Applied Surface Science 70/71 (1993) 407–412
North-Holland

applied surface science

Diamond tips and nanometer-scale mechanical polishing

Zhipeng Chang, Zili Ma, Jian Shen, Xing Chu, Changxin Zhu, Jing Wang, Shijin Pang
Beijing Laboratory of Vacuum Physics, Chinese Academy of Sciences, P.O. Box 2724, Beijing 100080, China

and

Zengquan Xue
Department of Radio Electronics, Peking University, Beijing 100871, China

Received 1 September 1992; accepted for publication 18 November 1992

The major application of the scanning tunneling microscope (STM) is as a local surface topography and structure probe. The scanning tip usually is made of metal such as tungsten (W), platinum–iridium (Pt–Ir), gold (Au), etc., by electrochemical etching or mechanical cutting. STM has also recently demonstrated its potential application as a powerful tool for nanometer-scale surface modification. We made a kind of diamond STM tips by the MPCVD technique. The tips are quite stable, and hard enough to polish the metal film surface to produce relatively flat areas. Both SEM and STM were employed to check and analyze the tips and the results of polishing.

1. Introduction

The application of STM in the field of biological research has become very common in the last several years. Suitable sample substrates for STM experiments must fulfil three requirements at least: they should be electrically conductive, stable in air, and atomically flat over a large region. However, there are quite few suitable materials (that fulfil the above requirements) in air at present, such as crystal graphite. It is very difficult to find suitable and alternative natural substrates for STM working in air. Therefore, developing a special nanometer-scale surface polishing technique to make artificial STM substrates becomes much imperative. Fortunately, the scanning tunneling microscope (STM) has shown great promise for surface modification in the nanometer regime recently [1–8]. It can fabricate or produce some nanometer-scale holes or hillocks on various surfaces by direct mechanical contact, voltage pulse, or large tunneling current, etc. [1–6], and even can do atomic manipulation [7,8]. Hence it is reasonable to imagine that the STM itself can be used as a polishing tool to make artificial nanometer-scale flat STM substrates. Based on the above suggestions, a new concept can be brought forward, i.e. nanometer-scale mechanical polishing. In this new field of STM application, STM with a special hard tip which is different from normal STM tips is used as the main polishing tool. As normal STM tips become easily damaged by mechanical contact with the sample surfaces in most cases, fabricating and using a hardened tip are necessary for the polishing. So far we have successfully developed a method to make a kind of stable and hardened diamond STM tips, and also done some nanometer-scale mechanical polishing work on gold film on mica surfaces with these tips.

2. Experimental

In the experiments, the diamond tips' fabrication processes can be divided into two steps. Firstly, we made a very sharp tip (fig. 1) by traditional electrochemical etching, the radii of the tips' apexes are less than 100 nm. The tip material is tungsten (W) wire (0.2 mm), the etching solution is 2 mol/ℓ NaOH. After etching, the tips were transferred into an MPCVD (microwave plasma chemical vapor deposition) system chamber to grow the diamond grains on them. MPCVD is a very mature diamond film deposition technique. In our laboratory, continuous diamond films can be manufactured on Si surface routinely and repeatedly by this technique [9].

Fig. 2 is a typical SEM (scanning electron microscope) picture of the diamond films on crystal silicon substrate. The diamond grain sizes of the films ranged from 0.1 μm to several μm, depending mainly on the deposition conditions. The MPCVD diamond films were characterized by Raman spectroscopy. In the Raman spectrum, a sharp peak at 1332 cm^{-1} verifies the samples as diamond films of good quality. The other reason that made us choose MPCVD to fabricate the diamond tips, is the problem of conductivity. Although natural diamond is a good insulator, the conductivity of this kind of diamond films made by the MPCVD technique is high enough for the STM experiment [10]. The conductance mechanism is not very clear as yet, possible explanations might be impurities, surface structural and chemical modifications like reconstructions, defects, hydrogenation or amorphic structures, etc. [11–14].

