Fabrication of arrays of nanometer size test structures for scanning probe microscope tips characterization*

A. L. Bogdanov, D. Erts,[a] B. Nilsson, and H. Olin
Department of Physics, Chalmers University of Technology, S-412 96 Gothenburg, Sweden

(Received 1 June 1994; accepted 19 August 1994)

A problem in scanning probe microscopy (SPM) is the unknown shape of the probing tip. Generally, the image is a convolution between the shape of the tip and the surface. Information of the shape of the probe may be gained by imaging very sharp tips. Here we present a method for making two-dimensional arrays of very sharp tips. The tip arrays were made of silicon using electron beam lithography with subsequent ion-beam etching. To achieve the best possible resolution, ultrasonic excitation was used during development of the bilayered PMMA resist. Thus, openings in the resist with size nearly equal to the spot size of the writing e-beam have been obtained. A further decrease of the radius of the tips was obtained by the choice of appropriate thickness for the masking NiCr layer. The tips were conical with a height up to 100 nm with a radius of the tip down to 10 nm. The tips were suitable for study of the shape of AFM probe tips, under condition that the tip array samples were rinsed in water prior to the measurement. Without the rinsing procedure, strong sticking forces between the probe and the sample would have eroded both of them. The regularity of the array provided an easy way to calibrate the lateral motion of the scanner.

I. INTRODUCTION

Scanning probe microscopes (SPM) is a growing family of instruments that scan with various sharp probes over samples to obtain images of the surface. The earliest and the most developed is the scanning tunneling microscope (STM).[1] In the STM, the tip to sample distance is kept constant by a feedback system using piezoelectric actuators. By scanning the tip over the surface, a map of the surface is obtained from the feedback signal. Inspired by the success of the STM, a number of other scanning probe microscopes have been developed.[2] The atomic force microscope (AFM) invented in 1986 by Binnig *et al.*[3] is one of the most common of these microscopes. In the AFM, a sharp tip is dragged along the sample, similar to an ordinary profilometer, but with a much less force applied (about 10 nN). The AFM image is built up from contours of constant tip–sample force. (see Fig. 1).

A general problem in all these SPMs is the unknown shape of the tip, that is eventually convoluted with the shape of the sample.[4–13] To make an *in situ* measurement of the tip, one could image a surface with structures that are sharper than the probing tip, giving, in the ideal case with delta function peaks protruding from the surface, an image of the probing tip.

There are several techniques for making samples covered with sharp tips, for example, evaporation of a material through micrometer sized holes,[14] localized deposition via a chemical vapor deposition process,[15] and etching of silicon surfaces.[16,17] However, work on structures or tip arrays dedicated for analysis of SPM tips have only recently been made.[4,5,11]

Such a structure for tip imaging was recently reported by Montelius and Tegenfeldt.[11] They made samples consisting of cylindrical tips with a diameter of approximately 40–50 nm and a height of 120 nm. Their tips were made by plasma etching an InP sample partly covered with aerosol-produced silver particles which served as an etch mask. Another reported structure for tip imaging is an array of micrometer sized polysilicon pillars having undercut sidewalls.[4,5] Here a method for making a regular two-dimensional array of sharp silicon tips is presented together with illustrating AFM images.

II. EXPERIMENTAL

A. Tip array fabrication

Electron-beam exposures were performed in a JEOL JBX-5DII with LaB_6 cathode. The electron-optical system was adjusted to give a beam diameter of 10–12 nm FWHM. The acceleration voltage was 50 kV. The tip arrays were exposed as dots with displacement accuracy of 5 nm provided by the exposure tool. A lift-off process with double layer PMMA resist[18] was used to fabricate NiCr protection mask on silicon for subsequent chemically assisted ion-beam etching.

