Compact substrate heater for use in an oxidizing atmosphere

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A compact heater, designed for the deposition of thin films at high temperatures in an oxidizing atmosphere or in vacuum, is described. The heater, including an oxygen-resistant case and the attached substrate, can be loaded into a vacuum deposition chamber through a small-diameter load-lock port, and will operate in 0–1 atm of oxygen at temperatures up to at least 800 °C. Heat is generated resistively, and the substrates are heated directly by thermal conduction. The heater was built specifically to heat substrates during the growth of high-temperature superconducting thin films.

I. BACKGROUND

The vacuum deposition of high-transition-temperature (high-T_c) superconducting thin films puts extraordinary constraints on the operation of vacuum system heaters. Because they are oxides, these ceramic superconductors, such as $YBa_2Cu_3O_7$, cannot be grown *in situ* in a true high-vacuum environment. Oxygen must be present during growth to form these compounds in the proper crystalline phase. Oxygen partial pressures up to 0.5 Torr, and temperatures in excess of 700 °C, are typical deposition conditions used for the growth of these materials.[1,2] Following deposition, the films are usually cooled from the growth temperature at oxygen pressures of a few hundred Torr. An anneal at 400–500 °C in 1 atm of oxygen completes the process. Oxygen partial pressures over 10^{-4} Torr usually cause severe problems for most heater materials heated to such high temperatures. The heater described here has been designed to withstand such extremes in temperature and oxygen partial pressure.

A major advantage of this heater is that heat is generated resistively, and substrates are heated directly by thermal conduction. In many vacuum-chamber heaters, heat is transferred to the substrate by infrared radiation. The source of infrared radiation may be a sealed quartz lamp,[3] or heated tungsten or platinum windings,[4] for example. A major disadvantage with such a design, especially for the growth of high-T_c superconductors, is that most of the substrates of choice are transparent to visible light, and poor absorbers of infrared radiation. Without an intermediate heat absorber, this method can result in very inefficient heating of the substrates, and also very large errors in thermometry, especially if one employs pyrometric techniques.[5]

II. DESIGN CONSIDERATIONS

A sketch of the heater is included as Fig. 1. The heater is easily scaled in size to accommodate any vacuum system. The rather compact design shown in Fig. 1 represents the implementation at the authors' laboratory, intended for use in an UHV ion-beam sputter-deposition system with a load-lock entry system. A load-lock port, while minimizing incursions into the UHV environment, imposes severe constraints on a heater system which is brought in and out with the substrate. In particular, the size of the heater is usually severely restricted, and the electrical contacts and thermometry to the heater assembly must be made and broken within the evacuated chamber, that is, the connections cannot be hard wired.

The materials chosen for the components of this heater, as well as some design features, are key to its operation at high temperatures and in oxygen. The main body of the heater, a cylinder about 3.2 cm in diameter and 1.1 cm long, is fabricated from boron nitride (BN). This synthetic material, an analogue of graphite, is commercially available from the Carborundum Company in several grades. The grade of BN used in this heater is Carborundum's AX05 grade. BN has a unique combination of properties, detailed in Carborundum's product literature, that is critical to the successful operation of the heater.[6] BN has a high thermal conductivity (approximately 100 $W\,m^{-1}\,K^{-1}$ at 25 °C and 25 $W\,m^{-1}\,K^{-1}$ at 1500 °C for AX05 grade) so that, when heated, the BN main body readily conducts heat to the substrate. BN is also electrically insulating ($\rho > 10^{14}$ Ω cm at 25 °C), so that heater wires wrapped in grooves around the main body are electrically insulated from one another. BN is also resistant to oxidation, being capable of withstanding temperatures of approximately 850 °C in 1 atm of oxygen (up to 900 °C in 10^{-10} Torr of N_2). Finally, although BN is a soft material (Knoop hardness of 3.4–4.9 kg/mm^2), it is easily machined, such as by drilling holes, tapping threads, and cutting grooves, allowing other components to be fixtured to it.

