Improvement of bonding force between abrasive grains and matrix in Cu/diamond composite fabricated by centrifugal mixed-powder method

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Abstract

As drilling process for carbon fiber reinforced plastic (CFRP), gyro-driving drilling machine has been proposed. Recently, Cu-based diamond grinding wheels for this drilling machine have been fabricated by using centrifugal mixed-powder method (CMPM). However, because diamond abrasive grains in this grinding wheel are dispersed into pure Cu matrix, drop out of the diamond grains occurs due to low wettability between Cu and diamond. In this study, improvement of bonding force between Cu matrix and diamond grains in Cu/diamond composite fabricated by the CMPM is investigated. As improvement method, Ti particles are added into mixed-powder used for the CMPM. This addition of Ti particles forms thin TiC layer at interface between the Cu matrix and the diamond grain. Thickness of the TiC layer increases as the amount of Ti particles in the mixed-powder increases. As the results, the bonding force between the Cu matrix and diamond grains is improved by the addition of Ti particles. The bonding force is increased as Ti concentration in the mixed-powder increases up to 2 mass%. When the Ti concentration exceeds 2 mass%, the bonding force is saturated. Also, the Cu/diamond composite containing Ti makes larger worn groove on counter disc used for wear test comparing with the composite without Ti. Therefore, the addition of Ti particles into the mixed-powder can be expected to enhance grinding ability of Cu-based diamond grinding wheel fabricated by the CMPM.

Key words : Composite, Centrifugal mixed-powder method (CMPM), Bonding force, Bonding test, Cu, Diamond, Wear property

1. Introduction

Carbon fiber reinforced plastic (CFRP) is well known as attracted structural materials because the CFRP has low density and high strength (Schulte and Baron, 1989). However, the CFRP has problem for the application of industrial product. One of the problems of the CFRP is difficulty of mechanical machining due to its too high strength. Therefore, machining process for the CFRP has been investigated to overcome this problem (Bhatnagar et al., 1995).

As one of important machining process for the CFRP, drilling process has been reported (Malhotra, 1990; Lin and Chen, 1996; Chen, 1997). Malhotra (1990) has investigated effects of drilling conditions on tool wear and quality of a hole drilled on CFRP. In that study, drilling process for the CFRP has some problems such as burr and delamination. As well as report by Malhotra (1990), Lin and Chen (1996) have also reported delamination and fiber pullout as typical problems of drilling the CFRP. Because of these, precision drilling process for the CFRP is nowadays demanded.

Recently, as novel drilling machine for the CFRP, gyro-driving drilling machine has been proposed by Takekoshi (2011). Figure 1 shows a schematic illustration of this drilling machine (Watanabe et al., 2011). The gyro-driving drilling machine makes a hole by gyro-movement of grinding wheel. According to the previous studies (Takekoshi, 2011; Watanabe et al., 2011), it has been reported that this drilling machine can make a hole for CFRP without burr and delamination. In our previous study (Watanabe et al., 2011), Cu-based and Al-based diamond grinding wheels for the...
gyro-driving drilling machine have been fabricated by centrifugal mixed-powder method (CMPM). The CMPM is centrifugal casting combined with powder metallurgy (Watanabe et al., 2009). Figure 2 is a set of schematic illustrations showing fabrication process of the metal-based grinding wheel by the CMPM. At first, a mixed-powder of abrasive grains and metal matrix particles is prepared and is subsequently inserted into a spinning mold (Fig. 2(a)). Then, the molten metal matrix is made by melting furnace of centrifugal machine, and is subsequently poured into the spinning mold with the mixed-powder (Fig. 2(b)). At that time, the molten metal matrix is impregnated into the mixed-powder, and the metal matrix particles are melted by heat of the molten metal matrix. Finally, the metal-based grinding wheel can be obtained (Fig. 2(c)). Watanabe et al. (2011) have successfully fabricated some grinding wheels by the CMPM and have suggested the application for the gyro-driving drilling machine. Furthermore, Watanabe et al. (2015) have developed Al/c-BN grinding wheel by the CMPM, and they have succeeded drilling for the CFRP without burr and delamination. In addition, Watanabe et al. (2013) have developed centrifugal sintered-casting, which is CMPM combined with sintering process, and have successfully fabricated Al-based diamond grinding wheel. Also, Kunimine et al. (2015) have prepared Cu-based diamond grinding wheel by the centrifugal sintered-casting, and they could make holes for the CFRP. Therefore, the CMPM is effective technique to fabricate the grinding wheel.

