ISSN 1516-392X

NANOFRETTING BEHAVIOR OF SI(100) AND ITS COATING[1]

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Abstract
With an atomic force microscopy, the nanofretting behaviors of Si(100) and its coating were investigated at various displacement amplitudes (0.5~250 nm) under atmosphere and vacuum conditions. It was found that the adhesion force may induce the increase in the maximum static friction force and prevent the contact pair from slipping. The nanofretting damage on silicon may experience two processes, namely as the generation of hillocks at low load and the formation of grooves at high load. A ultrathin hard DLC coating could reduce about 75% of the nanofretting damage on Si(100) substrate.
Key words: Nanofretting; Si(100); DLC coating; AFM.

1 *Technical contribution to the 18th IFHTSE Congress - International Federation for Heat Treatment and Surface Engineering, 2010 July 26-30th, Rio de Janeiro, RJ, Brazil.*
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1 INTRODUCTION

Nanofretting refers to cyclic movements of contact interfaces with the relative displacement amplitude in nanometer scale, where the contact area and normal load are usually much smaller than those in fretting.[1] Because of the temperature variation and mechanical vibration, nanofretting of monocrystalline silicon may exist in the contact interfaces of nano/microelectromechanical systems (NEMS/MEMS). Therefore, with the development in NEMS/MEMS, the understanding and control of the nanofretting behavior of monocrystalline silicon has become an important issue of concern.[2-3]

After the concept of nanofretting was proposed by Zhou and Qian[4] in 2003, people have performed tangential and radial nanofretting tests on kinds of materials. In 2005, Varenberg et al.[5] reported their studies on nanoscale fretting wear behavior of monocrystalline silicon (100). In 2007, Qian et al.[1] presented their research results on the tangential nanofretting behaviors of NiTi shape memory alloy. They found that nanofretting was different from fretting in aspects of the variation of tangential force versus number of nanofretting cycles, the value of friction coefficient, and the wear mechanism. These differences were further attributed to the single-asperity contact in nanofretting and multi-asperity contact in fretting.

In this paper, the nanofretting behaviors of Si(100) and its coating were investigated by an atomic force microscopy (AFM). It was found that the adhesion force revealed a strong effect on the regimes of tangential nanofretting. Different from fretting, the tangential nanofretting damage on silicon may experience two processes, namely as the generation of hillocks at low load and the formation of grooves at high load. A ultrathin hard coating could effectively prevent the nanofretting damage of Si(100) substrate.

2 MATERIALS AND METHODS

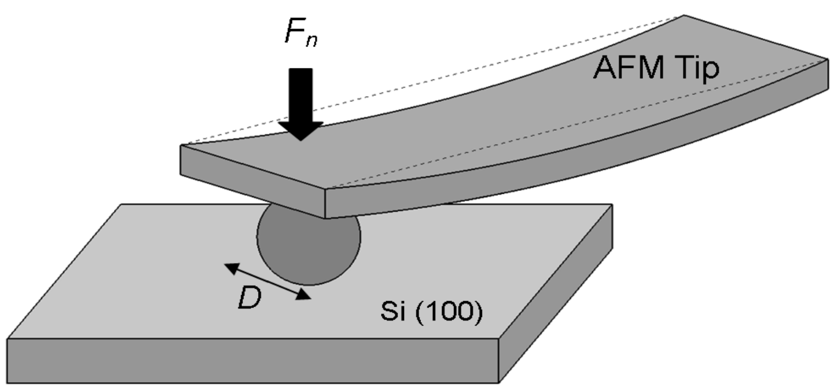


Fig. 1. The schematic illustration showing the nanofretting test.

