ISSN 1392–1320 MATERIALS SCIENCE (MEDŽIAGOTYRA). Vol. 11, No. 4. 2005

Investigations of Structure and Tribological Properties of the Lightweight c-BN-based Composites

Lembit KOMMEL[*], **Eduard KIMMARI**

Department of Materials Engineering, Tallinn University of Technology, Ehitajate tee 5, 19086 Tallinn, Estonia

Received 01 June 2005; accepted 21 July 2005

The present work is designed to provide understanding in the synthesis of ceramic-metal hard materials composites. The materials of interest are nitrides and carbides such as BN, TiC along with Fe, Cr, and Ni binder metals. This report deals with the structure and properties of cubic boron nitride (c-BN) based composites that were produced by self-propagating high-temperature synthesis (SHS). Scanning electron microscopy (SEM) technique was used to characterize the microstructure of material. Tribology properties of the composite have been tested in dry sliding conditions according to standard ASTM-B 611-85 and slurry hydroerosion in a sodium solution.

Keywords: powder technology, SHS, cubic boron nitride, composite, sliding wear.

1. INTRODUCTION

Boron nitride is a binary chemical compound, consisting of equal proportions of boron B and nitrogen N. Cubic boron nitride (c-BN), which is chemically and thermally inert, is second only to diamond in hardness, it has been attracting attention as a superhard material [1].

Cubic boron nitride (c-BN), known as the second hardest material, has good thermal stability and chemical inertness. Cubic boron nitride c-BN has the same structure as diamond. Cubic boron nitride was first synthesized in 1957, but it is only in the last decades that commercial production of c-BN has developed [2].

Cubic boron nitride based composites with addition of titanium nitride TiN, titanium carbide TiC and titanium silicocarbide Ti_3SiC_2 (also called titanium silicon carbide) as binder phase components were studied in [3]. These composites were produced by high pressure (7 GPa) hot pressing (1750 °C). Titanium nitride TiN and titanium carbide TiC react with c-BN, forming new phases in wide range of temperature and pressure [3].

Different technological methods can be used to fabricate boron nitride c-BN composites. Conventional production routes used to fabricate these materials, such as powder metallurgy, are usually energy, time, and capital consuming, hence reducing cost efficiency, due to the significantly high strength and melting temperature of initial materials [4]. Self-propagating high-temperature synthesis (SHS), also called combustion synthesis, which has been used in present study, provides an economical and energy efficient process route for the preparation of composites [4].

It is essential to have a proper understanding of this class of superhard material in order to realize its full potential.

2. EXPERIMENTAL PROCEDURE

2.1. Preparation of composites

The powder of cubic boron nitride c-BN with average grain size of 20 μm was used for composite fabrication. The powder contains also large grains of c-BN that reached 50 μm in cross-section.

The composition of initial powders mixture included cubic boron nitride c-BN – 25 wt.%, and binder phase components titanium Ti – 20 wt.%, iron Fe – 29.5 wt.%, chromium Cr – 11.5 wt.%, nickel Ni – 9 wt.%, and graphite – 5 wt.%. Mixing during 5 hours in a planetary machine was used for powder mixture preparation.

The powder charge was put into the steel container and compacted by vibration. The heating of container with the powder charge in vacuum furnace up to temperature of about 1150 °C initiates the SHS-process. SHS-process proceeds as exothermic reaction, i.e. with heat release, and the process temperature increase up to 1300 °C with low rate of self-propagated heat wave. Immediately after SHS-process the heated container with the material was subjected to densification.

The synthesized plate of composite material was cut into sections for specimen preparation by diamond grinding.

2.2. Characterization techniques

Several analytical techniques were used to characterize the morphological features of the c-BN composites: microstructure was studied using optical light microscope (OLM) Nikon CX, scanning electron microscope (SEM) Gemini LEO Supra-35 was used to observe surface morphology in sub-micro and nanometer range, and X-ray diffraction technique (XRD diffractometer Bruker AXS D5005) was used for characterize the surface chemistry of composites.

Mechanical properties of c-BN composites were studied by data obtained from microindentation tests performed using Zwick Z 2.5/TS1S installation according to the standard DIN 50359.

Microhardness was measured according to ASTM E384-89 standard test method. Buehler Micromet-2001 tester and Vickers diamond indenter were used for microhardness evaluation under different test loads, depending on object brittleness, hardness and dimensions. Up to 10 measurements of each phase have been performed for mean value evaluating.

