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Microwave Characterization of $YBa_2Cu_3O_{7-x}$ Thin Films Grown *in situ* by Off-Axis Magnetron Sputtering (*).

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Summary. — Superconducting films of $YBa_2Cu_3O_{7-x}$ were deposited *in situ* on $LaAlO_3$ substrates using single-target 90° off-axis sputtering. The obtained films have tipical T_c values of 91 K. Surface resistance measurements on as-grown films reach 1.1 mΩ at 77 K and 10 GHz; whilst on ion-etched patterned resonant lines *Rs* (77 K, 10 GHz) it is about 10 mΩ.

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PACS 84.30.Ey – Microwave circuits (*e.g.*, parametric, solid state).
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1. – Introduction.

A large variety of microwave devices made of high-T_c superconducting material have already been investigated by different authors, and their behaviour compared with those made up of conventional metals. It was found that, at liquid-nitrogen temperature and below, the properties of the superconductor-based devices are better up to frequencies of 50–100 GHz. There is a lower surface resistance and therefore a lower loss offering good prospects for their application as microwave devices. In this paper we present the growth characteristics of sputtered YBCO films and an investigation of R_s taken at 77 K and 10 GHz both on an as-grown film using a resonant-cavity technique and on a patterned microstrip line resonator.

Among the deposition methods which have been used to make $YBa_2Cu_3O_{7-x}$ *in situ* films included laser ablation [1, 2], evaporation [3, 4], composite-target sputtering [5-7], and molecular-beam epitaxy [8] or others [9-11], we have used a 90° off-axis sputter technique in which almost the exact composition of the target is obtained in a rotated substrate block to improve uniformity. The off-axis method

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employed benefits both from high pressure and off-axis geometry. The term *in situ* means that the $YBa_2Cu_3O_{7-x}$ phase is formed during growth and it takes place on the surface plane of the film in the presence of an incoming flux of atoms and ions, except for the final oxygen occupancy which takes place during the cool-down. Typical growth rates are such that it takes several seconds to deposit a monolayer. The temperature must be high enough during that time so that the mobility of the constituents is sufficiently great to allow them to order on their respective sites. In the *ex situ* technique superconductor films are deposited at or near room temperature with the correct composition, but with disordered or amorphous phases. In that case post-deposition annealing process was required to produce the desired crystalline films.

2. – Deposition process.

The thin films were deposited using a planar magnetron sputter gun in a commercial K. J. Lesker deposition system. The sputter gun was mounted in a cryopumped vacuum chamber; the base pressure was $3\cdot 10^{-8}$ Torr. Stoichiometric 3 in. diameter target was prepared from citrate pyrolysis powders[12]. The sputtering atmosphere varied between 40 to 60 mTorr O_2 and 160 to 240 mTorr Ar. An RF power of 100 W generated a cathode self-bias of -70 V and gave a deposition rate for the off-axis geometry of about 0.1 Å/s which depends on the total pressure and Ar/O_2 ratio. In our case it ranges from 200 to 300 mTorr with 20% O_2 and 80% Ar. During the measurement sputter gun was switched on and shuttered. The substrates were bonded by silver paste on the holder. During film growth the substrate block temperature was held constant between 730 and 750 °C. The films obtained were 0.6 μm thick on $LaAlO_3$ (100) substrates. After deposition, the chamber was

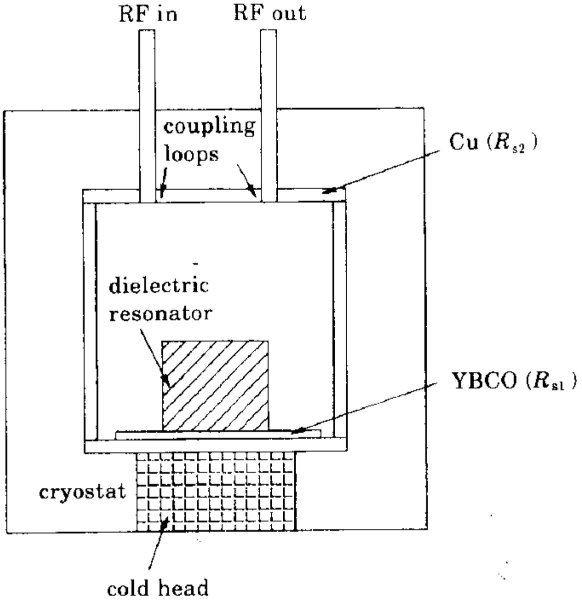

Fig. 1. – Cylindrical cavity layout.

