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Description	

A Si nanopillar grown on a Si tip by atomic force microscopy in ultrahigh vacuum for a high-quality scanning probe

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We grow a Si nanopillar on a commercial Si tip on an atomic force microscopy (AFM) cantilever using AFM in ultrahigh vacuum for a high-quality scanning force probe, and observe noncontact-AFM (nc-AFM) images of Si(111)7 × 7 and Ge deposited Si(111) with the nanopillar. We observe it *ex situ* by transmission electron microscopy to confirm its growth and crystallinity. The nc-AFM image clearly showed the high performance of the nanopillar as a probe with respect to the spatial resolution, image stability, and reproducibility. This nanopillar growth technique can elongate the lifetime of the cantilever and be applied to other materials. © *2005 American Institute of Physics*. [DOI: 10.1063/1.1866213]

For the last decade noncontact atomic force microscopy (nc-AFM) has successfully demonstrated the great imaging capability with atomic resolution for various sample surfaces, including insulators.[1,2] Additionally the nc-AFM has excellent features for surface spectroscopy utilizing the force between a nc-AFM tip and a sample surface. For example, by changing a separation and/or a bias voltage between the tip and the sample, we can simultaneously obtain the characteristics of the force and the tunneling current with atomic resolution, including two-dimensional maps of these values.[3–7] This nc-AFM performance is of great significance from surface analytical viewpoints of topography and electronic states. Moreover, recently the atom manipulation has been achieved by the nc-AFM.[8]

On the other hand, we have to take great care of the atomic structure and composition of the tip for surface imaging, force spectroscopy, and atom manipulation with atomic resolution, because the force interaction is crucially governed by the electronic states on the tip apex on an atomic scale. Furthermore, since the van der Waals interaction acts over several tens of nanometers, the total force between the tip and the sample depends on the shape and composition of the tip shank as well as the tip apex. Since, in general, commercially available [001]-oriented Si tips are used as a nc-AFM tip, which may be contaminated or blunted during transfer in air, special tip preparation techniques before nc-AFM scanning are really required to acquire the atom-resolved images and measurements with high stability and reproducibility.

In this letter, in order to prepare a well-defined Si tip, we present a nano-scale Si crystal pillar epitaxially grown on the commercial Si tip by retracting the tip after touching it to a heated Si wafer using the AFM in ultrahigh vacuum (UHV). This method is similar to the so-called crystal pulling method for macro-scale crystal growth (but not to the scale). The apex should be surrounded with atomically flat facets in a thermodynamically stable manner after its crystal growth in UHV. Thus this apex is expected to have a sharp corner at a facet-meeting point in specified crystal orientations, though the reconstruction on a small top (001) facet of [001]-oriented Si tip has not been evaluated yet to our knowledge. Here the apex is observed by transmission electron microscopy (TEM), and nc-AFM images are stably obtained with the nanopillar on an atomic scale.

A homemade nc-AFM/STM operated in 5×10^{-11} Torr utilizing a piezoresistive Si cantilever with a [001]-oriented Si tip (Veeco) was used.[9] The resonance frequency and the spring constant of cantilever were 185 kHz and 9 N/m, respectively. Since the Si tip, as supplied through air, was apparently oxidized and contaminated, it was cleaned by Ar ion sputtering and heating in an UHV chamber of a scanning Auger electron microscope (ULVAC-PHI SAM670) and evaluated with the SAM;[10] the cantilever can easily be heated by passing a small current into the piezoresistor up to about 600 °C. After forming a clean thin Si oxide layer on it as a protection layer by admitting pure oxygen gas into the SAM chamber ($\sim 10^{-5}$ Torr) and heating it at about 600 °C, we transferred it from the SAM to the nc-AFM through air. Subsequently it was heated at about 600 °C in the nc-AFM chamber to remove the thin oxide layer before nc-AFM scanning.

