



Effect of mechanical interaction on the tribochemical wear of bare silicon in water

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ABSTRACT

Nanowear tests of bare silicon were conducted in water using an atomic force microscope with microspherical SiO₂ tips. The effect of mechanical interaction, such as contact pressure, sliding velocity, and sliding cycles, on the tribochemical wear of bare silicon was examined. Experimental results indicated that contact pressure significantly influenced the wear of bare silicon. When the maximum contact pressure P_{max} increased by 146%, the corresponding wear depth of bare silicon rose by 264%. Sliding velocity slightly affected the wear of bare silicon. The wear trench depth decreased by 40% as the sliding velocity increased by approximately three orders of magnitude. The wear cycles exhibited a relatively stronger effect on the wear of bare silicon than the sliding velocity. When the number of sliding cycles increased to 1000, the average wear depth of each cycle decreased by roughly 90%. The high-resolution transmission electron microscopy observations of wear trenches confirmed the facilitative effect of mechanical interaction on the tribochemical reaction of the Si/SiO₂ interface. Further analysis indicated that increasing contact pressure may increase the contact area and shear stress. A large contact area may result in the formation of more Si–O–Si bonding bridges between Si/SiO₂ interfaces, and high shear stress can facilitate the hydrolysis of Si–Si back-bonds through water molecules. Both processes aggravate the wear of bare silicon. High sliding velocity may restrict the formation of interfacial Si–O–Si bonding bridges to some extent and further decrease wear depth. Results may provide insight into the tribochemical wear mechanism of silicon and optimize the process parameters of silicon chemical mechanical polishing.

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1. Introduction

Single-crystal silicon, which is the most essential semiconductor material, is widely applied in integrated circuits [1]. The application of silicon in ultra-large-scale integrations requires the sub-nanometer surface roughness and ultra-precision surface flatness of the silicon surface [1,2]. Chemical mechanical polishing (CMP) is the most widely used technique that affords a silicon surface with global subnanometer-scale planarization [3,4]. During the CMP process, the native oxide on the silicon wafer is removed, and the wafer maintains a fresh surface (bare silicon) throughout the entire process [5]. The material removal from silicon in CMP is an intricate tribochemical process [6]. Process parameters, such as down pressure, plate speed, and polishing time, influence the quality and efficiency of CMP significantly [7].

Over the last decades, numerous studies have been conducted using polishing machines to analyze the effect of process

parameters on silicon CMP [6–9]. The removal rate for Si(100) CMP increased sub-linearly with the applied pressure, plate speed, and slurry concentration [7]. In a similar manner, the removal rate with soft abrasives was significantly affected by the increase of down pressure, high slurry concentration, and rotational velocity in mechano-chemical polishing [8]. Experimental and theoretical studies showed that aside from the process parameters of pressure and velocity, other important input parameters, such as wafer hardness, pad hardness, pad roughness, abrasive size, and abrasive geometry, can affect the material removal rate dramatically [9]. These studies suggested that optimizing mechanical interaction can improve the quality of silicon CMP significantly. However, the effect of mechanical interaction on the tribochemical removal of bare silicon has not been fully understood.

Various studies investigated the microscale wear of silicon under different mechanical conditions [10–18]. When an as-received silicon surface is scratched by a diamond tip in various environment, a hillock-like wear trench will form on the surface without material removal. This protrusive structure is composed of amorphous silicon and is substantially dependent on contact

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pressure, scratch speed, and scratch cycles [12–14]. As a comparison, when an as-received silicon surface is scratched by a silica tip in air under low contact pressure, a groove-like wear trench is generated on the surface with material removal [15]. However, most of these studies used as-received silicon covered with native oxide. Studies on bare silicon (without native oxide on the surface) are limited. The wear of silicon in water is highly affected by the native oxide on the silicon surface [15,19]. Therefore, to better simulate and optimize silicon CMP, it is crucial to understand the effect of mechanical interaction on the tribochemical removal of bare silicon.

In the present research, the nanowear of bare single-crystal silicon (without native oxide on the surface) was investigated through atomic force microscopy with micro-spherical SiO₂ tips under various contact pressures, sliding velocities, and sliding cycles. High-resolution transmission electron microscope (HRTEM) observations were performed on the cross-section of the wear trenches. The effect of mechanical interaction on the tribochemical wear of bare silicon was discussed. This work may provide insight into the tribochemical mechanism of single-crystal silicon and optimize the process parameters of silicon CMP.

2. Material and methods

As-received single-crystal silicon wafer (Si-100; covered with native oxide) with a thickness of approximately 0.5 mm was purchased from MEMC Electronic Materials, Inc., USA. Bare silicon samples were obtained by etching the as-received silicon in hydrofluoric acid (40 wt% aqueous HF) for 2 min, ultrasonic cleaning in ethanol for 3 min, rinsing with ultrapure water, and drying with the nitrogen. The surface water contact angle of the silicon sample was approximately 83° after the treatments. All nanowear tests of bare silicon were performed against a micro spherical SiO₂ tip (Novascan Technologies, Inc., USA) through an atomic force microscope (AFM; SPI3800N, Seiko, Japan). The radius of the SiO₂ tip was approximately 1.25 μm. A silicon nitride AFM tip (MSCT; Bruker, USA) with a curvature radius of 20 nm was employed to scan the 3-D surface topography of the silicon samples.

