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A Novel Radiative Substrate Heater

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Abstract

In this work, we have developed a new low cost radiative substrate heater for deposition of high-T_c superconducting thin films. It has all the features required for the preparation of high quality superconducting thin films. It is possible to reach ∼800 °C substrate temperature with it by using only one 250 W halogen projector bulb.

Key Words: Substrate heater, Magnetron sputtering, $YBa_2Cu_3O_{7-\delta}$ thin film.

1. Introduction

For deposition of high-T_c superconducting thin films, there are many parameters that must be controlled. One of them is the substrate temperature [1]. Small changes on the substrate temperature result in a significant variability of film properties. It is known that for the in-situ growth of high-$T_c$superconducting thin films, substrates must be heated to temperatures between 600 °C and 800 °C. Many of the highest quality films reported have been grown at temperatures between 700 °C and 770 °C in low pressure ($\leq$ 1 mbar) oxygen atmosphere [2, 3]. Thus the performance of the heater in oxygen atmosphere is an important parameter. These necessities lead to the requirement for a substrate heater, which supplies elevated temperatures, either in vacuum or oxidizing atmospheres.

There are many types of heater design in the literature for the deposition of high-T_c superconducting thin films [4, 5]. The problems with heaters that supply resistive or radiative heating are the short lifetime of the heating element, inhomogeneous heating of substrate, unsuitability for high pressure sputtering and necessity of high power.

In this paper, we describe the construction and the performance of the heater that operates at temperatures above 750 °C with power of 250 W.

2. Heater Construction

A 250 W halogen bulb* was used as the heating source. The bulb was installed into a paraboloid-shaped cavity inside a brass block. In order to increase the reflectance of the cavity, the inner surface was highly polished, then plated with Ni-Cr alloy via electrolysis. Thus the background heating in the deposition chamber was reduced to a negligible value. Since the reflectance of the cavity decreases as the surface

*PHILIPS Projection Lamp, 77485, 24 V, 250 W.

temperature of the cavity increases, a closed cycle water-cooling system was designed and implemented to keep the cavity surface about room temperature while the substrate temperature is kept around 800 °C.

The halogen bulb is placed on the fundamental axis of the parabolic cavity, which had a 5 mm focal length. For maximum power transmission, the bulb must be positioned such that its filament is at the focus of the cavity. For this purpose a bulb holder was designed as shown in Figure 1. It is a hollow cylindrical brass which can be screwed up and down in the brass block. The electrical connections of the bulb were done via this part. The bulb leads are connected to the 2 mm thick copper wires by means of crimped copper tubing. The space between the cylinder and electrical feedthroughs filled with high temperature air-set

