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Transport Critical Current in MOCVD $YBa_2Cu_3O_7$ thin films using a pulse technique.

F. Weiss, G. Delabouglise, O. Lebedev, K. Fröhlich*, E. Mossang, J.P. Sénateur, O. Thomas

INPG, ENSPG, LMGP, UA 1109 CNRS, BP46, 38402 St Martin d'Hères, (France)

*IEE, Slovak Academy of Sciences, Dubravska Cesta 9, 84239 Bratislava, (Czechoslovakia)

Abstract

Transport currents in epitaxial thin layers of $YBa_2Cu_3O_7$ have been measured by a pulse technique which avoids heating at the contacts. We give a complete description of the experimental set up, which allows the measurement of critical currents as a function of temperature, applied field and angle. Thin films have been obtained by Metalorganic Chemical Vapor Deposition on different kinds of substrates: (100) $SrTiO_3$ and (100) MgO. These films have been patterned by chemical etching and bridges of typically 100μm width have been obtained. The microbridges for I-V measurements can be as wide as 100μm since the contact heating during critical current measurements is considerably reduced in the pulse method and large current intensities (1A and over) can be used without damage. Typical results of Jc(T, H) and of critical current anisotropy will be given for selected MOCVD samples.

1. Introduction.

Critical current measurements in high temperature superconductors (HTS), as a function of the magnitude and angle of the applied magnetic field, represent a major task in the studies of flux pinning mechanisms and in the development of superconducting materials for future applications.

Different determination techniques of Jc have been used. They present often some discrepancies due to the validity and sensitivity of the detection criteria used to define Jc. Very attractive are indirect determinations of Jc (e.g. from DC, AC magnetization or screening measurements (1,2)) which allow to use as produced samples without further preparation. They give a mean value for Jc, because the induced screening currents run around the whole sample. They are however dependent on the theoretical model used for the critical current evaluation and are complicated by the fact that, due to the high anisotropy of HTS materials, different components of the critical current are often complemetary induced.

The most direct method to determine the strenght of the superconducting current, being independent of any theoretical model, is certainly represented by transport measurements. In function of temperature, applied field and angle between the crystal structure and the applied field, the measured critical current behaviour describes different pinning situations which have been analyzed by various authors.(3-6). The direction of the external magnetic field determines the shape of the flux lines and their direction within the crystal structure, while the applied current determines the Lorentz force and the direction of the observed pinning force.

The four terminal method involved to perform these measurements needs patterning and contacting techniques (7,8) in some way complicated due to the particular nature of HTS materials. In order to measure a sample, over a wide range of temperature, applied field or angle, with reasonably low current intensity, the measuring bridges have to be very small, because the critical current densities at low temperature are very large. On the other hand, with large transport currents, the contact resistance between superconductor and current leads must be very low to avoid excessive contact heating and needs a careful technology.

In order to reduce these two correlated problems of bridge width and contact resistance, short current pulse measuring techniques can allow an easy characterisation of HTS samples. With that technique, large transport currents can be used to measure the transport critical current in epitaxial thin layers of HTS layers, with contact resistances of relatively less good quality than in direct current measurements. This technique can also be associated to a wet etching patterning technique,

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easier to perform, because the bridge widht is no more crucial.

In the present paper we describe the characteristics of our pulse measuring system and give some typical results for HTS $YBa_2Cu_3O_7$ layers grown on MgO and $SrTiO_3$ substrates by Metal Organic Chemical Vapor Deposition (MOCVD).

2. Experimental details

2.1 Film synthesis

Thin films (A, B and C) of $YBa_2Cu_3O_7$ were deposited on substrates of MgO (A) and $SrTiO_3$ (B,C) by MOCVD, in a hot wall system utilizing (thd) precursors of Y,Ba and Cu.(9). The thickness of the films is 150 nm for (A) and 40 nm for (B and C). X-ray diffraction patterns demonstrated that these layers are strongly textured with the cristallographic *c*-axis perpendicular to the film surface. Scanning Electron Microscopy (SEM) analysis exhibited a density of precipitates around $10^6/cm^2$ (presumably copper oxide) on the flat layer surface. For very thin layers (e.g. samples B and C) however, the surface roughness decreases considerably. Zero-resistivity temperature were at 78K for sample A and around 87K for sample B and 89K for sample C.