---

[*] Corresponding author. Tel.: + 372-6203356; fax: +372-6202020.
E-mail address: *kommel@edu.ttu.ee* (L. Kommel)

Sliding friction experiments were conducted in dry conditions, in open air using block-on-ring contact geometry against steel disc. Following test conditions have been used: room temperature, atmospheric pressure, normal load of 40, 180, 220 and 320 N (in static state), sliding velocity of 2.2 m/s, sliding distance 4 km.

The coefficient of friction in the dry sliding condition was obtained as the ratio of the frictional force to the contact force. The frictional force values were continuously determined using a dynamometer.

3. RESULTS AND DISCUSSION

3.1. Structural characteristics

The SEM-image presented in Fig. 1 gives the general view of the microstructure of cubic boron nitride based composite.

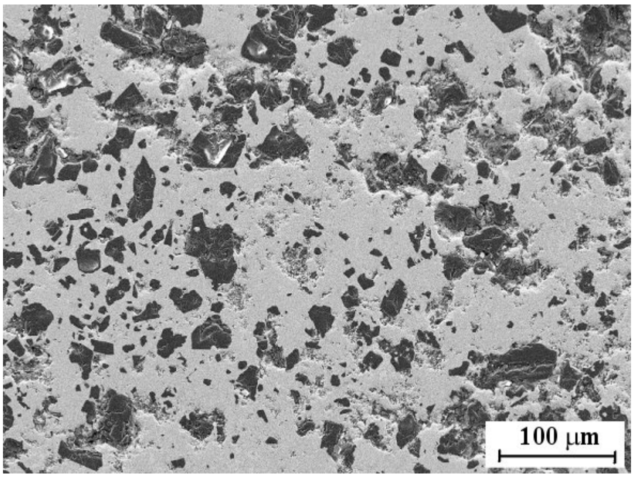

Fig. 1. SEM-image of c-BN based composite

The composite microstructure is characterized by uniformly distributed cubic boron nitride c-BN grains with cross-section in range 5 – 50 μm (dark areas in Fig. 1.), which are bound by metal matrix (light-gray colored areas in Fig. 1).

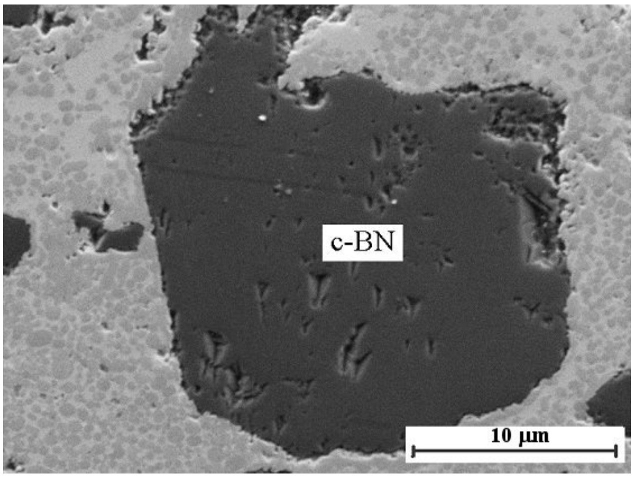

Fig. 2. SEM-image of an area of composite microstructure

The SEM-micrograph in Fig. 2 allows distinguishing the phases of composite microstructure. Three main phases can be explored: grains of cubic boron nitride c-BN, binder phase (light-gray colored areas), and spherical inclusions in binder phase (dark-gray colored areas).

In [3] it was established that the forming of new phases is occurred at the interface between c-BN grain and binder phase in result of composite preparation by hot pressing. Additional SEM-investigations of current study confirmed that synthesized by SHS-technology c-BN based composites have the same feature: a new phase is formed at the c-BN/binding phase interface (see Fig. 3).

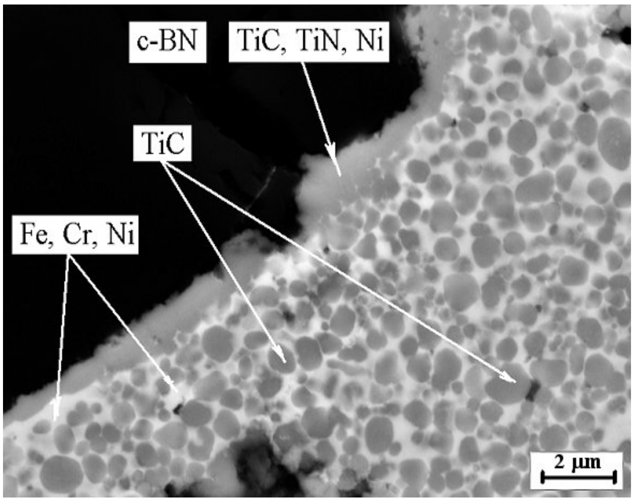

Fig. 3. SEM-image of composite microstructure in the zone nearby c-BN grain

There are numerous spherical inclusions up to 600 nm in diameter in the binder phase. The chemical content of the phase in the zone nearby c-BN grain was established.

