Observation of Shapiro-steps in AFM-plough Micron-size YBCO Planar Constrictions

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***Abstract*— Using an Atomic Force Microscope (AFM), we successfully ploughed micron size planar constriction type junctions on $YBa_2Cu_3O_{7-x}$ thin films. The 100 nanometer (nm) thin films are deposited on MgO substrates by an Inverted Cylindrical Magnetron (ICM) sputtering technique. The films are then patterned into 8-10 micron size strips, using photolithography and dry etching. A diamond coated tip was used with the AFM in this process. We were able to observe well defined current–voltage (I-V) characteristics and Shapiro-steps, successfully demonstrating a possible Josephson effect in these constrictions.**

***Index Terms*—AFM lithography, Josephson junction, stripline, YBCO**

I. INTRODUCTION

NOVEL electronic superconducting devices and sensors are regularly fabricated from high-temperature superconductors (HTS) and especially $YBa_2Cu_3O_{7-x}$. These devices are based on active elements called Josephson junctions. To date, various reliable processing methods for the production of Josephson junctions have been developed and reported.

Most HTS Josephson junctions are fabricated by using artificial grain boundaries, including bi-crystal, step-edge, multilayer ramp-edge, and biepitaxial junctions [1]-[4]. However, the fabrication of HTS tunnel junctions have turned out to be extremely difficult, because an epitaxial layer structure, with several different processing steps, is required [5].

The extraordinary behavior of the Josephson junction is also demonstrated in other grain boundary weak link structures, such as the Superconductor-Normal-Superconductor (SNS) structure [6], the point contact [7], variable thickness bridge [8], microbridge [9], and nanobridges [10]. The advantages of micro- and nanobridges are the low capacitance values, and the small dimensions compared to other types of weak links.

In order to fabricate nanobridges, suitable nanostructure techniques are needed. Bridges with sizes down to a few nm were successfully fabricated with focused ion beam (FIB) [11], and electron beam lithography (EBL) [12].

AFM lithography has shown to be a fascinating tool for material structuring and pattering with nanometer precision, and the technique was used to fabricate nano-plough junctions on aluminum [13], as well as variable thickness bridges on MgB_2 [8]. Earlier we have used the technique to fabricate nano- structures on YBCO thin films. However, we could not establish the Josephson effect in these junction [14].

In this paper, we report on the observation of Shapiro-steps and possible Josephson effect in the recently fabricated AFM-plough micron size constrictions on YBCO thin films, using hard diamond coated tips to scratch on the YBCO surfaces with high precision of alignment. The advantages of applying AFM lithography instead of EBL and FIB lithography are that no extra processing steps, such as adding layers of photoresist on the YBCO surface, are required. Also, we believe that the technique does not damage the superconducting properties of the constriction region as is the case with the laser-etch junctions [15].

II. EXPERIMENTAL PROCEDURE

A. Thin film preparation

YBCO thin films were grown on (100) oriented MgO substrates by Inverted Cylindrical Magnetron sputtering. Before the deposition, the thicknesses of the films were between 100 nm – 120 nm. The substrates were cleaned with acetone in an ultrasonic bath for 10 minutes, and then blown dry with nitrogen. The substrates were glued on a heater plate with silver paste. The YBCO ceramic target–substrate distance was 30mm. Pre-sputtering was applied for 15 minutes to eliminate any contamination on the surface of the target.

The substrate temperature was 740° C, the total pressure of the 1:1 argon/oxygen gas mixture was 320 mTorr, and the dc sputtering power 72 W with a current of 400 mA and a voltage of 180V. The deposition rate was approximately 2.7 nm/min. After the deposition, the films were cooled and annealed at 460° C for 30 min in a 1 atm oxygen environment before it was allowed to cool down to room temperature.

The surface morphology of the films was characterized by AFM. X-ray diffraction was employed to characterize the epitaxial growth, and to detect the presence of any unusual phases.

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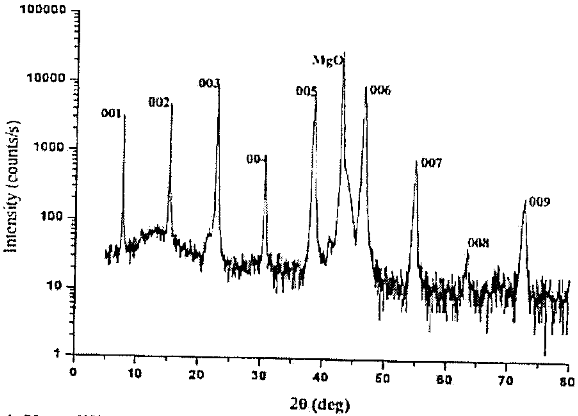

Fig. 1. X-ray diffraction patterns of YBCO thin films on (100) MgO substrates.

