# Tribological Characteristics of Magnetron Sputtered $MoS_2$ films in Various Atmospheric Conditions

**Seock-Sam Kim**
*Department of Mechanical Engineering, Kyungpook National University, Taegu 702-701, Korea*
**Chan-Wook Ahn, Tae-Hyung Kim***
*Graduate School, Department of Mechanical Engineering, Kyungpook National University, Taegu 702-701, Korea*

The friction and wear behaviors of magnetron sputtered $MoS_2$ films were investigated through the use of a pin and disk type tester. The experiments were performed for two kinds of specimens (ground (Ra 0.5 $\mu$m) and polished (Ra 0.01 $\mu$m) substrates) under the following operating condifions : linear sliding velocities in the range of 22~66 mm/s (3 types), normal loads varying from 9.8~29.4 N (3 types) and atmospheric conditions of air, medium and high vacuum (3 types). Silicon nitride pin was used as the lower specimen and magnetron sputtered $MoS_2$ on bearing steel disk was used as the upper specimen. The results showed that low friction property of the $MoS_2$ films could be identified in high vacuum and the specific wear rate in air was much higher than that in medium and high vacuum due to severe oxidation. It was found that the main wear mechanism in air was oxidation whereas in high vacuum accumulation of plastic flow and adhesion, were the main causes of wear.

**Key Words :** Magnetron Sputtering, $MoS_2$ Film, High Vacuum, Coefficient of Friction, Specific Wear Rate, Adhesion, Transfer Film

## 1. Introduction

Tribosystem is important in space applications as relative motions cause energy dissipation and wear. Therefore, tribosystems in space require withstand high performance, high precision and high endurance. In particular, space applications must withstand pressures ranging from atmosphere to ultra-high vacuum, wide range of temperature from $-100$ ℃ to $+150$℃, thermal cycling, thermal shock and dusty conditions (Voevodin et al., 1999 ; Fusaro, 1995).

In vacuum condition, oil and other liquid lubricants become impractical because of their contamination of components caused by condensed vapors. For these reasons, researches on solid lubricants have increased (Roberts, 1990 ; Kato et al., 1990; Roberts, 1989). Kato et al. (1990) suggested an in-situ tribocoating system that could supply a solid lubricant constantly in space and Miyoshi (1999) reported many problems of tribosystems in space and emphasized the role of solid lubricants in vacuum environment.

Among many kinds of solid lubricants, magnetron sputtered molybdenum disulfide ($MoS_2$) film is Known to be superior to resin bonded $MoS_2$ because of its high load-carrying capacity, ultra-low friction, constant composition and film thickness in vacuum (Suzuki, 1998). Also, it is used in the field of precise instruments such as satellite bearings and it is expected that the future demand of magnetron sputtered $MoS_2$ films will continue to increase in industries. Therefore, the

* Corresponding Author,
**E-mail** : kthcjh@hanmail.net
**TEL** : +82-53-957-4361; **FAX** : +82-53-950-6588
Graduate School, Department of Mechanical Engineering, Kyungpook National University, Taegu 702-701, Korea. (Manuscript **Received** November 14, 2001; **Revised** June 10, 2002)

main purpose of this study is to understand the friction and wear characteristics of magnetron sputtered $MoS_2$ films on bearing steel substrate and to investigate the wear and fracture mechanisms in air, medium and high vacuum.

## 2. Experimental Apparatus and Procedures

The schematic diagram of wear tester in vacuum is shown in Fig. 1. The test machine can be operated in various environments, such as medium vaccum ($10^{-1}$ Pa) by rotary pump and high vacuum ($10^{-4}$ Pa) by rotary and diffusion pump.

Specimens used in this study were magnetron sputtered $MoS_2$ films on AISI 52100 steel (disk) and $Si_3N_4$ (pin) as shown in Fig. 2.

Figure 3 shows the schematic diagram of magnetron sputtering process and the thickness of the $MoS_2$ film used in this experiment was about 1 $\mu$m.

The experimental conditions were as shown in Table 1.

