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Al_2O_3 –cBN composites sintered by SPS and HPHT methods

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a r t i c l e i n f o

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A B S T R A C T

Spark Plasma Sintering (SPS) and High Pressure–High Temperature (HPHT) methods were chosen in order to study the behaviour of metastable cubic boron nitride (cBN) during sintering of $Al_2O_3 + 30$ vol.% cBN composites at low and high pressure. Relatively low sintering temperatures were used to avoid the undesired reverse transformation of cBN to hexagonal, graphite-like form. The microstructure, and phase composition as well as physical and mechanical properties of the resulting Al_2O_3 –cBN composites were investigated. In the ball-on-disc tests, the coefficient of friction and the specific wear rate of the sintered samples were determined by rotating the sample against a stationary alumina ball. The best composites obtained by SPS (at 1300 ◦C/75 MPa) had slightly lower density, Young's modulus and hardness than those obtained by HPHT but their wear resistance was much better. Formation of hBN was suppressed up to 1300 ◦C during SPS consolidation at the pressure of 75 MPa.

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1. Introduction

High hardness, fracture toughness, wear resistance and low coefficient of friction are the basic materials characteristics most desired for advanced ceramic composites, especially for cutting tools applications. Furthermore, the materials to be used for the cutting inserts should be resistant to oxidation and chemically inert to the workpiece material even at high temperatures. At the moment, there is no universal material that could be used as a cutting blade for all machining applications. The metal processing industry is still dominated by sintered carbide tools, but for special purposes, especially for high cutting speeds and high feed rates, oxide-based ceramic materials are particularly important. Their basic components are usually Al_2O_3 or ZrO_2 . Advanced ceramics containing Al_2O_3 exhibit most of the required properties such as high thermal and wear resistance, good chemical stability and moderate to high mechanical strength. The use of ceramic cutting tools in certain cases allows for dry machining (without harmful cooling liquids) and efficient finishing operations (eliminating of grinding operations) which gives measurable economical gain as a result of reduction in machining costs. Alumina and its composites are some

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of the best candidates for universal cutting tool material, however, their fracture toughness is low because dislocation movement is limited by their ionic and/or covalent bonds. The brittleness and poor damage tolerance have limited their application as advanced engineering materials particularly for cutting applications so far [\[1\].](#page--1-0)

A wide range of various ceramic matrix composites (CMC's), reinforced by addition of silicon carbide (SiC), titanium diboride (TiB₂) and other hard particles to Al_2O_3 matrix, were investigated to improve mechanical properties of alumina based materials. The composites were fabricated mainly with the pressure assisted methods (e.g. hot-pressing). In most cases the significant enhancement in hardness, fracture toughness or/and the wear properties in comparison with the monolithic Al_2O_3 was achieved [1-3].

Another group of tool materials are superhard composites based on polycrystalline diamond (PCD) or polycrystalline cubic boron nitride (PcBN). The hardness of cubic boron nitride is greater than that of any other known material except diamond. In addition it is less reactive with ferrous materials than diamond, which makes it the most widely used tool material in material removal applications for ferrous workpieces [\[4\].](#page--1-0) Polycrystalline cBN (PcBN) composites are divided into two broad classes of materials characterized by the low (∼40–50 vol.%) and high (> 80 vol.%) cBN content. Increase in the amount of the hard phase in the composite results in higher hardness, mechanical strength and fracture toughness, however, it

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also decreases the thermal and chemical resistance of the material. In composites with high cubic boron nitride content, the bonding phase only activates the sintering process and fills the gaps between cBN grains and thereby increases the fracture toughness but does not significantly influence other properties of the composite, which are determined by the cBN phase. The control of thermal and chemical properties of the composite relies on the content of the bonding phase, which needs to be relatively high. Therefore, composites consisting of relatively low content of cBN grains are frequently used for high speed machining, where the temperature at the cutting edge can reach 1000 °C and high thermal and chemical resistance of the tool is more important than its mechanical strength. PCD and PcBN are among the most expensive tool materials because diamond and cubic boron nitride phases are metastable and for their processing the High Pressure–High Temperature (HPHT) conditions are required [\[5–7\].](#page--1-0)