The resistive behavior of the films was measured. Silver (Ag) contact pads with thicknesses of 600 nm were evaporated onto the films surface and subsequently annealed at 470° C in a 1 atm oxygen environment for 30 min, in order to obtain low-resistivity contacts.

B. Micro-plough constriction fabrication

The YBCO film was patterned into microstrips with 8–10 μm widths, using standard photolithography and argon ion milling.

Photoresist (ma-P 1225) was spun onto the film surface at 4000 rpm to a thickness of 2 μm, and soft-baked on a hot-plate at 100° C for 10 min. The resist is exposed to 360 nm UV through a chrome contact mask for 25 sec, and then developed for 50 sec in ma-D 331 developer. The resist was then hard-baked at 110° C for 30 min.

Argon ion milling was performed using a Kaufman-type ion source with an incidence angle of 45° to the film surface. The rf power was 50 W, the argon gas pressure 0.25 mTorr, and the voltages of the electrodes were 750 V.

In order to minimize the loss of oxygen from the superconducting layers during the milling process, the film was mounted on a water-cooled copper sample holder with thermal paste.

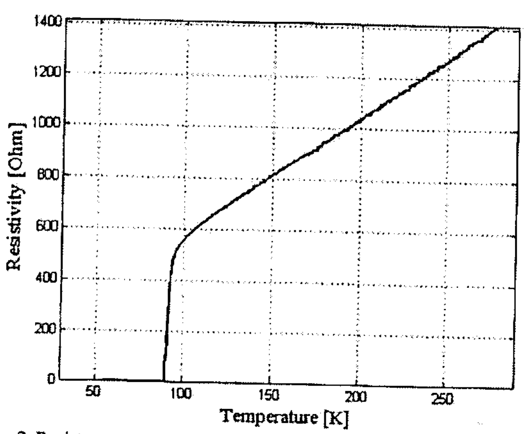

Fig. 2. Resistance versus temperature characteristic for 100 nm YBCO film.

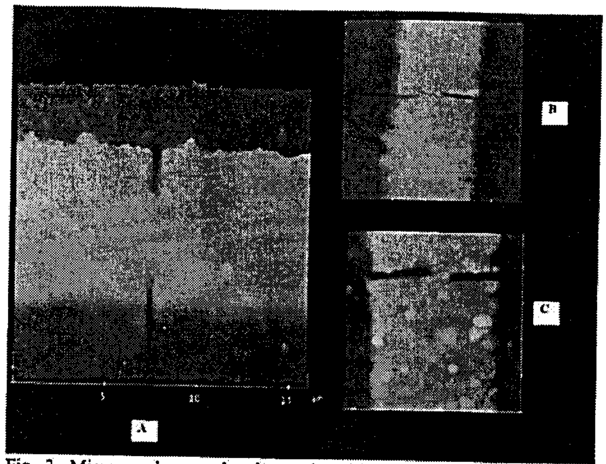

Fig. 3. Micro- and nano-ploughs made with AFM nanolithography. The widths of the constrictions are: a) 4 μm , b) 2 μm , and c) 750 nm .

The etching was terminated when an open circuit on the substrate at the sides of the YBCO striplines was measured. Photoresist residue was removed by immersing the film in mr-Rem 660 photoresist remover for 10 min, and then immersing it in acetone for 10 min in an ultrasonic bath.

We used a diamond coated tip as a cutting tool to define the plough. The AFM was operated in contact mode, with the tip vertically displaced toward the YBCO surface with a loading force of 11 μN, sufficient to completely remove the YBCO layers.

The YBCO stripline is imaged first, and then the middle of the stripline is moved to the centre of the image. At the start of the nanolithography process the tip is placed at the centre of the image at the centre of the stripline width. The constriction width W is controlled by displacing the tip by W/2 to the left side on the stripline. The tip is then driven into the YBCO surface, and displaced on the same line for a few hundred cycles. The tip velocity was 4 μm/s. The constriction is completed by applying the same nanolithography on the right side of the stripline. Constrictions with fully controlled width and depth were achieved by adjusting the tip movement and the scan speed of the plowing operation. The YBCO residuals from the cuts were cleaned by dipping the film in acetone in an ultrasonic bath for a few minutes. The drawback of this technique is the wear and resolution degradation of the tips after it has been used for a few times in the lithography.

