Application of atomic force microscopy nanomachining to the fabrication of metallic nanodots, nanowires, and nanoelectrodes

*Ju-Hung Hsu (許如宏), Chun-Yu Lin (林俊羽), Yu-Ju Chen (陳右儒), and Heh-Nan Lin (林鶴南)

Department of Materials Science and Engineering, National Tsing Hua University (清華大學材料科學工程學系)

(NSC92-2120-E-007-005)

We report a simple method to fabricate metallic nanostructures by a combination of atomic force microscopy (AFM) nanomachining and subsequent lift-off process. By controlling the force of an AFM tip on a thin PMMA resist, nanoscale holes and grooves are first created. By coating a metal film and performing the lift-off process, metallic nanodots with a size of 70 nm and nanowires with a width of 75 nm are successfully fabricated. In addition, nanoelectrodes with a gap of around 50 nm are also produced by cutting a nanowire with the tip. The present method demonstrates the feasibility and effectiveness of using single-layer resist in AFM mechanical lithography instead of two-layer resist reported in the literature.

keywords : atomic force microscopy (AFM), nanomachining, nanodots, nanowires, nanoelectrodes

1. Introduction

Scanning probe lithography has been used extensively in recent years for the fabrication of nanostructures and nanodevices.[1] Among the various techniques, the atomic force microscopy (AFM) nanomachining has been adopted since early years due to its ease of operation and applicability on insulating substrates.[2,3] By controlling the contact force between the AFM tip and the sample, desired nanostructures can be created. In this simplest scheme, nevertheless, the tip is vulnerable and not suitable for long time use, especially on hard materials.

By incorporating the notion of optical or electron-beam lithography, modifications of AFM nanomachining have consequently appeared. With the use of a thin resist on the substrate, the so called one-layer approach, holes or grooves can be first created by indenting or plowing the tip into the resist.[4–6] Creation of nanostructures with the one-layer approach by a combination of AFM nanomachining, coating, and lift-off, however, has been absent in the literature to our knowledge.

The one-layer approach has a claimed disadvantage that the tip still experiences damage. A further modification is the use of a two-layer resist. In this methodology, the nanomachining is performed on the top layer and the tip damage is therefore minimal since the second layer is a soft material. With following chemical etching (into the second layer), coating, and lift-off, metallic nanostructures have been successfully generated.[7–9] As the two-layer approach is more complicated, it is desirable that the one-layer approach can be realized by standard lift-off process. In this article, the goal is achieved and metallic nanostructures including nanodots, nanowires, and nanoelectrodes are successfully fabricated.

2. Experiment

The experimental procedure is show in Fig. 1. A solution of 1.25 w.t.% polymethyl methacrylate (PMMA) (molecular weight 960,500 g) in chlorobenzene was spin-coated on a sapphire or silicon substrate. The different substrate could be used for specific goal. For example that sapphire was for nanomaterial growth. After a 30-min soft baking at 150 °C, the PMMA film was produced and also free of pin-holes. The thickness and root-mean-square roughness were around 50 and 0.25 nm, respectively, as determined by AFM measurement.

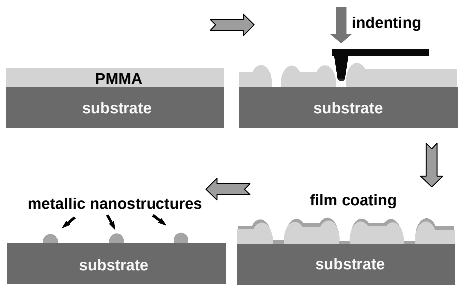

Fig. 1. Schematic diagram of the experimental procedure.

A commercial AFM (Smena-B, NT-MDT, Russia) and rectangular silicon probes (NSC15, MikroMasch, Russia) with a tip radius of 10 nm were employed for the experiment. The probes had listed spring constant of 40 N/m and resonant frequency of 325 kHz. Nevertheless, the measured frequencies were far below the listed value. Since the spring constant is proportional to the square of the resonant frequency for a rectangular probe, the corrected value of 32.5 N/m, which corresponded to a resonant frequency of 293 kHz measured from software, was used for related force calculation. For accurate force measurement, the scanner was first calibrated by a standard grating (TGZ02, MikroMasch, Russia). The detector sensitivity was then characterized by performing a force-distance curve on a silicon substrate with the identical probe used in the indentation described below.

