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R. Torii

S. R. Bandler

Tamar More
University of Portland, more@up.edu

F. S. Porter

R. E. Lanou

See next page for additional authors

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Authors
R. Torii, S. R. Bandler, Tamar More, F. S. Porter, R. E. Lanou, H.J. Maris, and G. M. Seidel

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Removal of superfluid helium films from surfaces below 0.1 K

R. Torii, S. R. Bandler, T. More, F. S. Porter, R. E. Lanou, H. J. Maris, and G. M. Seidel
Department of Physics, Brown University, Providence, Rhode Island 02912

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We have constructed an apparatus that is able to maintain a helium-free surface at low temperature ($T \leqslant 0.1$ K) in a cell containing superfluid helium. We discuss the considerations involved in the design of this device, and describe tests that we have made to confirm that a film-free surface has been produced.

I. INTRODUCTION

When superfluid helium is placed in a beaker a film quickly climbs up the beaker walls above the level of the liquid bath.[1] The film is held to the surface by van der Waals' forces and has a thickness d under saturated conditions that depends weakly on the height h above the bath according to the relation $d \sim h^{-1/3}$. The film thickness depends on the wall material, but for most substances the film is roughly 250-Å thick at a height of 10 cm. Under the influence of a driving force the film can flow up to a critical velocity, which is usually ~ 50 cm s^{-1}.

In some low-temperature experiments the existence of the film has been used to advantage. In studies of spin-polarized atomic hydrogen,[2] e.g., the film keeps the hydrogen from making contact with a solid surface to which it would be strongly attracted. However, in other cases the film may cause problems. When temperature gradients are essential in the presence of a film, the film flow, and the associated evaporation and reflux of gas atoms may introduce a large unwanted heat transport.

We have recently proposed a new experimental method for the detection of low-energy solar neutrinos.[3,4] For this method to have the desired sensitivity it is essential that the film be eliminated. In the experiment a neutrino deposits energy into a large bath of superfluid helium which is at a temperature below 0.1 K. As a result of this energy deposit helium atoms are evaporated from the surface of the liquid bath. These atoms are adsorbed onto a calorimeter, consisting of a thin large-area silicon wafer with an attached thermistor, and the number of atoms is determined from the temperature rise. Since the silicon must be physically suspended inside an enclosure containing liquid helium and must be thermally attached to a heat sink, preferably the mixing chamber of a dilution refrigerator, the calorimeter would normally be covered with film. But the film at low temperature has a heat capacity very much larger than that of the silicon. The heat capacity of the film is primarily due to the surface excitations, ripplons, and per unit area is given by[5]

$$C_r = 0.404 \frac{k^{7/3} T^{4/3} \rho^{2/3}}{\hbar^{3/4} \alpha^{2/3}}, \tag{1}$$

where k is Boltzmann's constant, T the temperature, ρ the density, and α the surface tension. At low temperatures this is numerically

$$C_r = 2.06 \times 10^{-9} T^{4/3} \text{ J K}^{-1} \text{ cm}^{-2}. \tag{2}$$

The heat capacity of pure silicon per unit volume is

$$C_s = \frac{1944 T^3}{V_m \Theta^3}, \tag{3}$$

where V_m is the molar volume and Θ is the Debye temperature which for silicon is 640 K. For a Si wafer of thickness 0.020 cm, the heat capacity C_s per unit area is

$$C_s = 1.28 \times 10^{-8} T^3 \text{ J K}^{-1} \text{ cm}^{-2}. \tag{4}$$

A comparison of Eqs. (2) and (4) shows that with helium film on both sides of a 0.02-cm-thick wafer at a temperature of 0.1 K the ripplon heat capacity is 15 times larger than that of the silicon, and at 0.01 K it is 700 times larger. The temperature change δT is related to the energy δE deposited in the structure by $\delta T = \delta E / C$, where C is the total heat capacity. It is clear that a substantial increase in sensitivity of the measurement of small energy deposits can be achieved by removing the helium film.

