Growth of Superconducting Nano Films of YBaCuO by Spray Pyrolysis Technique and Study of Quantum Interference Effects

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Abstract
Nano films of YBaCuO superconductors are grown by spray pyrolysis technique. Annealed films are superconducting with $T_c(0)$ at 86 K. In this paper we report observation of superconducting quantum interference effect in a rf-SQUID like geometry fabricated in 5µm thick YBaCuO film prepared by spray pyrolysis technique (top down approach). The film fabrication process, I-V characteristics are discussed along with V-$\Phi$ characteristics at different temperatures and noise spectrum at different frequencies of rf squid.

1. Introduction.
High temperature superconductors (HTS) are granular in nature and the grains are coupled through Josephson Tunneling. YBaCuO Superconducting thin films fabricated from spray pyrolysis technique. Superconducting quantum interference effects have been observed in case of thin films which are made from bottom up approach [1-4]. However, very little work has been reported on the study of quantum interference effects in spray films of high temperature superconductors (HTS) [5].

In this paper we report observation of superconducting quantum interference effect in rf-SQUID like geometry fabricated in 5µm thick YBaCuO film prepared by spray pyrolysis technique. The film fabrication process and I-V characteristics are discussed. The latter aspect is important if the film is to be used for future device applications.

2. Experimental Details.
The films used in this investigation are prepared by spray pyrolysis technique [6, 7]. The set-up is as shown in the figure 1.

Although the technique of thin film preparation by spray pyrolysis is very simple, it has several sensitive parameters to be controlled carefully for obtaining a good quality film. The process depends sensitivity on the details of automatization, distance between the nozzle and the substrate and the temperature of the substrate. One has to optimise several other parameters such as flow rates of solution as well as carrier gas, concentration of solution, alcoholic content added to the solution and duration of spray.

Fig 1 is a schematic diagram of spray pyrolysis set-up used in the present investigation. A simple nozzle made of Pyrex glass with a fine orifice is used to spray alcoholic solution containing nitrates of Yttrium, Barium and Copper in the atomic ratio 1:2:3. The solution is prepared by dissolving suitable amounts of Y(NO$_3$)$_3$; 6H$_2$O; Ba(NO$_3$)$_2$ and Cu(NO$_3$)$_3$, 3H$_2$O in triply distilled water. The solution is diluted with 20% Methanol in order to enhance the speed of evaporation during deposition. The films are deposited by downward spray of the solution on single crystal SrTiO$_3$ (100) &MgO (100) substrate which are kept at 400°C. The temperature of the substrate is continuously monitored during the spray by a chromelalumel thermocouple and is controlled within ±10°C. Oxygen gas has been used as carrier gas. After deposition of the film, the substrate are heated for 10 minutes in air at 800°C (pre-heating). The cycle of spraying and pre-heating is repeated up to 4 times in order to control the thickness of the film. This renders the good quality film with thickness of 5µm. The film thus prepared is given heat treatment in two stages as follows.

Sample annealed in an Oxygen environment at 950°C for 15 minutes followed by slow cooling (1°C per minute) to 550°C.

The sample is maintained at 550°C for 1 hour and is then slowly cooled to room temperature with the rate of 1°C per minute.
Figure 1. Schematic diagram of spray pyrolysis set-up for film preparation.

3. Result and Discussion.

Low resistance contacts are made using air drying silver paste. The resistivity of the sample is measured using a conventional four probe technique. The temperature of the sample is determined by thermometer within an accuracy of ± 0.2K. Fig 2 shows R-T curve with Tc (0) at 86 K. The in-set shows the geometry of the rf- SQUID with dimensions of 50µm × 50µm. I-V characteristics of micro bridge carved on thin film is shown in the figure 3.

Figure 2. Resistance Vs Temperature curve Y-Ba-Cu-O film prepared by spray pyrolysis method.

Figure 3. I-V Characteristics of micro bridge carved on a thin film at 77K

The rf- SQUID consist of a single Josephson junction integrated into a superconducting loop that is inductively coupled to the inductance L_T of the LC tank circuit. The tank circuit is driven by an rf current, and resultant rf voltage is periodic in the flux applied to the SQUID with a period Φ_0. In order to see the V-Φ behaviour of the rf-SQUID, a conventional commercial rf-SQUID electronics is used [8] with some modifications. Magnetic field is applied through an external af- oscillator. Schematic diagram of experimental set-up for observing rf-SQUID characteristics is shown in Fig 4.

Figure 4. Schematic diagram of rf-SQUID electronics set-up for seeing V-Φ Characteristics

rf- SQUID can be operated in two modes[9,10]. One is in hysteretic mode for which β_L > 1 and the other is in non- hysteretic mode for which β_L <1. Historically most of the low Tc rf-SQUID were operated in the hysteretic mode although there are advantages to the non hysteretic mode. Thus when the tank circuit is driven at constant frequency (19.6 MHz), the variation in the resonant frequency cause the rf-voltage to be periodic in Φ_0. While working in hysteretic mode the SQUID makes transition between quantum states and dissipates energy at a rate that is periodic in Φ_0. This periodic dissipation in turn modulates the quality factor Q (32) of the tank circuit so that it is driven on resonance with a current of constant amplitude, the rf- voltage is periodic in φ as shown in the figure 5 for different temperatures.

Figure 5 : V-Φ Characteristics at different temperatures.
The noise spectrum of rf-SQUID is shown in the figure 6. The SQUID has a flux noise of $2 \times 10^{-4} \Phi_0 / \sqrt{(Hz)}$ about 20 Hz. At Lower frequencies 1/f noise is found to predominate.

Figure 6: Noise spectrum of rf-SQUID at different frequencies

4. Conclusion.
Good quality superconducting nano films of YBaCuO made by spray pyrolysis and annealed suitably are superconducting with $T_C(0)$ at 86 K. RF-SQUID using grain boundary weak links in a micro bridge of YBaCuO superconducting film has been developed and it is operated at 77 K successfully. The SQUID has a flux noise $2 \times 10^{-4} \Phi_0 / \sqrt{(Hz)}$ about 20 Hz. At Lower frequencies 1/f noise is found to predominate.

5. References.