Considering the strength and the thermal conductivity of the grinding wheel, Cu is more suitable rather than Al as its matrix metal. However, wettability between molten pure Cu and diamond is low (Mortimer and Nicholas, 1970). Mortimer and Nicholas (1970, 1973) have reported that the addition of carbide element into molten Cu can improve the wettability between Cu and carbon. On the other hand, Scott et al. (1975) have investigated effects of Ti or Cr concentration in Cu alloy on the wettability and the bonding force of Cu with diamond. According to that study (Scott et al., 1975), the addition of Ti or Cr in Cu alloy can improve the wettability and the bonding force. The similar phenomenon has been reported for the wettability between Cu and graphite by Mao et al. (2015). Hence, it can be expected that the addition of Ti particles into the mixed-powder of Cu particles and diamond grains improves bonding force between a diamond grain and Cu matrix. However, effects of Ti addition into the mixed-powder on microstructure and Cu/diamond bonding force of the grinding wheel by the CMPM are unclear. Especially, report...
concerning with evaluation of the bonding force is limited.

In this study, improvement of the bonding force between Cu matrix and diamond grains in Cu/diamond composite by the CMPM is attempted by the addition of Ti particles into the mixed-powder. To evaluate this bonding force, bonding tests are carried out by using shear-test machine. As will be shown later, it is found that the Ti addition into the mixed-powder can improve the bonding force between Cu matrix and a diamond grain, and that grinding ability of the Cu/diamond composite is also improved by the increase of this bonding force.

2. Experimental procedure
2.1 Preparation of Cu/diamond composites by CMPM

Mixed-powders with different Ti concentrations were made from pure Cu particles, diamond grains and Ti particles. Sizes of the pure Cu particles, the diamond grains and the Ti particles are 25 μm, 150-170 μm and 45 μm, respectively. These particles were mixed by using Turbula mixer. Compositions of the prepared mixed-powders are shown in Table 1.

CMPM to fabricate the Cu/diamond composites was performed by using vacuum centrifugal casting machine shown in Fig. 3 (a). The mixed-powder was inserted into mold, and then the mold with the mixed-powder was set to the vacuum centrifugal casting machine. After that, pure Cu ingot with 80 g in weight was melted at 1200°C using the induction furnace of the casting machine, and subsequently the centrifugal force of 80 G (G is ratio of centrifugal force to gravity) was induced to the mold and the crucible. At this time, the molten pure Cu is poured into the mold, and the pure Cu particles in the mixed-powder are melted by this molten Cu. Finally, sample of Cu/diamond composite was obtained as shown in Fig. 3 (b). The obtained sample has dispersed part of diamond grains to centrifugal direction. As shown in Table 1, four kinds of the samples with different Ti concentrations were prepared under the same casting condition.

Table 1 Compositions of mixed-powder and casting conditions.

<table>
<thead>
<tr>
<th>Sample name</th>
<th>Composition of mixed-powder Cu particles</th>
<th>Ti particles</th>
<th>Ti concentration for Cu particles</th>
<th>Diamond grains</th>
<th>Cu ingot</th>
<th>Temperature of melting furnace</th>
<th>Centrifugal force</th>
</tr>
</thead>
<tbody>
<tr>
<td>CuD</td>
<td>1.32 g</td>
<td>-</td>
<td>-</td>
<td>0.18 g</td>
<td>80 g</td>
<td>1200 °C</td>
<td>80 G</td>
</tr>
<tr>
<td>CuD-1Ti</td>
<td>1.31 g</td>
<td>0.01 g</td>
<td>Cu-1%Ti</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>CuD-2Ti</td>
<td>1.29 g</td>
<td>0.03 g</td>
<td>Cu-2%Ti</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>CuD-4Ti</td>
<td>1.27 g</td>
<td>0.05 g</td>
<td>Cu-3%Ti</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

2.2 Microstructural observation, bonding tests and wear tests for Cu/diamond composites

To make microstructural observation and bonding tests, the dispersed part of diamond grains was cut from each
sample. The microstructural observations were carried out on the dispersed part using optical microscopy (OM) and scanning electron microscopy (SEM). Moreover, using energy dispersive X-ray spectroscopy (EDX), distributions of carbon and Ti in the dispersed part were investigated.

