High pressure sintering of TiB_2 ceramics at different temperatures

Andrei V. Kapylou[1*], Vladimir S. Urbanovich[1], Vladimir A. Kukareko[2]

[1]Laboratory of Refractory Ceramics and Nanomaterials, The Joint Institute of Solid State and Semiconductor Physics, National Academy of Sciences of Belarus, 19 P. Brovka Street, Minsk BY-220072, Republic of Belarus

[2] The Joint Institute of Mechanical Engineering, National Academy of Sciences of Belarus, 12 Akademichnaya Street, Minsk BY-220072, Republic of Belarus

Received 30 August 2007; received in revised form 25 February 2008; accepted 30 March 2008

Abstract

In this paper the effect of high-pressure sintering (HPS) temperature on the microstructure, physical and mechanical properties of TiB_2 ceramics has been investigated. Initial TiB_2 powder with the average particle size of 5 μm was sintered in a modified high-pressure anvil-type apparatus under static pressure of 4 GPa in the temperature range of 1400–1800°C. It is shown that HPS allows preparing full-dense TiB_2 ceramics with fine-grained structure. The density of samples rises with increasing the sintering temperature up to 1800°C while the maximal microhardness is observed on samples prepared in the temperature range of 1500–1600°C. XRD analysis has shown that this fact is connected with an increase of the level of internal stresses in these samples.

Keywords: titanium diboride, high-pressure sintering

I. Introduction

Because of its properties (high melting point, high hardness, good corrosion resistance and excellent electric conductivity) TiB_2 ceramics is suitable for many applications including cutting tools, wear-resistant parts, armor materials and cathode materials for Hall–Heroult cells [1–3]. Bulk TiB_2 ceramics without additives is usually produced by hot pressing [4,5]. However, up to now, the use of TiB_2 ceramics is rather limited due to the difficulties that exist in preparing a full-dense material. The high-pressure sintering processing (HPS) has been considered as an effective method for producing full-dense TiB_2 ceramics without any additives. The main features of HPS are a low sintering temperature and high sintering rate.

High pressure sintering kinetics of TiB_2 powder with a mean particle diameter of 60 μm was studied earlier [6]. It was shown that the densification process consists of two stages which proceed with different rates. On the first stage, densification rate is fast and the activation energy is low. It was assumed that the features of the first stage are caused by fragmentation and creep. On the second stage, densification is provided by the afterflow and substitutional diffusion in the metallic sublattice.

Structural transformations of titanium diboride under high pressures and temperatures were studied by Olejnik et al. [7]. It was shown that the plastic deformation of TiB_2 under the pressure lower than 7.7 GPa and the temperature lower than 2200°C occurs by creep and intensive fragmentation of grains. The processes of primary and collective recrystallization play an important role during the formation of the grain structure of TiB_2 ceramics under high pressures and temperatures.

The aim of this study was to investigate the effect of the HPS temperature on microstructure and physical and mechanical properties of TiB_2 ceramics. The results about features of the sintering process, microstructure and mechanical properties are reported.

II. Experimental

TiB_2 powder produced by the "Donetsk Factory of Chemical Reagents" with the average particle size of 5 μm was used as a starting material in the present in-

* Corresponding author: tel: +375 17 284 11 64,
fax: +375 17 2840 888, e-mail: *kopylov@ifttp.bas-net.by*

vestigation. Green pellets with a diameter of 11 mm and thickness of 3 mm were formed in a steel die under the pressure of 1 GPa. A modified high pressure anvil-type apparatus [8] was used for high-pressure sintering. Such type of apparatus is simple to manufacture and widely used for both industrial synthesis of super-hard materials and research. Hexagonal BN covering was used to avoid reaction between contact material and TiB_2 pellets. The samples were subjected to a pressure of up to 4 GPa and temperature from 1400 to 1800°C during 2 min. Control of the sintering temperature and pressure was carried out using a computerized system [9].