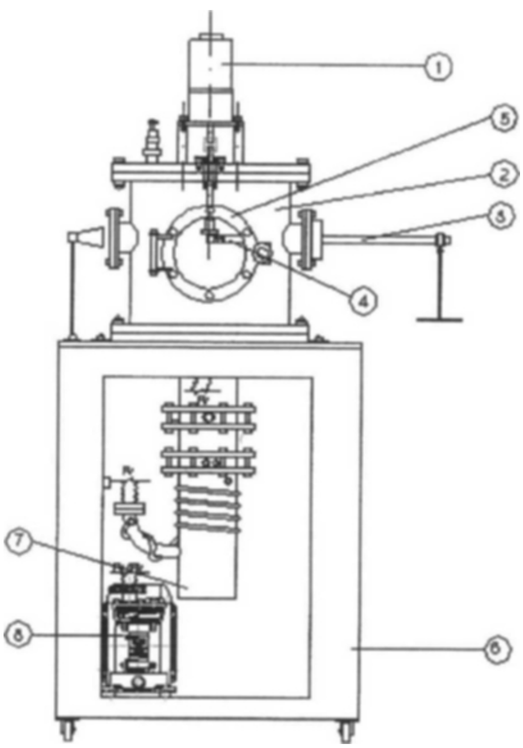


**Fig. 1** Schematic diagram of the testing apparatus ① motor ② vacuum chamber ③ dead weight ④ load cell unit ⑤ door ⑥ frame ⑦ diffusion pump ⑧ rotary pump

**Table 1** Experimental conditions of magnetron sputtered $MoS_2$ film

| Substrate roughness | Grinding (0.5 $\mu$m Ra) | Polishing (0.01 $\mu$m Ra) | |
|---|---|---|---|
| Normal load | 9.8 N | 19.6 N | 29.4 N |
| Environment | 105 Pa | $1.3\times10^{-1}$ Pa | $1.3\times10^{-1}$ Pa |
| Sliding speed | 22 mm/s | 44 mm/s | 66 mm/s |

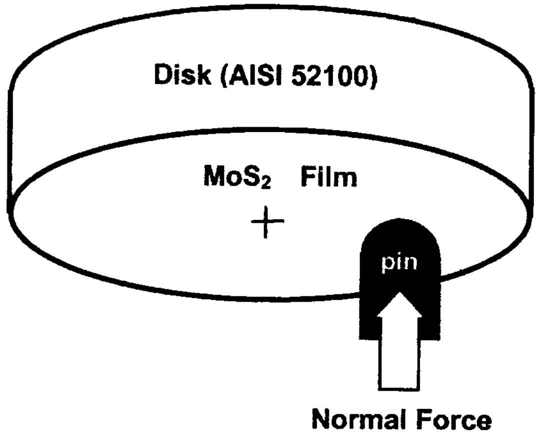


**Fig. 2** Schematic illustration of the contact configuration

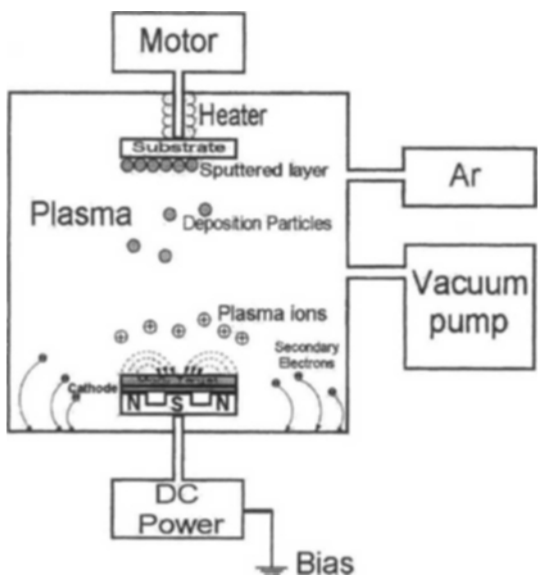


**Fig. 3** Schematic diagram of magnetron sputtering system

## 3. Results and Discussion

### 3.1 Frictional behavior with various substrates of different surface roughness

Figure 4 shows the coefficient of friction with different substrate surface roughness in air. In the initial stage, low coefficient of friction was maintained but the average coefficient of friction increased abruptly and reached about 0.8 after the $MoS_2$ film failed. The low friction characteristic was kept up at the first stage but the friction increased rapidly after 250~500 cycles and the coefficient of friction for the ground substrate was more or less higher than that for the polished substrate. However significant difference in the frictional behavior with respect to the substrate surface roughness could not observed.

