High T_c bi-epitaxial junctions and dc SQUIDs structured by focused ion beam etching

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Abstract—Focused ion beam etching has been used to pattern dc SQUIDs into previously characterised template bi-epitaxial grain boundary junctions. Using this technique the screening parameter β_L can be optimised for a chosen temperature (in our case 30 K). Electrical characteristics, including noise measurements, are presented. A minimal white noise level of $22 \mu\phi_c \cdot \text{Hz}^{-1/2}$ ($1.8 \cdot 10^{-29} \text{ J} \cdot \text{Hz}^{-1}$) has been obtained at 20 K. Using bias current modulation the $1/f$ noise could almost completely be suppressed down to 1 Hz in the entire temperature range (10-65 K).

I. INTRODUCTION

Junctions based on grain boundaries show a large spread in the critical current density J_c . If induced by a template layer, typically a spread in J_c of a factor 10 is observed. On the other hand, $I_c R_n$ products are fairly reproducible and are about 0.5 mV at 4.2 K. Because of the spread in J_c , dc SQUIDs based on this kind of grain boundary may not show a good match of the critical current I_0 of the separate junctions and the SQUID inductance L_{SQ} . Lowest expected white noise levels, thus optimal energy sensitivity, of the dc SQUID are expected for a value of the screening parameter $\beta_L = 2I_0 \cdot L_{SQ} / \phi_0$ equal to one according to Tesche and Clarke [1]. A second problem for these SQUIDs concerning voltage modulation and noise levels might be the Thermally Activated Phase Slippage (TAPS) across the grain boundary, which is frequently observed for grain boundary junctions. For currents near zero value, this phase slippage causes a voltage drop across the junction for temperatures between T^* (minimum temperature at which this voltage drop is observed) and T_c (the critical temperature of the electrodes).

II. EXPERIMENTAL

Template grain boundary junctions were prepared using (001) oriented MgO substrates, a 10 nm thick CeO_2 template layer and an $\text{YBa}_2\text{Cu}_3\text{O}_{7-d}$ toplayer (100 nm). The CeO_2 layer was structured using CaO lift-off. The grain boundary was aligned along the [100] direction of the substrate. Structuring

Manuscript received October 16, 1994.

This work has been supported by the "stichting voor Fundamenteel Onderzoek der Materie" (FOM), the "Nederlandse organisatie voor Wetenschappelijk Onderzoek" (NWO) and the "Nationaal Onderzoeksprogramma voor Hoge T_c supergeleiding" (NOP-hoge T_c).

Fig. 1. SEM picture of the dc SQUID described in the paper. Patterning of the SQUID into the template grain boundary junction was done using a focused ion beam. The bars equal 11 μm . The grain boundary is marked (a).

of the junctions was done using Ar ion beam etching. A detailed description of the fabrication process and electrical characterisation of these 5-20 μm wide junctions structured across the grain boundary is given elsewhere [2]. Dc SQUIDs were patterned in these characterised junctions using a 25 kV Focused Ion Beam (FIB) equipped with a Ga liquid metal ion source. SQUID hole and slit dimensions (determining the main part of the SQUID inductance [3-5]) were adjusted to obtain $\beta_L = 1$ at 30 K. Figure 1 shows a SEM picture of the resulting SQUID presented in this paper. The bars equal 11 μm . The SQUID hole and slit plus the lines defining the washer are etched away using the FIB. The top of the picture shows $\text{YBa}_2\text{Cu}_3\text{O}_{7-d}$ grown on the CeO_2 template layer, the bottom shows $\text{YBa}_2\text{Cu}_3\text{O}_{7-d}$ grown directly on the MgO substrate. The grain boundary is marked (a). Alignment problems of the ion beam caused a small asymmetry of the junctions. Etching depths are 200 nm. The slit is structured using minimal ion beam diameter (50 nm), resulting in a line width of 200 nm. The lines defining the washer and the SQUID hole are etched using an ion beam spot size of 130 nm. Dimensions of the SQUID hole are $34 \times 36 \mu\text{m}^2$. The resulting calculated SQUID inductance is 70 pH, consisting of a slit inductance of 15 pH and a hole inductance of 55 pH.

Electrical characterisation including noise measurements were performed in a variable temperature He gas flow

Fig. 2. HREM picture of an $\text{YBa}_2\text{Cu}_3\text{O}_{7-d}/\text{CeO}_2$ bilayer grown over a step (step height 18 nm) in the MgO surface. The step in the MgO surface does not seem to influence the orientation of the $\text{YBa}_2\text{Cu}_3\text{O}_{7-d}$ grown on top of this step.

cryostat. From the inside outwards, the sample was shielded from external noise sources by a BiSrCaCuO, a mu-metal and an Al can. Magnetics fields perpendicular to the substrate surface were applied using a coil mounted around the sample inside the BiSrCaCuO can.

