



Diamond–Ceramic Bonding Phase Composites for Application in Cutting Tools

LUCYNA JAWORSKA^{1,2}

¹ Institute of Advanced Manufacturing Technology, Materials Engineering Department, Kraków, Poland

² Pedagogical University, Institute of Technology, Kraków, Poland

e-mail: lucyna.jaworska@ios.krakow.pl

Abstract

Commercially available polycrystalline diamond compacts (PCD), consisting of a polycrystalline diamond-cobalt layer on a WC-Co substrate are used in drilling and machining applications. The presence of a cobalt phase in the diamond layer has the effect of significantly reducing thermal resistance. The thermal stability of a PCD material can be defined as its resistance to graphitization in an inert atmosphere at elevated temperatures. One of the possibilities to increase the thermal resistance of PCD materials is to reduce the cobalt content. The second one is the manufacturing of diamond compacts with a non-cobalt bonding phase. Presented in this paper are new trends for this type of material and my own experimental results of high-temperature sintering of diamond with carbides and/or silicides. The WC-Co layer allows for brazing of the PCD material to the tool body. Direct brazing of diamond materials to metal bodies is difficult and requires special solders and special conditions. Due to differences in thermal and mechanical properties between diamond and WC-Co, residual stresses develop in the interface region during the cooling from sintering, brazing or cutting temperatures. In an effort to reduce residual stresses, functionally graded material (FGM) diamond compacts with a ceramic bonding phase were designed and obtained. The results of X-ray phase composition analysis, electron microscopy microstructure observations, and selected physical and mechanical properties together with thermal resistance studies are presented.

Keywords: Polycrystalline diamond compacts, Binding phase, Tool materials

KOMPOZYTY DIAMENTOWE WIĄZANE FAZĄ CERAMICZNĄ DO ZASTOSOWANIA NA NARZĘDZIA SKRAWAJĄCE

Komercyjnie dostępne polikrystaliczne kompakt diamentowe (*polycrystalline diamond compacts*, PCD), składające się z polikrystalicznej warstwy diamentowo-kobaltowej na podłożu WC-Co wykorzystywane są do wiercenia i obróbki maszynowej. Obecność fazy kobaltowej w warstwie diamentowej ma wpływ na znaczne zmniejszenie odporności termicznej. Stabilność termiczną materiału PCD można zdefiniować jako jej odporność na grafityzację w atmosferze obojętnej w podwyższonych temperaturach. Jedną z możliwości zwiększenia odporności termicznej materiałów PCD jest zmniejszenie zawartości kobaltu. Inną jest wytwarzanie diamentowych kompaktów z niekobaltową fazą wiążącą. W niniejszym artykule przedstawione zostały nowe trendy w przypadku tego typu materiału i własne wyniki doświadczeń dotyczące wysokotemperaturowego spiekania diamentu z węglkami i/lub krzemkami. Warstwa WC-Co pozwala na lutowanie materiału PCD do narzędzia. Bezpośrednie lutowanie materiałów diamentowych do narzędzi metalowych jest trudne i wymaga specjalnych lutów i warunków lutowania. Z powodu różnic właściwości cieplnych i mechanicznych diamentu i WC-Co, podczas chłodzenia z temperatur spiekania, lutowania i cięcia, w obszarze międzyfazowym powstają naprężenia resztkowe. Starając się zredukować te naprężenia, opracowano i wytworzono diamentowe kompakt z ceramiczną fazą wiążącą i stopniowaną funkcjonalnością (FGM). Zaprezentowano wyniki rentgenowskiej analizy składu fazowego, mikroskopowych obserwacji mikrostrukturalnych (SEM), a także badań odporności termicznej oraz wybranych właściwości fizycznych i mechanicznych.

Słowa kluczowe: kompakt polikrystalicznego diamentu, faza wiążąca, materiały narzędziowe

1. Introduction

Sintered polycrystalline diamond (PCD) compacts are widely used as cutting tools, wire drawing dies and drill bit rocks. Polycrystalline diamond (PCD) cutting tools are currently used in two distinct markets; woodworking and metalworking. Abrasive workpiece materials, together with the use of higher cutting speeds and feed rates, result in a much shorter tool life of PCD cutting tool materials [1].