II. APPARATUS

The most direct method of removing a superfluid helium film at low temperatures from an object connected to a liquid reservoir is to heat the intervening surface so as to evaporate the helium film. This must be accomplished while keeping the object, in our case the silicon wafer/calorimeter, at low temperature, below 0.05 K. Since the evaporation rate from the heated intermediate surface must equal the rate at which the film flows onto it from the bath, the temperature of the evaporating surface must be typically in the range around 0.5 K. Also, the evaporating helium atoms cannot be allowed to recondense on the film-free object, i.e., the wafer must be isolated spatially as well as thermally from the evaporation section, but at the same time must be physically connected to it.

The means by which this separation is achieved is illustrated schematically in Fig. 1. The silicon wafer is suspended above the helium bath by a copper rod that runs through the top of the experimental cell and is attached to the mixing chamber of a dilution refrigerator. Superfluid film that flows from the top of the cell towards the wafer is intercepted by a stainless-steel tube surrounding the copper rod. The flowing film is thus forced onto a large heated area that serves as the evaporator. This area consists of a horizontal copper plate and a section of vertical tube also made of copper. The evaporator is heated by a coil wound onto the vertical copper tube. The evaporated helium at-

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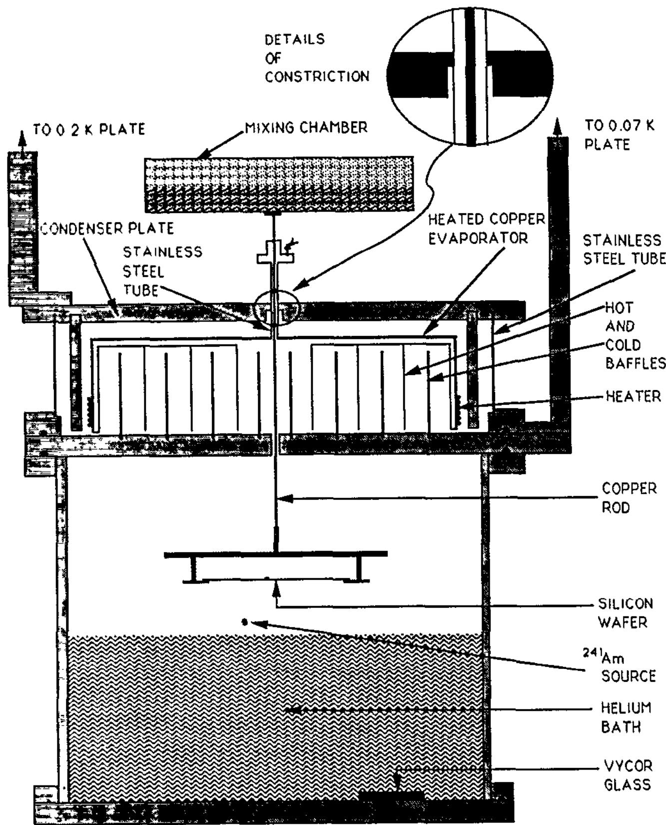

FIG. 1. Schematic design of the film burner and the experimental cell (not to scale). Only one of the heat links to the 0.07- and 0.2-K plates is shown. The blowup shows the limiting circumference restricting the film flow onto the evaporator.

oms recondense primarily on the top of the cell (the condenser), and it is from this region that the large heat of recondensation must be removed. Gas atoms are prevented from making their way to the film-free wafer by a series of interleaved baffles at high and low temperatures that we describe in detail later.

The removal of film by evaporation while restricting the gas flow through recondensation is similar in concept to the design of a film-flow suppressor developed by Wheatley and collaborators[6,7] to decrease the ^{4}He component in the vapor stream from a still of a dilution refrigerator. In the present case, however, there are more stringent conditions on what happens to the refluxing gas. Also, the temperature of the surfaces kept free of film must be cooled to low temperature.