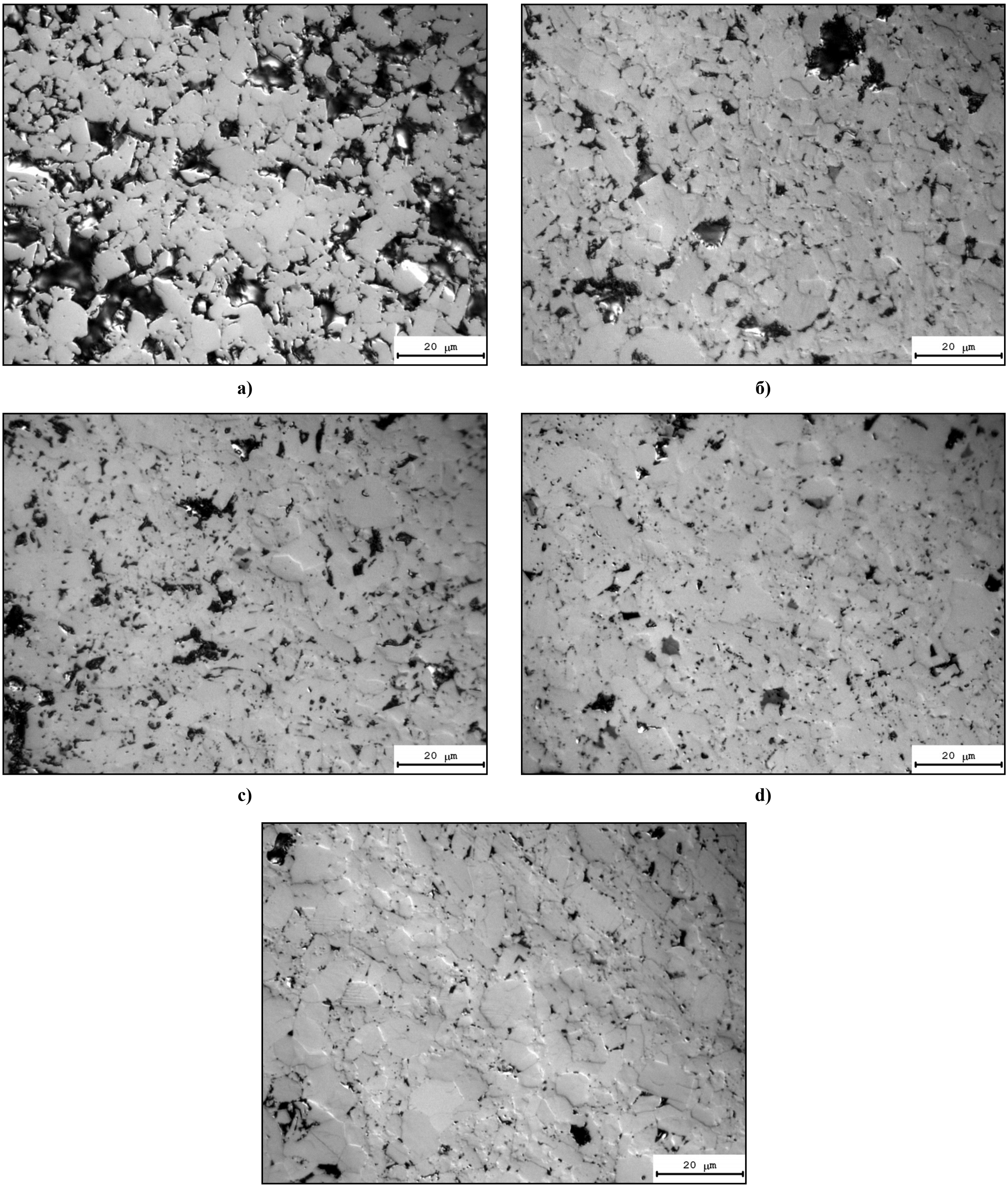

Figure 1. Microstructure of TiB_2 ceramics sintered under the pressure of 4 GPa at different temperatures: a) 1400°C, b) 1500°C, c) 1600°C, d) 1700°C and e) 1800°C

The density of the sintered samples was measured using Archimedes' method in carbon tetrachloride at room temperature. The microhardness measurements were carried out at a load of 1 N and the indentation time of 10 s on the samples polished with diamond powders to a smooth mirror surface. The X-ray diffraction (XRD) analysis was used to confirm the phase composition and determine change of the (211) peak broadening versus sintering temperature. X-Ray diffractometer DRON-2 with CuK_α radiation (step 0.1° and exposition in a point - 20 sec) was used. Microstructural observations were carried out using LEICA DM IRM inverted microscope for materials research. Polishing of the samples was carried out using diamond pastes.

III. Results and Discussion

Fig. 1 displays the evolution of the microstructure of TiB_2 ceramics versus the high-pressure sintering temperature. Microstructure analysis has shown that the HPS process allows preparing TiB_2 ceramics with full-dense fine-grained structure. The mean grain size appeared to be less than 10 µm. Insignificant grain growth is observed with increasing the sintering temperature while the structural inhomogeneity is reduced. The beginning of the formation of the grain structure is observed in the temperature range of 1400–1500°C. At higher temperatures recrystallization processes take place.

