TEM Evaluation of Interface of SiC-Whiskers-Reinforced Si_3N_4

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Abstract

An important aspect for mechanical properties in ceramic composites is to evaluate the effect of whiskers/matrix interface characteristics. This work presents microstructural analysis of whiskers/matrix interface of silicon nitride based ceramic composites, reinforced with silicon carbide whiskers. A high-resolution transmission electron microscope (HRTEM) has been used for such evaluation, showing mainly the presence of glassy phase at the whiskers/matrix interface and boundaries / triple junction of matrix grains. The predominant whiskers/matrix vitreous interface indicates weak interaction between whiskers and surrounding matrix, which can activate crack deflection and crack bridging toughening mechanisms, causing significant increase of fracture toughness in ceramics composites.

Keywords: Ceramics composites, Transmission electron microscopy, Silicon nitride, Silicon carbide whiskers, Interface whiskers/matrix.

Introduction

Research on ceramic composites has been widely studied and analyzed. Processing and characterization at room and high temperature are the main fields of study, taking into account the potential application of composites as structural materials. The applications of Si_3N_4-$SiC_{(w)}$ and Al_2O_3-$SiC_{(w)}$ composites include turbine components, cutting tools, etc., due to their high mechanical and thermal properties (1;5-6;14-15).

An important factor to be considered during processing of whisker reinforced ceramic composites is the need of a combination of whisker and matrix characteristics, bearing in mind the existing difference in terms of thermal expansion, elastic modulus and chemical compatibility, during the densification process (14). A better chemical compatibility is essential for high mechanical properties, considering the surface area and small diffusion distance involved in the use of whisker as reinforcement.

The preparation of ceramic composites with good chemical and thermal stability is possible, as long as an adequate choice of ceramic matrix is made, selecting those which provide smaller interdiffusion and interfacial reaction. Previous research used alumina, mullita and zirconia, with whiskers as reinforcement (11).

Some researchers (7;13) have examined the effect of the chemical surface of whiskers on mechanical strength and fracture toughness of composites, showing the importance of whiskers/matrix interface characteristics, affected by factors as formation of chemical bonding and differences of coefficient of thermal expansion.

Different toughness mechanisms can become active on structural ceramic composites, depending upon the type of bonding at the interface, the aspect ratio and volume fraction (2). As examples, there are crack deflection and crack bridging, both requiring a relatively weak interface between whiskers and matrix (3;10).

This work has a objective to evaluate the whiskers/matrix interface characteristics of silicon nitride based ceramics composites reinforced with silicon carbide whiskers from 10 and 30 vol.% by high-resolution transmission electron microscope (HRTEM).

Materials and Methods

The matrix was prepared using silicon nitride (H.C.Starck), aluminum nitride (H.C.Starck) and yttrium oxide (H.C.Starck). As reinforcement, silicon carbide whiskers were used (ICD-Group Inc.). Table I shows the used compositions for ceramic composites.

Table I: Compositions used in the fabrication of the ceramic composites

	Matrix (% weight)			Whiskers (% vol.)
samples	Si_3N_4	AlN	Y_2O_3	SiC
A	90	5	5	10
B	90	5	5	20
C	90	5	5	30

The powders plus whiskers were ball-milled for 24 h, using high purity alumina milling balls and dry ethanol. The slurry was dried and each sample die pressed at 50 MPa, followed by cold isostatic pressing at 300 MPa. Hot-pressed sintering was performed in graphite resistance furnace in nitrogen atmosphere, using a heating rate of 20 °C min, with holding time of 30 min at 1750 °C.

The samples were prepared from thin slices of each composition, mechanically polished to about 150 µm thickness and followed by dimpling and subsequent ion-beam thinning.

High resolution transmission electron microscopy (HRTEM) images were taken from interface and grain boundaries of Si_3N-$SiC_{(w)}$ composites, using a 300 kV Hitachi TEM. Bright and dark-field images and electron diffraction-patterns were used in the analysis.

Results and Discussion

Composition A

For composition Si_3N_4-10 vol.% $SiC_{(w)}$, figure 1, the presence of thin films of vitreous phase at whiskers/matrix interface were observed, indicating an weak interaction between whiskers and surrounding matrix.

