Fine optimization of Josephson critical current in SQUID devices by thermal annealing

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Abstract. In the present article an experimental study regarding optimization and fine-tuning and of Superconducting QUantum Interference Device (SQUID) parameters by a thermal annealing is presented. It allows to modify the parameters in order to meet a specific application or to adjust the device parameters to reduce the magnetic field noise preventing the work instability conditions due to a critical current value that exceed the expected one. In particular, we carried out the measurements of the critical current and the voltage swing as a functions of the annealing temperatures or time showing the effect of such procedures on the magnetic field spectral density of the SQUID magnetometers. The experimental results demonstrate that it is possible to optimize the main device parameters reducing the SQUID noise to a suitable value improving the stability of working operations.

1. Introduction
The Superconducting QUantum Interference Device (SQUID) is almost 60 years old but nowadays many scientific and technological researches are interested in its applications due to the outstanding magnetic field sensitivity. In fact, SQUID sensors still remain the best choice in the application that requires both extreme sensitivity and practicality. Even if the SQUID are successfully employed in many fields such as the nanomagnetism, geophysics or basic physics [1-4], the most well-established applications are the magnetoencephalography [5-8] and in quantum computation where the SQUIDs are employed as flux quantum bits (qubit) [9-11]. Here, a very good control of the most important parameters of the SQUID play a key role together with a good reliability and robustness. In the flux qubit, for example, the circuit is designed to give a double-well potential which height depends on the critical current of the Josephson elements.

A SQUID is based on a superconducting ring that includes two Josephson junctions which phase difference is related to the flux of magnetic field that pass through the superconducting ring. A fundamental feature of a SQUID is the inductance parameter that is proportional to the critical current via the SQUID loop inductance (\(\beta_L = LI_0/\Phi_0\)). Once the design is established, the inductance value is fixed, while is still possible to act on the critical current allowing to modify \(\beta_L\) and, as a consequence, the most
characteristics of a SQUID such as the voltage swing, the modulation depth, the magnetic flux noise spectral density as well as the double-well potential in the case of rf-SQUID [1]. An easy technique to obtain the critical current change is the thermal annealing. In this paper, we report an experimental study about the fine control and the optimization of the main SQUID parameters. In particular, we show that by thermal annealing it is possible to modify the critical current of SQUID magnetometers continuously with a resolution of a few percent up to a reduction of about one order of magnitude of the initial critical current. In such a way, it is possible to improve the stability and noise performance of a SQUID magnetometer as it is evident from the voltage-flux characteristic and the spectral density of the magnetic flux and field noise.

2. Fabrication and device design

The fabrication of the SQUID magnetometers exploits the all-refractory niobium technology and is based on different steps to obtain a fully integrated device [12-13]. The process starts with the trilayer (Nb/Al-AlOx/Nb) deposition on oxidized silicon wafer after defining the pattern by optical photolithography. During the trilayer deposition, the three metallic film are deposited without vacuum interruption by dc-sputtering in a high vacuum station at basic pressure of less than 3 \times 10^{-8} Torr thanks to both turbo molecular pump and ion pump action. The sputtering process is performed in a plasma of argon, whose pressure is fine controlled by a quartz crystal valve keeping in the vacuum chamber a stable pressure of 1.5 mTorr. The Nb base layer, having a thickness of 200 nm, is deposited at a rate of 1.5 nm/s. After that the initial conditions restored, the aluminum layer having a thickness of 7 nm is deposited at an effective rate of 0.09 nm/s, by rotating the wafer carrier to ensure film uniformity. The AlOx tunnel barrier is achieved by thermal oxidation by filling in the vacuum chamber dry oxygen at pressure of 250 mbar for 1 hour. In such a way, a critical current density of 70 A/cm² is expected. Finally, the Nb top electrode, 350 nm thick, is deposited in the same conditions of base electrode. After the deposition, a lift-off process is performed in acetone to define a final trilayer geometry. An optical photolithography step defines a square window type areas of 16 µm² where, by a Selective Niobium Anodization Process (SNAP), the Josephson junctions are realized. A further insulation is provided by a 120 nm thick SiO₂ film deposited by a rf-magnetron sputtering. To remove top Nb in selected areas to prepare the contact pads for electrical wiring a Reactive Ion Etching (RIE) process in CF₄ plasma is performed. The next step realizes the shunt and damping resistors by using a dc-magnetron sputtering housed in vacuum system depositing a thin film of Au-Pd having a thickness of 300 nm and deposited at a rate of 2.0 nm/s. Again the lift-off process defines a shape of these resistors. The sheet resistance is 1.0 Ω/sq at T = 4.2 K resulting in a resistor value R of about 3 Ω. The last fabrication step consists of wiring deposition including the realization of the SQUID washer; by using the system provided of an ion gun, the etching cleaning process is carried out before to deposit by dc sputtering, a 500 nm thick Nb film at a rate of 1.0 nm/s, whose geometry is defined, as in the previous steps, by an optical photolithography step and a subsequent lift-off process. This fabrication process allows to obtain all refractory niobium junction exhibiting a quality factor \( V_M > 80 \) mV at T = 4.2 K.

