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In Situ Direct Imaging of Scanning Tunneling Microscope Tip Apex

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A scanning tunneling microscope (STM) tip apex is directly imaged by the nano needle structure grown on the sample surface (Needle Formation and Tip Imaging : NFTI). The nano needle structure, 3 to 10nm in height and 2 to 3nm in diameter, is formed on the silicon(111)7$\times$7 surface by applying bias voltage of around -10V to the tip. On the contrary, when positive high voltage is applied to the tip, a nano needle structure is grown on the tip apex. The correspondence between the tip apex structure and the STM image is evaluated, and it is ascertained that an atomic scale minitip creates atomic resolution images. The growth process of nano needle on the tip is also observed by NFTI method.

1. INTRODUCTION

Scanning tunneling microscope (STM) has made remarkable progress in observing surfaces with atomic resolution. One of the most important factors for obtaining high resolution is the tip apex condition. Several tip observation methods have been reported using transmission electron microscope,[1)] field ion microscope[2)] and STM.[3)] In these techniques, however, tips have to be withdrawn from the sample surfaces during evaluation, which makes it impossible to observe the change of the tips and the sample surfaces continuously by those conventional technologies.

This paper describes the novel *in situ* STM tip observation technology by forming a sharp nano needle structure on the sample surface (Needle Formation and Tip Imaging : NFTI). Generally speaking, the STM image is obtained as a convolution of tip apex shape and the sample surface structure. Therefore, if the sample surface within the observation area includes sharper structures compared with the tip apex, the tip apex is inversely imaged by these structures, as schematically shown in Fig.1. The tip is scanned by the sharper structure and a tip image appears at the position where the structure exists as indicated by an image contour in Fig.1. Though a similar inverse imaging technique has been applied to atomic force microscope tips using columnar structures formed on the sample surface[4)], high enough resolution has not been achieved yet because of the large dimension of the columnar structures.

In this study, needle structures were formed on the silicon surface by applying high negative voltage to the tip, which were sharp enough to delineate tip apex shape. Using these needle structures, the tip apex features were evaluated with atomic resolution and it was shown that the tip apex shape corresponds to the STM image quality. Furthermore, when positive high voltage was applied to the tip, the needle structures were grown on the tip apex, and the growth process was observed *in situ* by NFTI method.

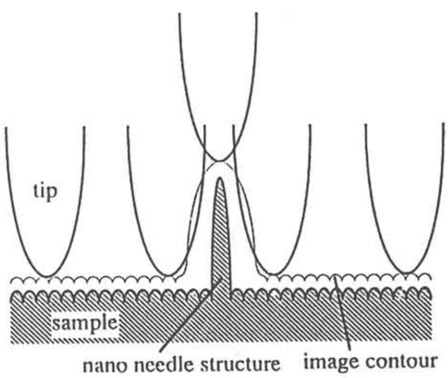

Fig. 1. Schematic drawing for explanation of tip imaging principle. The needle structure formed on the surface images the tip apex feature.

2. EXPERIMENTAL

The nano needle structures were formed by slowly increasing the tip bias voltage up to around -10V and kept for about 10 to 30s on a Si(111)7$\times$7 reconstructed surface, while maintaining the tunneling current at 0.2nA. The chamber pressure was below 7×10^{-11}Torr throughout the process. Electrochemically etched tungsten wires and mechanically sharpened platinum-iridium wires were used for tips. All the STM images were taken at the tip bias voltage of -2.0V and the tunneling current of 0.2nA.

3. RESULTS AND DISCUSSION

An STM image of the Si(111)7$\times$7 reconstructed surface is shown in Fig. 2, including three tip apex images taken by NFTI method. These needle structures were formed from the top right to the bottom left of the imaged area, the heights of which measured 3nm, 3nm and 1.5nm, respectively, by applying a tip voltage of -10V for 30s, 30s and 10s, respectively. Tip images taken by these needle structures are almost identical, which indicates that the shape of the needle structure is reproducible.

