

TEM STUDY OF SiC - PLATELETS / Si₃N₄ CERAMIC COMPOSITES

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Silicon nitride based ceramics are attractive materials for structural applications because they show low density, high strength and good oxidation resistance at high temperatures. Improved mechanical and creep resistance through the incorporation of reinforcements such as whiskers and platelets have been reported by previous works^[1-3]. Silicon nitride and its related composite materials can be used at high temperatures, so it is important to study the creep and thermal shock resistance^[1,4]. The determination of microstructural parameters, such as the existence and location of the glassy phase are very important to understand the material behavior, because liquid phases formed by additives during the sintering process remain at grain boundaries and triple junction after cooling.

In the present work 20v/o silicon carbide platelets reinforced silicon nitride was investigated. Samples were prepared by hot pressing silicon nitride powders (SNE 10, Ube Corp., Japan) containing 5 and 2 wt% of aluminium and yttrium oxide, respectively, as sintering additives. As reinforcing phases 20 v/o of silicon carbide platelet single crystals (32-50 μm , American Matrix, Knoxville, TN, USA) were added. The material was hot pressed at 1800°C in graphite dies. The processing procedure was described in previous works^[1,5].

X ray diffraction patterns of hot pressed composites indicated the presence of β -Si₃N₄ and α -SiC only.

The cross sectioned samples were prepared by polishing and plasma etching (100 s in a gas mixture of CF₄ and O₂) and analyzed by scanning electron microscopy. The fracture surfaces were also analyzed by SEM. Samples for transmission electron microscopy (TEM) analyses were prepared by using a conventional thinning procedure, that includes dimple grinding and argon ion milling, and finally they were coated with a

thin carbon film. TEM was used to investigate the presence and localization of crystalline and amorphous phases formed during material processing.

Figure 1 and 2 show the SEM-micrographs of fracture surfaces, where SiC platelets can be seen in the β -Si₃N₄ matrix. The β -Si₃N₄ needle-type grain with an average grain size of approximately 0.3 μ m is depicted in figure 3. The plasma etching process reveals the glassy phase (light grain boundary phase) and the silicon nitride grains (gray regions).

The TEM results show the silicon nitride matrix and a secondary phase (probably containing yttrium) at grain boundaries and triple points (figure 4 and 5). From the diagram spots in figure 6 it was possible to determine that the β -Si₃N₄ grain is oriented in the [0001] direction. A Difpat software^[6] was used to perform the crystalline orientation.

The TEM analyses (defocus and dark field with electron incoherently scattered) show that the secondary phase is a glass phase distributed among the silicon nitride grains and between the silicon carbide platelets and silicon nitride grains (figure. 7 and 8)

The existence of a glass phase film along the platelets and the silicon nitride grains, and its behavior at high temperatures can explain the experimental data for this material^[1,4].

References

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5. G.Schubert, W.Acchar, H.Hübner and R.Wagner, Herstellung and Hochtemperatureigenschaften von plateletverstärktem HPSN. Work presented in Seminar "Verstärkung keramischer Werkstoffe" Hamburg-Germany, 1991.
6. Software developed by Graham Carpenter and Laris Benkis at Metallurgical Laboratory CANMET , Ottawa-Canada.



