Characterization Of Diamond-Dispersed Cu-Matrix Composite

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Dispersion of diamond in metal matrices is expected to improve friction and wear properties. In this paper, cluster diamonds (average grain size: 5nm) are dispersed in pure copper matrix by a P/M method. A mechanical milling method is employed for the composite powder mixing. First, the change in morphology after milling and sintering is observed by SEM. Next, the influence of milling time and cluster diamond content on the friction coefficient and the specific wear rate of the composite are examined. The friction coefficient measured by a ring-on-disk method decreases with the increase in both milling time and diamond content. About 30\% reduction of friction coefficient compared with the pure copper is achieved for the composite (diamond content: 1vol\%) after a milling time of 40 hours. The specific wear rate also decreases with the milling time. However, the rate increases significantly as the diamond content increases. The experimental results show the possibility of a solid lubricant of diamond-dispersed metal matrix composites.

\textit{Keywords: Cluster Diamond, Mechanical Milling, Solid Lubricant, Metal Matrix Composite}

I. Introduction

The cluster diamond (CD) produced by a detonation method is composed of hundreds of carbon atoms, and the CD has the following characteristics\textsuperscript{(1),(2)}:
(1) ultrafine particle (5nm in average),
(2) almost spherical shape,
(3) excellent abrasive,
(4) excellent lubricant,
(5) single crystal.

Several researches have been done to exploit these unique properties of the CD. For example, practical applications such as abrasives in a resin, surface coating of tools, lubricants bonded by a wax, etc. have been studied so far. However great success has not been achieved because of the difficulties of processing technology.

To utilize the CD powder as solid materials, the powder should be consolidated. Casing and powder metallurgy (P/M) are considered as a effective consolidation method. However, the P/M method is superior to casting from the viewpoint of processing temperature. Low process temperature is important for the processing technology of the CD powder, which has a low burning temperature (535°C).

One of the promising applications of the CD powder is as solid lubricant. Recently, a very low friction coefficient of a CD dispersed metal matrix composite (MMC) was reported, and the possibility to realize the ultra-solid lubrication where the friction coefficient should be smaller than 0.01 has been discussed based on the obtained result from the MMC\textsuperscript{(3)}. The matrix of the MMC was aluminium. However, in general, copper alloy matrices show smaller friction coefficient than Al matrix.

In this paper, a solid lubricant MMC by P/M method is studied. The CD is dispersed into the pure copper matrix. Ultrafine powder particles like CD exist as agglomerated secondary particles (Fig.1). To mix the ultrafine powders with matrix powders uniformly against the cohesive power is very difficult by conventional mechanical mixing methods. A mechanical milling (MM) method usually used for mechanical alloying is applied because of the entrapping effect. First of all, the surface characteristics of
the MMC is investigated by changing the MM time and the CD content. Next the flow characteristics of the MMC is measured. And, finally the friction properties are discussed.

II. Materials and experimental procedures

The specific surface area of the CD and the copper powders were 300–390m²g⁻¹ and 0.21m²g⁻¹ respectively. The copper powder was prepared by the gas atomized method, and has an average particle size of 6.63 μm. The composition of the powder is listed in Table 1.

The CD and the copper powders are mixed by steel balls in a container filled with Argon gas. The diameter of the steel ball was 10mm. The rotating speed of the container was 500rpm.

After milling, the mixed powder was consolidated by hot pressing (pressure: 0.8GPa, temperature: 450°C). The dimension of the consolidated MMC disk was 25 × 5mm.

Three point bending test was carried out to obtain stress-strain curves. The specimens for the bending test was produced by slicing the sintered MMC. The specimen was 0.3mm in thickness, 3mm in width and 20mm in length. The distance between supporting points of the bending test was 7mm. The punch was pushed down from 0 to 3mm at the speed of 0.5mm/s.

