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Microwave-Assisted Molten-Salt Facile Synthesis of Chromium Carbide (Cr_3C_2) Coatings on the Diamond Particles

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Abstract: Chromium carbide (Cr_3C_2) coatings on the diamond were fabricated using the mixtures of molten salt of NaCl and BaCl_2 , powders of metallic chromium and diamond as raw materials in the temperature range of 750–900 °C by the microwave-assisted molten-salt synthesis. The morphology, microstructure and phase composition of the surface layer were characterized by Scanning Electron Microscopy, Energy-dispersive Spectrometry and X-ray diffraction. The results show that the surface of the diamond particles could be coated with Cr, forming a uniform and

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continuous Cr_3C_2 -coated layer. The coatings thickness on the diamond can be controlled by adjusting the heating temperature and time. The coating thickness expanded from 0.73 to 2.30 μm when the temperature was increased from 750 °C to 900 °C, and increasing rapidly during the temperature range of 800 °C–850 °C, the coating thickness expanded from 1.30 to 2.80 μm when the holding time was increased from 0.5 h to 4 h. The results illustrate that the microwave-assisted molten salt synthesis plays a positive role in chromium coatings on the diamond, offering a potent method for the surface metallization of diamond.

Keywords: chromium carbide coatings diamond microwave heating molten salt

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Introduction

For the diamond compacts, diamond particles are usually bonded with substrates using a binder. In order to increase the bonding strength of the diamond particles, it is useful to coat them with a material having high affinity to both of the diamond and the binder [1, 2]. The transition metal carbides have properties of very high melting points (~1727–3727 °C), high hardness (1200–3000 kg/mm²), high elastic modulus (300–700 GPa), good heat and electrical conductivity, and anti-erosion and corrosion, making them widely being used in a variety of industrial applications. Metal carbides coatings can extend the lives of the materials by allowing mechanical properties of the substrate materials to be maintained while protecting them against wear or corrosion. The coating can protect the diamond particles from graphitization at high temperature and promote the interfacial bonding without deteriorating the thermal conductivity of the metal matrix. Metal carbides can be prepared by using chemical vapor deposition, physical vapor deposition, precipitation of salts containing metal, carbon and oxygen followed by reduction and annealing, mechanical alloying and high energy milling from mixed powders of metal and carbon, etc [3, 4].

Thermal treatments have been proposed to coat the diamond particles with carbides. In the treatments, the

diamond particles react with elemental substances or oxides at a higher temperature, resulting in the surface of the diamond being coated with the carbides generated from the reaction [2, 5, 6]. However, diamond and metals are chemically incompatible, it is very difficult to make the diamond to be soaked by metal substrate, resulting in a lower adhesive strength between the diamond particles and the metal support, and a loss of diamond particles from the metal support during the grinding and cutting [7, 8]. It is well established that the diamond surface metallization is an effective method to improve the adhesive properties between diamond and surface carbide layer [9].

In order to improve the adhesive force between the metal layer and the diamond, several diamond surface metallization approaches, such as dc magnetron sputtering technique, electroless plating, electroplating, vacuum micro-deposition technology, vacuum plating, and salt bath plating, have been reported in recent years [5, 9–12]. Active elements, such as Cr, B, Ti, Mo, and W, are coated on the surface of diamond particles before sintering and infiltration to improve the interface bonding property diamond/metal composites [2, 12–23]. These metal elements can act as middle layers that strengthen the interface and protect the diamond powder from the atmosphere and reduce the degree of graphitization at high temperatures. However, the conventional powder metallurgy methods still have drawbacks of longer sintering time, higher temperature and higher energy consumption, which will produce defects in the diamond structure or form excessive interface products of a chemical reaction between the coating elements and diamond or matrix, causing a significant decrease of thermal conductivity of diamond or matrix, or even resulting in the reduced thermal conductivity of the composite.

Molten salt synthesis is now a well-established low-temperature technique, attracting increasing interest in recent years, in which the molten salts (e.g. alkali chlorides and sulphates) are utilized as a solvent or reacting species, or sometimes both, has been demonstrated to be one of the simplest methods to prepare pure and stoichiometric powders of multicomponent oxides. Since the diffusion rate of the components in molten salts are much higher than those in the solid state reaction, various oxide powders and the transition metal carbides can be prepared at relatively lower temperatures (about 950°C compared to the more typical synthesis temperature of 1400–1700°C for carbothermic reduction) and shorter reaction times (about 1 h compared to the more typical synthesis time of over 4 h for carbothermic reduction) by the molten salt synthesis method. The salt bath plating by the molten salt method not only can form a good

combined layer, but also can control the thickness of coating by adjusting the plating temperature and holding time, having advantages of lower energy consumption, and lower cost [2, 22, 24–28].