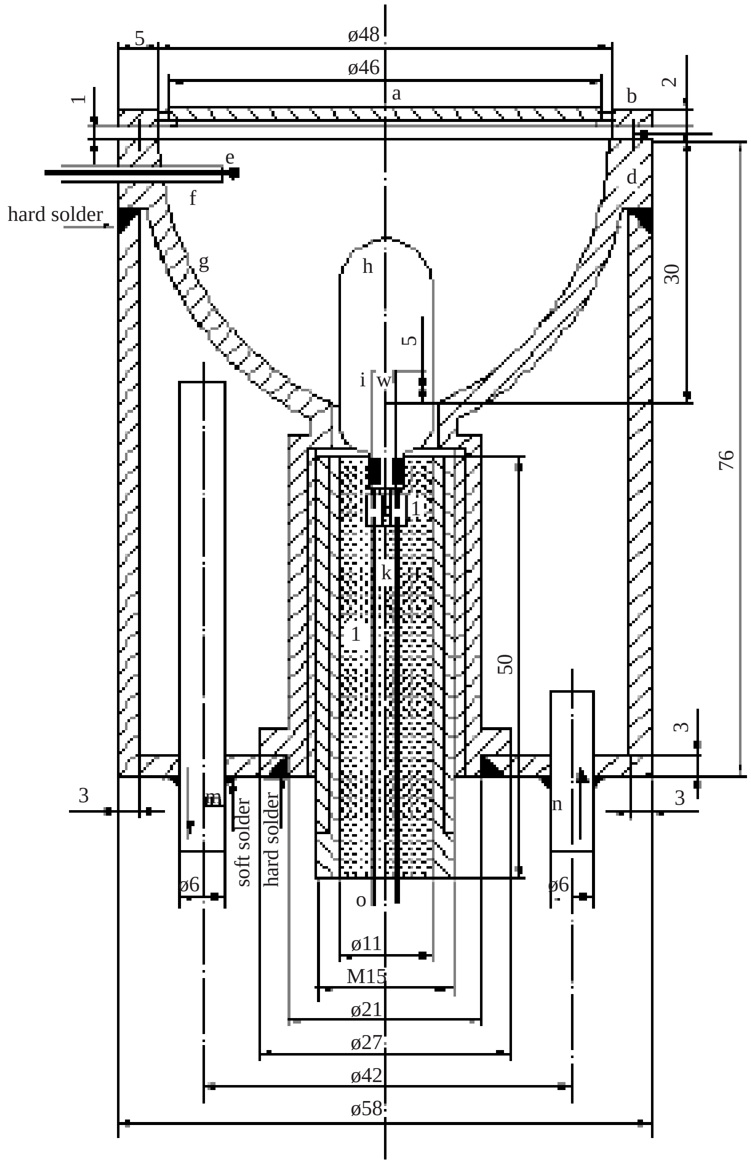

Figure 1. Block diagram of the heater (all dimensions are in millimeters): a) Ni Holder; b) Stainless steel ring; c) Four Ni-Cr wire supports; d) Brass block; e) K-type (chromel-alumel) thermocouple; f) Ceramic tube; g) Ni-Cr plated parabolic cavity; h) 250 W halogen bulb; i) Bulb filament at the focus of the paraboloid cavity; j) Crimped copper tubing; k) Copper wires; l) Cement filled bulb holder; m) Cooling water outlet; n) Cooling water inlet; o) Electrical feedthroughs.

cement[†]. The holder was baked in a box furnace at ~350 °C for 24 hours to dry and degas the cement. As the cement is a good heat conductor (as well as an excellent electrical insulator), it effectively aides in the cooling of the bulb pins, enhancing the lifetime of the bulb. As shown in Figure 1, there is a hole in which a thermocouple is placed to monitor the deposition temperature indirectly. So that this thermocouple can be used to measure the substrate temperature, it must be calibrated against another thermocouple, which measures the substrate temperature directly, prior to the deposition process.

3. Performance

Test were carried out to characterize the performance of the heater. Heater temperature was controlled by a commercial temperature controller[‡] to within an accuracy of ±2 °C. A cold junction-compensated K-type (chromel-alumel) thermocouple was used to measure the heater temperature. The thermocouple was bonded on the center of the Ni holder with silver paste. As could be seen in Figure 2, the dependence of the temperature to the power for two extreme pressures does not play a restrictive role in the temperature range between 400 °C and 800 °C. This interval is important for high-T_c superconducting thin film preparation. In addition to the deposition process at about 750 °C, there is also the post annealing process at 500 °C, at which the heater is also expected to be effective. Temperature gradient on the substrate is another important parameter that affects the quality of the samples. In order to examine the variation of the temperature along the holder surface, we have performed some experiments and obtained the results shown in Figure 3. During these experiments, the thermocouple was bonded at different locations on a Ni holder with silver paste. One can easily estimate from Figure 3 that in the area of 10310 mm^2 at the centre there is approximately 10 °C temperature variation. Hence the heater performs with an acceptable temperature variation over the width of the holder.

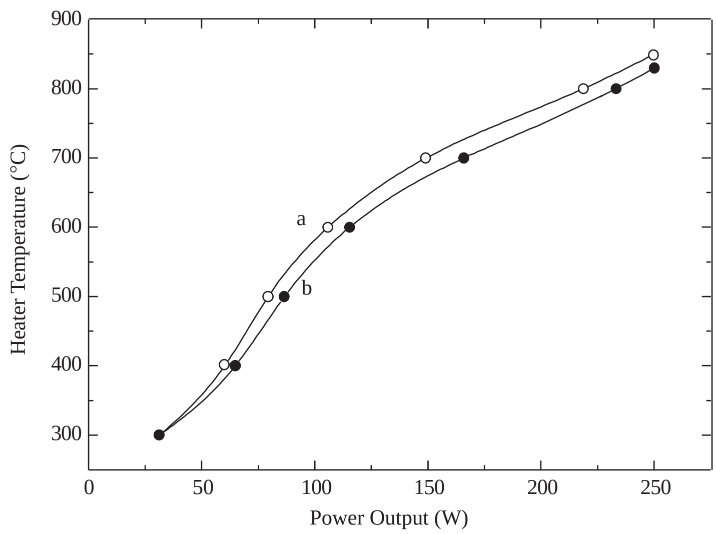

Figure 2. Heater surface temperature versus applied power to the filament. a) Ar + O_2 gas mixture pressure is 1 μTorr; b) Ar + O_2 gas mixture pressure is 200 mTorr. The lines are for the eye.

†Omegabond 400, High Temperature Air Set Cement. [thermal conductivity: 11 Btu·in/ft^2·hr·°F; Volume Resistivity: 10^8–10^9 Ω·cm (at 21 °C) and 10^5–10^6Ω·cm (at 399 °C)]. (Omega Engineering, Inc., One Omega Drive, P.O. Box 4047, Stamford, CT 06907-0047 USA.)

‡ABB COMMANDER 300, Universal Process Controller. (ABB Kent-Taylor Ltd., St. Neots, Cambs., England, PE19 3EU, UK.)