Figure 1- SEM micrograph of the fracture surface



Figure 2 - SEM micrograph of the fracture surface

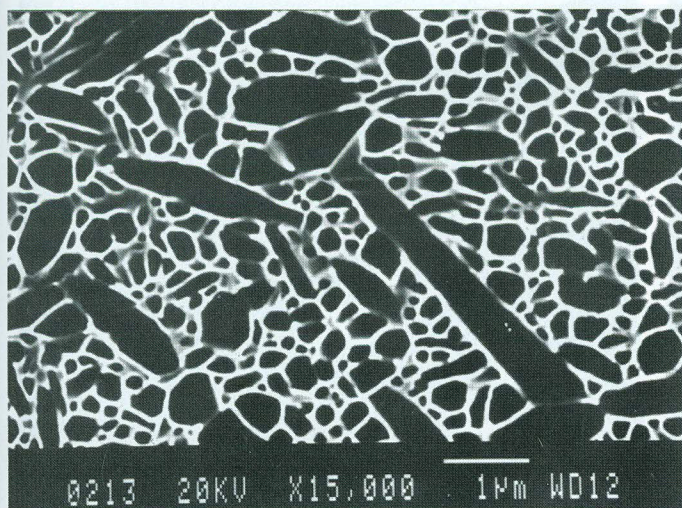
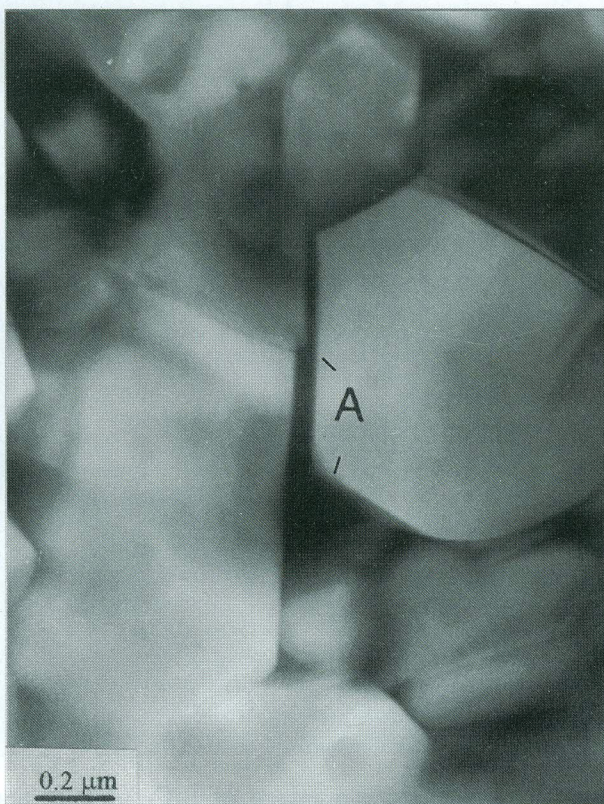


Figure 3 -SEM micrograph of the Si₃N₄ matrix

Figure 4 - Bright field image of Si₃N₄ grains and amorphous phase (A).



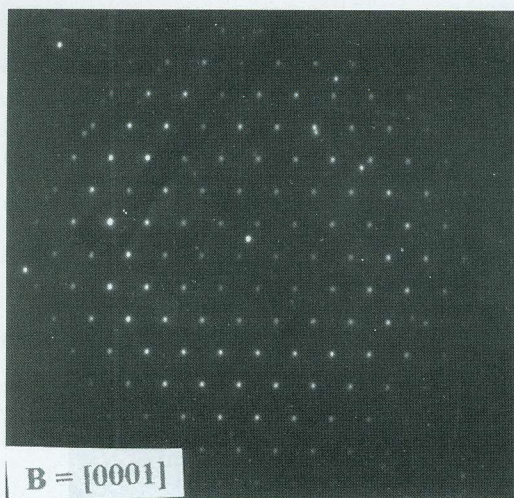


Figure 5 - SAD pattern of Si_3N_4 -grain.

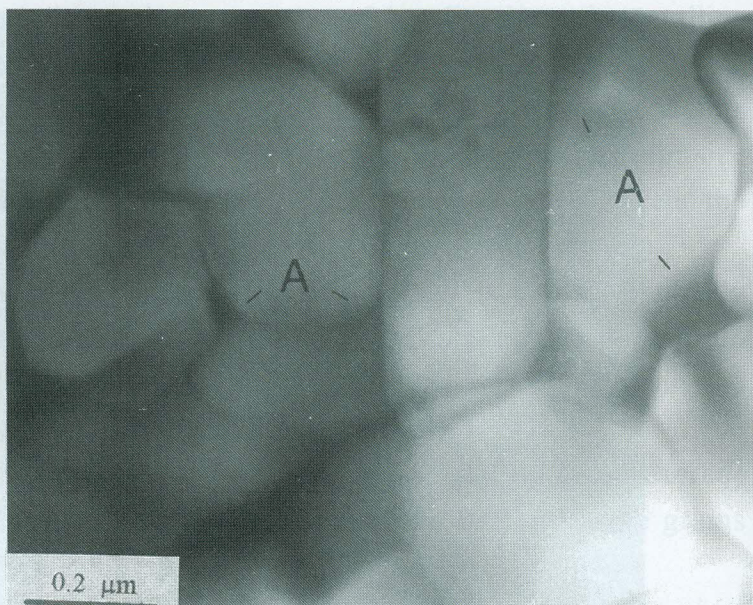


Figure 6 -Bright field image of Si_3N_4 grains and amorphous phase (A).

