Functional Probes for Scanning Probe Microscopy

K Akiyama¹, T Eguchi², T An³, Y Fujikawa¹, T Sakurai¹ and Y Hasegawa²*
¹Institute for Materials Research, Tohoku University, Sendai, 980-8577 Japan
²Institute for Solid State Physics, The University of Tokyo, Kashiwa, 277-8581 Japan
*corresponding e-mail: hasegawa@issp.u-tokyo.ac.jp

Abstract. Functional probes such as a metal-tip cantilever and a glass-coated tungsten tip for scanning probe microscopy (SPM) were fabricated utilizing focused ion beam method. Using the functional probes, we obtained results which were hard to reach by usual SPM probes.

1. Introduction

With scanning probe microscopy (SPM), it is possible to obtain valuable properties of materials with nano-scale spatial resolutions. In spite of recent developments of SPM, the scanning probe, one of the key elements in SPM, has not been improved so much. Etched tungsten or platinum-iridium tips are commonly used in scanning tunneling microscopy (STM), and in atomic force microscopy (AFM) commercial Si cantilevers are often used. Both probes are sufficiently qualified for conventional usage of SPM. However, for ultimate and specialized performances of SPM, developing new and appropriate probes is required [1, 2]. We believe that fabrication of unique functional probes opens up new possibilities of SPM.

Here we report new SPM probes specially developed for Kelvin probe force microscopy (KFM), AFM lithography and synchrotron-radiation-light-irradiated scanning tunneling microscopy (SR-STM). A cantilever with a sharp metal tip is effective in precise measurements / control for KFM and AFM lithography [3]. A glass-coated tungsten tip is suitable for reduction of background noises in SR-STM [4]. The preparation method and performances of the functional probes are described in this paper.

2. Fabrication of functional probes

To fabricate a sharp tip apex, which is crucial for high spatial resolution of SPM, we used focused ion beam (FIB). For instance, to make the metal-tip cantilever, first a thin metal wire (5 μm or 10 μm in diameter) was attached to a regular Si cantilever using a micromanipulator, and then the wire was sharpened by FIB. The glass-coated tungsten tip was also sharpened by FIB after a chemically etched tungsten tip was covered with glass layer. The glass-covered tip was formed by inserting the etched tip into a glass tube and pulling apart the tube under heating with a gas burner. After thinned by repetitive heating, the glass layer was removed from the tip apex (~5 μm from the apex) with FIB, and simultaneously the tip was sharpened.

Chemical etching is a simple technique to sharpen SPM probes and thus has been used as a standard method. Various kinds of metal wire can be etched into a tip shape electrochemically with appropriate electrolyte solutions and etching voltages, and in the case of the glass-coated tip, coated glass can be removed by chemical etching using a HF solution. It is, however, difficult to control the
etching with micron-size precision unless special masking technique is used. Here, instead, we used FIB, which is often used to make samples for transmission electron microscopy (TEM). In FIB, the beam size of Ga ions is focused into a size of ~0.1μm, and the beam position can be controlled with a sub-micron precision. The shape of samples, which can be milled by the beam in the submicron precision, is monitored with a secondary electron image during the milling process. The spatial resolution of the image is limited by the beam size, and thus almost same as that of scanning electron microscope (SEM). In the fabrication process, we examine the sharpness of the tip from a top-view image of secondary electrons.

In the FIB setup used in this study (Seiko Instruments, JFIB- 2300) the area in which the beam scans for the milling can be adjusted in any desired shapes. To make a sharp tip, we milled the tip from its longitudinal direction with the milling area set in a doughnut-shape. By milling the circular edge of the tip with the doughnut-shape milling area and gradually shrinking its size, the tip can be sharpened. The final size of the doughnut shape to make nanosize tip apex is 0.4μm in the inner diameter and 0.9 μm in the outer diameter. At the final stage of the FIB milling, we do not observe the tip shape with the secondary electron image since the thin apex would be easily milled and become blunt by the observing.

