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In-situ TEM studies on stick-slip friction characters of nanocrystallited carbon films

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Carbon films with two different kinds of *sp*^{2} nanocrystallited structure were investigated to study the stick-slip friction with the in-situ and ex-situ tests. In-situ TEM observation and nanofriction tests revealed that the origins of stick and slip varied with shear stress and film deformation, which was affected by the film density, surface roughness and hardness. Ex-situ nanofriction tests showed the same sliding behavior with the in-situ results. This study first clarified the mechanism of stick-slip friction with the in-situ TEM observation, which plays the important role for the micro and nano application of *sp*^{2} nanocrystallited carbon films.

Keywords: *sp*^{2} nanocrystallited carbon film; stick-slip friction; contact area; deformation; in-situ TEM

1. Introduction

Stick-slip friction is a common phenomenon during the sliding process, which means the contact interface exhibited stick and slip alternately [1]. The occurrence of stick-slip friction would increase the instability and reduce the tribological performance [2]. And there are many studies on stick-slip behavior at macro and atomic scales for the materials of amorphous structure [3], oriented crystalline structures like graphene [4] and graphite [5]. However, the dynamic transition of frictional contact interface during the stick and slip process is still a black box. For now, the transmission electron microscope is an important equipment to in-situ observes the nanostructure in atomic scale [6]. For the sp^2 nanocrystallite embedded carbon film prepared by electron cyclotron resonance (ECR) plasma sputtering system, the self-lubrication of sp^2 bonds can reduce the friction coefficient [7], sp^3 bonds can enhance the hardness and reduce the wear [8]. However, the carbon film exhibited different extent of stick-slip friction at microscale with nanoscratch test, and the mechanism of origin is still unclear. Therefore, in this paper, sp^2 nanocrystallited carbon films were prepared by different irradiation particles and energies, the origin of stick-slip behavior of sp^2 nanocrystallited carbon films were studied with in-situ TEM friction tests and verified with the ex-situ friction tests.

2. Methods

2.1. Preparation of sp^2 nanocrystallited carbon film

The sp^2 nanocrystallited carbon films were prepared with electron cyclotron resonance (ECR) plasma sputtering system. To attract the electrons for electron irradiation, mirror-confined magnetic field was used and positive biases of +0 V, +20 V, +50 V and +80 V were applied on the substrate, the deposition time was 40 min for the film thickness of about 100 nm. To attract the ions for ion irradiation, divergent magnetic field was used and nagtive biases of -0 V, -20 V, -50 V and -80 V were applied on the substrate, the deposition time was 30 min for the film thickness of about 100 nm.

2.2. In-situ TEM nanofriction test

The in-situ nanofriction properties of the sp^2 nanocrystallited carbon films were tested using a picoindenter (BRUKER, Hystron, PI95) in the HRTEM (ThermoFisher, Titan3 Themis G2) with electron accelerating voltage of 80 kV. Electron irradiated sp^2 nanocrystallited carbon film with substrate bias of +20 V and ion irradiated one with bias of -20 V were chosen for the in-situ TEM nanofriction test. The films for in-situ tests were deposited on the silicon wedge substrate with plateau width of 150 nm, and then the film is thin enough for the transmission of electrons without any further thinning process to induce the surface damage.

The schematic and apparatus for the in-situ TEM nanofriction test were shown in Fig. 1. The film on silicon wedge substrate can be directly mounted on the holder for nanofriction test and TEM observation without any surface damage. The in-situ tests were performed with a cube corner tip (curvature radius of 40 nm) indenting and sliding against the film surface. 2D MEMS transducer control the normal and the lateral forces and displacements, and the load resolutions for normal and lateral directions are 4 nN and 20 nN, respectively. The nanofriction tests were performed in load control mode and the applied loads were 10 μN, 20 μN, and 40 μN. The sliding distance was set as 200 nm, and the sliding time was 15 seconds.