The rate of volume flow of the film is given by

$$\dot{V}=v\,dS, \tag{5}$$

where v is the critical velocity of 50 cm s^{-1}, d is the film thickness ~250 Å, and S is the minimum perimeter that limits film flow in the vertical direction. In this design the restricting circumference was created by a 0.50-cm i.d. section of the condenser plate of the cell, shown in the blowup of Fig. 1. The diameter could not easily be made smaller given the space required for the copper cooling rod and for the electrical leads to the thermistor and other components.

It is well known[8] that the thickness of the flowing film can be considerably increased if the surface on which it flows is covered by a thin film of solid air. The experimental cell in our apparatus has a large volume and can only be pumped out through a small diameter tube. Consequently, we were concerned that the air remaining in the cell might condense and form a solid film in the region of the minimum perimeter, thereby increasing the film flow rate onto the evaporator. This would have the serious consequence that the power required to eliminate the film would increase, and the refrigerator would then be unable to reach the desired temperature. To guard against this possibility we glued some pieces of porous Vycor glass to the inside wall of the cell. This glass provides a very large surface area (several hundred m^2) compared to the surface area of the cell. As a result most of the air should condense in the pores of the glass.

The calculated power to evaporate the estimated $\dot{V}=2.0\times10^{-4}$ cm^3 s^{-1} is 400 μW. This power applied to the evaporator must be removed from the top of the cell, which forms the condenser. The condenser should be at a sufficiently low temperature so that the density of gas atoms is low at the entrance to the maze formed by the baffles. The condenser was operated at 0.2 K, and the heat introduced into it via the condensation process was transported by three copper rods, each of 2.5-cm diameter, to a plate, with sintered silver for heat exchange purposes, inserted at the appropriate point in the dilute line of the dilution refrigerator slightly below the still.

With the temperature of the condenser at 0.2 K the rate at which atoms are thermally reevaporated from the condenser is very small compared to the rate at which atoms from the evaporator condense. However, the liquid helium bath, the main body of the cell and the low-temperature baffles cannot be at 0.2 K since at that temperature the vapor pressure of helium is marginally too high to maintain the wafer free of helium. At low temperatures the density of helium atoms in the saturated vapor can be estimated by assuming the gas is ideal and the difference in internal energy of the gas and the liquid is predominantly given by the latent heat. In that case

$$n_s=(2\pi mkT/h^2)^{3/2}e^{-l/kT}.$$

On substituting numerical values (the latent heat per particle is $l/k=7.16$ K) the density becomes

$$n_s=1.5\times10^{21}T^{1.5}e^{-7.16/T}\ \mathrm{cm}^{-3}. \tag{6}$$

Thus, at $T=0.2$ K the gas density is of the order of 10^5 cm^{-3} and so about 10^9 atoms will strike the wafer per cm^2 per second. This would result in a monolayer of helium forming on the wafer in about ten days. However, at 0.1 K where according to Eq. (6) the equilibrium density of gas atoms is 10^{-11} cm^{-3}, the rate at which atoms reach the wafer is completely negligible.

To keep the lower part of the cell $\leqslant$0.1 K as required, we used the following design. The lower part of the experimental cell was separated from the upper condenser section by a section of stainless-steel tube (see Fig. 1) to give a low thermal conductance. To cool the lower part of the

cell three copper posts ran from it to a plate, with sinter, in the dilute line of the dilution refrigerator. The cell could be maintained under these conditions at a temperature of 0.07 K. The heat input from conduction through the stainless-steel tube is estimated to be 40 μW.

The total rate of evaporation of helium atoms from the evaporator is $5\times10^{18}\ s^{-1}$, and we wanted to keep the total number hitting the wafer below $10^9\ s^{-1}$. If we make the pessimistic assumption that all atoms managing to find their way through the baffles will hit the wafer, then we require the baffles to capture all but 1 in 10^9 of the atoms that enter it. The cold part of the baffles consisted of sections of copper tube running up from a heavy copper plate at 0.07 K. The hot part of the baffles was made up of sections of stainless-steel tube, running down from a stainless-steel plate. One can make rough estimates of the efficiency of the baffles based upon the geometry and the literature values[9] for the probability of condensation of helium atoms incident onto a helium film. When a helium atom condenses in a film it may produce a phonon or roton which subsequently can reevaporate another helium atom. This introduces a significant uncertainty in the estimate of the baffles efficiency. The baffles that we built were perhaps overdesigned, but we felt that a large margin of error was advisable.

