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Stereological Study of Whisker Reinforced Silicon Nitride Ceramics

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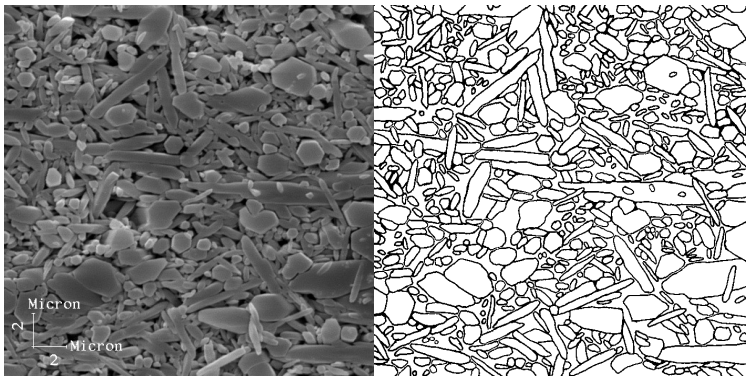
Abstract – A whisker reinforced silicon nitride composite was processed with 5 wt% of β-Si_3N_4 whiskers and 3 % de MgO. The material was hot press sintered at 1750 ºC by 30 minutes. The microstructure was revealed by HF etching, digitalized and analyzed. It was observed the silicon grains and the removal of the intergranular phase. The average aspect ratio of 2.57, the fracture toughness of 5.9 MPa.$m^{1/2}$ and a flexure strength of 863 MPa.

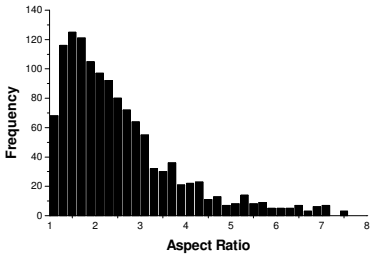
Silicon nitride (Si_3N_4) posses high strength, low thermal expansion coefficient and high fracture toughness, when compared to other advanced ceramics [1]. These properties, especially the fracture toughness, are very dependent on the idiomorphic microstructure, which is formed by α→β phase transformation during the liquid phase sintering. It is a tridimensional rod-like grain network that causes crack deflection or the whisker/grains pull out toughening mechanisms [2].

The quantification of the microstructure is quite complex, since it is necessary to infer a space distribution from a single plane observation of a non-equiaxed grain. Also, typical etching methods remove the intergranular phases formed during sintering [3], which can alter the grains dimension and morphology. This study had two objectives: to exemplify the difficult in quantifying the Si_3N_4 microstructure and to demonstrate that digital image analysis can lead to very good representation of the grains and pores structure, from which a statistical analysis can be performed.

An α-Si_3N_4 powder was mixtures with β-Si_3N_4 whiskers (5 %wt) and MgO (3 %wt) for 12 hours, dried, desaglomerated and hot pressed at 1750 ºC for 30 min. Then, it was cut, grinded, polished and etched with HF at 110 ºC. The microstructure is shown in Figure (left), where the tridimensional rod-like grains can be observed. The longer grains are the whicker reinforcement added. It is also noted that the intergranular phases were removed, leaving pores behind. The digital image at the middle is considered to a very representation of micrograph. To the right, a statistical analysis was performed on 717 grains. It showed that, on the observation plane (perpendicular to the hot pressing direction), there was a monomodal distribution, most of the grains had the aspect ratio between 1.5 to 3 and the average aspect ratio of 2.57. The present microstructure resulted in a fracture toughness of 5.9 MPa.$m^{1/2}$ (Indentation Method), which was attributed the crack deflection mechanism, and an average four-point flexure strength of 863 MPa.

To quantify the microstructure of whisker (or short fiber) reinforced materials, such as Si_3N_4, it is necessary the aids of software analysis. Even though the intergranular phase has been removed by the etching used, it was still possible to clearly observe the grain microstructure.





References

[1] X.-J. Liu, Z.-Y. Huang, Q.-M. Ge, X.-W. Sun, L.-P. Huang, J. Eur. Cer. Soc. 25 (2005) 3353.
[2] G. Ziegler, J. Heinrich, G. Wötting, J. Mat. Science, 22, (1987) 3041
[3] C.V. Rocha, DSc. Thesis, Universidade Federal do Rio de Janeiro, Brazil, (2004)