As a Si source material for Si nanopillar growth and a test sample for nc-AFM observation, a cut of *n*-type Si(111) wafer was used, which was degassed in UHV at 600 °C for 12 h and flashed at 1200 °C by direct current heating to clean it. After the sample was cooled to room temperature, we confirmed the Si(111)7 × 7 reconstruction by the nc-AFM on an atomic scale. After the sample temperature was raised to about 570 °C in the nc-AFM head, the Si tip on the cantilever, which was not intentionally heated, was brought close to the heated Si(111) sample [Fig. 1(a)], and slowly touched it using a fine positioning mechanism of the nc-AFM without any intentional excitation of cantilever oscillation for nc-AFM imaging [Fig. 1(b)]. Subsequently, the tip was slowly retracted from the sample to grow a Si nanopillar

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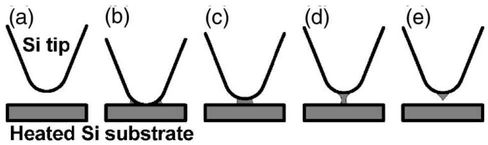

FIG. 1. Schematic diagram of a Si nanopillar growth on a Si tip by AFM in UHV. The Si substrate was heated at about 570 °C.

as a neck between the tip and the sample [Fig. 1(c)]. While bringing and retracting the tip, the current passing between the tip and the sample was being monitored with a current amplifier connected to the tip. We applied a voltage of about −3 V to the tip with respect to the potential of the center of sample, which was half of the voltage applied across the sample for heating. When the tip touches the Si substrate, the current of the order of 10 nA passes roughly in proportion to the contact area. Since the substrate is being heated, the Si atoms on the substrate surface in almost surface melting are easily mobile, and furthermore, the Si atoms are pulled toward the tip due to the electric effects induced by an applied bias voltage between the tip and the substrate. Thus, after the tip touches the substrate, Si atoms near the contact on the substrate move to the Si tip, leading to increase in the contact area, exhibiting increasing current. By slowly retracting the tip under a tension of sub-microNewtons through the cantilever bending, the contact area decreases gradually [Fig. 1(d)], and at last, the contact neck between the tip and the substrate is broken, resulting in a Si nanopillar on the Si tip [Fig. 1(e)].

The Si cantilever with the nanopillar was taken out of the nc-AFM chamber, and the Si tip was observed using a scanning electron microscope (SEM) (Hitachi S-5200), as shown in Fig. 2(a); we did not find any notable changes at the tip apex. Next the cantilever was cut in air to fit into a sample holder of TEM (Hitachi H9000). Figure 2(b) shows a TEM image of the grown Si nanopillar on the Si tip. The lattice fringes of (111) plane look continuous from the original Si tip to the grown nanopillar, slightly covered with an amorphous carbon layer. Since the Si nanopillar was grown and annealed in UHV, it is estimated that the tip apex was clean in the nc-AFM chamber. The lattice fringes indicate that the nanopillar grew to the [001] direction with a length of ~10 nm, a radius of ~5 nm, and a good crystallinity. Under well-controlled conditions, a longer nanopillar can be grown: SEM observation, not shown here, revealed the longer nanopillar growth.

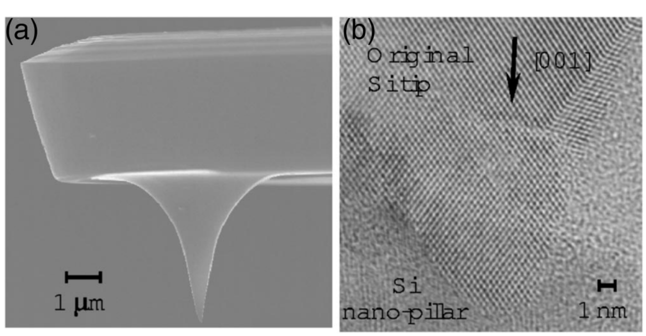

FIG. 2. (a) SEM image of a Si tip on the end of a piezoresisitive Si cantilever, taken after Si nanopillar growth. (b) TEM image of the grown Si nanopillar on the Si tip at 300 keV. The fringes correspond to the (111) plane of Si diamond structure.

