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Fabrication of metal nanowires by atomic force microscopy nanoscratching and lift-off process

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Abstract
A convenient method for the fabrication of metal nanowires by a combination of atomic force microscopy nanoscratching on a single-layer resist and lift-off process is reported. Various metal nanowires, including Au, Cu, Ni, Al, and Ti, with widths as small as 50 nm are successfully created. The electrical resistivities of the nanowires have also been obtained and found to be in good agreement with reported results.

(Some figures in this article are in colour only in the electronic version)

1. Introduction

Metal nanowires have been the focus of extensive research activities in recent years due to their unusual electrical [1, 2], magnetic [3] and optical properties [4, 5], and applications as nanointerconnects [6, 7], chemical sensors [8–10], etc. Fabrication techniques are naturally of great interest, and various physical, chemical or even biological approaches have been developed. In particular, scanning probe lithography (SPL) [11–13] has been employed for more than a decade, and is in continual use and improvement due to the advantages of ease of operation and relatively low cost.

Among the various SPL techniques, atomic force microscopy (AFM) nanomachining has been adopted since the early years [14–18]. By controlling the contact force between the AFM tip and the sample, desired nanopatterns can be created [15]. Furthermore, with the use of a resist and the application of a subsequent lift-off process, metal nanowires with widths down to 40 nm have been fabricated [16–18]. However, the resists in reported works usually have a bi-layer or tri-layer structure [16–18] and are more complicated than a single-layer resist. In addition, dry or wet chemical etching steps are also required in the processing.

The generation of metal nanowires based on AFM nanoscratching on a single-layer resist has been realized by electrochemical deposition of metal [19, 20]. Nevertheless, the electrochemical process needs a conductive substrate and the deposition parameters are also material dependent. Recently, we have successfully fabricated metal nanowires with a width of around 100 nm on insulating substrates by using a single-layer resist and a straightforward e-beam evaporation procedure [21]. The whole process is indeed simple and effective, and is readily applicable to many metal coatings without complicated parameter adjustment. In this paper, much improved production of various metal nanowires, including Au, Cu, Ni, Al, and Ti, with widths down to 50 nm is presented. The electrical resistivities of the nanowires have also been measured and compared with reported results in the literature.

2. Experiment

The resist film was made of poly(methylmethacrylate) (PMMA) with a thickness of around 50 nm, and prepared by spin-coating from a 1.25 wt% solution in chlorobenzene onto an Si substrate with a 1 μm-thick layer of thermal oxide. A commercial AFM (Smena-B, NT-MDT, Russia) and rectangular silicon probes (NSC15, MikroMasch, Russia) with a tip diameter of 20 nm were employed for the experiment. The AFM was operated in the intermittent-contact mode for imaging.

The experimental procedure is depicted in figure 1. To create a metal nanowire, a straight nanogroove with the desired

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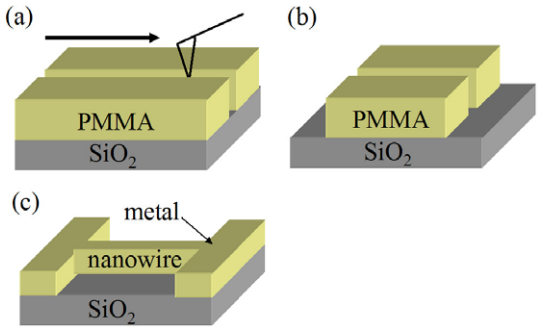

Figure 1. Schematic diagram of the experimental procedure. Note that the contact pads can also be defined first if a specific wire length is not required.

length was first generated by the AFM tip on the resist film at a speed of 2 μm s^{-1} and with a force of around 1 μN.[2] The force was determined from a force–distance curve and was large enough to scratch the resist off the substrate. For electrical characterization, two large regions for contact pads were created at the ends of the nanogroove by additional scratching steps with a blunt AFM tip and a sharp needle tip. (This step could also be performed prior to nanogroove creation if a specific wire length was not required.) A metal film was then coated on the sample by e-beam evaporation. The sample was soaked in acetone in an ultrasonic bath to remove the resist and finally dried by nitrogen gas. The current–voltage (I–V) relationship of the created nanowire was measured in a two-point geometry with the use of a Keithley 2400 source-measure unit.

3. Results and discussion

AFM images of the PMMA film and a nanogroove array after nanoscratching are shown in figures 2(a) and (b), respectively. The nanogrooves appear as though they are elevated lines in figure 2(b) due to the pile-up of the displaced polymer, which was also observed in previous works [15]. A zoomed image of a nanogroove is shown in figure 2(c) and the corresponding cross-section is plotted in figure 2(d). Due to the finite size of the tip apex, the observed depth in the plot is only 15 nm. Although this value is much less than the polymer thickness, the lift-off process was nevertheless successful. The scanning electron microscope (SEM) image of the Au nanowires fabricated from the pattern is shown in figure 2(e). A zoomed image is presented in figure 2(f), and the width is around 70 nm.

