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Fast technology for fabrication of thick single $Bi_2Sr_2CaCu_2O_{8+x}$ mesas on a Cu substrate

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Abstract

We continued the elaboration of the wet etching method for the fabrication of standalone $Bi_2Sr_2CaCu_2O_{8+x}$ (BSCCO) mesastructures, allowing us to get mesas with large thicknesses of the order of 1–15 μm. Here we performed the next step: fabricating such mesas on a thick copper substrate using the electroplating technique. This allowed us to get rigid structures that resist mechanical damage and also have a reliable heat sink, thus reducing the overheating of samples at large bias currents. Samples with a 30 μm thick copper layer were fabricated. Current–voltage curves of superconducting samples at various cryogenic temperatures were measured and studied, and a significant dependence on the thermal interface was observed. For one mesa, radiation of the order 0.1 μW was detected outside the cryostat by a Golay cell.

Keywords: intrinsic Josephson junction, BSCCO mesa, wet etching

(Some figures may appear in colour only in the online journal)

Introduction

Since the first observation [1] of THz generation in $Bi_2Sr_2CaCu_2O_{8+x}$ (BSCCO) mesas, significant progress in generation power from such structures has been achieved [2–6], with powers approaching the mW range [5]. BSCCO is a layered high-temperature superconductor with an internal Josephson effect [7], where a series chain of Josephson junctions is formed on an atomic scale. Artificially fabricated stacks made of high-T_c materials are not yet available, but the recently elaborated preliminary mask technology [8] can be used. The hybrid junctions between low-T_c and high-T_c superconductors demonstrate a small critical current density and low I_cR_n product [9], which is why naturally grown BSCCO monocrystals is the most efficient way to increase radiation power and synchronize outcome from neighboring layers. It is understood that in normal conditions a triangular array of Josephson vortices is formed in BSCCO layers, which can suppress radiation from neighboring layers; moreover, special conditions are required to synchronize all layers to oscillate in phase to get a noticeable power. Currently, one of the most efficient synchronization channels in BSCCO is supposed to be a hot spot [10, 11], where a part of the mesa is in the resistive state while another is superconductive. This appears for rather thick structures, and to get coherent generation one needs at least 100–200 layers [12]. The already achieved characteristics of BSCCO oscillators allow for the fabrication of practical THz receivers and spectrometers [13–19], with the possibility to tune the oscillation frequency by a laser beam [20] in addition to tuning it by current. However, the greatest shortcoming of the current technology is that mesas are usually fabricated by ion milling on top of larger BSCCO fragments [1, 21, 22]. This, on one hand, allows us to make just a few rather thin mesas, usually not thicker that 1 μm due to the slowness of the technological process; on the other hand, superconducting material between the mesa and the sample holder with poor thermal conductance leads to overheating of the mesa at large bias currents, thus narrowing the temperature range of effective THz radiation.

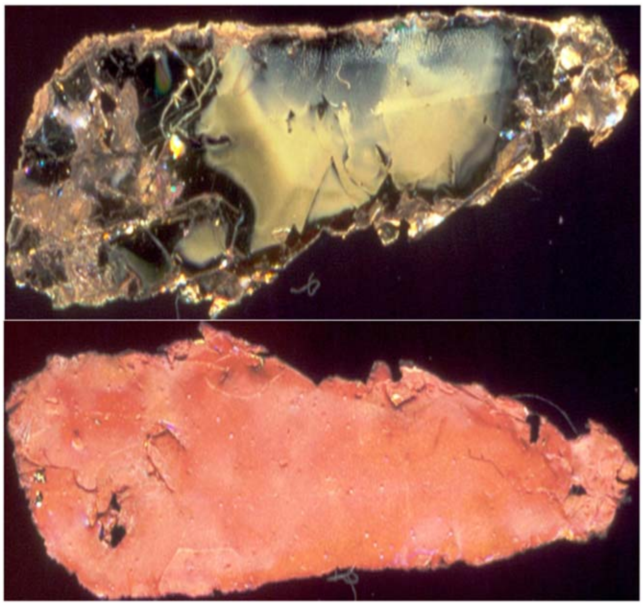

Figure 1. The BSCCO sample with a deposited 50 nm gold layer (top) and 30 μm copper layer (bottom).