The principal heat input to the wafer and cooling rod connecting it to the mixing chamber was the result of the 0.32-cm o.d. stainless-steel tubing, in Fig. 1, used to stand the cooling rod off from the condenser plate through which it passed. This heat input is approximately 0.5 μW. Electrical connections to the bolometer on the silicon wafer passed through a header at the top of this tube, and then ran inside of this tube down to the wafer.

The configuration of the dilution refrigerator used to cool the experimental helium cell with its film evaporator is illustrated in Fig. 2. The refrigerator was constructed in our laboratory and for the most part is of conventional design. Below the still there is first a continuous counterflow heat exchanger, and next the plate with sinter in the dilute line for removing the 500 μW from the condenser at 0.2 K. After this there is another section of continuous heat exchanger, followed by the plate maintaining the helium cell at 0.07 K. This plate extracts ~40 μW. Below this is another continuous exchanger, two step exchangers, and finally the mixing chamber. The refrigerator is intended to operate at circulation rates up to 1 mmol s^{-1}. The temperatures given above are for a rate of 0.5 mmol s^{-1}. At this rate the wafer reaches 0.045 K, a temperature limited by the lack of sufficient heat exchangers below the 0.07-K plate to cool the returning concentrated ^{3}He properly. At lower circulation rates without the evaporator operating the mixing chamber goes below 0.02 K.

III. RESULTS

In Fig. 3 we show experimental results for the temperature of the evaporator as a function of heater power. The qualitative features of this curve are readily understandable. For a very small power applied to the heater (region I) the temperature increases fairly rapidly with applied

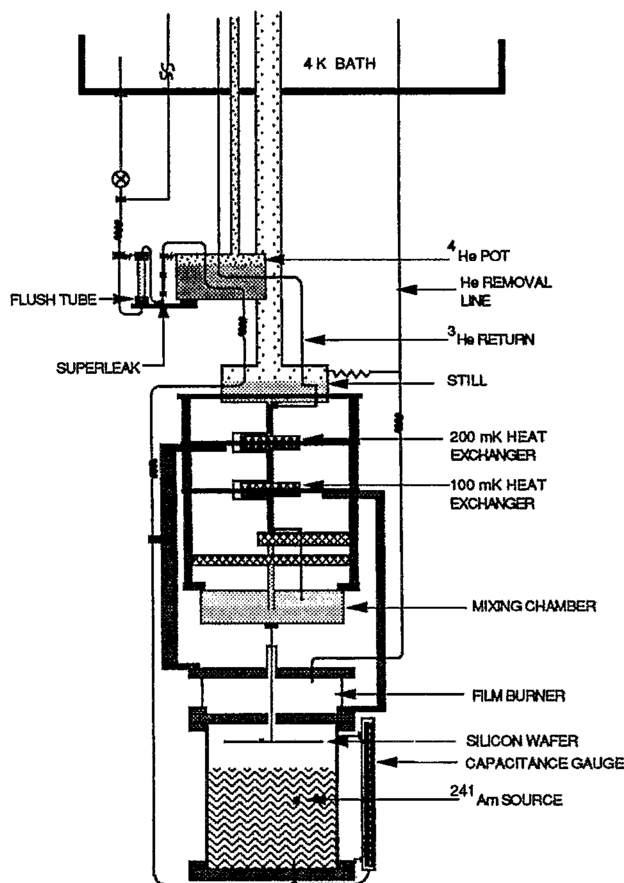

FIG. 2. Schematic design of the dilution refrigerator and heat links to the cell.

