

Preparation and characterization of SiAlON matrix composites reinforced with combustion synthesis rod-like SiAlON particles

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ABSTRACT

SiAlON matrix composites were prepared from a precursor reaction mixture of α -Si₃N₄, AlN, Al₂O₃ and Y₂O₃ powders and reinforced with different proportions of elongated SiAlON particles obtained by combustion synthesis. Green cylindrical specimens were consolidated by slip casting from well dispersed suspensions. The dried samples were densified by hot pressing at 1650 and 1700 °C and then characterized for density, phase composition and microstructure. Hardness tests, fracture toughness by indentation and pin on disc wear, were also chosen to characterize their mechanical properties. The effects of the processing variables (sintering temperature, proportion of added rod-like SiAlON particles) on the microstructural features and on the mechanical properties were studied and a good correlation between them was found.

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1. Introduction

The ceramics based on Si₃N₄ constitute an important class of structural materials. They are adequate for many commercial applications that require high resistance to high temperatures, chemical stability, and high toughness and wear resistance [1–3]. The densification of pure silicon nitride is extremely difficult because of the covalent nature of the bonds between Si and N. According, the densification of Si₃N₄-based materials is only possible with the help of high-pressure (as in the case of the hot pressing) or by adding oxide additives (namely Al₂O₃ and Y₂O₃) that, upon heating, react with SiO₂ present at the surface of each particle of Si₃N₄, forming a glass phase that enhances the densification process [1–6]. The components of this glass phase might also enter in the structure of Si₃N₄ forming the denominated SiAlON solid solutions. There are two phases of major importance in engineering: α - and β -SiAlON [6,7]. The β -SiAlON phase is formed by the substitution of the bonds Si–N for Al–O, the basic structure being characterized by the stack sequence of the ABAB... plans and is defined by the general formula Si_{6–z}Al_zO₂N_{8–z} [6,7]. α -SiAlON is the solid solution formed by the substitution of the bonds Si–N for Al–O and Al–N, the electronic charge balance being established by the addition of metallic cations in the interstitial network channels giving rise to a more complex structure, which is characterized by the stack sequence of the ABCDABCD... plans. The general formula can be represented by Me_xSi_{12–(m+n)}Al_{m+n}O_nN_{16–n} in which

Me is a metal like Ca, Mg or a rare earth element, except Ce and La, with m representing the number of bonds Si–N substituted for Al–N and n the number of bonds Al–O [6,7]. The different structures of the two polymorphs tend to reflect in the grains' morphology and on the mechanical properties. α -SiAlON is harder and usually occurs in equiaxed grains that confer low toughness, while the softer β -SiAlON tends to develop in elongated grains that enhance toughness.

Recently, Chen et al. [8–10] have shown that rod-like SiAlON particles could be easily prepared by combustion synthesis, suggesting them as reinforcing agent for SiAlON matrix composites. In a follow up work, the same authors demonstrated that the toughness of α -SiAlON could be improved to the same level of that of β -SiAlON by incorporating 20% of rod-like α -SiAlON particles in a α -SiAlON matrix [11]. However, adding elongated particles to a green compact hinders its densification process as predicted by the constrained network model of Lange [12] and requires the use of an external applied pressure. Other approaches to improve the fracture toughness of α -SiAlON were undertaken and some recent studies showed that α -SiAlON could also develop into elongated morphology by carefully controlling the nucleation and growth process [13–16]. These discoveries represent great breakthroughs, suggesting that hard and tough α -SiAlON ceramics can be developed.

In the present work, SiAlON-based ceramic matrix composites reinforced with different added amounts of rod-like SiAlON particles obtained by combustion synthesis process (CS) were consolidated by slip casting. The green bodies were densified by hot pressing, according to the approach proposed before [11].

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However, in that work the precursor mixtures were consolidated from the dry powders. The aim of the present work was to evaluate the influence of the added amount of reinforcement and of the colloidal processing route on the microstructure and the mechanical properties of the sintered composites. Hardness tests, fracture toughness determined by the indentation method and pin on disc wear, were chosen to characterize their mechanical properties.

2. Experimental

2.1. Composition

The starting powders used to prepare the SiAlON ceramics were α -Si₃N₄ (H.C. Stark, Germany, d_{50} = 0.38 μ m), Al₂O₃ (Alcoa Chemicals, USA, d_{50} = 0.38 μ m), AlN (H.C. Stark, Germany, d_{50} = 2 μ m) and Y₂O₃ (H.C. Stark, Germany, d_{50} = 0.75 μ m).

In the present case, the composition of the ceramic matrix was kept constant (76.92 wt.% Si₃N₄ + 13.46 wt.% AlN + 5.77 wt.% Y₂O₃ + 3.85 wt.% Al₂O₃), corresponding to m = 1.5 and n = 1.0 in the general formula. Y with an ionic radius of 0.89 Å was selected as the stabilizing cation for the α -SiAlON phase. It is known that the thermodynamic stability area of the phase diagram on the α -SiAlON plane increases with decreasing the size of the stabilizing cation and with increasing temperature [6].