In the present paper we continue the elaboration of wet etching technology [23], which is fast and allows us to fabricate standalone BSCCO mesas with a large thickness of the order of 1–15 μm. Currently, by the applied electroplating technique, several tenths of mesas on a thick 30 μm copper substrate are fabricated, thus leading to rigid structures that resist mechanical damage and also have a reliable heat sink. By varying the means of attaching the mesas to a sample holder, we study the effect of overheating on current–voltage characteristics (IVCs) with various shapes of negative differential resistance branches with maximal voltage from 2–7 V depending on the sample. While the samples are not inserted into any antenna or waveguide system, a little power of the order 0.1 μW is detected by the room temperature detector outside the cryostat.

Fabrication

Initially, a piece of BSCCO single crystal, grown from melting in a gold crucible [24–26], was repeatedly flaked by thin layers until a flat surface was visible. After that, a fragment with a thickness of the order of tenths of microns and lateral dimensions of the order of a few millimeters was chosen. The fragment was glued to the holder and cleaved. Just after that, a 50 nm thick gold layer was deposited on the cleaved surface of the sample using the thermal evaporation technique in vacuum (see figure 1, top). Next, a 2 μm thick copper layer was deposited *in situ* on the top of a gold layer using the thermal evaporation technique in vacuum. The growth of the contact substrate to the BSCCO structure was carried out by electroplating of the copper layer with a thickness of 28 μm above the 2 μm copper layer (see figure 1, bottom). For the electroplating of copper, a special electrolyte that would not destroy the BSCCO structure (with pH > 2) was chosen. Precipitation was carried out at temperature $T = 20\,^{\circ}$C, for $t = 6$ h, and at a low current density ($\sim$0.07 mA mm^{-2}), which ensured high uniformity in thickness, absence of defects, mechanical strength, and chemical purity of the precipitated copper. It also did not affect the properties of the BSCCO structure. Furthermore, the second plane of the BSCCO was cleaved, and a layer of 50 nm thick gold was deposited on the cleaved surface of the sample using the thermal evaporation technique in vacuum. The forming of contact pads of gold with a diameter of 400 μm was carried out by means of contact photolithography followed by liquid dynamic etching of a gold layer on a BSCCO single crystal using a photoresist mask in a special etchant based on free iodine that did not interact with the BSCCO structure. Subsequently, liquid etching of the BSCCO structure was carried out in hydrochloric acid (1:8 water solution) over roughly 30 min, followed by removal of the photoresist [23]. As a result of the used technology, several tenths of single mesas were fabricated (figure 2), thus leading to rigid structures that resist mechanical damage and also have a reliable heat sink due to the deposited copper substrate.

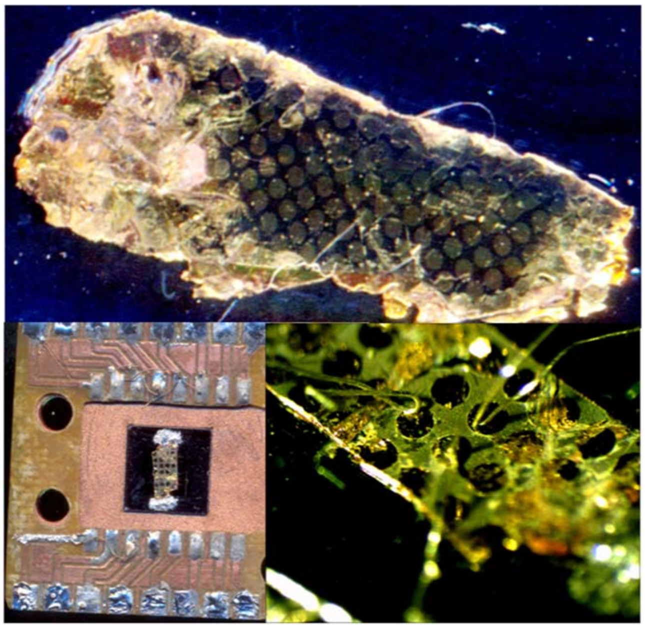

Figure 2. Circular single BSCCO mesas after wet etching, the fragment on a Si substrate attached to a copper holder by rubber glue, and mesas connected by wires.