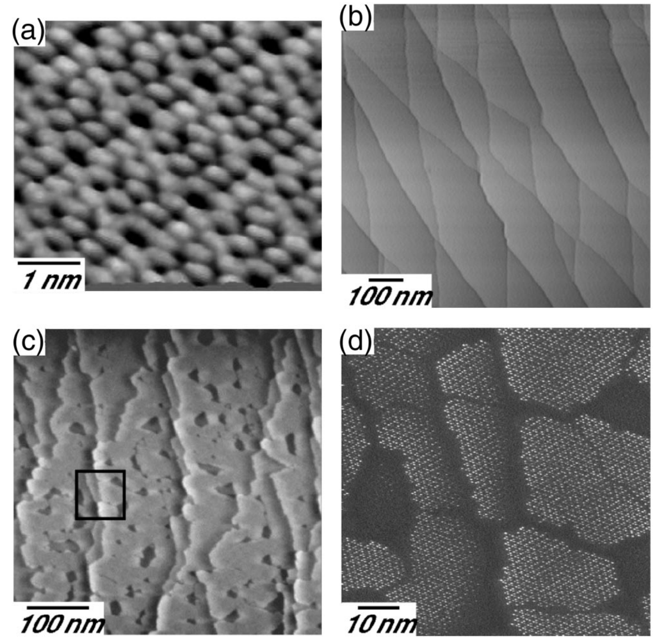

FIG. 3. nc-AFM images using a Si nanopillar for Si(111)7×7 surface (a), (b), and for Si(111)7×7 covered with 2–3 BL Ge (c), (d). (a) Scanning area: 5 nm$\times$5 nm. Imaging conditions: the resonance frequency shift Δf: −18.2 Hz, the oscillation amplitude of cantilever A:20 nm, and the sample voltage V_s:−1.0 V. (b) 900 nm$\times$900 nm. Δf:−12.5 Hz. The scanning time was about 3 h. (c) 500 nm$\times$500 nm. Δf:−13.5 Hz, A:20 nm, and V_s: −1.0 V. The scanning time was about 2 h. (d) 77 nm$\times$77 nm. Δf: −16.3 Hz. The scanning time was about 30 min.

By using a Si nanopillar we have observed the nc-AFM images under the conditions of a constant resonance frequency shift of the cantilever and a constant oscillation amplitude of the cantilever at several bias voltages between the tip and the sample. In general, after a Si tip is used for a long period in critical conditions or accidentally crashed with a sample, the atomic sharpness of the tip is degraded or lost. Then the resonance frequency shift to be set for imaging should be increased to obtain an atomic resolution, for example, from a few hertz to 1 kHz. This means that the tip becomes blunt and the tip should be brought closer to a sample to enhance the chemical interaction between a tip apex atom and a sample atom against the van der Waals interaction between the bulky tip and the sample. However, by growing a Si nanopillar as we propose, the resonance frequency shift for atom imaging decreases. This means that the sharpness of the Si tip can be restored and used for a long time.

Figures 3(a) and 3(b) show nc-AFM images of Si(111)7×7 taken with the Si nanopillar. Then the atomic resolution was stably obtained as in Fig. 3(a), and a wide area scanning over several hours was also stably performed as in Fig. 3(b) without tip-induced artifacts. According to our experience using the Si nanopillar as a scanning probe, the image noise due to tip apex instability decreases dramatically and the images are really reproducible with atomic resolution. Figures 3(c) and 3(d) show other examples of nc-AFM images of a Ge deposited Si(111)7×7 surface, taken with the Si nanopillar grown using a clean Si substrate in advance. The Ge on Si was deposited at a substrate temperature of 450 °C with a Ge coverage of 2–3 bilayers (BL). Figure 3(c) shows a wide scanning image of the Ge overlayer on a surface of Si(111) with terraces and steps. Although the surface is rugged with triangle depression regions surrounded by Ge step-flowing overlayers and Ge islands, the nc-AFM image was stably obtained. When we decreased the scanning area, we obtained an atom-resolved image as shown in Fig. 3(d), where the Ge atoms reconstructed in 7×7 on the top terrace. The area corresponds to the middle part denoted by a rectangle in Fig. 3(c). Under the imaging conditions described in the caption of Fig. 3, the Ge atoms at corner adatom sites in faulted half unit cell were clearly enhanced with atomic resolution.[11] We can also find the domain boundaries and deep trenches on the terraces with a high image quality. These images seem to demonstrate the power of the Si nanopillar as a scanning force probe. On the other hand, the atomic arrangement on top of the nanopillar is not clear, because appropriate analytical methods are not available. At present we are undertaking the evaluation of the atomic arrangement by comparing slight change in atomic contrast in nc-AFM images depending on the imaging conditions, which will be published elsewhere.

In summary, we grew a Si nanopillar on a commercial Si tip on an AFM cantilever using AFM in UHV, and observed nc-AFM images of a Si(111)7×7 surface and a Ge deposited Si(111)7×7 using the *in situ* grown Si nanopillar. The nc-AFM images clearly showed the high performance of the nanopillar as a scanning probe with respect to the spatial resolution, the image stability, and reproducibility for a long time. This technique can greatly elongate the lifetime of the cantilever. Furthermore, it is noted that this technique can be used to fabricate a nanopillar of other materials than Si.

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