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Mechanical behavior of diamond matrix composites with ceramic $Ti_3(Si, Ge)C_2$ bonding phase



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ABSTRACT

Polycrystalline diamond, PCD, compacts are usually produced by high pressure–high temperature (HP–HT) sintering. This technique always introduces strong internal stresses into the compacts, which may result in self-fragmentation or graphitization of diamond. This may be prevented by a bonding phase and $Ti_3(Si,Ge)C_2$ was so investigated. This layered ceramic was produced by Self Propagating High Temperature Synthesis and the product milled. The $Ti_3(Si,Ge)C_2$ milled powder was mechanically mixed, in the range 10 to 30 wt.%, with 3–6 µm diamond powder (MDA, De Beers) and compacted into disks 15 mm in diameter and 5 mm high. These were sintered at a pressure of 8.0 GPa and temperature of 2235 K in a Bridgman-type high pressure apparatus. The amount of the bonding phase affected the mechanical properties: Vickers hardness from 20.0 to 60.0 GPa and Young's modulus from 200 to 500 GPa, with their highest values recorded for 10 wt.% $Ti_3(Si,Ge)C_2$. For this composite fracture toughness was 7.0 MPa m^{1/2}, tensile strength 402 MPa and friction coefficient 0.08. Scanning and transmission electron microscopy, X-ray and electron diffraction phase analysis were used to examine the composites.

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1. Introduction

The global market for cutting tools is divided into two main groups: high speed steels and hard metals, but ceramics have now reached 8.5% [1] and for diamond and tools with diamond-like coatings it is expected to grow from an estimated \$905 million in 2010 to \$1.7 billion in 2015 with a compound annual growth rate of 14.1 over the next 5 years [2]. In particular, the move towards dry and high speed machining will suit PCD and PCBN tooling, due to their long tool lives and relative insensitivity to the application of coolants or the effects of greater cutting temperatures, that dramatically affect the cutting performance of other types of tooling materials. PCD tools have replaced hard metals, ceramics and natural diamond in a range of high-performance applications including turning, boring, milling, slotting and chamfering of e.g. non-ferrous materials, high-silicon aluminum, metal matrix composites, ceramics, reinforced epoxies, plastics, carbon fiber-reinforced plastics and engineered wood products, because of their high hardness/ strength, low friction coefficient and chemical stability.

Selecting the optimum grade of PCD tooling for a specific application is generally a function of surface finish requirements and tool life expectations [3,4]. It is perceived that diamond tipped tools tend to react with ferrous materials at high temperatures and hence are not suited for

* Corresponding author. *E-mail address:* szutkows@ios.krakow.pl (M. Szutkowska). machining steels. In order to avoid graphitization of the diamond matrix, it is necessary to keep the cutting temperature below the critical level of diamond graphitization. But, using cryogenic CO₂ cooling, is possible to cut high strength cast iron [5]. In spite of the highest hardness and wear-resistance among commonly available materials, low fracture toughness and impact strength of diamonds limit their wide application in industry. Further, polycrystalline diamond compacts are usually manufactured by high pressure-high temperature (HP-HT) sintering, which introduces high internal stresses into the compacts, which may result in their self-fragmentation or graphitization of the diamond. By combining binder phases with diamond powder during the sintering process, these critically important properties have been improved, as also electrical conduction. The latter property is especially important because it allows EDM machining of these composites. They can withstand much higher operating temperatures without markedly reducing their strength and wear resistance [6,7].

Cobalt-containing PCDs, however, are chemically stable only up to 700 °C. A mismatch of thermal expansion coefficients between diamond and the cobalt binding phases ($4.8 \cdot 10^{-6} \text{ K}^{-1}$ for diamond and $12 \cdot 10^{-6} \text{ K}^{-1}$ for cobalt) induces internal residual stresses which may generate microcracks in the composite [8,9]. Additionally, cobalt is increasingly expensive and also detrimental to health [10]. Thus diamond tool composites with a ceramic bonding phase, having a lower thermal expansion mismatch, have been proposed [11–13]. Experience with silicon and titanium carbide binder phases shows that an

important feature of the binding material is its ductility and thus further research is timely.