2.2 Patterning and contacting .

Different patterns for the transport Jc measurements were produced by photolithographic methods. The patterning process was considerably simplified and included the use of a standart photoresist (Photogelt PH4) which can be applied as an aerosol, revealed in a 2% KOH solution and easily removed by acetone.

The four terminal structure with a narrow constriction was produced by a chemical wet etching procedure. Against ion milling, this technique is presumed to be less performant (because of bottom etching) in the definition of steps and precise lines, especially for very narrow bridges (a few microns). In the present case however, large constrictions (100 μm) have been patterned and side effects due to the wet etching process can be neglected, if they exist. The selected etching solution (3% HCl + 2% HNO_3) allows to dissolve the superconducting phase $YBa_2Cu_3O_7$ as well as eventually other coprecipitated phases from the phase diagram (generally CuO or $BaCuO_2$ or .Y_2BaCuO_5)

The dimensions of the current bridge used were 100x500 μm. After patterning, the superconducting properties of the sample were not affected (same critical temperature Tc, same surface morphology). By SEM inspection it appears that the bridges are well defined with sharp steps, even sharper for thinner layers. An increase of the width of the stripline, as performed in this study, reduces the risk of failure and reduces also the probability to degrade the current carring capacity of a sample just by inclusions or small inhomogeneous or defective regions in the current path.

Current contacts, generally well optimized in order to improve contact resistances (7), were simply realized with Indium solder on the film surface without need of gold or silver clading sheat. Further connection to the measuring system were made with copper wires .This contacting procedure speeds up the sample mounting, without observable degradation in the sample properties.

2.3. Measuring system.

Critical current measurements were made in a classical four terminal configuration. A sketch of the measuring system is given in fig.1.

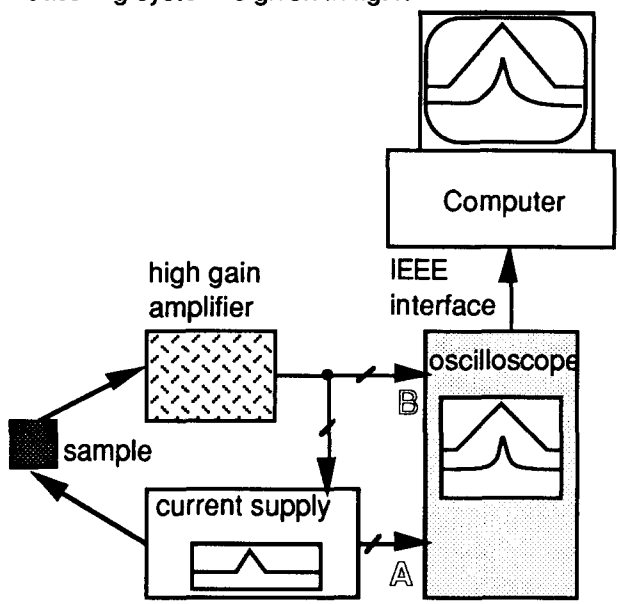

Figure 1. Lay out of the measuring system.

The current supply generates a current pulse, with a well defined slope in a time range varying from 1 ms to 100 ms. The current amplitude can be adjusted in different ranges from 10 mA to 40A depending on the nature of the analyzed samples (from thin layer to bulk materials).
The voltage issued from the superconducting sample passes trough a high gain (10^4) amplifier and is recorded together with the current pulse on a memory oscilloscope.

Raw data can be stored and analyzed using a computer connected to the oscilloscope.
A feedback loop, between voltage amplifier and current supply, allows to invert the testing current as soon as a given electrical field (e.g. 10 μv) is detected on the voltage leads. Contact overheating can be this way monitored by reducing both the voltage criterion or the current pulse.time.
A voltage criterion of 1 μV increase can be easily detected to obtain the critical current. Thus, depending on the separation of the voltage pads (typically 500 μm), a,criterion of 20 μV/cm sensitivity was commonly used to characterize the superconducting samples.

