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NIM B
Beam Interactions with Materials & Atoms

Nuclear Instruments and Methods in Physics Research B 235 (2005) 452–455

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Observation of an HCI-induced nano-dot on an HOPG surface with STM and AFM

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Available online 23 May 2005

Abstract

We have observed highly oriented pyrolytic graphite (HOPG) irradiated with highly charged ions (HCIs) with a scanning probe microscope. The same impact site was observed with both the scanning tunnelling microscope (STM) and atomic force microscope (AFM) modes. In the non-contact mode AFM image, protrusion-like dots have been observed as well as in the STM observations. The dot size (diameter) and height observed with the AFM mode were nearly equal to that with the STM mode. The present observation clearly indicates that an HCI impact induces topographic modification on an HOPG surface.
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PACS: 61.82.Bg; 68.37.Ef

Keywords: Highly charged ions; Scanning tunnelling microscopy; Ion-surface scattering

1. Introduction

Collisions of slow highly charged ions (HCIs) with surfaces have received great attention in recent years and have been extensively studied with various methods, such as scanning probe microscopy, secondary particle observation, photon spectroscopy and so on (for these studies, for example, see [1] and references therein). Through previous observations with a scanning probe microscope (SPM), it has been shown that single HCI impact can induce surface modification. However, the mechanism for inducing such modification is not known, and further systematic investigations are necessary.

In the present study, we have observed highly oriented pyrolytic graphite (HOPG) irradiated

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doi:10.1016/j.nimb.2005.03.223

with slow HCIs using both the scanning tunnelling microscope (STM) and atomic force microscope (AFM) modes of an SPM. Up to now, several groups [2,3] have performed similar observations. In those studies, each impact site was observed as a protrusion-like dot with a height of ~1 nm and a diameter of several nm in the STM observation. However, no surface deformation was observed with an AFM. Consequently, it was concluded that the protrusion observed with a STM did not correspond to topographic surface modification but to the change in the electronic density of states.

In order to clarify these interesting but puzzling observations, the same nano-dot induced on an HOPG surface was observed with both the STM and AFM modes. The present study is different from the previous ones in the following points: (1) higher charge states such as Xe^{46+} were used to induce larger modification; (2) the STM and AFM images of the same impact site were observed with the same conductive cantilever; (3) AFM observations were performed with the non-contact (NC-AFM) mode while the previous observations were performed with the contact mode in which strong repulsive force was applied on the sample surface. The present results are discussed in comparison with previous measurements.

2. Experiments

We have been using two ion sources, a high-T_C superconductor EBIS [4] and the Tokyo-EBIT [5] depending on the charge state used. The former can produce relatively intense but relatively low charged ions and was used for charge states of up to 40+. On the other hand, the latter can produce highly charged but relatively low intensity ion beam and was used for charge states higher than 40+. HOPG cleaved in the air with adhesive tape was irradiated with HCIs from one of those ion sources. After irradiation, the HOPG sample was exposed to air, transported and set on a sample holder of the ultra high vacuum SPM.

The SPM used is JEOL JSPM-4500A, which can be operated with both the STM and AFM modes. When a conductive cantilever is used, one can observe the same position of a sample both with the STM and AFM modes at atomic resolution without changing the tip. This is quite important for the present study since we are interested in the difference between the STM and AFM images of the same dot. In the present experiment, a Si cantilever coated with Pt was used. Since the tip curvature radius of the coated cantilever is rather large (~40 nm) as compared with a tungsten tip (10–20 nm), it may be difficult to observe and discuss detailed structure of the dot. However, the present observations are considered to give important and useful information since the same dot was observed with the same tip for both the STM and AFM modes.

It is noted that the STM and AFM images were not observed simultaneously; the STM observation was done in the constant current mode while the NC-AFM observation was done in the frequency modulated (FM) mode.

3. Results and discussion

Fig. 1(a) and (b) show the STM and AFM images of the HOPG target irradiated with 138 keV Xe^{46+} ions, respectively. Each dot in the image corresponds to the nano-structure induced by single HCI impact. The size of the dot in the STM image was nearly equal to those obtained by several other groups [2,3,6,7]. From the distribution of the step and the dots, it was more or less certain that the NC-AFM and STM images were for the same area of the sample. As seen in Fig. 1(b), the nano-dots produced by HCI impact were

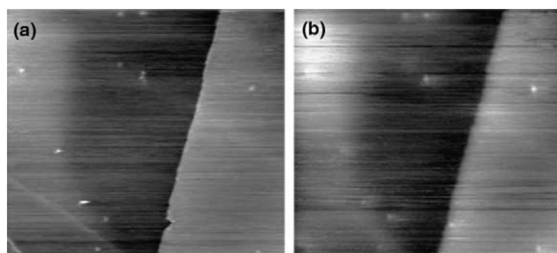

Fig. 1. STM image (a) and NC-AFM image (b) of HOPG irradiated with Xe^{46+}.

observed also with the AFM mode. The present observation has clearly shown that the nano-dot corresponds to topographical change on the surface.

