

Atomic removal via mechanochemical reactions

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Ultra-precision of nanomanufacturing process down to the atomic level is of paramount importance for new development of nanoelectronics with unique functionalities. The ultimate precision that can be achieved for the crystalline materials would be the topographic control down to atomic level. Achieving such an ultimate precision requires physical means or processes to reliably and reproducibly remove atoms at a specific location with an arbitrary shape without causing subsurface damages or disorders. The influence of mechanochemical interaction at the material interface on its atomic level removal behavior during this process cannot be ignored. Here, we investigate the atomic level removal behavior of typical crystalline materials (such as silicon, graphene) under different conditions, reveal the atomic removal mechanism under the mechanochemical action. Furthermore, a mask-less and chemical-free nanolithography process is demonstrated for regio-specific removal of atoms on crystalline materials via shear-induced mechanochemical reactions. Since the chemical reactions involve the topmost atoms exposed at the interface, removal of the atomic layer is possible and the crystalline lattice beneath the processed area remains intact, keeping perfect crystalline order without subsurface structural damage for crystalline silicon or controlling the atomic structures of the fabricated graphene step edges. Molecular scale simulations explain the atom-by-atom removal process, where the atoms are removed preferentially through the formation and dissociation of interfacial bridge bonds. Based on the parametric thresholds needed for the atoms removal, the critical energy barrier for stress-assisted chemical dissociation of Si-Si bonds and C-C bonds is determined respectively. The mechanochemical nanolithography method demonstrated here could be extended to nanofabrication of other crystalline materials.

1. Introduction

The ultra-precision of nanofabrication at the atomic level is essential for advancing nanoelectronics with novel capabilities¹, particularly in the semiconductor industry. As the functional chip structures shrink to nanoscale, manufacturing processes require controllable atomic-level material removal in surface planarization of semiconductor wafers² or the structural fabrication of lamellar materials³ facing the next generation of chips.

Here, we demonstrated how to control the atomic layer removal for the typical semiconductor and lamellar materials such as single crystal silicon and graphene based on the mechanochemical reactions that occurred in the scanning probe lithography (SPL). On the one hand, the fact that SPL handles single polishing abrasive to simulate the chemical-mechanical polishing (CMP) process helps for exploring the critical factors determining the material removal and then advancing the polishing technology. On the other hand, an SPL-based nanolithography technique was developed to pattern the lamellar materials at nanoscale where the lattice structure at the produced edge can be governed. The findings provide a theoretical foundation and new research avenues for atomic-level nanofabrication processes.

2. Atomic removal of bulk crystal materials

2.1 Key factors impacting atomic removal of bulk Si crystals

Pure mechanical interaction generally induces lattice distortion, slip, dislocations at the atomic scale, plastic deformation, flow, and even crack initiation at the visual level, so it is highly changing to remove the atomic layer through mechanical machining. Mechanochemical reactions occurring at the contact interface have been proven to be one of the most common and effective contributors to weakening the chemical bonds and then facilitating the stripping of atoms from the substrate surface⁴. For instance, through adjusting the mechanochemical synergy, CMP has been developed as a most effective technique to achieve atomic-level precision in polishing silicon-based integrated circuits⁵.

Silicon, one of the typical semiconductor materials, presents significantly different removal/wear behaviors depending on the dominant mechanisms. Under the conditions with pure mechanical interaction (i.e., scratched using inert diamond asperity), material removal only occurs as the applied contact pressure is above the yield stress of crystal silicon (~11 GPa). Otherwise, hillock growth due to amorphization instead of material removal is observed at the low

contact pressures. When the silicon surface is sliding against a chemically active asperity, such as silicon oxide, the mechanochemical reactions at the contact interface of the silicon substrate and silicon oxide asperity will be activated with the association of water molecules contributed from ambient air or liquid water⁶. Then the atomic material can be removed at a contact pressure as low as ~ 0.25 GPa. And most importantly, the Si atoms beneath the removed area keep the perfect crystallographic order⁷.