The application of polycrystalline diamond cutting tools increases in metalworking, because of the higher consumption of aluminium in each new vehicle, which generates sav-

ings on fuel and a reduction in CO₂ emissions. In the timber industry, the development of new construction materials, such as HPL flooring and fibre cement board cladding, has forced the development of new cutting tool materials to cut and shape these [2]. During the high pressure - high temperature synthesis process (at about 5-6 GPa, 1670-1770 K) fine, submicrometer and micrometer diamond powders are mainly obtained from carbon precursors. For cutting tool applications, large diamond crystals or polycrystals are used. These are obtained by the sintering of diamond powders.

Hall has reported solid state sintering of diamond powder without additives under high pressure [3]. For cutting tool

edge applications, diamond compacts have been used since the early 1970 s. Ultra-high pressures and temperatures are used for the sintering of diamond powders without additives or with a minor amount of additives. Because of the high energy cost for this process, these materials were not popular until the end of the 20th century. Materials without bonding phases are very hard but brittle. For this type of solid-state sintering, there is a problem of the graphitization process due to the stresses. Diamond is thermodynamically metastable phase at atmospheric conditions. During the high-pressure sintering process, part of the particle is in a compressive force state where the particle is in contact with the other, and part of the particle is in a tensile force state on the diamond crystal surface surrounded by voids. For the same particle, there are thermodynamic conditions for the appearance of a diamond phase and a graphite phase. For ultra-high pressures over 9 GPa, diamond particles are crushed and voids are filled, there is a better stress distribution in the sintered material, and the graphitization process is limited. Another method of obtaining binder-less polycrystalline diamond consists in a direct synthesis without catalysts from graphitic carbon at 10 GPa and 2270 K. Very fine microstructures (particle size 10-20 nm) without secondary phases, such as graphite or hexagonal diamond, are observed. The material is characterized by extremely high hardness [4, 5]. There are several kinds of natural binder-less polycrystalline diamonds: "carbonado", "ballas" and "bort", but these materials are inhomogeneous and contain impurities. New nano-polycrystalline diamonds have excellent thermal stability and have no hardness anisotropy.

Katzman and Libby have reported liquid phase sintering of the diamond-cobalt system [6]. Hibbs and Wentorf have developed a method of cobalt infiltration into diamond layers under high pressure conditions [7]. They obtained two layers of compact; the first layer was diamond with cobalt and the second was WC-Co. Diamond powders were sintered and bonded to a WC-Co substrate at the same time by infiltration of Co from the substrate during the sintering process. The carbide substrate is very useful for tool producers because of the possibility of brazing to the tool body. Sufficiently high wettability of diamond materials by molten metal fillers is the principal requirement for successful brazing. The majority of solders do not provide sufficient diamond wettability. The diamond brazing process requires special solders and special apparatus for brazing. Carbide brazing does not require special conditions and is cheaper.

The microstructure and mechanical properties of polycrystalline diamond (PCD), synthesized at high pressure and high temperature, are strongly influenced by a type of applied binding material phase. The most popular commercial PCDs are two-layer materials with a cobalt phase, similar to Hibbs and Wentorf's diamond/WC-Co. Cobalt provides good wetting of diamond crystallites. This property allows the production of compacts characterized by a low amount (below 10 wt%) of the bonding phase, resulting in their high hardness. But when the compact is cooled from sintering temperature to room temperature, very high radial compressive stresses are induced in the diamond layer, while much lower radial tensile stresses are induced in the cemented tungsten carbide substrate [8]. However, the high brittleness of these binding materials results in poor toughness of such

PCDs, undermining their practical applications. In addition, cobalt-containing PCDs are chemically stable only up to 900°C, while working temperatures may rise even higher. Today, these materials are the most popular PCDs. The main difference in currently-used materials, as compared to Hibbs and Wentorf's material, is in the "multi-modal" grade mixture of grains of different sizes, which enables the gaps between larger particles to be filled by smaller particles [9].