It was found that in the case of $MoS_2$ film sputtered on the ground substrates, the gradient of coefficient of friction was less steep than that of polished substrates because the fractured $MoS_2$ debris remained on the rough surfaces for a longer time. This pattern could be observed in medium and high vacuum conditions.

### 3.2 Frictional behavior of magnetron sputtered $MoS_2$ films in various atmospheric conditions

Figures 5 and 6 show the variation of coefficient of friction with respect to the atmospheric condition at the normal load of 29.4 N and the sliding speed of 66 mm/s. Coefficient of friction in medium and high vacuum was remarkably low in comparison with that in air and the critical sliding distance up to which the low-friction property of $MoS_2$ film was maintained was also much longer. In high vacuum, the coefficient of friction of both specimens with ground and polished substrates remained quite low until it increased after 1500~200 cycles owing to the failure of $MoS_2$ film. In medium vacuum, the coefficient of friction was somewhat unsteady and high up to 2000 cycles but showed stable behavior after that. In air, however, after 500 cycles the coefficient of friction increased

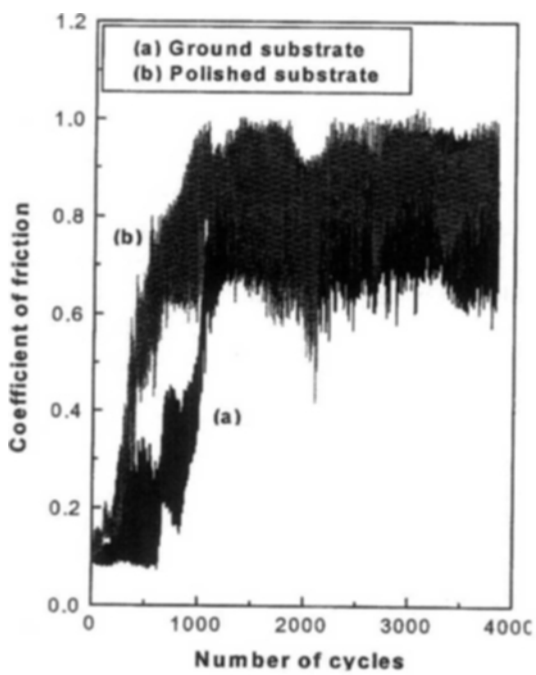


**Fig. 4** Coefficient of friction as a function of the sliding cycles under the load of 29.4 N and the sliding speed of 22 mm/s in air

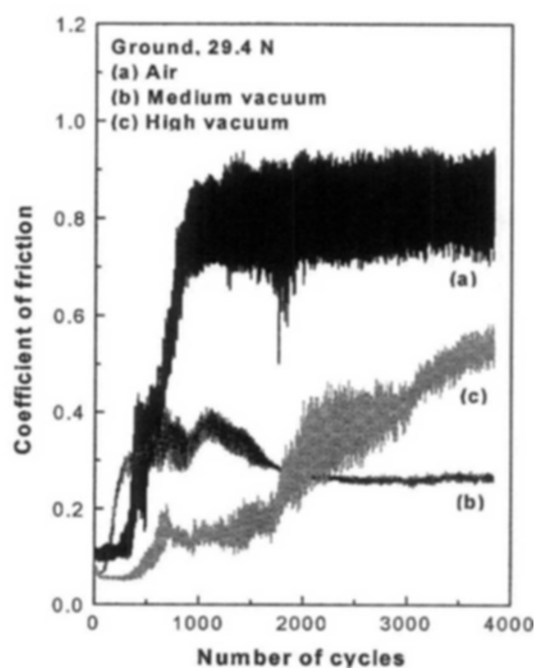


**Fig. 5** Coefficient of friction as a function of the sliding cycles for various atmospheric conditions at the sliding speed of 66 mm/s

