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Journal of the European Ceramic Society 33 (2013) 2191–2198

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cBN reinforced Y-α-SiAlON composites

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Received 14 December 2012; received in revised form 7 March 2013; accepted 9 March 2013
Available online 6 April 2013

Abstract

Dense α-Sialon–cBN composites were produced by FAST/SPS–sintering at 1575–1625 °C. The hardness of the materials increases only up to 21 GPa for materials with 10 vol.% cBN. On the other hand the fracture toughness increases up to nearly 8 MPa $m^{0.5}$ with 30 vol.% cBN. The reason for the increase in fracture toughness is attributed to crack deflection at cBN grains due to the weak bonding of the grains in the matrix. The weak interfaces are also responsible for the moderate increase in hardness. Detailed investigation of the interface between cBN and the matrix was carried out by TEM.

Keywords: Sialon; Boron nitride (cBN); Spark plasma sintering (SPS); Hardness; Toughness and toughening

1. Introduction

Silicon nitride ceramics have been widely investigated due to their advantageous properties of high strength, fracture toughness, thermal shock, chemical resistance and hardness at both room and elevated operating temperatures.[1–4] α-Sialon ceramics have improved hardness compared to β-silicon nitride ceramics, making them superior as cutting tool materials. The advantageous properties of Sialon ceramics have led to the advancement of Sialon as a cutting tool material selected for cast-iron and difficult to machine super alloys.

Improvements in the wear resistance of ceramic cutting tools can be achieved through the addition of hard particulates with good adhesion to the ceramic matrix. One possible candidate for reinforcing is cubic boron nitride (cBN) which is the second hardest material known to and used commercially by man after diamond.[5] It is known that cBN has a tendency to transform into its more stable hexagonal phase when sintered under relatively low pressures and temperatures above 1300 °C.[6] Therefore the use of spark plasma sintering (SPS) as a consolidation technique was selected due to the high heating and cooling rates achievable, which in turn reduces the exposure time of the cBN grains to high sintering temperatures, suppressing the transformation from the cubic to the hexagonal phase.[7]

Research into the fabrication of cBN containing Sialon composites has mainly been carried out with β-Sialon as the matrix material, where it was found that these β-Sialon/cBN composites exhibited maximum hardness, fracture toughness and flexural strength values of 15.4 GPa (Hv_5), 6.8 MPa $m^{0.5}$ and 432 MPa respectively with a 10 vol.% cBN addition.[8] Additionally, the reinforcing cBN grains were observed to transform into the hexagonal structure at an approximate sintering temperature of 1650 °C which consequently decreased the hardness of the composites. This effect became more pronounced with increased sintering temperature. Furthermore it was observed that increased heating rates could retard this transformation.[9] These data also revealed that the period of time spent above the cBN to hBN transition temperature is the decisive factor to the degree of transformation experienced. However, the use of SiO_2-containing phases (SiO_2, SiAlON and mullite) as the parent matrix results in a significant reduction in the hBN

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http://dx.doi.org/10.1016/j.jeurceramsoc.2013.03.014

transformation, in turn making cBN stable at higher temperatures as compared to other cBN containing composites (Al_2O_3, Al_2O_3-Ni, TiN and WC-Co).[10]

Zhao and Wang[11] specified that a 50 vol.% cBN containing β-Sialon composite with a relative density of 98.6 ± 0.9% could be obtained through SPS (Dr Sinter) at 1450 °C; additionally it was claimed that a $HV_{0.5}$ of 48.0 ± 0.9 GPa and fracture toughness of 11.5 MPa $m^{0.5}$ were obtained from a load of 4.9 N. The exceptionally high micro hardness values obtained by Zhao et al., indicate that the measurements were obtained solely on the cBN grains which had an average size of 7.5 μm. That value is close to the Vickers hardness of pure cBN. Currently no data exists concerning the interaction and behaviour of cBN in α-Sialon matrix compositions.

This study investigates the reinforcement of Y-α-Sialon ceramics through the addition of ≈10 μm cBN grains as the reinforcing agent.

2. Experimental procedure

The starting powders used to fabricate the Y-α-SiAlON matrices were α-Si_3N_4 (SN-10, UBE), AlN (Grade H, Tokuyama), Al_2O_3 (AKP 50, Sumitomo-Chemical) and Y_2O_3 (Grade C, H.C. Starck). The varying compositions of the Sialon ceramics were calculated on the basis of the overall formula of the Sialon phase $Y_{m/3}Si_{12-(m+n)}Al_{(m+n)}O_nN_{16-n}$.

