SPM observation of slow highly charged ion induced nanodots on highly orientated pyrolytic graphite

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Abstract. We have observed nanodots on a highly orientated pyrolytic graphite (HOPG) surface produced by highly charged ion impacts using a scanning tunneling microscope. Previous measurements have confirmed the dominant role of the potential energy or the incident ion charge state on the size and height of the observed nanodots. The present results extend these previous measurements to much lower kinetic energy. It appears that there is no observable influence on the lateral size of the nanodots due to the incident ion kinetic energy down to approximately 200 eV. In contrast some slight reduction in the nanodot height was observed as the kinetic energy was reduced.

1. Introduction

The interaction of slow highly charged ions with solid surfaces has received increasing attention in recent years [1]. Single ion implantation using doubly charged P ions has been used to enhance the properties of silicon semiconductor devices [2]. This technique has been proposed as a possible way to develop a quantum computer [3] and experiments taking advantage of the enhanced secondary electron signal from HCI surface interaction are currently in progress [4]. The implantation process causes damage to the solid lattice through a combination of elastic and electronic collisions processes. A HCI has a large potential energy which is deposited mainly in the surface region resulting in a complex interaction.

The current experiment is a scanning tunneling microscopy (STM) investigation of damage sites on HOPG after bombardment with slow Ar\textsuperscript{q+} ions (q = 1, 8, 9, 11) at various kinetic energies (approximately 200 eV to 20 keV). HOPG has been extensively studied due to its ease of preparation and stable flat surface. Experiments with both swift heavy ions and slow singly and multiply charged ions (see for example the review by Neumann [5] and references therein) have been reported. In all cases blister type defects, nanodots, have been found on the surface.

Several experiments have also been performed with HCI [6–12] with the resulting nanodots studied by STM and atomic force microscopy (AFM). It was found that the potential energy of the incident ion has a strong influence on the surface modification, i.e. the size of observed nanodot features increases with increasing charge state of the ion. In a series of experiments with Xe\textsuperscript{23+} over a kinetic energy range from 1–100 keV, Nakamura \textit{et al} [11] found that the
kinetic energy of the incident ion had no influence on either the height or the lateral size of the observed nanodots. They observed a strong increase on the nanodot size with increasing ion charge state although the height of the nanodot remained constant. The aim of the current research is to extend these previous measurements to lower kinetic energy (< 1 keV) to clarify the role of potential and kinetic energy in the limit of slow HCI collisions on HOPG.

2. Experimental Method
In all experiments ions were created using the RIKEN high-Tc superconducting EBIS [13] operating in leaky mode. The EBIS was typically operated at an electron beam energy of 7 keV and an electron beam current of 10–15 mA. Argon gas was injected into the EBIS via a needle valve to a pressure of around $1.0 \times 10^{-8}$ Torr (background $1.5 \times 10^{-9}$ Torr). HCI's were extracted from the EBIS at an extraction voltage of 2 kV and energy selected by a 90° magnet. The ion beam line was connected directly to the specimen treatment chamber of the ultra high vacuum scanning probe microscope (JEOL JSPM-4500A).

A specially constructed target holder was attached to this chamber and lowered into the ion beam during the experiment. To adjust and check the ion beam a MCP detector was fixed above the target region. Both the target and the MCP were shielded by a grounded metal plate with two small beam apertures (diameter 2 mm) separated vertically by 40 mm. Before irradiation the beam quality was optimized while observing the signal on the MCP. When sufficient beam quality was achieved the target holder was translated upwards by 40 mm so that the target region was moved into the ion beam. For Ar$^{8+}$ typically around $2 \times 10^4$ counts per second were observed at the MCP. During longer runs the beam intensity was checked periodically at the MCP and adjusted accordingly.

After irradiation the HOPG samples were transferred from the SPM treatment chamber to the observation chamber. All observations were thus carried out in situ, under ultra-high vacuum. The vacuum in the target and observation chambers was typically in the range of $10^{-9}$ Torr. The irradiated samples were then imaged using both mechanically cut Pt/Ir and electrochemically etched W tips. Constant current STM imaging was performed, with the tunneling current held at 1 nA and a bias voltage of 0.5 V between the sample and tip.

3. Results and Discussion
A typical STM image of a single nanodot on the HOPG surface is shown in figure 1. Line scans through the center of the dot structure were used to extract the dot height and base width from the STM image data. For each charge state and kinetic energy approximately 30 dots were analyzed and the average height and width calculated. The dependence of the dot height and width on the charge state of the incident ion is plotted in figure 2. Although there is some small change in kinetic energy for each charge state the effect should be negligible. The error bars represent the standard deviation of the measurements.

It seems that for the low charge states studied, there is no observable dot size dependence on the incident ion charge state within the experimental distribution. For the dot height the present results show a very slight reduction in the average values with lower charge states but the large spread in the data makes it unclear if there is any charge dependence. It seems that the kinetic energy of the incident ion plays a more important role in this regime. To clarify this effect figure 3 shows the same results plotted as a function of kinetic energy.

Although the distribution of the data is quite large, the general trend suggests that the dots get slightly taller with increasing kinetic energy. On the other hand, the results for the ion size are more ambiguous. No significant change is seen over the range of kinetic energies studied.

It seems that at the low kinetic and potential energies studied there is no significant dependence of the dot width on the kinetic or potential energy. Some slight increase in the dot height was observed both for higher charge states (larger potential energy) and for higher
Figure 1. A typical STM image of the ion impact site on HOPG, in this case for 180 eV Ar\textsuperscript{9+} incident ion. Lateral dimensions are 10 × 10 nm, darker regions are lower and brighter regions higher in this grayscale representation.

Figure 2. The potential energy dependence of the dot size and height for incident Ar ions with different charge states (q = 1, 8, 9, 11). The error bars represent the standard deviations.

impact kinetic energy although the large spread in the data makes it difficult to draw any firm conclusions. These results are in contrast to the previous results of Nakamura et al [11] which showed no kinetic energy dependence and a strong charge state dependence on the nanodot size. However, these results were obtained at much higher charge states (up to Xe\textsuperscript{44+}) and at much higher kinetic energy (1 to ≈ 100 keV).

Previous AFM studies with singly charged ions at low kinetic energy showed that the nanodot was of a topographical nature [14]. There was some doubt after several experiments failed to find any dots with AFM [6, 10]. A combined STM/AFM measurement reported by Terada et al [12] proved conclusively the topographic nature of the dots for HCl (Xe\textsuperscript{46+}). The origin of this type of surface deformation is still unclear with several electronic process having been proposed [8]. However, the present results seem to suggest some kinetic energy dependence when the incident ion has a kinetic energy of less than several kV. In this regime collisional effects may play some
Figure 3. The kinetic energy dependence of the dot size and height for incident Ar ions with different charge states \((q = 1, 8, 9, 11)\). The error bars represent the standard deviations.

role although this needs more detailed further study before any meaningful comparison with theory can be performed.

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