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Cryogenic substrate cooling or substrate heating without vacuum feedthroughs

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It is well known that substrate temperature during thin film deposition can dramatically affect film morphology. Cooled substrates are also useful for making films in metastable phases¹ and for making amorphous films. However, provisions for heating or cooling are often initially omitted from deposition systems because of the added cost and complexity. We describe a simple way of retrofitting systems to allow substrate cooling to 124 K or heating to 130 °C *without the use of any vacuum feedthroughs* and for a modest cost. Our implementation is for bell-jar evaporators, though simple variations will work for virtually any system which pumps to a suitable vacuum in two hours.

It is essential to delay substrate cooling until the deposition chamber is suitably evacuated; otherwise condensation will form, ruining the deposited film. Similarly, it is essential to delay substrate heating to avoid excess oxidation.

Our system includes two copper pieces: a "hot/cold" block and a separate substrate block, which remains near room temperature until desired. The hot/cold block is heated or cooled outside the deposition system. It is then transferred to the deposition system, where it is held magnetically above the substrate block while the chamber is pumped down. At this point the magnetic hold of the hot/cold block is released, it falls into contact with the substrate block, and substrate heating or cooling begins. This type of mechanical control of heavy objects with external magnets has only become practical within the last 15 years, with the advent of high energy product rare earth magnets.²

Tests of several geometries resulted in the final design shown in Fig. 1. The substrate block is a 3.2-mm-thick piece of oxygen-free, high-conductivity (OFHC) copper (width: 5 cm, length: 14 cm). Two OFHC arcs (cut from a used gasket for a 25.4 cm CF flange) are silver soldered to the edges of the block to provide added stiffness.³ This assembly is suspended from convenient support points within the deposition chamber by lengths of 0.8-mm-diam stainless steel wire (Loos and Co., Wire Division, Pomfret CT) which are attached to holes at the ends of the arcs.

The hot/cold block is made from a 10.9 cm × 7.8 cm × 3.8 cm piece of copper, with a shallow hole in the middle of the top surface. A thin-wall stainless tube (1.3 cm o.d., 0.25 mm wall, with vent slots) is seated in this hole. [The difficulty of soldering such a large piece of copper, led us to use Stycast 2850FT epoxy for this joint (Emerson and Cuming, Woburn, MA); using catalyst 43 as the curing agent extends the temperature range well above 150 °C]. The top end of the stain-

less tube is attached to a set of two NdFeB disk magnets, each 12.7 mm thick and 51 mm in diameter (Magnet Sales, Culver City, CA), which are held securely together by their magnetic interaction. The tube is mechanically attached to the magnets, using readily available components: we epoxy a set-screw collar to the end of the tube, then tie four 1.0-mm-diam stainless steel wires around the tube below the collar, and thread them under a hose clamp which is tightened around the magnet. (All stainless hose clamps are available from ABA of America, Rockford, IL.) This arrangement, though unconventional, is easy to setup and works very well. To prevent the magnets from directly contacting the bell jar, a layer of 1.5-mm-thick teflon is epoxied to the top surface of the magnets, extending slightly beyond their diameter. The deposition system we used for testing has a base pressure of 2×10^{-6} Torr when empty, and this same base pressure was achieved during our tests.

For cooling mode, the bell jar, implosion shield, and hot/cold block are setup adjacent to the deposition system as shown in Fig. 2. The hot/cold block assembly is suspended at the top of the bell jar by magnetic attraction to a second pair of disk magnets on the top of the implosion shield. The hot/cold block itself is thus suspended within an open-topped styrofoam container. The magnetic force is easily able to support the 2.9 kg hot/cold block despite the 2 cm magnet separation imposed by the intervening bell jar and implosion shield. (In fact, the hot/cold block can even be retracted after it is dropped, for separations up to 5 cm.)

To minimize condensation on the hot/cold block, air is expelled from the bell jar by flowing He gas from below. The styrofoam container is then filled with liquid nitrogen through a flexible tube attached to a pressurized storage dewar. While the block cools (~15 min), a very light flow of He is sufficient to avoid formation of frost. Then the implosion shield / bell-jar / hot/cold block assembly is lifted free of the liquid nitrogen, lowered over the deposition system, and pumping is commenced. At this point, the hot/cold block is suspended above the substrate block by the magnets. Inevitably, a small amount of frost does form on the hot/cold block (though none on the substrate block) as the chamber is pumped down. However, the vapor pressure of ice⁴ at our base temperature of 124 K is only 10^{-11} Torr.

