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Improved step edges on LaAlO$_3$ substrates by using amorphous carbon etch masks


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We report a technique for the fabrication of sharp and straight step edges on LaAlO$_3$ (LAO) substrates by ion milling. An electron beam lithography defined amorphous carbon film was used as an etch mask. It had very low ion milling rate and was easily prepared and removed. Atomic force microscopy was used to determine the step profile. YBa$_2$Cu$_3$O$_y$ step edge junctions fabricated at the LAO steps show promising results. An $I_R$ product of 1 mV was obtained at 30 K. A Fraunhoyer-like magnetic field dependence of $I_c$ was obtained up to $\pm 2\Phi_0$. One weak link or possibly identical weak links in series for these step edge junctions were observed from the current-voltage ($I-V$) curves as well as from the magnetic field dependence of the $I-V$ curves.

Progress has been made in the fabrication of high-$T_c$ Josephson junctions during the past few years. Step edge junctions$^1$ have received revived interest due to the possibility of large integration, low $1/f$ noise$^2$ in SQUIDs and high sensitivity in magnetometer applications.$^3$ Sharp and straight step edges on the substrates are crucial for the junction quality and reproducibility. Preparation of step edges on currently used substrates for YBa$_2$Cu$_3$O$_y$ (YBCO) thin films is non-trivial. As no selective dry etching process is currently available for these substrates, one has to use ion milling to create the sharp steps. Unfortunately, all presently used substrates have very low ion milling rates, which makes it difficult to use a photoresist mask (that has a several times higher etching rate). A metal mask and a small amount of oxygen in the process gas are usually required for making sharp steps,$^4$ but it may introduce residual metal mask debris on the substrate.$^5$ Recently, Sun et al.$^6$ used a diamond-like carbon film as an ion milling mask. It has a very low ion milling rate and can be easily removed. A disadvantage is that a special technique has to be used to deposit diamond-like carbon films.

In this letter, we report on the use of an amorphous carbon (a-C) film as the ion milling mask to make step edges on LaAlO$_3$ (LAO) substrates. The a-C film is chosen because it has a very low ion milling rate — less than half of that of LAO, and it can be very easily prepared and removed. The a-C mask was patterned by using electron-beam lithographed lift-off stencils. Electron beam lithography allows to avoid a waviness of the edge line.

The a-C films were made by electron gun evaporation in a vacuum system with a base pressure of $4\times 10^{-7}$ mbar. During evaporation, the substrate holder was water cooled. X-ray diffraction studies show that our carbon film is amorphous. The dark, shiny carbon film has a very smooth surface [Fig. 1(a)], and is very tough in an ultrasonic acetone bath that allows us to use a lift-off process to define an 80-nm-thick a-C film pattern on the LAO substrates. A lift-off mask consisting of copolymer (poly[methyl methacrylate/methacrylic acid]) and PMMA (poly[methyl methacrylate]) double layer resist was created by a direct electron beam exposure and subsequent development.

Figure 1(a) shows an atomic force microscope (AFM) image of the a-C film on a LAO substrate after the lift-off process. Sharp silicon AFM tips (ultralevers), with a force constant of 0.03 or 0.06 N/m, were used in a Park Scientific Instruments AFM (Universal System). The tips have a half open angle of $10^\circ$. The typical radius of curvature of the tip is 10 nm. The force applied in all images was about 5 nN and the scan frequency was 0.5 Hz.

A normal incident ion beam with a voltage of 500 V and a current density of 0.67 mA/cm$^2$ was used to transfer the pattern from the a-C film to the substrate for ion milling. During the ion milling, the Ar pressure was kept at 1.5$\times 10^{-4}$ mbar and the water cooled substrate holder was rotated. The milling rate was about 4–5 nm/min for the a-C film and 12–13 nm/min for the LAO substrate. After ion milling, the a-C mask was removed by an oxygen reactive ion etching (RIE) process. Figure 1(b) is the AFM image of the step edge on LAO. The image shows a clean, sharp, and straight step edge. We also show the AFM image in Fig. 1(c) of another step edge prepared on an MgO substrate by using an aluminum mask defined by photolithography. In this process, the photoresist mask seems to erode severely, resulting in a wavy and jagged step. A cross-sectional analysis of the step edge in Fig. 1(b) gives a uniform edge slope of about $70^\circ$ and a step height of 180 nm. However in Fig. 1(c) the edge slope is nonuniform and not very sharp, ranging between $40^\circ$ and $50^\circ$.

