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Short communication

Grain size dependence of hardness and fracture toughness in pure near fully-dense boron carbide ceramics

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ABSTRACT

Room temperature fracture toughness and hardness of spark plasma sintered pure B_4C ceramics with grain sizes ranging from 120 nm to 17 μm have been studied. Vickers indentation and single edge V-notched beam (SEVNB) techniques have been used to measure hardness and fracture toughness, respectively. A critical analysis of the results derived from these two techniques has been carried out and the conditions for proper comparison of the derived results are discussed. The results have shown that hardness follows the Hall–Petch dependence with either grain size or twin spacing when the effect of porosity is corrected for. On the contrary, fracture toughness is found to be essentially grain size independent. The value of this quantity is $\sim$2 MPa $m^{1/2}$.

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1. Introduction

The increasing demand for super-hard ceramic materials is a reality nowadays. This fact implies that a precise characterization of their mechanical properties becomes critical. To this purpose, an accurate analysis of hardness and toughness as well as the wear resistance and their grain size dependence is essential for advanced applications. Boron carbide is a keystone in the world of super-hard ceramics because of the combination of outstanding mechanical properties with other properties like low density, high melting point and high elastic modulus. Most scientists and technologists consider that this ceramic will be of especial interest for many strategic applications [1–4].

In spite of many attractive properties of these super-hard ceramics, their major drawback is their brittle nature, characterized by a low value of the fracture toughness. The determination of this quantity can be carried out through several techniques. One popular choice is the so-called indentation method because specimen preparation is simple, requiring only well-polished surfaces and small specimens and finally it is less time-consuming compared to conventional methods. But up to now a universally agreed standard for indentation fracture toughness determination does not exist and the whole technique can be criticised on various grounds [5]. For instance, there are several theoretical models in the literature relating the surface crack lengths measured after indentation to the indenter load and material parameters and it is not clear which is appropriate or correct. The most commonly observed crack systems produced by Vickers indentations are median-radial cracks or Palmqvist cracks. One of these is often assumed to be the relevant geometry, which may or may not be the case for the material in question [6–10].

The correlation between the indentation fracture toughness and its fracture toughness measured by single edge V-notched beam (SEVNB) test in super-hard ceramics has not been studied yet. In the case of pure boron carbide, a relatively wide range of fracture toughness values has been reported and measured by the indentation method [2,3,11–19]. The values of fracture toughness measured by Lee and Speyer [11] were between 2.75–3.15 MPa $m^{1/2}$ while the density was between 90–93%. They measured fracture toughness by the Anstis equation, based upon median-radial cracks. Ghosh et al. [12] used the Evans and Charles fracture toughness equation, also assuming median-radial cracks which was concluded to be the crack geometry in their B_4C specimens. They reported values between 2.69–3.00 MPa $m^{1/2}$ for B_4C specimens with grain sizes of 1.6–2.7 μm and densities of 96–99.2%. Hayun et al. [13] estimated the K_c values with two different equations when Palmqvist cracks were observed after subsequent polishing. The K_c values were between 3.9 and 4.9 MPa $m^{1/2}$ for fully dense boron carbide specimens with grain size of 4 μm. Sairam

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et al. [14] used the Anstis methodology for fracture toughness calculations. They demonstrated that near full density B_4C with grain size 2–6 μm exhibits a fracture toughness of 2.8 MPa m$^{1/2}$ and it increases with decreasing density up to a maximum of 5.8 MPa m$^{1/2}$ corresponding to 91% dense boron carbide. Moshtaghioun et al. [15,16] showed the same tendency in fracture toughness values obtained by the Anstis equation. They reported apparent fracture toughness values as high as 4.81 MPa m$^{1/2}$ for sub-micrometric B_4C with a density of 90.3% while the fracture toughness decreases to values in-between 3–3.65 MPa m$^{1/2}$ for nearly fully-dense sub-micrometric B_4C specimens (with grain size of 300–800 nm) dropping to 1.88–2.10 MPa m$^{1/2}$ for fully dense B_4C specimens with grain size of 25–50 μm. Ji et al. [17] demonstrated that B_4C ceramics with relatively high density (99.7%) and average grain size of 2.36 μm exhibit Vickers hardness of 37.8 GPa and fracture toughness of 4.7 MPa m$^{1/2}$ measured based on radial-median cracks. As for nano-B_4C's, Reddy et al. [18] measured toughness values between 3.6–4.7 MPa m$^{1/2}$ by means of the Laugier equation assuming the Palmqvist crack geometry in samples with a relative density of 92.8% and grain size in the interval 40–150 nm. The value of fracture toughness reported by Moshtaghioun et al. [19] for nano-B_4C specimens with density of 94.6–97.5% and grain size of 100–180 nm obtained by the Anstis equation was in the range of 4.12–4.6 MPa m$^{1/2}$. According to these studies, the presence of porosity in boron carbide ceramics seems to play an important role in determining the hardness and apparent fracture toughness [14–19]. making it difficult to elucidate the grain size dependence of these quantities. It is also well-known that indentation induces amorphization of B_4C [12,15]. The size and shape of the amorphized zone as well as the amorphization intensity beneath a Vickers indentation have been evaluated by Subhash et al. [20] and the damage evolution implies the initiation and formation of radial cracking from the amorphized zone. The amorphization during indentation can affect hardness and apparent fracture toughness as well [20].