Microwave heating has gained growing attention during the past decade, a number of potential applications of microwave heating in different fields have been thoroughly reviewed [29–39]. It is well-established that intrinsic advantages in utilizing microwave technologies for processing materials compared with conventional heating include penetrating radiation, controlled electric field distribution, and selective and volumetric heating due to the difference of heating mechanism between them. For example, microwave sintering technology has unique attributes of higher heating rate, shorter processing time, it is possible to reduce internal stresses, which contribute to cracking of parts during sintering, and to create a more uniform microstructure, resulting in the improved mechanical properties and reliability by eliminating temperature gradients, and has been developed as a new technique for controlling microstructure to improve the properties of sintered materials, which has been widely used in many fields [33, 35, 38–40].

Crystalline Cr_3C_2 has wide applications in coatings and structural reinforcement, attracting wide research due to its excellent properties of high melting point, hardness, strength, toughness, chemical stability, and corrosion resistance [41]. The chromium carbide was usually synthesized by using the carbon thermal reduction method using chromium oxide and carbon powders as raw materials. For chromium carbide produced in the molten salt, Dai et al. [42] investigated the production of nano-sized chromium carbide powders from $\text{Cr}_2\text{O}_3/\text{C}$ precursors by direct electrochemical reduction in molten calcium chloride. The results showed that the single phase Cr_7C_3 powders with a mean diameter of <200 nm were prepared after electrolysis at 2.8 V for 5 h in 850°C CaCl_2 melt. For chromium carbide fabricated by microwave processing, Zhao et al. [43] and Zhao and Hu [44] reported the synthesis of chromium carbide nanopowders from nanometer Cr_2O_3 and nanometer carbon black by microwave heating technique, and using micron-sized chromic oxide (Cr_2O_3) and nano-sized carbon black as raw materials via mechanical alloying assisted microwave heating route, concluding that the synthesis temperature and time required by the microwave heating method is at least 400°C lower and 3 h shorter than those required by the conventional approaches for preparing chromium carbide, making the distribution of particle size more uniform, Gunnewiek and Kiminami [45] reported the fast synthesis of porous chromium carbide by microwave-assisted carbothermal reduction from Cr_2O_3

and carbon black in only 20 min of reaction time, further proved the advantages of microwave heating for the preparation of chromium carbide coatings.

Although the copper-diamond composites with chromium carbide coatings on diamond particles by vacuum pressure infiltration, spark plasma sintering technique, or conventional sintering method [12, 17, 46, 47], silicon carbide coating on the diamond particles [48], TiC coating on the diamond particles [20] by microwave heating have been reported; and silicon carbide (SiC)-coated graphite composite powders was synthesized using graphite flakes and silicon powders as raw materials by the microwave-assisted molten salt synthesis [49], there has been little discussion about the chromium carbide coatings on the diamond particles by microwave sintering process in a molten salt mixture.

Herein, on the premise of making full use of the advantages of microwave sintering and molten salt synthesis mentioned above, a facile microwave-assisted molten salts synthesis was developed for the low-temperature preparation of chromium carbide coatings on the diamond particles. In order to evaluate the effects of plating temperature and time on the coating thickness, the Cr/diamond samples were heated at temperatures of 750, 800, 850, and 900°C for 1 h, respectively, and also heated at 850°C for 0.5 h, 2 h, and 4 h, respectively. Scanning Electron Microscopy (SEM), Energy-dispersive Spectrometry (EDS), X-ray diffraction (XRD) was adopted to analyze the morphology, microstructure and phase composition of the surface layer.

Experimental

Materials and apparatus

Diamonds with average diameters of 125 µm (Henan Huanghe Whirlwind CO., LTD.), Chromium powder (reagent grade, 99 % pure, Reagent Chemicals Co., Ltd Tianjin.) NaCl (reagent grade, Reagent Chemicals Co., Ltd Tianjin.) and BaCl₂ (reagent grade, Reagent Chemicals Co., Ltd Tianjin.) were used for the salt molten coating process by microwave and conventional heating. Figure 1 shows an SEM image of the uncoated diamond.

