NOTICE

**CERTAIN DATA
CONTAINED IN THIS
DOCUMENT MAY BE
DIFFICULT TO READ
IN MICROFICHE
PRODUCTS.**

CONF-910162--1

Microstructure and Properties of Alumina-Whisker- Reinforced Tetragonal
Zirconia Polycrystal Matrix Composites*

J. M. Roberts**, J. P. Singh

CONF-910162--1

DE91 006036

Argonne National Laboratory, Argonne, Illinois 60439

and

R. O. Scattergood

Department of Materials Science and Engineering.

North Carolina State University, Raleigh, North Carolina 27610

The submitted manuscript has been authored
by a contractor of the U. S. Government
under contract No. W-31-109-ENG-38.
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.

RECEIVED 1/13/91

JAN 13 1991

To be published in the Proc. of 15th Annual Conference on Composites and
Advanced Ceramics, American Ceramic Society, Cocoa Beach, Florida,
January 13-16, 1991.

MASTER

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

*Work supported by U. S. Department of Energy under Contract W-31-109-Eng-38.

**Work performed in partial fulfillment of requirements of M. S. degree, Department of Materials Science and Engineering, North Carolina State University.

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.

Microstructure and Properties of Alumina-Whisker- Reinforced Tetragonal
Zirconia Polycrystal Matrix Composites*

J. M. Roberts**, J. P. Singh

Argonne National Laboratory, Argonne, Illinois 60439

and

R. O. Scattergood

Department of Materials Science and Engineering,

North Carolina State University, Raleigh, North Carolina 27610

Introduction

With the increasing demand of today's technology, there is a growing need for high temperature materials with improved fracture toughness. Whisker-reinforced ceramic composites are materials with potentially high fracture toughness¹⁻³. Many whisker-matrix systems are possible, but the choices for toughened composites are limited by such considerations as chemical compatibility and thermal expansion mismatch. In view of the high fracture toughness of tetragonal zirconia polycrystals (TZP) matrix, many investigators³⁻⁵ have successfully attempted to improve the mechanical properties of yttria stabilized tetragonal zirconia polycrystals (Y-TZP) with

the addition of strong, single crystal SiC whiskers. The primary objective of this work was to place Al_2O_3 whiskers into an Y-TZP matrix and study the toughening effects on the new composite. This particular system was chosen because of the high strength and reasonably high toughness inherent within Y-TZP and potentially high strength obtainable within a single crystal alumina. This, combined with an agreeable thermal expansion mismatch, could theoretically produce a composite of exceptionally high toughness⁶.

Experimental Procedure

The alumina whiskers used in this study were supplied by Vista Chemical Company and were experimental products still in the developmental stage⁷. The whiskers are alpha-phase alumina having a rhombohedral shape. Average diameter of the whiskers was reported as 4-7 μm with an aspect ratio in the range of 10-13.

The matrix powder used was tetragonal zirconia polycrystals (TZP), labeled as TZ-3Y by its manufacturer, Toyo Soda Manufacturing, and consists of fine particles (<0.5 microns) of TZP⁸. The material is a stabilized zirconia of almost 100% tetragonal phase.

Various amounts (0-25 vol%) of alumina whiskers were successfully blended with TZP powder via a wet mixing technique using an appropriate solvent and a high-speed laboratory blender. The wet slurry was dried in thin layers to prevent separation of the constituents by sedimentation. After drying, the composite powders were uniaxially hot-pressed in a 1.5 inch diameter BN-

coated graphite die at approximately 1500°C and 25 MPa for 1 hour in a vacuum atmosphere. Sufficient composite powder was used in order to produce a specimen disk ~0.15 inch thick.

The hot-pressed disks were subsequently cut into ~3 x 4 x 40 mm bars. Density measurements were made on the bars using Archimedes Method with deionized water as the suspension medium.

Individual bars were then utilized in various mechanical properties tests. The flexural strength was measured in a four-point bending mode using an Instron testing machine with a loading span of 9.5 mm, support span of 19 mm, and a crosshead speed of 0.5 mm/min. Fracture toughness was measured by the diamond point hardness (DPH) technique⁹, and Young's modulus was measured by the sound velocity measurement using a pulse-echo technique¹⁰.