Fig. 2 presents the evolution of the relative density and microhardness of TiB_2 ceramics versus the high-pressure sintering temperature. It is shown that the relative density of TiB_2 samples rises with increasing the sintering temperature and reaches the maximal value of 99.3 % at 1700–1800°C. At the same time, the highest microhardness (about 33 GPa) was achieved in the temperature range 1500–1600°C. This fact can be explained using the data obtained by XRD analysis (Fig. 3). The

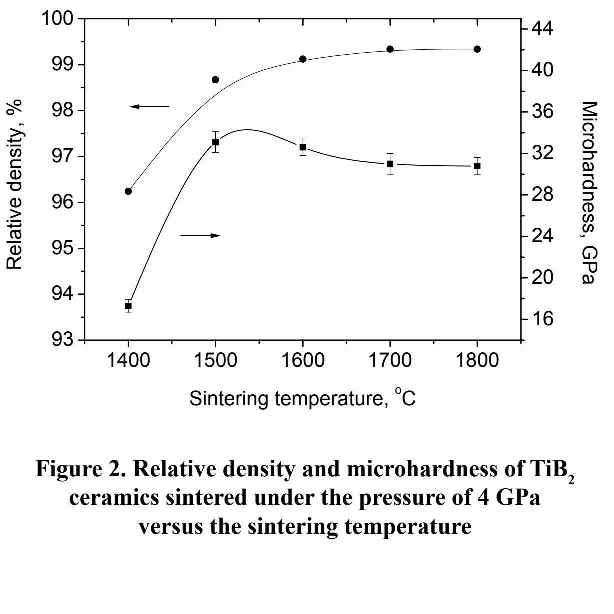

Figure 2. Relative density and microhardness of TiB_2 ceramics sintered under the pressure of 4 GPa versus the sintering temperature

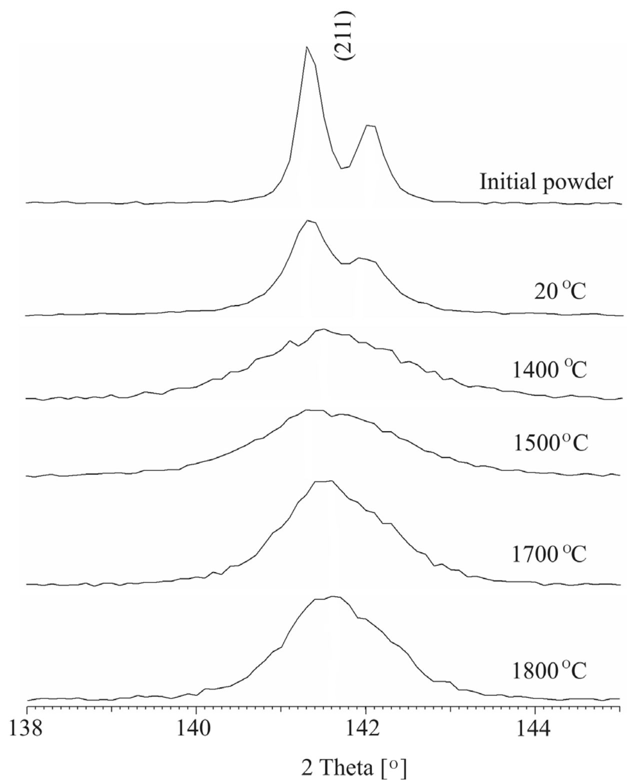

Figure 3. Profile of the (211) peak of TiB_2 sintered under the pressure of 4 GPa versus the sintering temperature

evolution of the (211) peak broadening with raising the sintering temperature demonstrates that the increase in microhardness is accompanied by the lattice deformation, which is caused by an increase of the level of internal stresses in the samples. Then, the reduction in microhardness is related to the beginning of the recrystallization process and relaxation of internal stresses.

VI. Conclusions

The effect of the HPS temperature at the pressure of 4 GPa on microstructure and physical and mechanical properties of TiB_2 ceramics was investigated. It was shown that HPS permits preparing full-dense TiB_2 ceramics with fine-grained structure. Insignificant grain growth is observed with increasing the sintering temperature while the structural inhomogeneity is reduced. The beginning of the formation of grain structure is observed in the temperature range of 1400–1500°C. At higher temperatures recrystallization processes take place. The density of samples rises with increasing the sintering temperature up to 1800°C while the maximal microhardness is observed in samples sintered in the range 1500–1600°C. The XRD analysis has shown that this fact can be ascribed to an increase of the level of internal stresses in these samples.

Acknowledgements: This research was supported by the Belarussian State Programme "Nanomaterials and Nanotechnologies" under grant # 2.02.

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