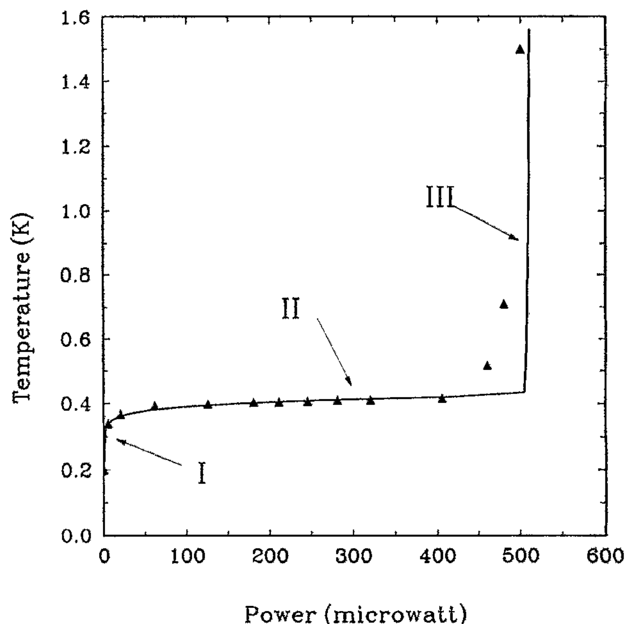

FIG. 3. Temperature of the evaporator as a function of the power applied to the evaporator heater. The triangles are experimental data and the solid curve is the results obtained from the model described in the text.

power. In this range the temperature is so low that negligible evaporation occurs. The heat that is supplied then escapes primarily by conduction through the stainless-steel tube that provides the support for the evaporator. The variation of the evaporator temperature with power primarily reflects the thermal conductance of this support. For higher powers (region II in Fig. 3) the temperature becomes almost independent of power. In this range the temperature has become high enough that evaporation is the dominant heat-loss mechanism. Since the evaporation rate increases very rapidly with increasing temperature, a change in power leads to only a very small change in the temperature of the evaporator. Throughout this region the evaporator and the silicon wafer are still covered with a helium film. At a critical power the temperature begins to rise rapidly (III). In this region all film that flows onto the evaporator is evaporated. The excess heat (i.e., that which is in addition to the amount required to evaporate the flowing film) then has to be conducted away by the support system. When this region is entered the film that is on the wafer also flows up on to the evaporator and evaporates. This process rapidly removes from the wafer all helium except for a very thin film (one or two layers) which is below the critical thickness required for superfluidity.

We have constructed a model of this process, and the results of a calculation based on this model are shown in Fig. 3. The model makes use of the thermal conductances of the materials involved (based on literature values). Although straightforward in principle, the model had to allow for the temperature variation that occurred over the surface of the evaporator. This area of $\sim 200\ \text{cm}^2$ was divided into several elements at different temperatures, and these temperatures were determined by requiring that the net heat flux into each element be zero. The heat fluxes included contributions from heat conduction, evaporation film, and from the heater on the evaporator.

In this calculation the evaporation rate as a function of temperature was estimated by the following method. For a film in equilibrium with saturated vapor, the rates of evaporation and condensation must be equal. If we make the approximation that the sticking coefficient is unity, the rate of evaporation is therefore determined. We then assume that when the vapor is removed the rate of evaporation from the film remains the same. The only adjustable parameter in the calculation is the value of $\dot{V}$. This was taken as $2.4 \times 10^{-4}\ \text{cm}^3\,\text{s}^{-1}$, a value that agrees well with the estimate made earlier from the geometry of the film-flow constriction. The film is completely evaporated when the temperature rises at a power of 500 μW. We believe that the discrepancy between the model and the experimental data in the transition region between II and III is due to the very long time constant (of the order of an hour) to reach equilibrium in this range of applied power. Thus, the experimental data have been taken before a steady state is reached.