2. Reactions in the traditional diamond-cobalt system

Cobalt belongs to the iron group of materials, which are known to be good solvents/catalysts for diamond synthesis and also for the binder phase during the diamond powder sintering process. Metal binders, such as Co, Ni and Fe, are able to dissolve carbon. The sintering technology is based on the infiltration of cobalt into diamond grain surfaces, the solution of carbon in the cobalt and reverse crystallization of the carbon into diamond. These materials are characterized by a special microstructure composed of fine initial diamond grains surrounded by the metal and new very small diamond crystals.

The utilization of binder materials for the sintering of diamond compacts has three-fold benefits: a reduction in sintering temperatures and pressures, cleaning the diamond surface of graphite, and electron discharge cutability for tool making [9]. Cutability of tool materials is a very important property, especially for diamond, because diamond is not electrically conductive. For the diamond with the cobalt bonding phase, the electro-discharge method of cutting is widely used. Traditional mechanical methods of cutting are inefficient.

One of the possibilities to increase the thermal resistance of PCD materials is to reduce the cobalt bonding phase content. Diamond compacts usually contain over 85 vol.% diamond crystals. The second one is the preparation of materials with a non-metallic bonding phase without negative effects on diamond graphitization and with compatibility of the thermal expansion coefficient to the diamond coefficient [1]. A mismatch of thermal expansion coefficients between diamond and the catalyst (binding) metals ($4.8 \cdot 10^{-6} \text{ K}^{-1}$ for diamond and $12 \cdot 10^{-6} \text{ K}^{-1}$ for cobalt) may induce internal residual stresses and may generate microcracks in the polycrystalline diamond [8]. Therefore, a new material characterized by at least as good wetting of diamond but producing synthesis reaction products both less brittle and chemically stable at even higher temperatures than those containing cobalt would be of interest.

3. Diamond sintering with carbides forming metallic and non-metallic bonding phases

Instead of dissolving carbon in metals, other metals can be used, for example carbide-generating elements such, as Ti, Zr, Hf, W and Mo. Due to the high melting temperatures of these metals and their compounds for the diamond sintering process, higher temperatures are needed than for a diamond with the cobalt bonding phase (up to 2670 K and pressures higher than 7.8 GPa).

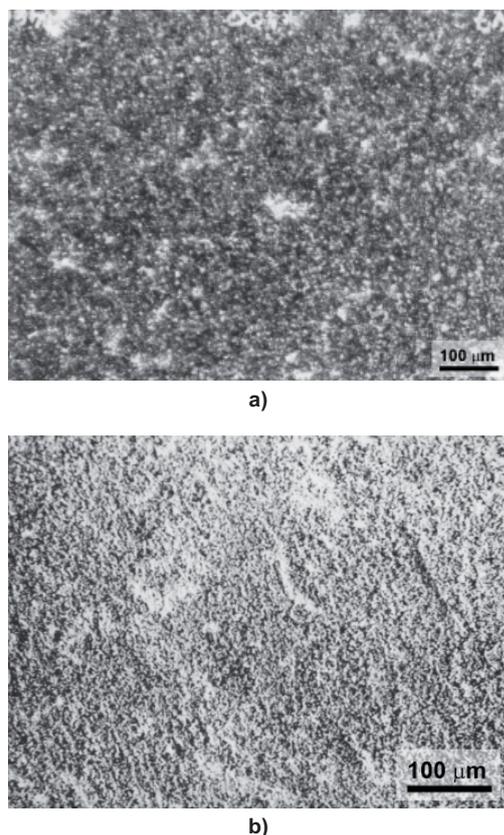


Fig. 1. Microstructure of diamond compacts with 5 wt% Ti: a) compact of diamond and titanium mixture, b) compact of diamond with deposited titanium.

The result of sintering is a diamond-metal carbide compact and, possibly, some remaining metal. For the majority of these elements, the sintering process is carried out below melting temperature, in solid state, without an infiltration process. The final product contains a diamond skeleton with pores filled with carbide and broken particles of diamond (Figs. 1a and 1b).

Due to the metal plastic behaviour, voids between diamond grains were filled by bonding phases, so for these systems there are secured thermodynamic conditions for the presence of diamond. The method of metal addition has a strong influence on the properties of the compacts (Table 1). For example, metal added (below 5 wt%) to the diamond powder by means of PVD (Fig. 1b), chemical and other deposition processes results in a homogeneous distribution of metal in the sintered body and good mechanical properties. These compacts contain a titanium carbide phase in the grain boundaries after sintering.