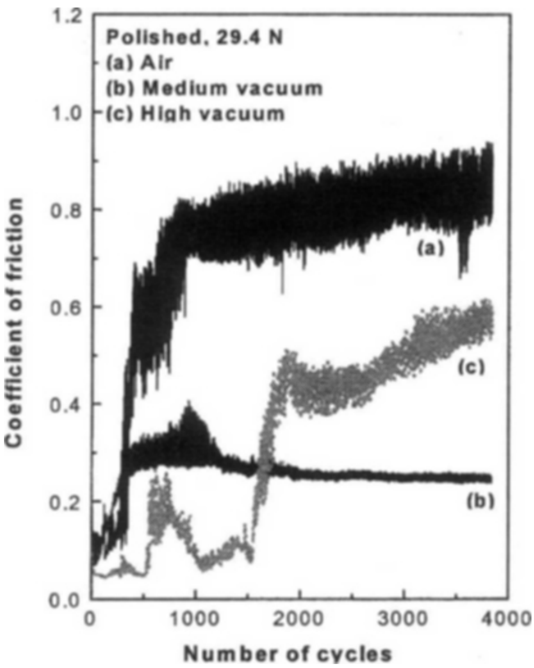


**Fig. 6** Coefficient of friction as a function of the sliding cycles for various atmospheric conditions at the sliding speed of 66 mm/s

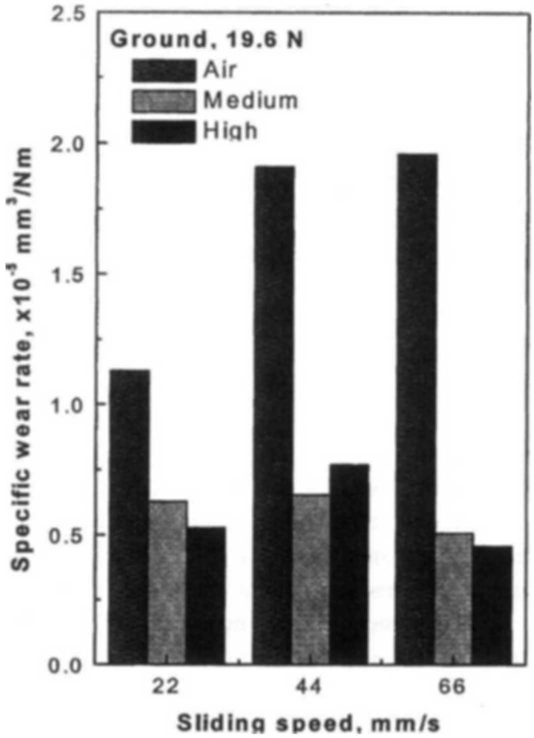


**Fig. 7** Specific wear rate of magnetron sputtered $MoS_2$ for ground substrate after 4000 cycles

steeply and reached about 0.8 after 1000 cycles.

$MoS_2$ film reacts with oxygen and vapor chemically in air which produces $MoO_3$ as follows :

$$2MoS_2+4H_2O+9O_2 \rightarrow 2MoO_3+4H_2SO_4 \quad (1)$$

By this chemical reaction, the nature of $MoS_2$ structure is affecfed significantly and consequently the $MoS_2$ film loses its initial property with low shear strength. For this reason high coefficient of friction is observed in air. On the other hand, the effect of oxygen and vapor does not exist in vacuum condition and the crystal direction of $MoS_2$ molecules is transformed to the sliding direction by shear. Therefore, it was thought that the coefficient of friction in vacuum was distinctively low compared with that in air.

### 3.3 The variation of specific wear rate in various atmospheric conditions

The specific wear rate of $MoS_2$ film was investigated in various atmospheric conditions.

Figure 7 shows the variation of specific wear rate for ground substrate specimens with increasing sliding speed at the normal load of 19.6 N in various experimental conditions after 4000 cycles. It was found that the specific wear rate in vacuum was much smaller than that in air. It was thought that the main reason was the formation of a transfer film due to low shear strength and adhesion of the $MoS_2$ film.

In air, shear resistance increases owing to the oxidation of $MoS_2$ film and the film loses its ability as a solid lubricant, whereas in vacuum $MoS_2$ film is transferred to the counterpart specimen and the transfer film is built up. Hence, it is thought that low friction property can be maintained much longer and the specific wear rate is very low in vacuum because of the transfer film formed on the $Si_3N_4$ pin surface.

Figure 8 shows the variation of the specific wear rate for polished substrate specimens with the increasing sliding speed at the higher normal load of 29.4 N in various experimental environments after 4000 cycles. The specific wear rate in air increased with increasing normal load but in vacuum it showed very low values in spite of the increase in normal load due to the transfer film which could be maintained for a long time. Consequently, the specific wear rate in air increased with increasing normal load but in vacuum increase in the normal load did not affect the specific wear rate because the load bearing capacity and the transfer film were well maintained in vacuum.