Combining the above experimental results and the further molecular dynamic (MD) simulations (Fig. 1a), the mechanochemical removal mechanism is deeply revealed⁸. Under a relatively low contact pressure, the Si substrate elastically deforms exerted by a SiO₂ nanoparticle (Fig. 1b) and completely recovers, except a few Si atoms removed from the surface, after the load is released (Fig. 1c). The key process of mechanochemical removal of atomic layers from silicon interfaces can be understood as three stages: (i) generation of surface hydroxyls through the reaction of surface atoms with water molecules (Fig. 1d), (ii) formation of interfacial bridging bonds through the dehydration reaction between two surface hydroxyls at the interface (Fig. 1e), and (iii) mechanical shear under the effect of dissociation of the substrate bonds (Fig. 1f), leading to the removal of Si atoms from the substrate (Fig. 1g). In the third stage, water molecules from the gas phase further react with the newly exposed surface atoms to form Si-OH groups.

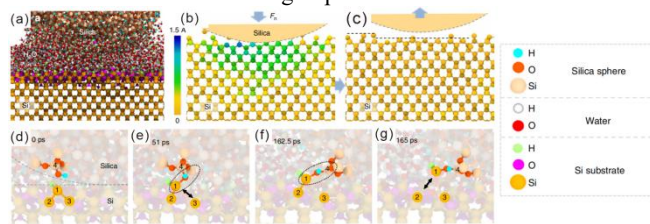


Fig. 1 Atomic removal of Si surface against silica nanosphere in MD simulation⁸. (a) Sliding model in MD simulation. (b) Deformation of Si substrate under load condition. (c) Removal of Si atoms and release of substrate deformation after unloading. (d-g) Mechanochemical reaction process in MD simulation.

Then, the three critical factors for activating mechanochemical reactions on the silicon surfaces can be summarized, involving chemical active counter-face, mechanical interaction and water molecules. The mechanochemical removal occurs at extreme low contact pressures when against reactive SPL probes such as Si, SiO₂, Al₂O₃ and CrO₂ probes rather than the inert diamond and diamond-like carbon coated probes. The atomic removal rate of silicon increases exponentially with the increase of contact pressure, which can be well described based on the stress-assisted chemical reaction kinetics. Through the analysis of the Arrhenius equation⁹, it was confirmed that the inter-interfacial water molecules can significantly lower the critical energy barriers of the mechanochemical reactions, allowing the interfacial shear to effectively activate the interfacial reactions even under the low loadings. The growth of absorbed solid-like water facilitates silicon atom removal; however, a too thick liquid-like water layer uncleared at the two contact surfaces suppresses the formation of an interfacial chemical bridge, resulting in less removal or even no atomic removal at liquid water conditions¹⁰. Otherwise, the chemistry of the silicon

surface strongly impacts the atomic removal. The native oxide layer has a higher activation energy barrier, so atomic removal occurs much more readily when the silicon substrate is exposed¹¹.

The atomic removal of silicon can be controlled through tuning mechanical interaction or chemical reactions. For instance, silicon removal can be enhanced in the higher pH liquid conditions. The size of the abrasive particles significantly influenced the removal process, primarily due to the increased contact area, which enhanced the formation of interfacial siloxane bonds at the Si/SiO₂ interface¹². These bonds are critical in mechanochemical reactions¹³, with their quantity largely determining material removal efficiency. As the contact area expanded, more siloxane bond bridges formed between the two contact surfaces, corresponding to more silicon atom removal. The influence of water temperature on the silicon atom removal is complex depending on the surface chemistry of itself. The mechanochemical removal of the oxide-covered Si underwent a process from no obvious surface damage to significant material removal, but that occurring at all temperatures decreased gradually on the oxide-free Si surface. The opposite temperature dependence can be explained by the growth or degeneration of the Si-OH surficial groups on these two surfaces¹⁴. The tribochemical removal of silicon presented strong crystallography-induced anisotropy. The removal rate is largest on the Si (110) surface, middle on the Si (111) surface, and smallest on the Si (100) surface due to the various atomic planar density and interplanar spacing in different crystal planes¹⁵.

2.2 Atomic layer removal via mechanochemical reactions

It is conceivable that the atomic layer materials can be removed layer-by-layer due to the weak interlaminar Van der Waals force. Here, the controlled mechanochemical reactions at the contact interface offer potential channels to achieve the precise removal of multiple atomic layers or even a single atomic layer from the bulk crystalline surfaces¹⁶. The MD simulations prove this image and visualize this atomic layer process. Under humid air and lower loading conditions, only the Si atoms in the first layer interacting with the adsorbed water molecules and the counter-face can readily undergo chemical reactions and be removed preferentially, while the second layer of atoms is essentially free from mechanical action and still maintains an intact lattice structure if the mechanical interactions can be controlled accurately⁸. Such a mechanism of action makes it possible to achieve high-precision controlled atom layer removal from the silicon surface.