In figure 2 (bottom) the mesas with contact wires are demonstrated. While the bottom electrode of the structure is a copper plate, and it is easy to make electrical contact with it, the size of the top electrode of each mesa is rather small and it is difficult to make proper contact using, for example, Ag conducting glue. We chose to use an additional Si substrate with gold contact pads and to attach a copper plate with mesas to it by indium. After that, using split electrode bonding, thin Ag wires were attached to contact pads at the Si substrate, and elastic Ag wire was connected via clamping to the top electrodes of mesas (see bottom right photo in figure 2). The main problem of such wiring was that the contact was rather unstable from cooling due to thermal drifts and vibrations of the dry cryostat plate; therefore, in the future it is necessary to find a better solution for wiring. Nevertheless, in each cooling

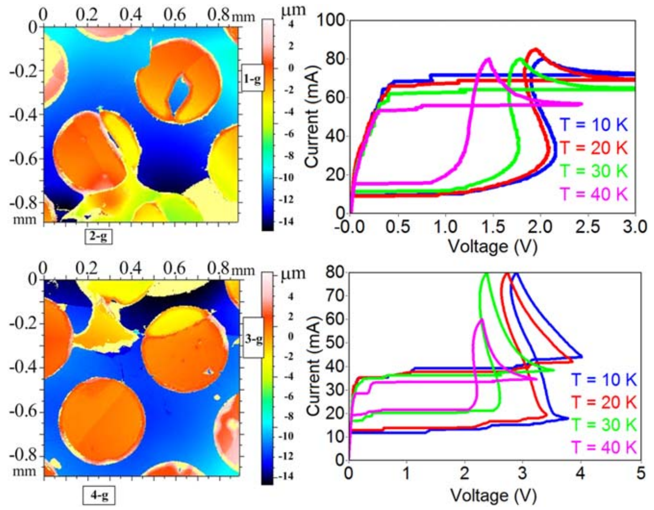

Figure 3. Talysurf profiles of several BSCCO mesas with thicknesses from 9.4 to 12 μm (left), and the IVCs of mesas 2-g and 3-g at various temperatures.

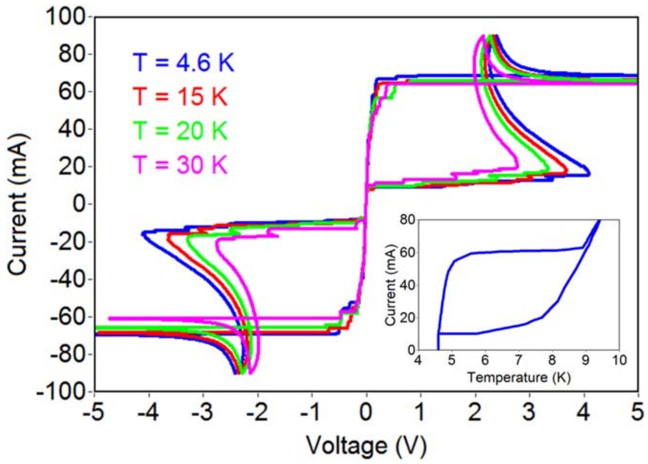

Figure 4. The IVCs of the 4-i BSCCO mesa (Si substrate attached with glue) at various temperatures of the cryostat plate. Inset: temperature at the copper sample holder below mesa during IVC measurement.

cycle at least one of the three to four connected structures was connected properly, and we got suitable IVCs from all six measured mesas, which were chosen by visual control of their uniformity. The Si substrate was attached to the copper sample holder by two different means. Initially, we used a rubber glue, but later it was substituted by indium, which radically changed the thermal contact and thus affected the IVCs. This is shown below.

Measurements

All mesas were measured with a Talysurf 2000 white light interferometer, and numbered. Two examples of mesa profiles with thicknesses from 9.4 to 12 μm and their IVCs at various temperatures of the cryostat plate are shown in figure 3. Since significant heating of the sample holder can be observed at large currents, all dependences are marked for the initial temperature of the sample holder for zero current. It should be noted that all working mesas demonstrated IVCs with significant hysteresis and a negative differential resistance branch, which is explained by the appearance of a hot spot in the mesa [10, 11, 27]. The statistics of the obtained structures were collected (see table 1), and the characteristics of the samples were compared with the analogs described in a literature. The critical current density of the obtained samples $J_c = 30–70$ A cm^{-2} is close to the literature data [4, 6, 17, 28–32] $J_c = 80–250$ A cm^{-2}. The gap voltage in terms of one junction layer (we calculated the approximate number of junctions, assuming a one-layer height of 1.5 nm) $V_g = 0.26–0.4$ mV differs from the values given in the literature ($V_g = 1–1.4$ mV). The reasons for these differences are clarified below.