The two compositions investigated had significantly different values of m and n and were named M1025 ($m = 1.0$ and $n = 2.5$) and M2045 ($m = 2.0$ and $n = 4.5$), respectively. The oxygen content of the nitride starting powders corresponding to 2.5 wt.% SiO_2 and Al_2O_3 in the Si_3N_4 and AlN powders respectively was accounted for in the calculation of the Sialon compositions. An additional 2 wt.% of Y_2O_3 was added in order to aid densification through the formation of a permanent liquid phase. The composition of M2045 was designed for comparison to that presented by Ye et al.[8] however the location of the M2045 Sialon composition is slightly above the Sialon plane in the Jänecke prism due to the addition of the 2 wt.% Y_2O_3.

The starting powders were mixed using a planetary ball mill for 4 h at a speed of 200 rpm. Agate milling media (bowl and 1 cm balls) was used along with isopropanol as the medium, after which the mixture was dried through the use of a rotary evaporator. The wear of the agate media was found to be negligible. The reinforcing cBN grains (Grade 9, Element 6 Pty. Ltd.) were introduced into the Sialon matrix in 10, 20 and 30 vol.% additions through the use of a dry Turbula mixing route (60 rpm for 2 h).

Sintering of discs 20 mm in diameter and approximately 5 mm thickness was carried out with the use of a SPS furnace (HP-D5 FCT, Germany) under vacuum with a constant dwell time of 5 min and uni-axial pressure of 50 MPa. The heating profile consisted of a two-step heating ramp rate, firstly 250 °C/min up to 1350 °C and secondly 50 °C/min up to the desired sintering temperature, cooling however was constant at 200 °C/min. The sintering temperatures investigated were 1550, 1575, 1600 and 1625 °C. The densities of the sintered compacts were measured through the use of the Archimedes method.[12] The crystalline phases were identified through XRD analysis (D2, Bruker) with Cu Kα radiation (30 kV and 10 mA). Measurements were taken between 10° and 60° with a step size of 0.02°. Microstructural investigations were carried out using a scanning electron microscope (FEI Quanta 400 FEG) accompanied with both secondary and back scattered electron detectors. Transmission electron microscopy investigations were also conducted to obtain a better understanding of the transformation/reaction zone between the Sialon matrix and cBN grains. TEM specimens were prepared by FIB (FEI Helios Nanolab 650) at 30 kV with final polishing at 500 V. The TEM investigation was carried out using a JEOL JEM 2100 microscope. Confirmation of boron nitride crystalline phases was completed through matching of experimental SAED patterns to simulated SAED patterns using JEMS simulation software package.[13] The Vickers hardness (Hv_5) was measured using a Leco V-100-A2, where at least five indentations were used per hardness value. The indentation fracture toughness was determined from crack measurements under the same load calculated through the description given by Anstis et al.,[14] where the main requirements to be met are a homogenous microstructure with a parallel mirror polished surface free of pores and cracks.[15] A Young's modulus of 300 GPa was used for the Sialon matrix.[16,17]

The values determined by indentation fracture toughness have limitations; a detailed evaluation of this method was given by Quinn and Bradt.[18] Nevertheless the method is mentioned in prCENT/TS 14425-1 as a possible method for evaluating the fracture toughness of ceramic materials where the only exclusions are very tough or porous materials. This is not the case for our materials. The absolute values determined should be used with caution however trends with changing composition and sintering the values do reflect and can be used for comparative purposes with other ceramic materials and composite measured through particular method. Furthermore the investigations of the crack path verify the indentation fracture toughness results.

3. Results and discussion

3.1. Densification

The sintering conditions and achieved densities are given in Table 1. The densification curves of the pure matrices are given in Fig. 1. It is well recognised that the intensive densification of Sialon begins with the formation of the oxide liquid phase at about 1200–1250 °C.[2–4,19,20] Both composites start to densify in this temperature range predominantly through the rearrangement of the Si_3N_4 particles. However, the onset temperature pertaining to the oxide liquid formation is lower for M2045 as well as the intensity of densification being higher than that of M1025, which is expected since it is more oxygen-rich than M1025 Sialon. However, the initial peak of densification rate occurs at approximately 1380 °C for both materials (Fig. 1). After the first maximum of the densification rate (dL/dt; Fig. 1) a steady increase in densification is observed with increasing sintering temperature, as seen from the continuous piston travel with increasing sintering temperature (travel; Fig. 1). However, the densification rate of M2045 reduces in the temperature