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Analysis of Laser-Brazed Diamond Particle Microstructures

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Brazing diamond particles to a steel substrate using Ni-based filler alloy was carried out via laser in an argon atmosphere. The brazed diamond particles were detected by scanning electron microscope (SEM), X-ray diffraction (XRD), and energy dispersive X-ray spectroscopy (EDS). The formation mechanism of carbide layers was discussed. All the results indicated that a high-strength bond between the diamond particles and the steel substrate was successfully realized. The chromium in the Ni-based alloy segregated preferentially to the surfaces of the diamonds to form a chromium-rich reaction product, and the bond between the alloy and the steel substrate was established through a cross-diffusion of iron and Ni-based alloy.

Keywords: laser brazing, diamond, Ni-base alloy, interfacial microstructure.

1. INTRODUCTION

Because of their hardness, diamonds are widely used for cutting, grinding, and polishing. They are frequently used in the form of composite structures, wherein diamond particles are bonded using plastic, ceramic, or metallic bonding matrices. Among these three categories of bonding agents, metallic bonding agents offer the optimal mechanical and thermal properties and are the best bonding material for the fabrication of diamond tools. An important category of diamond tools have a monolayer of diamond particles, typically fabricated with electroplating. However, electroplated diamond tools have significant problems, such as small holding space and low bonding strength between the particles and metal matrix. This causes the diamond particles to become easily dislodged during grinding, which is a key reason their use is restricted in modern machining industries [1 – 7].

In order to alleviate the setback mentioned above, high temperature brazed diamond grinding tools have garnered attention, and have been extensively researched in the engineering and technology fields over the last twenty years. The particles, the brazing alloy, and the substrate for this type of grinding tool bond tightly together through a chemical reaction. Currently, high-temperature brazed monolayer diamond particle tools are primarily fabricated by vacuum and high-frequency induction brazing [6 – 7]. However, these two brazing methods have limitations in practical applications. During brazing carried out in a vacuum environment, heating and cooling times add to the length of the production cycle, and synthetic diamond particles tend to degrade quickly and transform to graphite when exposed to high temperatures for several minutes. Due to the restrictions of furnace sizes, large soldering tools are difficult to employ. Additionally, plastically deformed substrates should not be used, as high temperature brazing can cause shape distortion of the substrate. When brazing is carried out by high-frequency induction, the heating and cooling cycles are short, and the entire brazing cycle is reduced significantly. However, with the different shapes of tools brazed by high-frequency induction, the induction loops have to be redesigned and reproduced. For large uniform-surfaced tools, the process mentioned above is complex. An alternative is to apply heat to activate the brazing process in a narrowly selected area and carry out the brazing process continuously to heat the entire brazed area. Laser beams with high energy density and low energy input have the properties to perform in such an application. These attributes can achieve the goals of a short heat treatment duration, small heat zone, and consequently, minimal deformation of the substrate and high efficiency production of diamond tools. It was the goal of this study to evaluate the feasibility of laser processing in the fabrication of diamond-brazed tools [8 – 9].

2. EXPERIMENTAL PROCEDURES

The brazing alloy used in this project was Ni-Cr-B-Si powder. The solidus temperature of the alloy was 1020 °C. The diamond particles were not coated, and the particle sizes ranged from 315 to 400 *um*. The substrate used was 45 steel with dimensions of 50 mm × 6 mm × 6 mm. Before brazing, the substrate and the diamond particles were ultrasonically cleaned in acetone. Then, the diamond particles were mixed with the brazing powder in a determined ratio and applied to the steel substrate. Finally, the assembled specimens were subjected to laser brazing.

Laser brazing was carried out using a cross flow CO_2 numerical control laser (TJ – HL-T5000). The laser beam was a TEM00 (Transverse Electro-Magnetic) mold. The power input was 600 – 800 W, the scanning velocity was 25 – 35 mm/min, and the beam was 3 mm * 6 mm. Argon with a flow rate of 100 cm^3/s was used as a shielding gas to protect the diamond particles and brazing alloy from oxidation. Laser brazing was a quick but unbalanced heat-circulation process.