The bottom resist layer was spun from 2.9% solution of a 185 K PMMS in xylene to produce a film of approximately 50 nm thickness. After baking of the bottom layer for several minutes on a hot plate the top layer was applied. The top layer resist was 1.8% solution of a 350 K PMMS in xylene. Poor solubility of PMMA in xylene prevented intermixing of the top and the bottom layers. The double layered resist was finally baked on the hot plate for 10 min at 170 °C. The exposure dose per dot was $1-1.25\times10^{-16}$ C, which corresponds to surface dose of 200–250 $\mu C/cm^2$.

An ultrasonic excitation during development of the resist by 10% water solution in IPA was used to provide higher contrast and thus better aspect ratio in the narrow pitches. The ultrasonic helps to untie dissolved polymer molecules at the pitch bottom, where due to a big surface curvature the surface tension makes the molecule's release harder.[19]

*Published without author corrections.

[a]Also at Institute of Chemical Physics, University of Latvia, LV-1586 Riga, Latvia.

 0734-211X/94/12(6)/3681/4/$1.00

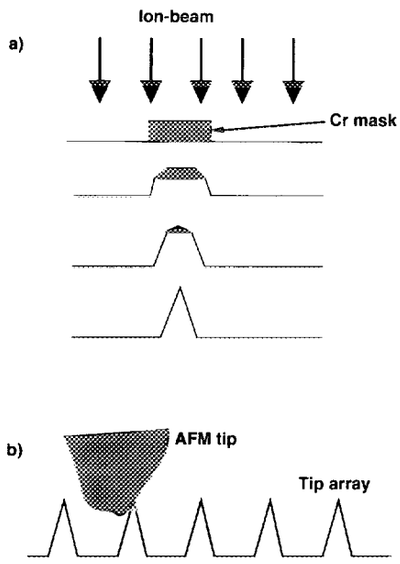

FIG. 1. (a) Principle of the fabrication of the tip array. (b) AFM probe imaging principle using a tip array.

A 15 nm layer of NiCr was thermally evaporated on the developed resist in vacuum with residual pressure of 10^{-5} mbar. The lift-off was made in 60 °C warm acetone, again with the assistance of the ultrasonic bath. The resulting NiCr pattern on the silicon was then used as a mask for Cl assisted Ar ion milling. Ar ions with an energy of 500 eV were used at normal incidence. The chlorine gas was fed through an O-shaped pipe with small holes evenly distributed over the circumference. The pipe was fitted near the substrate surface symmetrically around the ion beam axis. The ion current and the chlorine flow were chosen to provide the steepest slope of the etched profile.

B. Atomic force microscopy

Commercially available scanning probe microscopes (Nanonscope III by Digital Instruments, Santa Barbara, CA, and Universal System by Park Scientific Instruments, Sunnyvale, CA) were used in the experiments. The tips used were standard Si_3N_4 tips (Digital Instruments or Park Scientific Instruments) or sharper silicon tips (Ultratips, Park Scientific Instruments, and Nanoprobes, Digital Instrument). The Si_3N_4 tips had an aspect ratio of about 1:1 while the sharper silicon tips had a cone angle of 20° and a radius of 20 nm. The force applied in all images was between 5 and 20 nN and the scan frequency was between 0.2 and 1 Hz. Force calibration was performed before each run. The shapes of the AFM-tips were estimated by scanning electron microscopy (SEM).

III. RESULTS AND DISCUSSION

The array tips had a height of 100 nm and a radius of 10–25 nm as revealed from SEM images (Fig. 2). The base had a diameter of about 35 nm giving an aspect ratio between height and base up to 1:3. This could be compared to the Si_3N_4 tips with a radius of 50–100 nm and an aspect ratio