In the nanowear tests, load F_n , which was applied to the SiO₂ tip, was set between 0.2 μN and 3 μN. The relative sliding velocity v varied from 0.1 μm/s to 100 μm/s. The relative sliding length L was 500 nm, and the number of reciprocating sliding cycles N ranged from 1 to 2000. All the nanowear tests were conducted in ultrapure water using a fluid cell at room temperature (Fig. 1). The ultrapure water was produced by a laboratory water purification system (Master-S; Hi-tech, China), and its resistivity was 18.2 MΩ·cm at room temperature. Before conducting the wear tests, the bare silicon samples, cantilever holder, and fluid cell were cleaned completely to prevent impurities from accessing the sliding interface. The water contact angle of bare silicon was around 74° after storing the bare silicon in water for 1 h. Thus,

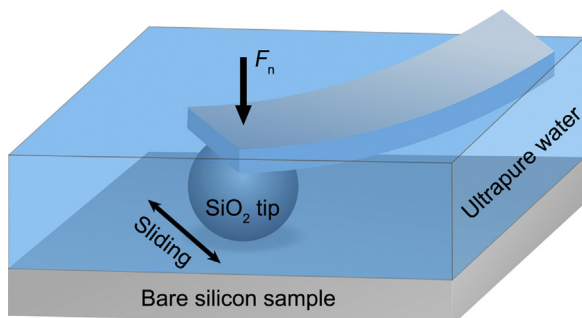


Fig. 1. Nanowear tests of the Si/SiO₂ interface in water by atomic force microscopy.

each nanowear test was controlled within 1 h to prevent the influence of surface chemical change on the wear tests. To avoid the possible influence of crystal orientation on the nanowear of bare silicon, all the scratches in this study were along $\langle 100 \rangle$ direction on the bare Si(100) surface.

HRTEM (Tecnai G2 F20, FEI, USA) was used to characterize the cross-sectional microstructure of the wear trench on the bare silicon surface. Before the transmission electron microscopy (TEM) detection, the cross-sectional TEM samples were prepared using a focused ion beam miller (Nanolab Helios 400S; FEI, Holland).

3. Results

3.1. Nanowear of bare silicon under various contact pressures

Nanowear tests of the Si/SiO₂ interface were conducted under the applied loads of 0.2–3 μN in water to examine the effect of contact pressure on the tribochemical wear of bare silicon. The number of sliding cycles N was set to 100 during the tests. It was reported that the elastic moduli of SiO₂ tip and bare Si(100) substrate are 72 GPa and 130 GPa, respectively [20,21]. The Poisson's ratios associated with them are 0.17 and 0.28, respectively [21,22]. Based on the pull-off force measurement, the adhesion force between SiO₂ tip and bare silicon substrate in water approaches zero [23]. Therefore, for the applied loads of 0.2–3 μN, the maximum contact pressure P_{max} between the bare silicon surface and the SiO₂ tip was estimated to be 0.39–0.96 GPa according to the DMT contact model [24]. These values were considerably below the yield stress of the bare single-crystal silicon (roughly 10.9 GPa) [20].

Fig. 2 shows the typical AFM 3-D surface topographies and average cross-sectional profiles of the wear traces on the bare silicon surface. The variation of wear depth d and wear volume V of the bare silicon with maximum contact pressure P_{max} are shown in Fig. 3. The wear depth almost showed a linear increase with P_{max} (Fig. 3a). When P_{max} increased by 146%, the corresponding wear depth of the bare silicon increased by 264%. The wear volume of the bare silicon exhibited a super-linear increase. These results suggest that contact pressure plays a significant role in the tribochemical wear of bare silicon in water.

3.2. Nanowear of bare silicon at various sliding velocities

Relative sliding velocity can affect the material removal at microscale [25]. Nanowear tests of Si/SiO₂ interface were conducted in water at various sliding velocities to examine the effect of sliding velocity on the nanowear of bare silicon. Fig. 4 presents the typical AFM 3-D surface topographies of the wear traces on the bare silicon surface at various sliding velocities. Fig. 5 presents the variation of wear depth d and wear volume V of the bare silicon as a function of sliding velocity. When the SiO₂ tip slid on bare silicon surface at the lowest velocity of 0.1 μm/s, the depth of the wear scar was approximately 9.1 nm. With the increase in sliding velocity, the wear of bare silicon was gradually slight. When the velocity was 25 μm/s, the wear depth was roughly 5.3 nm. However, as sliding velocity increased further, the wear depth of bare silicon barely changed, maintaining its stability till the highest velocity of 100 μm/s (Fig. 5a). Overall, the depth of wear trench decreased by 40% as sliding velocity increased by around three orders of magnitude. The variation trend of the wear volume of bare silicon was similar with that of the wear depth (Fig. 5b). These results demonstrate that the tribochemical wear of bare silicon will be slightly affected by the relative sliding velocity to a certain scale.

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