

CONF-901125--1

REINFORCED CERAMICS EMPLOYING DISCONTINUOUS PHASES*

Paul F. Becher
Metals and Ceramics Division
Oak Ridge National Laboratory
Oak Ridge, Tennessee

CONF-901125--1

DE91 000399

ABSTRACT

The fracture toughness of ceramics can be improved by the incorporation of a variety of discontinuous reinforcing phases and microstructures. Observations of crack paths in these systems indicate that these reinforcing phases bridge the crack tip wake region. Recent developments in micromechanics toughening models applicable to such systems are discussed and compared with experimental observations. Because material parameters and microstructural characteristics are considered, the crack bridging models provide a means to optimize the toughening effects.

INTRODUCTION

Advances in processing and in nondestructive evaluation are providing means to reduce the flaw and defect sizes in as-fabricated components to ensure the desired (high) strength levels. This is also resulting in some improvements in (narrowing of) the strength distributions of the as-fabricated components. However, generation of new flaws and growth of existing flaws during service degrades the strengths and the strength distributions. Materials with high fracture toughness and R-curve behavior are much less sensitive to strength degradation in service and also are found to exhibit narrower strength distributions and to be more resistant to slow crack growth and thermal shock.

A number of approaches have been devised to improve the fracture resistance of brittle ceramic systems. Here we will briefly examine the use of microstructural modification and reinforcement by second phases, separately and as coupled processes. The aim is to address how these processes are influenced by key microstructural and compositional characteristics utilizing both results from micromechanics analysis and experimental studies.

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED
MASTER 102

*Invited paper to be presented at Ceramic Science and Technology Congress,
Orlando, Fla., November 13-15, 1990.

"The submitted manuscript has been
authorized by a contractor of the U.S.
Government under contract No. DE-
AC05-84OR21400. Accordingly, the U.S.
Government retains a nonexclusive,
royalty-free license to publish or reproduce
the published form of this contribution, or
allow others to do so, for U.S. Government
purposes."

RESULTS AND DISCUSSION

Toughening by Whisker Reinforcement

Bridging of the crack surfaces by and pullout of strong whiskers imposes a closure force on the crack. These processes and deflection of the crack tip by the whiskers can contribute to the toughness of whisker-reinforced ceramics.¹⁻⁴ The bridging and pullout contributions to the toughness for uniaxially aligned whiskers are:

$$\Delta K^{wr} = 2\beta \sigma^c (2D_B/\pi)^{1/2} = K_{IC}^c - K_{IC}^m, \quad (1)$$

where K_{IC}^c is the overall toughness of the composite, K_{IC}^m is the matrix toughness, σ^c is the closure stress acting on the bridging zone of length D_B , and β is a constant dependent upon the bridging stress profile. With whisker bridging, the crack opening displacement at the end of the whisker bridging zone will equal the tensile displacement in the whisker when it fails ($\ell_{DB}\sigma_f^w/E^w$ where ℓ_{DB} is the debonded length of the interface, σ_f^w is the whisker's tensile strength, and E^w is its Young's modulus). Employing this crack opening displacement relationship, one finds that D_B is proportional to the product of the ratios of the whisker to interface fracture energy (γ^w/γ^i), the composite to whisker Young's modulus (E^c/E^w), and whisker radius divided by the whisker content (r/V^w).¹

Substantial toughening by whisker bridging relies on whisker-matrix interface debonding. The stresses acting on the bridging whiskers immediately behind the crack tip are then substantially reduced and increase with increasing distance behind the crack tip. The maximum closure stress acting at the end of the bridging zone is the product of the tensile strength of the whiskers and the fraction of bridging whiskers. Employing a bridging stress that increases linearly from zero at the crack tip to the maximum value at the end of the zone, the toughening contribution of bridging whiskers is found to be:

$$\Delta K^{wr} = \sigma_f^w [V^w r (\gamma^w E^c / \gamma^i E^w) / 6(1 - v^2)]^{1/2}, \quad (2)$$

where v is the Poisson's ratio of the composite. An important attribute of this micromechanics model, described in detail earlier,¹ is the fact that it provides a basis for designing such systems using material properties and characteristics.