Table 1. Selected properties of diamond compacts with 5 wt% titanium.

Form of titanium additive	density ρ^* [g/cm ³]	Compressive strength R_r^{**} [MPa]	Hardness HV^{***} [GPa]
Ti powder	3.506	115.97	36.82 ± 1.80
PVD coating	3.511	90.85	42.34 ± 1.12
TiH ₂ powder	3.526	141.37	58.9 ± 2.1

* Density measured using the hydrostatic method.

** Average of five measurements, $\alpha = 0.05$.

*** Compressive radian strength.

But because of the high number of M-C_d-M connections in the material, it is not characterized by good wear or cutting properties: For cutting materials, better results are obtained for C_d-C_d (diamond-diamond) connections. The addition of metal powder - or metal hydride powder - to diamond powder, mechanically mixed, results in not so homogeneous metal distribution, but materials are characterized by better wear and cutting properties, because of the direct diamond connections [10]. For Ti addition below 5 wt%, there is no higher thermal resistance observed. The cutting process requires the use of a cooling liquid, such as for the materials with the cobalt bonding phase.

At the end of 20th century, new commercial diamond materials with a silicon binder material were produced. During the sintering process, silicon forms a SiC carbide. Diamond-silicon carbide composites find a wide use in industrial applications. The most popular method of obtaining such a composite is infiltration of Si into diamond and the consequent reaction of Si with the carbon in the diamond. The SiC formation is associated with an increase in volume, which could result in blocking of pore channels, which would affect the material porosity and roughness of the tool surface [11].

This material is used in drill applications because, whilst it does not preserve good surface quality of machining materials during turning or milling, it is characterized by a higher temperature resistance in comparison to diamond compacts with cobalt bonding phases.

4. Composites with carbide bonding phases

For carbide bonding phases, in spite of the different thermal expansion coefficients of diamond and carbides, large thermal stresses may accumulate inside the compacts because carbides are not ductile materials.

In composites with SiC binding material, a tendency to graphitization is observed. In the sintering process, there is no infiltration or liquid presence, as for Si binding material (Figs. 2a and 2b). During sintering, in the case of SiC bonding phase, there is no fragmentation of SiC crystallites, due to its high strength properties at high temperature [12].

Y.S. Ko *et al.* have confirmed in their works the presence of graphite for diamond-SiC material (50-90 wt% diamond) [13]. Graphite content decreased with the addition of 1 wt% Al.

The participation of graphite increases with an increase in the proportion of SiC binding material, Fig. 3. Diamond crystals are surrounded by large SiC grains. At the grain boundaries, graphite strips occur as a result of the graphitization process.

Due to the presence of graphite in the composition of compacts (Fig. 3) the abrasive wear resistance of composites with a SiC bonding phase is predicted to be lower than the composites with TiC and Ti₃SiC₂ bonding phases.

The situation for the TiC bonding phase is different from that for SiC [14]. The TiC bonding phase fills well the areas between diamond crystals. The initial TiC particle size was of 3 μm; after the sintering process, it was in the range 0.042–0.45 μm. The composites with TiC have very good mechanical properties, but lower wear resistance. There is a fragmentation of TiC crystallites during the sintering process. Small particles below 0.5 μm are removed from the