The p-Si(100) wafers of 0.5 mm thickness were received from MEMC Electronic Materials, Inc., USA. The ultra-thin DLC films were prepared on Si(100) wafers by a physical vapor deposition system. Raman spectroscopy and X-ray photoelectron

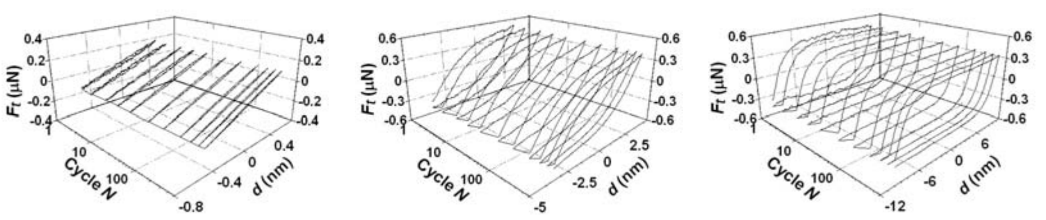
ISSN 1516-392X

spectroscopy (XPS) were used to analyze the composition and the microstructure of the DLC film. The hardness of DLC films was measured as about 85 GPa.

All the nanofretting tests and in situ topography scanning were performed by an AFM equipped with a vacuum chamber. Fig. 1 schematically shows the nanofretting of spherical tips against Si(100) surface. The spherical tips included SiO_2 (Novascan Technologies, USA) and diamond microsphere (Microstar Technologies, USA), respectively. Their radii R ranged from 0.15 μm to 1.0 μm. During the nanofretting, the spherical tips moved horizontally on silicon surface with a displacement amplitude D under a normal load F_n. The applied normal loads F_n were varied between 0.5 μN and 70 μN. The displacement amplitudes D were ranged from 0.5 nm to 250 nm. The frequency was 2 Hz and the number of nanofretting cycles N was varied between 1 and 500. After nanofretting, the topography of scars was scanned by a sensitive silicon nitride tip. All the nanofretting tests were performed in atmosphere with a relative humidity of 50%-60%, or in vacuum with a pressure below 5.0×10^{-6} torr (6.7×10^{-4} Pa).

3 RESULTS AND DISCUSSION

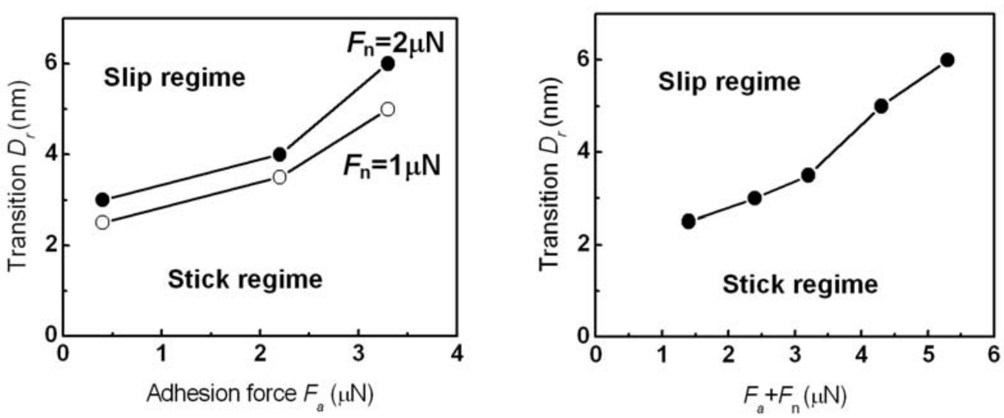
3.1 The Effect of Adhesion Force on the Regimes of Tangential Nanofretting



D=0.5 nm *D*=3.5 nm *D*=10 nm

Fig. 2. The frictional logs of Si(100)/SiO_2 pair at various displacement amplitudes D; R=0.43μm.

As shown in Fig. 2, the shape of F_t–d curves in nanofretting was found to vary from line shape to parallelogram with the increase in displacement amplitude D, which corresponds to the transition from the stick regime to slip regime. [6-7]



(a) (b)

Fig. 3. The transition displacement D_r between stick regime and slip regime versus (a) adhesion force F_a and (b) F_a+F_n curves.

