of the thermal and mechanical properties of the material.

Based on increases observed in thermal conductivity, fracture strength, and toughness and decrease in thermal expansion, the SiC whisker-reinforced aluminas should exhibit greater thermal shock resistance than the unreinforced alumina. Measurements of  $\Delta T_c$  values based on strength losses after quenching samples into boiling water baths confirm the greater thermal shock resistance of the composite as compared to unreinforced alumina.<sup>36</sup> Also as predicted, the  $\Delta T_c$  values are dependent upon thickness  $t$  of the flexure bar in both the composite and the alumina ceramic, Figure 6. At small sample thicknesses, there is a

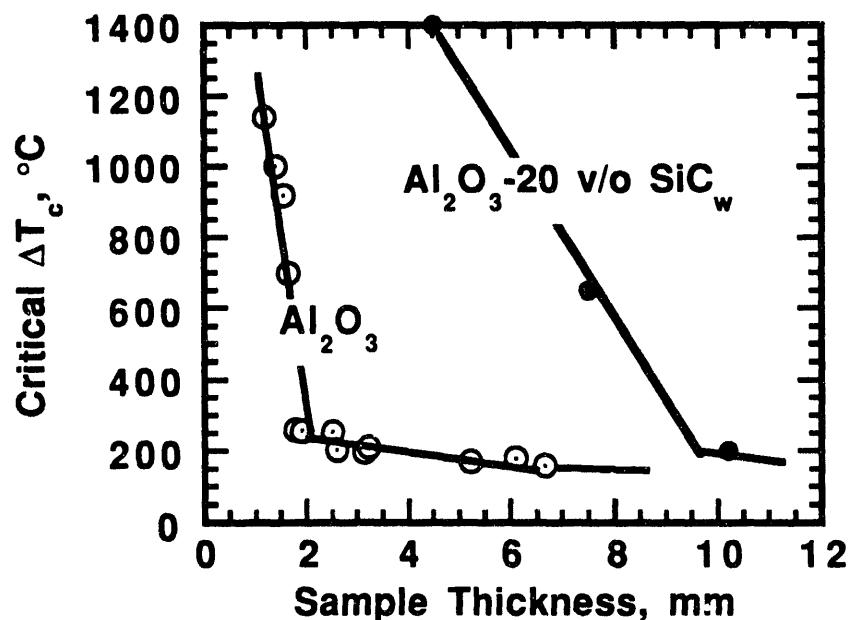


Figure 6. The thermal shock resistance is substantially increased by whisker-reinforcement. Even sections that are multiple millimeters thick do not undergo strength losses after quenching from several hundred degrees centigrade into a boiling water.

rapid decrease in  $\Delta T_c$  values with increase in sample thickness followed by a transition to a region where  $\Delta T_c$  becomes essentially independent of sample size. As expected, the composite exhibits greater resistance to

thermal shock at much larger sample sizes. Finally, the composite also exhibits excellent resistance to repeated thermal shock cycles;<sup>36a</sup> this may not be surprising in view of its excellent resistance to mechanical fatigue. The ability to tailor thermal properties (e.g., Figures 4 and 5) combined with the excellent mechanical properties of whisker reinforced ceramics obviously provide an opportunity for developing thermal shock resistant structural ceramics.

#### Elevated Temperature Mechanical Properties

Previous studies show that the high flexure strengths (650 to 800 MPa) at room temperature are retained to quite high temperatures (e.g., 1000° to 1100°C in air) in SiC whisker-reinforced fine grained (< 5 µm) alumina.<sup>37</sup> Above 1100°C, the strengths decrease with increase in test temperature. Similarly, the fracture toughness of this same composite remains constant for temperatures up to ~1100°C in air; above 1100°C, the measured toughness increases. Each of these changes above 1100°C is due to the contributions of creep deformation primarily associated with grain boundary sliding as a result of the fine grain size of the alumina matrix.

Tests involving slow crack growth and surface reactions in air at elevated temperature revealed that alumina-20 vol% SiC whisker composites have remarkable resistance to strength degradation. At test temperatures of 800, 1000, and 1100°C, the fracture strengths were virtually unaffected after exposing the composites to applied flexure stresses equivalent to two-thirds the fast fracture strengths for times of up to 1000 h.<sup>37</sup> Delayed failure tests at 1000 and 1100°C resulted in no failures in samples exposed to stresses ≤ 75% of the fast fracture strengths for times up to 14 weeks.<sup>22</sup> Only when the applied stress was > 75% of the fast fracture strength at 1000 and 1100°C, was slow crack growth induced failure observed. At temperatures of 1200°C and above, the fine grained alumina composites exhibited time dependent failure over an applied stress range of 40 to 100% of the fast fracture strength.