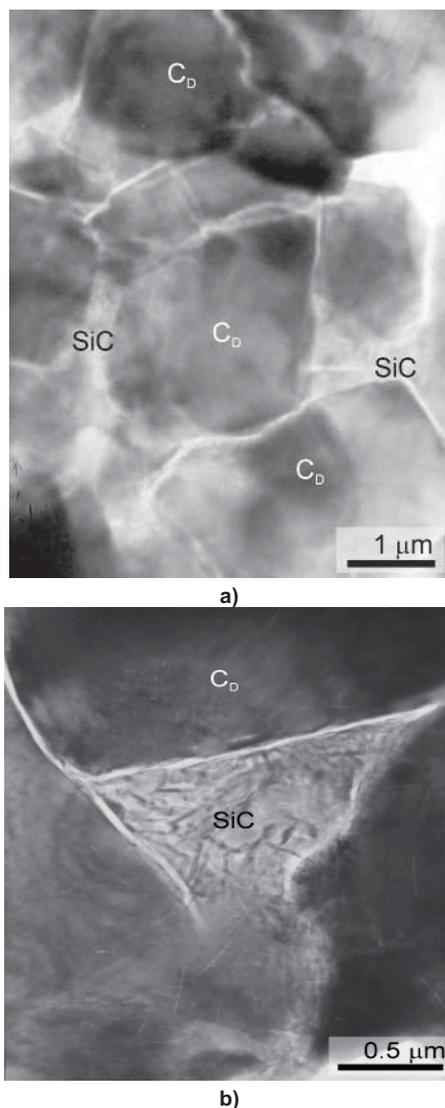


Fig. 2. (a) Electron microstructure of diamond (El.Six, MDA 36) - SiC (Goodfellow, 5µm) composite; (b) Microstructure of the bonding phase in diamond = SiC composite; C_D - diamond.

cutting edges made of this material, during the machining process. This has a negative effect on the roughness of the workpiece.

As carbides can change their stoichiometry, an interaction between diamond and carbide is possible. For the thermal resistance studies, the diamond composites with 30 wt% bonding phase were annealed for 30 min at 1470 K in a 0.8 Pa vacuum using a furnace at a heating rate of 10 K/min. The results of these studies are presented in Table 2. Diamond compacts with 30 wt% SiC are characterized by high levels of graphitization.

Table 2. Changes in density and hardness of diamond composites, before and after heat treatment.

Type of bonding phase	Density before heat treatment [g/cm ³]	Density after heat treatment, 1473 K [g/cm ³]	Calculated graphite content* [wt%]	Hardness HV before heat treatment** [GPa]	Hardness HV after heat treatment** [GPa]
TiC 30 wt%	3.55	3.52	2.38	45.5 ± 2.4	34.0
SiC 30 wt%	3.09	3.06	3.57	32.5 ± 4.3	20.1
Ti ₃ SiC ₂ 30 wt%	3.61	3.59	0.94	34.1 ± 4.32	28.0

* Difference in the percentage share of graphite before and after heat treatment at 1473 K.

** Average value of five hardness measurements.

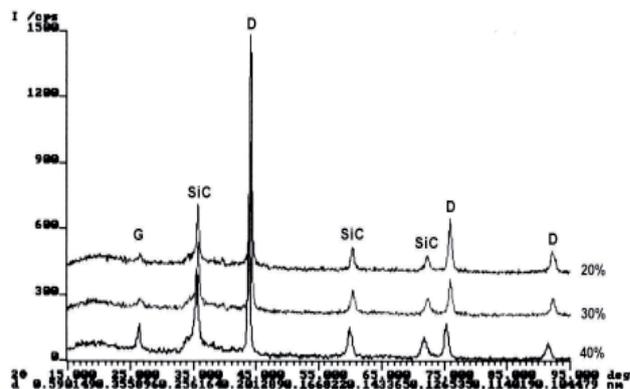


Fig. 3. X-ray pattern of diamond composites with 20 wt%, 30 wt%, 40 wt% SiC powders, $I = f(2\theta)$, in the range 15-95° (D - diamond, G - graphite).

The best compacts for practical application seem to be those with the largest addition of Ti_3SiC_2 , as these show the highest hardness at a negligible loss of wear properties as compared with other diamond compacts with carbide content [15].

Composites were prepared from diamond powders of 3-6 µm (MDA, Element Six) and 2-4 µm Ti_3SiC_2 (SHS technique) average crystallite size. These were sintered using a Bridgman-type high-pressure apparatus at 8.0 ± 0.2 GPa and at temperature of 2070 ± 50 K. Samples were heated in an internal graphite heater with an inside diameter of 15 mm. Samples for Vickers hardness measurement were prepared through lapping on a cast iron plate with the diamond paste.