Experimentally, based on the scanning method to manipulate the SPL probe (Fig. 2a), we successfully completed the controlled removal of single or multiple silicon atomic layers by tuning the main influence factors such as mechanical stress, scanning speed, and chemical reactions with quantitative water-associated action (Fig. 2b). High-resolution transmission electron microscopy (HRTEM) provides direct evidence that the outermost atomic layers of the silicon (100) surface were removed without any damage or defects to the underlying subsurface structure. This is accomplished through SPL tip-assisted etching, a maskless and chemical-free nanolithography technique capable of directly etching single-crystalline Si wafers.

Moreover, this method can be extended to other materials that

undergo shear-induced mechanochemical reactions. We conducted nano-removal experiments on the CaF_2 (111) surface using SiO_2 tips in a PH-controlled liquid bath to investigate the effect of solution pH on atomic layer removal¹⁷. They discovered that pH significantly impacts the surface wear of CaF_2 , and that under optimal pH conditions (~ 9.5), SiO_2 probes can achieve the removal of a single atomic layer on the CaF_2 (111) surface without causing corrosion-induced wear or subsurface crystal structure damage due to mechanical action. The related results are presented in Fig. 2c. Then, we optimized the polishing slurry based on the obtained optimum pH condition and reduced the surface roughness down to the atomic level.

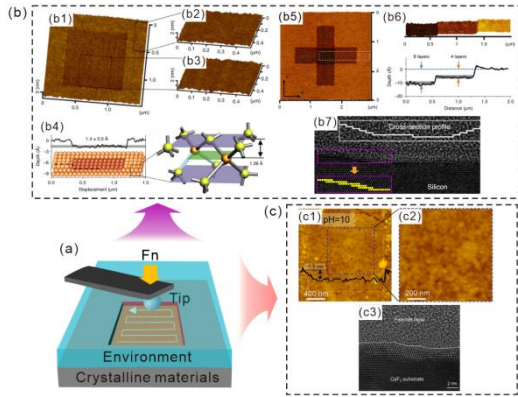


Fig. 2 (a) Schematic of SPL tip-assisted etching. (b) Single-layer or multilayer removals of silicon atoms by scanning⁸. (b1-4) SPM image and corresponding cross-section profile of the single atomic layer removal on the Si (100) surface. (b5-6) SPM images and corresponding cross-section profile of the multilayer removal. (b7) Cross-section TEM image of the mechanochemical removal region. (c) Single-layer atomic removal of CaF_2 surfaces scanning against SiO_2 tip under optimal pH¹⁷. (c1) Surface topography of CaF_2 surfaces after area scanning in KOH solution. (c2) AFM images of the scanning regions marked by blue dotted line frames in (c1). (c3) TEM image of the cross-section of the scanning area.

3. Controlled structural machining of lamellar materials via mechanochemical reactions

3.1 Atomic removal mechanism of lamellar materials

The applications of lamellar materials in microelectronics need the manufacturing of tiny structures in which processes the removal precise down to atomic layer level and the atomic structure of the fabricated edge normally should be controlled. Studies have shown that several properties of lamellar materials, such as friction, fatigue, and mechanical strength, are closely tied to their thickness or the number of layers they possess¹⁸. Wear caused by surface defects or atomic step edges continues to pose significant challenges in practical applications¹⁹. Consequently, understanding the main factors to determine the atomic layer removal in lamellar materials and the corresponding mechanisms is crucial.

The dependence of thickness (number of layers) on the atomic removal was found to be nonmonotonic depending on the type of lamellar materials and the surface chemistry of the substrate. For pure graphene, atomic layer removal becomes more difficult as the thickness goes up. In comparison, the fluorinated graphene (FG) nanosheets deposited on a hydrophilic silicon surface are removed

readily as the FG thickness changes from monolayer to 7 layers²⁰. Density functional theory (DFT) simulations reveal that this inverse thickness dependence is mainly attributed to the changes in the energy of FG when attached to the substrate. Substrates with higher surface energy increase the interfacial adhesion energy between the FG monolayer and the substrate, thereby reducing the structural flexibility of the FG layer, leading to less fatigue during reciprocating frictional cycles and ultimately improving wear resistance. The graphene oxide (GO) also presents the similar inverse dependence of thickness on atomic layer removal as the thickness increased to 4 layers²¹. For thicker GO, the critical load corresponding to the removal initiation increases again for the larger number of layers (Fig. 3a-b). The removal of GO is strongly influenced by the interfacial interaction with the substrate. Increasing the surface energy of the substrate enhances the adhesion energy between the GO layers and the substrate, thereby increasing the removal resistance (Fig. 3c-d). When a hydrophobic substrate with much lower surface energy is applied to deposit the GO nanosheets, the removal occurs readily, and only positive thickness dependence can be observed.