The phase identification was performed by x-ray analysis using a Philips diffractometer with a range of 2θ varying from 25 to 65° at a scan rate of 2°/min. The microstructure of the various specimens was analyzed by optical and scanning electron microscopy. Grain size and morphology, fracture surface and whisker morphology were studied.

Results and Discussions

Microscopic evaluation of the whiskers and composites indicated that the as-received whisker did not have any pores in them as shown in Fig. 1, while the whiskers in the hot-pressed composites show the presence of

porosity. As shown in Fig. 2, the whiskers seems to have degraded during hot-pressing and ~0.6-19 μm size pores were developed.

Both hot-pressed TZP and its composites with alumina whiskers had densities greater than 99% theoretical and have submicron size grains (Fig. 3). The fractographic evaluation indicate that many of the alumina whiskers (which did not have any pores) deflected the main crack while the cracks went straight through porous whiskers without any deflection (Fig. 4).

An x-ray analysis of the hot-pressed TZP and its composites indicated that the addition of alumina whisker resulted in an increase in monoclinic ZrO_2 phase in the tetragonal matrix. The amount of monoclinic phase (x_m) present was determined by the method of Garvie and Nicholson¹¹ using the following equation:

$$x_m = [I_m(111) + I_m(\bar{1}\bar{1}\bar{1})]/[I_m(111) + I_m(\bar{1}\bar{1}\bar{1}) + I_t(111)]$$

where, I_m and I_t are the intensities for monoclinic and tetragonal phases, respectively. Figure 5 shows the monoclinic phase observed on the fracture surface of the composites as a function of the whisker content. As shown in the figure, the fraction of monoclinic phase in the TZP matrix increased from ~5% to 18% as a result of 20% alumina whisker. The increase in the amount of transformed monoclinic phase Probably results from the induced internal stresses due to mismatch between thermal expansion coefficients of the TZP matrix and alumina whisker.

Figure 6 shows the dependence of elastic modulus on alumina whisker content. As expected, the elastic modulus increases with increasing

whisker content since the elastic modulus of alumina is ~400 GPa which is larger than that of TZP which is 200 GPa.

Figure 7 is a plot showing the variation of fracture toughness, K_{IC} , of composites with alumina whisker content. In view of the large scatter in data, it is difficult to predict exact dependence of K_{IC} on the whisker content, specially at whisker contents < 20%. On the other hand, at ~25% whisker content, the average K_{IC} has increased from ~8.7 to 10 MPa $\sqrt{m}$. It is believed that the increase in K_{IC} at 25% whisker content is partly due to crack deflection¹² (Fig. 4) toughening resulting from the presence of alumina whiskers. In addition, tetragonal to monoclinic transformation of ZrO_2 as discussed earlier is also believed to have contributed to the toughening process¹³⁻¹⁴. The increase in toughness of TZP due to 25 vol% addition of alumina whiskers is a modest 13%. The larger increase in toughness was not achieved because of the absence of other toughening mechanisms such as whisker pull out and crack bridging which require strong whisker. In the present case, the whiskers were weak due to the presence of large porosity. Furthermore, the surface degradation of the whiskers (Fig. 2) during hot-pressing made the surface very rough which would make it difficult for whiskers to pull out.

It is to be noted that the toughness data has a large scatter. This is due to the fact that whiskers in the hot-pressed composites developed inhomogeneous distribution of pore structure. As a crack propagates through the composite, whiskers are encountered at various orientations with varying degree of porosity. If the crack encounters a particular whisker at or near a pore, the crack propagates through the whisker while

expending little energy in doing so. In this case, little toughening occurs. On the other hand, if the crack encounters an intact whisker, or contacts a whisker far from any included pore, the crack is deflected around the whisker. This results in toughening due to additional work required in propagating a deflected crack. Therefore, for the same specimen, fracture toughness ranged from a small value to a large value depending on whether the crack interacted with a porous whisker or a dense whisker. This resulted in a large scatter in the data.