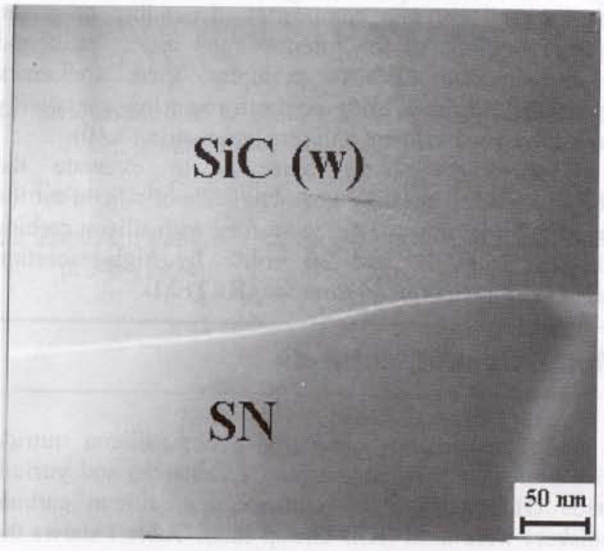

Figure 1 – Bright-field image of composition Si_3N_4-10 vol.% $SiC_{(w)}$ showing thin films of vitreous phase (2nm) at whiskers/matrix interface.

In such case, depending upon the load condition, crack deflection and crack bridging toughening mechanisms would prevail.

Composition B

The composition Si_3N_4-20 vol.% $SiC_{(w)}$, figure 2, showed the presence of vitreous phase at triple junction of the matrix grains and whiskers/matrix interface. The Figure 3 presented the electron diffraction pattern triple junction of the grain matrix at point 2.

In Si_3N_4 based ceramics composites, to which sintering aids were added during sintering process, a non-crystalline phase is generally dispersed throughout the microstructure, at triple junction grains and at grain boundaries.

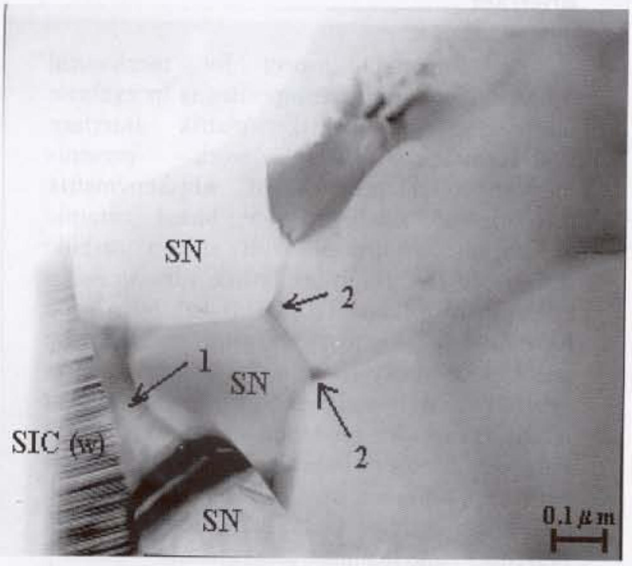

Figure 2 –Bright-field image of composition Si_3N_4-20 vol.% $SiC_{(w)}$. The presence of vitreous phases at triple junction of the matrix grains (points 2) and whiskers/matrix interface (point 1) is observed.

Figure 3 - Electron diffraction pattern triple junction of the grain matrix (point 2) at figure 2.

Also the whiskers surface revealed the high concentration of planar faults, related to internal structural characteristics, giving rise to intense streaking in the corresponding electron-diffraction pattern, showed in figure 4. This one-dimensional disorder perpendicular to the growth axis does not allow assigning a single well-defined polytype to the whiskers (12;8).

Some authors (4;9) have defined that the whiskers surface have tiny microfacets of alternating planes. The nature of the surface is clearly visible in low angle SEM micrographs. The notches dimensions formed in the sequence of the stacked polytypes are correlated with distances between the planar faults intersecting the whiskers surface.