Four different compositions for the ceramic matrix composites were studied, which only differ in the amount of added reinforcing phase (5, 10, 20 and 30 vol.%), as detailed in Table 1. The first two digits in the sample codes stand for the added amount of reinforcing, while T1 and T2 stand to sintering temperatures (1650 °C and 1700 °C, respectively) as indicated in the last column. The phase assemblage of the rod-like particles produced by CS presented in Fig. 1 shows that β -SiAlON is the predominant crystalline phase.

Table 1
Experimental conditions and the respective sample codes.

Sample code	Added amount of reinforcing agent (%)	Temperature (°C)
05T1	05	1650
05T2	05	1700
10T1	10	1650
10T2	10	1700
20T1	20	1650
20T2	20	1700
30T1	30	1650
30T2	30	1700

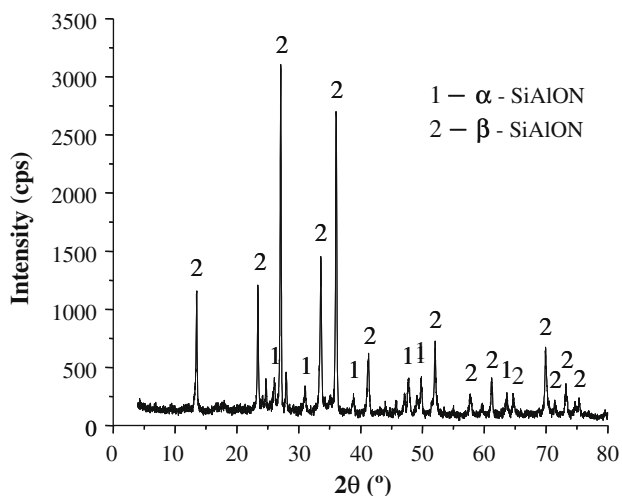


Fig. 1. X-ray diffraction pattern of the rod-like particles prepared by CS.

This information is important to better understand the phase assemblages detected in the sintered composites.

2.2. Materials and methods

2.2.1. Preparation of reinforcing phase

Rod-like SiAlON particles were prepared by combustion synthesis (CS) from a stoichiometric mixture of Si₃N₄, AlN, Al, Y₂O₃, Si and NH₄F powders corresponding to m = 1.5 and n = 1.0 in the general formula. The mixture was homogenized by ball milling in the ethanol for 8 h and then dried and put into a porous crucible with a diameter of 25 mm and a height of 40 mm. The crucible filled with the powders mixture was then placed into a high-pressure chamber described elsewhere [8–10]. The chamber was evacuated up to a vacuum of 10⁻⁴ MPa and then pressurised with nitrogen at the required pressure of 4 MPa. The mixed powders were then ignited by passing an electric current of 20 A through a tungsten coil.

2.2.2. Preparation of the green compacts

The powders of the precursor α -SiAlON matrix and the elongated SiAlON particles used as reinforcing agent were first mixed and homogenised in ethanol and then dried, deagglomerated and passed through an 80 μ m sieve [17,18]. The as obtained powders were then dispersed in an organic solvent consisting of an azeotropic mixture of 60 vol.% methylethylketon (MEK) (Riedel-de Haën, Germany) and 40 vol.% ethanol (E) (Merck, Germany) with the help of a dispersant, Hypermer KD1 (Imperial Chemical Industries PLC, England) [18–20]. Deagglomeration was carried out in a planetary mill for 4 h at a rotating speed of 150 rpm to prepare a stable suspension.

Slip casting experiments were carried out by pouring the suspensions into Teflon rings set on absorbent plaster blocks. The green bodies obtained by slip casting were let to dry at room temperature for 24 h and then completely dried in an oven at 110 °C for further 24 h [18–20].

2.2.3. Densification by hot pressing

The cylindrical slip cast samples were placed inside an appropriate graphite of a hot pressing machine equipped with a graphite resistance furnace and sintered at 1650 °C and 1700 °C. A constant heating rate of 15 °C/min was used up to the maximum temperatures. As soon as the maximum temperatures were achieved, a load of 20 MPa was applied and kept constant for a period of 60 min, at the end of which the power was switched off, letting the samples to cool inside the furnace. Table 1 summarises the experimental conditions used during the steps of preparation and sintering of the samples.

2.3. Characterization techniques

2.3.1. Microstructural characterization

The green compacts density was measured by the Archimedes method in mercury. The sintered density was measured using the same principle by immersing the samples in ethylene-glycol.

X-ray diffraction studies of the as prepared powders and of the sintered bodies were carried out using a high resolution Rigaku Geigerflex D/Mac, C Series diffractometer equipped with a graphite monochromator in the diffracted beam. XRD patterns were recorded with Copper K α radiation (λ = 1.5406 Å) produced at 30 kV and 25 mA scanned the diffraction angles (2θ) between 4° and 80° with a step size of 0.02° 2θ per second.

The microstructures of polished and chemically-etched surfaces by molten NaOH for 50 s, were observed by scanning electron microscopy SEM/EDS (Hitachi S-4100, Japan).

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