In figure 4 the IVCs of mesa 4-i are shown together with the temperature at the sample holder (in the inset). One can see that at a low base temperature of 4.6 K the temperature at the ohmic line reaches 9 K, which should not be a surprise taking into account the large dc power. The results presented in figures 3 and 4 correspond to the case where the Si substrate was attached to a copper holder by a rubber glue (figure 5(a)).

We changed the mounting of the substrate to the holder and used indium instead of glue, as shown in the schematic in figure 5(b). Following this, the IVCs are changed significantly (see figure 6). First, the gap voltage increased by almost a factor of two compared to previous measurements (figure 4). This is due to a decrease in sample overheating during the jump to the resistive state (1–2 K instead of 4–5 K). Thus, in the absence of additional overheating, the gap voltage per junction $V_g = 0.6–0.7$ mV is close to the literature data. It is obvious that the main difference from other papers is due to the record thickness of the structures; moreover, the maximum voltage observed in our experiments is 7 V, while according to the published data it does not usually exceed 1 V. The increase in the thickness of the structures with effective synchronization in different layers of the mesa should increase the power generated from such structures and also improve the matching of the structure with the external electrodynamic environment.

By comparing figures 4 and 6 one can see a less-pronounced negative differential resistance branch. Additionally, with the increase in temperature, the decrease in the negative differential resistance branch is less-pronounced than in the case of attaching by glue. This signals that for a more efficient thermal interface, the hot spot, efficiently synchronizing radiation inside the layers, may persist until higher temperatures. IVCs for two types of Si substrate attachment are compared in figure 7 for the 3-j BSCCO mesa.

While the obtained samples were not intended for efficient power radiation since no antennas and lens were supposed, we decided to check for possible microwave emission coming outside the cryostat using a Golay cell. The calculated diagram of the mesa radiation has a maxima at 45° from the

Table 1. Statistics of measured structures, attached by glue, and their parameters.

Mesa number	Thickness (μm)	Number of junctions	I_c (mA)	J_c (A cm^{-2})	Gap voltage (V)	Gap voltage per junction (mV)
2-g	12	8400	70	76	1.9	0.26
3-g	12	8400	40	43	3.2	0.38
4-i	13	9100	70	76	2.5	0.27
3-i	13	9100	35	38	3.2	0.35
3-j	15	10500	40	43	2.8	0.26
1-k	12	8400	40	43	3.4	0.4

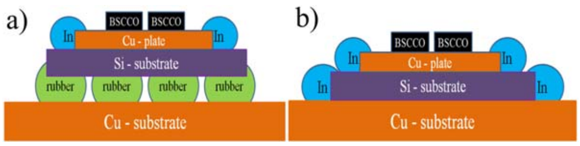

Figure 5. Schematic view of the Si substrate, attached to the copper holder by (a) rubber glue and (b) indium.

plane, and therefore only a small amount of radiation can be detected outside the cryostat window with the holder, parallel to the cryostat plate. Nevertheless, several attempts to detect THz emission from the 3-j mesa was successful, and a power of the order 0.05–0.08 μW was detected. The largest power was observed during a slow sweep of bias current with the total sweep time for the IVC branch around one hour. In figure 8 the voltage at the backward IVC branch versus bias current is presented for two different scales. Radiation was observed at the plate temperature of 18 K when the Si substrate was glued to the copper sample holder. The first curve is shown in the full scale from 0 to 3.6 V, while the second curve is shown from 3.31 to 3.57 V to enhance the IVC steps. The emitted power versus bias current is shown in red with the scale at the right axis, with the maximal peak up to 0.18 μW. Meanwhile, the voltage curve looks smooth at lower values, and no steps can be observed; at larger values some steps can be distinguished, which have a certain correlation with the shape of the power versus the current curve (the step locations are marked by dashed lines). Radiation was observed when the substrate was attached to a copper holder by a rubber glue. Conversely, when the substrate was attached by indium, no radiation was observed, presumably due to a too-low holder temperature at a better heat sink or not enough overheating due to a too-efficient heat sink. Currently, further measurements at higher temperatures and with a modified set up are in progress.