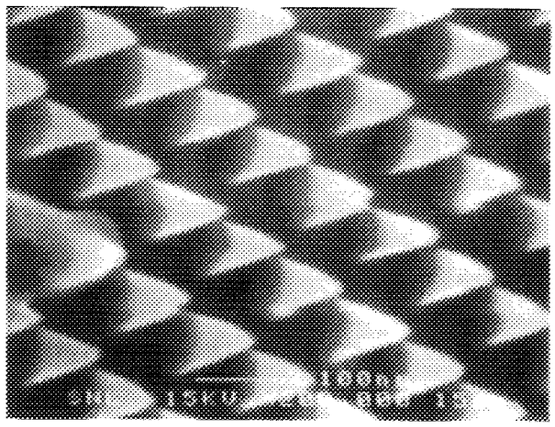

FIG. 2. SEM image of the Si tip array. Tilt angle is 60°.

close to 1:1. The sharper Si tips have a radius of about 20 nm and aspect ratios of about 1:3. The area of the sample covered with tips was only 10×10 μm^2, however, this area is easily found using an optical microscope.

Figure 3(a) shows an SEM image of a contaminated Ultratip before AFM scanning. The AFM image [Fig. 3(b)] shows clearly the corresponding probe images. Figure 3(c) is the SEM image of a Nanoprobe AFM tip and in Fig. 3(d) the corresponding AFM image is shown.

Figure 4(a) shows an AFM image of the tip array taken with a fresh Si_3N_4 tip. During scanning the array tips were

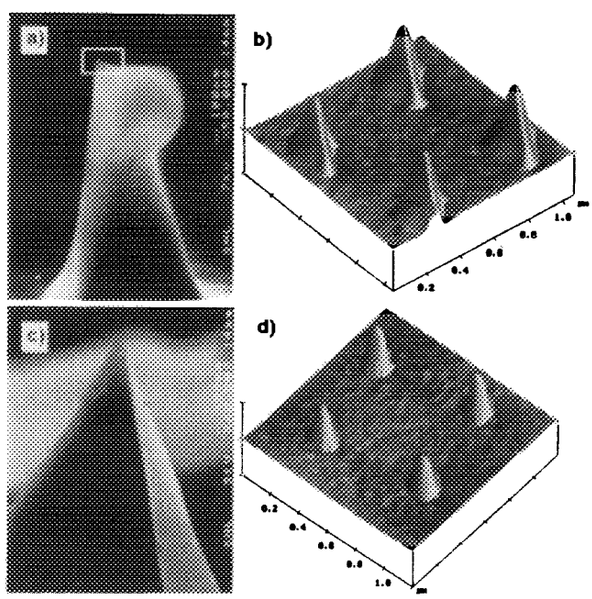

FIG. 3. (a) SEM image of a contaminated silicon Ultratip taken before AFM imaging. The small white square indicates the parts of the AFM tip that are measured by the tip array. (b) The AFM image of the tip array using this tip. The height scale is 110 nm/div. (c) SEM image of a silicon nanoprobe taken before AFM imaging. (d) The AFM image of the tip array using this tip. The height scale is 130 nm/div.