Epitaxial, $c$-axis oriented, 150-nm-thick YBCO films were deposited on top of the LAO substrates (with steps) by pulsed laser deposition. An oxygen pressure of 0.8 mbar and a substrate temperature of 780 °C were used during deposition. For good ohmic contact, the YBCO film was coated in situ with a 20-nm-thick gold layer. The YBCO film was patterned by using a photoresist mask and Ar ion milling. Two control strips, 4 $\mu$m wide, were patterned to determine the film quality on the milled and unmilled parts of the substrate. Two stripes, 4 and 8 $\mu$m wide, crossed the step. In addition, two 4-$\mu$m-wide strips in parallel formed a $50\times 5 \mu$m$^2$ large dc SQUID at the step edge. After patterning of the YBCO, a layer of gold, 250 nm thick, was evaporated through a metal mask to form the contact pads. A short ion milling was then

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used to remove the \textit{in situ} gold layer on top of the YBCO film. Figure 1(d) shows the AFM image of the 4-\(\mu\)m-wide strip of YBCO crossing the step edge.

The current-voltage (I-V) characteristic of each strip was measured using a four-probe method. A criterion of 1 \(\mu\)V was used for the determination of the critical current (\(I_c\)). The normal resistance (\(R_n\)) was determined at sufficiently high bias where the \(dV/dI\) value was a constant. The \(T_c\)'s of the films were around 90 K. Critical current densities for both the milled and unmilled parts were higher than \(2\times10^6\) A/cm\(^2\) at 77 K. In Fig. 2, we show the I-V characteristics of the 4-\(\mu\)m-wide junction at 80 and 52 K. These characteristics can be described by the resistively shunted junction model. Some excess current is also observed. Interestingly enough, we can observe the effect of only one weak link from these I-V curves (and also from the magnetic field dependence of the I-V curves, see below). In principle, two weak links (or more) should be formed in series across the step, i.e., one at the top and one at the bottom of the step. Possible explanations might be that the two weak links have identical critical currents or the critical currents are very disparate (i.e., one weakly and one well coupled junction). The 8-\(\mu\)m-wide junction as well as the SQUID have similar I-V characteristics.

An \(I_cR_n\) product of 1 mV was obtained at a temperature of about 30 K for both the junctions and the SQUID. The \(I_cR_n\) product (filled symbols) and \(R_n\) values (open symbols)
FIG. 3. The $I_cR_n$ product (filled symbols) and $R_n$ (open symbols) measured at different temperatures for a 4-μm-wide junction (circles) and a dc-SQUID (squares).

As the temperature was decreased, the $I_cR_n$ product increased linearly and $R_n$ remained almost constant. The $R_n$ value of the 4-μm-wide junction is about twice as large as that of the SQUID (formed by two 4-μm-wide junctions), while the $I_c$ is about half, resulting in about the same $I_cR_n$ product values vs temperature. This might be due to the clean and straight step edge fabricated on the LAO substrate. Sun et al.\(^6\) reported that a-axis grains at the step edge grow with their plates aligned to the cubic axis of a SrTiO\(_3\) substrate, regardless of the direction of the step edge. They speculate that such structures might be related to a deterioration of the junction characteristics in case of a wavy or jagged edge. Thus a straight step edge over the length scale of the a-axis plates is preferred, so as to keep the a-axis plates aligned to the step edge direction.

The magnetic field dependence of the $I$-$V$ curves for the 4-μm-wide junction measured at 73 K is shown in Fig. 4. A nearly symmetric magnetic field dependence of $I_c$ with a main peak in the center was observed. One can notice that at high bias the $I$-$V$ curves are smooth vs magnetic field, which indicates one weak link or two (or more) identical weak links in series. The magnetic field dependence of $I_c$ lost its periodicity above about $\pm 2\, \Phi_0$, which may be the signature of a current nonuniformity of length scale $\approx 1\, \mu$m. $I_c$ can only be suppressed to about 40% in the first minimum. This might be connected to the excess current through the junction. There might be some superconducting channels with higher critical current densities in the grain boundary region. Junctions on other chips, that had smaller critical current densities did, indeed, display minima approaching zero value of current.

An important issue for the fabrication of step edge junctions is the reproducibility both for junctions on the same chip and from chip to chip. We have prepared two additional samples with up to 30 junctions on each chip. We can get similar $I_cR_n$ products for junctions on the same chip. However, the values of $I_cR_n$ product differed from chip to chip.

In conclusion, we have developed a process for the fabrication of sharp and straight step edges on LAO substrates (and similar substrates). It is based on easily deposited amorphous carbon films which are used as ion milling masks.

FIG. 4. Three-dimensional plot of the magnetic field dependence of the $I$-$V$ curves for a 4-μm-wide junction at 73 K.

YBCO step edge junctions fabricated on these LAO steps show promising results. An $I_cR_n$ product of 1 mV was obtained at a temperature of about 30 K. Nearly symmetric magnetic field dependencies of $I_c$, with a main peak in the center, were observed for single junctions. From the measured $I$-$V$ curves as well as from the magnetic field dependencies of the $I$-$V$ curves, we conclude that the YBCO step edge region consists of one weak link or identical weak links in series.

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