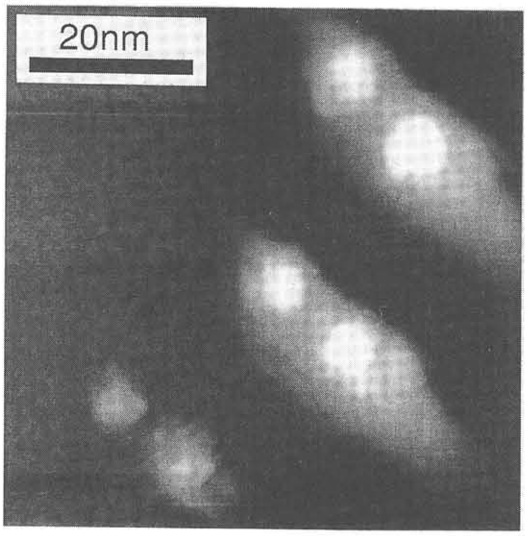

Fig. 2. Tip images observed by NFTI method. The needle structures were grown by applying tip voltage of -10V for 30s ,30s and 10s from the top right to the bottom left of the imaged area.

The relationship between the tip feature and STM image was investigated by NFTI method. The nano needle structrue was grown by applying a tip voltage of -10V for 10s and the tip image was observed as shown in Fig. 3(a). Figure 3(b) is an STM image of a Si(111)7×7 reconstructed surface using this tip. The resolution was poor and white dots were observed due to the instability of the tip apex. In order to change the tip apex condition, a -4V tip voltage was applied for 60s at the area 30nm apart from the needle structure, where the applied tip voltage had no effect on the needle structure. Figure 3(c) shows the tip image using the same needle structure. The most remarkable change of the tip apex is the growth of a very sharp minitip on the apex as indicated by an arrow, which might consist of single atom. Figure 3(d) indicates the STM image of the same area as Fig. 3(b) using the modified tip. The quality of the image was obviously improved. Steps and Si atoms were clearly resolved and white dots disappeared, and the improvement of the STM image is attributed to the minitip. These results clearly demonstrate a good correspondence between the tip feature and the STM images.

The nano needle structrue for NFTI observation, 9nm in height, was formed on Si(111) surface by applying a tip voltage of -7V for 10s to evaluate the nano needle growth on an STM tip. Figure 4(a) shows a side view of the tip apex. The figure shows that this tip had a very flat plane at the top, the diameter of which was around 50nm. After applying +10V for 30s to the tip at the point 50nm apart from the needle structure, the tip image was observed as shown in Fig. 4(b) using the same needle structure. The most remarkable change is the appearance of a nano needle structure grown on the tip apex, which extended about 5nm measured from the original top plane of the tip. By further applying a tip voltage of +10V for 30s, the nano needle structure on the tip apex was grown to around 9nm, which was almost equal to the height of the sample needle structure. As a result, the sample needle structure could not reach the original tip apex plane and only the tip needle structure was imaged. In Fig.4(c) the sample surface around the needle structure was observed, which did not appear in Fig.4(a) and (b) because this area was overlaid with the tip image. The diameter of the needle structure can be evaluated from Fig.4(c), where the observed feature, measuring 5nm in diameter, is a convolution of the sample needle structure and the tip needle structure. Assuming that the both needle structures are identical, the diameter of the needle is half of the observed feature. Therefore, the needle structure is 9nm in height and 2 to 3nm in diameter, which is much sharper than the normal STM tip. Thus, the sharpness makes it possible to delineate the tip apex structure.

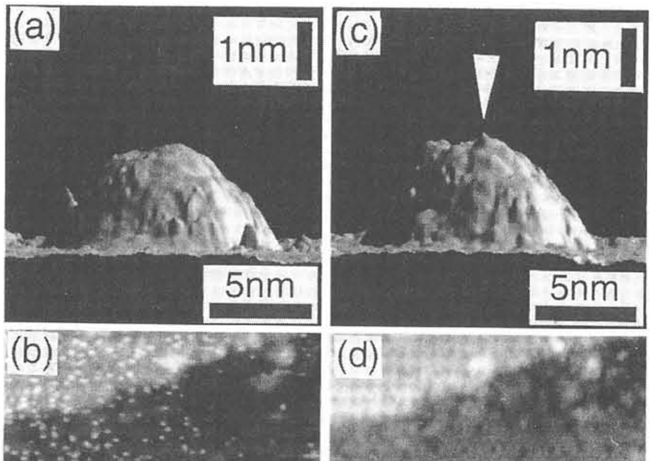

Fig. 3. The relationship between the tip structure and the STM images. (a) a side view of tip apex. (b) An STM image of Si(111)7×7 surface observed using the tip shown in (a). (c) A side view of the tip after modification by applying a tip voltage of -4V for 60s. Two minitips were grown at the top of the tip. (d) An STM image of the same area as (b) using the tip shown in (c). The resolution was improved.