High temperature microwave furnace (HAMiLab-V3000, SYNOTHERM Co., Ltd Changsha, China) was used for heating the material to the salt-melt temperature, and the Multimode cavity of high temperature microwave furnace with 2.45 GHz of maximum 3 kW

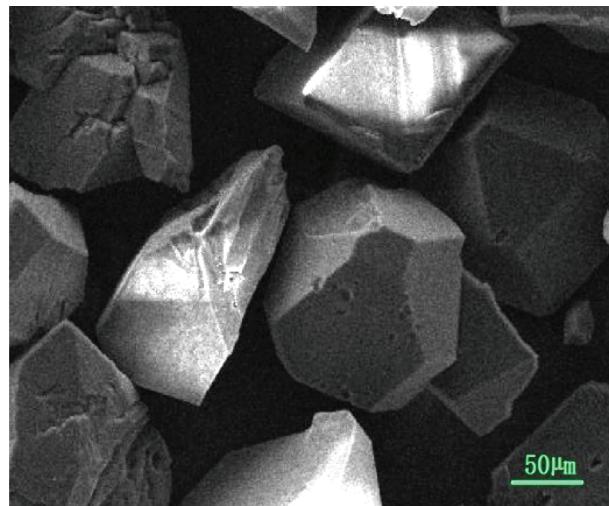


Figure 1: SEM micrographs of uncoated diamond.

output power was illustrated in Figure 2, which was cooled via water circulating in the double wall. SiC slice was used to increase the overall heating rate in the microwave cavity. Dilute hydrochloric acid (reagent grade, Reagent Chemicals Co., Ltd Tianjin.) and Acetone (reagent grade, Xin Guan Chemical Co., Ltd Wei Fang) were used for washing the diamond. Thermostatic water bath (HH-1, Hangzhou David Scientific Instrument Co., Ltd.) and Ultrasonic cleaning (PS-1001T, Hefei climbed Ultrasonic Technology Co., Ltd) were used for removing excess powder and dissolving the molten plating

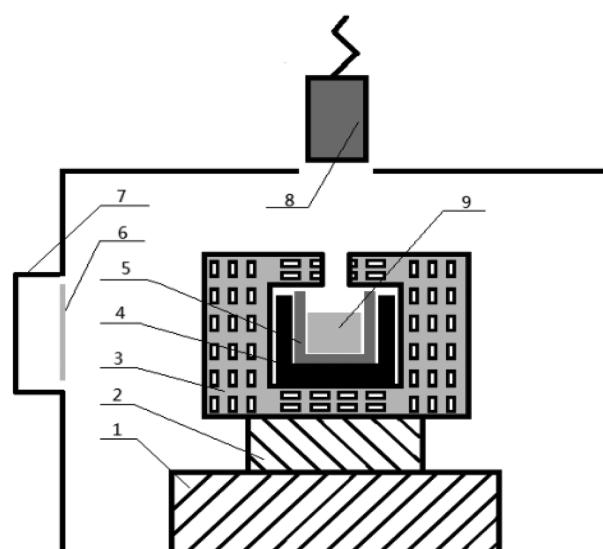


Figure 2: The cavity of high temperature microwave furnace (1-Rotating stage, 2-Loading ring, 3-Alumina insulating sheath, 4-SiC slice, 5-Corundum crucible, 6-Quartz glass, 7-microwave generator, 8-Infrared pyrometer, 9-Samples).

Pretreatment of diamond

Before the diamond deposition, the pretreatment of the diamond was carried out to increase the surface activity. The diamonds were immersed in dilute hydrochloric acid for half an hour, and then cleaned with acetone to remove the remaining wax, catalysts and other impurities, and then washed with distilled water two times and dried in digital blast oven for obtaining the suitable experimental samples.

Preparation of chromium carbide coating on diamond powders

The diamond coating was prepared by heating the well-blended mixtures of 20 g molten salt of NaCl and BaCl_2 (1:1), 10 g metallic chromium and 10 g diamond powder by a microwave furnace at the heating rate about $30^\circ\text{C}/\text{min}$. The chromium was embedded in the salt mixture, because this procedure was assumed to be protective against a reaction of chromium with the oxygen in the atmosphere. The gaseous precursor chosen for diamond coating was a gas mixture of Ar , H_2 (volume ratio: Ar 85%, H_2 15%). The flow rates of Ar and H_2 were 1700 sccm (standard $\text{cm}^3 \text{min}^{-1}$) and 300 sccm, respectively. The coating pressure was maintained at the standard atmospheric pressure. The coating temperature was determined by an infrared pyrometer (Opiris IRtec 10:1 M, Germany). In order to study the effects of plating temperature and time on the coating thickness, the Cr/diamond samples were heated at temperatures of 750, 800, 850, and 900°C for 1 h, respectively, and also heated at a temperature of 850°C for 0.5 h, 2 h, 4 h, respectively.