When the whiskers fail at the end of the bridging zone, those whiskers which fail at some distance away from the main crack plane can then be pulled out of the matrix. This pullout process contributes to the fracture resistance of the systems. For pullout, the crack opening displacement at the end of the pullout zone must equal the length of the pullout ℓ_{po} . Here we will assume this length is equal to the debond length and that the effective closure stress due to pullout is the product of the frictional shear resistance of the debonded interface τ^i and the areal fraction of whiskers that are pulled out, A^{po} . A toughening contribution of a pull-out zone which exhibits a linearly decreasing stress profile along its length is:

$$\Delta K^{po} = [A^{po} \tau^i E^c (\ell_{po})^2 / 2(1 - v^2) r]^{1/2}, \quad (3)$$

where ℓ_{po} is equated to ℓ_{DB} which equals $(r\gamma^w/6\gamma^i)$, e.g., ref. 5) when the debonding criteria is based on debonding occurring just ahead of the crack tip.²

Combining the whisker bridging and whisker pullout toughening contributions, one can predict the experimental toughness values for whisker-reinforced ceramics as seen in Fig. 1. The results shown here are for composites containing the same SiC whiskers which have relatively smooth, straight, rod-like morphologies and diameters near $0.8 \mu\text{m}$. Introduction of other whiskers with rough surfaces, different surface chemistries, or large defects will yield quite different results. Surface roughness and surface chemistry of the whiskers can affect a change in the ability to initiate interfacial debonding. Restricting interface debonding will diminish the bridging and pullout toughening contributions. This can be seen from Fig. 1 where pretreatment of the whiskers resulted in a lower toughness for the alumina composite. Note this data point corresponds to the prediction in the absence of pullout; fracture surface observations confirm this. These findings indicated that the micromechanics analysis, based on various materials properties, provides a very useful tool to either modify the composites to improve toughness or to examine composites to determine what parameters/characteristics are limiting the toughness achieved.

Matrix Microstructure Effects

In the present discussion, grain size effects on toughness are related to bridging ligaments formed by matrix grains which are left intact behind the crack tip.⁶⁻⁸ The toughening analysis is analogous to that for the whisker pullout described above. The bridging stress supported by these ligaments is, as with whisker pullout, a product of the frictional stress required to pull out each debonded bridging grain times the areal fraction of bridging grains, $A^{gb} \tau_{gb}$. The grain bridging zone length is again equated to the crack opening displacement at the end of the zone to completely pull out the bridging grains. Assuming that half the grain must be pulled out to disrupt a ligament, u will be equal to one half the grain size (d). Employing Eq. (1) again, the incremental increase in fracture toughness due to grain bridging ΔK^{gb} is:

$$\Delta K^{gb} = [A^{gb} \tau_{gb} E^c d / 2(1 - v^2)]^{1/2}, \quad (4)$$

yielding a grain bridging toughening contribution with a strong grain size dependence. This model is consistent with experimental observations, Fig. 2⁹, for grain sizes below those resulting in spontaneous microcracking.¹⁰⁻¹²

Crack bridging phenomena and toughening effects similar to those from both whisker and the above grain size related matrix bridging effects can be expected to result from other microstructural feature changes. For example, the development of more thermal shock resistant alumina ceramics led to microstructures containing plate-like alumina grains in a medium-sized (5 to 10 micron) equiaxed grained

matrix. These materials had excellent thermal shock resistance.¹³ Fracture toughness measurements using large precracked samples gave values of 7 to 8 MPa m^{1/2} for samples containing ~25 vol % large (up to 100 to 300 micron wide by up to 10 to 30 micron thick) single-crystal alumina plates. Aluminas with similar equiaxed grain size but without these plate-like grains had toughness values of only 4 to 4.5 MPa m^{1/2}. Furthermore, the addition of SiC platelets to alumina is found to result in increases in fracture toughness values that are comparable to those achieved with SiC whiskers. In both of the above examples, the propagating crack is deflected along the interface between the matrix and the large plate-like grains. Thus, fracture around the plate-like grains and second phases produces platelet features which bridge the main crack and results in the high toughness due to crack bridging and pullout mechanisms. Additional examples of toughening by crack bridging are found in Si₃N₄ ceramics which are reinforced by the *in situ* growth of elongated whisker-like grains. Such self-reinforcement processes prove to be a potent toughening approach yielding silicon nitride ceramics with toughness values of ≥ 10 MPa m^{1/2}.¹⁴⁻¹⁷ Again, crack bridging and pullout by these grains contribute to the improved toughness.

