Fabrication of mesoscopic Nb wires using conventional e-beam lithographic techniques

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Conventional electron beam lithography has been used to fabricate mesoscopic Nb wires with a superconducting transition temperature above 7.0 K. The typical line width and the thickness were 200 nm and 45 nm respectively. Nb was deposited in an ultra-high vacuum evaporation chamber using electron gun heating. All samples exhibited a normal-superconducting transition. The transition temperature decreased with thickness and line width. To demonstrate the feasibility of two angle evaporation techniques we also fabricated small Nb/(Al-)AlOₓ/Nb tunnel junctions.

85.25.K, 73.63, 73.23.H.

A number of nanometer-scale devices such as single-electron transistors [1], superconducting quantum bits [2], and other mesoscopic superconducting devices [3] have been realized using the self-alignment technique which provides submicron accuracy [4]. Self alignment is achieved by the shadow evaporation technique, commonly used with the resist polymethylmethacrylate (PMMA) and co-polymer [P(MMA-MAA)] as a double layer stencil mask patterned by electron-beam (e-beam) lithography. Until now this conventional shadow evaporation technique has been applied successfully for the soft metals such as Al, Cu and Pb. For the refractory metals such as Nb, W or Ta, however, this technique is known to be difficult to apply. In particular Nb is a promising alternative to the soft metals for superconducting nano devices such as single-electron transistors and quantum bits, because of its large superconducting gap and high stability under thermal cycling. A demonstration that it is possible to fabricate nanostructures by conventional e-beam lithography would therefore be highly significant.

The problems with the application of the conventional technique have been ascribed to the partial decomposition of the PMMA-co-polymer double layer during the evaporation of the refractory metals [5]. The resulting outgassing from the resist [6] and consequent contamination of the deposited Nb would then explain the changes in the electronic properties of the deposited Nb. Contrary to this common experience, we were able to fabricate mesoscopic Nb wires with zero-field critical temperatures T_c higher than 7.0 K by using the conventional shadow evaporation technique.

The results presented here were obtained with an e-beam lithography process which did not have any special features but followed the most common procedures with conventional recipes. We deposited a double layer of PMMA-P(MMA-MAA) on the oxidized Si substrate with the thickness of the oxidization layer of about 250 nm. The spinning rates for PMMA and P(MMA-MAA) were 3000 rpm and 6000 rpm, respectively, and the spinning time was 30 seconds for both. The thickness of the PMMA and P(MMA-MAA) were measured to be about 270 nm and 300 nm, respectively. The resist was baked at 160°C for about 60 minutes. We then drew a pattern of wire (see Fig. 1) by using a scanning electron microscope (JEOL, JSM 840A). The line width was about 200 nm. To develop the upper layer of the PMMA resist we immersed the sample in a mixed (1:2) solution of methyl iso-butyketone and isopropyl alcohol for 12 seconds. To develop the lower layer of the P(MMA-MAA) resist and to form an undercut, the sample was dipped into a solution (1:2) of methyl glycol and methanol for 15 seconds.

The Nb (99.9 %, Goodfellow) was evaporated onto the patterned substrate in an ultra-high vacuum (UHV) chamber equipped with a cryo-vacuum pump (Cryo-Torr High vacuum pump, CTI-Cryogenics) and a liquid nitrogen trap. The pressure of the UHV chamber was 2 x 10⁻⁸ mbar, the evaporation rate was about 0.45 nm/s and the power of the electron gun was 2 kW. A crucial feature in the evaporation chamber was the 40 cm distance between the substrate and the Nb crucible. This relatively large separation reduced the indirect heating of the samples when evaporating Nb. All samples were made using the same evaporation parameters.

We have measured the change of sample resistance with temperature, R(T), for the five samples fabricated (see Fig. 2 and Table 1). The typical dimension of the wires is about 200 nm in width and 11 µm in length while the width of the 2D film is about 10 µm.

![FIG. 1. Scanning electron micrograph of a Nb wire with thickness 45 nm and width 200 nm.](image-url)
Details of sample parameters are summarized in Table 1. The sample resistance has been measured by the conventional lock-in amplifier technique in a four probe measurement configuration. The bias current level was 10 nA for wires and 100 nA for 2D films. A calibrated diode sensor (DT470, Lake Shore Cryotronics) was used to monitor the temperature.