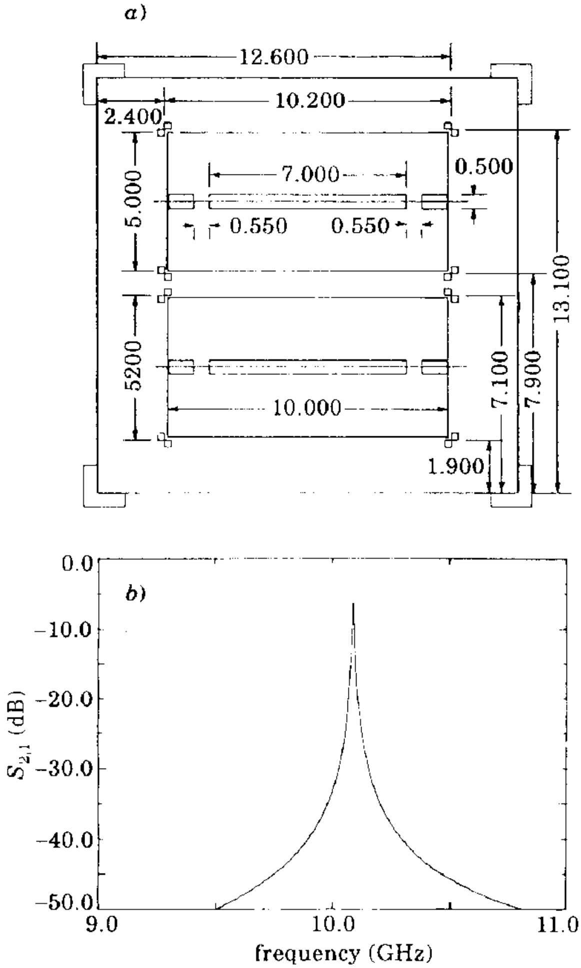

Fig. 2. – a) Patterning layout. b) S_{21} CAD simulation.

immediately vented to 20 Torr of pure oxygen and the substrates were allowed to cool slowly to room temperature. As well known a key role is played by the oxygen partial pressure and by temperature during the deposition process but the exact effects of both of them on superconducting properties are not yet completely clarified. Concerning the oxygen partial pressure, referring to the phase diagram of the Y-Ba-Cu-O system based on the data of Hammond and Bormann [13, 14], we know that the phase formation is complicated by the presence of plasma which activates the oxygen. The presence of oxygen plasma enables the *in situ* growth to occur at lower oxygen pressure. Concerning the substrate temperature we know that when it is below a certain threshold a purely *a*-axis–oriented film is formed. At higher temperature purely *c*-axis–oriented films are grown. The exact value of these parameters depends strongly on the deposition atmosphere and geometrical conditions and should be optimized for each deposition system.

3. – Results and discussion.

Among the microwave measurement methods for testing materials properties the dielectric-resonator method is one of the simplest. This measurement has been made by using a metallic cylindrical cavity including a dielectric resonator as shown in fig. 1 in which

$$1/Q = A\,\mathrm{tg}\,\delta + B_1 R_{s1} + B_2 R_{s2}\,, \tag{1}$$

where $\mathrm{tg}\,\delta$ refers to the dielectric resonator, R_{s1} to the superconductor and R_{s2} to the copper. By choosing appropriate dielectric constant and resonator dimension, it is possible to measure over the range 2 to 40 GHz. For thin-film measurement at 10 GHz we have used TiO_2 as dielectric which has $\varepsilon_r = 85$ and dimensions 7 mm diameter and 1 mm height. Typical results, obtained with a scalar network analyser within a cryostat equipped with high-frequency probes, are depicted in the following table:

Sample	$R_s\,(m\Omega)$	T_{c_0}(K)	ΔT_c(K)
FY0993	1.25	91.0	≤ 1.0
FY0294	1.10	90.2	1.2
FY0394	1.30	90.2	1.0

In this case the structure frequency, the quality factor and the contribution of each part of (1) are given by a computational method, 2D finite element method. Of course the structure geometry design must be adjusted according to the characterization. For ε_r and $\mathrm{tg}\,\delta$ accurate measurements have been done using a dielectric resonator.

Subsequently, the films were patterned using a conventional photolitographical process (AZ1818) and an ion etching obtained by an Ar/O_2 mix. The patterning layout is depicted in fig. 2*a*) as well as an Eesof CAD simulation of S_{21}, fig. 2*b*). Figure 3 shows the S_{21} measured at 77 K, and from that measurement it is

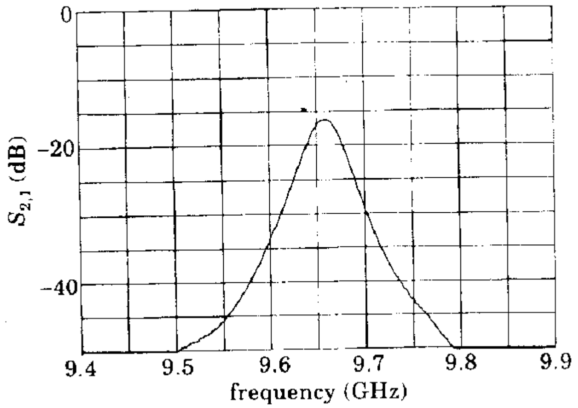

Fig. 3. – S_{21} measured on microstrip line resonator.

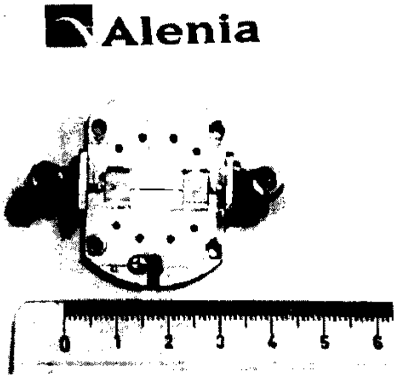

Fig. 4. – Photograph of the resonant line.

possible to obtain the surface resistance value given by

$$R_s = \pi Z_0 W/\lambda Q_0 \approx 10\ \mathrm{m\Omega}\,.$$

Figure 4 shows a photograph of the resonant line.

4. – Conclusions.

We have grown YBCO thin films and performed microwave measurements on as-grown and patterned films at 10 GHz and 77 K. From these measurements the surface resistances were determined in the as-grown case using a numerical calculation and in the patterned one using the standard R_s definition for a microstrip line resonator. A significant drop in R_s value of an order of magnitude in the patterned film is attributed to the detrimental effect of the ion etching over the superconducting properties as already observed on the same samples by magnetic measurements.

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