ISSN 1516-392X

In order to understand the role of adhesion force in the nanofretting behavior of material, the transition displacement D_r was obtained through three SiO_2 tips (R=0.43 μm) with the adhesion forces of 0.4 μN, 2.2 μN and 3.3 μN on Si(100) surface, respectively. As shown in Fig. 3, with the increase in adhesion forces, the transition displacement amplitude D_r would move towards high values of displacement amplitude. During the nanofretting process of Si(100)/SiO_2 pair, the adhesion force may induce the increase in the maximum static friction force and prevent the contact pair from slipping. For instance, when the adhesion force varied from 0.4 μN to 3.3 μN, the maximum static friction force increased from 0.46 μN to 1.33 μN for an applied normal load of 2 μN. As a result, with the increase in adhesion force F_a, the tip was more difficult to slip.

3.2 The Damage Mode of Nanofretting

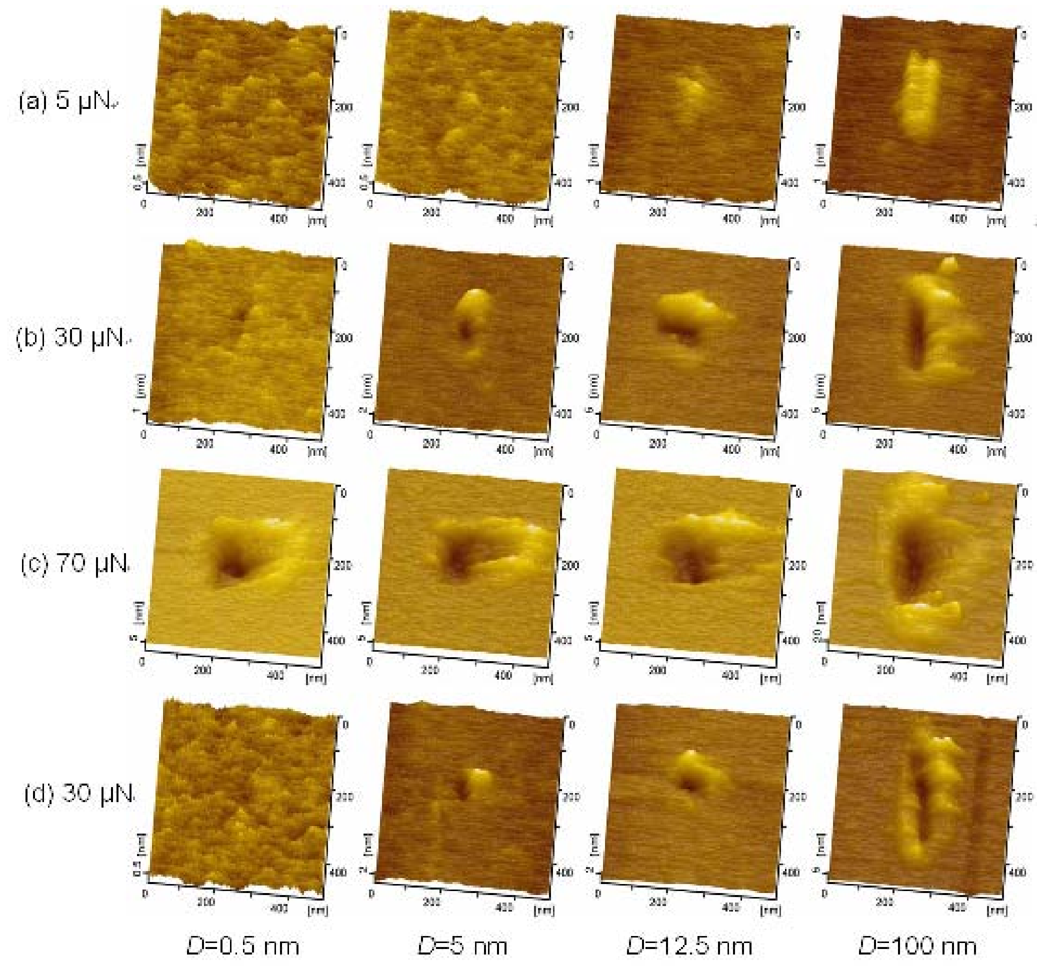