In order to measure the bonding force between Cu matrix and a diamond grain in the Cu/diamond composites, the diamond grains were exposed from Cu matrix by electro-polishing in electrolyte of nitric acid and ethanol (volume ratio is HNO₃ : C₂H₅OH = 1 : 4). Voltage, temperature and duration were 10 V, 0 °C and 25 min, respectively. Bonding force between Cu matrix and a diamond grain was measured using shear-test machine. A schematic illustration showing principle of the bonding test is presented in Fig. 4. Hard steel needle of the shear-test machine moves toward a diamond grain. After that, the needle induces shear force and removes the diamond grain from Cu matrix. In this study, the shear force to remove a diamond grain from Cu matrix is measured as the bonding force. Movement speed of the hard steel needle was 0.1 mm/s and number of tested diamond grains was 100.

Wear tests were performed for CuD and CuD-2Ti samples using pin-on-disc type wear machine. Counter disc for the wear tests was SKD11. Sliding speed, sliding distance and load for the wear tests were 4.7 m/s, 6420 m and 5.39 N, respectively. Wear resistance is usually evaluated by weight or volume loss of specimen. However, weight loss of the samples in this study was too small. Because of this, wear resistance of the samples was evaluated by measuring cross-sectional area of wear grooves formed on the counter disc.

3. Results and discussion

3.1 Microstructure of Cu/diamond composites fabricated by CMPM

Figures 5 (a), (b), (c) and (d) are SEM photographs showing microstructures of CuD, CuD-1Ti, CuD-2Ti and CuD-4Ti samples, respectively. In each figure, left-hand photo is secondary electron (SE) image and right-hand image is backscatter electron compositional (BEC) image. As can be seen in Fig. 5, diamond grains are homogeneously distributed in the dispersed part of all samples. Some dimples are observed in Cu matrix of the CuD sample while the samples containing Ti have no dimple. From this result, it is seen that the drop out of diamond grains is prevented by Ti addition. Moreover, it is observed from BEC images that secondary phases, which are observed with gray contrast, are formed at interface between Cu matrix and diamond grain in the samples containing Ti. As will be mentioned later, the secondary phase is TiC layer generated by reaction of Ti particles and diamond grains.

To investigate the details of the secondary phase formed at the interface between Cu matrix and diamond grain, SE and BEC images of an exposed diamond grain in the CuD-1Ti sample are shown in Figs. 6 (a) and (b), respectively. As well as Fig. 5, the secondary phase is observed on surface of diamond grain. Furthermore, in order to make the composition of the secondary phase clear, compositional analysis for square region surrounded by broken line in Fig. 6 (a) is performed using EDX. Figures 7 (a), (b) and (c) are SE image, carbon and Ti images on diamond grain surrounded by broken line in Fig. 6 (a), respectively. Thin layer is formed on surface of a diamond grain. Moreover, it is seen from composition analysis shown in Figs. 7 (b) and (c) that this thin layer is TiC. Hence, it is found that Ti addition into mixed-powder forms TiC layer at interface between Cu matrix and diamond grain.

Mortimer et al. (1973) have investigated the wetting behavior of Cu-Ti alloy on graphite plate or cylinder. In that study, they have reported that Cu-Ti alloy wet the graphite plate and forms TiC layer at interface between Cu-Ti alloy and graphite plate. In addition, they have found that the thickness of the TiC layer is increased with increasing Ti
concentration in the Cu-Ti alloy. Also, Mao et al. (2015) have investigated about the wettability of Cu-Ti alloy on graphite plate by melting Cu-Ti alloy on graphite plate, and they have mentioned the formation of TiC layer at interface between Cu-Ti alloy and graphite plate. Therefore, the formation behavior of TiC layer around diamond grain in this study is in good agreement with the results of those previous studies.