After brazing, the samples were cut, mounted, and polished for evaluation of the interfacial microstructures

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between the particles and the brazing alloy, which were examined by scanning electron microscopy (SEM) (Japanese Electronics Company JSM-6300). The XRD (X-ray diffraction Japanese Neo-Confucianism Company D’max) and EDS (American KEVEX Corporation) were used for analyzing the phase components of the reaction products and the elemental line distributions.

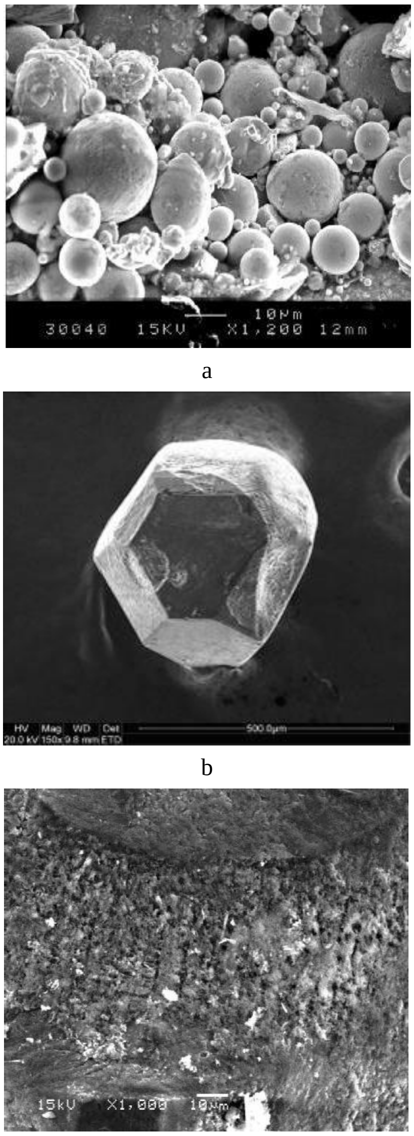
3. RESULTS AND DISCUSSION

3.1. Linear energy

In order to investigate the effects of the laser brazing under different process parameters, linear energy was introduced in this study [6]:

$$\rho = \frac{p}{bv} \tag{1}$$

where ρ is the linear energy (J/mm^2), p is the laser power (W), b is the diameter of laser spot (mm), and v is the scanning speed (mm/s).



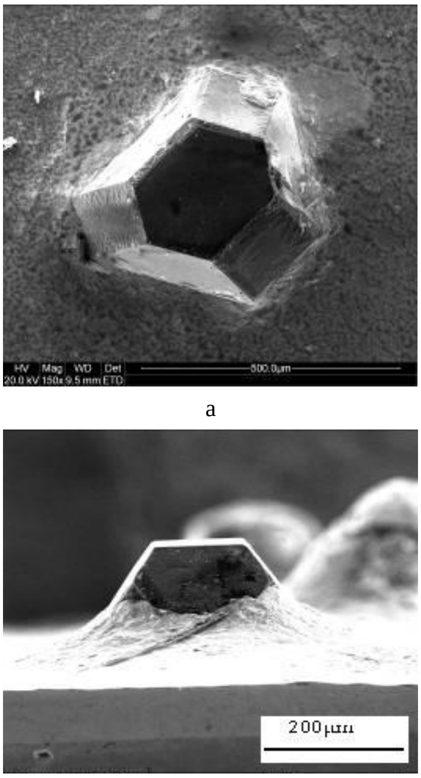
a

b

c

Fig. 1. Analysis of the experiment: a – un-melted filler alloy; b – graphitized diamond; c – local amplification image

Different results of laser brazing under different process parameters are defined by the linear energy. If the linear energy is less than 243.5 J/mm^2, the diamond particles become dislodged while grinding on marble. An example is illustrated in Fig. 1 a, where solder balls can be seen in the resulting hole.If the linear energy is more than 253.5 J/mm^2, the diamond particles become graphitized, as can be seen in Fig. 1 b and c. The substances in the surface of diamond are flocculent if we amplified the graphitized area. If the linear energy is between 243.5-253.5 J/mm^2, a stronger laser-brazed diamond is realized. Figure 2 shows the combined morphology of the Ni-Cr alloy and the diamond particles after laser brazing, a phenomenon could be observed that much crescent Ni-Cr alloy coated on the diamond grain edge. Obviously, the wetting of Ni-Cr alloy to diamond is good, there could be some reactions between the Ni-Cr alloy and the diamond grain. In addition, there are no cracks or thermal damage on the diamond surfaces, and the shapes of diamond particles are intact.