Once the desired vacuum level is achieved, the exterior magnets are removed, allowing the hot/cold block to drop onto the substrate block and begin cooling it. If desired, after deposition, the hot/cold block can be retracted by replacing the exterior magnets on the top of the implosion shield.

For substrate heating, a similar procedure is used, except

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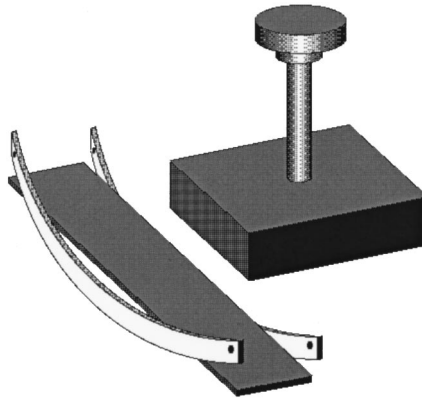


FIG. 1. Drawing of substrate block (left) and hot/cold block (right). Substrates (not shown) are mounted on the bottom of the substrate block, which hangs inside the deposition chamber, supported by four stainless steel wires attached through the holes at the ends of the stiffening arcs shown here. The hot/cold block is attached to a pair of strong permanent magnets by a thin-wall stainless tube, as shown. The block is heated or cooled outside the deposition chamber, then loaded into the chamber and suspended magnetically above the substrate block. Once the chamber has been pumped down, the magnetic hold is released, and heat transfer between the hot/cold block and the substrate block begins.

the hot/cold block is externally heated by resting on a copper plate which is brought to the desired temperature by a hot plate. Our thermometry limited us to tests below room temperature. However, there should be no difficulty in heating the block to 150 °C, and even higher if the joint between the block and the stainless tube were made with silver solder. (Although the magnets are somewhat isolated from the heat applied to the block, magnets made from SmCo would be better for such high temperatures since NdFeB loses magnetization if heated to 150 °C.)

To promote thermal contact between hot/cold block and the substrate block, we place three 6 mm lengths of 1-mm-diam indium wire in a triangular pattern on the sample block

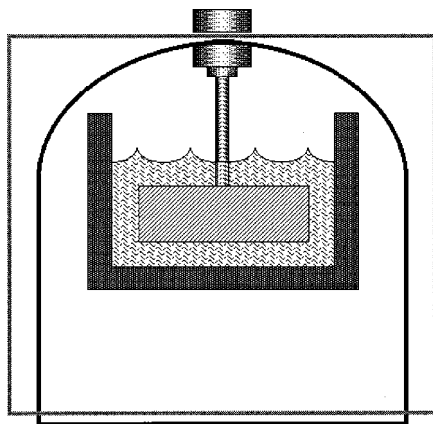


FIG. 2. Schematic cross-sectional view of setup for cooling of hot/cold block. The outermost gray line represents the implosion shield, the black line represents the bell jar, and the dark stippled area represents a styrofoam container filled with liquid nitrogen. The block is suspended within the nitrogen by attraction to an external pair of magnets placed on the top of the implosion shield. The bell jar is filled with helium gas during cooling to minimize frost formation.

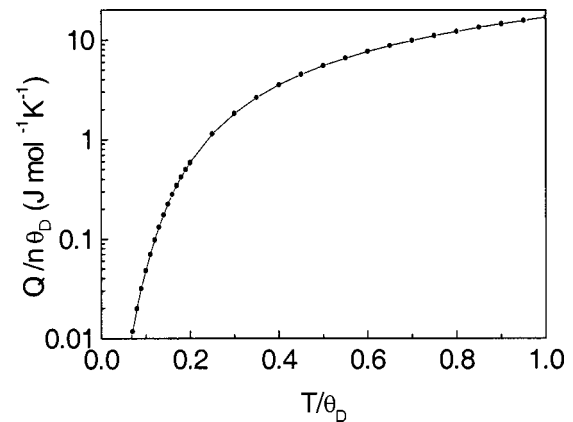


FIG. 3. Graph of molar thermal energy content (normalized by dividing by θ_D) vs normalized temperature. Dots show the result of numerical evaluation of Eqs. (1) and (2); the line is a guide to the eye. This graph allows easy calculation of the result of thermal equilibration for substances which are well described by the Debye theory.