Note that all the samples become superconducting, but with a transition temperature \(T_c\), which depends on the sample dimension. One of the samples, the 2D Nb film with a thickness of 135 nm (2D-1) showed \(T_c = 9.1\) K, close to that of bulk Nb, 9.26 K (see the inset of Fig. 2). \(T_c\) was defined as the temperature where the sample resistance is reduced to one half of its resistance at 10 K. We have found that the superconducting transition temperature decreases with Nb thickness or width as shown in Fig. 2. Wire-3 with dimensions typical of those in applications had a \(T_c\) of 7.0 K and the resistivity ratio \(\rho_{295K}/\rho_{10K}\) of 1.5.

The reason for the reduction of \(T_c\) from the bulk value is worth considering. For this purpose we plot \(T_c\) vs the inverse mean free path \(1/l\) in Fig. 3 where \(l\) has been calculated from the resistivity at 10 K using the Drude formula \(\rho l = 8.7 \times 10^{-12} \Omega\text{cm}^2\) for Nb [7]. The Fig. 3 shows that \(T_c\) is decreased as the mean free path \(l\) decreased. Our results are similar to the previous experimental observations [8] where the authors concluded that \(T_c\) depends on the disorder of the sample. The authors of Ref. 8 suggested that disorder in Nb smears out the density of states of Nb metal and consequently reduce the density of states at the Fermi level, resulting in a decrease of \(T_c\). Since all our samples have been evaporated under an equal evaporation condition, the reduction of \(T_c\) of our samples is consistent with the suggestion that it depends mainly on disorder of the sample.

To demonstrate the feasibility of the conventional two angle evaporation technique and to take advantage of the high critical temperatures reached we have also fabricated Nb/(Al-)AlO\(_x\)/Nb junctions with a single electron transistor geometry (see the inset of Fig. 4). The 15 nm thick Al layer was evaporated on the 67 nm thick Nb layer. The Al layer was subsequently oxidized in a static oxygen pressure of 100 mbar for 5 minutes. Then the third layer of Nb was deposited at a different angle. We measured the current vs voltage \((I-V)\) characteristic at 4.2 K for the Nb/(Al-)AlO\(_x\)/Nb junctions with a room temperature junction resistance 60 k\(\Omega\) (Fig. 4). The \(I-V\) curve clearly shows a superconducting gap, the size of which is very close to \(4\Delta_{Nb}\) where \(\Delta_{Nb}\) is the bulk Nb superconducting gap energy (\(\Delta_{Nb} = 1.5\) meV). These results are promising for the fabrication of Nb based nano-scale devices.

In conclusion, using the conventional e-beam lithographic technique we have fabricated mesoscopic lithographic Nb wires with a width of about 200 nm showing \(T_c\) higher

FIG. 2. Resistance vs temperature for various samples. Inset: resistance vs temperature for 2D-1 on an expanded scale.

FIG. 3. \(T_c\) vs inverse mean free path \(1/l\) at 10 K. The mean free path \(l\) at 10 K has been calculated from the formula \(\rho l = 8.7 \times 10^{-12} \Omega\text{cm}^2\). \(T_c\) of bulk Nb is 9.26 K. The dashed line is a guide for the eye.

FIG. 4. \(I-V\) characteristic of the Nb/(Al-)AlO\(_x\)/Nb junctions at 4.2 K. \(\Delta_{Nb} = 1.5\) meV. Inset: scanning electron micrograph of the Nb/(Al-)AlO\(_x\)/Nb junctions fabricated by the conventional two angle evaporation technique. The length of the white bar is 1 \(\mu\)m.
than 7.0 K. In addition we could also make mesoscopic
Nb/(Al-)AlO$\text{x}$/Nb tunnel junctions showing a superconducting gap close to that of bulk Nb at 4.2 K.

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| TABLE I. Results of the various samples. We denoted as 2D films the macroscopic samples with width 10 $\mu$m. $w$, $t$, $L$, $\rho$ and $l$ are the sample width, thickness, length, resistivity and mean free path at 10 K. $\rho_{295K}/\rho_{10K}$ is the resistivity ratio for the two temperatures, 295 K and 10 K. |
|---|---|---|---|---|---|---|---|
| Sample | $w$ ($\mu$m) | $t$ (nm) | $L$ (nm) | $T_c$ (K) | $\rho$ ($\mu\Omega$cm) | $l$ (nm) | $\rho_{295K}/\rho_{10K}$ |
| 2D-1 | 10 | 135 | 300 | 9.1 | 7.6 | 11 |
| 2D-2 | 10 | 67 | 160 | 8.9 | 15 | 5.7 | 2.2 |
| Wire-1 | 0.23 | 67 | 11 | 8.0 | 27 | 3.2 |
| Wire-2 | 0.23 | 45 | 11 | 7.3 | 23 | 3.8 | 1.6 |
| Wire-3 | 0.20 | 45 | 11 | 7.0 | 33 | 2.6 | 1.5 |

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