The friction coefficient and the specific wear rate were obtained by a ring-on-disk method in atmosphere. A ring made of W1-8 (ASTM) had a length of 10mm and a inside and a outside diameters of 14 and 18mm respectively. The surface roughness of the ring and the disk was finished between Rmax0.2 and Rmax0.3 μm. The disk was driven at the rotating speed of 240rpm while the ring was fixed. The pressure of 98N or 9.8N was applied to the disk through the ring. The specific wear rate was calculated using the equation, V/(P·L), where P, L and V stand for applied pressure, sliding distance and removed volume respectively. In this experiment, the L=120m was selected.

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III. Result and Discussion

1. Surface Characteristics

1-1 SEM Observation
(a) Mixed Powder
The particle shape and the surface condition of the mixed powder of copper and CD were observed with changing milling time (0–60h) and CD content (0–10vol%). SEM micrographs are shown in Fig.2.

Fig.2(a) shows the mixed powder particles with different milling time. As shown in (i), the particle shape of the original copper powder is almost spherical, and the surface is smooth. In 10 hours, deformation of the spherical shape occurred; uneven and coarser surfaces and small fractured pieces are observed (ii). After that, the particle shape became smaller and the copper surface coarser with time. This is due to fracture during the milling process. However, in 40 hours, the particle size becomes slightly bigger than before, and the surface smoother. This might be caused by rejoining at elevated temperature. Between 40 and 60 hours of milling time, outstanding changes are not observed except some flattening tendency of the particle.

Fig.2(b) shows micrographs of the mixed powder particles obtained by 40-hour milling. The CD content is varied from 0 to 10vol%. The surface roughness becomes coarser as the CD content increases. This is because of the increase in the amount of entrapped CD on the surface of the Cu particles which prevented the deformation and the rejoining of the fractured particles. As the result, the particle size becomes smaller.

The change in the average particle size is shown in Fig.3. The size decreases with the milling time and then begin to increase slightly after 10hours. For the higher CD contents, the smaller size was obtained.
(b) Sintered MMC
The CD dispersed MMC is produced by hot pressing of the mixed powders. The electrolytically polished surface of the MMC was observed by SEM. The microstructures of the MMC with different milling time and content are shown in Fig.4. The bright spot in the micrograph indicates the place of CD densified area. As for the bright spot, as the number increases, the size becomes smaller, and the distribution becomes more uniform with the increase in milling time. This means that the mechanical milling method is effective and that longer milling time is better.
Fig. 2 SEM micrographs of mixed powder with different MM time and CD content.

1-2. Residual Stress

The residual stress of the powder was measured by the Hall method and sintered MMC by sin²ψ method using X-ray. The residual stress in the Cu and CD composite particle increased as the CD content and the milling time increased. However, the difference caused by these factors almost disappeared after sintering. For example, the residual stress of the sintered MMCs with different milling time had little correlation with the particle residual stress, and distributed in between -110—130MPa. The generation of the compressive stress could be attributed to the production method of the MMC and also to the preparation method of the specimen.

2. Hardness

The Vickers Hardness was measured by keeping the load at 300g for 25s. The hardness change for the 1vol% MMCs with different milling time is shown in Fig.5. The hardness rises rapidly up to 10 hours of milling time, after that, the hardness rises slowly. Strain hardening
introduced by the plastic deformation of Cu particle and CD dispersion resulted in the increase in the hardness of the MMC. The slowdown in increase of hardness after 10 hours is due to the decrease in the plastic deformation of the Cu particle.

Next, the influence of the CD content on the hardness was also examined. As shown in Fig.6, the hardness increases linearly up to HV170. The dispersion of the hardest material, the CD, has the effect of elevating the hardness. Small scattering of the measured hardness together with the microstructures shown in the Fig.4 indicate the uniform dispersion of CD into the Cu matrix.