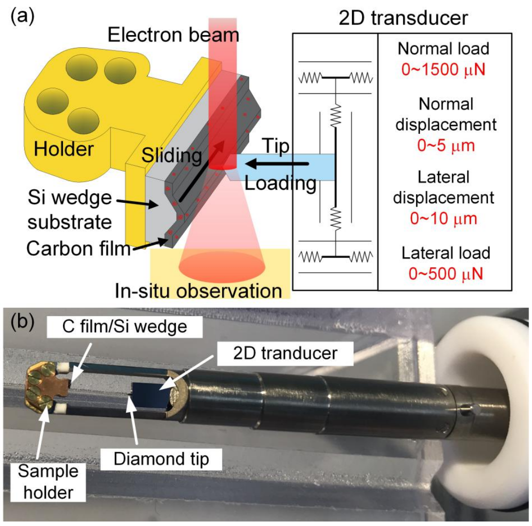

Figure 1: The schematic and apparatus of in-situ TEM nanofriction test

2.3. Ex-situ nanofriction test

The ex-situ nanofriction properties of the sp^2 nanocrystallited carbon films were tested using a triboindenter (BRUKER, Hystron, TI950). The Berkovich tip with nominal tip radius of 100 nm was used to sliding against the film surface. The sliding loads were set as 200 μN, 500 μN, 1000 μN, and 1500 μN. During the nanofriction tests, 2D transducer was used, which enabled to measure the lateral force during the sliding process. The load resolutions for normal and lateral directions are 1 nN and 3 μN, respectively. The sliding distance was set as 10 μm, and the sliding time was 15 seconds. After the sliding process, the wear depth was acquired by scanning the section profiles of the scratch track with the Berkovich tip. The images were obtained with scan size of 15 μm×15 μm to cover the whole sliding distance, and the loading force was 2 μN. Then, the wear depth was obtained by made sectional lines across the wear tracks. In order to increase the reproducibility and accuracy of the measurements, we performed the double check measurement.

3. Results

The in-situ nanofriction tests were performed to investigate the origin of stick-slip friction. The electron and ion irradiated carbon films with +20 V and -20 V tested separately under the loads of 10 μN, 20 μN and 40 μN, and the in-situ friction coefficient were shown in Fig. 2. In Fig. 2(a), the electron irradiated carbon film showed severe stick-slip friction. The degree of stick increased with the increasing of load, and the tip was fully adhered when the load was 40 μN. The video of dynamic sliding process under the load of 10 μN showed that the contact between the tip and the film surface first strengthened with a gradually increased stick area and bended the nanocrystallited film with the trend of sliding, when the shear strength exceeded the adhesive strength, the tip suddenly slipped over with a much smaller contact area. Then the tip went into another stick-slip stage. Four different stick-slip stages were marked with I~IV in the figure and the relative contact status observed with TEM images were shown with inserted figures I~IV respectively. In the inserted figure I, the tip was just contact with the surface without applying load. In the inserted figure II, when the stick increased to a critical stage, the contact area was large. In the inserted figure III, when the slip happened, the contact area was obviously smaller than before, which meant the unstable sliding process of the electron irradiated ones. In the inserted figure IV, after the nanofriction test, there was abrasive wear particle showed on the surface. In Fig. 2(b), the ion irradiated carbon film also showed that the contact strength increased at first, and then the tip slid against the film with slight vibration and smoother sliding process. The video of dynamic sliding process showed that after sliding process, a long wear track was generated with the tip ploughing against the film. The different states of the friction curve had been marked V~VIII. It can be found from the inserted figures VI and VII that the contact area of ion irradiated ones was almost the same during the stick and the slip process.