It is anticipated that the MAX-phase (layered ternary carbides and nitrides) will fulfill current expectations, because they combine some of the best properties of ceramics and metals. MAX phases are nanolayered ceramics with the general formula $M_{n + 1}AX_n$ (n = 1–3), where M is an early transition metal, A is an A-group element (mostly group IIIA or IV A), and X is either carbon and/or nitrogen [14,15]. In 1996, there was a remarkable breakthrough in this area by Barsoum and El-Raghy [16], who reported a successful synthesis of a fully dense, single phase Ti₃SiC₂. There are now more than 50 M₂AX, or 211 compounds, six M₃AX₂ or 312 compounds: Ti₃SiC₂, Ti₃GeC₂, Ti₃AlC₂, Ti₃SnC₂, Ta₃AlC₂, and V₃AlC₂ and seven M₄AX₃ or 413 compounds: Ti₄AlN₃, Ti₄GaC₃, V₄AlC₃, Ti₄SiC₃, Ti₄GeC₃, Ta₄AlC₃, and Nb₄AlC₃ [17]. The bonding is mostly metallic, with covalent and ionic contributions, very strong M-X bond, together with M-A bonds that are relatively weak, especially in shear. Like metals, they are good electrical and thermal conductors, readily machinable, tolerant to damage, and resistant to thermal shock. The MAX phases are relatively soft (2–8 GPa hardness). Similar to ceramics, they are refractory (their decomposition temperature is higher than 2000 °C), oxidation resistant, guite stiff and relatively light (~4.5 g/cm³). The MAX phases are in general quite stiff and elastically isotropic. Some of them are also resistant to thermal shock, oxidation, fatigue, and creep [18]. Because of the attractive set of properties of layered ternary carbides (MAX-phases), the Ti-Si-Ge-C binder system for diamond was chosen for our investigations.

2. Experimental procedure

2.1. Material production

Commercially available synthetic diamond powder (Element Six MDA 36, 3–6 µm grade) and titanium (AEE, USA), germanium (AEE, USA), graphite (Merck, Germany) and silicon were the starting materials. Silicon powder, of 99.9% purity, was obtained by an autogenic milling of semiconductor waste produced by the Polish Nitrogen Factory. To produce the MAX phase Ti₃(Si,Ge)C₂, the elements, in stoichiometric proportions, were first mixed with isopropyl alcohol, and silicon carbide balls, to produce a suspension which was homogenized for 24 h and then dried, in a way which prevented a second segregation. This powder was processed by the relatively low-cost Self Propagating High Temperature Synthesis method at the Faculty of Materials Science and Ceramics of the AGH University of Science and Technology. About 500 g of the reaction mixture was used for each SHS process carried out in a vacuum. Synthesis reaction of the powder mixture was initiated in a reactor chamber with high alumina refractory brick and graphite foil lining. The exothermic reaction started when the temperature in the reactor rose to about 1100 °C, the reaction front propagated across the chamber, resulting in full transformation of the material. The product was crushed in an Abbich mortar to produce powder with 0.5 mm grain size and next was milled for 20 h in a rotary-vibratory mill with WC grinding media in anhydrous isopropyl alcohol to reduce the particle size. The alcohol was then evaporated. The grain size distribution and average size of particles were measured using the Shimadzu (type SA-CP3) apparatus. Densities of powder and compacts were measured using AccPyc 1340 helium pycnometer, manufactured by Micrometrics Inc. The powder, of 4.96 g/cm³ density, was 5.2 µm in size. It was mixed with the diamond powder and pressed at 90 MPa into cylinders 15 mm diameter and about 5 mm in thickness to attain 10 20 and 30 wt.% of Ti₃(Si,Ge)C₂. To obtain crack-free samples with the highest values of density and mechanical properties, the optimum sintering temperature was established experimentally. The compacts were sintered at the high temperature of 2230 \pm 50 K at the high pressure of 8 \pm 0.5 GPa in a Bridgman-type toroidal apparatus for 30 s (Fig. 1).



Fig. 1. HP–HT sintering system: a) scheme of the Bridgman's apparatus: 1,4 – pyrophyllite gasket; 2,6 – graphite heater; 3 – sintered sample; 5 – molybdenum plate, b) gasket with sample before sintering.

2.2. Characterization techniques

After sintering, specimens were lapped on a cast iron disk, ground with DIA-Pro type diamond abrasive (size of diamond grains about $9 \,\mu$ m) and polished with a OP-S type diamond abrasive (size of diamond grains about 0.04 μ m) (Fig. 2).

Apparent density, ρ , of the sintered composite samples was determined by saturation in vacuum according to PN-EN 1389:2005 [19].

XRD measurements were taken using a PANAlytical Empyrean system with Cu K α radiation. Phase compositions of the SHS powder and sintered compacts were identified using the database of International Center for Diffraction Data. The quantitative compositions of the SHS product and the diamond-Ti₃(Si,Ge)C₂ composite were determined by the Rietveld method with X'Pert Plus program. X-ray diffraction of



Fig. 2. DTi₃(Si,Ge)C₂ specimen after lapping and polishing.

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