III. RESULTS AND DISCUSSION

A. Thin film characterization

The surface morphology of the YBCO thin films was investigated using AFM. The films had smooth surfaces, with surface roughness values between 4-6 nm. No droplets or outgrowths were observed on the surface of the films.

The X-ray diffraction patterns of 100 nm YBCO thin films are shown in Fig. 1. The films were c-axis oriented, strong and weak (00l) peaks of the superconducting YBCO phases were observed, as well as a few peaks of some impurities.

The amplitudes of the impurity peaks are much smaller than the amplitudes of the peaks of superconducting YBCO phases.

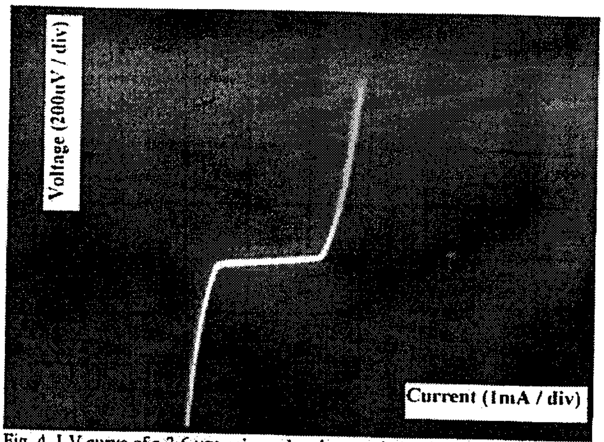

Fig. 4. I-V curve of a 3.6 μm micro-plough constriction junction at 57 K.

Fig. 2 shows the temperature dependence of the electrical resistance. The films show metallic-like resistive behavior in the normal state and have sharp superconducting transitions.

The residual resistance ratio R (300)/R (100) was 2.83, the zero resistance temperature (T_{c0}) was 90.2 K, and the width of the superconducting transition about 1 K. These results confirmed that the films had good superconducting properties.

B. Possible Josephson effect in micro-ploughs

After successful fabrication of the ploughs with AFM nanolithography (see Fig. 3), the film was mounted on a PC board. Gold wires were bonded from the silver contact pads to copper striplines on the PCB. The sample was then positioned inside the cold finger cryo-cooler unit for testing.

We used the Mr. SQUID Electronics unit [16] as excitation source. It generates a triangular waveform to test the device, and we monitored the I-V response on the oscilloscope window.

The current-voltage (I-V) characteristics of a 3.6 μm width micro-plough, in the absence of microwave power, at 57 K are shown in Fig. 4. The critical current was measured to be 1.58 mA at 47 K. This temperature was the lowest that the specific cry-cooler could reach. The normal resistance of the junction was 1.2 Ω, and the I_cR_n product was 90 μV at 77 K.

As a result of thermal fluctuations or flux flow a rounding effect can be present in the I-V characteristics of a junction [17]. However, our I-V curves exhibit sharp knees, possibly ruling out any thermal fluctuations or flux flow effects. This means that the I-V curves are most probably representing true Josephson behavior.

However a mere observation of I-V characteristics with dc currents may not necessarily mean the demonstration of the Josephson effect. In order to establish the Josephson effect, one should demonstrate ac and dc Josephson effects, and the magnetic modulation of critical currents. In this paper we adopt the method of the observation of Shapiro-steps, which is a direct consequence of the Josephson effect.

Accordingly, measurements on the I-V characteristics of the micro-plough junctions have been performed in the presence of an external microwave power source.

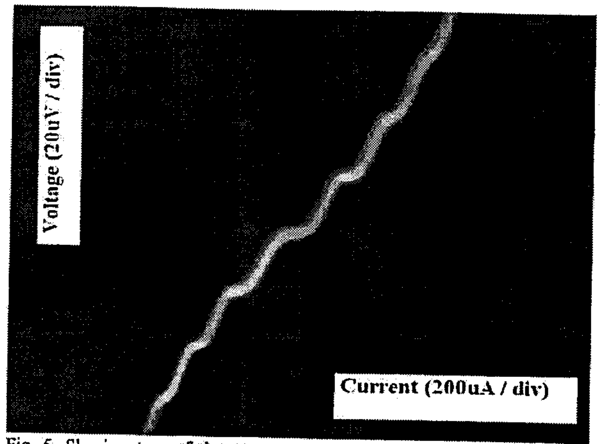

Fig. 5. Shapiro-steps of the same constriction junction (Fig. 4) exposed to 10.225 GHz MW power at 54 K.