So far the minimum width obtained is around 50 nm (see below), but a smaller value can be expected with the use of a thinner PMMA film. It was also found that the nanowire width was mainly affected by the tip quality, and the scratching force only had a minor effect, although a high force could cause a substantial tip wear and also an increase of the width. In addition, it has been verified that more than 50 nanowires such as those shown in figure 2(e) could be fabricated by a single

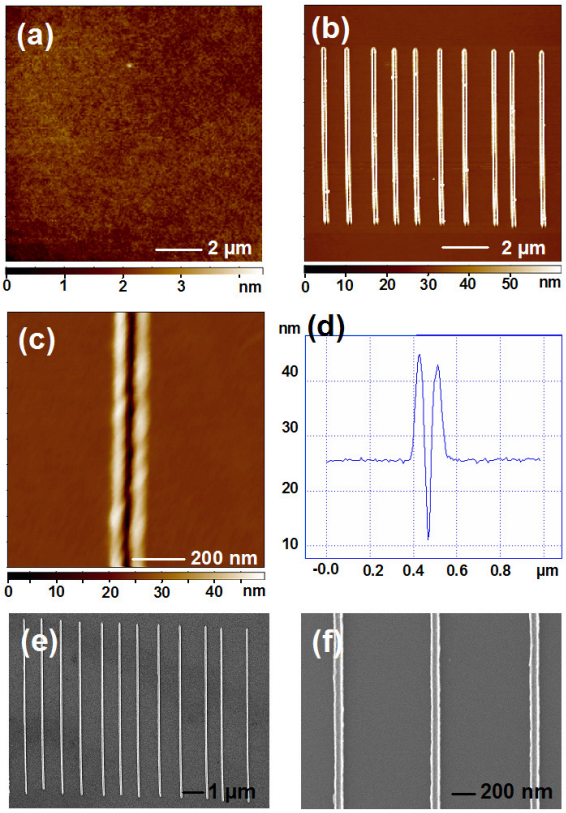

Figure 2. (a) AFM images of the PMMA film, and (b) the result of a nanogroove array after nanoscratching. (c) A zoomed image of a nanogroove, and (d) the corresponding cross-section plot. (e) The SEM image of the Au nanowires fabricated from the pattern, and (f) a zoomed image showing that the width is around 70 nm.

tip with the widths controlled below 70 nm. Further use of the same tip caused an increase of the width that originated from tip wear-off after repeated scratching.

With the addition of contact pads, electrical resistances of the fabricated metal nanowires could be obtained. Figure 3(a) shows the SEM image of an Au nanowire between electrodes that has a width, a thickness, and a length of 65 nm, 15 nm, and 11.5 μm, respectively. The I–V curve of the nanowire is shown in figure 3(b). A linear relationship is clearly seen, and the resistance is 1462 Ω. With the formula $\rho = R\frac{A}{L}$, where ρ is the electrical resistivity, R the resistance, A the cross-section area, and L the length, the resistivity of the Au nanowire is therefore 12.4 $\mu\Omega$ cm, which is roughly five times higher than the bulk value. As a second example, the SEM image and the I–V curve of an Ni nanowire are shown in figures 3(c) and (d), respectively. The Ni nanowire is 50 nm in width, 10 nm in thickness, and 7.0 μm in length. The resistance is 7531 Ω and the resistivity is 53.8 $\mu\Omega$ cm.

In total, nanowires of five different metals with widths as small as 50 nm have been fabricated. The dimensions and the electrical resistivities of representative results are summarized in table 1,[3] along with comparative resistivities

[2] The 1 μN force is substantially lower than the minimum indentation force of 3.8 μN for the creation of nanodots observed in our previous study as listed in [21]. The discrepancy is believed to be due to the different nature of indentation and scratching in nanomachining, but the detail has not been explored.

[3] The listed resistances of the nanowires were measured within a couple of hours after lift-off and showed little variation in further measurements except for the Cu nanowires, of which the resistances increased by orders of magnitude within a few days under ambient conditions.

Table 1. Widths, lengths, thicknesses, resistances and resistivities of the fabricated nanowires (see footnote 3) and comparative resistivities reported in the literature. Note that only reported results from nanowires with widths and thicknesses less than 100 nm are listed.

	W (nm)	L (μm)	T (nm)	R (Ω)	ρ ($\mu\Omega$ cm)	ρ ($\mu\Omega$ cm) in literature	Bulk[a] ρ ($\mu\Omega$ cm)
Au	60	3.9	15	595	13.7	7[b]	2.35
	65	10.2	15	1 255	12.0	6[c]	
	65	11.5	15	1 462	12.4	30[d]	
	70	9.8	15	987	10.6	13[e]	
Cu	50	3.5	15	1 310	28.1	17[f]	1.67
	50	4.4	15	2 227	37.0		
	50	6.5	15	2 793	32.2		
	60	6.4	20	926	17.4		
Ni	50	7.0	10	7531	53.8	9[c] 3×[g]	6.84
	55	15.6	10	10 205	36.0		
	60	3.7	10	3 267	53.0		
	65	5.4	10	4 486	54.0		
Al	50	8.0	10	2 545	15.9	NA	2.65
Ti	50	12.0	10	358k	1492	1300[h]	42
	55	10.9	10	213k	1074		

[a] Reference [22].
[b] Reference [1].
[c] Reference [23].
[d] Reference [24].
[e] Reference [25].
[f] Reference [26].
[g] Reference [27].
[h] Reference [28] (see footnote 4).