3. Results and discussion

3.1. KFM observation with a tungsten-tip cantilever
For the KFM measurement, a sharp tip is needed for high spatial resolution and a metal tip is required for quantitative analysis. In the past, KFM measurements were carried out using a metal-coated tip or highly-doped Si tip. The coating makes the tip blunt; a drawback for achieving high spatial resolution. Furthermore, the metal coating might be easily removed during scanning when it is rather thin. In the case of the semiconductor tip, field penetration may hamper quantitative analysis of the potential.

To these problems the metal-tip cantilever fabricated by the FIB technique gives a perfect solution. In Fig. 1, a secondary electron image of a tungsten-tip cantilever fabricated in this study is presented. A sharp tungsten tip is found next to the original Si tip. The curvature radius of the tip is very sharp and found as sharp as 3.5 nm by TEM observation. Using this cantilever, we observed an electrostatic potential profile on the Ge/Si(105) surface in high spatial and energy resolutions. We successfully detected a small potential variation on the surface due to charge transfer among the dangling bond states of the surface [5].

3.2. AFM lithography with Au-tip cantilever
Using the same method, we can fabricate a cantilever with a tip of any material as far as its thin wire is available. We have fabricated a Au-tip cantilever, as shown in Fig. 2, for AFM lithography. AFM lithography is a method to deposit a submicron-size chunk of material from the tip by applying a positive voltage pulse on the tip and to draw a designated pattern with the dot. The material transfer from the tip to the substrate should be due to field evaporation, but direct contact (tapping) may also contribute to the phenomenon. So far the method has been performed by several groups [6, 7] using a Au-
coated tip in the amplitude-modulation mode (AM) of AFM. The radius of the Au coated tip was at least several tens of nm, and thus the minimum width of the drawn Au lines on a substrate is limited by the tip size.

In the same milling process of the tungsten-tip cantilever it is possible to make a Au-tip cantilever with a sharper apex than the coated one since the process basically doesn't depend on materials. Using the Au-tip cantilever in non-contact (nc) or frequency-modulation AFM mode, which has better force and distance regulations than the AM mode, we think that Au lines with the width of less than 10nm can be drawn on a substrate. We are now performing the lithography experiment with the Au-tip cantilever in nc-AFM mode.

If the nanometer-scale lithography is realized, we plan to use the Au lines as electrodes connecting to single molecules or nanosize materials for characterization of their electrical properties. As for the conductance measurement of single molecules, one of the big problems is the unknown number of the molecules sandwiched between electrodes [8]. A theoretical work predicts similar conductance curve even for the situation in which the molecule is absent. Using our nc-AFM lithography with the Au-tip cantilever, we can hopefully draw electrodes on appropriate positions of the sample surface after observing the target single molecule with AFM images.

3.3. SR-STM observation with a glass-coated tungsten tip

By utilizing the same method of the probe fabrication, we have also developed a glass-coated tungsten tip (Fig. 3) for SR-STM [4].

SR-STM is a STM combined with a synchrotron light source aiming at elemental analysis with the resolution of STM by detecting photo-induced electrons emitted from the surface with an STM tip as a detector. To achieve high spatial resolution the tip must be covered with an insulating layer to block undesired secondary electrons coming from a large subsurface area.

When we used polymer for covering a tungsten tip, the coated polymer contaminated the surface. Thus we devised a tungsten tip coated with glass except its apex.

With this new probe, we have obtained clean and atomically-resolved images of the Si(111)-(7x7) surface. Furthermore, the glass-coated tip is found effective in blocking the excess electrons, compared with an uncovered one, by a factor of ~40. Using this new tip, we successfully obtained images showing spatial distribution of specific element (Ni) with a spatial resolution less than 20 nm on a sample having a regular array of 1-micron-square Ni dots [9].

4. Conclusion

Functional SPM probes were fabricated by the FIB method and utilized in various purposes of SPM. The metal-tip cantilever works quite well as a KFM probe and a probe for AFM lithography. We found that the glass coated tungsten tip rejects excess secondary electrons effectively to give elemental resolution in nanoscale spatial resolution.

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