Characterization of diamond coatings

Scanning electron microscope (XL30 ESEM-TMP, Dutch Philips CO., LTD.) was used to characterize the surface and cross-sectional morphologies of the diamond coatings. An energy dispersive X-ray spectrometer (EDS) (PHOENIX, American EDAX CO., LTD.) was utilized to perform Microanalysis of the selected areas of the samples.

The diamond coatings were analyzed with an X-ray diffraction (XRD, Germany BRUKER CO., LTD) apparatus at a grazing angle of 2° (scan step size: 0.02°). The cross-sectional material component analysis was determined by EDS Liner Scanning (PHOENIX, American EDAX CO., LTD.).

Results and discussions

Heating chromium powder by microwaves

The microwave coupling capability of materials depends on their dielectric properties: coupling increases when the dielectric loss is more significant [36]. Theoretically, bulk metals with higher conductivity will reflect microwaves or microwaves cause sparking of metallic materials at low temperature, which cannot be effectively heated by microwaves, however, it is well established that metal powders can be readily heated by microwaves [40]. Therefore, the microwave absorption ability of the chromium powders and the temperature rising curve of chromium powders were investigated. Figure 3 shows the temperature rising curve of chromium powders heated by microwaves, illustrating that the chromium powders can be heated up rapidly. The temperature of the chromium powders increases slowly from 0°C to 600°C in about 300 s, while increasing rapidly from 600°C to 1000°C in just 100 s, suggesting that 600°C is the critical point for chromium powders absorbing the microwave energy. The results demonstrate that it is feasible to coat diamond with chromium in a molten salt mixture heated by microwaves.

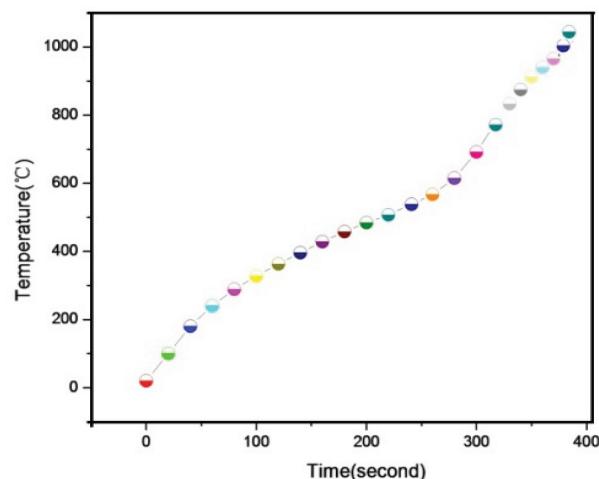


Figure 3: The temperature rising curve of chromium powder heating in the 2.45 GHz microwave furnace.

Coating diamond with chromium in a molten salt mixture by microwave heating

The Cr-coated diamond was prepared by using the well-blended mixtures of 20 g molten salt of NaCl and BaCl_2 (1:1), 10 g metallic chromium and 10 g diamond powder

as raw materials heated by microwaves at the heating rate about 30 °C /min. Figure 4 exhibits the SEM micrographs with and without Cr coating on diamond surfaces. It can be seen in Figure 4(a) that the surface of the uncoated diamond was clean and smooth. In contrast, it was observed in Figure 4(b) that the surface of the coated diamond was rough and a large amount of residues was retained, which confirms that chemical reactions occurred at the diamond interface. Compared with the transparent yellow color of the uncoated synthetic diamond, the color of coated diamond is opaque dark gray after the formation of Cr layer. The Cr coating is uniform and macroscopically homogeneous on the surface of diamond via the molten salt technique by microwave heating. It can be observed that the diamond particles still keep their initial shapes (regular polyhedrons) after coating deposition and is not damaged by high temperature burning. The surface defects of uncoated diamond such as stairs, pits, cracks are repaired during the salt-melting deposition under microwave heating.

Figure 5 shows the EDS of Cr-coated diamond by microwave heating at the temperature of 850 °C for 1 h, indicating that the surface coating mainly contains chromium and carbon and the ratio of the two atoms is close to 3:2, and suggesting that the elemental Cr in the surface layer has been reacted to form the Cr_3C_2 phases. It is well documented that chromium carbides show complex structures, presenting three stable phases: cubic Cr_{23}C_6 , two orthorhombic Cr_7C_3 and Cr_3C_2 [3, 50]. Cr_3C_2 is the most stable transition metal carbide exhibiting high hardness, good strength, high Young's modulus, high corrosion and erosion properties, good chemical stability and high oxidation resistance [41].