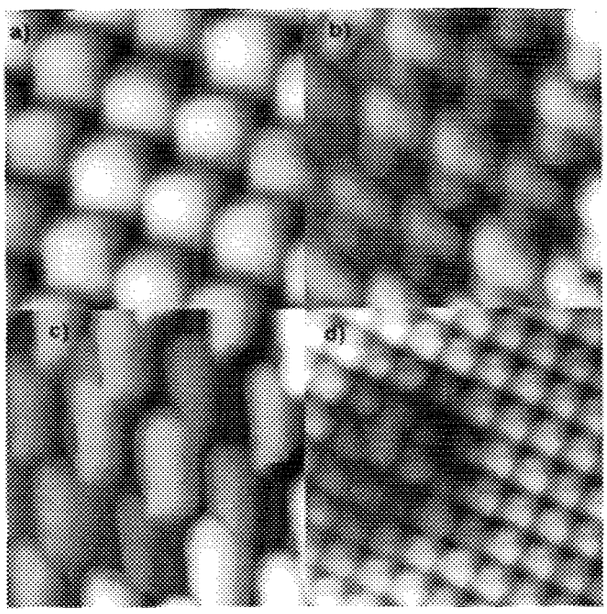

FIG. 4. AFM images showing the etching effect on the tip array using a Si_3N_4 tip. Distance between tips: 100 nm. (a) Image after one scan. (b) After 42 scans the image is strongly altered. (c) After 192 scans. (d) Image with a larger scan area which contains the smaller areas shown in (a)–(c). The small areas have been scanned 290 times. Note also that the tip has been changed by comparing (a) and the nonscanned area in (d). The height difference between the tips in scanned and nonscanned area is 40 nm. (The force constant of the cantilever was 0.032 N/m, the tip-sample force was 5 nN, and the scan frequency was 0.8 Hz.)

eroded, and after 48 images in the same area the image has changed dramatically [Fig. 4(b)]. Prolonged scanning resulted in a further degradation [Fig. 4(c)]. The etching effect is clearly seen in the larger scale image [Fig. 4(d)]. The scanning tip has also changed as seen from a comparison between Fig. 4(a) and the nonscanned region in Fig. 4(d).

This etching effect is a common problem in scanning probe microscopy and a great deal of work is going on to increase the understanding of these phenomena and even to explore the effect in order to create nanometer sized structures. In our case the sticking force between the Si surface and the Si or Si_3N_4 tip is too large, resulting in poor quality of the images. To image Si successfully we rinsed the sample in distilled water prior to imaging. Figure 5 shows an AFM image of the array after rinsing in distilled water. No erosion effect was observed during the 2 h that the imaging was performed.

Another way to circumvent the problem with the strong sticking force between tip and sample is to image using the so-called tapping mode.[20] In this mode the tip is not in contact with the surface during lateral translation; instead the tip is made to oscillate along the Z axis. Figure 6 shows an AFM image taken in the tapping mode. The characteristic triangular shape of the silicon tip used in the tapping mode is clearly visible. During the experimental run (about 1 h) any degradation of the tips was not observed.

Other materials can be used for making tip arrays. To

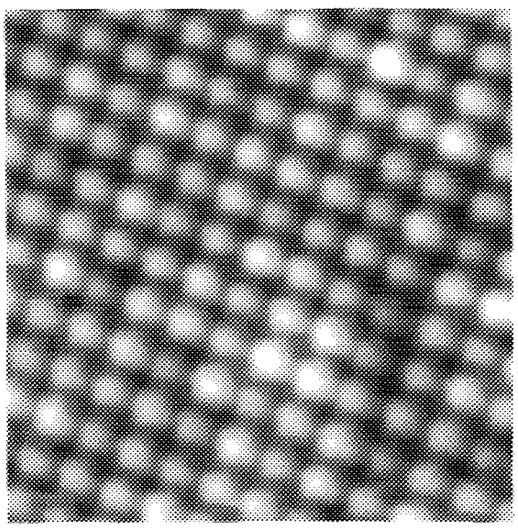

FIG. 5. AFM image of the tip array after rinsing the sample in water. Distance between tips: 100 nm. No degrading tip was observed during scanning. (The force constant of the cantilever ws 0.21 N/m, the tip-sample force was 5 nN, and the scan frequency wsa 0.5 Hz.)

avoid the above mentioned problems with sticking to the silicon surface, other materials like Al_2O_3, MgF_2, or glass may be used. For STM-tip characterization a nonoxidizing metal could be used.

The necessary recalibration of the piezo scanners is easily accomplished using these tip arrays. A lateral accuracy of 5 nm is provided by the lithography tool.