The device consists of a single square coil pick coil \( (A_p=16 \text{ mm}^2) \) connected in series with a multiturn input coil which is magnetically coupled to the SQUID loop in a washer configuration. An integrated feedback coil in bipolar configuration to reduce cross talk effect and an additional positive feedback (APF) circuit consisting of a square coil surrounding the pickup coil and a resistor network are integrated in same chip containing the magnetometer [12] (figure1).
3. Thermal annealing procedure and experimental results

The Josephson current in a SQUID is due to the tunnelling of Cooper pairs through a thin barrier between two superconductors when an overlap of the relative macroscopic wave-function occurs. The thickness of the insulator barrier sets the maximum value of this current known as Josephson critical current which density shows an exponential decay according to the equation $J_c = J_{c,0} e^{-\alpha t}$, where $\alpha$ is a coefficient depending on superconductor and insulator materials and $t$ is the thickness of the insulator barrier [14]. Therefore, even a small change in the barrier thickness affects appreciably the density of critical current that, especially in the fabrication of a large number of Josephson devices, can result different with respect to the design value. It is well known that a thermal annealing process allows increasing, in a reliable way, the thickness of the insulator barrier, so a change of Josephson critical density can be obtained also after the fabrication [15-18]. In fact, the thermal treatment can increase the thickness of the tunnel barrier (AlOx) inducing the percolation of reactive oxygen coming from surface niobium oxide through the niobium granular structure or the reaction between the unbound oxygen embedded at interstitial places close to the tunneling barrier and aluminum atoms.

So, a well-controlled thermal annealing can be a suitable tool to change the critical current of Josephson junction and, in particular, of SQUID magnetometers. To this aim, to study experimentally the effect of the annealing on fully integrated SQUID magnetometer described in previous section, we have employed a programmable hot plate (CEE Brewer Science 1100) having a resolution of 0.1 °C resolution and a maximum temperature up to 280 °C using it in hard contact mode by a suitable vacuum to ensure a uniform and reliable temperature. An alternative way to do an annealing of the devices is to use a laser that allows you to perform a selectable annealing with a high spatial resolution (few µm), but the heating temperature is more difficult to control [19-20].

After each annealing step, the SQUID magnetometers have been characterized at liquid helium temperature in a two concentric shields made of a superconductor (lead) and a high permeability material (μ-metal) and using a radio frequency filter on electrical connections to the readout electronics [21-22]. The white magnetic flux noise has been measured by using a direct coupled scheme based on a very
low noise readout electronics and in Flux-Locked-Loop (FLL) scheme to linearizes the SQUID output increasing the linear dynamic range[23-24]. The whole amplification stage is integrated on a miniaturized detection circuit and carefully shielded by a copper box. In particular, the full device characterization consisting of the measurements of the critical current, voltage-magnetic flux characteristic, spectral density of the magnetic field and flux noise has been carried out.