A microscope analysis indicated that the bonding material has a multiphase composition including Ti_xSi_y intermetallic, TiC and SiC carbides. It was additionally determined that at the diamond interface, a layer of SiC dominates with only a minor fraction of TiC, while the centers of channels separating the diamond crystallites are filled mostly with Ti_xSi_y intermetallic. For solid state sintering, better results are observed for a higher bonding phase content. There is no infiltration. The microstructure of this composite is shown in Fig. 4. In Table 3, the comparisons of selected properties for diamond- Ti_3SiC_2 with various amounts of binding material are presented. Selected properties for 30 wt% diamond- Ti_3SiC_2 material are presented in Table 4.

5. Compacts with oxide and nitride compounds

A calculation of the phase equilibrium for Al_2O_3 -diamond and Si_3N_4 systems was carried out. The Gibbs free energy minimization technique was used for all elements. A program

Table 3. Influence of Ti_3SiC_2 weight share on Young modulus and density of diamond compacts.

Ti_3SiC_2 content [wt%]	Density, [g/cm ³]	Young modulus, [MPa]
10	3.63	373
20	3.60	356
30	3.61	550

Table 4. Selected physical and mechanical properties of PCD with 30 wt% Ti_3SiC_2 bonding phase.

Density* [g/cm ³]	Hardness** [HV]	Young Modulus [GPa]	$R_{0.05}$ *** [MPa]	K_{Ic} **** [MPa·m ^{1/2}]
3.608	44.42 ± 3.1	567.0	253.08	8.0

* Average measurement for three samples.

** Average of five hardness measurements, $\alpha = 0.05$.

*** Compressive radian strength, average value of three measurements.

**** Fracture toughness, notched beam method.

was written for calculations using the chemical equation for multi-compound and multi-element systems, on the basis of modification of the VCS algorithm (Villars, Cruise, Smith). These calculations were performed at temperature of 2070 K at a pressure of 8 GPa. According to the calculations of chemical equilibrium in Si_3N_4 -diamond and Al_2O_3 -diamond systems, these compounds cannot react with diamond [16]. Similar calculations show the diamond/cBN (110) junction to be thermodynamically unstable [17]. Experiments confirmed that diamond compacts with TiN, Si_3N_4 , $Ti(C,N)_{70:30}$ and Al_2O_3 do not sinter at 2070 K and 8 GPa. However, the compacts with SiAlON were obtained. For 30 wt% SiAlON multiphase composition of material was confirmed by the X-ray diffraction method (diamond, Al_2O_3 , $Si_3Al_3O_3N_4$) [16].

6. Diamond compacts with intermetallics and silicides

Analyses of processes for various bonding phase materials indicate that intermetallic compounds from Mo–Si, Nb–Si, Ti–Si systems ensure lower sintering conditions. The most promising seem to be $MoSi_2$, $TiSi_2$ and $NbSi_2$ [18]. These materials are characterized by the multiphase composition of non stoichiometric silicides and carbides. The microstructure (growth and shape of crystallites) of the bonding phase confirms the presence of liquid during the process. The presence of SiC as well as MoC or NbC carbides in the PCD compacts prepared with silicides obtained from SHS reaction products indicates that the conditions of PCD synthesis enabled a reaction between binder material and diamond. The hardness and Young's modulus of the PCD synthesized with silicides were found to be comparable with the properties of commercial cobalt-containing PCD.

For other intermetallic materials such as $Nb_{19}Ti_{19}Cr_{64}$, diamond compacts with good wear and temperature resistance were also obtained. The process for these systems is reactive sintering. It is important that the bonding phase contain a carbide- or solid solution- forming element (Ni, Co, Fe, V, W, Ti, Mo, Cr, Ta, Zr, Si) and that the bonding phase

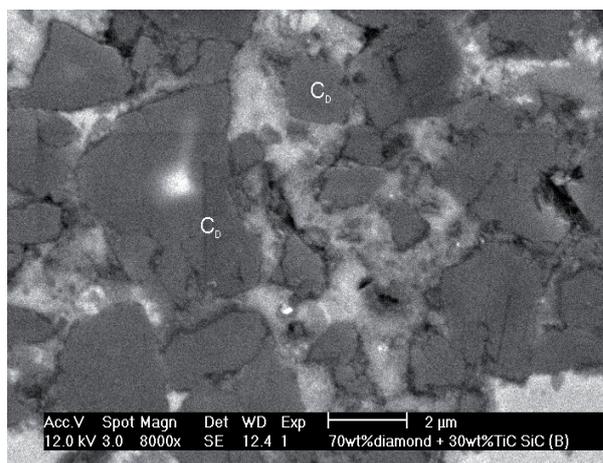


Fig. 4. SEM microstructure of diamond with Ti_3SiC_2 binding material, C_D – diamond grains.

has a low melting temperature, below the temperature of the sintering process. This allows a reduction in the conditions of the sintering process.