In this work the studies of alumina based CMC's containing 30 vol.% of cBN, sintered atlow pressures (35 and 75 MPa—provided by SPS) and ultra-high pressure (7.5 GPa—provided by HPHT) are presented. A fine alumina powder grade was chosen as the matrix material for preventing the undesired reverse phase transformation from ultra hard cBN to graphite like hBN, which could occur at high temperatures and at relatively low pressures typical of the SPS method [\[8,9\].](#page--1-0) The use of only 30 vol.% of cBN aims to increase thermal and chemical resistance of the composite. Moreover, the relatively small content of dispersed phase decreases the probability of direct contacts between their particles. Cubic boron nitride sintering temperature is higher than 2000 ◦C. In the case of low temperature SPS sintering process weak bonding between the cBN grains may yield to low strength.

2. Experimental

Fine alumina powder (Al $_2$ O $_3$ - α , TM-DAR grade, 0.1 μ m, TAIMEI, Japan) characterised by low sintering temperature (∼1300 ◦C), and cubic boron nitride cBN powder (Micron + ABN grade, $3-6 \mu m$, Element Six, Ireland) were used as starting materials. The mixture containing Al_2O_3 + 30 vol.% cBN was prepared using Fritsch Pulverisette 6 planetary ball mill equipped with zirconia grinding vessel and balls. The powders were homogenised with rotation speed of 200 rpm for 2 h in acetone. The composites were sintered with two different methods.

2.1. High Pressure–High Temperature sintering (HPHT)

Sintering process was carried out using the Bridgman-type toroidal apparatus. In this system quasi-isostatic compression of the preliminary consolidated powders is achieved as a result of plastic deformation of the mineral gasket material (usually metamorphic stones) between anvils [\[10,11\].](#page--1-0) Electrical heating of the sample material is provided by a high-power transformer and graphite heater.

Homogeneous mixtures were pressed into pellets with the diameter of 15 mm and the height of 5 mm under the pressure of 200 MPa. The pellet was placed into the graphite heater in the central part of the HPHT cell assembly. The consolidation was carried out at the pressure of 7.5 GPa the temperature ranging from 770 to 1880 \degree C. Duration of the HPHT sintering process was 60 s.

2.2. Spark Plasma Sintering (SPS)

The material was sintered employing an SPS equipment (HPD5 type, FCT system, Germany). The powder was placed in a graphite die (inner diameter of 20 mm), then uniaxially pressed at 35 or 75 MPa and heated up to sintering temperature with a heating rate of 100° C/min. A graphite sheet with 0.5 mm in thickness was inserted between the raw material powder and the graphite die to prevent punch scuffing and to lighten the sintered sample extraction from the matrix. The graphite die was also wrapped with carbon blankets in order to minimize the heat loss during the sintering process. The samples were sintered in the temperature range from 1100 to 1500 \degree C for 4 min in argon.

2.3. Investigation methods

XRD patterns were obtained using the PANalytical Empyrean diffractometer with the copper radiation (λ_{Cu} =1.5406A). The quantitative phase analysis of the studied material was carried out using Rietveld refinement and HighScore PANalytical software. Microstructure of the materials was studied with a scanning electron microscope (JEOL JSM-6460LV). Density of the sintered samples was measured by the Archimede's method while their theoretical density was calculated applying the rule of mixtures and assuming densities of 3.98 g/cm³ for Al₂O₃ and 3.48 g/cm³ for cBN. Young's modulus of the composites was determined by ultrasonic wave transition method measuring the velocity of ultrasonic

Fig. 1. Density and Young's modulus of Al₂O₃ + 30 vol.% cBN composite sintered by HPHT and SPS methods under the ultra-high and moderate pressure in the wide range of temperatures. Optimal sintering temperature for HPHT (1) and SPS (2) are indicated.

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