The heating element consists of 18 gauge (1-mm-diameter) Kanthal AF or A-1 wire that is wound in a spiral groove cut in the outside of the BN main body. Kanthal, an alloy of iron, chromium, and aluminum, is available commercially from Kanthal Corporation.[7] Two features of Kanthal are noteworthy for this application. First, Kanthal wire has a high resistivity ($\rho = 145$ $\mu\Omega$ cm at 20 °C and is not very temperature dependent) so that

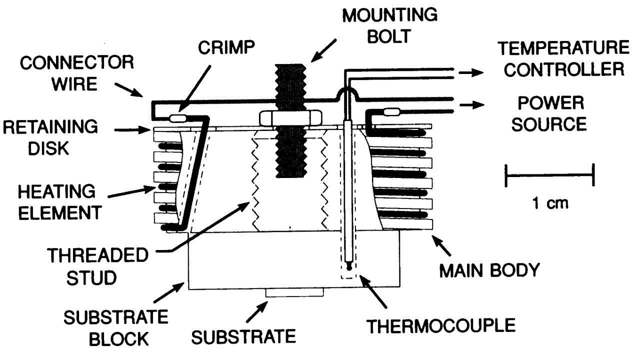

FIG. 1. Cut-away drawing of the compact substrate heater. The scale shown is approximate.

only short lengths are necessary to make a suitable heating element. Second, Kanthal forms a protective natural oxide barrier so that it can operate effectively in an oxygen atmosphere. In fact, when used in a reducing atmosphere (such as N_2 or Ar), the manufacturer recommends occasionally heat treating the wire at 1050 °C in air for 7–10 h to maintain the protective oxide layer.[8] Such a preoxidizing treatment also prevents the wire from fusing when heated in a vacuum. The heating element for the heater described here was preoxidized by operating the heater in air as prescribed. The substrate block temperature during this treatment was 760 °C (measured by an internal thermocouple), while the Kanthal-wire heating element temperature was 1100 °C (measured by a dual wavelength optical pyrometer).

The substrate block is fabricated from Haynes alloy 214. This alloy of mainly nickel, chromium, aluminum, and iron is commercially available from Haynes International, Inc.[9] Like Kanthal, the Haynes alloy also develops a protective oxide layer when heated in oxygen, thus being very resistant to the degradation effects of oxidation. As illustrated in Fig. 1, the substrate block is machined as a short cylinder with a threaded stud. The stud is screwed into a threaded hole in the BN body as shown. A retaining disk is also fabricated from Haynes alloy and serves to reinforce the structure, as the BN is not as strong as the metal components. A nut on the mounting bolt holds everything together. The Kanthal wire on the right-hand side of Fig. 1, shown as coming up in a groove on the edge of the retaining disk, may be secured under this nut.

The key materials used in this design (BN, Kanthal, and Haynes alloy) have been used in other heater designs,[10,11] especially since the advent of high-T_c superconductors. A unique feature of the present design, in addition to the compact size, is that the Haynes alloy substrate block is heated directly via thermal conduction through the BN body. There is no need to electrically insulate or physically separate the block from the heating element with insulators such as quartz, sapphire, or ceramic sheet. The Haynes alloy substrate block threads directly into the electrically insulating, but thermally conducting, BN.

A thermocouple is fed through holes drilled in both the BN and the Haynes alloy substrate block. This permits measurement of the block temperature, which can be used in a feedback loop with the heater power supply to control the temperature. The mounting bolt at the top of the heater is used to fixture the whole assembly in the vacuum system. All metal parts in the heater, including any required for fixturing, are fabricated out of Haynes alloy. An alumina disk (not shown in the figure) may be used to support the assembly and insulate it, both electrically and thermally, from the heater casing (not shown). Holes in the alumina disk can accommodate heater and thermocouple connectors which mate to similar connectors in the vacuum chamber. The heater casing acts as a heat shield, and may also contain additional radiation reflectors. In the present design, short pieces of 1-mm-diam platinum wire are used to electrically link the end of the Kanthal wire to the alumina disk connectors. The platinum wire is much more flexible than the Kanthal, and therefore easier to route to the connectors, while at the same time it is much less resistive, and thus does not heat up as much as the Kanthal. The wires are joined by wrapping platinum foil around the overlapping wire ends, and crimping the combination inside a small piece of stainless steel tubing. This mechanical method has proved to be simple and effective. Alternatively, one might spot weld the Kanthal and platinum wires together noting, however, that these materials are very different in their hardness and thermal properties.