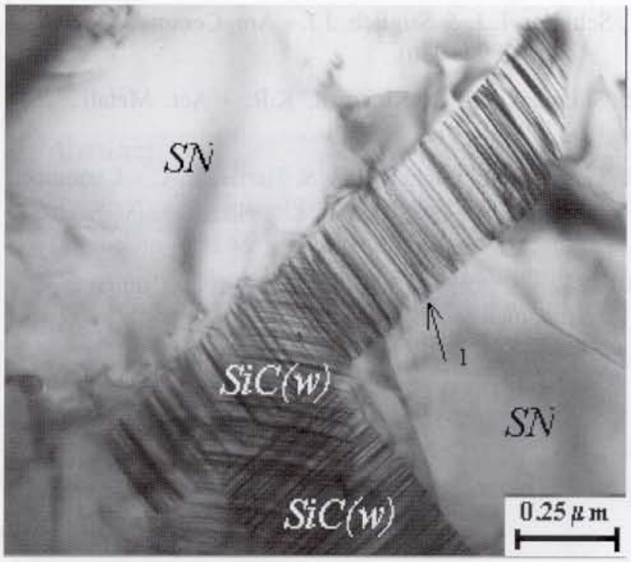

Figure 4 – The whiskers surface reveal the high concentration of planar faults, related to internal structural characteristics.

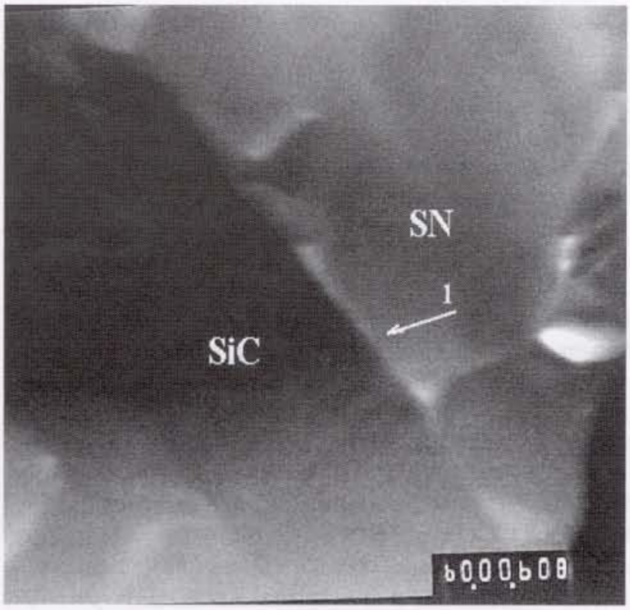

Figure 5 – Dark field images of composition Si_3N_4-30 vol.% $SiC_{(w)}$ showing presence of vitreous phases at whiskers/matrix interface (point 1).

Composition C

For dark field and bright field images, figure 5 and figure 6 respectively, was observed the presence of vitreous phase at whiskers/matrix interface in composition with 30 vol.% $SiC_{(w)}$.

However, given the oxygen-rich surface chemistry of as-received SiC whiskers, the presence of the continuous vitreous phase at the whiskers/matrix interface and grain boundaries is expected, particularly in the Si_3N_4 base composite, to which oxide sintering aids were added.

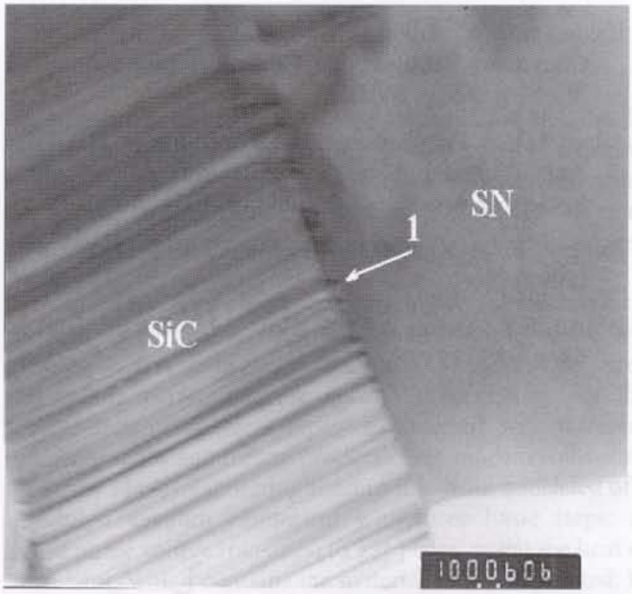

Figure 6 – Bright-field images of composition Si_3N_4-30 vol.% $SiC_{(w)}$ showing presence of vitreous phases at whiskers/matrix interface (point 1).

Conclusions

The analysis by HRTEM showed mainly the presence of glassy phase at whiskers/matrix interface and triple junction of matrix grains. The predominant whiskers/matrix vitreous interface indicates weak interaction between whiskers and surrounding matrix, which can activate crack deflection, pullout and crack bridging toughening mechanisms, resulting in significant increase of fracture toughness in ceramic composites.

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