Figure 6 shows the XRD pattern of the Cr-coated diamond under microwave heating at a temperature of 850 °C for 1 h. The patterns contain peaks corresponding to diamond and chromium carbide, indicating that the coating on the diamond particles exhibits high purity by microwave heating at the temperature of 850 °C under the argon-hydrogen atmosphere. Meanwhile, Cr_3C_2 are formed on the surface

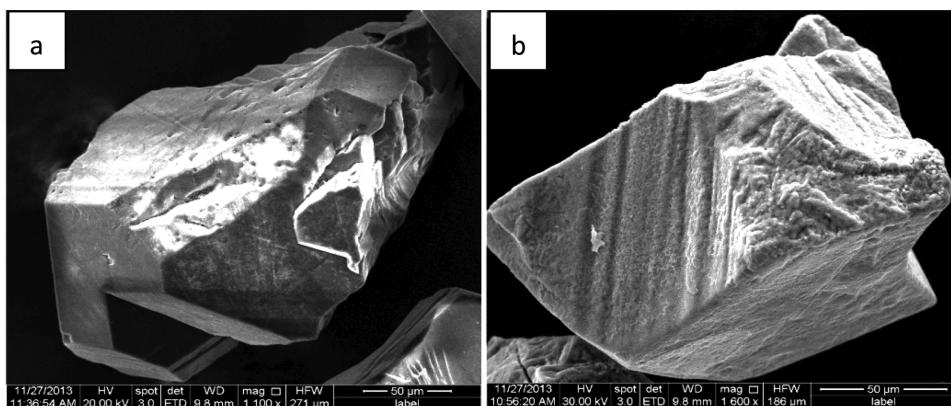


Figure 4: SEM micrographs of diamonds: (a) without coating and (b) with Cr coating.

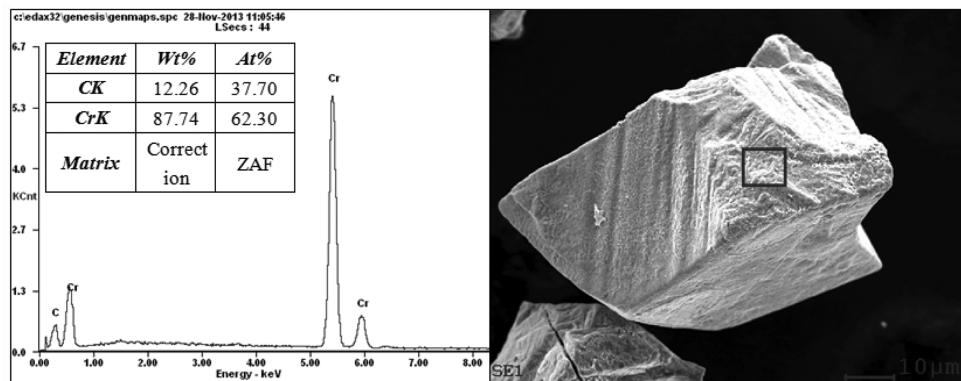


Figure 5: The EDS of coated diamond by microwave heating at the temperature of 850 °C for 1 h.

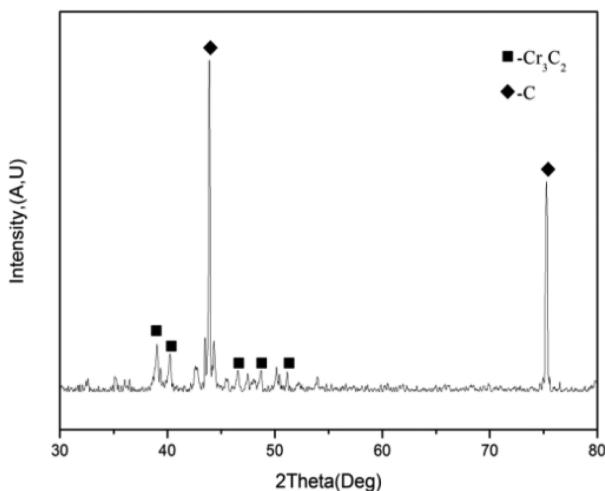


Figure 6: X-ray diffraction pattern of coated diamond by microwave heating at the temperature of 850 °C for 1 h.

of diamond particles, revealing that interfacial chemical reaction between Cr and C elements has occurred during the microwave salt-bath process.

Based on the above mentioned XRD, EDS, and SEM analyses, the result indicates that the surface of diamond particles could be successfully coated with Cr using microwave-assisted molten-salt synthesis method, and Cr_3C_2 was generated during coating simultaneously. The present result is almost similar to the result of the copper-diamond composites with chromium carbide coatings on the diamond particles by vacuum pressure infiltration technique, the coatings on the diamond particles were Cr_7C_3 , which were formed with a reaction medium of chromium in mixed molten salt of NaCl and KCl (1:1) [6], and were in good agreement with the result of Cr_2C_3 prepared from nanometer Cr_2O_3 and nanometer carbon black by microwave heating technique, in which chromium carbide

nanopowders were prepared by a microwave heating method at 1000 °C for 1 h [43, 44].

