INTERFACIAL COMPOSITION ANALYSIS OF SILICON NITRIDE WHISKER REINFORCED OXYNITRIDE GLASS SYSTEMS

E. Y. Sun, K. B. Alexander, P. F. Becher, and S. L. Hwang

Metals and Ceramics Division, Oak Ridge National Laboratory, Oak Ridge, TN 37831-6068

Microstructural and compositional analysis in many structural ceramics requires sophisticated analytical electron microscopy because of the light elements (O, N, C and B) present in the materials and the nano-scale amorphous phases at the grain boundaries. In the present study, scanning transmission electron microscopy (STEM) combined with energy-dispersive x-ray spectroscopy (EDS) and parallel-detection electron energy-loss spectroscopy (PEELS) was used to analyze the composition at interfaces in β - Si_3N_4 whisker reinforced oxynitride glass systems. Composition profiles were acquired by either manually stepping the probe across the interface or by sequential integration during slow-rate line-scanning with a < 2.0 nm probe in the STEM mode of a Philips EM400T/FEG microscope.

Silicon nitride, which has been recognized as one of the most promising candidates for high temperature applications, is typically densified by employing various sintering aids such as yttria and alumina. Reaction of the sintering aids and the oxide surface layer on Si_3N_4 particles forms secondary intergranular phases that are primarily silicon metal oxynitride glasses.¹ To study how the boundary phases influence crack-bridging toughening mechanisms, such as interfacial debonding and pull-out of the elongated β - Si_3N_4 grains,² model systems of β - Si_3N_4 whisker-reinforced SiAlYON glasses were fabricated. The nominal starting compositions of the glasses are listed in Table 1. For whiskers embedded in glasses with low Al:Y ratio (composite A and B), no interfacial layers were formed between the glass and the β - Si_3N_4 grains, as shown in the high resolution image in Fig. 1. In composites formed from glasses with high Al:Y ratio, the interfacial structure varied with the nitrogen content. A crystallized layer ~ 60 nm thick was present at the whisker/glass interface in the high-nitrogen systems (composite E) as shown in Fig. 2, whereas in the low-nitrogen glasses (composites C and D), no interfacial layer was observed. The composition profiles shown in Fig. 3 indicate that the interfacial layer formed in composite E contained Si and Al, with very low Y solubility, which is typical for SiAlON. SiAlON formation at interfaces during β - Si_3N_4 grain growth has also been observed via scanning electron microscopy (SEM) by Kramer *et al.*³ Interfacial debonding experiments performed on these composites indicated that glass chemistry had a strong influence on the debonding behavior.⁴ With the formation of a SiAlON solid solution layer at the interface, the energy required to debond the Si_3N_4 -glass interface increases significantly. In silicon nitride ceramics, such interfacial debonding processes are critical to the formation of bridging grains to achieve toughening effects. The above results suggest that the fracture toughness of silicon nitride might be optimized by tailoring the intergranular amorphous phases.

References

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Table 1. Composition of the oxynitride glasses.

Glass	Si eq%	Al eq%	Y eq%	N eq%	O eq%
A	55	10	35	10	90
B	55	10	35	20	80
C	55	25	20	5	95
D	55	25	20	10	90
E	55	25	20	20	80

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FIG. 1.-High resolution TEM image of silicon nitride/oxynitride glass interface in composite B.

FIG. 2.-TEM image of SiAlON formation at interface between silicon nitride whisker and oxynitride glass in composite E.

FIG. 3.-Composition profiles as determined by EDS analysis of cation species near interface in composite E.

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