Considering the basic requirements of the STM substrates, we selected gold film on mica surface as our polishing object, because gold film is a good electrical conductor and very stable in air. The procedures of gold deposition on mica are the following: (1) cut off several top layers of mica to produce a fresh and clean surface; (2) immediately put the fresh mica sample into a vacuum vapor deposition chamber (its vacuum pressure is 2×10^{-3} Pa) to deposit gold film on

Fig. 1. SEM picture of a tungsten tip manufactured by the electrochemical etching method.

mica simply by physical vapor deposition. The thickness of the film is from 0.1 to 1 μm. The surface of such films is quite rough, about 30 nm undulation can be observed on the surface with STM.

A domestic STM set-up (CSTM-9000, manufactured by the Institute of Chemistry, Chinese Academy of Sciences) was used in the experiment. For surface polishing, we interrupted the feed-back circuit of our STM electronic controller and lowered the tip down to the sample and made the distance of approach as close as we possibly could. Then the tips were made to reciprocate on the region of interest of the sample surfaces according to a certain frequency. SEM measurements showed that the diamond tips changed little after the polishing.

3. Result and discussion

It is not difficult for us to grow diamond grains on the side of tungsten wires by the MPCVD technique. Figs. 3 and 4 are two typical SEM pictures of tips with diamond grains. However, the SEM observation results demonstrated that most of the original tungsten tips were damaged during the diamond deposition procedure. The sharp apices of the W tips were cut off and the tips' top became a circular platform (about several micrometers in diameter). The reason might be the strong particle bombardment on the tips during the vapor deposition process. The original apices of the tungsten tips were cut off, but new apices sometimes were formed by the diamond grains growing on or near the top platform of the

Fig. 2. SEM image of a MPCVD diamond film, 21 μm × 15 μm area.

Fig. 3. SEM picture of a tungsten tip after growth of diamond grains; the insert is a magnified image of the top part (marked by a white rectangle).

Fig. 4. SEM picture of a tungsten tip after growth of diamond grains, the insert is a magnified image of the top part (marked by a white rectangle).

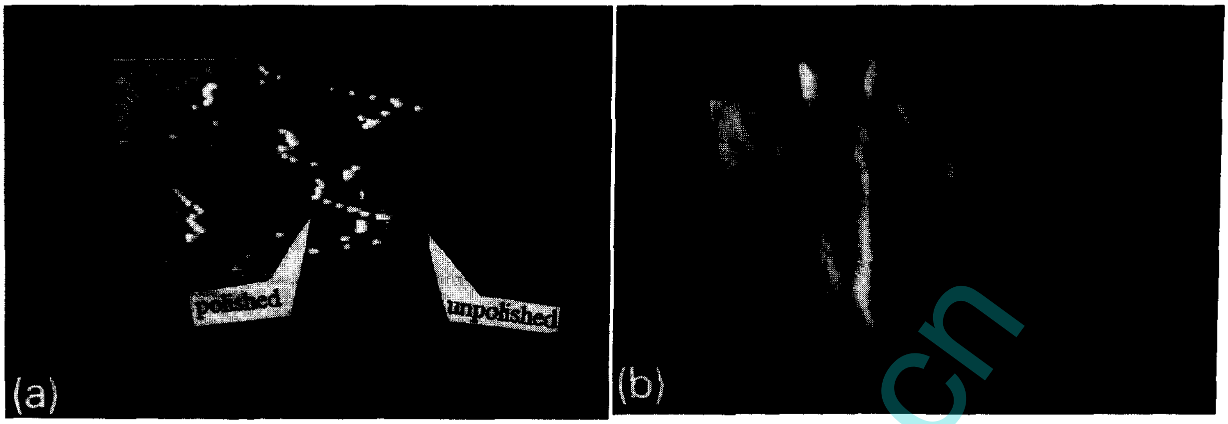

Fig. 5. STM image of gold film on mica after mechanical polishing by the diamond tip, the area is 240 nm × 124 nm: (a) grey-scale picture, (b) three-dimensional picture.

tungsten tips (see inserts of figs. 3 and 4). Actually, these new apices can replace the role of the original ones. Because most of the new apices cannot only produce a stable tunneling current under the normal STM work conditions (hundreds of millivolts to several volts bias voltage, 0.5 to 2 nA tunneling current), but also be sharp enough to obtain high-quality STM images, like fig. 4. The radius of the new diamond apex shown in fig. 4 is less than 100 nm; this apex actually is a corner point of a diamond grain. Obviously, this kind of new diamond tips are harder and more stable than the tungsten tips. Figs. 3 and 4 also show different densities and shapes of diamond grains along the side of tungsten tips; we attributed them to the different conditions and time of growth.

