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2008 Nanotechnology 19 315304

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Fabrication and characterization of high- T_c YBa₂Cu₃O_{7-x} nanoSQUIDs made by focused ion beam milling

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Received 26 March 2008, in final form 16 May 2008

Published 24 June 2008

Online at stacks.iop.org/Nano/19/315304

Abstract

We have fabricated high- T_c nanoscale superconducting quantum interference devices (nanoSQUIDs) with a hole size of 250 nm × 250 nm based on a 100 nm bridge at 77 K by focused ion beam milling and ion implantation. At 78 K, the curve of the voltage branch became roughly linear and agreed with the Josephson-like behavior. The sample exhibited strong flux flow behavior at temperatures under 76 K. The voltage flux characteristic curves, $V-I_{\text{mod}}$, of the nanoSQUID at different bias currents at 78 K were observed. Typically, critical currents of 15 μA and peak-to-peak values of the voltage flux transfer function of 3.7 μV were measured. The measured data strongly suggest that the weak link structure could be a superconducting metal with a critical temperature T'_c smaller than that (T_c) of other YBa₂Cu₃O_{7-x} (YBCO) films. This fabrication method of combining a nanobridge and ion implantation can improve the yield of nanojunctions and nanoSQUIDs.

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

1. Introduction

The superconducting quantum interference device (SQUID) is the most sensitive device for detecting weak magnetic flux change. SQUID magnetometers have unsurpassed energy sensitivity, and they have been employed in many applications [1–3]. Recent interest in the development of SQUIDs has been stimulated by the usefulness of these sensors to measure the magnetic nanoparticles and the magnetic flux induced by an atomic spin system [4–6]. In principle, nanoSQUIDs are good candidates. Up to now, several high- T_c Josephson junctions have been extensively investigated, including bi-crystal, bi-epitaxial, step-edge, and superconductor–normal–superconductor (SNS) type. High- T_c SQUID magnetometers are mostly fabricated on bicrystal, ramp edge and step edge substrates. However, it is not easy to

fabricate nanojunction and nanoscale SQUIDs (nanoSQUIDs) by these methods. Recently, an approach based on a nanoscale bridge (nanobridge) was developed to form a weak link as a Josephson junction. SQUIDs based on nanobridges, patterned with electron beam lithography, with focused ion beam (FIB) milling, and with carbon nanotubes as junctions, have been reported [6, 7]. The main advantages of the approach are that only a single high-quality superconducting layer is required and the junction locations are not restricted. It can avoid many of the complications encountered in other Josephson junction types.

An important figure of merit for DC SQUIDs is the flux power spectral density. The flux power spectral density $S_\Phi(f)$ is usually given by

$$S_\Phi(f) = \frac{S_V(f)}{\left(\frac{\partial V}{\partial \Phi}\right)^2}, \quad \text{with } \frac{\partial V}{\partial \Phi} \cong \pi \frac{\Delta V}{\Phi_0} \cong \frac{R}{2L} \quad (1)$$

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Figure 1. NanoSQUID fabrication with standard photolithography and FIB milling.

where $S_V(f)$ is the voltage noise power spectral density, $\partial V/\partial \Phi$ is the transfer function, R is the normal resistance of each weak link, and L is the loop inductance. We note that the geometrical inductance of a SQUID hole is conveniently estimated from

$$L_s = 1.25\mu_0 A_h^{1/2}, \quad (2)$$

where A_h is the area of the SQUID. The small A_h of nanoSQUID will lead to the large $\partial V/\partial \Phi$ and reduce the flux power spectral density according to equation (1). NanoSQUIDs are relatively insensitive to magnetic field because the effective area is very small. However, a nanoSQUID has a high sensitivity to a magnetic dipole located in the nanoSQUID hole. This makes Josephson junctions more attractive for a wide range of applications, such as in increasing the resolution of scanning SQUID microscopy (SSM), studying magnetic nanoparticles, and the measurement of the spin of a single atom. In this study, the characteristics of high- T_c nanoSQUIDs, fabricated by focused ion beam milling, are investigated; they include resistance versus temperature, voltage–current, magnetic modulation, amongst others.