Molten salt investigation revealed that transport reactions occur because metals dissociated to mobile cations and delocalized electrons, a state which is considered to be intermediate between ionic and metallic, facilitating the dissolution and transport of the chromium, and hence the formation of the chromium carbide coatings, through the diffusion of chromium cations from the molten salt to the surface of the diamond particles with subsequent reaction, demonstrating that the chemical reaction in molten salt system was easier and more uniform [25, 42]. Prior research showed that the advantages of salt bath plating are simple to operate, easy to control the coating thickness, and carbide layer can be directly formed on the diamond surface uniform and compact.

Meanwhile, the non-thermal effects of microwave show that the electromagnetic fields can reduce sintering activation energy of materials, improve diffusion of grain boundary and bring an extra driving force for mass transport, which result in low sintering temperature and short sintering time required for full densification of materials. Therefore, the microwave sintering is an energy and time saving sintering technology, and can be used to fabricate the materials with fine grains and homogeneous microstructure [33, 38, 39]. For the present study discussed above, Cr powders can absorb the microwaves, accelerating the motion and distribution of Cr metal and resulting in the coatings more uniform and compact [40]. Furthermore, it has been demonstrated that Cr_3C_2 can also couple effectively with microwaves [43, 44], which results in a more uniform temperature gradient within the billet, making the carbide layer formed on the diamond surface more uniform and compact.

Figure 7 illustrates the SEM images of Cr-coated diamond by (a) microwave heating and (b) conventional heating

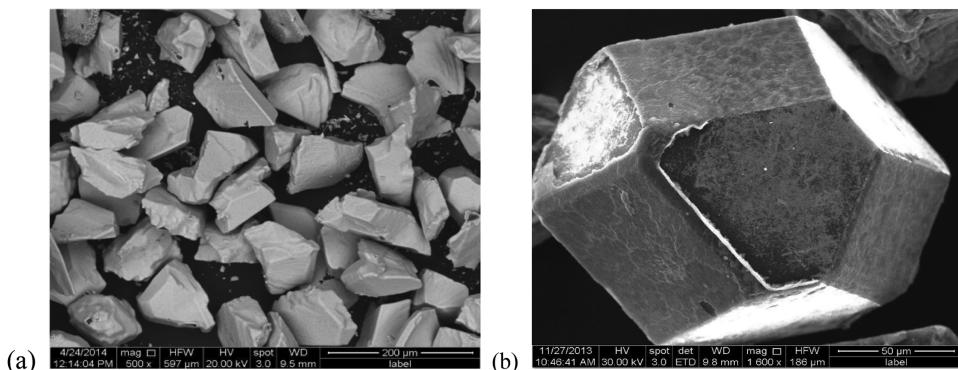


Figure 7: The SEM images of coated diamond by (a) microwave heating and (b) conventional heating at temperature of 850 °C for 1 h.

heating at the temperature of 850°C for 1 h. It can be found in Figure 7(a) that the diamond plating by microwave heating is more uniform and homogeneous than that of conventional heating (Figure 7(b)), illustrating that the chromium carbide coating can be synthesized at 850°C for 1 h by the microwave-assisted molten-salt synthesis. The reaction temperature of the present study was about 150°C lower than the work reported by Zhao et al [43],, in which chromium carbide nanopowders were prepared by a microwave heating method at a temperature of 1000°C for 1 h, highlighting the advantages of microwave-assisted molten-salt synthesis method developed in the present study.

Coating thickness of diamond by microwave heating

In order to evaluate the effects of heating temperature and time on the coating thickness, the Cr/diamond samples were heated at temperatures of 750, 800, 850, and 900°C for 1 h, respectively, and also heated at a temperature of 850°C for 0.5 h, 2 h, 4 h, respectively. Figure 8. shows the

coating thickness of SEM images by microwave heating at different temperatures of 750, 800, 850, and 900°C for 1 h, and Figure 9 shows the coating thickness of SEM images by microwave heating at the temperature of 850°C for different time (a) 0.5 h, (b) 2 h, and (c) 4 h. It can be seen in Figure 9 (a) that the coating adheres to the surface of the diamond which confirms a strong interfacial bonding between diamond, while it appeared obviously cracking phenomenon in Figure 9(c), because too thick coating has large thermal stress during the cooling process.