The AFM was operated in the tapping mode for imaging. The contact mode operation was not adopted since the PMMA surface was frequently damaged due to the large spring constant of the employed probes. Each indentation was realized by moving the scanner and thus the tip in the vertical (z-axis) direction toward the sample. A desired pattern was created by locating the tip on designated positions controlled by the software. A gold or copper film was then coated on the sample by ion sputtering or electron-beam evaporation. The sample was finally soaked in acetone to remove the PMMA and dried by nitrogen.

3. Results and discussion

3.1 Nanodot

As can be imagined, the lift-off process will fail unless the indented holes reach the substrate. A large indentation force is required from this aspect. On the other hand, the size of the nanoholes gets larger when the force is higher. Various moving distances were tested and the minimum distance for a successful lift-off was around 180 nm. Using this value, which corresponded to an indentation force of 3.8 μN, a nanohole array was generated on the PMMA film and the image is shown in Fig. 2(a). The pile-up around each hole is apparent, which is similar to those reported previously.[2, 5, 6, 8] After coating a 10 nm thick gold film and removing the PMMA, a gold nanodot array with a dot size of 70 nm and a period of 250 nm was fabricated and the result is shown in Fig. 2(b). In addition to regular array patterns, complicated dot patterns were also created. In Fig. 2(c), the scanning electron microscope (SEM) image of the pattern “NANO” is presented, showing the ease and effectiveness of the present method to generate nanoscale patterns.

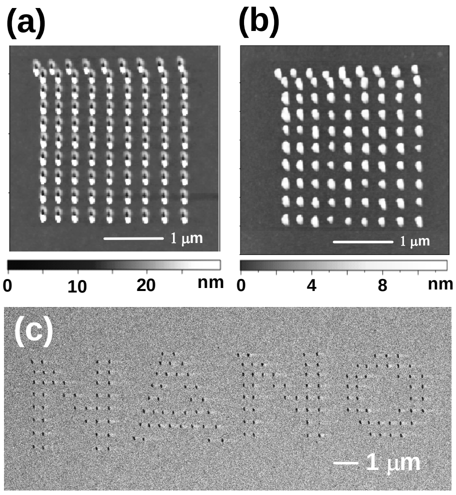

Fig. 2. AFM images of (a) a nanohole array on PMMA and (b) the corresponding gold nanodot array after lift-off. (c) SEM image of a gold nanodot pattern “NANO”.

With the use of the 3.8 μN indentation force, the tip damage was minimal even after hundreds of indentations. The SEM images of a new tip, the tip after 625 indentations and the resultant gold nanodot array are shown in Fig. 3. In addition, it has also been verified that more than 5000 nanodots of sizes less than 100 nm can be fabricated by a single tip. With the use of higher forces, on the other hand, the dot size increased as expected. The results for 4 different forces of 4.3, 5.6, 6.8, and 8.1 μN (top to bottom for every 2 rows), are shown in Fig. 3(d). It is interesting to note that the dots become elongated in one direction under high forces, which may be caused by the twisting of the tip due to a high load.

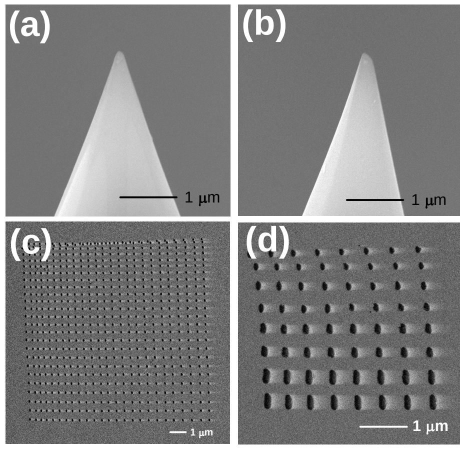

Fig. 3. SEM images of (a) a new tip, (b) the same tip after 625 indentations, (c) the resultant 25 × 25 gold nanodot array, and (d) a gold nanodot array created by 4 different forces of 4.3, 5.6, 6.8, and 8.1 μN (top to bottom for every 2 rows).