At applied stress levels < 75% of the fast fracture strength, failed samples exhibit permanent deformation indicative of creep-related failure. These observations are indicative of the beneficial effects of whisker reinforcement on the elevated temperature of mechanical properties. As seen above, degradation of mechanical properties at elevated temperatures is associated with the onset of creep. Thus studies of the role of microstructure and composition on creep processes would prove useful in improving the elevated temperature mechanical performance of these composites. Studies do show that creep rates of fine grained alumina are greatly reduced by incorporating SiC whiskers; at 1200 (Figure 7) and 1300°C the creep rates are two orders of magnitude less

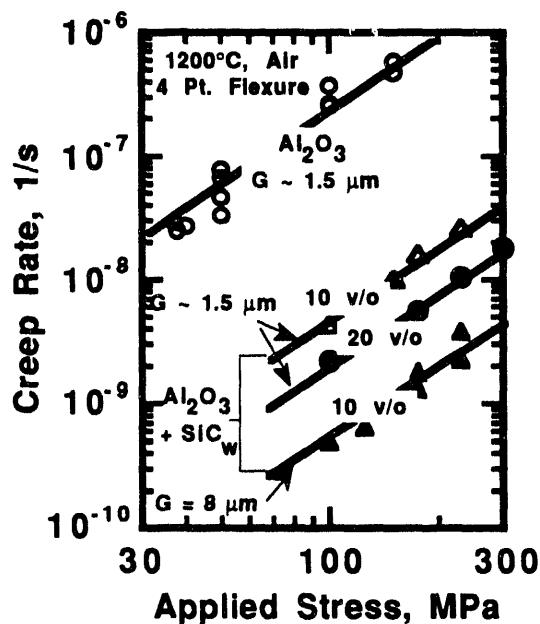


Figure 7. Creep rates at elevated temperatures are greatly reduced with the addition of whiskers. Matrix grain size can also be increased to obtain further creep resistance.

than those of the unreinforced aluminas.<sup>38</sup> Microscopy studies indicate that grain boundary sliding in the fine grained matrix is the primary mode of creep in these materials. A major contribution to reduced creep in reinforced alumina is a result of grain boundary pinning of the grain

boundaries that results both from whiskers penetrating across grain boundaries and those that lie in the grain boundary planes.

However, creep resistance of SiC whisker reinforced alumina composites in air can also be degraded when whisker content is raised to 30 vol% or greater.<sup>39</sup> This is a result of both increased cavitation associated with whiskers and enhanced surface oxidation and glass formation. At the same time, the use of certain densification aids (e.g., yttria) that interact with the oxidation of SiC whiskers and form amorphous phases are also found to increase creep rates.<sup>39</sup> On the other hand, increasing the matrix grain size can result in greater creep resistance in the composite.<sup>40</sup> Attention to the formulation and design of the composite provide an approach to increasing the resistance to creep and extend the life of composites at elevated temperatures.

## Conclusions

The successful application of whisker-reinforcement to improve the mechanical reliability of ceramics demands that attention be paid to the influences of whisker characteristics, matrix microstructure, and chemical composition. Progress in the area of reinforced ceramics is providing a wealth of new insights into the toughening of ceramic systems. The theoretical descriptions of the toughening response in whisker-reinforced ceramics provide details on how to design tougher materials and directions where more insight is needed, e.g., interfacial property-structure relationships. In addition, models for whisker reinforcement point to a need to develop techniques to synthesize whiskers where size and strength can be altered in a controlled manner.

As described herein, significant improvements in the fracture strength and its statistical distribution can be achieved in whisker-reinforced aluminas. These reinforced materials also exhibit fracture strengths that are much more resistant to subsequent degradation by crack growth due

to thermal shock, and subcritical and cyclic fatigue as compared to unreinforced ceramics. At elevated temperatures, these systems offer considerable potential as they can exhibit excellent strength retention and creep resistance. Attention must also be given to changes in phase content resulting from exposure to various environments at elevated temperatures. For example in whisker-reinforced alumina, increasing the whisker content may be attractive for enhancing the fracture toughness but can result in degradation of creep performance due to the promotion of oxidation and creep rates. Clearly, tailoring compositions and microstructures of these composites offers an avenue to developing advanced high temperature ceramics and ceramics for various structural applications.

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