Figure 2a shows the critical current behavior while the figure 2b depicts the voltage swing as a function of the annealing temperature ranging from 150 °C to 240 °C. Each annealing step lasts 30 minute in which the sample was heated, in hard contact mode, without the chip carrier. For both I_c and ΔV, we have obtained a reduction of about one order of magnitude with respect to the initial values. It is worth to note that in both cases a slow variation occurs for lower temperatures, while the values decrease faster above 180 °C. The same behavior has been observed for different devices. Therefore, we can supposed a not linear dependence of oxygen migration process towards the barrier by the annealing temperature. In the figure, each step increase of 10 °C.

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However, we would stress that it is possible to have a much small variation of critical current by decreasing the temperature steps. In particular, in the range of 180-240 °C we have been able to induce in a very reliable way a critical current variation as low as 2-3 % for a temperature step of 2 °C. Since in both dc and rf SQUID the inductance parameter (β_L=LI_c/Φ_0 being L the loop inductance) depends linearly on critical current, this procedure enable us to modify the β_L of a SQUID by about a factor 10. In the figure 3, we report the voltage flux characteristics and the spectral density of the field noise measured at liquid helium temperature of the SQUID under investigation. Figure 3a depicts the characteristic relative to the untreated sample while figure 3b reports measurements where the overall annealing process produced the best magnetic field noise (T=200 °C). In order to verify the possibility to modify in a wide range the parameter of the SQUIDs, we have choose a sample having a high critical current showing a peaked V-Φ characteristic and a high magnetic noise level. As it is evident from the figure 3a, the V-Φ curve shows a very sharp side due to high critical current. Note that the magnetic noise of the device without any annealing (fig.3a) is more than 4 time greater than that after the optimized annealing demonstrating full effectiveness in tuning and optimizing SQUID parameters.

From a practical point of view it should be emphasized that the noise of 13 fT/Hz^{1/2} shown by the SQUID magnetometer prevents MEG applications, while the same magnetometer after the annealing process can be applied in all high sensitivity applications including the MEG.
Figure 3. a) Spectral densities of the magnetic noise of a SQUID and the voltage swing as a function of the external magnetic flux of a SQUID magnetometer without annealing. b) The same device characteristics after an annealing at a temperature of 200 °C.

In order to obtain information also on the temporal dependence for a fixed temperature of the annealing process, we made a complete characterization on another SQUID fixing the annealing temperature and varying the time. In particular, we have chosen an annealing temperature of 170 °C and increased the time with a step of 30 minutes.

Figure 4 reports the critical current, the voltage swing and white spectral density of the magnetic field noise measured at T=4.2 K as a function of the annealing time for a fixed annealing temperature (T_a=170 °C).

Figure 4 reports the critical current, the voltage swing and white spectral density of the magnetic field noise measured at T=4.2 K as a function of the annealing time for a fixed annealing temperature (T_a=170 °C). Figure 4 reports the critical current, the voltage swing and white spectral density of the magnetic field noise measured at T=4.2 K as a function of the annealing time for a fixed annealing temperature (T_a=170 °C). From the figure, it is evident that there are slight variations as a function of the time. In particular, after 150 minute of annealing we have measured critical decrease of about 20% and even less for the voltage swing. A greater variation is observed for the magnetic field noise since even small decreases of critical current can have a significant effect on the APF gain and on the stability of the SQUID. The same measurements have been made also
for different annealing temperature and we have observed the same behavior. Due to the observed slight variation, this procedure can be effectively used to perform a fine-tuning of the device parameters.

4. Conclusions

In conclusion, an experimental study on the annealing of SQUID magnetometers have been presented. In particular, detailed measurements of main SQUID parameters such as critical current, voltage swing and magnetic noise as a function of the annealing temperature have been shown. These measurements highlight the possibility to optimize the performances and to fine-tune the most important parameters of a SQUID. We believe that this study is very important for application that require the optimization and the fine control of the SQUID key parameters. In addition to high sensitivity applications such as the biomagnetism, it can certainly be useful for superconducting quantum computing where a fine control of the superconducting quantum device parameters is required. In the last application, it is preferred an annealing by a laser, which allows to selectable heat the devices contained on the chip [19, 20].

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