The thermal expansion coefficients for the Kanthal and Haynes components are approximately 1.5×10^{-5}/K while that of BN is less than 1×10^{-6}/K. However, since the BN is a rather soft material, it yields readily to the more expansive metal components such as the Haynes sample block threaded into the BN body. The prototype heater assembled as described here has been cycled to approximately 700 °C hundreds of time without ever experiencing a failure due to thermal stresses on the BN.

There are many possible variations to the design described. Aside from different geometries that would be obvious, one could use different materials and still maintain the basic concept. Other alloys besides Kanthal, such as wire made with Haynes alloy, could be used as the heating element. Sheathed nichrome wire could be used,[12] through outgassing might be a problem in an UHV environment, while the large size and bending radius of sheathed nichrome make it difficult to use in a small, compact heater design as described here. Tantalum wire could be used in lower oxygen pressures (up to 5×10^{-4} Torr). Platinum-rhodium alloys could also be used as the heating element. However, their lower resistivities make a small heater difficult to fabricate because it is difficult to achieve high resistance in the windings. The BN heater body could be replaced but with some disadvantages. Other candidate materials would be other high thermal conductivity, low electrical conductivity materials, such as beryllia or aluminum nitride. Machinable ceramics or alumina could be used, though they are difficult to machine and do not have the high thermal conductivity of BN.

Special considerations are required for operation of this heater in a strict UHV environment. First, one of the alternate materials listed above may be preferable to BN

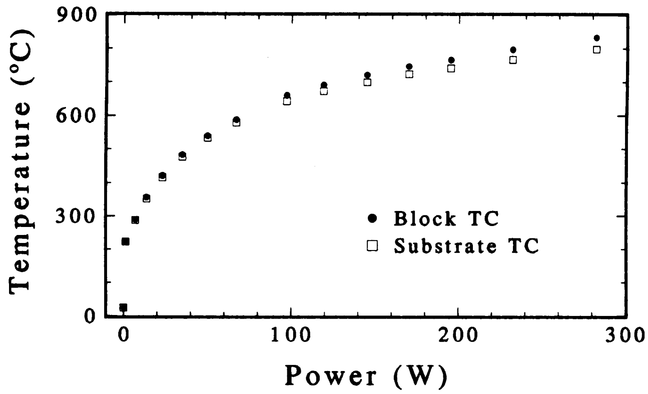

FIG. 2. Temperatures measured with thermocouples bonded to the substrate (substrate TC) and inserted into a hole in the Haynes substrate block (block TC).

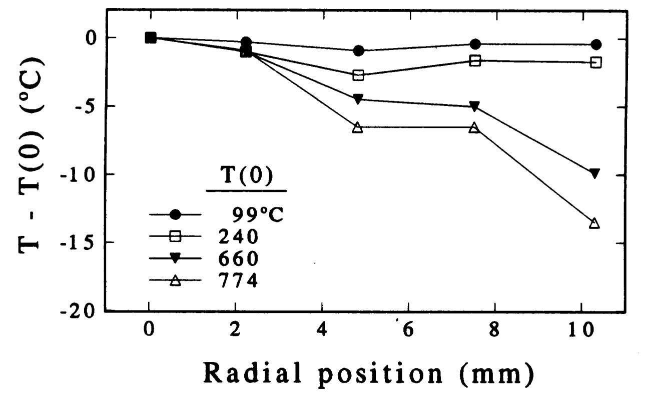

FIG. 3. Temperature profile measured in air across the Haynes substrate block; zero position is the center of the block and $T(0)$ is the temperature at the center.

