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Surface elemental imaging of Ge quantum dots on a Si surface by XANAM

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1. Introduction

In recent years, the importance of surface/interface elemental analysis at the single-digit nanoscale has increased with the demand for the application of single atoms, clusters, and nanocrystals to electronic devices. Up to date, we developed X-ray aided noncontact atomic force microscopy (XANAM), scanning probe microscopy combined with synchrotron X-ray designed for elemental analysis at the nanoscale. Previously, NC-AFM studies showed that the tip-surface force involves a component of covalent bond force in addition to van der Waals force, electrostatic force. According to the idea, we expected that the covalent bond force could be a sign of an elemental species on surfaces, with changing its force strength by X-ray absorption, at which electron transition from a core-level of sample's atom to the electron states in the covalent bond. (Fig. 1). We reported the presence of the X-ray induced change on the surfaces of gold, nickel, and germanium (Ge) by irradiating the X-rays tuned at the corresponding X-ray absorption edge energies of Au-L3, Ni-K, and Ge-K, respectively [1]. Further, the components of the tip-surface force were analyzed by the calculations based on the NC-AFM theory. The results indicated that at least two force components of the covalent bond force and the electrostatic force were involved in the signals, so that we thought it proved at last, the above hypothesis that the X-ray induced change could be used for the elemental analysis.

However, there has been an issue with the spatial resolution of this technique, XANAM as microscopy. The lower limit of the spatial resolution has not been clear yet due to the less measurement efficiency to acquire enough data points. The evidence is essential for future applications of XANAM to the research fields of semiconductors.

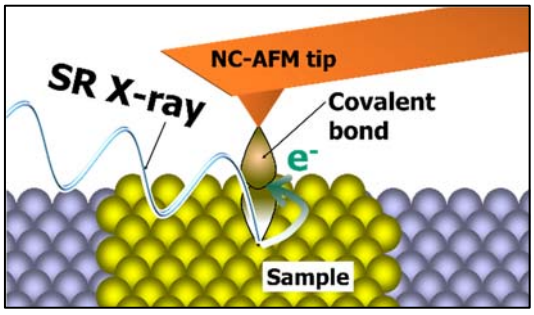

Fig. 1 Basic concept of XANAM

Here, we applied XANAM to Ge quantum dots prepared on a silicon substrate to examine the lower limit of the spatial resolution 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 noncontact atomic force microscope was operated in an ultrahigh vacuum (UHV) chamber equipped with beryllium windows for X-ray penetration. The data was recorded by Nanonis control systems (SPECS Zurich GmbH). An AFM-probe was fabricated by using a quartz tuning fork with a similar configuration of the qPlus sensor [2]. The typical resonance frequency of the sensor was ~30 kHz. As the force signal, frequency shift (Δf) of the AFM-probe from the resonance frequency was recorded. All the measurements were carried out under X-rays with the energies around the Ge-K absorption edge. The data was recorded by Nanonis control systems (Nanonis, SPECS Zurich GmbH).

3. Results and Discussion

XANAM imaging was carried out on Ge quantum dots on a Si substrate. Variation of the force in three-dimensions was measured by force volume spectroscopy, which is composed of the spectroscopic data of tip-surface force taken in two-dimensions on the surface. The data resolution in the horizontal direction was determined by the number of points at which the spectroscopy of Δf was

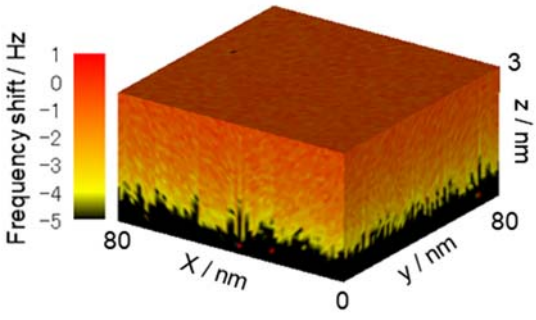

Fig. 2 A force volume data of Δf (x, y, z) obtained for Ge quantum dots (X-ray energy: 11103 eV, Volume size: 80 × 80 × 3.0 nm^3, Δf range: -5 ~ +1 Hz)

measured. In surface normal, the data resolution was the same as the data points in the tip sweep direction.