We applied 3 dBm microwave power in the 2 – 18 GHz range via a coaxial cable terminated with an antenna above the device. We observed well defined Shapiro-steps on the I-V curve at 10.225 GHz (see Figure 5). The response of the constriction to microwave radiations (Shapiro-steps) clearly gives positive evidence of the Josephson effect. This is because the observed step size satisfies (1). which can only be derived from the fundamental Josephson effect.

The step sizes can be used to calculate the theoretical constant $\frac{e}{h}$.

The voltage step is expressed as

$$V_0 = n\left(\frac{\Phi_0}{2\pi}\right)\omega_s \qquad (1)$$

which is equivalent to

$$V_0 = n\left(\frac{h\omega_s}{4\pi e}\right) = n\frac{hf_s}{2e} \qquad (2)$$

where $\Phi_0 = \frac{h}{2e}$. This equation can be rewritten to yield the theoretical constant $\frac{e}{h} = 2.41796\times10^{14}\,Hz/V$ as:

$$\frac{e}{h} = n\left(\frac{f_s}{2V_0}\right). \qquad (3)$$

The voltage V_0 is approximately $21\,\mu V$ with some degree of uncertainty and the frequency f_s =10.225 GHz. Substitution of these values into (3) results in $\frac{e}{h} = 2.4345\times10^{14}\,Hz/V$. This value corresponds quite well with the theoretically predicted value, constituting positive evidence for the Josephson effect. However, at this stage we have to recall a deeper argument by Likharev [18], that similar voltage steps can occur due to the motion of Abrikosov vortices in a weak link whose width is larger as compared to critical width w_c ($w_c/\xi \sim 4.4$), where ξ is the coherence length, which is a few angstroms. Therefore, with our constriction widths in micron size, we are in a regime where w/ξ is > 4.44, or the critical limit where Abrikosov vortex motion is possible. Such a phenomenon will also lead to voltage steps upon microwave irradiation of the junction, similar to the Shapiro-steps [18]. However, one should note that the abrikosov vortex motion is only possible in a junction free of inhomogeneities [18]. Even small inhomogeneities will pin the fluxons so that they can not move. In high-Tc cuprate superconductors, where the coherence length is very small, even a small defect can act as a pinning center. From an experimental point of view it is very unlikely to deposit an YBCO thin film that will be absolutely free of inhomogeneities. These inhomogeneities that naturally occur during deposition of thin films will pin any Abrikosov vortices and therefore there should not be any vortex motion. In this scenario, we believe that the observed steps are more likely the Shapiro-steps than the steps due to Abrikosov vortex motion.

Fig. 6 shows the temperature dependence of the critical current. The critical current $I_c(T)$ follows a quasi-quadratic dependence for temperatures near the critical temperature, $I_c(T) \propto \left(1-\frac{T_c}{T}\right)^2$.

IV. Conclusion

In conclusion, we have produced micron-size constriction type junctions on YBCO thin films by AFM-lithography. The junctions were tested for the Josephson effect, we have observed I-V characteristics and Shapiro-steps with external microwave irradiation of the constrictions, which demonstrate the possibility of the Josephson effect in these junctions. The voltage step value corresponds well with the predicted theoretical constant for Shapiro-steps. Measurements of the magnetic field modulation of critical currents in these constrictions are an important part of our immediate future work.

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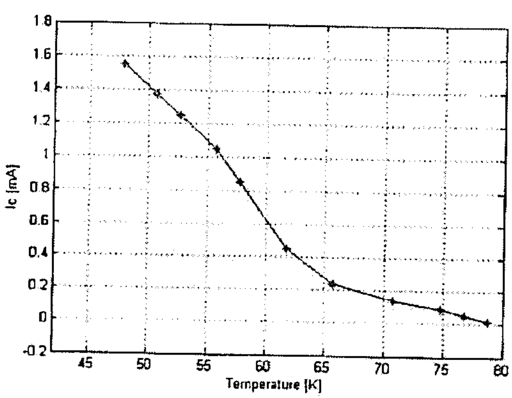

Fig. 6. Temperature dependence of the critical current in a 3.6 μm micro-plough junction.

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