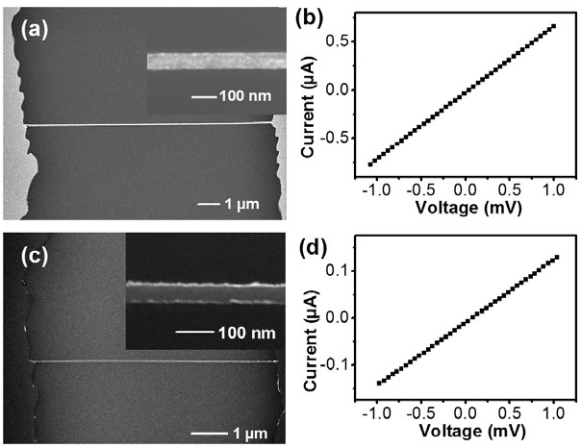

Figure 3. (a) The SEM images of an Au nanowire with a width of 65 nm across two electrodes, and (b) the corresponding I–V curve. (c) The SEM images of an Ni nanowire with a width of 50 nm, and (d) the corresponding I–V curve.

reported in literature [1, 22–28],[4]. (Note that the listed reported results are from nanowires with widths and thicknesses of less than 100 nm.) It can be seen that the present results are in good agreement with reported values although they are slightly higher. It is known that the increased resistivity in a metal nanowire is mainly due to surface and grain boundary scattering effects [1], and oxidation [26] if it occurs. Therefore, the origin of the present higher resistivities can be reasonably attributed to the small thicknesses of the fabricated nanowires compared with those reported. This point is also exemplified in the case of Cu, as the resistivity for the last Cu nanowire shown in table 1 is much lower than the others and also consistent with the listed reported value.

From table 1, it is also obvious that the resistivities of the nanowires are roughly 5–10 times higher than bulk values except for Cu and Ti. Therefore, it can be inferred that the influence of oxidation on the resistivities of Ni and Al nanowires is not significant in consideration of the non-oxidative nature of Au. This argument is consistent with known facts that the natural oxide layers of Ni and Al prevent further oxidation of the two metals. On the other hand, the more resistive behaviour of the Cu and the Ti nanowires when compared with bulk materials indicates that there is a substantial extent of oxidation in the two metals, which is again consistent with their more oxidative nature. It should be noted that the resistances of the Cu nanowires increased by orders of magnitude within a few days and the wires finally became non-conductive (see footnote 3). Such behaviour has also been observed previously [26].

Other nanostructures could also be created directly from the fabricated nanowires. By cutting a nanowire with the AFM tip, nanoelectrodes with a gap of around 60 nm could be generated routinely. On the other hand, a gap as narrow as 40 nm was achieved on an Au nanowire, and the result is shown in figure 4(a). Such nanoelectrodes are valuable for the construction and studies of nanoelectronic devices. Furthermore, repeated cuttings were employed to create rectangular nanostructures, and the result is shown in figure 4(b). The sharp square edges in these metal nanostructures are potentially interesting subjects for

[4] The resistivity is calculated from the I–V curve in figure 2, which shows a resistance of 30 kΩ for a Ti nanowire of 30 nm in width, 7 nm in thickness and 500 nm in length.

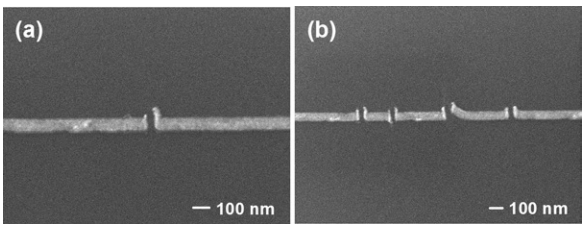

Figure 4. SEM images of (a) a pair of Au nanoelectrodes with a gap of 40 nm made by a cutting on an Au nanowire, and (b) rectangular nanostructures made by repeated cuttings.

experimental investigations of light distribution in the optical near-field [4].

4. Conclusion

In summary, a convenient and effective method to fabricate nanowires by a combination of AFM nanoscratching on a single-layer resist, e-beam evaporation, and lift-off is presented. Metal nanowires, including Au, Cu, Ni, Al, and Ti with widths as small as 50 nm have been fabricated. The electrical resistivities of the nanowires have been obtained and they are consistent with reported values. For the Au, Ni and Al nanowires, the resistivities are roughly 5–10 times higher than bulk values, whereas the resistivities of the Cu and Ti nanowires are even higher due to more extensive oxidation. In addition, other nanostructures such as nanoelectrodes have also been made by additional cuttings on a nanowire.

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