The intermetallic powders intended as a bonding phase may be prepared using the relatively low-cost Self-Propagating High-Temperature Synthesis (SHS) technique. SHS is especially useful for silicide production due to its low temperature of the ignition process.

The high porosity of such synthesized materials means they are easily milled, allowing the fine powders to be obtained to be mixed with diamond crystallites. A certain drawback of the SHS technique is that the combustion products, with the exception of the main phase, usually also contain other minor phases [18]. Diamond 30 wt% $MoSi_2$ compacts are obtained in the same conditions as diamond 30 wt% Ti_3SiC_2 compacts (see Chapter 3) [19]. Microstructure observations were performed using the Philips CM 20 TWIN (200 kV) transmission electron microscope with the Phoenix EDAX EDS attachment. Thin foils were prepared by mechanical polishing and ion milling with a Gatan DuoMill 600. The microstructure and electron diffraction of this composite is presented in Figs. 5 and 6.

X-Ray diffraction (using a Philips PW 1710 Co lamp and graphite monochromator) of diamond compacts with 30 wt% $MoSi_2$ confirmed that, besides the diamond phase, the composite contains mainly $MoSi_2$ and $Mo_{24}Si_{15}C_3$. $Mo_{24}Si_{15}C_3$ compound was described in Pearson's Handbook of Crystallographic Data for Intermetallic Phases, 1991. The material

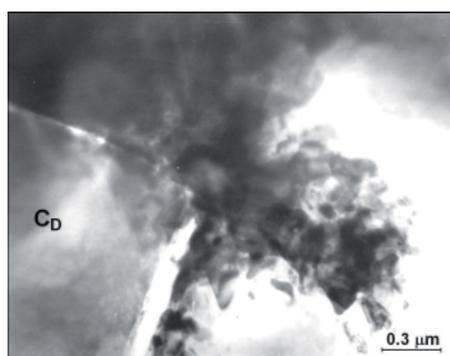


Fig. 5. Microstructure of diamond compact with 30 wt% $MoSi_2$, TEM, C_D – diamond grains.

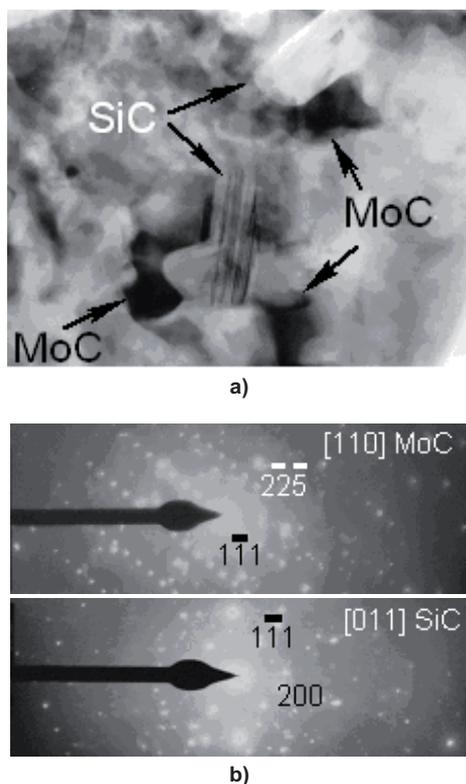


Fig. 6. (a) Microstructure of the bonding phase, near the diamond, for diamond with 30 wt% MoSi_2 , and (b) electron diffraction from dark (MoC) and light crystalline (SiC), TEM.

has a hexagonal structure ($a = 0.7286 \text{ nm}$, $c = 0.5046 \text{ nm}$). After thermal treatment at 1470 K for 30 min in vacuum, MoSi_2 content decreased whilst $\text{Mo}_{24}\text{Si}_{15}\text{C}_3$ content increased.

