BL-7C/ 2016G109

Improvements in data acquisition and processing toward XANAM imaging

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1. Introduction

Scanning probe microscopy (SPM) provides structural information about surfaces on flat substrates. In addition, as a spectroscopy using a probe, it is also used for analysis of surface physical properties and chemical properties. Especially, for the noncontact atomic force microscopy (NC-AFM), chemical information can be also obtained by force spectroscopy with theoretical analysis of the force components between the tip and the sample surface [1]. The method excellently showed a new possibility of the SPM and NC-AFM. To make it easier-to-use, we proposed a methodology of NC-AFM combined with synchrotron radiation (SR) X-ray to obtain data for chemical information on the surfaces using fundamental knowledge of X-ray elemental analysis, named "X-ray aided noncontact atomic force microscopy (XANAM)". Using the SR X-ray, the core-level electrons can be excited at energies of X-ray absorption edge for the sample surfaces. On the other hand, the presence of covalent bonding between the tip and the sample surface is commonly well-known. From the those, we proposed that the covalent bonding created between the tip and the sample surface can be used for the detection. If the electron excitation alters electron density in the covalent bond, the force between the tip and the surface can be changed, i.e., the electron excitation is detected by the AFM-tip, the atom species directly beneath the tip can be identified (Fig. 1). In the previous reports [2], X-ray energy dependency of the on a gold (Au) partially deposited silicon (Si) surface showed the clear different spectral features on Au and Si regions near the Au-L3 absorption edge energy. Besides, we confirmed the existence of covalent bonding force and found its strength was weakened by X-rays with energy near the Au - L 3 absorption edge. Although the above method was a spectroscopy on a fixed point on the surface, we also reported that it could be extended to two dimensions, providing the results comparable to the NC-AFM image data on the surface. However, the procedures have not been well-established. In this report, recent progress in the improvement of experimental procedures for XANAM imaging.

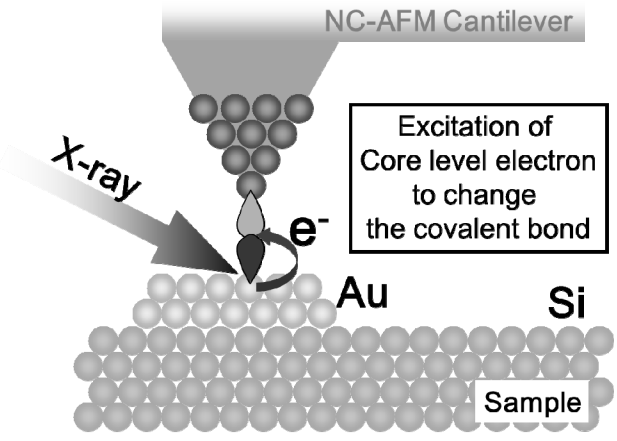

Fig. 1 Basic concept of XANAM

2. Experimental

The experiments were performed at BL-7C of the Photon Factory, Institute of Material Structure Science, High Energy Accelerator Organization (KEK-IMS-PF). A non-contact atomic force microscope was operated in a home-build ultrahigh vacuum (UHV) chamber equipped with two beryllium windows for X-ray penetration through the chamber. The microscope stage had four kinds of freedom to set the sample surface on the X-ray pass: X-, Y- and Z- movements and a rotation around vertical (Z) axis. We focused an X-ray beam on the sample position by a sagittal focusing of Si(111) double-crystal monochromator. In this measurement, a Au deposited substrate was used as a sample. An AFM-probe was fabricated by using a quartz tuning fork and an electrochemically-etched tiny tungsten (W) tip with the similar configuration of the qPlus sensor [3]. The typical resonance frequency of the sensor was ~29.3 kHz, respectively. As the force signal, frequency shift (Δf) from the resonance frequency was recorded. All the measurements were carried out with irradiating the X-ray beam with energies around the Au-L3 absorption edge energy. The data was recorded by Nanonis control systems (SPECS Zurich GmbH). The data was processed by the programs coded by Mathematica language (Wolfram).

3. Results and Discussion

Fig. 2(a) and (b) show results Δf on the surface under X-ray irradiation tuned at 11869 eV and 11923 eV, respectively. To obtain these data, the force spectroscopy measurements, the distance dependency of Δf was recorded in two-dimensionally on a Au deposited part in the same region. Because the Au region was a deposited part, it's height was higher than that of the underlying Si region. The height difference was corrected by shifting the data origin in each force spectrum. In this case, the origin was defined as the point where the amplitude of the sensor began to decrease in each spectrum. Hence, the height difference between Au and Si regions could be apparently cancelled. However, distinctive contrast differences between two Δf images were observed in the range of -45 ~ -17 Hz. The lower values indicated stronger attractive force. The contrast of Au region in Fig. 2(a) was brighter than that of Si region, while the contrasts in two regions became ambiguous in Fig. 2(b) as much as the Fig. 2(a). The X-ray energy for the former and the latter cases were 11869 eV and 11923 eV, respectively. Because the change in the contrasts of images appeared around the Au-L3 absorption edge of 11919 eV, it was proved that the Au regions were identified by XANAM using the Au-L3 absorption edge energy. At this stage, because the changes in a sub-micrometer scale of the Au region could be observed clearly, several hundred of spatial resolution could be estimated as the microscopic usage of XANAM. To obtain further clearer contrast changes, surface structural factors of height difference should be eliminated more precisely for estimation of the spatial resolution.

4. Conclusion

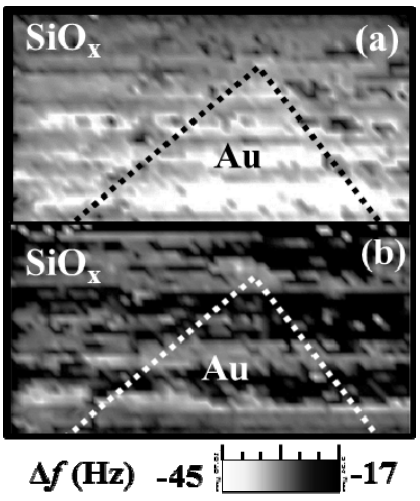

Fig. 2 XANAM images obtained by force spectroscopy measurements on the partially Au-deposited Si substrate surface under X-ray irradiation with the X-ray energy of (a) 11869 eV and (b) 11923 eV. The frequency shift data were recorded in the area of 1×2 μm^2.

We have developed XANAM to obtain elemental information on surfaces at the nanoscale using NC-AFM combined with SR X-ray. To obtain image data of XANAM for estimation of the spatial resolution, we adopted the two-dimensional force spectroscopy measurements as XANAM imaging under X-ray irradiation. Comparing with the two images with two X-ray energies of 11869 eV and 11923 eV, the force changes in the Au region was imaged with several hundred of spatial resolution.

Acknowledgements

This work was partially supported by Murata Science Foundation and Tatematsu Foundation, as well as the previous grants from the JSPS, JST, and NEDO. The experiments were carried out under the approval of Photon Factory Advisory Committee (PAC: Proposal No. 2012G099, 2014G114, and 2016G109).

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