

Effects of the characteristics of silicon nitride powders on the preparation of α' -sialon ceramics

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Effects of the characteristics of silicon nitride powders on the preparation of α' -sialon ceramics

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Oxygen impurity is always present at the surface of silicon nitride particles [1], which plays a very important role in the densification of silicon nitride or oxynitride ceramics by forming a eutectic oxide liquid phase with oxide sintering additives during firing. Furthermore, the α -silicon nitride content, particle size and other impurities also affect the densification behaviour. This letter reports the influence of the characteristics of silicon nitride powders on the preparation of α' -sialon ceramics, which promise improved thermal shock resistance and excellent mechanical properties at high temperatures [2].

The chemical composition and properties of the silicon nitride powders used are summarized in Table I, which shows the difference in oxygen impurity and α -phase content. After powder processing the oxygen content increases and the differences become smaller. Three sets of specimens were prepared by mixing 15.25 wt% AlN (Grade C, Starck) and 9.34 wt% Y₂O₃ (99.99% purity, Ventron) to 75.41 wt% Si₃N₄ using for the different sets powders A, B and C (see Table I). After sintering α' -sialon ceramics are formed [3]. Sintering was conducted in a gas pressure furnace with a dilatometer.

The reaction sequence of the formation of α' -sialon is determined by means of X-ray diffraction (XRD). The experimental results indicate that the oxide eutectic liquid phase appears above 1300 °C, the dissolution of silicon nitride into the liquid occurs at approximately 1380 °C and the

precipitation of α' -sialon follows immediately (Fig. 1). When the temperature rises to 1800 °C the formation of α' -sialon is nearly completed. Although the impurities affect the surface tension of particles and the composition of the liquid phase, and the solubility and mobility of silicon nitride in the liquid phase are consequently varied, no difference in the reaction sequence has been observed. Similar results have been observed after the addition of some oxides into the mixtures [4]. Thus, the formation of α' -sialon is assumed to be a process which requires the presence of a liquid phase, but is not strongly influenced by the quantity and composition of this liquid phase.

The densification of α' -sialon ceramics was followed by means of *in situ* dilatometry during sintering. The shrinkage behaviour is strongly affected by the powder characteristics, as can be seen in Fig. 2. The densification of these three samples starts approximately at or above 1380 °C, which corresponds to the dissolution of nitrides into and the precipitation of α' -sialon from the liquid. However, samples B and C exhibited a very poor sinterability. Up to 1800 °C only a relative density of <76% theoretical had been reached and a large number of open pores remained. When a high nitrogen pressure of 10⁷ Pa was subsequently employed at higher temperatures, no further densification was observed. In contrast, the densities decreased slightly, accompanied by a large weight

TABLE I Chemical composition of silicon nitride powders used in this investigation

	LC-12 A	DFV0014 B	DFV0015 C
O (wt %)	1.57	1.10	0.34
	1.80 ^a	1.50 ^a	0.61 ^a
C (wt %)	0.18	0.12	0.12
Fe (p.p.m.)	50	71	15
Al (p.p.m.)	44	12	8
Ca (p.p.m.)	4	3	3
$\alpha/(\alpha + \beta)$	95	71	73
FSSS (μm)	0.55	0.66	0.53

^aAfter powder processing.

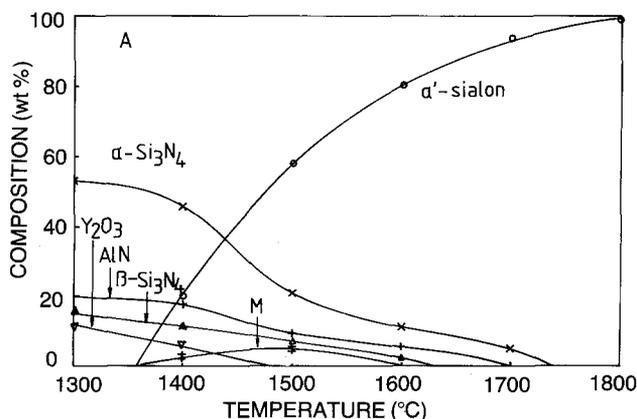
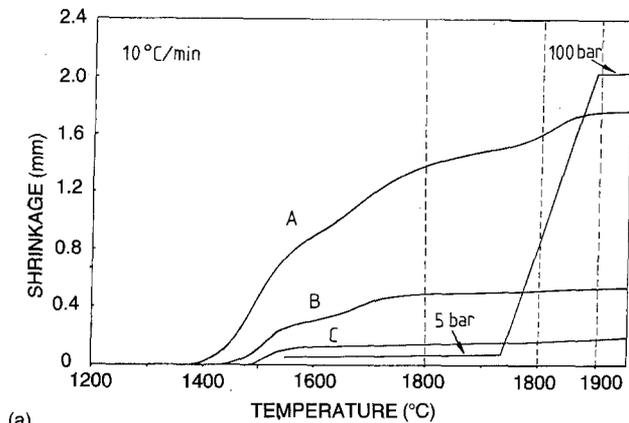
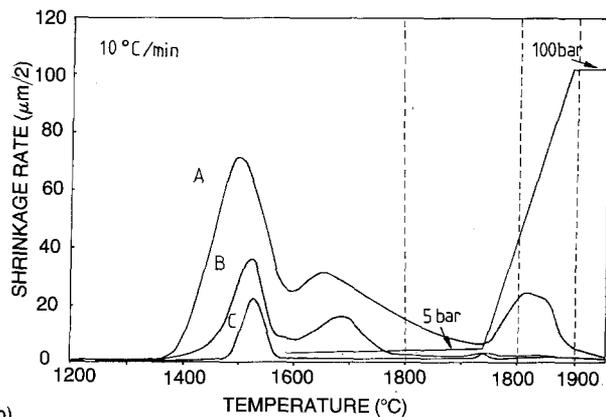


Figure 1 The reaction sequence of the formation of α' -sialon; heating rate 10 °C min⁻¹. M, Y₂O₃-Si₃N₄.