3.2 Nanowire

In addition to the fabrication of nanodots, nanowires can also be created by moving the tip along a straight line. In addition, the line width can be controlled by using different carving forces. The results of two nanowires are shown in Fig. 5, and the smallest width achieved so far is around 75 nm.

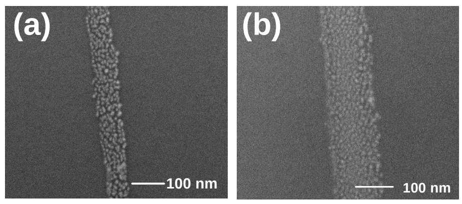

Fig. 5. SEM images of nanowires with different width:(a)75 nm and (b)125 nm.

By evaporating a 4 nm thick gold film and a 20 nm thick copper film, a copper nanowire with a width of 120 nm was produced after lift-off and the image is shown in Fig. 6(a). To verify the electric conduction of the wire, copper pads were also fabricated concurrently by scratching the PMMA with a needle before the AFM nanomachining. The obtained current-voltage curve is plotted in Fig. 6(b). The linear relationship, which corresponds to a resistance of 2.54 kΩ, ascertains the good conductive behavior of the wire. By assuming the wire with a length, a width, and a height of 4.7 μm, 120 nm, and 24 nm, respectively, the calculated resistivity is 1.56×10^{-4} Ω·cm. This value is two orders of magnitude higher than the bulk resistivity of copper. With a further effort, nanoelectrodes were also fabricated on a nanowire by direct cutting with the AFM tip. A pair of nanoelectrodes with a gap of around 50 nm was produced and the image is shown in Fig. 6(c).[11] Simultaneous current measurement during cutting is also plotted in Fig. 6(d), and the zero current after cutting certifies that the nanowire wire was cut off completely.

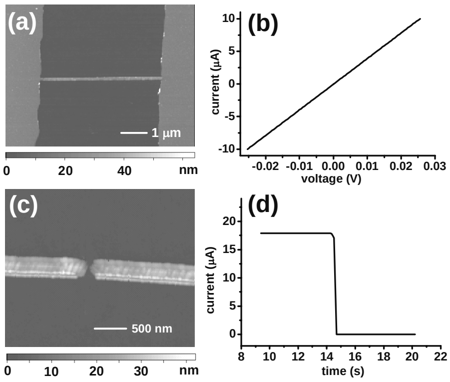

Fig. 6. (a) AFM image of a copper nanowire with a width of 120 nm, and (b) the corresponding *I–V* curve that shows a resistance of 2.54 kΩ. (c) A pair of nanoelectrodes created by direct cutting a nanowire, and (d) the simultaneous obtained current versus time relationship.

4. Force calculation

The indentation force is equal to the product of the tip spring constant and the (static) deflection. In Fig. 6, the force and the vibration amplitude are plotted against the *z*-axis position. (A positive position means that tip is away from the sample and the tip moving distance in an indentation is relative to the origin.) As can be seen clearly, the amplitude decreases as the tip indents the sample and remains zero after a distance *w*. On the other hand, the force (deflection) stays at zero and starts to increase linearly after this distance. As mentioned previously, the minimum moving distance for a successful lift-off is around 180 nm, which amounts to 3.8 μN from Fig. 7.

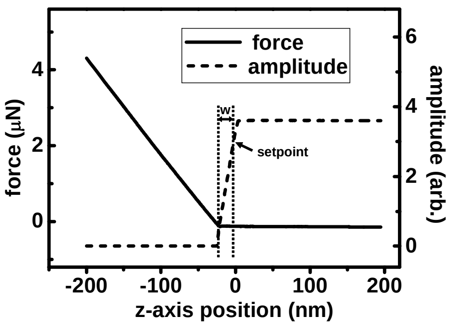

Fig. 7. Cantilever force (deflection) and vibration amplitude versus scanner z-axis position.