a

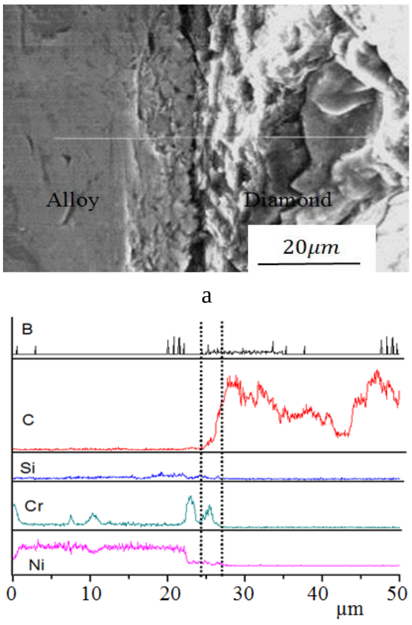
b

Fig. 2. The SEM image of the diamond: a – the top morphology, b – the side morphology

3. 2. Analysis of the interface between diamond particles and Ni-Cr alloy

Fig. 3 a shows a cross-sectional SEM micrograph of the bond between the diamond particles and the Ni-Cr alloy. The shallow gray areas in the center of the image are the reaction zones adjacent to the diamond particles. Fig. 3 b shows an EDS line scan across the interface that is shown in Fig. 3 a.

The EDS analysis shown in Figure 3.b indicates that the distribution of elements was not uniform. The interface between the diamond particles and the brazed alloy shows that the active element Cr segregated preferentially to the diamond particles, and the content of Cr atoms was much higher than that in the original alloy.



a

b

Fig. 3. Analysis of the experiment: a – micrograph of surface cross-section; b – concentration cirves of elements in interface across the surface

Clearly, a Cr-carbide layer formed between the diamond particles and the brazed alloy due to the fact that there was a strong affinity between the element Cr and C.

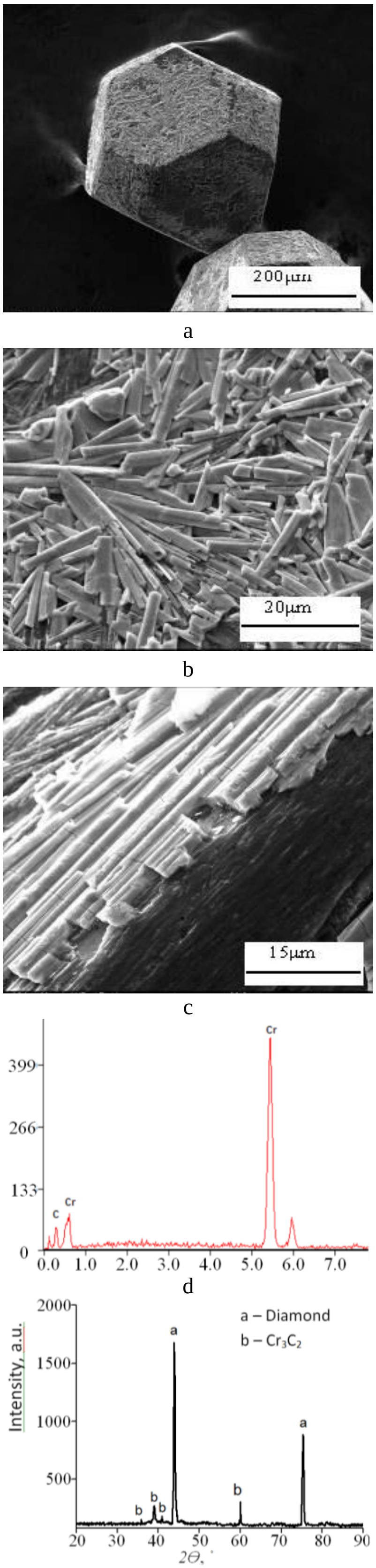
Moreover, the brazing alloys also included B and Si, whose functions are to reduce the alloy's melting point and enhance the fluidity and wetting quality. The Cr-C compound is fairly chemically stable, is anti-corrosive, and not easily dissolved. In order to observe the resultant interfaces, the laser-brazed specimens were immerged into the mixed acids for several hours and then rinsed with acetone. The interfacial characteristics of the grains were studied under SEM. Fig. 4 a shows the SEM image of the chromium carbide on the diamond surfaces. It is evident that there are laminated carbides on the particle surfaces, as shown in Fig. 4 b and c. During the ingredient analysis of the carbide, the corresponding element analysis by EDS indicated that the main composition involved Cr and C, as can be seen in Fig. 4 d. Table 1 shows that the atom ratios of C and Cr in the flake carbide deposited on the diamond particle surfaces was approximately 2:3, which is referred to as Cr_3C_2.