When the system is operating in region III only one or two atomic layers are expected to remain on the silicon wafer. These layers still have a significant heat capacity and must be removed. To do this we heated the wafer

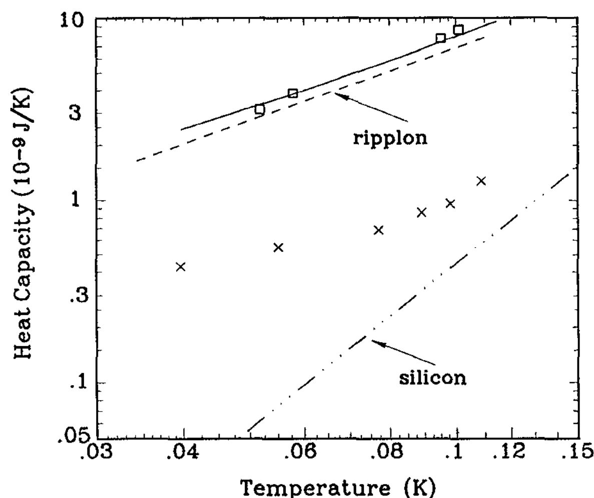

FIG. 4. Heat capacity of the calorimeter. Crosses and squares are the experimental data with and without the film burner in operation. The dashed-dotted line shows the heat capacity of silicon as calculated from Eq. (3). The calculated ripplon heat capacity for an area of 70 cm^2 is shown by the dashed line, and the solid line is the sum of the ripplon heat capacity and the experimentally measured heat capacity of the calorimeter with the film burner operating.

above 5 K for several minutes, while the helium bath temperature was maintained below 0.1 K and the evaporator kept in operation. Since the binding energy of helium to silicon is estimated[10] to be ~ 100 K, all the remaining helium atoms should be desorbed as a result of this procedure.

To test the operation of the film evaporator, or "burner," we have measured the heat capacity of the silicon wafer under the condition that it was covered with film, and also when the burner was operating and the wafer had been heated for a period to remove any residual helium. The wafer had a mass of 1.7 g, a thickness of 0.020 cm, and an area per side of 35 cm^2. The thermistor was a chip of neutron transmutation doped germanium, $1 \times 2 \times 0.2$ mm, provided by Haller.[11] Energy was added to the wafer by stopping in the silicon a 5.5-MeV alpha particle from an ^{241}Am source. The measured heat capacities are shown in Fig. 4, along with the expected lattice heat capacity for 1.7 g of pure silicon and the ripplon heat capacity for a 70-cm^2 surface area. We also measured the heat capacity of the wafer before any helium was introduced into the cell. This heat capacity was found to agree with that measured with the film burner in operation and helium in the cell. These results for the heat capacity indicate that the device we have constructed is able to maintain the wafer in a film-free state.

We have verified that the surface is free of helium by a second method. When there is a helium film on the wafer a pulse of heat applied to the wafer produces a burst of evaporated atoms which can be detected by another wafer located nearby. The *absence* of a signal of this type indicates that the film has been removed. Using this technique we have observed film-free conditions for more than 12 h.

In the present apparatus the time that the film-free condition can be maintained is limited by the need to refill the main storage Dewar.

As can be seen in Fig. 4, the heat capacity of the film-cover wafer is, to the accuracy of the measurements, equal to the sum of the ripplon heat capacity and the measured heat capacity of the wafer. However, the measured heat capacity of the film-free wafer is considerably larger than that of pure silicon. At 0.05 K the ratio is close to 10. The origin of the excess heat capacity of the bare silicon is uncertain. Care was taken with the addenda (thermistor, epoxy, leads) to keep their heat capacity below that of the silicon. The silicon was uncompensated and had a room-temperature resistivity of 1000 Ω cm so that bulk impurities are unlikely to be the cause.[12] The oxide layer on the surface would require a density of two-level systems ten times greater than has been measured[13] in bulk SiO_2 to account for the excess.

While the difference in heat capacities of the bare and film-covered wafer is not as large as predicted because of the excess heat capacity, these measurements do confirm that the apparatus can successfully keep an object completely free of film while suspended above a helium bath at low temperatures.

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