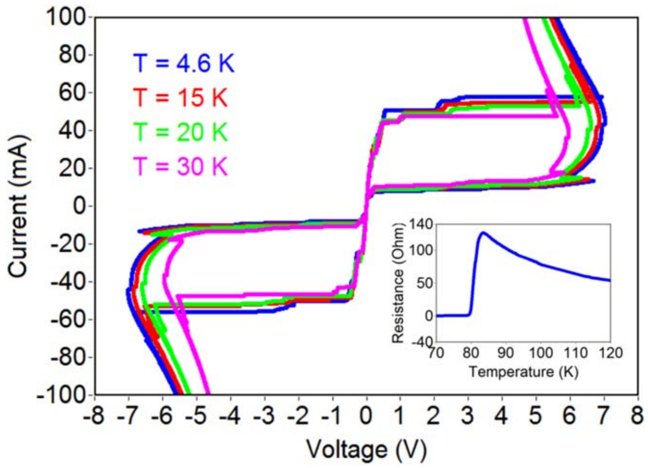

Figure 6. IVCs of the 4-i BSCCO mesa (Si substrate attached with indium) at various temperatures of the cryostat plate. One can see a less-pronounced negative differential resistance branch, but with almost twice the maximal voltage increase in comparison with the results shown in figure 4. Inset: critical temperature of the measured mesa.

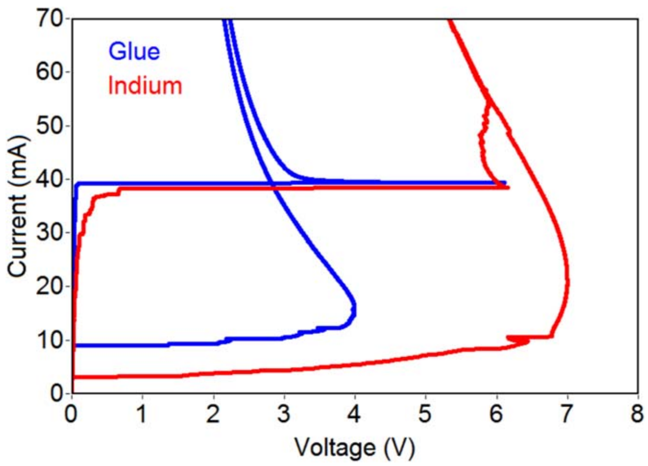

Figure 7. IVCs of the 3-j BSCCO mesa for two Si substrate attachments, leading to good and bad heat sinks ($T = 5$ K).

Conclusions

In conclusion, we report further progress of the wet etching method for the fabrication of standalone BSCCO mesastructures, allowing us to get mesas with a large thickness of the order of 1–15 μm. The mesas are fabricated on a thick copper substrate using the electroplating technique, which allows us to get rigid structures that resist mechanical damage and also have a reliable heat sink, thus reducing the overheating of samples at large bias currents. Current–voltage curves of BSCCO mesas at various plate temperatures are measured, and significant dependence on the thermal interface is observed. It is possible to detect power (of the order of 0.1 μW) outside the cryostat by a room temperature detector.

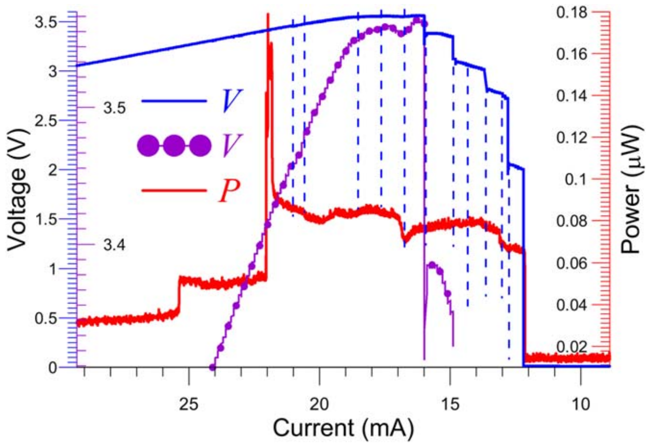

Figure 8. The voltage of IVC as a function of bias current at the 3-j BSCCO mesa (in two different scales (left axis): from 0 to 3.6, and 3.31 to 3.57 to enlarge the steps) and generated power, received by the room temperature detector outside the cryostat (right axis).

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