Fig. 4. AFM images of the scars on Si(100) surface after nanofretting at various displacement amplitudes D and normal loads F_n in atmosphere (a-c) and in vacuum (d). R= 0.15 μm and N= 100.

As shown in Fig. 4a, for the diamond tip with R =0.15 μm and under F_n =5 μN, no obvious damage was observed in stick regime ($D \leq$ 5 nm). With the increase in D, the hillocks were generated in the contact area of Si(100) surface. However, when the normal load F_n attained 30 μN, the grooves were observed in the wear area of Si(100) surface both in atmosphere and in vacuum, as shown in Fig. 4b and Fig. 4d. Finally, as shown in Fig. 4c, when the normal load F_n was increased to 70 μN, wider and deeper grooves were observed on Si(100) surface. The results indicated that the nanofretting damage of Si(100) surface was strongly depended on the displacement amplitude and normal load. With the increase in normal load, the nanofretting damage would undergo an evolvement from the generation of hillock to groove.

3.3 The Transition Between Two Damage Modes

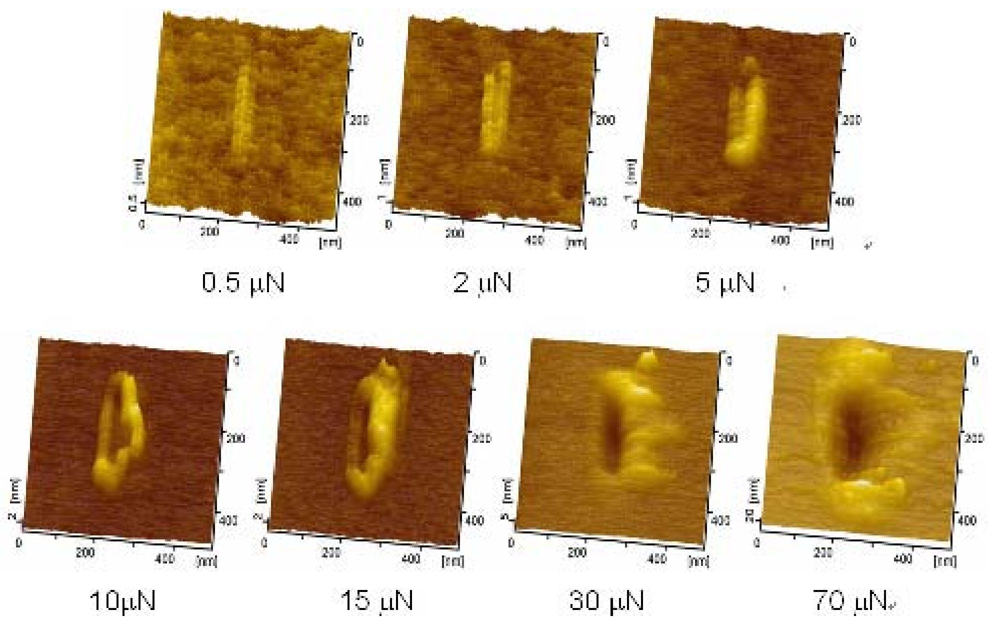
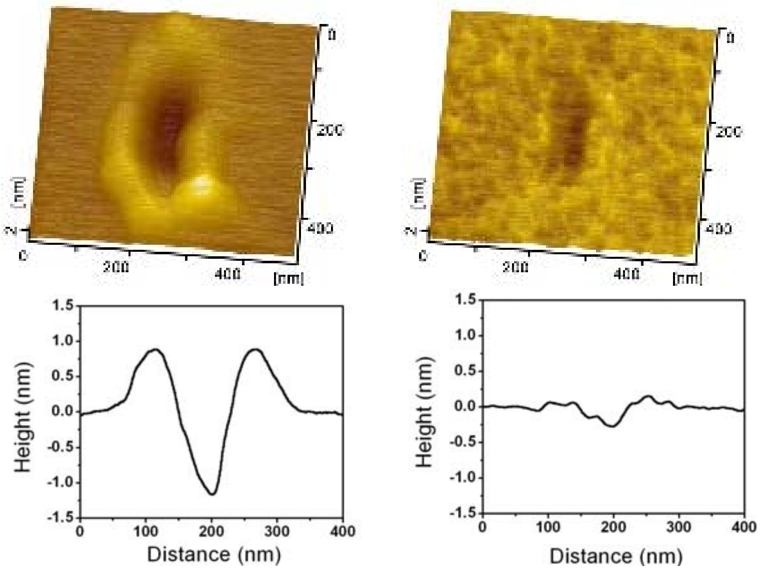