2. Experimental details

Figure 1 presents the process of fabrication of nanoSQUIDs with standard photolithography and focused ion beam (FIB) milling. The devices were fabricated onto 5 mm × 5 mm SrTiO₃ (STO) substrates. The off-axis rf-magnetron sputtering technique was proposed to prepare smooth YBCO films with excellent electrical properties. The gold layer is evaporated by the DC magnetron sputtering technique. A 150 nm thick YBCO film was covered with a 300 nm Au layer on top of an STO substrate [8]. The Au layer was used as a fabrication mask, a protective layer on the YBCO thin film, and to increase the accuracy of milling. High- T_c YBCO microbridges with a

width of 3 μm were fabricated using the standard techniques of photolithography and Ar⁺ ion milling at 350 eV on a liquid-nitrogen-cooled rotating holder. The Au masking layer was deposited sufficiently thick to protect the YBCO thin film, but also sufficiently thin that the nanobridges, which define the junction barrier, could be made with sufficient accuracy by the FIB. The experiments were performed on FEI dual beam systems (FEI Nova 200). The system combines an FIB (Ga⁺ ions) and a scanning electron microscope, which makes it possible to create nanostructures faithfully and reproducibly using an FIB, many factors have to be taken into consideration, such as the profile of the ion beam, the influence of dwell time, the redeposition of the sputtered material, the yield change with the ion incidence angle, and the unintentional sputtering by beam tails [9]. Figures 1(a)–(d) present a schematic diagram of the fabrication procedures. A focused Ga⁺ beam (30 keV) with a beam current of 10 pA was first used to mill a hole in the middle of the microbridge with a width of 3 μm, and then YBCO nanobridges with a 100 nm width were kept and the rest was milled. Finally, the nanobridge was 150 nm thick, 100 nm wide, and 250 nm long. Different hole sizes from 250 nm × 250 nm to 1 μm² were fabricated. Furthermore, the properties of the YBCO thin film were damaged by ion beam irradiation. To prevent further deterioration of the junction properties, the electric properties of the SQUID were measured before the scanning electron microscopy (SEM) image was obtained.

3. Results and discussion

The critical temperature, T_c , of the microbridge was measured before and after processing by standard lithography, and was found to be unchanged at approximately 88 K. The critical current densities were higher than 2 MA cm^{−2} at 77 K.

Figure 2. NanoSQUID images taken by SEM. (a) The top-view image of a SQUID with 100 nm width and a hole size of 250 nm $\times$ 250 nm. (b) The tilted-view image is tilted at 52° to show the underlying YBCO SQUID. (c) Top-view image of a SQUID with 100 nm width and a hole size of 150 nm $\times$ 150 nm. (d) Tilted-view image of SQUID with $\sim$ 95 nm width and a hole size of 600 nm $\times$ 700 nm.

Figure 2(a) presents top-view SEM images of a nanoSQUID. YBCO nanoSQUIDs with different hole sizes and with nanobridges of 100 nm width were made accurately by FIB milling. Figures 2(b) and (c) show the tilted-view SEM image of a nanoSQUID. The Ga^+ ion beam milled nanoscale holes of size 250 nm $\times$ 250 nm and 300 nm $\times$ 300 nm in the middle of the 3 μm microbridges. However, it also removed the top surface of the Au layer on nanobridges. The Au layer on YBCO nanobridges became a hollow shape, as shown in figure 2(b). We know that the dwell time can affect the line broadening and sputtering depth in a material during the milling process. Long dwell time leads to a deep sputtering depth, which reduces the fluctuation of beam current by reducing the scanning numbers of pixels [9–11]. On lengthening the dwell time, the damaged condition was not obviously observed. Figure 2(d) presents the tilted-view SEM image of a nanoSQUID with a hole size of 616 nm $\times$ 700 nm. The Au layer was not seriously damaged by the Ga^+ ion beam used for FIB milling.

Figure 3 reveals the typical current–voltage characteristics (IVCs) of a nanoSQUID at different temperatures. At 78 K, the curve of the voltage branch became roughly linear. The IVC curve showed Josephson-junction-like properties. Based on the fitting by the resistively shunted junction (RSJ) model under thermal noise, the nanoSQUID has a normal resistance of 320 m Ω and a critical current of 15 μA , giving the characteristic voltage of $I_c R_n = 0.5$ mV at 78 K. The IVCs do not follow the RSJ model, and exhibit strong flux flow behavior at temperatures under 76 K.