In this work, the effects of grain size and porosity (density) on the hardness and fracture toughness were investigated. To this end, a set of boron carbide specimens from micrometric to nanometric grain sizes were prepared and SEVNB fracture toughness tests were performed to measure fracture toughness values in order to evaluate the reported results of this quantity by indentation method while Vickers indentation was used for measuring the hardness.

2. Experimental procedure

Three available boron carbide powders with average particle size around 500 nm, 220 nm and 40 nm (Grade HD20, H. C. Starck, Germany and Tekna Plasma System Inc., Canada) were used as the starting materials. The powder was consolidated under vacuum in a SPS device (Dr. Sinter 515S, Kanagawa, Japan), using a cylindrical graphite die with an inner diameter equal to 15 mm. A pressure of 75 MPa was applied upon heating and released at the end of the holding time. In all cases the heating rate was 100 °C/min. During the sintering process, the temperature was measured by an optical pyrometer, which was focused on a bare hole in the middle part of the graphite die. Table 1 lists the specific SPS variables used and more details on processing and sample preparation are reported elsewhere [15,16,19].

The relative density of the sintered samples was measured by the Archimedes method, using distilled water as the immersion liquid. Microstructural analysis was performed on both fractured and polished surfaces by scanning electron microscopy (HITACHI S5200, University of Seville, Spain). The grain size was determined from the SEM pictures. The grain diameter was defined as that of the equivalent circle from image analysis. The polished surfaces were electro-chemically etched with a solution of 1% KOH.

Hardness (H) was measured by Vickers indentation on polished surfaces using a hardness tester (Struers A/S, DK-2750 Bullerup, Denmark) with indentation loads of respectively 4.91, 9.81 and 19.6 N and loading time 10 s.

Fracture toughness was measured by SEVNB method tested in four-point bending configuration. For fracture toughness determinations by SEVNB fracture toughness tests, as-cut beams were ground to beams with dimensions of 13 mm × 2 mm × 2.5 mm. The ratio between the notch depths and specimen thickness were between 0.2 and 0.3. The notches were sharpened by means of razor blades with a purpose-built sawing mechanism using diamond paste grades down to 1 μm so that the tip radii of the notches were less than ∼10 μm. The fracture toughness was measured in a four-point bending configuration, using a crosshead loading rate of 5 N/min and inner and outer spans of 5.8 and 10 mm, respectively. The tests were performed on a universal testing machine (Dension–Mayes, UK). For estimating the fracture toughness, the following relation was used [21]:

$$K_{C(S)} = \frac{P_f(L_o - L_i)}{BW^{3/2}} \frac{3\alpha^{1/2}}{2(1-\alpha)^{3/2}} f(\alpha) \quad (1)$$

$$f(\alpha) = 1.9887 - 1.326\alpha - \frac{\alpha(1-\alpha)(3.49 - 0.68\alpha + 1.35\alpha^2)}{(1+\alpha)^2} \quad (2)$$

where P_f, L_o, L_i, B and W are the experimentally measured fracture load, outer span, inner span, specimen width, and specimen depth, respectively. The pre-crack size (notch depth), a, enters the equation through α, where $\alpha = a/W$. The reported values are the average of at least five independent measurements.

3. Results and discussion

Table 1 lists the processing conditions, densities and average grain sizes of the different B_4C ceramics studied in this work with the standard deviation, and Fig. 1 shows SEM micrographs of all of them. It can be seen in Fig. 1a that sample 1 which is SPS-ed at 1800 °C has a coarse microstructure with average grain size of 17.2 μm. The presence of microtwins observed by SEM is a well-known feature in boron carbide ceramics [15,16,19]. In contrast, lower SPS temperatures are increasingly effective in retaining fine-grained microstructures (Fig. 1b–d). Sub-micrometric boron carbide specimens with average grain size of 688 nm and 370 nm were obtained at 1700 °C from two different B_4C powders. Moreover for all these three specimens, the relative densities were >98% and there were only a few isolated pores in the microstructures (Fig. 1a–c). In the case of nano-B_4C, a lower sintering temperature of 1600 °C was required to retain the nanostructured character. In addition, two-step SPS was performed to eliminate boria impurities and improve the sinterability [19]. In this condition, nano-B_4C specimens with an average grain size of 120 nm were obtained. Interconnected pores were present in microstructure (Fig. 1d) and the relative density was only 95%.

The experimental results of hardness for each indentation load of 4.91, 9.81 and 19.6 N which are named as HV0.5, HV1 and HV2 are shown in Table 1, as well as the values of fracture toughness determined through SEVNB tests. Fig. 2 displays the experimental hardness versus the applied indentation load. The results show a mild "indentation size effect" in which the apparent hardness increases with decreasing load [22,23]. However, the difference between the highest and lowest values for each material is only ∼10%.

The hardness also varies with grain size as seen in Fig. 3, which shows the results of experimental hardness (black circle points) versus the inverse square root of the grain diameter determined

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