Fabrication of Nb/Al(AlOx)/Nb DC SQUID by focused ion beam sculpturing

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Highly reproducible Nb/Al(AlOx)/Nb Josephson junction based direct current superconducting quantum interference devices (DC SQUID) were fabricated by three dimensional etching using focused ion beam. Hysteretic and non-hysteretic DC SQUID with critical current ranging from 25 to 1100 µA were fabricated by varying the Al barrier and oxygen exposure time. The fabricated DC SQUIDs have shown periodic flux dependence with high modulation factor reaching a value of 92% at 4.2 K.

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Direct current superconducting quantum interference devices (DC SQUIDs) are formed by enclosing two Josephson junction (JJs) in a superconducting loop. They were primarily used as sensitive magnetic flux detectors and as voltage standards, but in recent years demonstration of SQUIDs as nanoscale position sensors, qubit readout and scanning SQUID microscopy has re-opened the interest in these devices [1-4]. SQUIDs based on Nb/Al(AlOx)/Nb JJ have relatively high transition temperature (Tc), high flux-voltage modulation factor and good thermal recyclability and can be fabricated with a wide range of critical currents (Ic) [5]. Traditionally Nb/Al(AlOx)/Nb SQUIDs with size ranging from few to several hundred microns are fabricated in sequence of steps involving several photolithography and anodization processes, and it also requires in-situ etching for the deposition of top Nb electrode [6]. In spite of the superior properties of Nb/Al(AlOx)/Nb SQUIDs, the fact that a multistep-process is needed for device fabrication has limited its popularity.

The advancement in the nanofabrication techniques lead to the realization of nano sized SQUIDs based on Al/AlOx/Al junctions and Nb nanobridges [4, 6]. SQUIDs based on Al/AlOx/Al are fabricated using a single shadow evaporation process, but the relatively low transition temperature of Al limits the range of possible applications [4]. Although the SQUIDs based on the Nb nanobridges have relatively high Tc and are relatively easy to fabricate, these SQUIDs are highly hysteretic and the nanobridges typically lose their sinusoidal current phase relationship when cooled to very low temperatures [4, 8]. Compared to alternative nanofabrication techniques, focused ion beam (FIB) offers the possibility to etch the samples in 3D [6]. Recently Watanabe et al. have demonstrated the application of 3D FIB etching for the fabrication of JJs and single electron transistors (SET) [10, 11].

We report here a process to fabricate Nb/Al(AlOx)/Nb junction based DC SQUIDs using only a single photolithography step and a single 3D FIB etching. Moreover, we present the characteristics of the fabricated hysteresis and non hysteresis DC SQUIDs at low temperature (4.2 K and 0.1 K). Nb/Al(AlOx)/Nb trilayer was deposited on SiN coated Si substrate at an Ar pressure of 5 x 10^-3 mbar at room temperature by DC magnetron sputtering. Thickness of the base and top Nb electrodes was fixed as 350 nm, whereas the thickness of the Al barrier and oxidation conditions were varied for different samples. SQUID loop, magnetic flux line and bonding pads are patterned on the trilayer films using a single photolithography step. The SQUID loop has an area of 40 x 40 μm² and the flux lines were fixed 10 μm away from the SQUID loop. JJs in the DC SQUIDs were fabricated using the process similar to the one used by Watanabe et al. for the fabrication of trilayer SET [11]. The sample with the trilayer SQUID loop is mounted on wedge shaped (45°) sample holder. In the 3D FIB etching process the JJs were fabricated on the arms of the SQUID loop by etching in perpendicular and parallel directions to the sample surface plane. Initially using perpendicular etching with 2.8 nA Ga ion current a 1 micron wide section is formed on each arm of the SQUID loop. The width of these sections is further reduced down to 300 nm using a current of 28 pA. Then JJ’s were fabricated on the trilayer bridge by parallel etching, in which the top and the bottom Nb layer

FIG. 1: Scanning electron microscope (SEM) image of the Nb/Al(AlOx)/Nb Josephson Junction (JJ) fabricated by 3D FIB etching. The inset shows a schematic of JJ structure.
were etched with 1.5 pA Ga ion current as schematically shown in the inset of Fig. 1. Two nominally identical JJs as shown in Fig. 1 with lateral dimension of 0.12 μm² were fabricated in each arm of the SQUID loop.

Table I shows the critical currents measured in the sweep up and sweep down directions respectively. In all the DC SQUIDs except DS-B and DS-D, we have observed behavior is due to the decrease in the critical current density of the junctions determined from the I-V increased from 750 to 350 μA with the increase in the oxygen exposure time. As the fabricated JJs are of similar size the observed behavior is due to the decrease in the critical current density of the junction with the increases in the oxygen exposure time.

The flux response of the hysteretic SQUID was measured for I₀ of 750 and 765 μA in sweep up and sweep down directions.

