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Reprint of "Tribological behavior of monocrystalline silicon from single- to multiple-asperity scratch"☆

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ABSTRACT

During producing silicon surface and fabricating silicon devices, the machining, cutting and milling from nanoscale to macroscale are accomplished with friction and wear under single- and multiple-asperity contact. In the present study, the scratch tests on silicon surface under single- and multiple-asperity contact were contrastively investigated, and the transition and correlation between different contact modes were addressed. When surface wear (formation of groove or protrusive hillock) appears under single-asperity sliding on silicon, the friction force became load-controlled and increased linearly with the normal load. The transition of single-asperity to multiple-asperity scratch was observed with the increase in the applied normal force. Under the multiple-asperity condition, surface hillocks were created on the scratched area, which were detected only under single-asperity test before. This study will shed new light on the understanding of multi-scale processing of silicon surface, and provide new insight for enriching tribology theory.

1. Introduction

Single-crystal silicon has excellent electrical and mechanical properties, and serves as a prevalent material for producing micro/nanoelectromechanical systems (MEMS/NEMS) [1,2]. During producing silicon surface and fabricating silicon devices, the machining, cutting and milling from nanoscale to macroscale have been a much concerned issue [3–5]. The processing of silicon is accomplished with friction and surface wear on sliding interface. For a deep understanding of the tribological behavior, it is meaningful to study the transition and correlation between single- and multi-asperity sliding on silicon.

In the past years, single-asperity contact has been studied with the focus on the scratch and indentation at micro- and nanoscale [6]. Szlufarska et al. defined directly the single-asperity contact as a single and continuous contact area, and it provides a straight way to differentiate it simply from combination effect of multiple asperities [7]. Generally, single-asperity contact takes place in nanotribological tests realized by scanning probe microscope (SPM) or some nano-scratch testers, where the tip can be viewed as a single asperity and the contact area is at nanoscale. At nanoscale level, the surface forces, such as friction and adhesion, usually play a much more important role than the bulk forces because of the large surface-to-bulk ratio (a large fraction of atoms are exposed at the surface) under single-asperity contact [8–10]. For example, the surface force can usually lead to a higher friction force or friction coefficient especially at lower normal load. For revealing the friction mechanism of single-asperity sliding, different contact models, such as DMT, JKR and MD models, were proposed to simulate the friction with some measurable contact parameters [6,11]. In contrast, under the multiple-asperity sliding, the friction increases linearly with the applied normal load and surface wear usually occurs under a low nominal contact pressure, and it was stated that several local asperities bear the applied load and trigger severe wear under higher contact pressure [12]. Moreover, the adhesion force under multi-asperity sliding is usually much lower than the applied load and can even be ignored, and the friction force may be figured out theoretically by summing up the contribution of individual asperities based on statistics method [6,13].

However, the reported tribological study was generally focused on tests at either single- or multi-asperity contact. There is still some shortage of study on the correlation and the transition behaviors between the two modes. Single-asperity scratch tests by SPM tip were used to investigate the friction and wear of silicon materials in the past

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decades. Due to the mechanical ploughing by a sharp diamond tip, the formation of grooves via material removal was considered as the main type of silicon wear in early reports, and the abrasive wear associated with plastic deformation was suggested to occur especially at high applied normal loads [14,15]. In addition, protrusive hillocks were found to form on silicon surfaces after scratching by a blunt diamond AFM tip, where the corresponding maximum Hertz contact pressure P_{max} is less than the hardness of silicon [16]. The mechanical interaction-induced amorphization is found to play a key role in the hillock formation, while the contribution of oxidation is very limited [16,17]. In contrast, during multi-asperity sliding of silicon, severe plastic deformation, micro-fracture and phase transformation can be detected, although the nominal contact pressure is much lower than the yield stress of silicon [18,19]. By far, there is hardly any insight towards the correlation and the transition mechanism between the two modes.

In the present study, the atomic force microscope (AFM), nano-scratching tester (NST) and universal micro-tribometer (UMT) were used for investigating the friction and wear of monocrystalline silicon from nano- to macro-scratch tests. To simulate the single- or multi-asperity contact, the tips and ball with the radii R ranging from 20 nm to 2 mm were used. Based on the scratching at different test scales, the transition and the correlation performance in surface wear and friction of silicon were addressed.

2. Materials and methods

P-type Si(111) wafers with a thickness of about 0.5 mm and surface root-mean-square (RMS) roughness of 0.3 nm was used for the test. The silicon wafer was cut into many pieces with the size of about 1×1 cm^2, and then ultrasonically washed in acetone, alcohol and deionized water for 5 min in turn for removing the surface contaminations.

The atomic force microscope (AFM, SPI3800N, Seiko, Japan) was used for the nanoscrach test. A Si_3N_4 tip (MLCT, Bruker Corporation, USA) with the tip radius (R) of about 20 nm and a diamond tip (Micro Star Technologies, USA) with R of about 1 μm were employed for the tests on AFM, as shown in Fig. 1(a) and (b). The normal spring constant of the probe was calibrated by a cantilever with known spring constant (CLFC-NOBO, Vecco, USA) [20]. The line-scratch tests were performed under one-cycle scratching by the Si_3N_4 tip under the load from 0 to 60 nN and by the diamond tip from 5 to 150 μN, respectively (Table 1).