In our previous studies (El-Hadad et al., 2011; Yamauchi et al., 2015), Al matrix composites containing Al₃Ti particles have been fabricated by reaction CMPM. In this fabrication process, mixed-powder of Al and Ti particles is prepared and then molten Al is poured into the spinning mold with this mixed-powder. At that time, Al particles in the mixed-powder are melted by heat of the molten Al, and the Al₃Ti particles are formed by reaction of the molten Al and Ti particles. Namely, if the mixed-powder consists of reactive element powders, secondary phase is formed by pouring the molten matrix into the mold. Hence, it is natural that formation of TiC layer at interface between Cu matrix and diamond grain is observed in this study. However, Ti in the present samples forms only TiC layer without the formation of the other phase such as Cu₃Ti. It is considered that this comes from large carbide formation tendency of Ti. In some previous studies (Kaneko et al., 1963; Kuniya et al., 1985; Liu et al., 1989), carbide formation tendency of solute elements in molten steels or molten Cu has been reported. According to those studies, carbide formation tendency of Ti is larger than that of Cu. Because of this, Ti can form carbide preferentially rather than Cu. Therefore, the present samples containing Ti has only TiC layer around diamond grain.

Fig. 5 SEM photographs showing microstructures of (a) CuD, (b) CuD-1Ti, (c) CuD-2Ti and (d) CuD-4Ti samples. In each photograph, left and right photos are SE image and BEC image, respectively.
3.2 Effects of Ti addition into the mixed-powder on bonding force between Cu and a diamond grain

Figure 8 is a set of typical load-displacement curves obtained by bonding tests. In the bonding tests for the Cu/diamond composites, two kinds of load-displacement curves are obtained. One type is, as shown in Fig. 8 (a), the curve which load is immediately decreased after maximum load. Conversely, in the other curve shown in Fig. 8 (b), the load is slowly and unstably decreased after the maximum load. Hereafter, it is denoted shear behaviors in Figs. 8 (a) and (b) as mode A and mode B, respectively. Usually, maximum load of mode B is much higher than that of mode A. Although micrograph is not shown here, fragmentation of a diamond grain occurs under shear deformation of mode B while bonding test with behavior of mode A induces the debonding of a diamond grain from Cu matrix without its fragmentation. Because of this, if load-displacement curve presents the behavior of mode A, the maximum load corresponds to bonding force between Cu matrix and diamond grain. Meanwhile, in case of the behavior of mode B, this means that this test measures strength of diamond grain. Hence, the maximum load obtained under the behavior of mode A is used as the bonding force between Cu matrix and diamond grain.

Distributions of the bonding force between Cu matrix and diamond grain are shown in Fig. 9. Figures 9 (a), (b), (c)
and (d) are the distributions of the bonding force of CuD, CuD-1Ti, CuD-2Ti and CuD-4Ti samples, respectively. Since embedded depth of diamond grains into Cu matrix is not constant, the bonding forces are broadly distributed for all of the samples. Maximum peak of the distribution moves toward to higher load as the amount of Ti particles increases. Thus, Ti addition into the mixed-powder can improve the bonding force between Cu matrix and diamond grain. As mentioned in session 3.1, samples containing Ti have TiC layer at the interface between Cu matrix and diamond grain. Therefore, it is natural to be concluded that the bonding force between Cu matrix and diamond grain is improved by formation of TiC layer due to Ti addition into the mixed-powder.