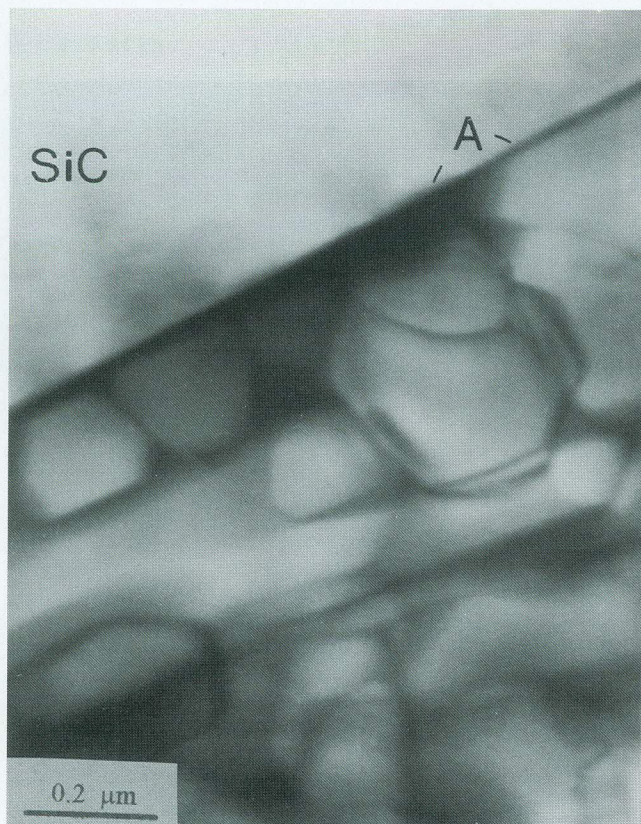


Figure 7 - Bright field image of SiC- platelet and Si_3N_4 -grains.

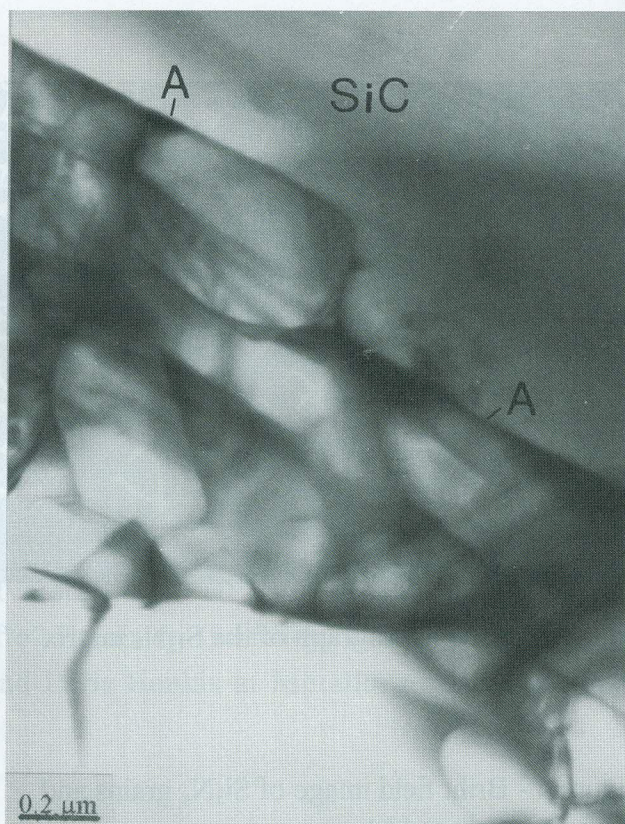


Figure 8 - Bright field image of SiC- platelet and Si_3N_4 -grains