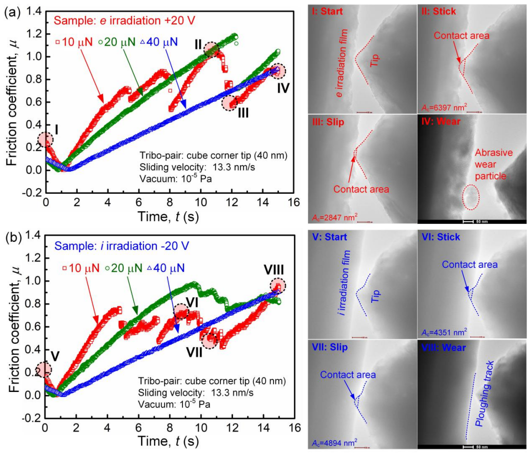

Figure 2: The in-situ nanofriction test results for the films prepared with +20 V and -20 V under the load of 10 μN, 20 μN and 40 μN, respectively, the inserted figures I~VIII show the in-situ nanofriction states.

Figure 3 exhibited two different plastic deformation type of electron and ion irradiated carbon films. For electron irradiated carbon films with lower density, higher surface roughness and lower hardness, the tip was easier to stick on the film surface due to the adhesion with a contact strengthening process, the contact area grew until the static force reached the critical shear value [9,10]. During this period, the area behind the probe showed breakage and the breakage broadened gradually, as shown in Fig. 3(a) and 3(b). Then the energy stored in the stick process dissipated [11], resulting in a large area of plastic deformation on the contact area, as shown in Fig. 3(c). For ion irradiated ones shown in Fig. 3(e)-3(h), the contact strengthened at first, and when the shear strength is high enough to break the stiction, the tip ploughed on the film surface. Because of the high film density, lower surface roughness and higher hardness, the contact area was changed small, and the carbon film exhibited a smooth and stable sliding process. For electron irradiated carbon films, the largest depth of adhesion deformation was 55.4 nm with the load of 10 μN, which was much deeper than the average contact depth of 16.8 nm at the stick stage. It meant the severe stick-slip friction was due to the adhesion [12]. For ion irradiated carbon films, the largest depth of ploughing was 10.5 nm with the load of 10 μN, which was shallower than the average contact depth of 14.5 nm at the stick stage. Therefore, the origins of stick and slip varied with shear stress and film deformation, which was affected by the film density, surface roughness and hardness. Higher film density, lower surface roughness and higher hardness promise the stable of contact area with ploughing of plastic deformation, and such kind of sp^2 nanocrystallited carbon films show the potential for smooth sliding as nanoscale surface.

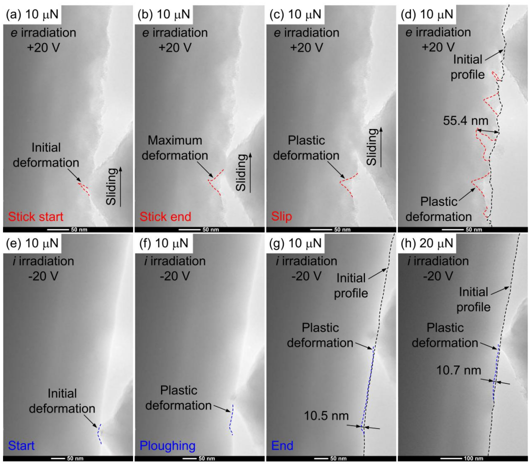

Figure 3: (a)-(d) Large area of plastic deformation caused by stick-slip friction, (e)-(h) Ploughing of plastic deformation with a stable sliding process.

4. Conclusion

Carbon films with two different kinds of sp^2 nanocrystallited structure were investigated to study the stick-slip friction with the in-situ and ex-situ tests. The ECR plasma sputtering system was used to prepare the carbon films with different sp^2 nanocrystallitze size, film density, surface roughness and hardness. In-situ TEM observation and nanofriction tests revealed that origin of stick and slip varied with shear stress and film deformation, which was affected by the film density, surface roughness and hardness. Ex-situ nanofriction tests on a series of sp^2 nanocrystallited carbon films with different irradiation energies showed the same scratch behavior with the in-situ results. This study first clarified the mechanism of stick-slip friction with the in-situ TEM observation, which plays the important role for the micro and nano application of sp^2 nanocrystallited carbon films.

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6. References

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