

酸性腐食環境における DLC コーティング材料のトライボロジー特性 Tribological Properties of DLC Coatings in Acidic Corrosive Environment

名大・工（学）*鳳 浩東 名大・工（正）梅原 徳次 名大・工（正）野老山 貴行

Feng Hao dong*, Umehara Noritsugu*, Tokoroyama Takayuki*

*Nagoya University

1. Introduction

Diamond-like carbon (DLC) is an amorphous carbon which contains sp^2 and sp^3 hybridized carbon atoms. Because of its low coefficient of friction (CoF), high hardness, high wear resistance, and chemical stability, DLC is usually used as coatings on the metals. In recent years, with the deepening of research on DLC, DLC has been gradually applied in automobile and medical fields. The goal is to reduce friction and increase efficiency. However, DLC is very sensitive to the environment it is in, and the tribological characteristics are different depending on the friction environments. Acidic environment is a common environment in industrial production. Nitric acid is a highly corrosive mineral acid, which is widely used in fertilizer industries. However, mechanical components in the production process may subject to severe corrosion due to its corrosive properties. To promote the further application of DLC in acidic corrosive environment, detailed research is indispensable.

According to the research of Nagai T. [1], DLC was coated on the surface of stainless steel and the corrosion resistance of DLC against hydrochloric acid, nitric acid, and sulfuric acid was evaluated. The results suggested that the DLC coating could effectively improve the corrosion resistance of metallic materials to acid solutions. In the study of Wu [2], dielectric barrier discharge was used to modify the surface of DLC and lower the CoF of DLC. In the process of dielectric barrier discharge, a liquid film was generated which contained a certain amount of nitric acid. The effect of nitric acid on the surface of DLC remains unclear. The previous studies have proved that DLC can prevent severe corrosive failure in acidic environment. However, the study about tribological properties of DLC in nitric acid is few.

The objective of present work is to study the tribological characteristics of DLC in different concentration of nitric acid. And the corrosion resistance of DLC will also be evaluated.

2. Experimental Procedures

2.1 Specimen

Tetrahedral amorphous carbon (ta-C) was deposited on SUJ2 disk using IBA-FAD system, as shown in figure 1. The SUJ2 disk is $\Phi 22.5 \times 4$ mm and was cleaned using ultrasonic cleaner with benzene and acetone respectively. The purpose of cleaning process is to remove oil and other impurities from the surface. In this equipment, the T-shaped filter was used to eliminate macro-droplets produced in the arc process to improve the coating quality [3]. The deposition will be carried out at a pressure of 4.0×10^{-3} Pa and argon gas will be used to clean the surface for 20 minutes. The applied substrate bias voltage was set as -100 V, and the coating time was 20 min. Besides, to improve adhesion between the substrate and ta-C, a titanium interlayer was introduced [4]. Titanium deposition time was 15 minutes, and sputter power was 600 watts.

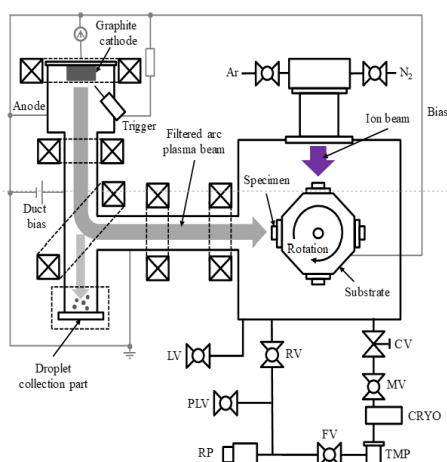


Fig.1 Schematic diagram of the IBA-FAD system

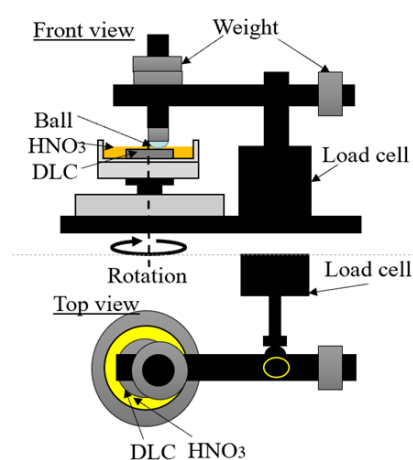


Fig.2 Schematic diagram of ball on disc tribometer

2.2 Friction Test

In this experiment, ball on disk tribometer was used to test friction behaviors, as shown in figure 2. The tribopair is ta-C disk versus Si_3N_4 ball. The ta-C disk will be immersed in the nitric acid. The nitric acid will be diluted with distilled water to pH1 and

pH2 acid. The friction test will be conducted in room temperature for 60 minutes. The normal load is 9.8N and the sliding speed of ball is 38 mm/s.