Figure 10(a) exhibits the relationship between coating thickness and plating temperature, which indicated the interlayer expanded from 0.73 to 2.30 μm, when the temperature was increased from 750°C to 900°C, suggesting that the formation of Cr₃C₂ interface species could be promoted by the heating time. Moreover, the coating thickness increased rapidly in the temperature range of 800 ~850°C, because the interface chemical reaction and the formation of Cr₃C₂ species were greatly affected by temperature in this range.

In order to reveal the effect of heating time on the coating thickness, the samples were heated by microwaves at a temperature of 850°C for 0.5 h, 1 h, 2 h and

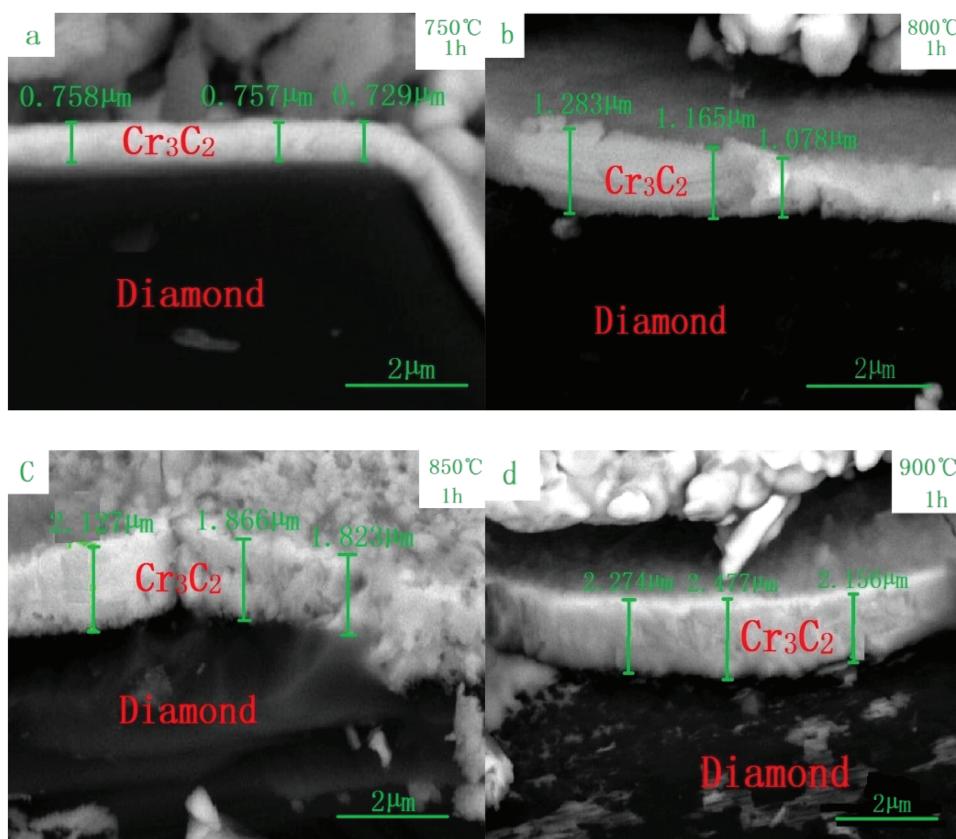


Figure 8: The coating thickness of SEM images by microwave heating at different temperatures for 1 h.