Coupled Toughening Responses: Whisker Reinforcement-Grain Bridging

As discussed earlier, the fracture resistance of ceramics, especially noncubic ceramics, will increase with increase in grain size as a result of grain bridging in the wake of the crack tip. Thus the overall fracture toughness of the composite can also be influenced by the intrinsic matrix toughness, the microstructural component of the matrix toughness, especially in the case of noncubic matrices, and the whisker reinforcement contribution.⁹ For simple additive combination of these toughening processes, the overall composite toughness is:

$$K_{IC}^c = K_{IC}^m + \Delta K^{gb} + \Delta K^{wr}, \quad (5)$$

where ΔK^{gb} is the fracture resistance derived from matrix grain bridging, Eq. (4)¹ and K_{IC}^m is the local fracture resistance of the matrix in the absence of grain bridging. Substitution of Eqs. (2) and (4) into Eq. (5) describes the combined toughness due to whisker reinforcement and matrix grain size effects:

$$K_{IC}^c = K_{IC}^m + [A^{gb} \tau_{gb} E^c d / 2(1 - v^2)]^{1/2} + \Delta K^{wr}. \quad (6)$$

This behavior is observed in SiC whisker-reinforced aluminas, Fig. 2.⁹ Similar coupled toughening behavior has been described² for the toughening response in SiC whisker-reinforced silicon nitrides. Using the data of Shalek et al.,¹⁸ one can see that the introduction of elongated silicon nitride grains within the matrix provides a crack bridging contribution which couples with whisker bridging to provide significant toughening. In the presence of equiaxed silicon nitride grains, the composite toughness is much less. Utilizing multiple toughening mechanisms, we see that the matrix microstructure can be readily modified to enhance the fracture toughness of a composite.

CONCLUSIONS

Reinforced ceramics including reinforcement by strong whiskers and other second-phase geometries, e.g., platelets, employ crack bridging and crack deflection processes to achieve improved fracture resistance. Similar effects can be achieved by changes in grain size, e.g., in noncubic ceramics, and/or grain shape, e.g., formation of elongated grains in Si_3N_4 and SiAlON and plate-like grains in alumina ceramics. The amount of toughness realized is dependent upon the properties and characteristics of the reinforcing phase and the interface as described by the micromechanics models developed for these systems.

Considerable improvements in fracture toughness can be achieved by incorporating these processes. In fact, coupling these individual mechanisms allows for even greater advances in the development of ceramics with high fracture resistance. However, the toughening response can only be optimized by attention to control of the appropriate microstructural, compositional, and interfacial characteristics.

ACKNOWLEDGEMENTS

The support of my colleagues, especially T. N. Tiegs, C. H. Hsueh, K. B. Alexander, P. Angelini, H. T. Lin, S. B. Waters, W. H. Warwick, and L. M. Evans, is gratefully acknowledged. This research was sponsored by the Division of Materials Sciences, U.S. Department of Energy, under contract DE-AC05-84OR21400 with Martin Marietta Energy Systems, Inc.

REFERENCES

1. P. F. Becher, C. H. Hsueh, P. Angelini, and T. N. Tiegs, "Toughening Behavior in Whisker Reinforced Ceramic Matrix Composites," *J. Am. Ceram. Soc.* **71**(12), 1050-61 (1988).
2. P. F. Becher, "Microstructural Design of Toughened Ceramics," to be published.
3. N. Claussen and G. Petzow, pp. 649-62 in *Tailoring of Multiphase and Composite Ceramics, Vol. 20, Materials Science Research*, eds. R. E. Tressler, G. L. Messing, C. G. Pantano, R. E. Newnham, Plenum Publishing, New York, 1986.
4. R. Warren and V. K. Sarin, "Fracture of Whisker Reinforced Ceramics," in *Applications of Fracture Mechanics to Composite Materials*, ed. K. Friedrich, Elsevier, Amsterdam, in press.
5. B. Budiansky, J. W. Hutchinson, and A. G. Evans, "Matrix Fracture in Fiber-Reinforced Ceramics," *J. Mech. Phys. Solids* **34**(2), 167-89 (1986).
6. M. V. Swain, "R-Curve Behavior in a Polycrystalline Alumina Material," *J. Mater. Sci. Lett.* **5**, 1313-15 (1986).
7. a. P. L. Swanson, C. J. Fairbanks, B. R. Lawn, and Y. W. Mai, "Crack-Interface Grain Bridging as a Fracture Resistance Mechanism in Ceramics: I, Experimental Study of Alumina," *Am. Ceram. Soc.* **70**(4), 279-89 (1987).