Table 1. The EDS results of carbide atom ratio

Element	Weight %	Atomic %
C	13.20	39.70
Cr	86.80	60.30

3. 3. Microstructures of the formed compounds in the interfacial region and XRD analysis of the compounds

In order to prove the existence of active element Cr in the interfacial region during brazing, the phases of the compounds were further analyzed using XRD. It was discovered that the compound regions contained only Cr_3C_2, as shown in Fig. 4 e.



a

b

c

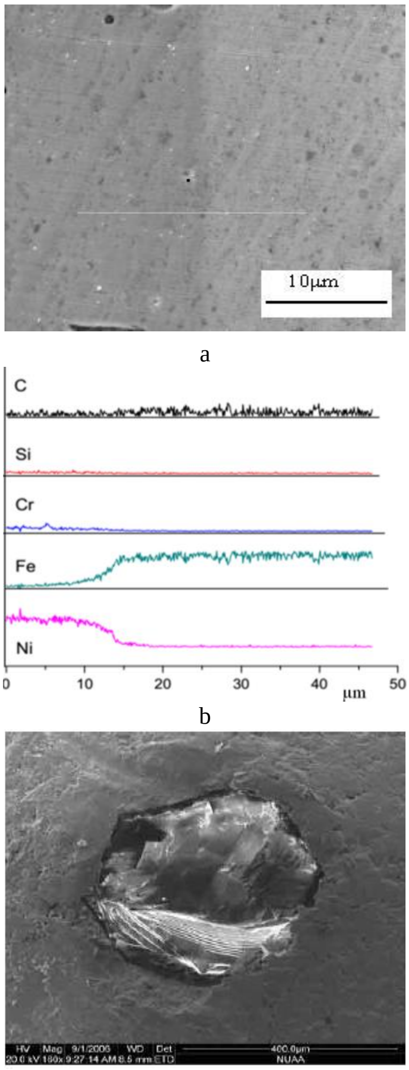
d

e

Fig. 4. Compound micrographs, EDS and XRD analysis of brazed diamond particles: a – morphology; b – aminated carbide; c – the topography between carbide; d – EDS of carbide in the surface and diamond particles; e – X-beam diffraction analysis

Therefore, the laminated compounds were confirmed to be Cr_3C_2. With the bridge effect of the special Cr-C compounds, the abrasive diamond particles bonded firmly with the filler alloy.

The growth characteristics of the carbides were observed in the deep corrosion of the diamond surfaces. The results showed that the Ni-Cr alloys reacted with C in the diamond surfaces to form nucleation of carbides and epitaxial growth (Fig. 4 c). The Cr_3C_2 linear expansion coefficient (9.70×10^{-6}/ °C) was between the diamond (approximately ($1.5 – 4.8 \times 10^{-6}$/ °C) and solder (approximately 1.2×10^{-5}/ °C). It was beneficial in reducing the residual stress that was caused by the difference in the linear expansion coefficient between the diamond particles and the filler metal [6 – 9].



a

b

c

Fig. 5. Micrograph and corresponding elemental distribution between filler metal and steel substrate: a – micrograph of filler metal and steel substrate; b – corresponding elemental distribution; c – fracture morphology of brazed diamond grain

3. 4. Interfacial reaction region between filler metal and steel substrate

In order to discover the level of bonding between the brazed alloy and the steel substrate, the microstructure and corresponding elemental line distribution images along the red line at the interface between the filler metal and the 45 steel substrates were acquired, as shown in Fig. 5 a and b Large quantities of Cr, Ni, Si, and Fe atoms were seen concentrated around the interface. It was apparent that a good chemical metallurgy bond had been formed between the alloy layer and steel substrate.

