Proc. Schl. Eng. Tokai Univ., Ser. E
31 (2006) 45 - 46

Fabrication of Silicon Boride Ceramics

by

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Abstract

The sintering of silicon tetraboride (SiB_4) and hexaboride (SiB_6) ceramics by hot pressing was investigated to determine their suitability for applications as high-temperature materials. The samples were hot-pressed at the desired temperature for 1 hour in vacuum under a mechanical pressure. The relative densities of both monolithic SiB_4 and SiB_6 sintered bodies increased with sintering temperature. In the case of sintering temperatures of 1723 and 1923 K, SiB_4 and SiB_6 sintered bodies having relative densities of more than 99% were obtained, respectively. The Vickers hardnesses at room temperature of the SiB_4 and SiB_6 sintered bodies were 27.5 and 14.5 GPa, respectively. The Vickers hardness of the SiB_6 sample sintered at 1923 K decreased from about 27.5 at room temperature to 12.5 GPa at 1273 K.

Keywords: Boride, Ceramic, Hardness, Sintering

1. Introduction

Silicon chemical compound of oxide, carbide, nitride, and silicide is the useful industrial materials. Several silicon boride phases have been registered in the X-ray Cards of the International Center for Diffraction Data. R.W. Olesinski and G. J. Abbaschian were reported with that SiB_3 and SiB_6 were the stable Si-B composite in the B-Si binary alloy phase diagrams[1]. Then, R. R. Dirkx and K. E. Spear reported the thermodynamic research data of SiB_3 and SiB_6[2]. Afterwards in 1989, that SiB_3 was SiB_4 that is rich in Si clarified on SiB_3 without existing. Among them, SiB_4 and SiB_6 have proved to be a potentially useful material because of its excellent chemical stability. Recently, studies have been carried out to determine the crystal structure of using silicon boride[3-5]. Unfortunately, although monolithic SiB_4 and SiB_6 are known to be chemical stability up to high temperatures. To date, there have been few reports regarding the properties of SiB_4 and SiB_6 sintered bodies. In this study, the sintering of the SiB_4 and SiB_6 ceramics were investigated to determine their suitability high-temperature utility.

2. Experimental procedures

SiB_4 and SiB_6 powder used commercial materials. The SiB_4 and SiB_6 average particle size and purity were 2-3 μm and purity of 98 %, respectively. The powder was packed in a carbon vessel in a resistance electric furnace equipped with carbon heating elements and hot-pressing sintered under vacuum at the desired temperature for 1 hour under a mechanical pressure of 40 MPa. The bulk density of the sintered body was measured by the Archimedes' method. The relative density of the sintered body was obtained by calculating the ratio of the bulk density to the theoretical density of SiB_4 or SiB_6. The Vickers hardness test was carried out under the conditions of a load of 9.8 N and a load time of 20 seconds at room temperature to high temperature in vacuum. Isothermal oxidation in air was carried out in an electric furnace maintained at the desired temperature (1 ± 1 K). A pure α-Al_2O_3 boat containing sintered body was pushed in and pulled out of the hot zone at appropriate intervals and mass gain data obtained to an accuracy of ± 0.1 mg using an electrovalance. The samples were subjected to X-ray diffraction (XRD) analysis for phase evolution using a powder X-ray diffractometer. The surfaces of the sintered specimens were observed using a scanning electron microscope (SEM) to estimate the microstructures.

3. Results and discussion

The relative density of both monolithic SiB_4 and SiB_6 sintered bodies increased with sintering temperature. In the case of sintering temperature of 1723 and 1923 K for 1 hour under a mechanical pressure of 40 MPa, both monolithic SiB_4 and SiB_6 sintered bodies having a relative density of more than 99 % were obtained, respectively. X-ray diffraction analysis showed no crystalline phase other than SiB_4 and SiB_6 in the sintered bodies, respectively. The polish surfaces of the dense sintered samples were observed using a SEM. The SEM photographs of the surface of the SiB_4 samples sintered at 1623 to 1723 K for 1 hour are shown in Fig. 1. The surface of the sintered body showed many pores in the sample sintered at 1648 K for 1hour. In the meantime, the sample of the sintered at 1723 K for 1 hour supposes that it is pore free and made

