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Two-zone furnace used to grow Tl-Ba-Ca-Cu-O films

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In this work, results are shown concerning the properties of two-zone reaction chamber used to grow Tl-Ba-Ca-Cu/Ag thin films prepared by spray pyrolysis. The advantage of using the two-zone thallination furnace is a better control of thallium oxide pTl_2O and oxygen pO_2 partial pressures. The sintering and thallination conditions have not been optimized yet.

1. Introduction

The main problem for preparing Tl-based films from deposited Ba-Ca-Cu-O precursors is the thallination step, in which thallium diffuses into the precursor creating thus the Tl-Ba-Ca-Cu-O superconductor. Because of high volatility of thallium at high processing temperatures, the final superconducting and structural properties of synthesized films depend very much upon the procedure by which the thallium vapor is transported from the Tl-source to the film surface.

The needed induced partial pressure of Tl_2O may be controled in a very fine way by regulating the temperature of the Tl-source and the amount of the initial Tl-content in the source. Another parameter, very important for a proper thallination is the time of the whole heat treatment.

Usually, to thallinate precursor Ba-Ca-Cu-O films, a one-zone thallination chamber is used, in which the precursor and the Tl-source are at the same temperature during the whole thallination. An advantage of such a arrangement is that the transport of Tl-vapor is relatively easy because the precursor film is in a direct contact with the Tl-source (usually a crude Tl-Ba-Ca-Cu-O pellet). A disadvantage is, that both, the sample and the source are at the same processing temperature, however, the temperature needed to develop the required partial pressure pTl_2O is lower by about 100^{o}C (around 750^{o}C) than that needed to sinter the samples (around 850^{o}C).

The situation is improved by using a 2-zone furnace, in which the Tl-source and the precursor film are two different temperatures which is more close to the required optimum state [1,2].It is a purpose of this work to show some preliminary results describing basic properties and behavior of such equipment.

2. Experimental

Precursor Ba-Ca-Cu-O films were deposited on Ag substrates from an aerosol by the spray pyrolysis technique using a solution of acetylacetonate of Ba, Ca and Cu dissolved in N-dimethylformamide with 0.01M concentration. The chemical composition of the solution was adjusted in a such way that the metal cation ratio Ba:Ca:Cu in the deposited precursor films was 2:2:3 according to the EDAX analysis. The aerosol was generated by a commercial ultrasonic nebulizer operating at 0.8 MHz.

The two-zone furnace was made out of four sections and separated into two chambers by a thermal insulating wall as shown in Fig.1. Each set of resistance wire has an independent electronic control system which allows not only to sustain two thermal regions at different temperatures but also to extend the length of thermal plateau of each region. In order to maintain the pTl_2O pressure as constant as possible, three axial quartz tube chambers are used inside the furnace, Fig. 1. The outside one serves like a thermalizator, whereas the central one is closed and connected to an external source of oxygen and a residuum thallium vapor trap. The inner chamber is partially closed and here it is where actually the thallination process takes place.

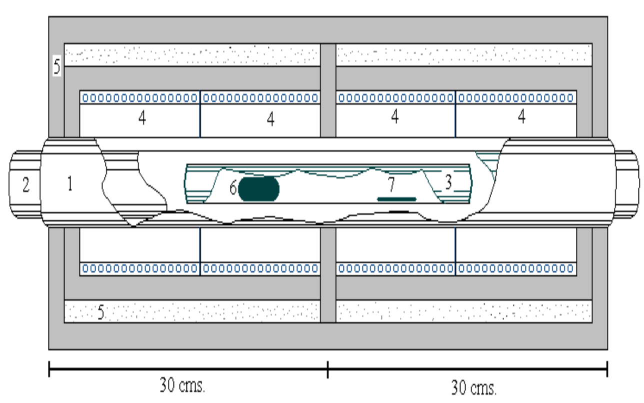

Fig. 1. Schematic diagram of the built two-zone furnace. 1, 2, 3-quartz tube chambers, 4-heat windings, 5-refractory ceramic fiber, 6-thallium source, 7-sample.

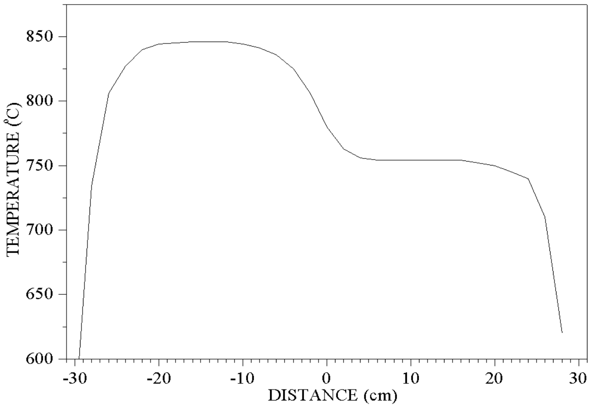

Fig. 2. Temperature profile of the built two-zone furnace.

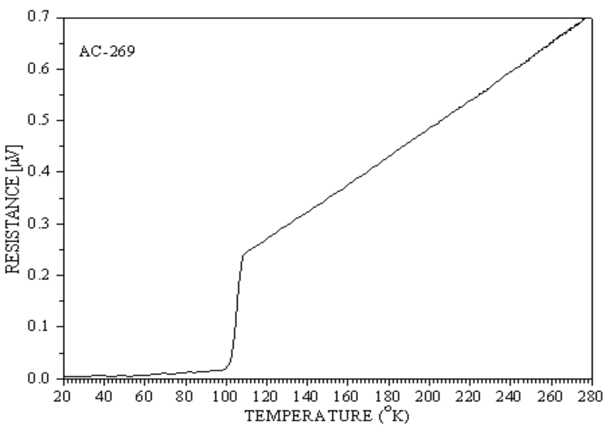

Fig. 3. Resistance v.s. temperature measurement of the sample AC-269.

As a thallium source we suppose to use either crude Tl-Ba-Ca-Cu-O pellet, or directly Tl_2O_3 powder. The thallination will be performed with one chamber at about 850^{o}C and another one at about 750^{o}C with the time of the heat treatment being about 10 to 100 min.

3. Results and discussion

The temperature profile of the built two-zone furnace is shown in Fig. 2. Under the criterium of 1^{o}C deviation from the programmed/monitored temperature value which is within the measurement error, the thermal plateau on axis of both parts is larger than 10 cm. This length is maintained even when 250^{o}C difference between the two chambers exists. The maximum variation in time is about ±1.5^{o}C and it is limited mainly by the type of the temperature controller.

4. Conclusion

A two-zone furnace has been built which enable us to process thermally Ba-Ca-Cu-O film precursors at two different temperatures, one is needed to induce an optimal partial pressure of thallium vapor ($p$$Tl_2O$), and the other is needed to sinter Ba-Ca-Cu-O precursors and to grow superconducting Tl-Ba-Ca-Cu-O films.

The difference between these two required temperatures is about 100^{o}C.Up to now, we were using the one-zone furnace, where both, the thallium source as well as the thallinated sample were on the same temperature (an example of the resistance v.s. temperature dependance of Tl-based film prepared in this type of reactor may be seen in Fig. 3).

We expect that by using the two-zone furnace to grow Tl-based films, we will be able to prepare phase pure films either of 1223, 2212 or 2223 Tl-system which are of primary interest for us.

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