PROPERTIES OF SILICON NITRIDE-SILICA CERAMICS SINTERED BY HIP

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ABSTRACT
High-purity Si₃N₄ containing 2.5 to 20 wt% SiO₂ additions were fabricated by hot isostatic pressing. Addition of SiO₂ promoted densification and delayed the α-β transformation rate. The fracture toughness was insensitive to the microstructure and glass content, but the Vickers hardness depended strongly on both. The room temperature strength decreased with SiO₂, probably due to increase in the initial flaw size. Vickers hardness showed no drastic degradation up to 1200°C regardless of SiO₂ content and sintering temperature.

INTRODUCTION
Si₃N₄ is one of the most important high-temperature materials. It has been successfully used as turbo-charger rotor for temperatures up to ~1000°C, but applications at higher temperatures are still limited due to strength degradation caused by the intergranular glassy phase. Therefore, great effort is being paid to improve the refractoriness of the intergranular glassy phase by, for example, the use of high-purity Si₃N₄ powders and suitable sintering aids.

Recently we succeeded in fabricating a series of high-purity Si₃N₄-SiO₂ ceramics [1,2,3], by hot isostatic pressing (HIP) of Si₃N₄ and SiO₂ powders using a glass encapsulation technique [4]. In these ceramics, the added SiO₂ remained as glassy phase at the grain boundaries, but fracture strengths of these ceramics were not degraded at 1400°C when the SiO₂ addition was ≤ 10 wt%.
As a part of the study towards better understanding of the basic properties of Si₃N₄ ceramics, this paper reports on the mechanical properties of Si₃N₄-SiO₂ ceramics from room temperature to high temperatures.

MATERIALS AND METHODS

The starting Si₃N₄ powder and SiO₂ powder both contained < 0.01% of cation impurities. The Si₃N₄ powder had an oxygen impurity content of 1.3 wt%, and a specific surface area of 10 m²/g. This powder was composed of 97% α-Si₃N₄ and 3% β-Si₃N₄. The SiO₂ powder had a specific area of 170 m²/g. These two powders were mixed by ball-milling for 12 h in ethanol using Si₃N₄ balls and polyethylene container. The Si₃N₄-SiO₂ ceramics were prepared by HIPing of these mixtures at 1900~2000°C in argon gas of 170 MPa for 1 h using the glass encapsulation method.

Density, phases of the sintered bodies were characterized by Archimedes method and X-ray diffraction [5], respectively. Fracture toughness and hardness were measured by Vickers indentation method with a 49 N load and 15 s loading time [6].

RESULTS AND DISCUSSION

Sintering behaviors

The densification and phase transformation behaviors of Si₃N₄ were found to depend strongly on SiO₂ content. Figure 1 shows the results at 1900, 1950 and 2000°C. Addition of 10 wt% SiO₂ led to complete densification at a sintering temperature 100°C lower than that required for full densification of additive-free Si₃N₄, and resulted in a retention of > 60% α-Si₃N₄: It

![Figure 1](attachment:image.png)