The nano-scratching tester (NST; CSM Instrument, Switzerland) and a diamond tip of $R = 2$ μm (Fig. 1(c)) were used for the scratch test at microscale. The normal load was set as 0.3–11.0 mN. The universal micro-tribometer (UMT-2, CETR, USA) and a Si_3N_4 ball with a diameter of 4 mm were used for the scratching under the normal load from 0 to 400 mN. All the scratch tests were carried out in air with the relative humidity of 50 ± 5% and temperature of 25 ± 2 °C. After the scratch tests, the surface was scanned in situ on AFM with a Si_3N_4 tip in vacuum to avoid the adhesion from adsorbed water.

Table 1
Comparison of various experiment conditions in the present study.

Experiment setup	Tip/ball radius	Contact load	Purpose in present study
AFM	~20 nm	0–60 nN	Single-asperity contact
AFM	~1 μm	5–150 μN	Single-asperity contact
NST	~2 μm	0.3–11 mN	Single- to multi-asperity contact
UMT	2 mm	0–400 mN	Multi-asperity contact

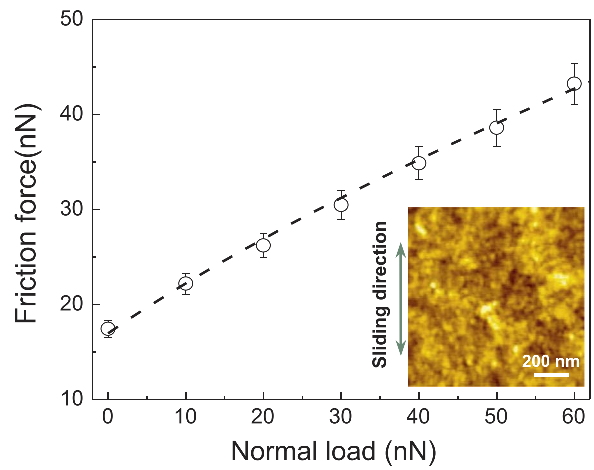

Fig. 2. The variation of the friction force with the applied normal load F_n measured by a Si_3N_4 tip ($R \approx 20$ nm). The dotted line is fitted by DMT model. The inset AFM image shows the area after the friction test, where the bidirectional arrow indicates the sliding direction.

3. Experimental results and discussions

3.1. Single-asperity scratch test

The silicon surface was scratched by a Si_3N_4 tip on the AFM under linearly loading from 0 to 60 nN. Fig. 2 presents the friction force and the surface topography after the friction test. No obvious surface wear can be found from the scratched area. It is noted that at the applied normal load $F_n = 0$, the friction force F_f is up to 17 nN. This can be ascribed to the contribution from adhesive contact at nanoscale [21–23]. For interpreting the variation in the friction, the contact model in the case of the contact and adherence of a sphere can be used

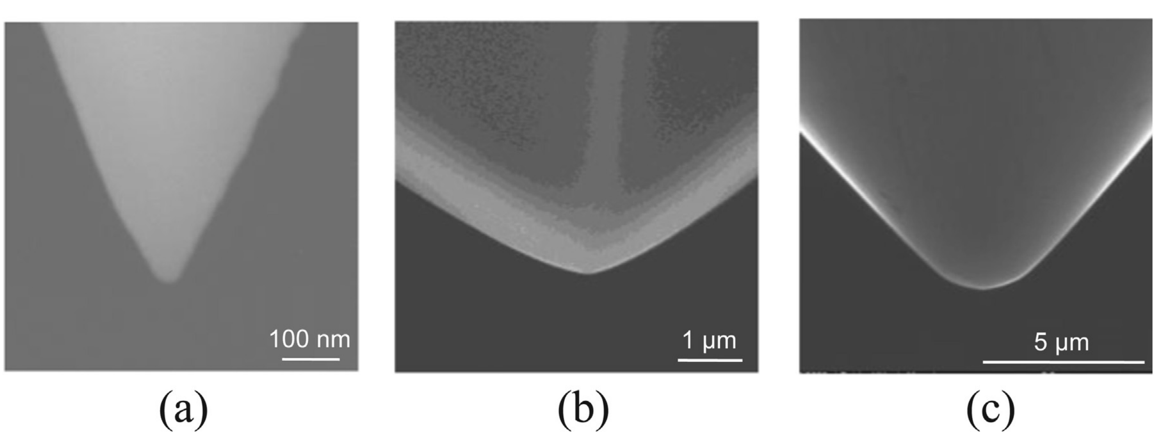

Fig. 1. SEM images of the tips used in the present study. (a) AFM Si_3N_4 tip with R of about 20 nm, (b) AFM diamond tip with R of about 1 μm and (c) the diamond tip with R of about 2 μm used on NST.