Hardness and Fracture Toughness of Pressureless-Sintered Boron Carbide (B₄C)

Hyukjae Lee and Robert F. Speyer*

School of Materials Science and Engineering, Georgia Institute of Technology, Atlanta, Georgia 30332

The elastic modulus of undoped B₄C (90% of theoretical density (TD)) was measured to be 330 GPa, whereas, for carbon-doped (3 wt%) samples (at 97% of TD), the value was 395 GPa. Figure 1 shows the results of Vickers hardness measurements for undoped samples, as a function of measured grain sizes. Hardness increased as grain size decreased in undoped samples sintered to the same density. Undoped samples exposed to very rapid heating rates had higher hardness values, regardless of their grain sizes. Carbon-doped samples sintered to 97% of TD showed no statistically significant trend, with the limited range of grain sizes, and higher hardness values than those of undoped samples; carbon-doped samples had hardness values of ~2400 kg/mm². Calculated fracture toughness values of undoped samples increased for larger grains (Fig. 2). Carbon-doped samples showed a statistically insignificant variation of fracture toughness with grain size, having values of ~2.9 MPa m¹/², which was higher than the value for undoped samples, with the exception of the largest grain sizes. The hardness and fracture toughness values of undoped samples sintered using various heating schedules, as a function of sintered density, are shown in Figs. 3 and 4, respectively. Hardness increased as sintered density increased, whereas the fracture toughness showed the reversed trend.

Doped samples were sintered using a rate-controlled sintering (RCS) schedule of 0.4%/min to 85% of the total allowed shrinkage and then 0.04%/min to 90% of TD, for comparison with undoped samples sintered in the same fashion. The average grain size was 2.03 μm, which was smaller than the 2.32 μm average grain size of the undoped sample exposed to the same heating schedule. A hardness value of 1780 kg/mm² was measured, which was approximately the same as in the undoped case. A fracture toughness value of 2.8 MPa m¹/² was measured, which was close to, but slightly higher than, the value of the undoped sample.

I. Introduction

A relatively wide range of fracture toughness values has been measured for B₄C by indentation and single-edge notched beam methods (2.9–3.7 MPa m¹/²). Fracture toughness is not greatly changed by heating to 1200°C, even in air. Experimental values of Young’s modulus are scattered in the range of 360–460 GPa for fully sintered compacts. The measured Young’s modulus decreases slightly when temperature or porosity increases. In this work, Vickers microhardness (Hᵥ) and fracture toughness (Kᵥ) were measured using a Vickers indenter (Model AMH-2000, LECO Co., St. Joseph, MI). Each polished sample was indented at five locations with a 29.4 N load for 10 s. The indentation fracture toughness was calculated by

\[ K_{Ic} = 0.018P \left( \frac{E}{cH} \right)^{1/2} = 0.0264a \left( \frac{EP}{c} \right)^{1/2} \]

where \( E \) is Young’s modulus (Pa), \( P \) the load (N), \( 2a \) the indentation diagonal length (mm), and \( 2c \) the crack length (mm). If the ratio of crack length to indentation length \((c/2a) = 0.264\), or if there was crack branching, the data were rejected. Young’s modulus was obtained from a load-versus-displacement measurement, using a 2000 N maximum load and a 0.08 mm/min displacement rate. The compression load was applied using an elastometer (Model Phoenix 1k, MTI, Roswell, GA). The head displacement was measured using a laser extensometer (Model 1532 LTS, Zyro, Middlefield, CT).

II. Results and Discussion

The B/C molar ratio in the samples evaluated was 3.76, which was within the range of a B₄C solid solution. X-ray diffraction results of B₄C compacts heated at 30°C/min to 2250°C show small amounts of graphite, in addition to B₄C.
Considerable interpretation was required to determine the crack lengths and sizes of the indented regions (Fig. 5). Deformation on the boundary region between the indented region and the surrounding area made it difficult to measure indentation diagonal lengths for hardness measurements. Fracture toughness ($K_{IC}$) decreased as the indentation load increased.

Compacts with smaller grain sizes had a greater concentration of grain boundaries. Thus, there was a greater frequency at which moving dislocations encountered grain boundaries. The resulting dislocation pileups at grain boundaries required larger stresses for further deformation, resulting in a greater measured hardness (Fig. 1). The increase in hardness of the undoped samples as the terminal density increased (Fig. 3) resulted from porous regions providing no resistance to the applied load (the average indentation diameter was ~60 μm, whereas the average grain size was <3 μm). The smaller grain size of the higher density samples might have contributed to higher hardness, as well. The carbon-doped samples showed a higher hardness (~2400 kg/mm²) than that of undoped samples (~1800 kg/mm²), because the sintered densities of the carbon-doped samples were higher than those of the undoped samples. No difference in hardness between a carbon-doped sample and an undoped sample sintered to the same density was observed. Although the presence of graphite would be expected to lower hardness, the smaller average grain size of the
doped samples may have been a compensating factor. The fracture toughness of the undoped samples increased for larger grains (Fig. 2). Schwetz et al. observed the same trend from injection-molded B$_4$C samples with a small amount of carbon addition; that is, dislocation pileups more rapidly accumulate at the grain boundaries of smaller grains, which fosters more immediate strain hardening. Thus, the solid displays a greater propensity to crack, rather than plastically deform, under the load of an indenter. The fracture toughness of the present undoped samples decreased as density increased; fracture toughness increased as hardness decreased (Eq. 1), and hardness decreased as porosity increased. In addition, the pores could function as crack arrestors, contributing to increased fracture toughness.

III. Conclusions

After various sintering heat treatments, the hardness of B$_4$C compacts increased and the indentation fracture toughness decreased as grain size decreased; dislocation pinning on more frequent grain boundaries caused strain hardening, which, in turn, fostered crack formation under lower loads.

References

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