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into the densification. The Vickers hardnesses at room temperature of the SiB_4 and SiB_6 sintered bodies were 27.5 and 14.5 GPa, respectively. The Vickers hardnesses of the SiB_4 and SiB_6 sintered samples decreased with increasing temperature. The Vickers hardness of the SiB_6 sample sintered at 1923 K decreased from about 27.5 GPa at room temperature to 12.5 GPa at 1273 K (Fig. 2). The sample of the SiB_6 sintered at 1923 K oxidized at 873 to 1273 K for 25 hours exhibited weight gain with increasing oxidation temperature; the oxidation changed of the sample accordance with the parabolic law during the initial oxidation stage. The weight gain of the sample oxidized at 1273 K for 25 hours was approximately 1.5 %. Based on the results of the X-ray diffraction analysis, silicon oxide and boron oxide were present on the surface of the sample oxidized at 873 to 1273 K. From the result of SEM, the surface oxidized at 673 K showed a structure almost identical to the surface of the as-received sample. It was found that a coarsely grained oxide layer was formed on the surface of the sample oxidized at 1073 K. On the other hand, the surface of the sample oxidized at 1273 K, a layer assumed to be a vitreous substance which covered the surface. The sample of the SiB_6 sintered at 1923 K showed a good oxidation resistance at 1273 K, because the surface film of oxide formed by oxidation acted as an oxidation resistant layer.

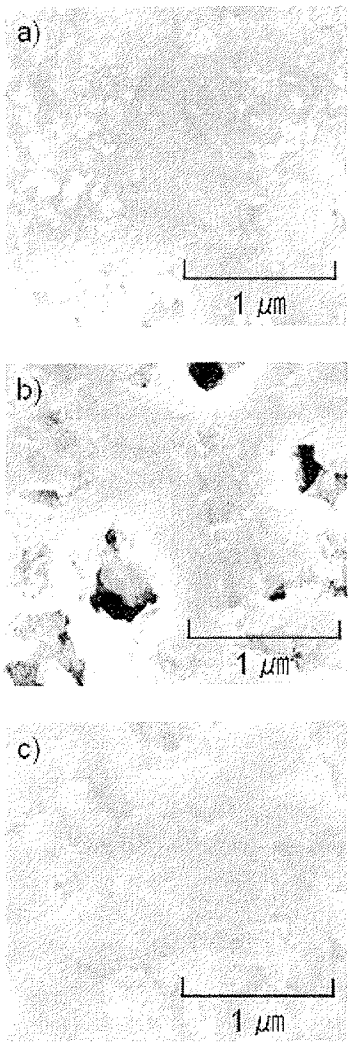

Fig. 1 SEM microphotographs of the surface of the SiB_4 sample sintered at a): 1623 K, b): 1648 K, and c): 1723 K for 1 h.

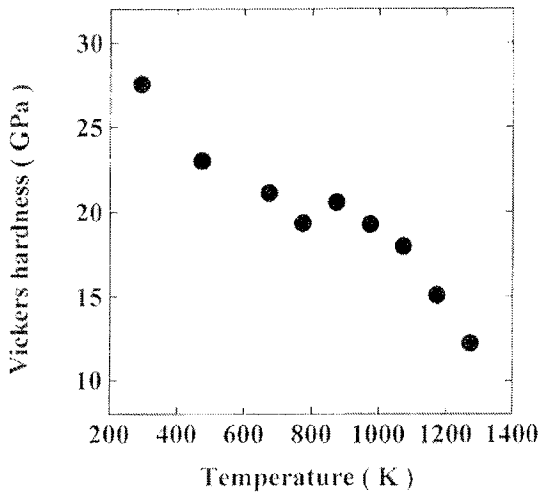

Fig. 2 The temperature dependence of Vickers hardness of the SiB_6 sample sintered at 1923 K for 1 h. (Load: 9.8 N for 30 s)

4. Summary

The sintering of the silicon boride (SiB_4 and SiB_6) ceramics by hot pressing method was investigated to determine their suitability for applications as high-temperature materials. The relative densities of both monolithic SiB_4 and SiB_6 sintered bodies increased with sintering temperature. In the case of sintering temperature of 1723 and 1923 K, a SiB_4 and SiB_6 sintered bodies having a relative density of more than 99% were obtained, respectively. The Vickers hardnesses at room temperature of the SiB_4 and SiB_6 sintered bodies were 27.5 and 14.5 GPa, respectively. The Vickers hardness of the SiB_6 sample sintered at 1923 K changed from about 27.5 GPa at room temperature to 12.5 GPa at 1273 K. The sample of the SiB_6 sintered at 1923 K showed a good oxidation resistance at 1273 K, because the surface film of oxide formed by oxidation acted as an oxidation resistant layer.

Acknowledgements

This work was supported by the Tokai University Research Grant Program. The author would like to gratefully thank Mr. N. Takashima, Mr. S. Tanaka, Ms. A. Kitashima, and K. Shimao of Tokai University for experimental support.

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