2.4. Experimental

The characterization of HTS films has been made in function of temperature, applied field and angle.

Samples are mounted on a static sample holder in a closed cycle Helium cryostat (10K<T<300K). The angular dependence was measured in fixed magnetic fields (up to 1T) as established by a split field rotatable electromagnet ($\Delta\theta$=0.5°).The current direction was chosen to be parralel to the rotation axis, in order to establish the maximum Lorentz force for any orientation and is always flowing in the *ab*-plane. Because of symmetry reasons, all the data are restricted to the range $0<\theta<90°$, where θ=0° corresponds to the applied field *H//c* and θ=90° to *H// ab*-planes.

3. Results and discussion.

Measurements on 3 films are reported.
The first film on a MgO substrate (sample A) has a relatively low critical current density of 4.10^5 A/cm^2. at 60K. The angular dependence of critical current *Ic* at different temperatures is plotted in fig.2 for *H*=1T.
SampleB and C have very large current densities. In fig.3.for sample B, at 50K, the critical current reaches a value of 500 mA in a superconducting section of 40nmx100μm. The critical current density is of the order of 3.10^6 A/cm^2 at 77K.
For sample C (the angular dependence of critical current *Ic* (θ) at an applied field of *H*=1T is shown in fig.4. at a temperature close to Tc. At 84.5K, the anisotropy factor is about 10 and the critical current density *Jc* is always around 10^5A/cm^2.

A maximum of *Ic* at θ=90° and a minimum at θ=0° are typically observed, in both type of sample, with a smooth angular dependence in between. As temperature increases, the peak at 90° becomes more prononced and emerges from a less angular dependent bakground. Near Tc at 76K (sample A), this peak, attributed to an intrinsic pinning phenomenon of vortices by the layers between the CuO planes, separates regions where, depending on the angle, the sample is above or below the irreversibility line.