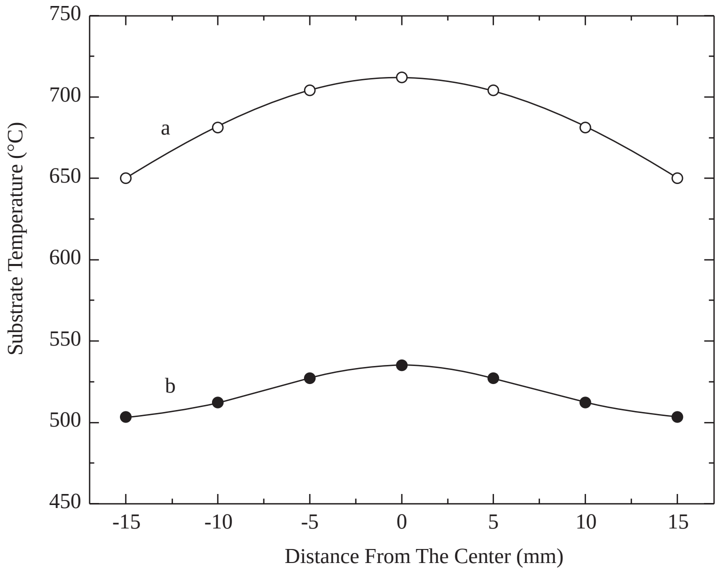

Figure 3. Variation of the temperature on the holder surface at Ar + O_2 gas mixture pressure of 1 μTorr. a) Set temperature is 700 °C; b) Set temperature is 525 °C. The lines are for the eye.

We have been using this substrate heater to prepare $YBa_2Cu_3O_{7-\delta}$ thin films in a magnetron sputtering system. For the production of thin films, we employed three different heating procedures. The first is the degassing and baking of the bonded substrate. For this purpose, the substrate is heated to 200 °C, the rise occurring within 15 minutes; kept at this temperature for 30 minutes; and then cooled down to room temperature in vacuum with pressure of 1 μTorr. In the second heat treatment, a three-step profile is followed. In the first step, the substrate is heated to 500 °C within 25 minutes. In the second step, the substrate is soaked at 500 °C for 15 minutes. For the last step, the substrate is heated to the desired deposition temperature at a 15 °C/min heating rate. Following these steps, the deposition is begun and continues over a period of 2 hours. During these processes the Ar + O_2 gas mixture is kept at a pressure 50 mTorr. Post-annealing at 500 °C for an hour at 760 mTorr O_2 completes the film preparation. At the end of post-annealing the heater is turned off.

Using the above described growth process, films fabricated using the parameters in Table were found to have a transition temperature of ~87 K and the resistance curve shown in Figure 4. The same halogen bulb was used during the characterization tests for the heater and film fabrication, and thus was used for more than 100 hours of lamp time.

Table. The optimized sputtering parameters for $YBa_2Cu_3O_{7-\delta}$ thin film fabrication.

Deposition temperature	730 °C
O_2 partial pressure	10 mTorr
Ar partial pressure	40 mTorr
R.F. power	50 W
Annealing temperature	500 °C
Post-annealing time	1 hour at 1 atm O_2 pressure

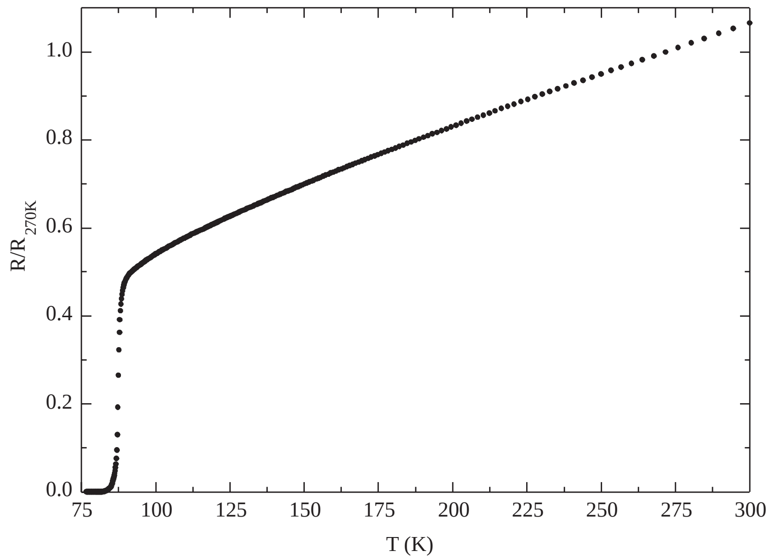

Figure 4. Normalized electrical resistance versus temperature for the prepared $YBa_2Cu_3O_{7-\delta}$ thin film.

4. Results

As a consequence of our investigations, we have designed and constructed a new low cost substrate heater. The results demonstrate that it is possible to reach to the temperatures necessary for the growth of superconducting thin films. As seen from the Figure 4, high quality $YBa_2Cu_3O_{7-\delta}$ thin films can be prepared with this substrate heater.

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