407  Solid State Lubricant Film Dispersed Nano-Cluster Diamond Powder

Takahisa Yamazaki* and Akio Suzumura**

*Technology Research Department, National Space Development Agency of Japan (NASDA)
Tsukuba Space Center, 2-1-1 Sengen, Tsukuba, Ibaraki 305-8505, Japan,
yamazaki.takahisa@nasda.go.jp
**Graduate School of Science and Engineering, Tokyo Institute of Technology
2-12-1, Ookayama, Meguro-ku, Tokyo 152-8552 Japan

Abstract: For space mechanical components, the characteristics of silver thin film dispersed nano-cluster diamond powder as a solid lubricant film have been investigated. The silver films dispersed the cluster diamond powder were made on the 440C stainless steel disks under vacuum of $3 \times 10^{-3}$ Pa at 1080K without graphitization of the nano-cluster diamond powder. Frictional characteristics of the films were investigated by a pin-on-disk friction test in injected nitrogen gas atmosphere (30%RH, 15% oxygen gas) and under ultra high vacuum of $2 \times 10^{-4}$ Pa. Initial Hertzian contact pressure was set almost 0.7GPa. The sliding speed between the pin and the disk was about 30mm/s. Compared with ion-plated silver film, the lifetime of the film dispersed the nano-cluster diamond powder was 100 times longer in nitrogen gas atmosphere. The initial friction coefficient showed 0.2, and then it showed stable 0.4 for a while. The cluster diamond powder is considered to prevent the wear of the silver film.

Key word: Nano-Cluster, Diamond, Solid Lubricant, Silver, Friction, Plating Film

1. INTRODUCTION

Ion-plated silver film is a solid lubricant under low temperature and ultra high vacuum. Silver film dispersed nano-cluster diamond powder is expected to improve the property of silver film. The addition of the nano-cluster diamond will provide lower friction coefficient and longer wear lifetime for the silver film, because this powder decreases friction coefficient when it is applied to liquid lubricants as an additive [1].

The nano-cluster diamond powder is a round shape. The diameter of it is about 5nm. It is easy to form secondary powder for the nano-cluster powder in ambient atmosphere. It is important to disperse into the silver film avoiding the secondary powder, because the secondary powder has grinding property. The plating of the silver films on 440C stainless steel disks were carried out by a brazing method.

Frictional characteristics of the film were investigated by pin-on-disk friction test in injected nitrogen gas and under ultra high vacuum at room temperature. The worn tracks were observed by an atomic force microscope (AFM) in order to evaluate the role as the additives of the nano-cluster diamond powder to the silver film.

2. EXPERIMENTAL

Nano-cluster diamond powder was supplied after washed in HNO$_3$ and H$_2$SO$_4$ bath at elevated temperature from Tokyo Diamond Tools Mfg. Co., Ltd.. Pure silver powder, the nano-cluster diamond powder and non-oxidized vanadium powder were well mixed in the ratio of 1:1:0.2. The silver powder was passed through 325mesh screen, and the mean diameter of the vanadium powder was 0.075mm. The mixed powder was screen-printed on one sheet of silver-copper eutectic foil supplied from Tokyo Braze Co., Ltd. The thickness of it was 0.05mm.

The 440C stainless steel disks of 2.8mm in thickness were polished up by emery paper. They
had not been heat-treated for surface hardening. They were coated with gold thin film, since the wettability of the 440C stainless steel for silver is not good. The silver-copper foil, on which mixed powder was screen-printed, was mounted on the disk. This specimen was heated at 1060K under vacuum of $3 \times 10^3\text{Pa}$ for 240s, then cooled down soon. This process includes the surface hardening of the disk. The silver-copper eutectic foil starts to melt at 1050K. The reason why the silver-copper foil was used is that the low temperature process prevents the nano-cluster diamond powder from the graphitization at 1173K as reported in reference [2].

Unidirectional sliding friction experiments were conducted at room temperature in injected nitrogen gas (about 15% oxygen gas) and under vacuum of $2 \times 10^3\text{Pa}$ using pin-on-disk friction apparatus. This apparatus measures the friction force occurring between a rotating disk and a static pin at constant sliding velocity. The pin mated with the disk was 440C stainless steel ball. Load and diameters of the balls were set to the initial Hertzian contact pressure of about 0.7GPa. The numbers of laps per minute were 60 (in nitrogen gas) and 120 (under ultra high vacuum). The sliding speeds were mainly 30mm/s (in nitrogen gas) and 60mm/s (under ultra high vacuum). In order to confirm the repeatability of the friction coefficient trace with the number of laps, the friction tests were carried out two times for each disk by using different sliding radii of 5mm and 7mm without changing the mated pin. The slow sliding speed was selected, because the friction force measurement was disturbed by the relatively large roughness of the silver film made by this method. The worn tracks were observed by an AFM after the friction test in order to investigate the effect of the nano-cluster diamond powder.