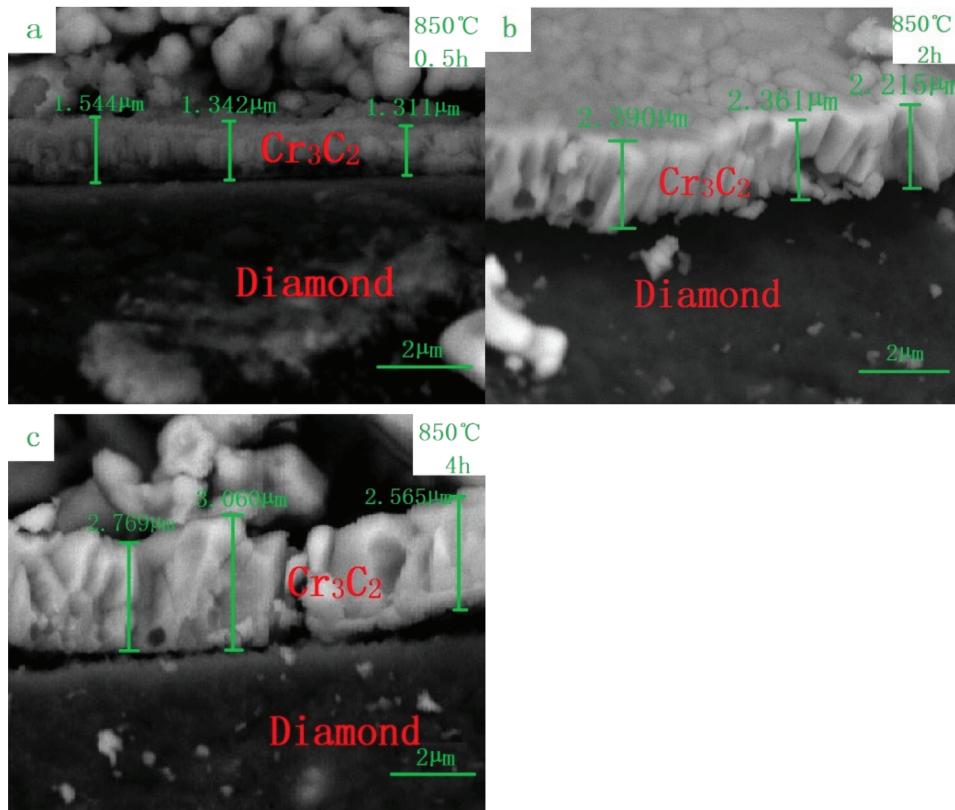


Figure 9: The coating thickness of SEM images by microwave heating at temperature of 850 °C for different time (a) 0.5 h, (b) 2 h and (c) 4 h.

4 h (Figure 10(b)). Figure 10(b) shows the relationship between coating thickness and holding time, which exhibits that the coating thickness expanded from 1.30 to 2.80 μm when the holding time was increased from 0.5 to 4 h. In addition, when thick carbide film formed on diamonds, the diffusion of Cr atoms goes through the carbide film became difficult, resulting in the coating thickness increased more and more slowly with the increasing of holding time. Chromium coating on diamond belongs to solid - solid phase reaction

and the reaction rates are affected by the diffusion rate of chromium atoms going through a phase [6, 46].

The results demonstrated that the plating temperature and time had substantial impact on the thickness of Cr coating, suggesting the thickness of coating on the diamond by microwave heating can be controlled by adjusting the heating temperature and holding time.

Compared with the methods of carbothermic reduction or via molten-salt synthesis by conventional heating, the microwave-assisted molten-salt synthesis has the

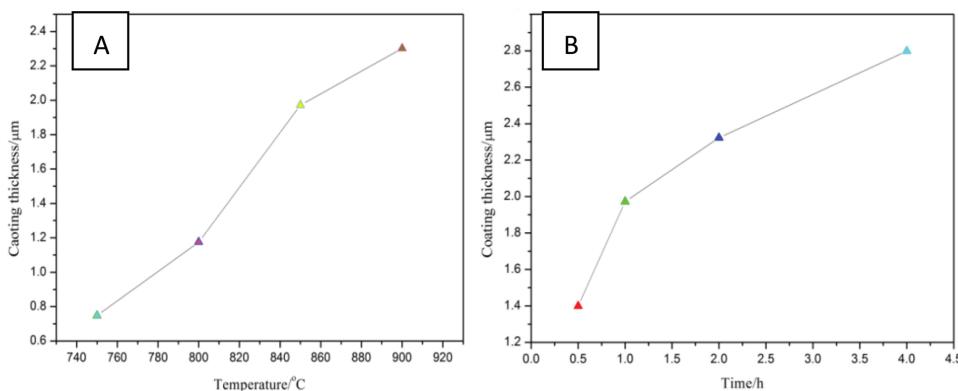


Figure 10: The relationship between coating thickness and (a) plating temperature and (b) holding time.

advantages of lower reaction temperature and shorter reaction time. In a word, the chromium carbide (Cr_3C_2) coatings on the diamond particles can be prepared by making full use of the advantages of microwave heating and molten salt synthesis.

Conclusions

A continuous chromium coating on the diamond was achieved using a molten salt technique by microwave heating in the temperature range of 750–900°C. The thickness of coating on the diamond by microwave heating can be controlled by adjusting the heating temperature and holding time. The interlayer expands from 0.73 to 2.30 μm , when the annealing temperature increases from 750°C to 900°C, the coating thickness increases rapidly during the temperature range of 800~850 °C, because the interface chemical reaction and the formation of Cr_3C_2 species are greatly affected during this temperature range, while, the coating thickness expands from 1.30 to 2.80 μm when the holding time increases from 0.5 to 4 h. The results suggest that the molten salt synthesis by microwave heating maybe a technically viable option for the diamond surface metallization.

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