III. RESULTS

High Resolution Electron Microscopy (HREM) was used to get more structural information on nanometer scale about the different layers and interfaces on the MgO substrate. The CaO lift-off technique was used to structure 15 nm thick CeO_2 layers into 20 μm wide striplines. This structure was covered by a 120 nm thick $\text{YBa}_2\text{Cu}_3\text{O}_{7-d}$ layer. TEM cross section preparation was described elsewhere in detail by Træholt et al. [6]. $\text{YBa}_2\text{Cu}_3\text{O}_{7-d}$ grown directly on the MgO is highly crystalline and c-axis oriented. Steps in the MgO surface of about 1 nm do not seem to induce grain boundaries in the $\text{YBa}_2\text{Cu}_3\text{O}_{7-d}$. No interface layer, caused by a reaction between CaO or water and the MgO, is observed.

Figure 2 shows an $\text{YBa}_2\text{Cu}_3\text{O}_{7-d}/\text{CeO}_2$ bilayer on a stepped MgO surface (step height about 18 nm). From the top to the bottom of the picture the $\text{YBa}_2\text{Cu}_3\text{O}_{7-d}$, the CeO_2 and the MgO are visible viewed along the [100] MgO axis. In the

bottom middle part of the picture, the step in the MgO surface is visible. Although the structure of the CeO_2 is not clearly visible, it is obvious that the step does not influence the orientation of the $\text{YBa}_2\text{Cu}_3\text{O}_{7-d}$ perpendicular to the substrate surface. The $\text{YBa}_2\text{Cu}_3\text{O}_{7-d}$ layer is growing nicely without noticeable defects over the entire layer up to the $\text{YBa}_2\text{Cu}_3\text{O}_{7-d}$ surface.

Junctions structured across these template grain boundaries show a large spread, typically a factor 10, in J_c and $R_n A$ values between different substrates. J_c and $R_n A$ values for 16 RSJ-like junctions on one substrate are given in figure 3 as a function of the position along the grain boundary (denoted by x). The critical current varies between 3×10^3 and 5×10^4 A/cm², although the variation of J_c over distances of 1 mm is much smaller and typically a factor 1.5. $R_n A$ products vary between 1×10^{-8} and 8×10^{-8} Ωcm^2 . Again a much smaller variation is observed for junctions closely together. In general junctions with high critical currents have low $R_n A$ products. The resulting $I_c R_n$ products vary between 0.1 and 0.9 mV. No differences in the average value and spread in J_c for junctions with different widths is observed. Probably the spread in J_c and $R_n A$ values is caused by a variation of the grain boundary thickness over the substrate.

The effect of the patterning process by the FIB on the

Fig. 3. The critical current density and $R_n A$ product of 16 RSJ-like grain boundary junctions on one substrate as a function of the position along the grain boundary measured at 20 K.

critical current of the junction is shown in figure 4. Shown are the critical current of the original junction before patterning and the total critical current of the dc SQUID after patterning. A reduction in T^* of about 10 K is observed. No change in the critical temperature of the $\text{YBa}_2\text{Cu}_3\text{O}_{7-d}$ electrodes is observed. The normal resistance $R_{n, \text{sq}}$ of the dc SQUID increased to 4.2Ω as compared to $R_{n0} = 3.3 \Omega$ of the original junction (both determined at 20 K). Because of the considerable decrease of T^* and the increase of R_n of 27%, as compared to the decrease of the total junction width of about 2%, it is concluded that the grain boundary is damaged by the Ga ions during aligning and structuring of the sample.

Applying a small varying magnetic field perpendicular to the grain boundary, IV curves and voltage modulation of the dc SQUID were measured as a function of temperature. At 10 K a maximum voltage modulation of $72 \mu\text{V}$ and a flux to voltage transfer $dV/d\phi$ of $230 \mu\text{V}/\phi_0$ were obtained. Up to 72 K, about 15 K above T^* , clear sinusoidal voltage modulation is observed. The maximum measured voltage

Fig. 4. The critical current of the original junction (before patterning) and of the dc SQUID (after patterning) as a function of the temperature.