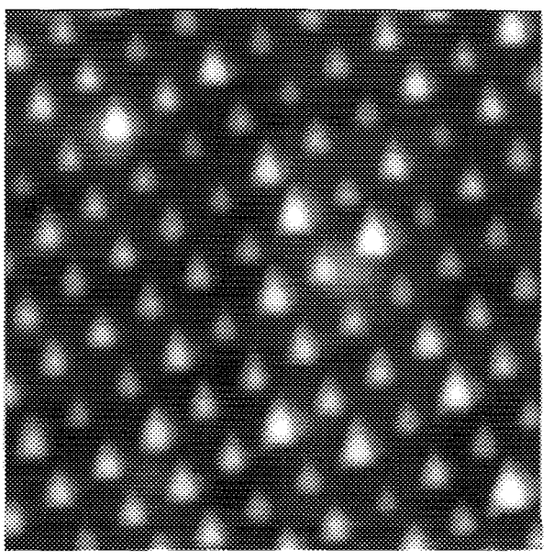

FIG. 6. Tapping mode AFM image of the tip array showing the characteristic triangular shape of the special tip used in this imaging mode. Distance between tips: 100 nm. No degrading was observed.

IV. SUMMARY AND CONCLUSION

A method to make two-dimensional arrays of sharp tips of silicon has been demonstrated. The tips have a radius of 10 nm and an aspect ratio of 1:3. The tip arrays are suitable for examining the shapes of AFM tips under condition that the tip array sample is rinsed in water prior to measurement. Without the rinsing procedure the strong forces between the probe and the sample will degrade both of them. The regularity of the array provided an easy way to calibrate the piezo for lateral motion.

ACKNOWLEDGMENTS

This work was supported by the Swedish Board for Industrial and Technical Development and by the Swedish Technical Science Research Council. The tip arrays were made in the Swedish Nanometer Laboratory.

[1]G. Binning, H. Rohrer, C. Gerber, and E. Weibel, Phys. Rev. Lett. **49**, 57 (1982).
[2]H. K. Wickramasinghe, J. Vac. Sci. Technol. A **8**, 363 (1990).
[3]G. Binning, C. Gerber, and C. F. Quate, Phys. Rev. Lett. **56**, 930 (1986).
[4]J. E. Griffith, D. A. Grigg, M. J. Vasile, P. E. Russell, and E. A. Fitzgerald, J. Vac. Sci. Technol. B **9**, 3586 (1991).
[5]D. A. Grigg, P. E. Russell, J. E. Griffith, M. J. Vasile, and E. A. Fitzgerald, Ultramicroscopy **42–44**, 1616 (1992).
[6]P. Niedermann, J. Microsc. **152**, 93 (1988).
[7]G. S. Pingali and R. Jain, Proc. SPIE **1923**, 151 (1992).
[8]G. Reiss, J. Vancea, H. Wittmann, J. Zweck, and H. Hoffmann, J. Appl. Phys. **67**, 1156 (1990).
[9]G. Reiss, F. Schneider, J. Vancea, and H. Hoffmann, Appl. Phys. Lett. **57**, 867 (1990).
[10]D. Keller, Surf. Sci. **253**, 353 (1991).
[11]L. Montelius and J. O. Tegenfeldt, Appl. Phys. Lett.**62**, 2628 (1993).
[12]S. M. Paik, S. Kim, and I. K. Schuller, Phys. Rev. B **44**, 3272 (1991).
[13]D. J. Keller and F. S. Franke, Surf. Sci. **294**, 409 (1993).
[14]C. A. Spindt, I. Brodie, L. Humphrey, and E. R. Westerberg, J. Appl. Phys. **47**, 5248 (1976).
[15]E. I. Givargizov, J. Vac. Sci. Technol. B **11**, 449 (1993).
[16]R. N. Thomas and H. C. Nathanson, Appl. Phys. Lett. **21**, 384 (1972).
[17]H. C. Gray, G. Campisi, and R. F. Greene, IEDM Tech. Dig. 776 (1986).
[18]S. Mackie and S. P. Beaumont, Solid State Technol. 117 (1985).
[19]W. Chen and H. Ahmed, Appl. Phys. Lett. **62**, 1499 (1993).
[20]Q. Zhong, D. Inniss, K. Kjoller, and V. B. Elings, Surf. Sci. **290**, L688 (1993).