It is instructive to use a simple argument to estimate the minimum force. In an indentation,

$$D = w + \delta + d = w + \delta + \frac{F}{k} \qquad (1)$$

where D is the tip moving distance toward the sample, δ the tip penetration depth, d the tip deflection, F the indentation force, and k the tip spring constant. Suppose at the minimum moving distance (~180 nm), the tip just indents to the bottom of the PMMA. With w and δ equal to 30 (from Fig. 6) and 50 nm (film thickness), respectively, the minimum force is therefore 3.25 μN. This value is close to but slightly less than the experimental value as expected. The calculation indicates that the 180 nm is a suitable distance for the fabrication.

There is also a theoretical formula that can be used to estimate the minimum force. In Fig. 6, the force and the distance have a linear relationship. From Sneddon formulism of indentation, the linear behavior indicates a rigid cylindrical punch with the following relationship: [12]

$$F = \frac{2E}{1-\nu^2} a\delta \qquad (2)$$

where E is the Young's modulus, ν the Poisson's ratio, a the cylindrical indenter radius, δ the penetration depth. With the parameters 3×10^9 Pa for E, 0.35 for ν,[13] 10 nm for a, and 50 nm for δ, the calculated minimum force is 3.42 μN. This value is again in good agreement with the experimental value.

5. Conclusion

To summarize, we have presented a method to fabricate metallic nanostructures by a combination of AFM nanomachining and standard lift-off process. By controlling the force of an AFM tip on a thin resist, nanoscale holes and grooves are first created. By coating a metal film and performing the lift-off process, metallic nanodots with a size of 70 nm are successfully fabricated. The width of nanowires could be controlled and got a smallest width 75 nm. Furthermore, nanoelectrodes with a gap of around 50 nm are also produced by cutting a nanowire with the tip. Theoretical calculations for the minimum indentation force have been elaborated and are in good agreement with the experimental value. In addition, tip wear has been proven minimal even after thousands of indentations, which is opposed to the common impression of high risk of tip damage for the one-layer approach.

Acknowledgement

This work was supported by the National Science Council under Grant No. 92-2120-E-007-005.

References

1. H. T. Soh, K. W. Guarini, and C. F. Quate, *Scanning Probe Lithography* (Kluwer, Boston, 2001).
2. X. Jin and W. N. Unertl, Appl. Phys. Lett. 61, 657 (1992).
3. Y. Kim and C. M. Lieber, Science 257, 375 (1992).
4. M. Wendel, S. Kühn, H. Lorenz, J. P. Kotthaus, and M. Holland, Appl. Phys. Lett. 65, 1775 (1994).
5. B. Klehn and U. Kunze, J. Appl. Phys. 88, 3897 (1999).
6. K. Wiesauer and G. Springholz, J. Appl. Phys. 88, 7289 (2000).
7. L. L. Sohn and R. L. Willett, Appl. Phys. Lett. 67, 1552 (1995).
8. V. Bouchiat and D. Esteve, Appl. Phys. Lett. 69, 3098 (1996).
9. S. Hu, A. Hamidi, S. Altmeyer, T. Köster, B. Spangenberg, and H. Kurz, J. Vac. Sci. Technol. B 16,

2822 (1998).
10. M. Rolandi, C. F. Quate, and H. Dai, Adv. Mater. 14, 191 (2002).
11. It should be noted that the cutting speed has a strong effect on the shape of nanoelectrodes. A slow speed usually generates a pair of curved electrodes.
12. I. N. Sneddon, Int. J. Eng. Sci. **3**, 47 (1965). The employed NSC 15 is a conical tip with a diameter of 20 nm and a half angle of 10°. Since the penetration depth is only 50 nm, the approximation as a cylindrical punch is plausible.
13. The Young's modulus for PMMA is from R. J. Young and P. A. Lovell, *Introduction to Polymers* (Chapman & Hall, New York, 1991), p. 420. The Poisson's ratio has minor effect in the calculation and is taken as that of polystyrene.