(a)



(b)

Figure 2 (a) Shrinkage and (b) shrinkage rate curves for the sintering of α' -sialon ceramics based on various starting silicon nitride powders, $10\text{ }^\circ\text{C min}^{-1}$.

loss (Fig. 3), probably due to a large internal surface area. The microstructure evolution suggests that for the sintering of α' -sialon ceramics contact-flattening is dominant in the early stage below $1600\text{ }^\circ\text{C}$, whereas further densification is brought about mainly by the Ostwald ripening mechanism, resulting in a fast grain growth [4]. The mixtures based on silicon nitride powders with a small amount of oxygen impurity and a low α -phase content exhibit a very poor sinterability. Thus, a certain amount of oxygen impurity and high α -phase content in silicon nitride powder are considered necessary to achieve fully dense α' -sialon ceramics.

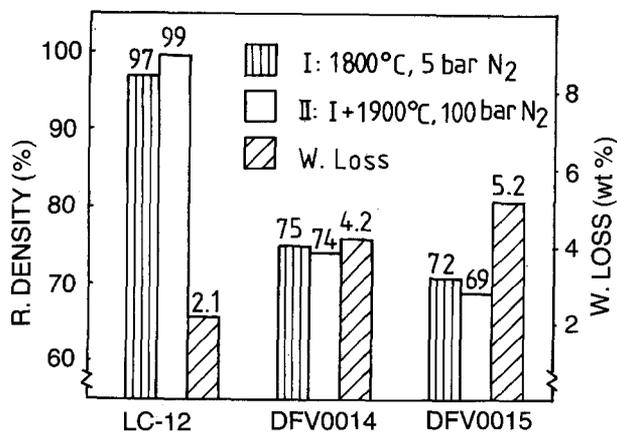


Figure 3 Densities and weight loss of α' -sialon ceramics based on various starting silicon nitride powders. (▨) I, $1800\text{ }^\circ\text{C}$, 0.5 MPa N_2 ; (□) II, $1900\text{ }^\circ\text{C}$, 10 MPa N_2 ; and (▩) weight loss.

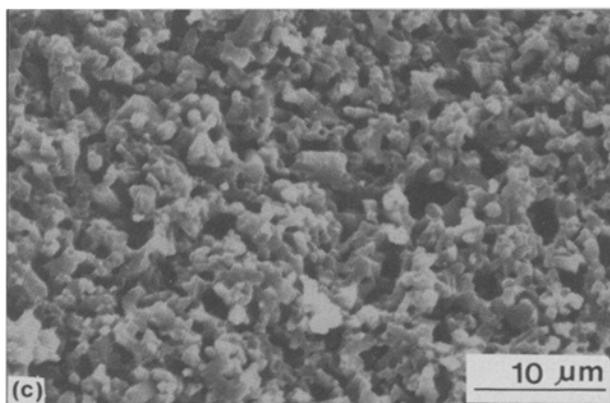
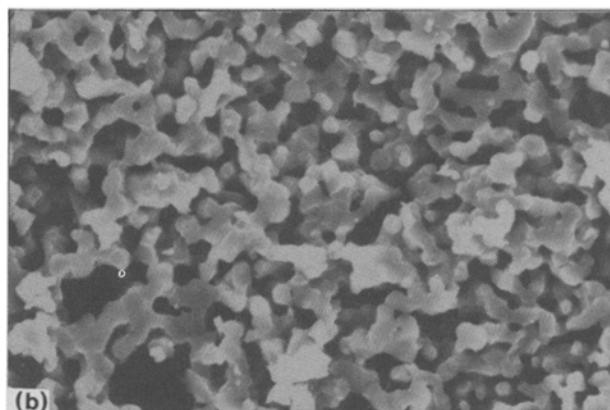
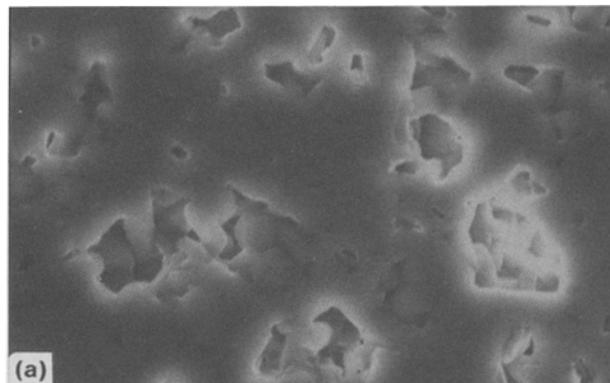


Figure 4 SEM micrographs of α' -sialon ceramics for samples (a) A, (b) B and (c) C.

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References

1. M. PEUCKERT and P. GREIL, *J. Mater. Sci.* **22** (1987) 3717.
2. K. H. JACK, in "Progress in nitrogen ceramics", edited by F. L. Riley, NATO ASI Series E65 (Martinus Nijhoff, The Hague, 1983) p. 45.
3. Z. K. HUANG, P. GREIL and P. PETZOW, *J. Amer. Ceram. Soc.* **66** (1983) C96.
4. G. Z. CAO, R. METSELAAR and G. ZIEGLER, in "Ceramics Today-Tomorrow's Ceramics", edited by P. V. Vincenzini (Elsevier Science Publishers B.V., Amsterdam 1991), p. 1285.

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