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SiC NANO-CERAMICS SINTERED UNDER HIGH-PRESSURE

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Abstract. Two SiC nanopowders with the average grain sizes of 16 nm and 45 nm were sintered under high pressure (2-8 GPa) and at the temperature of 1200 °C. The initial powders grain sizes were preserved in the sintered materials. The microhardness HV_{02} of the ceramics increases with sintering pressure and varied from 760 to 1730 for the initial powder of 16 nm, and from 420 to 1710 for 45 nm powder.

1. INTRODUCTION

Nanostructured ceramics, metals and composites offer superior or atypical properties (non-linear optical, electric and magnetic characteristics, inverse Hall-Petch effect) comparing to classic counterparts. One of the key problems in nanotechnology is production of bulk nanomaterials because of thermal instability of nanocrystals which leads to a spontaneous grain growth. We use high-pressure-high-temperature sintering of nanopowders to obtain a variety of bulk nanomaterials [1,2]. In this paper we present results of sintering of SiC nano-ceramics.

2. EXPERIMENTAL DATA

We used two SiC nanopowders composed of loose agglomerated chains of nanoparticles [3]. Table 1 lists: (i) average crystallite sizes, evaluated from X-ray diffraction patterns using the Scherrer formula, (ii) specific surface areas (SSA) and (iii) average particle sizes evaluated using the BET isotherm, of SiC nanopowders used for sintering.

Cylindrically shaped compacts of SiC nanopowders (diameter and height about 5 mm) were prepared under 1000 MPa in vacuum of 4× 10^{-8} MPa. The sintering processes were conduced in solid-state, high-pressure cell of toroid-type [1] with parameters: (i) pressure from 2 GPa to 8 GPa, (ii) temperature of 1200 °C, (iii) time 1 min. Microstructures of the SiC nano-ceramics were examined using scanning and transmission electron microscopes (SEM, TEM), Figs.1, 2. For selected sample average grain size was estimated using a computer system for image analysis [4]. Microhardness HV_{02} of the ceramics was measured with a Vickers indenter using a load of 2 N. The results are presented in Fig. 3.

3. RESULTS

1. We obtained bulk SiC nano-ceramics without a spontaneous grain growth, thus preserving that of the initial powder. The average grain size calculated using computer analysis of TEM pictures of the ceramic obtained from initial powder of 16 nm under pressure 2 GPa is 16±0.7 nm (see Figs. 1 and 2).
2. The properties of SiC ceramics depend on sintering pressure and size of the initial powder grains:

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Table 1. The characteristics of the SiC nanopowders.

Nr	Average crystallite size (X-ray diffraction - Scherrer formula) [nm]	SSA [m^2/g]	Particle size determined from SSA [nm]
1	16	106	18
2	45	30	62

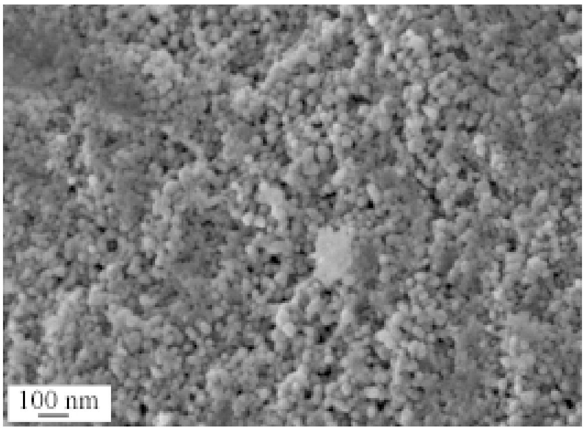

Fig. 1. SEM image of the fracture surface of SiC nano-ceramic with average crystallite size 16±0.7 nm (initial powder 16 nm, parameters of sintering process 2 GPa/1200 °C).