To demonstrate the bond strength of the brazing interface between the diamond particles and the filler layer using Ni-Cr powder mixture as the active brazing alloy, the brazed specimen was transversely sheared; the typical fracture surface of the particle is illustrated in Fig. 2. It was clear that the particles were not dislodged from the filler layer. Furthermore, the margin of the fracture region near the particles was completely covered with the filler layer. This suggested that the bond between the diamond particles and Ni-Cr powder was stronger than the strength of the molten filler alloy itself, meeting the requirements for grinding hard materials. Additionally, according to the features of the fractured surfaces of diamond particles (Fig. 5 c), it could be assumed that an intense chemical reaction took place between the diamond particles and the Ni-Cr powder filler in the laser brazing process. Otherwise, dislodging of diamond particles from the connecting layer would have been observed. Electroplated abrasive tools typically do not have a strong bond between the connecting layer and the diamond particles, causing the particles to come loose.

3. 5. Diamond kinetics analysis and Ni-Cr solder interface

The interfacial reaction between diamonds and Ni-Cr alloy is a process of reaction-diffusion that is caused by the diffusion of C atoms from the diamonds to the solder layer and the segregation of Cr atoms from the solder layer to the abrasive surface. The interface reaction diffusion layer is generated by two methods: First, by the physical phenomena caused by capillary liquid solder penetration; second, the diffusion of Cr solders alloys and the chemical reaction with the diamond particles. As such, this diffusion can also be called a reaction-diffusion, where its rate was significantly affected by the two factors of the chemical reaction rates and Cr atomic diffusion speed.

During the diamond laser soldering process, when the sample was heated after brazing to a certain temperature, the carbon atom or carbon atoms groups spread form the diamond to solder layer through the pore space and the space group due to the increased lattice vibrations of the diamond particles. Dangling bonds were present on the surface of the carbon atoms, and the chromium is transition metals with 3d-shell electron-deficient. The carbides with Cr atoms were formed at high temperatures by the interactions of d-sp^2 and d-sp^3. The formation of carbide organizations followed the relationship between the concentrations of each phase region of the Cr-C phase diagram, thus the phases of carbides from low concentration to high concentration were formed sequentially, and these phases developed in the direction of maximum thermodynamic stability. Therefore, the phases of carbides from the diamonds to the solder interface followed δ phase, α phase, and β phase, and because the chemical energy in the surface was higher than the inner chemistry, the diffusion activation energy of the surface atoms was lower than the internal atomic diffusion activation energy. There were many crystal defects in the diamond surfaces that were unevenly distributed. Therefore, the highest atomic chemical energy was in the distortion of the diamond lattice, while the arrangement of atoms in the crystal surface was irregular. There, the diffusion activation energy of the atoms was lower and the diffusion rate was faster. Therefore, δ phase (Cr_3C_2) separated out initially in the defects in the diamond surfaces. The carbide shape was from spotty to form flake shape with the time coursing (see Fig. 6), the entire process consisted of the diffusion and phase changes [7 – 10].

