Revisiting Surface Conditions of H/Si(111) after Wet-Chemical Treatment through Different SPM Modes

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High-performance solution processing is essential for next-generation electronic and optical devices, and Scanning Probe Microscopy (SPM) enables atomic-scale evaluation. This report aims to characterize wet-treated Si(111) surfaces in detail using multiple SPM modes.

Si(111) surfaces were treated by anisotropic etching in NH_4F [1]. These surfaces were first imaged in air by conventional amplitude-modulated Atomic Force Microscopy (AFM). Figure 1(a) shows a familiar step/terrace structure. The same surface was then examined by noncontact AFM (nc-AFM) and Scanning Tunneling Microscopy (STM). The nc-AFM/STM hybrid system was operated at room temperature under ultrahigh-vacuum conditions of 10^{-8}–10^{-7} Pa. For nc-AFM, the oscillation amplitude and frequency shift were set to 1.0 nm and −1.0 Hz, respectively, and for STM, the sample bias and tunneling current were −2.0 V and 0.6 pA. It is evident that the nc-AFM image in Fig. 1(b) resolves smaller particles or adsorbates with much higher resolution than Fig. 1(a). More importantly, these small particles visible in Fig. 1(b) are absent in the STM image in Fig. 1(c). A subsequent nc-AFM scan of the same area after acquiring Fig. 1(c) reproduced the result in Fig. 1(b), confirming that Fig. 1(c) does not arise from probe-induced manipulation of adsorbates but rather from the different imaging mechanisms of nc-AFM and STM.

The X-ray Photoelectron Spectroscopy (XPS) spectrum in Fig. 2, obtained with the same sample, reveals carbon and oxygen signals in addition to Si. This indicates that the small particles in the nc-AFM image in Fig. 1(b) likely correspond to organic contaminants or water molecules with low conductivity. Their probable origin is sample preparation in air, ranging from the NH_4F treatment itself to the subsequent transfer process. In contrast, the fine particles observed in the STM image in Fig. 1(c) are attributed to Si dissolved in NH_4F and redeposited on the surface. These results demonstrate that the combination of nc-AFM and STM provides complementary insights into the molecular-level distribution of adsorbates, which will be valuable for advancing solution-based processes such as wet cleaning.

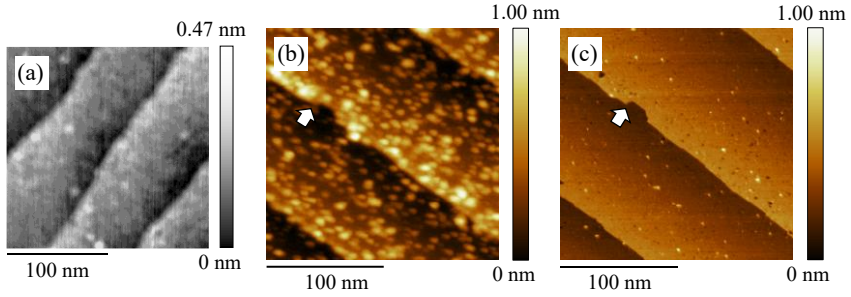

Fig.1. (a) Amplitude-modulation AFM, (b) nc-AFM, and (c) STM images of wet-treated H/Si(111) surface. The arrows indicate the same locations.

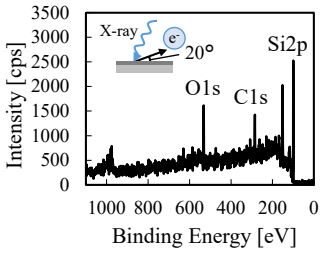

Fig.2. Survey scan of treated Si surfaces.

[1] G. S. Higashi, *Appl. Phys. Lett.* **58**, 1656 (1991).

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Supplementary Information

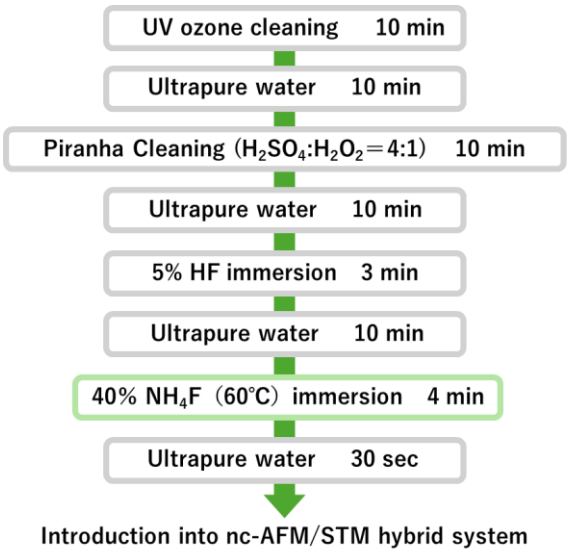

Fig.3. Flowchart of sample preparation method