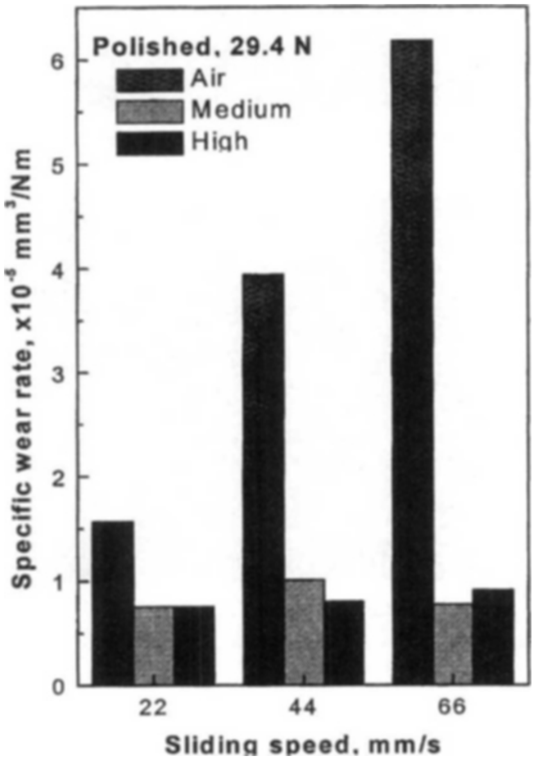


**Fig. 8** Specific wear rate of magnetron sputtered $MoS_2$ for polished substrate after 4000 cycles

### 3.4 Wear mechanisms by microscopic analysis

After the friction and wear test, worn surfaces of $MoS_2$ films were investigated by SEM. Figure 9 shows the worn surface of the disk at the normal load of 29.4 N and the sliding speed of 66 mm/s in air after 4000 cycles. According to Fig. 9, the worn surface was rough because the $MoS_2$ film was oxidized and many oxidative wear particles were scattered on the worn surface. However, in medium and high vacuum the number of oxidative wear particles decreased and worn surface became smooth. In medium vacuum, the middle of the wear track was much smoother than in air, but along the boundary of the contact and the non-contact area oxidative wear particles were observed. It was thought that the coefficient of friction rose to some extent in medium vacuum because of the presence of these wear particles.

The worn surface at the normal load of 19.6 N and the sliding speed of 22 mm/s in high vacuum is shown in Fig. 11. According to Fig. 11, the wear track in high vacuum was very smooth and severe plastic flow was observed along the boundary and in the middle of the wear track adhesion of $MoS_2$ film occurred. Therefore, it is thought that the main wear mechanism in high vacuum is accumulation of plastic flow and adhesion.