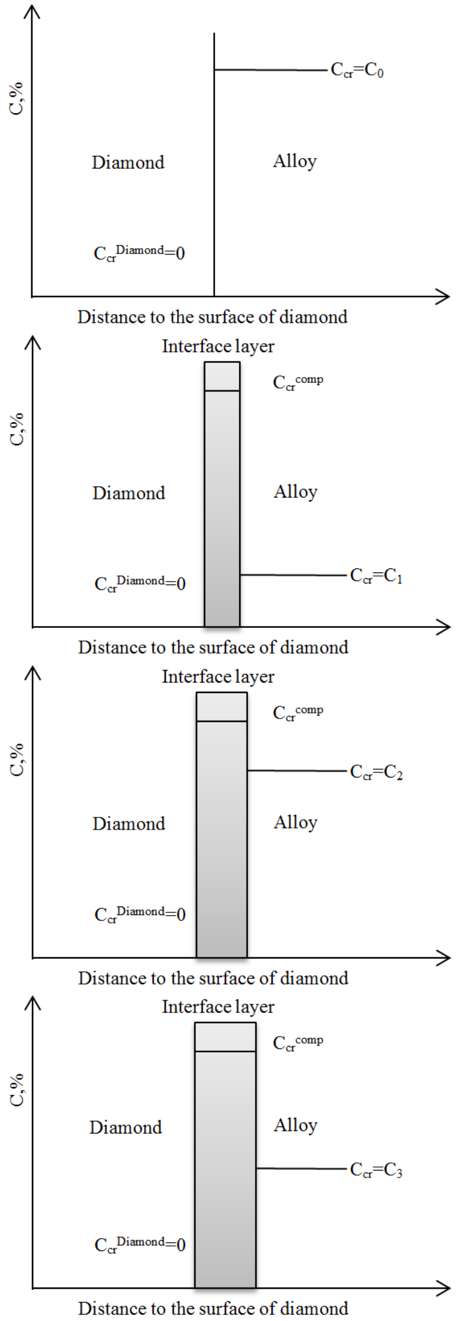


Fig. 6. Schematic of the reaction

When the carbide Cr_3C_2 crystal nucleus began to form with the diamond for particle boundary nucleation growth, large amounts of active elements were consumed at the interface of the Cr, reducing the amount of Cr, so that the concentration gradient of the element Cr in solder layer increased sharply. This provided the driving force through which the element Cr away from the abrasive continued to spread to the interface. The higher the Cr contents in the solder, the greater the concentration gradient and the faster the diffusion of Cr, which was advantageous to the thickening of the interface reaction layer. Figure 6 shows the interface reaction, where C_0 is the original Cr content of the solder layer, $C_{Cr}^{Diamond}$ is the Cr content of the diamond particles, C_{Cr}^{Comp} is the Cr content in the interface compound, and C_{Cr} is the Cr content of the solder layer near the interface layer with time.

As the temperature rose, the solder became molten and the reaction proceeded with the continued proliferation of Cr and C. Then, the Cr diffusion was mainly affected by the abrasive concentration at the interface of the solder and the concentration gradient of the Cr content. The diffusion process between Cr and C can be regarded as a mobile problem of phase interface under control conditions. Multi-phase reaction diffusion occurs in the diffusion process. The organization formed of multi-phase reaction layers should follow the relationship between the concentrations of each region of the Cr-C phase diagrams. Accordingly, different carbides are generated in the diamond surfaces from the inside to the outside sequentially: $Cr_3C_2 \rightarrow Cr_7C_3 \rightarrow Cr_{23}C_6$. The structure of carbides depends on the relative number of active Cr atoms and C atoms, where Cr_3C_2 is generated when the quantity of Cr atoms is insufficient; however, when the number of Cr atoms is sufficient, Cr_7C_3 is generated. Lastly, $Cr_{23}C_6$ is produced when there are too many Cr atoms. Due to the very short duration of reaction time during laser brazing and lack of dynamic response, the carbides of Cr_7C_3 and $Cr_{23}C_6$ don't have much time to form. As a result, only Cr_3C_2 was created in this study [4 – 7].

4. CONCLUSIONS

Based on this experimental study, the following was concluded:

1. The Ni-Cr alloy in the laser brazing process showed good soakage behaviour to diamond particles and the steel substrates. In the interfacial reaction region of the diamond particles and the filler metal, the chromium in the Ni-based alloy segregated preferentially to the surfaces of the diamonds and Cr_3C_2 was formed, through which strong bonds were accomplished between the filler metal and diamond particles.
2. In the interfacial reaction region of the steel substrate and the brazing alloy, the element diffusion effect also took place. Hence, the substrate and the filler bonded well. With the bridge effect of the Ni-Cr filler as the active brazing alloy, the abrasive diamond particles and the steel substrate formed a tight bond, which met the requirements for heavy load grinding.

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