Figure 2 shows a force volume data of Δf (x, y, z) obtained for Ge quantum dots obtained at the beamline under X-rays tuned at the Ge-K absorption edge energy of 11103 eV. The data resolutions in the horizontal and vertical directions are 1.25 nm/points and 0.1 nm/points, respectively. The volume size of data is $80 \times 80 \times 3.0$ nm^3. Compared with the previous experiments, the improved-acquisition rate of the force spectroscopy was about four times, and the recording time par data was 40 min. The color bar indicates the magnitude of Δf within the rage of -5 - +1 Hz. Horizontal images could be extracted at each point along the vertical direction, resulting in 30 slices of XANAM images from this volume data.

Figure 3 shows images of possibly two quantum dots regenerated from the volume data at the tip-surface separation (z) within the range of 0.4-1.2 nm. The image size is 40×40 nm^2. The images of (a), (b), (c), and (d) were obtained under X-ray irradiation with the energies of 11053 eV, 11103 eV, 11153 eV, and 11203 eV, respectively. The bright region was observed at the center in each image and was assigned to the quantum dots. The dark region around them was assigned to the bare SiO_x surface. We found that the topography of quantum dots was modulated by increasing the X-energy in the separation range of 0.4-0.7 nm. Compared with the images for 11053 eV and 11203 eV, the topography in images for 11103 eV and 11153 eV indicated small changes in its shape. However, the topography in the far region, more than 0.8 nm, it showed changes depending on the X-ray energy became ambiguous. In the far region, the electrostatic force is dominant, compared with the covalent bond force. Thus, it could be deduced under our hypothesis that the change in the covalent bond force was covered by the variation in the electrostatic force, resulting in the vague change.

Consequently, we found that the X-ray energy dependence of the force change at the nanoscale on the Ge quantum dots. By estimated from the data resolution, we thought that the spatial resolution of XANAM was achieved to at least a few nanometer scale.

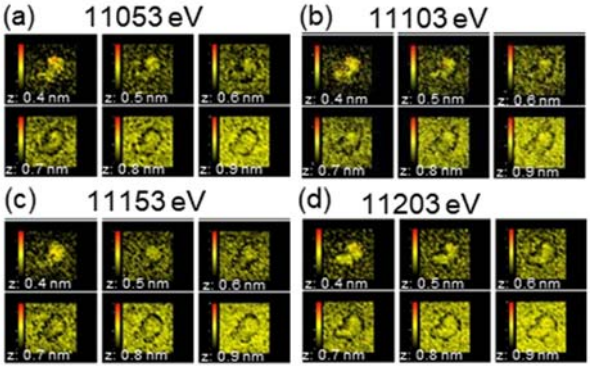

Fig. 3 Extracted-Δf (x, y) images of quantum dots prepared on a Si substrate obtained at X-ray energies of (a)11053 eV, (b)11103 eV, (c)11153 eV, and (d)11203 eV. (Image size: 40×40 nm^2, Δf range: -5 ~ +1 Hz)

4. Conclusion

We have developed XANAM to obtain elemental information on surfaces at the nanoscale using NC-AFM combined with SR X-ray. To evaluate the details of X-ray induced force change and the lower limit of the spatial resolution of XANAM as microscopy, we observed the Ge quantum dots on the Si substrate under X-ray irradiation. From the force volume date in three dimensions, the XANAM images of the Ge quantum dots were obtained. Further, the X-ray energy dependence of the images was observed, as observed in the spectroscopic data in the previous studies [1]. We concluded that tentatively the spatial resolution of XANAM could be achieved at least a few nanometer scale.

Acknowledgments

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