Fig. 5. AFM images of the scars on Si(100) surface after nanofretting under various normal load F_n in atmosphere. R=0.15 μm, D = 100 nm and N = 100.

To understand the nanowear process of Si(100), the nanofretting of Si(100)/diamond pair was conducted at various normal loads in atmosphere. As shown in Fig. 5, the transition of surface damage of silicon from hillock to groove was observed with the increase in normal load F_n. The hillock was generated on Si(100) surface for F_n<10 μN. As F_n = 10 μN, the cupped shape of scars appeared on the top of hillock. As F_n was higher than 15 μN, the groove was formed on Si(100) surface. With the increase in F_n, the height of hillocks first increased to 1.5 nm for F_n =5 μN and then presented a decrease. When F_n >15 μN, the surface height was below the original surface and the depth of groove attained 9.4 nm for F_n =70 μN. Clearly, the nanofretting damage of Si(100) surface may successively experience two progresses: hillocks and grooves. The hillock was usually generated under low normal load or in the initial nanofretting cycles. However, the groove was always formed under high normal load or after a large number of nanofretting cycles. Analysis indicated that the critical contact pressure for the transition of damage mode should be between 12.67

GPa and 15.96 GPa under the given condition, which is very closed to the hardness of Si(100).[8]

3.4 Comparison of the Nanofretting Damage on Si(100) and its Coating



(a) Si(100) (b) 2 nm DLC coating on Si(100)

Fig. 6. AFM images of the nanofretting scars on Si(100) and DLC surface in air.

To prevent the nanofretting wear on Si(100), a DLC coating of 2 nm in thickness was prepared on Si(100). As shown in Fig. 6, the groove with the depth of 1.2 nm on the Si(100) surface was formed after nanofretting under 2 μN by a SiO_2 tip. However, the depth of the groove on DLC coating was only 0.3 nm under the same conditions. Clearly, due to its high hardness, the DLC coating could reduce 75% of nanofretting damage on Si(100) substrate.

4 CONCLUSIONS

The nanofretting behaviours of Si(100) and its coating were investigated at various displacement amplitudes (0.5~250 nm) with an atomic force microscopy. The main conclusions can be summarized as following:

(1) The tangential nanofretting could be divided into stick regime and slip regime upon the transition criterion. The adhesion force may induce the increase in the maximum static friction force and prevent the contact pair from slipping.

(2) The nanofretting damage on silicon may experience two processes, namely as the generation of hillocks at low load and the formation of grooves at high load.

(3) A ultrathin hard DLC coating could effectively prevent the nanofretting damage of Si(100) substrate.

Acknowledgements

The authors are grateful for the financial support from the Natural Science Foundation of China (90923017, 50625515, 50821063).

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