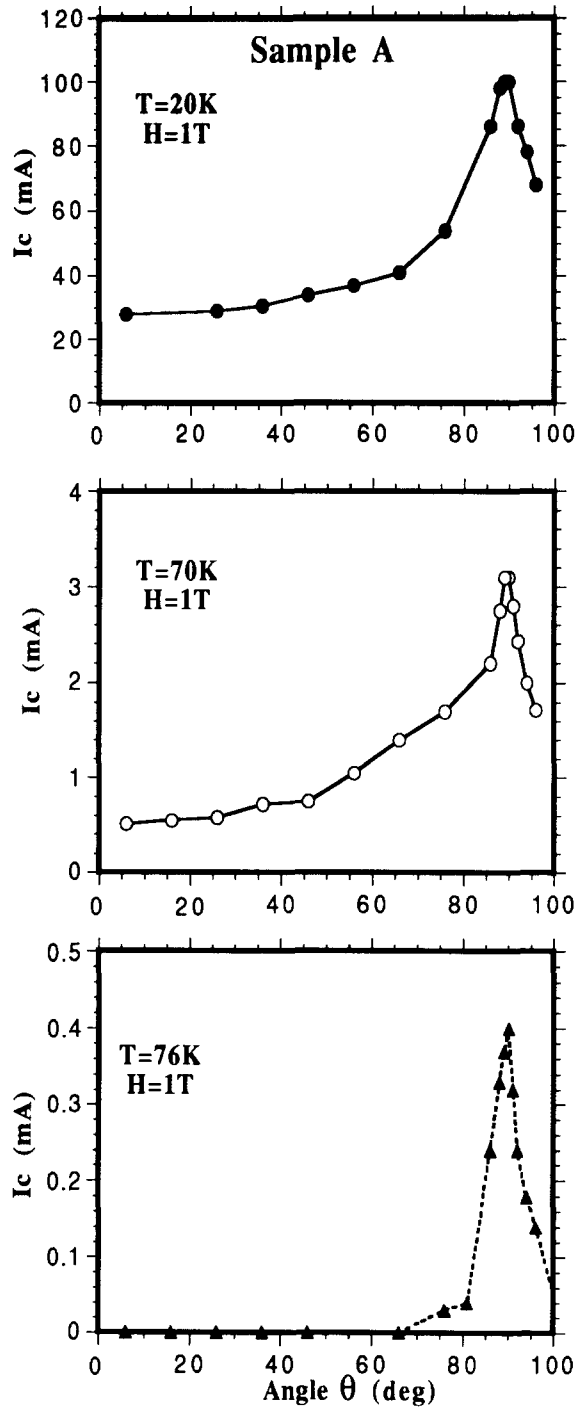

Figure 2. Angular dependence of critical current at 20K, 70K and 76K showing a more pronounced anisotropy as temperature increases.

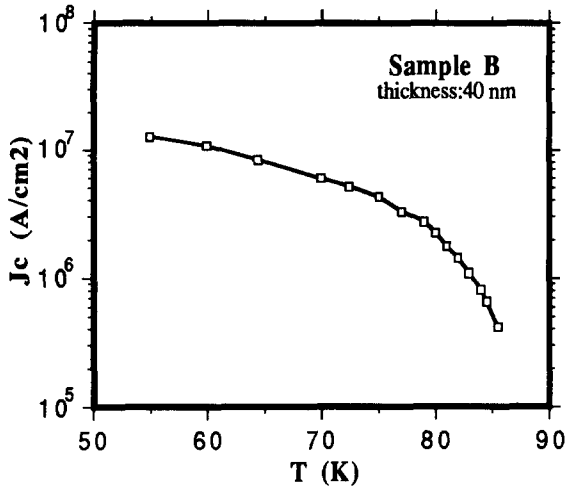

Figure 3. Critical current density of sample B in function of temperature, H=0.

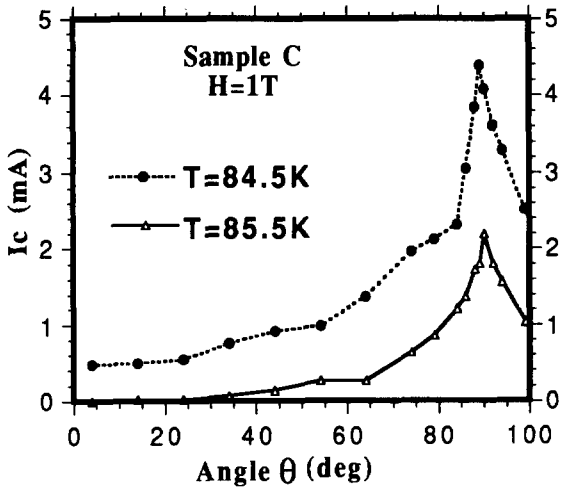

Figure 4. Angular dependence of *Ic* close to Tc for sample C.

Intrinsic pinning, as presented here exists only in a narrow angular region where the applied field lies in the *ab*-planes. The sharpness and height of this peak is certainly a criterion of sample quality and brooder peaks are generally related to less anisotropy (samples with more defects and smaller Tc's).

The half maximum half width Δθ (HMHW) of the *Jc*(θ) curve decreases with increasing applied field and can be strictly determined only when the other contributions have disappeared. Nevertheless for sample A, at 76K, it can be well established at Δθ=4° and is to be compared to the value of Δθ=0.5° typically determined on MOCVD films with X-ray texture scans. The residual discrepancy can be explained by the strong influence of defects, such as stacking faults on the intrinsic pinning mechanism.

Finally the smooth angular dependence of *Jc*(θ) suggests a predominant role of point defects in our samples. The surface roughness, which should induce a maximum at θ=0°, seems to have only little influence on pinning. Further studies are nevertheless needed to understand more precisely the different pinning mechanisms involved in HTS MOCVD thin layers.

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