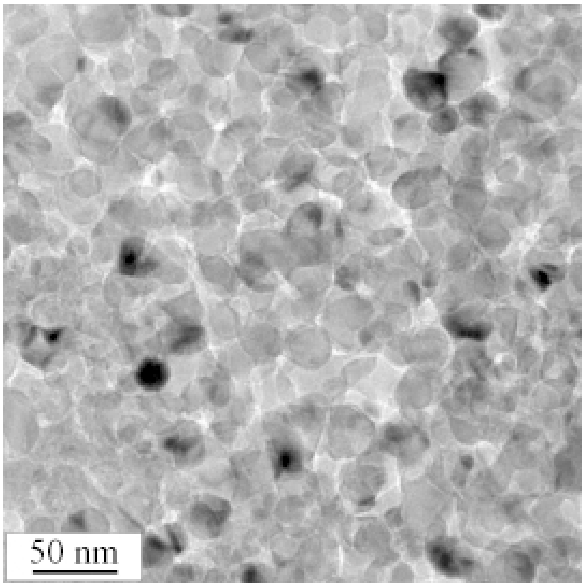

Fig. 2. TEM image of the SiC nano-ceramic with average crystallite size 16±0.7 nm (initial powder 16 nm, parameters of sintering process 2 GPa/1200 °C).

- In the sintering pressure range from 2 to 6 GPa process conditions and granularity of the starting powder influence properties of the nanoceramics. Samples obtained under higher pressure from finer nanopowder present higher mechanical strength. The microhardness HV_{02} of the ceramics increases with sintering pressure and varied from 760 to 1450 for the initial powder of 16 nm, and from 420 to 1310 for 45 nm powder (see Fig. 3).
- Nano-ceramics obtained under 8 GPa, apart from initial powder refinement, present non-porous microstructures in the micrometer-range and the same microhardness 1700 HV_{02}.

3. The mechanical strength of the materials that we obtained does not reach the standard hardness of SiC ceramics 25-30 GPa. This indicates that even under 8 GPa, at the temperature 1200 °C the diffusion processes are not effective

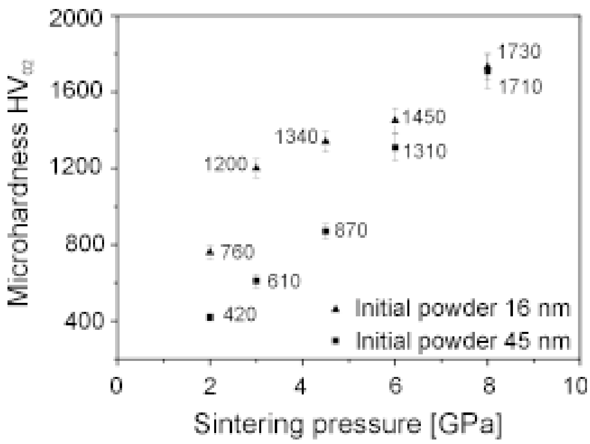

Fig. 3. Microhardness HV_{02} of the SiC nano-ceramics as a function of sintering pressure.

enough to create strong chemical bonding at the nano-particles interfaces during sintering time 1 minute.

Our latest results (in progress) indicate that the ceramics obtained under 8 GPa at 1600 °C present hardness higher than 30 GPa, up to 40 GPa. We suppose that the superior properties of SiC nano-ceramics result from extremely developed area of the grain boundaries. The high hardness appears in a relatively narrow range of pressure and temperature conditions. The work on fabrication of "super-hard" SiC nano-ceramics is currently on the way.

ACKNOWLEDGEMENTS

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REFERENCES

[1] S. Gierlotka, B.F. Pałosz, A Swiderska-Sroda, E. Grzanka, G. Kalisz, K. Fietkiewicz, S. Stelmakh and Ch. Lathe // *Solid State Phenomena* **101-102** (2005) 157.

[2] B.F. Palosz, S. Gierlotka, A. Swiderska-Sroda, K. Fietkiewicz, G. Kalisz, E. Grzanka, S. Stelmakh and W. Palosz // In: *Innovative Superhard Materials and Sustainable Coatings for Advanced Manufacturing*, ed. by J.Lee and N. Novikov (Springer, Netherlands, 2005), p.43.

[3] R. Dez, F. Ténégal, C. Reynaud, M Mayne, X Armand and N Herlin-Boime // *J. Eur. Ceram. Soc.* **22** (2002) 2969.

[4] T. Wejrzanowski, *Computer Assisted Quantitative Description of the Functionally Graded Materials*, *Master's Thesis* (Warsaw University of Technology, Warsaw, Poland, 2000).