Nanoscale elemental analysis and applications using STM combined with brilliant hard X-rays

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Analyses by scanning tunneling microscopy (STM) combined with brilliant X-rays from synchrotron radiation (SR) can provide various possibilities of original and important applications. The STM observation under inner-shell excitation at a specific core-level enables us to analyze the elements or control the local reaction with the high spatial resolution of STM [1].

We have recently demonstrated the elemental analyses with a spatial resolution lower than 2 nm on semiconductor surfaces [2]. The principle of our analyses is not to collect the secondary electrons by STM tip (that may damage the spatial resolution), but to extract the element-specific modulation of the "tunneling current" succeeding the core-excitation process, which contains truly local information. A key to accomplish successful results is to effectively increase the signal to noise (S/N) ratio. On this purpose, we developed a special SR-STM system.

The experimental setup is shown in Fig.1. To surmount a tiny core-excitation efficiency by hard X-rays, we focused two-dimensionally an incident beam having the highest photon density at the SPring-8. Many problems derived from the high brilliance (thermal and electrical noise, damage of STM scanner, instability such as thermal drift, etc.) were solved by the special apparatus and system [1]. Furthermore, we developed a special tip [3] (that can eliminate the noisy electrons coming from a wide area) and signal acquisition system that realizes a high signal to noise ratio to obtain a small modification of the tunneling current originating from the core excitation.

After first results on a semiconductor hetero-interface (Si(111)7x7-Ge) [1], second results on the nanoscale elemental analysis were acquired for metal-semiconductor interface (Ge(111)-Cu nanodomains) [2]. For both cases, the spatial resolution of the analysis was estimated to be 1~4 nm, and it is worth noting that the measured domains had a thickness of less than one atomic layer (Fig.2).

After progresses of the measurement system and techniques, we succeeded in obtaining a series of successive STM images at an atomically same area with serious drift or sample damages. Accordingly, we could acquire a linear dependence of the element contrast on the incident photon density. The photon density dependence of the elemental contrast will give an important clue to know the origin of the element contrast. Actually, our result on the linear dependence of the element contrast on the photon density suggests that we can deny a possibility of the local potential change derived from the core excitation, because the potential should give an exponential dependence of the contrast on the incident photon density.

Also we could recently measured scanning tunneling spectroscopy (STS), which have long been impossible because of instability due to brilliant X-ray irradiation. STS information gives us more direct hint to approach the mechanism of contrast to obtain a higher resolution. It is notable that the image in Fig. 2(c) shows the contrast originating from the chemical difference (that is not based on the surface step height), presenting the structures different from the conventional topographic (Fig. 2(b)) image.

Next, we have recently achieved a direct observation of the “X-ray induced atomic motion” with the track of the atomic motion at an atomic scale using the SR-STM system under the incident photon density of ~2x10$^{15}$ photon/sec/mm$^2$ [4]. This observation was enabled only by use of the in situ SR-STM system, because the STM images in the atomically same area should be compared before and after X-ray irradiation. In our STM images, the low-magnification images showed that the X-ray induced atomic motion rate is so low that structural changes are hardly detectable by other surface analysis techniques such as diffraction analysis. However, the magnified STM images revealed a clear change in the atomic structures after X-ray irradiation. Then, we developed a technique to recognize atomic motions directly to comprehend their behavior. By merging the STM images obtained before and after X-ray irradiation, the atomic motion track could be newly presented as several continuous lines (Fig. 3), whereas other stable atoms are shown as spheres. The appeared atomic track is the direct evidence and visualized information of the atomic diffusion at an atomic scale. It is worth comparing our results with past conventional thermal STM observations on the same surface [5], where the atomic motion was found to occur in the form of 2-dimensional domain and begin at ~220°C. However, our results show the atomic track having a local chain distribution. This locality in diffusion can be attributed to the anisotropy of the
surface structure, and probably the origin of atomic motion, to core excitation. In fact, considering the temperature increase of 92 K from the room temperature estimated from the X-rays irradiation, our atomic motion occurs at very low temperature in comparison with the past report.

Apart from the result on the elemental analysis, this finding on the atomic motion will serve to study the initial radiation effect on the optical devices such as mirror or grating at the X-ray sources of new generation such as X-ray free electron laser (XFEL). Also our observation of the damage barrier has potential importance as an indicator for a damage threshold in the near future for analyzing tiny materials using strong X-rays.

On the other hand, the above mentioned results will allow us to study the element-specific atomic control of local reaction with the spatial resolution of STM, giving hope of wide application. For example, the dense X-rays are suggested to have new applications, such as direct X-ray lithography. In other viewpoint, our results show a new application of the in situ SR-STM system. Our method for observing the atomic track will serve to provide new information not only for the radiation effects on various optical devices in new X-ray generators, but also for basic science by observing photon-matter interactions.

References

Figures

Fig. 1 Schematic view of experimental setup.

Fig. 2 (a) Line profile of beam-induced tip current image along the line shown in the bottom image. (b) Topographic image and (c) beam-induced tip current image of Ge (111)-Cu (-2V, 0.2 nA).

Fig. 3 Atomic motion tracks newly presented by merging the STM images before and after X-ray irradiation.