Figure 3. Voltage–current curves at different temperatures.

The characteristics of this device were investigated further by elucidating the temperature dependence of the normal-state resistance and the temperature and the critical current density dependence of the characteristic voltage $I_c R_n$, plotted in figure 4. The normal resistance $R_n(T)$ decrease with the decrease in sample temperature is characteristic of a metallic barrier. The behavior is similar to the temperature dependence of the resistivity of YBCO above T_c . The inset shows the temperature versus the characteristic voltage $I_c R_n$

Figure 4. Dynamic resistance versus temperature. The inset plots the temperature dependence of the characteristic voltage $I_c R_n$.

of the nanoSQUID. The linear relationship with temperature near T_c strongly suggests that the weak link structure could be a superconducting metal (S–S'–S link) with a critical temperature T'_c smaller than that (T_c) of other YBCO films. Here S' stands for weakly damaged. There are reasons to believe that the junctions are of S–S'–S type, rather than a standard RSJ model [10–12]. Figure 5(a) plots the voltage flux characteristic curve, $V-I_{mod}$, of the nanoSQUID at different bias currents at 78 K. The peak-to-peak voltage swing (V_{pp}) of sample 1 with 100 nm width and a hole size of 250 nm × 250 nm is around 2.5 μ V for a single microSQUID. Figure 5(b) shows that the V_{pp} of sample 2 with 300 nm × 300 nm with 95 nm width is approximately 3.7 μ V for a single nanoSQUID. The effective areas of the nanoSQUIDs are about 0.056 and 0.045 μ m².

The nano-Josephson junction is typically fabricated using a nanobridge with a width and length smaller than the effective penetration depth. The nanobridges have a notch shape which minimizes the length of the junction. The small dimension of the nanobridge behaves as a weak link, that is, the bridges are expected to show superconductor–insulator–superconductor (SIS) or superconductor–normal–superconductor (SNS) junction behavior as a result of coherent vortex motion or quantum phase slip [13]. However, the nanobridges herein are long and wide. Our data reveal S–S'–S behavior, in which the S' component was formed in the thinnest part of the Au by Ga⁺ ions from FIB [11]. The junction formation process was similar to the formation of a weak link Josephson junction by focused electron (ion) beam irradiation of a high- T_c microbridge. The irradiation damage reduces J_c and increases R_n . These effects are presumably caused by the disordering of oxygen atoms by electron (ion) irradiation. Hence, the structure becomes a weak link (S–N–S, S–I–S), which serves as a Josephson junction if the damaged region is sufficiently narrow [14–16]. Based on the experiment, we infer that FIB milling may cause Ga ion implantation of the nanobridges of YBCO thin film, further degrading the superconducting properties. The damaged area formed weak links. However, the structure of the thin film exhibited only partial damage. The data do not reveal the standard RSJ

Figure 5. (a) The voltage flux characteristic curve, $V-I_{mod}$, of the nanoSQUID at different bias currents I_b at 78 K. (b) shows that the V_{pp} of sample 2 is approximately 3.7 μ V.

behavior. In figure 2(d), we see that the Au layer was not damaged by FIB milling. The characteristics of a SQUID were not observed. The reason is that no weak links were formed in the device's nanobridges.

4. Conclusion

We have successfully fabricated a high- T_c nanoSQUID with a hole size of 250 nm × 250 nm based on a nanobridge at 77 K by focused ion beam milling. The sample exhibited strong flux flow behavior at temperatures under 76 K. The characteristics of the nanoSQUID revealed S–S'–S behavior. Peak-to-peak values of voltage flux transfer function of 3.7 μ V were measured. The effective area of the nanoSQUID is about 0.045 μ m². This fabrication method of combining a nanobridge and ion implantation can improve the yield of nanojunctions and nanoSQUIDs. This technique might provide a way to detect a small number of magnetic molecules on the sensitive region of a nanodevice.

Acknowledgment

The authors would like to thank the National Science Council of the Republic of China, Taiwan, for financially supporting

this research under Contract No. NSC95-2112-M005-016-MY3.

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