before pumpdown. When the hot/cold block falls, the indium is flattened, creating macroscopic contact areas. The substrate block reaches a base temperature of 124 K within 25 min of dropping the hot/cold block, and remains close to this temperature for at least 1 h. (We have made some measurements with a more complex system, which does not use the indium; instead, short inward-pointing arcs of copper are added to the substrate block to serve as gripping “claws.” The ends of the hot/cold block are slightly bevelled so that it wedges into place when dropped between the claws. This more complex arrangement provides only marginally better performance than the indium wire method.)

The theoretical base temperature is not as low as one might assume based on a simple weighted average of the initial temperatures of the two blocks, since the heat capacity decreases at low temperatures. For a quantitative comparison between the experimental base temperature and theoretical expectations, we begin by calculating the initial thermal energy content of each block, relative to $T=0$. This can be done by integrating the heat capacity:

$$Q = n \int_0^T C(T') dT', \quad (1)$$

where C is the molar heat capacity and n is the number of moles. (Here, we ignore the distinction between C_p and C_v , which is small for solids.) For copper, the heat capacity is well described by the Debye model:⁵

$$C = 9R \left(\frac{T}{\theta_D} \right)^3 \int_0^{\theta_D/T} \frac{x^4 e^x}{(e^x - 1)^2} dx, \quad (2)$$

where $R = 8.314 \text{ J K}^{-1} \text{ mol}^{-1}$ is the universal gas constant, and θ_D is the Debye temperature ($\theta_D \approx 343 \text{ K}$ for Cu.) Thus, Q can be evaluated numerically for the initial hot/cold block temperature of 77.4 K ($Q_{\text{hot/cold}} = 1.28 \times 10^4 \text{ J}$) and the initial substrate block temperature of 293 K ($Q_{\text{substrate}} = 2.11 \times 10^4 \text{ J}$).

For the convenience of other researchers designing similar systems, a graph of the results of the numerical evaluation of Eq. (1) is shown in Fig. 3. Note that the result of dividing Q by θ_D is a universal curve which describes the molar thermal energy content as a function of temperature for all materials which can be modelled with the Debye theory.

To these initial energies, we add the energy transferred to the blocks during the pump and cool down, which totals approximately 1.1×10^4 J (about 60% from radiation, 25% from conduction down the magnet support tube, and 10% from convection during the 30 s time interval between lifting the hot/cold block out of the liquid nitrogen bath and initiating pump down). The heat leak down the support wires is negligible, and heat flow through the gas after pumping has started is small, as is the thermal energy added during a typical evaporation. Thus, when base temperature is achieved (after 12 min of pumping prior to dropping and 25 min of additional pumping and cool down), the total energy content of the substrate block and hot/cold block is approximately 4.5×10^4 J.

This value can be substituted into Eq. (1), and the equation evaluated iteratively (or the solution determined graphically using Fig. 3) to find the corresponding temperature of 120 K, in good agreement with the experimental base temperature of 124 K. The most important design criterion is a maximum ratio of the mass of the hot/cold block to that of the substrate block, so as to provide a small total heat content spread throughout a large mass.

¹See, for example, K. D. Leedy and J. M. Rigsbee, *J. Vac. Sci. Technol. A* **14**, 2202 (1996).

²For a review of these materials, see J. F. Herbst, *Rev. Mod. Phys.* **63**, 819 (1991).

³We have also tested designs which omitted the stiffening arcs. In all designs, the hot/cold block must be supported by the substrate block, which necessarily flexes under the load. The stiffening arcs minimize this flexure, and so allow the use of a thin substrate block, which improves the base temperature, as discussed in the conclusion.

⁴R. J. Donnelly, "Cryogenics" in *A Physicist's Desk Reference*, 2nd ed. (American Institute of Physics, New York, 1989), p. 126.

⁵See, for example, M. A. Omar, *Elementary Solid State Physics* (Addison-Wesley, Reading, Ma, 1975), p. 84.