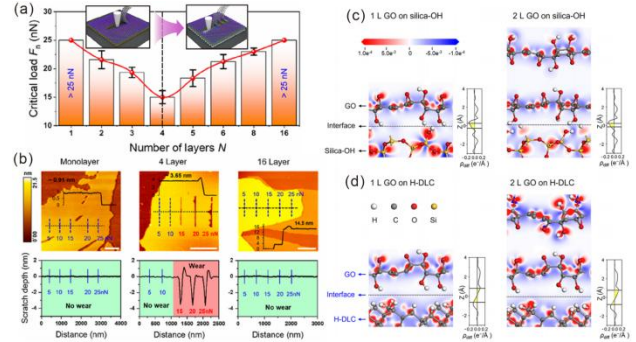


Fig. 3 Nanowear of few GO layers on Si substrate²¹. (a) Critical loads for nanowear initiation on GO as a function of the number of layers. (b) Topographies and the corresponding cross-section profiles of GO with different thicknesses after wear testing at applied loads ranging between 5 and 25 nN. (c-d) Thickness- and substrate-dependent interfacial interactions of GO determined by DFT simulations.

The atomic layer removal of graphene monolayer was detected to be strongly impacted by the chemical activity of the probe surface. Unlike purely mechanical damage (fracture and folding) caused by diamond probes, Si probes remove atomic layers from the edges of graphene primarily by inducing mechanochemical reactions at the interface²². This mechanism facilitates the easier removal of atomic layers from the graphene surface (Fig. 4b). DFT analysis further elucidated the mechanism of graphene atomic layer removal under mechanochemical interactions (Figs. 4g-h): compared to the pure mechanical interactions at the diamond/graphene interface, the Si tip preferentially binds to graphene's step edges, forming Si-O-C bond bridges with lower energy barriers. This promotes C-C bond breakage under mechanical stress, allowing for the atom-by-atom removal of graphene at the step edges (Fig. 4i).

It suggests that the atomic layer removal of 2D materials can be effectively achieved under minimal mechanical stress by creating highly chemically active interfaces. Under such conditions, atom removal is predominantly driven by interfacial bonding. However, it is worth noting that the influence of environmental factors on mechanochemical removal processes should not be overlooked in this

case. Ambient oxygen had a particularly significant impact, and its presence notably accelerated atomic layer removal from graphene. It can attributed this phenomenon to the combined effect of interfacial bonding and oxygen atom cleavage²³. In an oxygen-rich environment, oxidation originating from the graphene edge caused deformation or destruction of the honeycomb structure, and its C network was further distorted by the Si-O-C interfacial bonds. Moreover, the Si-O-C bonds enable to the transmission more mechanical energy from the AFM probe to the graphene layer, facilitating the atomic removal of graphene.

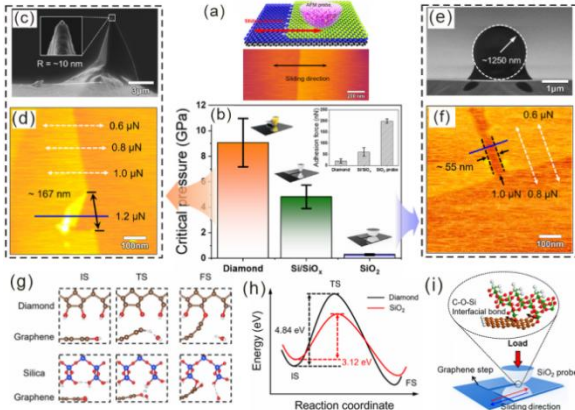


Fig. 4 (a) Schematic diagram and topography of the lateral force measured at the graphene step edge with an AFM probe. (b) Critical contact pressures for nanowear at the graphene step edges by diamond, Si/SiO_x, and SiO₂ probes. The inset shows the adhesion forces measured at the interfaces between the graphene substrate and probes. SPL images of graphene damage produced by the nanowear processes of diamond probes (c-d), and SiO₂ probe (e-f). (g) Results of the DFT calculation of the formation energy of bond bridge for the diamond/graphene interface and SiO₂/graphene interface. (h) Potential energy along the reaction coordinates leading to the formation of interfacial bond bridges at the graphene step edge scratched by diamond and SiO₂ probes. (i) Schematic diagram of the mechanochemically stimulated atomic attrition mechanism at the tribological interface of the SiO₂ probe and monolayer graphene substrate²³.