It appears that toughening contributions are being made, notably from the transformation of the tetragonal phase as well as from whisker reinforcement. But these contributions are offset to varying extent by weakened and porous whiskers. It is believed that large increase in fracture toughness is possible for these composites if the processing of whisker and/or composites are improved such that whisker degradations are eliminated or minimized.

Acknowledgement

This work was supported by the U. S. Department of Energy, under Contract W-31-109-Eng-38. The authors thank R. B. Poeppel, Manager of the Ceramic Section of the Materials and Components Technology Division at Argonne National Laboratory, for his encouragement and support of this work. Thanks are extended to Bill Carradine from Vista Chemical Company for providing alumina whiskers and to Ben Tani from Argonne National Laboratory for his help in x-ray analysis.

References

1. Becker, P.F. and Wei, G.C. "Toughening Behavior in SiC-Whisker-Reinforced-Alumina," J. Am. Ceram. Soc., 67 [12] C267-C269 (1984).
2. Lundberg, R. et. al., "SiC-Whisker-Reinforced Si₃N₄ Composites," Am. Ceram. Soc. Bull., 66 [2] 330-333 (1987).
3. Claussen, N. and Petzow, G., "Whisker-Reinforced Oxide Ceramics," Journal de Physique, 47 [2] C1-693-C1-702 (1986).
4. Claussen, N., Weisskopf, K. L. and Ruhle, M., " Tetragonal Zirconia Polycrystals Reinforced with SiC Whiskers," J. Am. Ceram. Soc. 69 [3] 288-292 (1986).
5. Akimune, Y., Katano, Y. and Shichi, Y., "Mechanical Properties and Microstructure of an Air-Annealed SiC-Whisker/ Y-TZP Composite," Advanced Ceramic Materials, 3 [2] 138-142 (1988).

6. Warren, R. and Sarin, V. K., "Fracture of Whisker Reinforced Ceramics," to be published in Application of Fracture Mechanics to Composite Materials, ed. by K. Friedrich, Elsevier, Amsterdam, The Netherlands.
7. VISTA Chemical Company, Internal Communication (1988).
8. Toyo Soda Manufacturing Company, Internal Communication (1988).
9. Evans, A. G. and Charles, E. A., "Fracture Toughness Determinations by Indentation," J. Am. Ceram. Soc., 59 [7-8] 371-372 (1976).
10. Kräutkramer, H. and Kräutkammer, H., Ultrasonic Testing of Materials, Springer-Verlag, New York (1983).
11. Garvie, R. C. and Nicholson, P. S., "Phase Analysis in Zirconia System," J. Am. Ceram. Soc. 55 [6] 303-305 (1972).
12. Faber, K. T. and Evans A. G., "Crack Deflection Processes I-Theory," Act. Met., 31[4] 565-576 (1983).
13. Evans, A. G., "Toughening Mechanisms in Zirconia Alloys," in Advances in Ceramics, vol.12, pp 193-212. Science and Technology of Zirconia II, edited by N. Claussen, M. Ruhle, and A. H. Heuer, Am. Ceram. Soc. Columbus, Ohio, 1984.
14. Heuer, A. H., "Transformation Toughening in ZrO₂ Containing Ceramics," J. Am. Ceram. Soc. 70 [10] 689-698 (1987).

Fig. 1. Scanning electron micrograph showing as-received alumina whiskers.

Fig. 2. Scanning electron micrograph of a alumina whisker reinforced TZP composite showing changed morphology of whisker in the hot-pressed composite.

Fig. 3. Scanning electron micrograph of polished and etched surface of a alumina whisker reinforced TZP composite showing TZP grains.

Fig. 4. Micrographs of polished surface of a alumina whisker reinforced TZP composite showing whisker / crack interactions: (a) crack deflected by a strong whisker, and (b) crack propagating straight through a weakened whisker near pores.

Fig. 5. Effect of whisker content on monoclinic phase observed on the fracture surface of the alumina whisker reinforced TZP composite.

Fig. 6. Effect of whisker content on the elastic modulus of the alumina whisker reinforced TZP composites.

Fig. 7. Effect of whisker content on the fracture toughness of the alumina whisker reinforced TZP composites.

END

DATE FILMED

02 / 04 / 91