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Synthesis and characterization of cBN/WCCo composites obtained by the pulse plasma sintering (PPS) method

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Abstract. The cBN/cemented carbide containing 30vol% of cBN particles was produced using a mixture of a 6wt% Co added-WC powder, with a WC grain size of 0.4 μ m and a cBN powder with a grain size ranging from 4 to 40 μ m. The mixture was sintered to produce a plate, 20 mm in diameter, 3 mm thick. The sintering processes were conducted at temperature of 1100°C under a load of 100 MPa. The phase composition, density, hardness and microstructure of the sintered parts thus obtained were examined. The fractures through the WCCo/cBN composite showed the cBN particles torn out from the cemented carbide matrix were only few, whereas most of them have cleaved along the fracture plane. This gives evidence that the bond at the WCCo/cBN interface is mechanically strong.

Introduction

Cubic boron nitride is a material next to diamond in hardness [1], but its thermal stability and chemical neutrality are superior to those of diamond. Because of these advantages cubic boron nitride is widely used for the manufacture of cutting tools.

Another material widely used for cutting tools is tungsten carbide which is the next hardest material to cBN. Tungsten carbide is commonly sintered with cobalt as the binding phase. Thanks to its high hardness, good resistance to frictional wear and high resistance to cracking, over 50% of cutting tools are made of cemented carbide. Its hardness can be significantly increased by replacing part of the carbide phase with cubic boron nitride which is several times harder. In addition, the cBN particles can considerably increase the fracture toughness of the material due to the crack deflection effect wherein the crack energy is absorbed [2].

The cBN/WCCo composite cannot however be produced by conventional sintering, which is conducted at a temperature between 1350 and 1500°C in the presence of the liquid phase [3], since within this temperature range, cBN undergoes transformation into hBN, and the presence of cobaltrich liquid phase enhances the transformation rate.

Composites with cBN particles dispersed within cemented carbides, known as CDCC (Cubic Boron Dispersed Carbides), were obtained by Martinez and Eceberria [2] by sintering using the method of hot isostatic pressing (HIP) at a temperature between 1100°C and 1200°C under pressure up to 200 MPa. The CDCC composites thus obtained were dense and no phase transformation of cBN into

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hBN took place during the sintering process. Shi et al [4] sintered the CDCC composites using a nanocrystalline (WC-10wt%Co) powder and a CBN powder with titanium-covered grains 150 μ m to 375 μ m in size. The sintering process was conducted by the Spark Plasma Sintering (SPS) method at a temperature of 1240°C. Yaman et al [5] obtained a CDCC composite using a WC-6wt%Co powder and a cBN powder with a grain size of 0.8 and 5 μ m, and sintering the powders by the SPS method at a temperature of 1300°C and pressure of 75MPa.

In the present experiments the CDCC composites were sintered by the Pulse Plasma Sintering (PPS) method which uses periodic high-current electric pulses generated by discharging a capacitor battery. The energy of several kJ, stored in the capacitors, is delivered during several hundred of microseconds, which creates specific sintering conditions. The PPS method has been used for sintering a wide variety of materials, such as WCCo/diamond [6] and Cu/diamond [7] composites, nanocrystalline sinters [8] and, in combination with the SHS reaction, for fabricating high-melting ceramics [9-11]. The aim of this work was to obtain sintered parts with density near theoretical value and with very good contact of cBN particles with the WCCo matrix.

Experimental procedure

The CDCC composites were produced using a mixture of a (WC+0.5wt%VC) powder, a Co powder, and a cBN powder of various grades (1/3 and 37/44). The (WC+0.5wt%VC) powder added with 6wt% of cobalt was mixed with cemented carbide balls (ball-to-powder mass ratio -1:1) in a turbular mixer at a rotational speed of 70 rev/min for 10h. Then the mixture was mixed with 30vol% of cBN powder particles using the same mixing parameters. The powder mixtures were sintered to obtain samples 20 mm in diameter and 3 mm high. The composites were sintered at temperature of 1100°C. The sintering process was conducted in a PPS apparatus in vacuum of $5x10^{-5}$ mbar, using a graphite die. The compositions of the mixtures of the powders are given in Table 1.

Label	Composition	dt (g/cc)
W6	(WC + 0.5wt%VC + 6wt%Co)	14.85
W6(1/3)	30vol% cBN(1/3) + (WC+ 0.5 wt%VC + 6wt%Co)	11.44
W6(37/44)	30vol% cBN(37/44) + (WC+ 0.5wt%VC + 6wt%Co)	11.44

 Table 1. Composition and designations of the powder blends.

The phase composition of the sintered materials was determined with a PHILIPS PW 1140 X-ray diffractometer equipped with a PW1050 goniometer using CoK_{α} radiation. The microstructure and the chemical composition were examined in a HITACHI S3500N scanning electron microscope. The hardness was determined using a ZWICK hardness-meter under a load of 1 kG. The density of the composites was measured by immersing the samples in water and using the Archimedes principle; the measured density values were compared with the theoretical density calculated from the rule of mixtures.

Results and discussion

The W6(1/3) composites sintered at 1100°C have the hardness of 2070 HK1 and that of the W6(37/44) composite was 1990 HK1. The density of the W6(1/3) and W6(37/44) composites is below the theoretical density (DT) and is equal to 92.6%DT in the W6(1/3) composite and 98.6%DT in the W6(37/44) composite, whereas without the cBN addition, the density of cemented carbide+6wt%Co sintered at 1100°C by PPS is 14.85g/cm³ (99.5%DT). This indicates that the addition of the cBN particles decreases the sinterability compared to that of cemented carbide itself.

The diffraction examinations of W6(1/3) and W6(37/44) composites did not show the presence of the hBN phase (Figure 1). It should however be realized that the diffraction peaks from the hBN phase may be very weak because of the very high massive absorption coefficient of the WC phase.

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Figure 1. XRD diagrams: (a) W6(1/3) composite, (b) W6(37/44) composite.

Observations of the microstructure of the composites sintered at a temperature of 1100° C neither did reveal the presence of the hBN phase. Figures 2 and 3 show SEM photographs of the surface of a fracture of the W6(1/3) and W6(37/44) composites. As can be seen, the cBN particles are uniformly distributed in the cemented carbide matrix. No pores and hBN precipitates are visible around the cBN particles which are firmly bound with the cemented carbide matrix. Only few cBN particles thorn out from the matrix can be seen on the fracture surface, while, during the fracture, most of them break in the transcrystalline manner and remain embedded in the matrix.



Figure 2. SEM of the surface of a fracture of the W6(1/3) composite.



Figure 3. SEM of the surface of a fracture of the W6(37/44) composite.

Conclusions

Composites containing the cBN phase dispersed in the cemented carbide matrix were consolidated by the PPS method under the conditions of the thermodynamic non-equilibrium of cBN. The composites sintered at a temperature of 1100°C under a pressure of 100 MPa for 5 min have a density near the theoretical value. SEM observations of the microstructure and diffraction phase examinations did not show the presence of the hBN phase. The specific conditions of heating the material to be consolidated using high-current pulses hamper the transformation of cBN into hBN and ensure a strong bond between the cBN particles and the cemented carbide matrix. No hBN precipitates can be seen around the boron nitride particles and and no pores are present around them. On the surface of the composite fracture, the SEM photograph reveals cBN particles and transcrystalline fractures of the cBN particles. The presence of transcrystalline fractures of the cBN particles indicates that the bonding forces between the cBN particles and the WCCo matrix exceed the matrix cohesion.

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