for use at the highest temperatures in oxygen. At a temperature of about 850 °C in air, BN starts to oxidize, forming B_2O_3. B_2O_3 melts at 450 °C and has a vapor pressure of about 2×10^{-9} Torr at 850 °C.[13] In addition, the phase stability and vapor pressure of BN under the use conditions must be taken into account. As calculated from changes in the Gibbs free energy,[14] BN decomposes into boron and nitrogen at 920 °C in 10^{-10} Torr of N_2, while the BN vapor pressure is much less than the N_2 pressure required for phase stability. A final consideration for UHV environments is the prevention of virtual leaks. Although not shown in Fig. 1, holes and slotted threads can be incorporated into the design to avoid such problems.

III. OPERATION

A dc power supply can be used to drive current, I, through the Kanthal wire having resistance R. Heating power, P, is generated simply by resistive heating: $P=I^2R$. If desired, ac can be supplied instead of dc. The nature of the power source is immaterial. The critical requirements are that sufficient current be supplied to reach the desired operating temperature, and that the current-carrying capacity of the wire not be exceeded. These properties depend on the wire diameter. The heat generated is conducted through the BN by direct thermal conduction to the Haynes substrate block. Substrates, on which the film is to be deposited, can be attached to the Haynes block in a number of ways. The preferred method of attachment is to use silver paint or paste to lightly glue the substrate in place on the Haynes block. The silver paint is baked out at 250–300 °C before loading the substrate into the vacuum chamber, leaving nearly pure silver as the actual attaching medium. Alternatively, the silver paint can be baked out in the vacuum system if outgassing of some organic solvents and binders is acceptable. The substrate is attached very well both mechanically (it never falls off) and thermally (note the small temperature difference between the block and substrate in Fig. 2, described below). Removing the substrate after deposition is very easy due to the softness of the silver. Fixturing clips can also be used to simply hold the substrate against the Haynes block. However, this technique is least efficient in conducting heat to the substrate and results in the greatest uncertainty as to the actual substrate temperature. A variation of this method is to sandwich a thin piece of silver or gold foil between the clip-held substrate and the heater surface.[15]

It is prudent to calibrate such a heater so that the true substrate temperature is known with a minimum uncertainty. The normal working thermometer when using this heater is a thermocouple inserted in a hole drilled into the Haynes alloy substrate block. This relies on the metallic alloy acting as a good blackbody in steady-state radiation balance with the thermocouple. To check this methodology, two additional thermocouples were silver-paint bonded to the Haynes alloy block. One of the additional thermocouples was bonded directly to the block, and the other was bonded to a small 0.5-mm-thick piece of $SrTiO_3$ (a typical substrate material for $YBa_2Cu_3O_7$ films) that was in turn silver-paint bonded to the substrate block. The heater was operated under typical deposition conditions in the vacuum chamber with a total gas pressure of 1 mTorr. The results of the calibration are shown in Fig. 2. The temperatures were measured using chromel-alumel thermocouples. The abscissa is the rms input power supplied by an ac voltage source at 60 Hz. The temperature readings for the thermocouple bonded directly to the substrate block were essentially the same as those for the thermocouple inserted in the drilled hole. The resistance of the short Kanthal heating element used for this calibration was approximately 1 Ω. These temperatures also agree with measurements taken using a two-color optical pyrometer. During normal operation of the heater, the block temperature, and therefore the substrate temperature, are controlled based on readings from the inner block thermocouple. For example, from the calibration for this heater, in order to achieve a substrate temperature of 700 °C, the temperature of the thermocouple inside the Haynes block should be maintained at 722 °C.

To check the temperature uniformity across the small 2.4-cm-diam substrate block, a number of chromel-alumel thermocouples were silver-paint bonded to the block surface at various radial positions. These measurements were performed in air with the heater casing removed. The results are illustrated in Fig. 3, where the temperature profile across the Haynes substrate block is shown for several temperatures.

ACKNOWLEDGMENTS

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