Fig. 5. The measured (downtriangles) and the calculated (boxes) maximum voltage modulation after Enpuku et al. [7] as a function of the temperature.

modulation was compared to the expected voltage modulation for a dc SQUID consisting of two junctions with equal critical current densities I_1 and I_2 respectively, as described by Enpuku et al. [7]. The results are given in figure 5. At temperatures of 25 K and higher the calculated and measured voltage show reasonable agreement. At temperatures below 25 K the measured voltage modulation exceeds the calculated one up to 40 % at 10 K. The agreement between measured and calculated voltage modulation indicates that although a large spread in critical current densities between different substrates and on one substrate is observed, the variation of the critical current density on a scale of tens of micrometers is much smaller. As discussed by Tesche and Clarke [1], the expected voltage modulation for a dc SQUID consisting of two junctions with critical current densities $I_1/I_2=9$, is about 50 % of the voltage modulation for a dc SQUID with equal critical current densities.

$\beta_{L, \text{meas}}$ as determined from the IV curves using $I_{\text{mod}}/I_c = \beta_{L, \text{meas}} / (1 + \beta_{L, \text{meas}})$ was compared with $\beta_{L, \text{calc}}$ as

Fig. 6. Measured and expected screening parameter $\beta_{L, \text{meas}}$ and $\beta_{L, \text{calc}}$ as a function of the temperature.

Fig.7. Flux noise as a function of temperature at 1 Hz (1/f noise) and the white noise level both for dc and ac current biasing.

determined from the critical current and the calculated SQUID inductance. Here I_{mod} is the minimal current at which voltage modulation is observed, and I_c the total critical current of the SQUID. The results are given in figure 6. At temperatures above 50 K, no reliable value of $\beta_{L,\text{meas}}$ can be obtained, because of the low values of I_{mod} and rounding of the IV curves near I_c . For temperatures below 40 K $\beta_{L,\text{meas}}$ is larger than expected from the critical current and the calculated SQUID inductance. This difference can not be explained by a correction for the excess current present in this junction (maximum value 6 μA at 35 K and below) or the kinetic plus magnetic inductance of the junction striplines and slit which is estimated to be about 10% of the slit inductance [8].

Temperature dependent noise measurements (1 Hz - 1 kHz) have been obtained in a flux locked loop, both for dc and ac current bias at maximum voltage modulation. For dc current bias the noise powerspectrum in general consists of a 1/f part for frequencies up to $\nu_{1/f}$ and a white noise level at higher frequencies. For increasing temperature, $\nu_{1/f}$ decreases. By applying ac current biasing the 1/f noise could be suppressed considerably over the entire temperature range. Figure 7 shows the fluxnoise as a function of temperature at 1 Hz and the white noise level, both for dc and ac current biasing. For temperatures of 40 K and higher the 1/f noise could be totally suppressed indicating that asymmetric critical current fluctuations are the main reason for the 1/f noise at these temperatures. A minimum value of $40 \mu\phi_0 \cdot \text{Hz}^{-1/2}$ at 1 Hz and 40 K is obtained. The white noise level is slightly increased by ac current biasing, and shows a broad minimum around 20 K for dc biasing. A minimum white noise level of $22 \mu\phi_0 \cdot \text{Hz}^{-1/2}$ ($1.8 \cdot 10^{-29} \text{ J} \cdot \text{Hz}^{-1}$) at 20 K is obtained. This value is about a factor 10^2 higher than the theoretical white noise limit for this dc SQUID [9], but comparable to values reported in literature [10].

IV. CONCLUSIONS

Structuring the CeO_2 layer with the CaO lift-off technique does not result in an observable structural degradation of the $\text{YBa}_2\text{Cu}_3\text{O}_{7-d}$ layer grown on top of this structures. Large steps in the MgO surface covered by CeO_2 do not influence the orientation of the $\text{YBa}_2\text{Cu}_3\text{O}_{7-d}$ layer grown on top of this steps. Patterning a dc SQUID using focused ion beam etching into a previously characterised template grain boundary junction and thereby matching the SQUID inductance to the critical current is possible. The junctions show a decreased value of T^* and I_c and an increased R_n value, probably caused by a change of the grain boundary by the 25 keV Ga ions. Voltage modulation up to 72 K, about 15 K above T^* of the dc SQUID, was observed. A minimal white noise level of $22 \mu\phi_0 \cdot \text{Hz}^{-1/2}$ at 20 K was obtained. Using ac bias current modulation, the 1/f noise could be almost completely suppressed down to 1 Hz over the entire temperature range, indicating that most 1/f noise is caused by asymmetrical critical current fluctuations. Using ac current biasing, a minimal flux noise level of $44 \mu\phi_0 \cdot \text{Hz}^{-1/2}$ at 1 Hz and 40 K was obtained.

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