The critical current of the SQUID decreased from 750 to 25 μA after anodization process. Device DS-A after anodization process is characterized using FIB. Device DS-A after anodization process was measured for 'hysteretic' and 'non-hysteretic' respectively. The parameter ∆Ic is given by ∆Ic = Ic1 - Ic2, where Ic1 and Ic2 are the critical currents measured in the sweep up and sweep down directions respectively.

The critical current of the SQUID decreased by variation from the mean value. ± was found to be highly reproducible with less than 6% variation from the mean value.

Table I: The parameters of the different DC SQUIDs fabricated using FIB. Device DS-A after anodization process is labeled as device DS-B. The parameter D = Pτ, where P is the oxygen partial pressure and τ is the oxygen exposure time, characterizes the oxidation dose. In the Ic column H and NH stands for 'hysteretic' and 'non-hysteretic' respectively. The parameter ∆Ic is given by ∆Ic = Ic1 - Ic2, where Ic1 and Ic2 are the critical currents measured in the sweep up and sweep down directions respectively.

| Device | Nb/Al/Nb (nm) | D (torr s) | Ic (μA) | ∆Ic (μA) | J0 (kA cm⁻²) |
|--------|---------------|------------|---------|----------|--------------|
| DS-A   | 350/5/350     | 4.8        | 750 (H) | 25       | 300          |
| DS-B   | 350/5/350     | 4.8        | 390 (NH)| 160      |              |
| DS-C   | 350/5/350     | 12         | 334 (NH)| 130      |              |
| DS-D   | 350/5/350     | 480        | 25 (NH) | 2.4      |              |
| DS-E   | 350/3/350     | 6          | 1070 (H)| 70       | 450          |

**TABLE I:** The parameters of the different DC SQUIDs fabricated using FIB. Device DS-A after anodization process is labeled as device DS-B. The parameter D = Pτ, where P is the oxygen partial pressure and τ is the oxygen exposure time, characterizes the oxidation dose. In the Ic column H and NH stands for 'hysteretic' and 'non-hysteretic' respectively. The parameter ∆Ic is given by ∆Ic = Ic1 - Ic2, where Ic1 and Ic2 are the critical currents measured in the sweep up and sweep down directions respectively.
The voltage modulation factor of the SQUID calculated using \((V_{\text{max}} - V_{\text{min}})/V_{\text{max}}\) × 100% in this region is found to be 96% (where \(V_{\text{max}}\) and \(V_{\text{min}}\) are respectively the maximum and minimum voltage measured with respect to the flux changes). The flux response curve becomes symmetric and the modulation factor decreases to 19% at \(I_b\) of 765 \(\mu\)A and the flux response curve for the sweep up and down direction become identical as shown in Fig. 3.

The flux response of the non-hysteretic DC SQUID (DS-B) is shown in Fig. 4b. This SQUID did not exhibit flux sensitivity for \(I_b\) below 350 \(\mu\)A and above 450 \(\mu\)A, and showed periodic modulation of the voltage within this range as shown in Fig. 4b. In this non-hysteretic SQUID no switching like behavior is observed and the voltage is rather smooth and symmetric with respect to \(V_{\text{max}}\) with relatively high modulation depth. The flux response curves of this SQUID are identical irrespective of the direction of the \(I_f\) sweep and hence the flux response with respect to sweep up direction is only plotted here. The calculated modulation factor is plotted against \(I_b\) in Fig. 4b. The modulation factor increased from 60% at \(I_b\) of 350 \(\mu\)A and reached a maximum value of 92% at \(I_b\) of 392 \(\mu\)A. After reaching the maximum the modulation factor gradually decreases with the further increase in \(I_b\) as shown in Fig. 4b. The relatively high modulation factor indicates that the two JJs of the SQUID have similar critical currents, demonstrating thus the good repeatability of the FIB fabrication technique.

The operation of the DC SQUID (DS-A) was tested from 4.2 K down to 0.1 K. The critical current of the junction increased from 750 \(\mu\)A at 4.2 K to 1880 \(\mu\)A at 0.1 K. The flux response of this SQUID measured at 0.1 K had shown periodic voltage modulation with a modulation factor of 80%. These results demonstrate that DC SQUIDs fabricated in the present process have very high modulation factor over a wide range of temperatures compared to the DC SQUIDs made of Nb nanobridges.

In summary, Nb/Al(AlOx)/Nb DC SQUIDs with nanoscale JJs were successfully fabricated using relatively simple process consisting of a single photolithography step and a single 3D FIB etching step. Using this process hysteretic and non-hysteretic DC SQUIDs were fabricated by varying the Al barrier thickness and oxidation conditions. Hysteretic SQUIDs exhibited asymmetric flux response curve with high modulation factor. Non-hysteretic SQUIDs have shown smooth symmetric flux response, and the voltage modulation factor in this case reached a maximum value of 92% near the critical current of the SQUID.

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