2.3 Coating characterization and surface analysis

The hardness and Elastic modulus of ta-C coatings were evaluated by nano-indentation method with a Berkovich indenter (ENT-1100a, Elionix, Japan). The hardness of ta-C coating is 34.8 GPa and Elastic modulus is 378.4 GPa. The thickness of ta-C coatings was measured by laser microscope. The thickness of ta-C coating is 360 nanometers.

3 Result and Discussion

3.1 Tribological performance

Figure 3 shows the friction coefficient of ta-C coatings in pH1, pH2, and distilled water. In the running-in stage, the friction coefficient is a little higher compared than the whole friction process, which is because of micro-asperities and the associated interlocking phenomenon between the contact interfaces and tribo-pairs. The average values of friction coefficient (calculated from the last 1500 sliding cycles) in pH1, pH2, and distilled water are 0.082, 0.072 and 0.044, respectively.

3.2 Corrosion

Using laser microscopy to observe the wear track of ta-C coatings, we can find that the pitting corrosion occurred obviously in pH1 nitric acid environment, as shown in figure 4. Iterative Self-Organizing Data Analysis Technique (ISODATA) is an automatic threshold selecting method to segment images and create binary images. Using this method, the defect density of surface before friction and after friction was calculated. The results show that the defect density did not change significantly in the environment of distilled water and pH2 nitric acid. But in the environment of pH1 nitric acid, the defect density increased significantly because of corrosion on the surface.

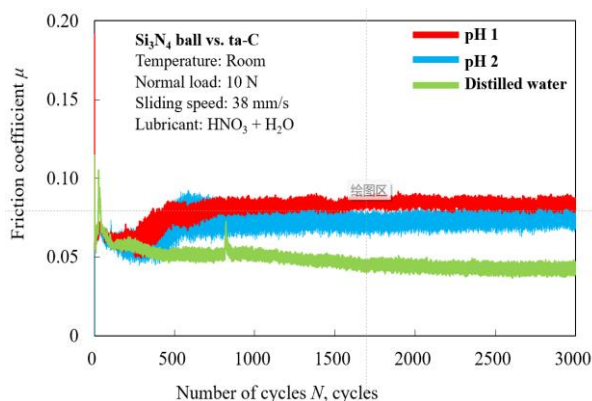


Fig.3 Friction coefficients with sliding cycles.

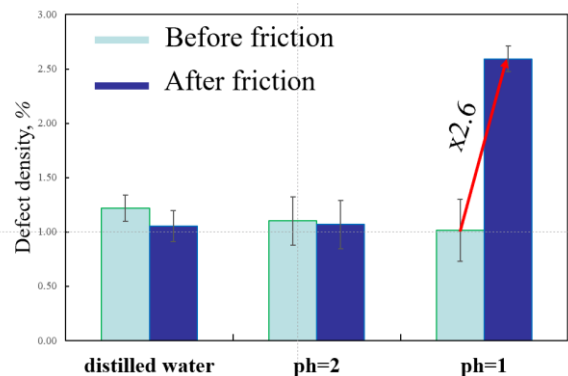


Fig.4 Defect density with different environment

4 Conclusion

In this study, ta-C coating was deposited on the surface of SUJ2 disk. A titanium interlayer was introduced to improve the adhesion between ta-C coating and substrate. Through the observation of laser microscopy, we found that titanium interlayer can effectively prevent the occurrence of delamination in the acidic environment. In pH1 nitric acid environment, pitting corrosion was evident. By using ISODATA method to calculate the defect density on the surface, we found that the defect density did not change significantly in the environment of distilled water and pH2 nitric acid. But in the environment of pH1 nitric acid, the defect density increased significantly because of corrosion on the surface.

Reference

- 1) Nagai T, Hiratsuka M, Alnazi A, Nakamori H, Hirakuri K. Anticorrosion of DLC coating in acid solutions. *Applied Surface Science*. 552, (2021) 149-373.
- 2) Wu W, Murashima M, Saso T, Tokoroyama T, Lee W-Y, Kousaka H, et al. New in situ superlow-friction method for nitrogen-containing diamond-like carbon coatings using dielectric barrier discharge treatment in ambient air. *Tribology International*. 174, (2022) 107-749.
- 3) Liu X, Yamaguchi R, Umehara N, Deng X, Kousaka H, Murashima M. Clarification of high wear resistance mechanism of ta-CN_x coating under poly alpha-olefin (PAO) lubrication. *Tribology International*. 105 (2017) 193-200.
- 4) Lee KR, Yong Eun K, Kim I, Kim J. Design of W buffer layer for adhesion improvement of DLC films on tool steels. *Thin Solid Films*. 377-378, (2000) 261-8.