Due to the reaction sintering process, the phase composition and microstructure for diamond 30 wt% Mo_5Si_3 is similar, because of the bonding phase reaction with carbide in the diamond [20].

7. Functionally graded diamond

A homogenous mixture was prepared of 70 wt% diamond powder (3–6 μm , MDA Element 6) and 30 wt% Ti_3SiC_2 powder (below 2 μm , SHS method, AGH) [21].

The mixture of powders was dispersed by the ultrasonic method in a solution of water with organic additives. This suspension, with 0.3 wt% of solid content, was introduced into special dismountable centrifugal tubes. The deposition process was carried out in a UP 65M ultra-centrifuge (made in Germany) with a rotational speed of 20000 rpm for 20 minutes. The deposited materials were removed from the centrifugal tubes and put directly into graphite heaters ($\Phi = 15 \text{ mm}$, $h = 5 \text{ mm}$). After this, the materials were dried and put into a special assembly for HT-HP sintering. The samples were HP-HT sintered in this special assembly. Compacts were obtained at a pressure of $8.0 \pm 0.2 \text{ GPa}$ and temperature of $2070 \pm 50 \text{ K}$ in a Bridgman-type high-pressure apparatus.

The parameters of the powder mixture deposition (duration of deposition, rotational speed) have a significant influence on the phase distribution gradient of the materials. FGM macrostructure is shown in Figs. 7a and 7b.

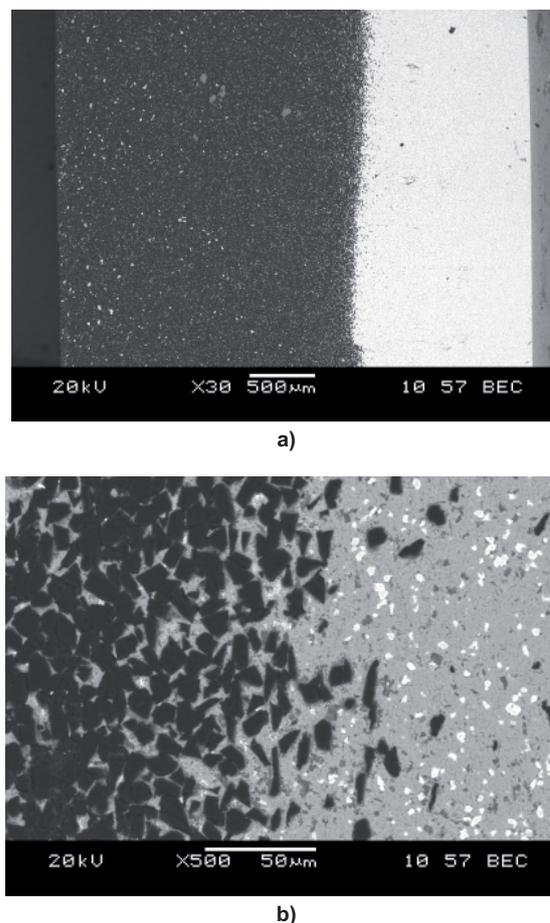


Fig. 7. Macrostructure of diamond- Ti_3SiC_2 graded material (a) Microstructure of the contact zone (b) for diamond (black) and Ti_3SiC_2 (light) phases in the composite.

FGM materials allow the replacement of traditional layers by diamond-WC/Co layers to occur. The binding material takes the role of the diamond bonding phase and substrate for edge-brazing to tool body.

8. Conclusions

For the diamond sintering processes with liquid phase participation, practically non-porous materials are obtained, due to the infiltration process.

Infiltration of carbide-forming elements was carried out and a consequent reaction with the carbon was observed in the carbide bonding phase. Carbide formation may be associated with an increase in volume, which could result in blocking of pore channels, which would affect the material porosity and roughness of the tool surface.

In composites with a carbide binding material, a tendency to graphitization is observed, due to the sintering in the solid state. For TiC-diamond composite, there is a fragmentation of TiC crystallites. In the case of the SiC bonding phase, there is no fragmentation of SiC crystallites during sintering, due to its high strength properties at high temperature.

For the intermetallic materials and silicides, materials are characterized by the multiphase composition of non stoichiometric silicides and carbides.

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