Figure 10 shows average bonding force between Cu matrix and a diamond grain as a function of Ti concentration for Cu particles in the mixed-powder. As seen in Fig. 10, the average bonding force is increased as the Ti concentration increases up to 2 mass%. Kuniya et al. (1985) have investigated the bonding strength between carbon fiber and Cu-Ti alloy. In that study, it has been reported that the bonding strength between the carbon fiber and the Cu-Ti alloy is increased as Ti concentration in the Cu-Ti alloy increases. The result obtained in this study is agreed with the report by Kuniya et al. However, when the Ti concentration exceeds more than 2 mass%, the average bonding force is saturated. As already mentioned above, Ti addition improves the bonding force between Cu matrix and a diamond grain by
formation of TiC layer at Cu/diamond interface. Thus, once the diamond grain is surrounded by TiC layer, the bonding force would be constant. This is the reason why the average bonding force is saturated at more than 2 mass%Ti. From these results, it is found that suitable Ti concentration in the mixed-powder for the Cu/diamond composite fabricated by the CMPM is 2 mass%.

### 3.3 Effects of Ti addition into the mixed-powder on wear properties of Cu/diamond composites

Increase of the bonding force between Cu matrix and diamond grain would improve wear properties of the Cu/diamond composites. To investigate effects of Ti addition into the mixed-powder on wear properties of the composites, wear tests are made for CuD and CuD-2Ti samples. Average frictional coefficients of the CuD and the CuD-2Ti samples are 0.29 and 0.39, respectively. The frictional coefficient of the CuD-2Ti sample is larger than that of CuD sample. This difference in the frictional coefficient is caused by the drop out of diamond grains during wear tests. Since the CuD sample has no Ti, diamond grains would be easily debonded from Cu matrix during wear test. As a result, because worn surface of the CuD sample becomes smooth, frictional coefficient becomes low. Conversely, since the CuD-2Ti sample can keep diamond grains on worn surface without debonding, these diamond grains would increase frictional coefficient. Hence, this result is one of prove that Ti addition into the mixed-powder improves bonding force between Cu matrix and diamond grain.

Weight losses by wear tests for CuD and CuD-2Ti samples are 0.002 g and 0.003 g, respectively. Since these values are too small, the weight losses of the both samples are almost same. In addition to the weight loss, cross-sectional area of worn groove on counter disc formed by wear test is measured to evaluate grinding ability of the Cu/diamond composites. The cross-sectional area of the worn groove was calculated by making its cross-sectional profile using digital microscope as shown in Fig. 11. Cross-sectional areas of worn groove formed on counter disc by wear tests for the CuD and the CuD-2Ti samples are 0.026 mm² and 0.036 mm², respectively. This means that the CuD-2Ti sample makes larger worn groove on counter disc than CuD sample. In the other word, under the same weight loss by the wear test, CuD-2Ti sample can grind the counter disc more effectively rather than CuD sample. From these results, it can be expected that Cu/diamond composite containing Ti have better ability as grinding wheel.

![Cross-sectional profile of worn groove](image)

**Fig. 11** Measurement method of cross-sectional area of worn groove on counter disc. Left photo shows worn counter disc and right graph is cross-sectional profile of worn groove. Hatched region in the right graph corresponds to cross-section of worn groove.

### 4. Conclusions

Improvement of bonding force between Cu matrix and diamond grains in Cu/diamond composites fabricated by centrifugal mixed-powder method (CMPM) was investigated. As a method to improve the bonding force, Ti particles are added into the mixed-powder used for the CMPM. Main results are as follows. 
(1) Ti addition into the mixed-powder forms thin TiC layer at interface between Cu matrix and a diamond grain. Thickness of the TiC layer is increased as Ti concentration in the matrix increases.
(2) Bonding forces between Cu matrix and diamond grain were evaluated by shear test. The bonding force is increased with Ti concentration in Cu matrix increases up to 2 mass%. As exceeding Ti concentration of 2 mass%, the bonding force is saturated. This is because that once the diamond grain is surrounded by TiC layer, the bonding force would be constant.

(3) Frictional coefficient of the CuD-2Ti sample containing Ti is larger than that of CuD sample. This is because that the drop out of diamond grains is prevented by improving the bonding force between Cu matrix and diamond grain. Moreover, the CuD-2Ti makes larger worn groove on counter disc comparing with CuD sample. Therefore, it can be expected that Cu/diamond composite containing Ti have better ability as grinding wheel.

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