- b. Y. W. Mai and B. R. Lawn, "Crack-Interface Grain Bridging as a Fracture Resistance Mechanism in Ceramics: II, Theoretical Fracture Mechanics Model," *Am. Ceram. Soc.* **70**(4), 289-94 (1987).
- 8. C. Cm. Wu, S. W. Freiman, R. W. Rice, and J. J. Mecholsky, "Microstructural Aspects of Crack Propagation in Ceramics," *J. Mater. Sci.* **13**, 2659-70 (1978).
- 9. P. F. Becher, E. R. Fuller, Jr., and P. Angelini, "Matrix Grain Bridging in Whisker Reinforced Ceramics," to be published.
- 10. R. W. Rice, S. W. Freiman, and P. F. Becher, "Grain-Size Dependence of Fracture Energy in Ceramics: I, Experiment," *J. Am. Ceram. Soc.* **64**(6), 345-50 (1981).
- 11. a. R. Steinbrech, R. Khehans, and W. Schaarwachter, "Increase of Crack Resistance During Slow Crack Growth in Al_2O_3 Bend Specimens," *J. Mater. Sci.* **18**, 265-70 (1983).
b. R. Khehans and R. Steinbrech, "Memory Effect of Crack Resistance During Slow Crack Growth in Al_2O_3 Bend Specimens," *J. Mater. Sci. Lett.* **1**, 327-29 (1982).
- 12. B. Mussler, M. V. Swain, and N. Claussen, "Dependence of Fracture Toughness of Alumina on Grain Size and Test Technique," *J. Am. Ceram. Soc.* **65**(11), 566-72 (1982).
- 13. T. N. Tiegs and P. F. Becher, "Particulate and Whisker Toughened Alumina Composites," pp. 479-85 in *Proceedings of 22nd Automotive Technology Development Contractors' Coordination Meeting*, P-155, Society of Automotive Engineers, Inc., Warrendale, Pa., 1985.
- 14. K. B. Alexander, P. F. Becher, and S. B. Waters, "Characterization of Silicon Carbide Platelet-Reinforced Alumina," pp. 1032-33 in *Proceedings of the XIIth International Congress for Electron Microscopy*, ed. G. W. Bailey, San Francisco Press, San Francisco, 1990.
- 15. C. W. Li and J. Yamanis, "Super-Tough Silicon Nitride with R-Curve Behavior," *Ceram. Eng. Sci. Proc.* **10**(7-8), 632-45 (1989).
- 16. G. Himsolt, H. Knoch, H. Huebner, and F. W. Kleinlein, "Mechanical Properties of Hot-Pressed Silicon Nitride with Different Grain Structures," *J. Am. Ceram. Soc.* **62**(1), 29-32 (1979).
- 17. M. H. Lewis, "Microstructural Engineering of Ceramics for High-Temperature Application," pp. 713-30 in *Fracture Mechanics of Ceramics*, eds. R. C. Bradt, A. G. Evans, D. P. H. Hasselman, and F. F. Lange, Plenum, New York, 1986.
- 18. P. D. Shalek, J. J. Petrov, G. F. Hurley, and F. D. Gac, "Hot Pressed SiC Whisker/ Si_3N_4 Matrix Composites," *Am. Ceram. Soc. Bull.* **65**(2), 351-56 (1986).