3. EXPERIMENTAL RESULTS

3.1 Friction Test in Nitrogen Gas

Figure 1 shows the friction coefficient trace with the number of laps for ion-plated silver film in injected nitrogen gas. These pin-on-disk friction tests were carried out using balls of 6mm in diameter and 5N load. The maximum Hertzian pressure was 0.77GPa. Sliding speed was 30mm/s. The load was determined by the pin-on-disk test apparatus specification. The wear lifetime is short within 100 laps as shown in the Fig.1. The initial friction coefficient is 0.450. It increases soon and rises up to 0.7. Figure 2 shows the friction coefficient trace in nitrogen gas with the number of the laps for the silver film dispersed cluster diamond. The initial friction coefficient is 0.2, and soon increases to 0.45. It continues the value of 0.45 until 6000 laps. Then, it increases gradually. It rises up to 0.7 at about 8500 laps. The wear lifetime of the silver film dispersed nano-cluster diamond powder is 100 times longer than that of the ion-plated silver film.

![Fig. 1 Friction coefficient against ion-plated silver film in nitrogen gas atmosphere.](image1)

![Fig. 2 Friction coefficient against silver film dispersed nano-cluster diamond in nitrogen gas.](image2)
Silver film provides good lubricant property under ultra high vacuum, because the film can avoid oxidation. According to a test result obtained in NASA [3], the initial friction coefficient for the ion-plated silver film was 0.2. Then it rose slowly to 0.3 till over $2 \times 10^4$ laps.

The silver film dispersed nano-cluster diamond powder was tested in vacuum chamber under $2 \times 10^9$Pa. The pin-on-disk friction test apparatus in vacuum chamber was different from the one in injected nitrogen gas before mentioned. At first, the test was conducted under the conventional friction test condition of NASA. The wear lifetime of the film was very short as 100 laps. The test condition of high sliding speed and heavy load was not proper to the silver film made by this plating method, because the film was so thick that the contact area of the pin became large and the friction coefficient was near 0.5, and because the pin often scratched the film. Then, 440C stainless balls of 8mm in diameter as a pin and load of 2N were selected for the friction test under ultra high vacuum. The maximum Hertzian contact pressure was calculated to be 0.70GPa. Deformation depth of the silver film was calculated to be 300nm. The sliding speed was 60mm/s. The measurement of the friction force between the pin and the disk was carried out every 1s.

Figure 3 shows the friction coefficient trace with the number of laps. Initially, the friction coefficient shows 0.1. While 300 laps, it shows under 0.2. This effect can be observed every silver film dispersed nano-cluster diamond powder. After 1000 laps, the pin scratches the film often, and wide track is observed. The friction coefficient changes widely, but several times it shows near 0.1. Until 4000 laps, the average friction coefficient is about 0.4. This indicates the nano-cluster diamond provide wear-less property in silver film. After that, the friction coefficient of the film recovers to 0.2 several times and the disk rotates $3 \times 10^4$ laps.

3.3 AFM Observation Results

Figure 4 shows the AFM image of the worn track after the pin-on-disk friction test in injected nitrogen gas. The view image area is 500nm $\times$ 500nm. On the right side of the image shown by an upward arrow, a track edge is observed. The hollow of about 100nm in diameter is observed in the bottom of the figure shown by a downward arrow. The hollow is considered to be the mark where the nano-cluster diamond powder was there. The nano-cluster diamond powder is considered to be dispersed not so well in this plating process, because the area of the mark in the worn track is much larger than the diameter of the nano-cluster diamond. In the middle of the figure, a mountain is observed. A nano-cluster diamonds are considered to be in the mountain. The cluster diamonds are so hard that silver film is plastically deformed every lap of the pin. The void will appear between the diamond and the film. At last, the diamond powder will be pulled out in the film, and the worn track will become rough. The pin scratches the silver film, so the friction coefficient jumps up to over 0.7.

![Pin-on-Disk Test under UHV, 0.7GPa](image)

Fig. 3 Friction coefficient against silver film dispersed nano-cluster diamond under ultra high vacuum.

In the silver film plating process, vanadium powder will work to disperse the nano-cluster diamonds into the silver film, since the hydrogen atoms, which the vanadium powder contains, terminate the dangling bond hands at the diamond surface at elevated temperature and pre-
vent the formation of secondary powder of the nano-cluster diamonds. The vanadium atoms react with carbon atoms at the nano-cluster diamond surfaces. Wettability of the nano-cluster diamond with silver is improved by the formation of the vanadium carbide. This wetting process is expected to disperse well the nano-cluster diamond into the silver film. The AFM observation indicates the dispersion of the nano-cluster diamond into the silver film is not enough for apart to a single piece. Further investigation is need for the problem is that the temperature of the plating process is so low that surface roughness becomes large and the flow of the molten silver metal is not so good.

Fig. 4 AFM observation image of silver film dispersed nano-cluster diamond after friction test in nitrogen gas.

4. CONCLUSIONS

Nano-cluster diamond gives the silver film the low friction coefficient of about 0.1 and long lifetime. The plating process of the silver film dispersed nano-cluster powder like brazing prevents formation of the secondary powder of the nano-cluster diamond. If the suitable thin film process is made, the silver film dispersed nano-cluster diamond is superior to the ion-plated silver film.

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