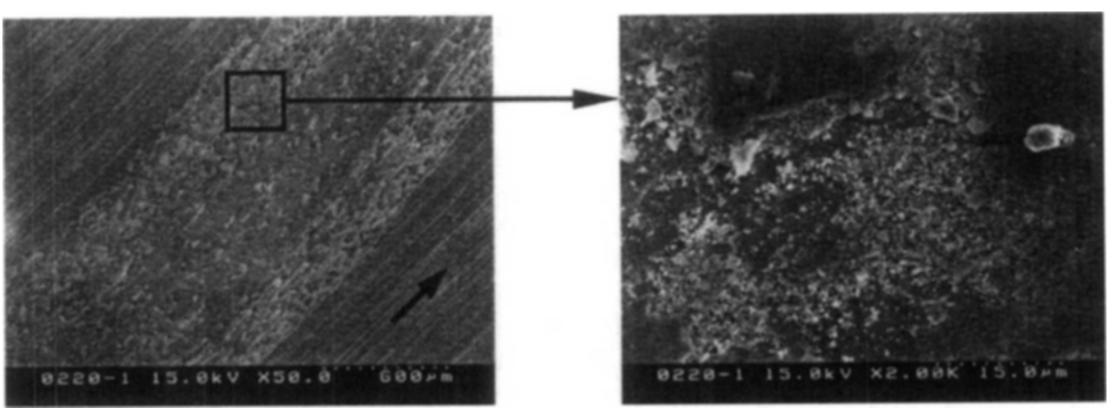

**Fig. 9** Oxidative wear particles on the worn surface in air (ground substrate, 29.4 N, 66 mm/s)

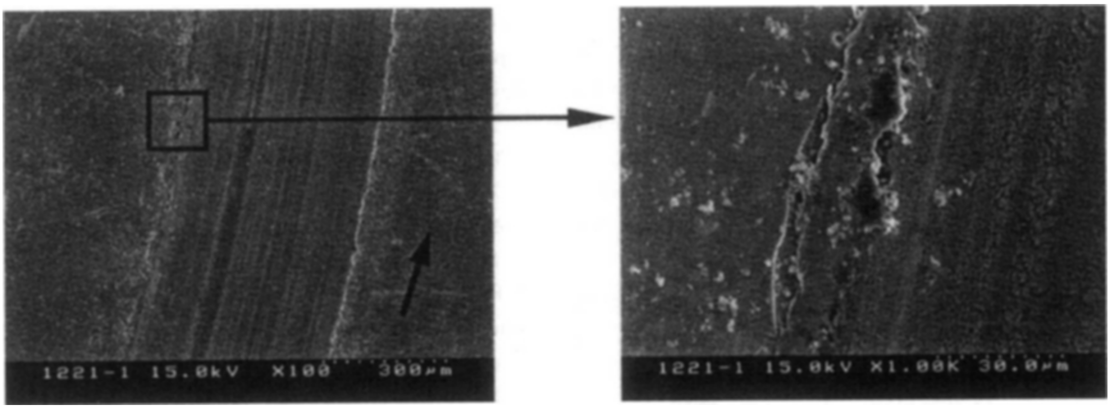


**Fig. 10** SEM photograph of the worn surface in medium vacuum (polished substrate, 29.4 N, 66 mm/s)

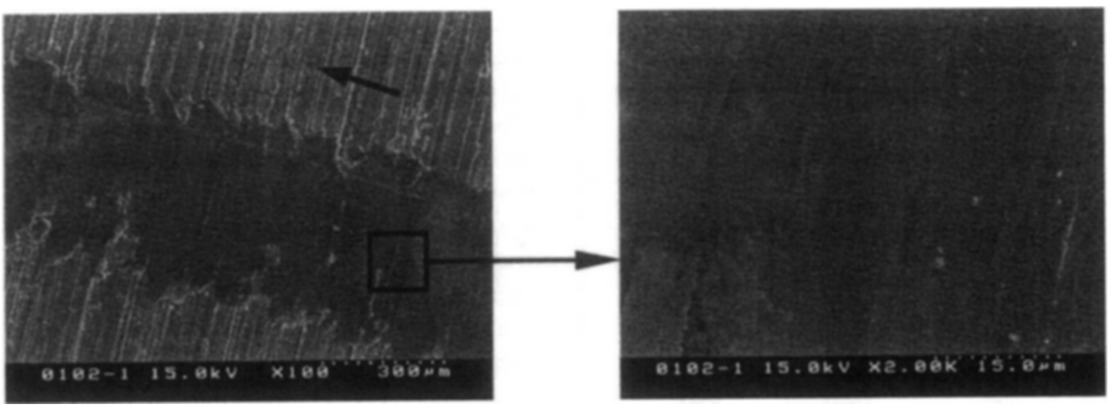


**Fig. 11** Accumulation of plastic flow along the boundaries in high vacuum (ground substrate, 19.6 N, 22 mm/s)

## 4. Conclusions

In this study, the friction and wear behaviors of magnetron sputtered $MoS_2$ films were investigated and compared in air as well as medium and high vacuum conditions.

The main results obtained from the experiments were as follows :

(1) Coefficient of friction of the ground substrate was somewhat higher than that of the polished substrate. However, the variation of the frictional behavior with the change of substrate surface roughness was not clear.

(2) The low-friction property of $MoS_2$ film was most obvious in high vacuum and the $MoS_2$ film could not work well as an effective solid lubricant in air due to severe oxidation. Specific wear rate in air was very high but decreased sharply as the environment was changed to high vacuum owing to the inherent low shear strength of $MoS_2$ film and the formation of a transfer film.

(3) The main wear mechanism of worn surface in air was oxidation wear caused by oxygen and vapor. As the atmospheric condition was changed to high vacuum, the effect of oxidation decreased and accumulation of plastic deformation and adhesion were found to be profound in high vauum.

## Acknowledgment

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