The gold grain sizes of the films on mica are about 30 to 50 nm, and the undulation of the surface is about 30 nm, according to our STM measurements. Fig. 5 is a STM image (displayed both as a grey-scale picture and as a three-dimensional one), which shows that some area of gold film on mica had been mechanically polished by the diamond tip on a nanometer-scale, the scanning region is 240 nm × 124 nm. Obviously, a polishing mark can be seen in this picture, on the left edge of the polishing area, the tip fabricated a straight-line shape protrusion along the edge. On the right of this line shape protrusion, a relatively flat area (although on the right edge the region is irregular) was produced, the undulation of this area being only about 3 to 5 nm. The irregularity of the right edge might be due to the special shape of the tip and the local topography of the gold film surface.

Various tip materials and polishing objects ought to be chosen in future research, in order to increase the rate of successful fabrication of diamond tips and obtain better polishing results.

4. Conclusion

We have successfully fabricated a kind of diamond tips by the MPCVD technique. The tips have sufficient conductivity and sharp apices. These tips are quite stiff and hard. Nanometer-scale mechanical polishing by these tips on gold film surfaces can produce relatively flat surfaces. This kind of artificial flat surfaces might be used as substrates of biological STM samples in the future.

Acknowledgements

We gratefully acknowledge Professor Chunli Bai (Institute of Chemistry, Chinese Academy of Sciences) and Dr. N. Liu for stimulating discussions and their kind help in the experiments and Ms. Xinli Jiang for SEM measurements.

References

[1] D.W. Abraham, H.J. Mamin, E. Ganz and J. Clarke, IBM J. Res. Develop. 30 (1986) 492.

[2] E.J. van Loenen, D. Dijkkamp, A.J. Hoeven, J.M. Lenssinck and J. Dieleman, Appl. Phys. Lett. 55 (1989) 1312.

[3] T.R. Albrecht, M.M. Dovek, M.D. Kirk, C.A. Lang and C.F. Quate, Appl. Phys. Lett. 55 (1989) 1727.

[4] Y.Z. Li, L. Vazquez, R. Piner, R.P. Andres and R. Reifenberger, Appl. Phys. Lett. 54 (1989) 1424.

[5] J.P. Rabe and S. Buchholz, Appl. Phys. Lett. 58 (1991) 702.

[6] H.J. Mamin, S. Chiang, H. Birk, P.H. Guethner and D. Rugar, J. Vac. Sci. Technol. B 9 (1991) 1398.

[7] D.M. Eigler and E.K. Schweizer, Nature 344 (1990) 524.

[8] J.A. Stroscio and E.M. Eigler, Science 254 (1991) 1319.

[9] Z. Chang, N. Liu, G. Chen and S. Pang, Vac. Sci. Technol. (China) 12 (1992) 275.

[10] W. Zimmermann-Edling, H.-G. Busmann, H. Strang and I.V. Hertel, in press.

[11] H.-G. Busmann, H. Sprang, I.V. Hertel, W. Zimmermann-Edling and H.-J. Güntherodt, Appl. Phys. Lett. 59 (1991) 295.

[12] T. Tsuno, T. Imai, Y. Nishibayashi, K. Hamada and N. Fujimori, Jpn. J. Appl. Phys. 30 (1991) 1063.

[13] K.F. Turner, B.R. Stoner, L. Bergman, J.T. Glass and R.J. Nemanich, J. Appl. Phys. 69 (1991) 6400.

[14] S.P. Mechandru and A.B. Anderson, Surf. Sci. 248 (1991) 369.