3.2 Nanostructure fabrication of lamellar materials via mechanochemical reactions

Nanofabrication of these nanoelectronic devices that rely on various nanostructures remains challenging due to the ultra-high mechanical strength and chemical inertness of lamellar materials. However, SPL-assisted etching via shear-induced mechanochemical reactions offers a promising solution, enabling precise atomic layer removal from the surfaces of these materials under low loadings.

Based on the above experimental results, we developed a scanning probe lithography (SPL) method facilitated by a mechanochemical atomic attrition process for fabrication of nanopatterns into single-layer graphene on the graphite basal plane along the crystallographic orientation of zigzag and armchair edges without mechanical damages in the surrounding area²⁴ (Fig. 5a). This SPL-based mechanochemical etching method will enable the construction of various nanodevice structures with specific edge orientations, which allows exploiting their electronic properties. Based on the mechanochemical reactions, atomic layer removal initiating from step edge in this technology allows to directly fabricate nanoscale structures for graphene, which has extreme high

mechanical strength and is difficult for machining. This technology can be applied to directly fabricate various GO nanopatterns, such as triangular-wave, square-wave nano-channels, step nano-channels, and nano-channel arrays (Fig. 5b), both at the step edge and on the in-plane surface²⁵. After that, deoxygenated GO nanostructures with complete edge structures can be prepared by high-temperature annealing (Fig. 5c). These works have a wide range of applications in graphene-based micro/nanodevices, sensors, and gas detection systems based on a wide range of nanostructures²⁶. For instance, the fabricated structures can be applied to gas sensors, and the sensitivity to detect gas adsorption for the structure with a zigzag structure is several times higher than that with an armchair structure.

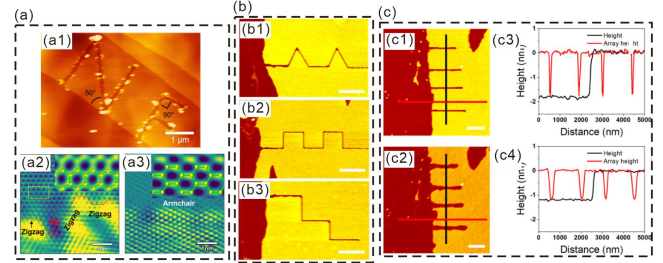


Fig. 5 (a) Nanostructures fabricated in single-layer graphene by mechanochemistry-associated SPL²⁴. (a1) Nano-pattern of alphabet N and F engraved in the graphene sheet. (a2-3) High-resolution c-AFM images of the step edges of the nanochannels shown in (a1). (b) Nanostructures fabricated on the GO layer. Nanochannels fabricated: (b1) triangular wave nanochannel, (b2) square wave nanochannel, (b3) step nanochannel. (c) Deoxygenated GO nanostructures with complete edge structures prepared by high-temperature annealing: Nanochannel array before (c1) and after (c2) high-temperature annealing. Height profiles of the GO nanochannel array measured before (c3) and after (c4) high-temperature annealing²⁵.

4. Conclusion

The ultimate precision in the development of nanofabrication of chips is to accomplish precise morphological control down to the atomic level, and the understanding of the atomic layer removal mechanism of crystalline materials is particularly important under this processing precision. Our works systematically investigate the atomic level removal behavior of typical crystalline materials (e.g., silicon, graphene) under the influence of different factors, and reveal the key role of interfacial mechanochemical reactions in the atomic level removal, which can achieve single-layer atomic removal from the surface of the materials through interfacial bonding interactions. Additionally, we demonstrate an SPL tip-assisted, mask-free, and chemical-free nanolithography technique that enables precise atomic removal in specific regions via shear-induced mechanochemical reactions, while maintaining the structural integrity of the crystal subsurface. This method holds significant promise for ultra-precision polishing of bulk crystal surfaces or the nanoscale patterning of two-dimensional materials.

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