Preparation techniques of thin film ITC superconductors

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Abstract. This paper considers the several deposition techniques currently in use for deposition of thin film superconductors with reasonably good results.

Keywords. Ultra-high vacuum systems; ITC superconductors; thin film preparation.

1. Introduction

High temperature superconductors, which at present have the highest technological interest, are mainly those based on alloys of Cu oxides with Ba and rare earths with Bi, Sr, Ca or with Tl, Ba, Ca. They show critical temperatures in the range 80° to 125°K, and show common characteristics such as (i) stratified structure, (ii) electrical conductivity in the copper oxygen regions and (iii) anisotropic electrical properties. The process of producing films of those superconductors must fulfil the following requirements: (i) correct stoichiometry; (ii) correct crystal structure and (iii) sufficient oxygen for the build-up of the correct phase. Furthermore all processes should be done *in situ* avoiding any post-annealing at high temperature in a separate oven. Many different deposition techniques are presently used for the deposition of thin film superconductors with acceptably good results. Here we consider the techniques more widely used.

2. Evaporation techniques

The evaporation of materials using an electron gun was historically the first technique used to produce high T_c superconductors 1-2-3 (IBM, Stanford, Cornell). In this case a co-deposition or sequential deposition technique from 3 different electron beam sources is used.

Typically individual sources for Y, Ba and Cu were used; sometimes Ba was substituted by BaF₂ with advantages connected to the lower deposition temperature required and to the stability of films during the process. For Ta and Bi compounds a sequential deposition technique is not often used due to the number of sources required and the high vapour pressure of Ta. Good results have been achieved with MBE techniques using both effusion cells and electron beam sources. These techniques seem to be very promising.

Today various companies can offer among their product line UHV systems in which both high power (10 kW) electron guns or effusion cells are used as deposition sources, as well as sample manipulators that can allow high temperature processes (up to 1000°C) with rotation capability to allow the necessary film uniformity. The key issues of the above processes are: a fine control of the individual sources (related to the thickness) and the need of oxygen during deposition. The thickness control can be easily monitored using techniques based on quartz frequencies/thickness relationship. The results are much better than those achieved through vapour diagnostic controls because of the contamination of the sensor heads and the difficulties of the multisources

system geometry. The second issue is more delicate due to the general incompatibility between the presence of oxygen in a chamber and the simultaneous operation of an electron gun. To solve this problem many different oxygen sources have been used. It is advisable to use both shower sources and oxygen ion guns. This latter has the advantage of having the best control of the oxidation and of being used as etcher when needed.

3. Sputtering

Practically all known techniques have been used, from RF diodes to magnetrons DC or RF, with good results. Both multiple individual element sources (alloys and oxides) or single sources have been used. Using a single source with stoichiometry target, problems related to resputtering may arise due to negative ions with consequent stoichiometry change.

Initially this problem was solved using targets with appropriate composition, out of stoichiometry. Later proper techniques and targets commercially available from companies like Saes Getters Milano, have been developed to minimize the bombardment problems on the deposited film keeping the substrates out of the plasma and using the appropriate working pressures. With this process it is possible to produce a film with a correct target composition.

The single target techniques is widely used in Japan both for rare earth-based superconductors and for tellurium-based ones. This technique is now spreading also in Europe and USA.

Pressure control is very important and therefore stable pumping speed and flow control are necessary to guarantee the final results.

4. Laser ablation

A new technique now being developed uses a target of the required composition, bombarded by a high energy laser (pulsed at 1 or 2 joule for some nanoseconds) and the results seem to be very promising. This technique is relatively simple and reproduces very well the structure and composition of the target; it has been used successfully on rare earth-based superconductors and is now being optimized on Ta and Bi compounds. Today some companies can offer deposition chambers with suitable target manipulators and windows that can accept a laser beam, as well as heated sample holders, but the results of this technique have not been proved yet; therefore no process details or suggestions can be given.

5. Systems configuration

Superconductivity researches are still involving very new techniques which depend on several and different variables but a very important one is the "quality" of the vacuum which must be assured by UHV technology. Vacuum quality is needed during the whole process and therefore the system has to be based on a multichambers configuration concept which assures a complete *in situ* clean process using load-lock and transfer mechanism devices. A 10^{-12} torr base pressure which assures the best process environment is achieved with a Varian multi-chambers configuration system.