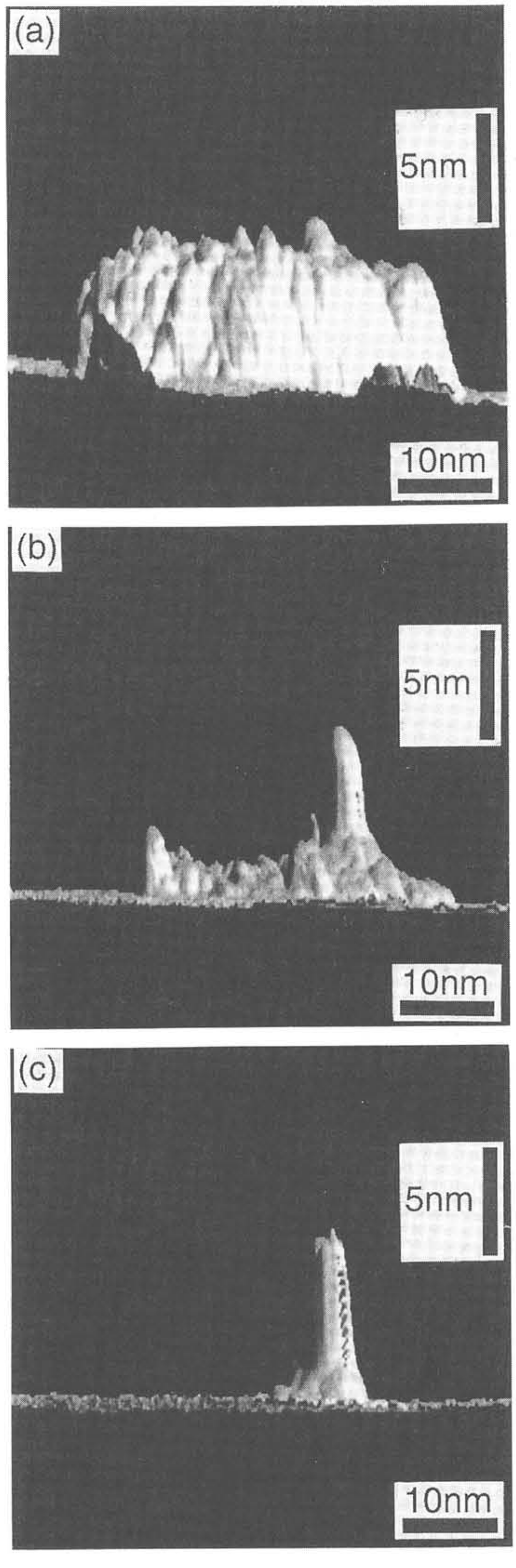

Fig. 4. Growth process of a nano needle structure on the tip apex. (a) A tip image before growth. (b) After applying +10V for 30s to the tip. The nano needle is grown by 5nm. (c) After applying +10V for 30s to the tip again. The nano needle extends 9nm. The original tip apex disappeared.

It is not clear yet whether the needle structure consists of the tip material or silicon atoms from the Si(111) sample surface. However, the needle formation phenomenon was observed regardless of the tip material, either tungsten or platinum. Furthermore, the sample surface became quite rough around the needle structure and many silicon addatoms are removed from their reconstructed position. Considering all these facts, the material of the needle structure would be originated from the sample silicon atoms. Aono et al. reported that silicon atoms right under the tip can be easily removed by applying a high bias voltage between the tip and the sample.[6] The applied voltage used in this work is high enough compared with the threshold voltage they reported. In addition, a field gradient is generated by the tip voltage on the sample surface near the tip. Therefore, if the dipole moment is induced in the silicon atoms due to the field, the atoms are attracted towards the tip. Such a surface diffusion was also reported in the case of cesium atoms adsorbed on the gallium arsenide(110) surface.[7] In this work the surface silicon addatoms may be removed from the surface due to the high field, and then migrate towards the tip, resulting in formation of a nano needle structure. The detailed mechanism is under investigation.

4. CONCLUSION

An STM tip apex was successfully imaged *in situ* by the nano needle structure on Si(111)7×7 surface, which was grown by applying a tip voltage of -10V for several seconds (Needle Formation and Tip Imaging : NFTI). The relationship between the tip apex structure and the STM image was evaluated, and it was experimentally ascertained that an atomic scale minitip creates atomic resolution images. The nano needle structure was also grown on the tip apex by applying a high positive voltage to the tip, and the growth process was observed by NFTI method *in situ*.

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