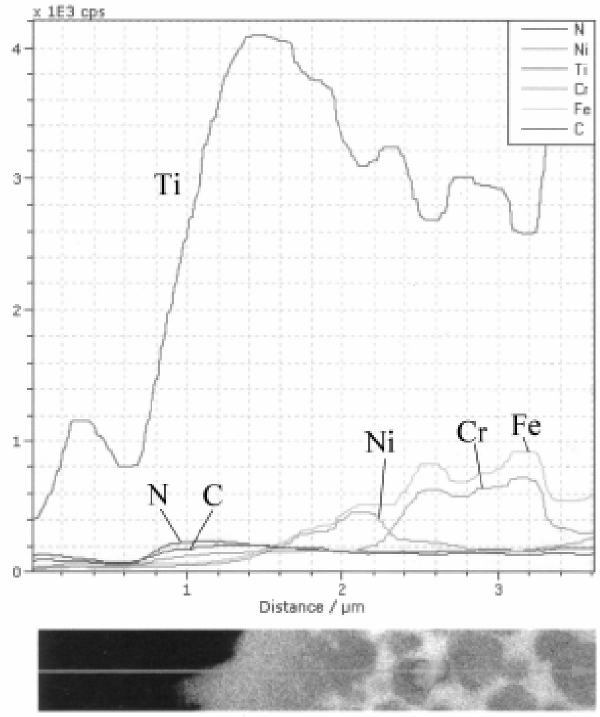

Fig. 4. Chemistry of the near c-BN zone in composite (black colored area – c-BN grain)

The traces from the Fig. 4 indicate that the content of titanium Ti and nickel Ni increase rapidly in the area of c-BN/binder phase interface, then decrease in the area of binder phase. The traces indicated the content of iron Fe and chromium Cr reach the maximum in area of binder phase. The content of nitrogen N and carbon C is about the same through all zones in Fig. 4.

A result of solid state sintering diffusion the nitrogen N combines with titanium Ti to form titanium nitride TiN, that along with nickel Ni and titanium carbide TiC are the constituent of the near c-BN grain phase.

The main components of binder phase are the iron Fe and chromium Cr. The spherical inclusions in binder phase are the sub-micrometer scale particles of TiC. Titanium carbide is one of the most attractive ceramic materials of reinforcement by its high hardness, oxidation resistance thermal stability and low density [2, 5, 6].

The origin of the titanium carbide TiC particles occurred as a result of the SHS-process. It was established that the growth of the TiC particles take place by the coalescence. The process of coalescence is presented in Fig. 5.

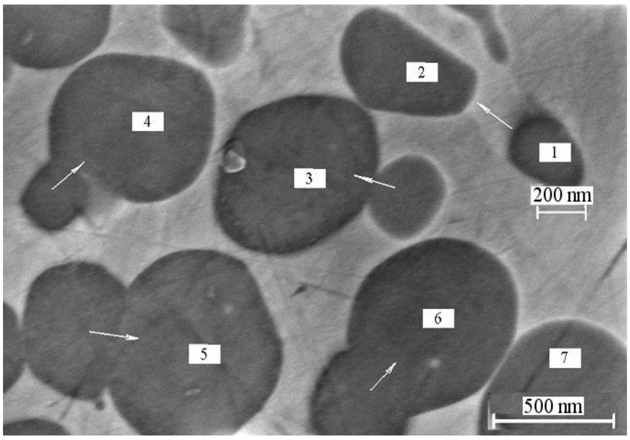

Fig. 5. SEM photograph illustrating fuse coalescence of TiC particles

It is known that solubility is inversely proportional to the grain size [7]. Ultrafine grains have the highest solubility in surrounding matrix. The small grains diffuse to a large one as it is shown in Fig. 5 (grains 1 and 2). If the grains are smaller than the critical size they tend to shrink. Larger grains join each other to form a new grain.

3.2. Mechanical properties

Microhardness of the binder phase was measured 1465 HV0.05. Microhardness of titanium carbide TiC grains was impossible to measure using Buehler Micromet-2001 because of their nanometer-scale size. The hardness of bulk TiC is 3200 HV0.02 as reported in [8]. It is expected that dispersion of hard particles such as TiC in binder improves the hardness, wear resistance and compressive strength [5]. The grains of cubic boron nitride c-BN had the microhardness of about 7000 HV0.05, which is comparable with that of diamond.