Fig. 2(a) and (b) show the results of close-up observations of the same nano-dot with the STM and AFM modes, respectively. It is clear that the structure is not a crater but a protrusion also in the NC-AFM image as well as the STM image. In contact-AFM observation, it is known that the same disordered area can be observed both as a crater and as a protrusion depending on the scan direction by the influence of lateral forces [8], so that not only quantitative but also qualitative interpretation of the image is quite difficult. Since the lateral force effect is negligible in the NC-AFM mode, the present result clearly shows that the HCI-induced topographic features are protrusions.

The present results are listed in Table 1 together with several previous observations for the collisions of HCI with HOPG. In the previous studies, topographical change was not observed with an AFM even though protrusions were observed with an STM. The difference between the present and the previous results may be due to the following reasons.

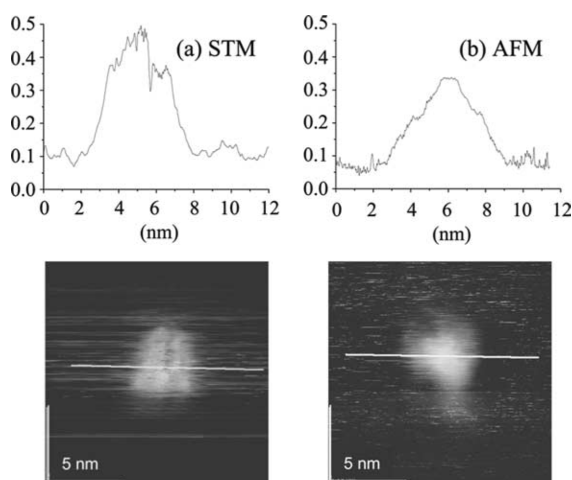

Fig. 2. STM image (a) and NC-AFM image (b) of the same dot observed with the same tip. For each figure, the lower panel represents two dimensional image and the upper panel the cross section at the line in the lower panel.

(1) The potential energy of the incident ion is quite different. In the present study, Xe^{46+} with a potential energy of 66 keV was used while for example Ar^{9+} with a potential energy of 1 keV was used by Gebeshuber et al. [3]. (2) The kinetic energy of the incident ion is also different; 138 keV in the present study while 0.15 keV for [3] and 4 keV for [6]. It is conceivable that the larger potential and kinetic energies in the present study induced larger topographical change which can be easily observed with an AFM. (3) In the present study AFM observation was done with the NC-AFM mode while contact-AFM was used in the previous studies. In the contact AFM mode, strong repulsive force is applied on the sample surface, which may damage the nano-dot produced by HCI impact. In addition, it was reported by several groups that the contact AFM mode did not have true atomic resolution and thus it could not observe atomic scale defects although it could observe periodic structure of a surface [9]. Although we tried the observation with the contact AFM mode, no topographic feature was recognized. Judging from these results, the contact AFM mode may be unsuitable for observations of nano-dots on HOPG while it was successfully used for observations of HCI-induced nano-structure on mica [8]. It is noted that tapping mode AFM has been used by An et al. [10] to observe HOPG irradiated with 1 keV Ar^{+}, and that they have observed protrusions with a height of 0.08–0.15 nm. The height of the dot in the present NC-AFM observation has been about 0.5 nm, which is much larger than that observed by An et al. It is considered that larger potential energy or larger kinetic energy or both in the present experiments induced larger topographic deformation. According to the observations by Minniti et al. with an STM [7], the dot diameter and height increase with increasing charge state even when the kinetic energy is as high as 276 keV (see Table 1). It may be concluded from the results discussed above that the potential energy or the charge state has more direct and stronger influence on the nano-dot size rather than the kinetic energy.

Table 1
Size of the nano-dot induced by HCI impact on HOPG

Incident ion	Potential energy (keV)	Impact energy (keV)	Dot size (nm)		Dot height (nm)		AFM mode	Ref.
			STM	AFM	STM	AFM		
Xe^{46+}	66	138	∼6	∼6	∼0.5	∼0.5	FM	Present
Xe^{44+}	51	276	∼7[a]	–	∼0.8[a]	–	–	[7]
Xe^{23+}	6.8	276	∼3[a]	–	∼0.5[a]	–	–	[7]
Ar^{8+}	0.6	4	∼1.2[b]	None	∼0.5[b]	None	Contact	[2]
Ar^{8+}	0.6	0.4	∼1[c]	–	Not specified	–	–	[6]
Ar^{9+}	1	0.15	∼1.2[d]	None	∼0.3[d]	None	Contact	[3]

[a] Estimated from Figs. 2 and 3 of [7].
[b] Estimated from Figs. 3 and 4 of [2].
[c] Estimated from Fig. 1 of [6].
[d] Estimated from Fig. 9 of [3].

Acknowledgements

This work was supported by Special Research Projects for Basic Science of RIKEN, and partially supported by the CREST program, "Creation of Ultrafast, Ultralow Power, Super-performance Nanodevices and Systems" in the Japan Science and Technology Agency.

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