3. Stress-Strain Curve

Stress-strain curves for the MMCs with different CD contents are shown in Fig.7. There is little difference of the flow stress among these curves. However, the elongation differs significantly depending on the CD content. The higher CD content causes lower elongation.
Fig. 7 Bending stress-strain curves for various CD contents. (MM time: 40h)

For the MMCs with 0 and 1 vol% of CD, bigger strain than 0.07 is possible. For 2, 5, and 10 vol% of CD, the elongations were 0.035, 0.008 and 0.004 respectively.

For the MMCs with high CD content, the decrease in elongation is also observed with the increase in milling time of the mixed powder.

4. Friction and Wear Properties

The friction coefficient of the MMC with different milling time was measured. The obtained results are shown in Fig. 8. The symbols of ○ and □ stand for the coefficient for the different loads 98N and 9.8N respectively. The material without milling is the sintered pure copper. The increase in milling time lowers the friction coefficient, which is independent of the load value. The friction coefficient drops rapidly at the early stage of milling. Little difference of the friction coefficient is observed between 40 and 60 hours. This tendency coincides with those of structural and hardness changes caused by milling time. The friction coefficient is affected strongly by the state of CD dispersion and hardness of the matrix.

As can be seen in Fig. 8, the friction coefficient is strongly affected by the load. In the case of 9.8N, the friction coefficient decreases from 20 to 40% compared with the load 98N. Worn surfaces are shown in Fig. 9. Judging from these micrographs, the main mechanism of the wear is considered to be adhesive wear in the friction layer. The load of 98N causes a thick friction layer where plastic deformation takes place. The reduction of the thickness of the layer is observed for the lighter load and the increase in mechanical milling time. However, after
10 hours of milling time, nearly the same worn trace were observed. The specific wear rate decreased from $5.3 \times 10^{-6}$ to $3.1 \times 10^{-7}$ as the MM time increased from 0 to 10 hours, but the decrease tapers off after 10 hours of milling.

The relation between the CD content and the friction coefficient is shown in Fig. 10. The friction coefficient decreases from 1.1 to 0.85 when the CD content range from 0 to 10 vol%.

The change in specific wear rate as a function of the CD volume fraction is also shown in Fig. 10. The specific wear rate decreases at first and then begins to increase at 1 vol%. Two main causes are considered to explain this phenomena. One is the adhesion and the other is the falling off of the powder particle. The adhesive wear is supposed to decrease monotonically with the increase in CD content. On the contrary, the wear by the falling off continues to increase. These are based on the observation of the powder particles (Fig. 2), and also from the stress-strain curve (Fig. 7). Owing to these opposite tendencies, the curve of specific wear rate in Fig. 10 are obtained.

Worn surfaces with 10vol%CD content are shown in Fig. 11. Several traces of the adhesive wear were observed for the MMC with 1vol%CD (Fig. 9(b)), while several traces of the falling-off and innumerable minute abrasion traces are observed for the MMC with 10vol%CD. However, the minute abrasions cannot be considered as the main cause of the wear.

The friction properties of the CD-dispersed Cu matrix composite were measured. However, the improvement of the properties were not so significant as expected. To realize the ultra solid lubrication by this kind of MMC, studies on the Cu alloy matrix systems and on the MMCs with extremely high CD content should be necessary along with the equipment for friction test under ultralight load.

### IV. Conclusion

On the basis of the experimental study on the CD dispersed Cu matrix composite, the following conclusions are obtained.

1. Mechanical milling method is effective in achieving uniform dispersion of the ultrafine CD into the matrix powder.

2. The hardness of the MMC increases with the increase in CD content and milling time. However the elongation shows the opposite tendency.

3. Compressive residual stress was observed on the surface of the MMCs produced by P/M method.

4. The longer milling time is desirable from the viewpoint of the friction property. For example, the 14% reduction of the friction coefficient and 90% reduction of the specific wear rate were achieved in 10 hour mechanical milling for the MMC.

5. The increase in the CD content decreases the friction coefficient while the specific wear rate increases. The 10vol%CD addition resulted in 25% reduction of the friction coefficient and increase in the specific wear rate from $2 \times 10^{-7}$ to $9 \times 10^{-6}$.

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