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

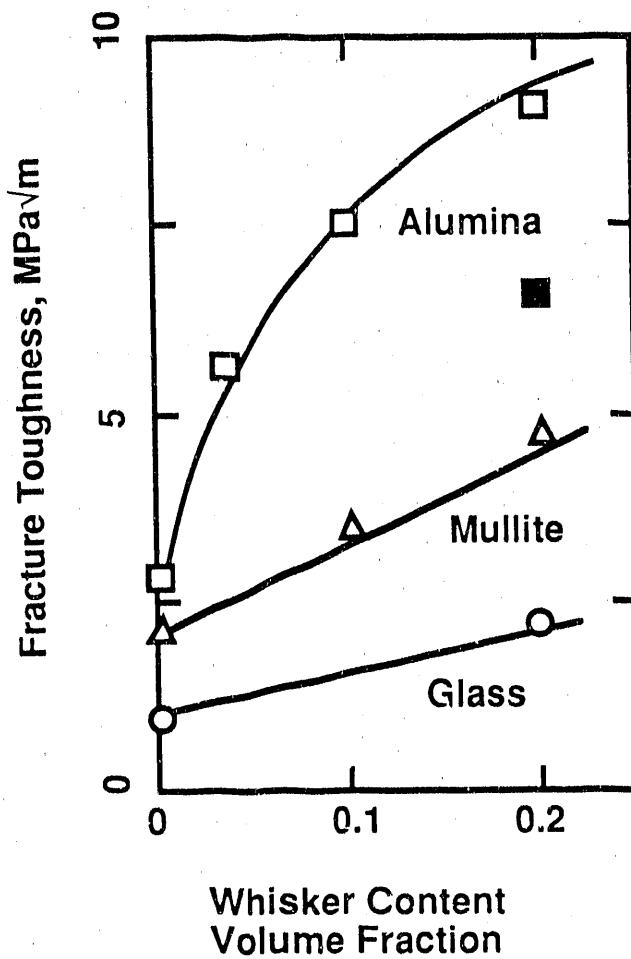


Figure 1 The addition of SiC whisker reinforcements results in a substantial increase in the fracture toughness of various ceramics including polycrystalline oxide (shown above) and nonoxide and glass (above) matrices. Both the magnitude of the toughness and the influence of various material properties on the measured fracture toughness values are in keeping with the behavior predicted by whisker bridging, Eq. (2) (mullite and glass matrices), and whisker bridging plus pullout, Eq. (3) (alumina matrix). Open symbols represent measured values obtained for composites containing as received whiskers ($r=0.4 \mu\text{m}$); the close symbol is for data obtained with preoxidized whiskers in alumina. This composite exhibits negligible whisker pullout.

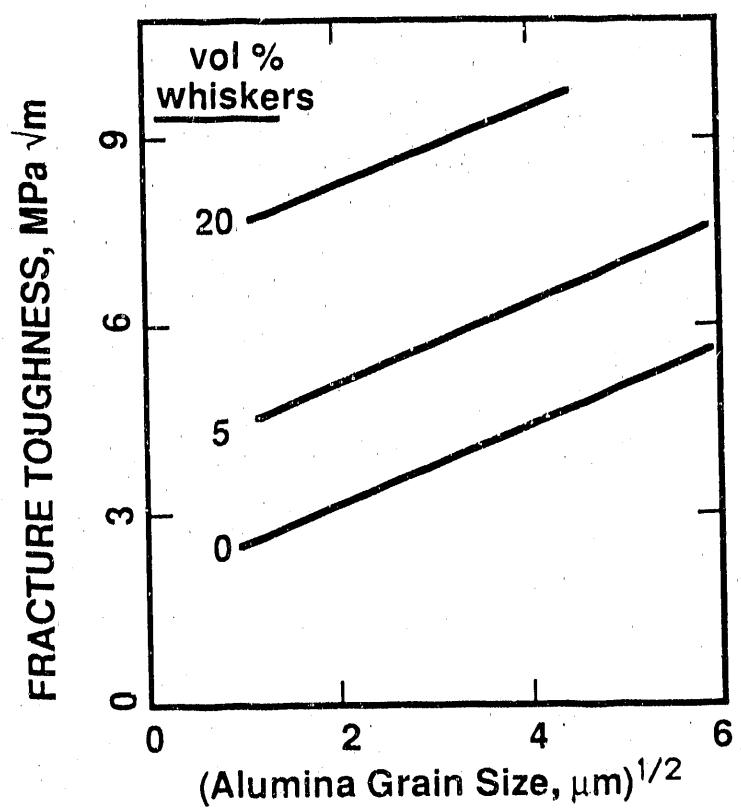


Figure 2 The fracture toughness of both monolithic alumina and SiC whisker (0.4 μm radius) reinforced alumina is strongly influenced by the microstructure of the alumina. Increase in the alumina grain size introduces matrix bridging of the crack and an increased toughness in a manner predicted for a grain bridging contribution, e.g., Eqs. (4) and (6).

END

DATE FILMED

10/26/90