The universal hardness *HU* of c-BN composite was measured about HU = 5500 N/mm^2, and the plastic part of universal hardness HU_{plast} = 8500 N/mm^2. The indentation modulus Y_{HU} = 190 kN/mm^2. The indentation modulus Y_{hu} is comparable with the modulus of elasticity (Young’s modulus) *E* of the material [9].

3.3. Tribological properties

The dry sliding experiments were performed at different normal loads applied to the specimen of c-BN composite material.

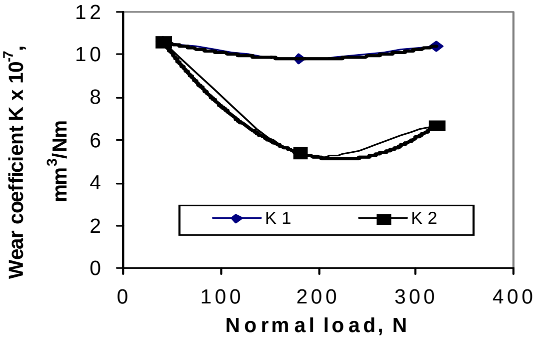

Fig. 6. Wear coefficient *vs.* normal load for c-BN composite

The experimental results obtained are presented in Fig. 6.

The weight loss of the specimens changed from 0.8 mg to 6.1 mg with the normal load increase from 40 N to 320 N in the first series (curve K1), while from 0.8 mg to 3.9 mg in the second (curve K2) (see Fig. 6). The experiments of the second series were performed with the same specimens and on the same part of surface as in the first series. Additionally the weight loss of the material was measured at normal load of 180 N and 220 N. Accordingly obtained experimental data the curves of wear coefficient were drawn. The wear coefficient values in Fig. 6 are in order of 10^{-7} mm^3/(Nm) for a wide range of applied loads, which suggested high resistance to dry sliding wear of the c-BN composites. The friction coefficient values varied in the range of 0.23 – 0.25 at normal load of 150 N and 8 km of sliding distance.

4. CONCLUSIONS

1. Multiphase metal-ceramic composites were prepared by SHS-technology from powders of cubic boron nitride c-BN as initial ceramic phase and titanium Ti, iron Fe, chromium Cr and nickel Ni as metallic phase.
2. Refractory carbides and nitrides are formed in result of SHS in the structure of composite. This microstructural modification, including the formation in binder of spherical sub-micrometer scale sized titanium carbide TiC particles, improved the hardness and wear resistance.
3. Cubic boron nitride based composite is an attractive advanced material for applications where low density, high hardness, and good oxidation and wear resistance are required.

REFERENCES

1. **Watanabe, S., Wheeler, D., Abel, F., Street, K., Mitoshi, K., Murakawa, M., Miyake, M.** Surface Chemistry, Microstructure and Tribological Properties of Cubic Boron Nitride Films *NASA Technical Memorandum 113163* January 1998: 20 p.

2. **Haubner, R.** Superhard Materials *International Journal of Refractory Metals and Hard Materials* 20 2002: pp. 91 – 92.
3. **Benko, E., Klimczyk, P., Morgiel, I., Wlochowicz, A., Barr, T.** Electron Microscopy Investigations of the cBN-Ti Compound Composites *Materials Chemistry and Physics ISSN 0254-0584* 81 Issue 2 2003: pp. 336 – 340.
4. **Slokombe, A., Li, L.** Selective Laser Sintering of TiC-Al_2O_3 Composite with Self-propagating High Temperature Synthesis *Proceedings of the International Conference on Advances in Materials and Processing Technologies, AMPT'99 and 16th Annual Conference of the Irish Manufacturing Committee IMC16* Vol. 2 1999: pp. 1261 – 1269.
5. **Matsuura, K., Hikichi, Y., Kudoh, M.** Combustion Synthesis of Carbide Particle Dispersed Aluminide Intermetallics *Materials Science Forum* 475 – 479 2005: pp. 1605 – 1608.
6. **Pirso, J., Letunovitš, S., Viljus, M.** Friction and Wear Behavior of Cemented Carbides *Wear* 257 2004: pp. 257 – 265.
7. **Bollina, R., German, R.** Heating Rate Effects on Micro-structural Properties of Liquid Phase Sintered Tungsten Heavy Alloys *International Journal of Refractory Metals and Hard Materials* 22 2004: pp. 117 – 127.
8. CRC Materials Science and Engineering Handbook, 2001.
9. Boron Carbide. Ed. **Kislij, P., Kuzenkova, M., Bodnaruk, N., Grabchuk, B.** Kiev, 1988: 216 p. (in Russian).

DOI: 10.5755/j02.ms.26560