Microstructural Development and Secondary Phases in Silicon Nitride Sintered with Mixed Neodymia/Magnesia Additions

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ABSTRACT
The pressureless sintering of silicon nitride powder with mixed neodymia/magnesia additives is reported. Of the sixteen additive combinations studied the majority give rise to rapid densification after firing in 1 atm nitrogen for 30 min at a temperature of 1650°C and densities of 3050 kg m⁻³ are achieved. Such rapid densification is attributable to a large volume of liquid formed during densification. The α→β transformation is controlled by the additive level and combination ratio and, in certain compositions, the optimum requirement of high density and virtually complete transformation are obtained simultaneously. The resulting β-grain morphologies are acicular and interlocking suggesting that ceramic compositions within this easily densified silicon nitride/additive system should have good mechanical properties. Preliminary work shows that the intergranular glass can be devitrified to give more beneficial crystalline phases.

1. INTRODUCTION
Silicon nitride-based ceramics have achieved considerable commercial success because of their high strengths, wear resistance and resistance to thermal shock. A significant feature of the fabrication process for β'-sialons, for example, is the ease with which they can be densified with low levels of additives by pressureless sintering.¹ Furthermore, an
interlocking fibrous grain morphology, which gives rise to their high strengths, is observed simultaneously with completion of densification. Rapid densification is obtained with $\beta'$-sialons because of the large volume of liquid formed during sintering but because aluminium and oxygen are absorbed from this liquid into precipitating $\beta'$-sialon grains, the amount of liquid remaining after complete densification is small. Moreover, since any remaining grain-boundary glass can be converted into a beneficial refractory phase, such as yttrium aluminium garnet, high strengths can be maintained to temperatures of up to 1300°C.2

Wötting and Ziegler4 have demonstrated the importance of the shape and aspect (length to diameter) ratio of $\beta$ silicon nitride grains in determining strength and resistance to fracture of material pressureless sintered with mixed yttria–alumina additives. These authors have also shown that grain morphology and aspect ratio can be controlled by the amount of additive used during sintering. Thus there is a clear need to identify additive systems which promote a fibrous grain morphology.

Magnesia as a single additive to silicon nitride promotes good densification but an equi-axed low $\beta$ phase material, whereas yttria additions result in a fibrous high $\beta$ phase material but densification is poor.5 The object of studies being carried out at NIHE Limerick is to examine different additive systems for silicon nitride which do not involve the use of alumina and previous work6 concentrated on mixed magnesia–yttria additions. Results obtained showed that although a fibrous high $\beta$-phase grain morphology could be easily developed, densification was difficult. More recently, mixed magnesia–neodymia additions have been investigated and this paper reports the initial results obtained.

2. EXPERIMENTAL

The silicon nitride powder used in this work was supplied by H. C. Starck (Berlin) (LC 12 grade). Analar grade magnesia and 99.99% pure neodymia were used as mixed additives.

The powders were blended in iso-propanol for 5 h after which the alcohol was evaporated off and the powders dry mixed for a further 30 min. Quantities of powder were then die-pressed and embedded in a mixture of 50 wt % boron nitride + 50 wt % silicon nitride and