ISSN 0020-4412, Instruments and Experimental Techniques, 2009, Vol. 52, No. 2, pp. 301–307. © Pleiades Publishing, Ltd., 2009.
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LABORATORY TECHNIQUES

A Dilution Microcryostat–Insert

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Received September 23, 2008

Abstract—An autonomous dilution microcryostat in which ^{3}He circulates due to its condensation in the volume cooled by sorption evacuation of ^{3}He from a separate bath. A specific feature of this apparatus is the position of the sample holder in its upper part, which facilitates access to it. The holder is connected to the mixer through a heat conductor made from annealed copper wire with a length of ~0.5 m. The cryostat operates while inserted into a nitrogen-free portable (35 l) helium-filled vessel. The operating cycle of the cryostat includes the procedures of desorption, condensation, and cooling of the ^{3}He-filled bath to 0.35–0.40 K and the mixer to 0.05–0.10 K (lasting ~1.5–2.0 h) and also a period of maintaining the temperature below 0.1 K (12–14 h). The amount of liquid helium in the portable cryostat is sufficient for operating for 6 days. The lowest reached temperature of the holder is 0.04 K. When the power dissipated in the holder is 0.5 μW, its temperature does not increase above 0.1 K. The instrument is mainly designed for cooling sensitive radiation detectors.

PACS numbers: 07.20.Mc

DOI: 10.1134/S002044120902033X

INTRODUCTION

Paper [1] describes refrigerator units for two versions of a portable cryostat based on ^{3}He dilution in ^{4}He. The operation of these units is based on the evacuation of their mixture vapors through condensation on a cold wall [2, 3]. Evacuation of vapors of liquid ^{3}He from a separate bath by a sorption pump is used to cool the condenser. To facilitate the operation of these devices, they are inserted into a portable vessel with liquid helium. Such cryostats are primarily intended for cooling radiation detectors. It is reasonable to place detectors or other samples under study at the top of a cryostat outside of the portable vessel in order to simplify the admission of radiation to the low-temperature zone and ensure easy access to samples during their mounting. Below, an apparatus that satisfied these requirements is described.

DESIGN OF THE MICROCRYOSTAT

Figure 1a shows a general view of the assembled microcryostat–insert. During operation, the apparatus is inserted into a nitrogen-free cryostat filled with liquid helium, which is produced at NPO Geliimash (Fig. 1b). The parameters of the portable cryostat are as follows: capacity of 35 l, evaporability of helium of ~1 l/day, and diameter of the throat of 58 mm. The height with the inserted microcryostat is 1 m.

The housing of the insert consists of upper part *2* (Fig 2a), to the flanges of which upper cap *1* and outer stainless-steel tube *25* (with a diameter of 56 mm and a wall 0.3 mm thick) running downward are attached. The cap and the tube are sealed with indium. To guarantee the stability of the tube walls under evacuation, reinforcing rings are soldered on it. An adapting copper ring is soldered to the bottom of the tube with the Ag–Cu solder. Copper tube–screen *20* covering the low-temperature part of the device is hermetically soldered to the copper ring. Tube *20* is vacuum-tightly soldered to the housing of sorber units *16*, which is positioned at the bottom of the cryostat, with an indium–tin solder with a melting temperature of 105°C. This is the final soldering procedure during assembling. During dismantling, this seam is unsoldered, thus allowing taking away the outer tube together with the copper tube and have easy access to internal elements.

Upper part *2* is manufactured from stainless steel and contains four hermetic cavities separated from one another. Three of them (cavities *27*) have toroidal shapes, are positioned in the bottom part, and are intended for keeping working gases—^{4}He and ^{3}He—and their mixture 30–40% ^{3}He + 70–60% ^{4}He. The volumes are closed by valves shutting them off the gas-supplying line and are connected to the corresponding low-temperature units through stainless-steel capillaries with a diameter of 1 mm and 0.1-mm-thick walls. The volumes are filled with gases when the low-temperature part of the insert is cooled to helium temperature, and then the apparatus is disconnected from the gas station. During subsequent warming up of the apparatus, they are held at a pressure of 25–50 atm inside the cryostat.

The upper cylindrical cavity contains sample holder *3* surrounded by screens *5–7* with removable covers having temperatures of 70–100, 4.2, and 0.35–0.45 K, respectively. Heat conductor *4* made from a 2-mm-diameter copper wire runs downward from the sample holder to the mixer, and annealed copper tubes run from

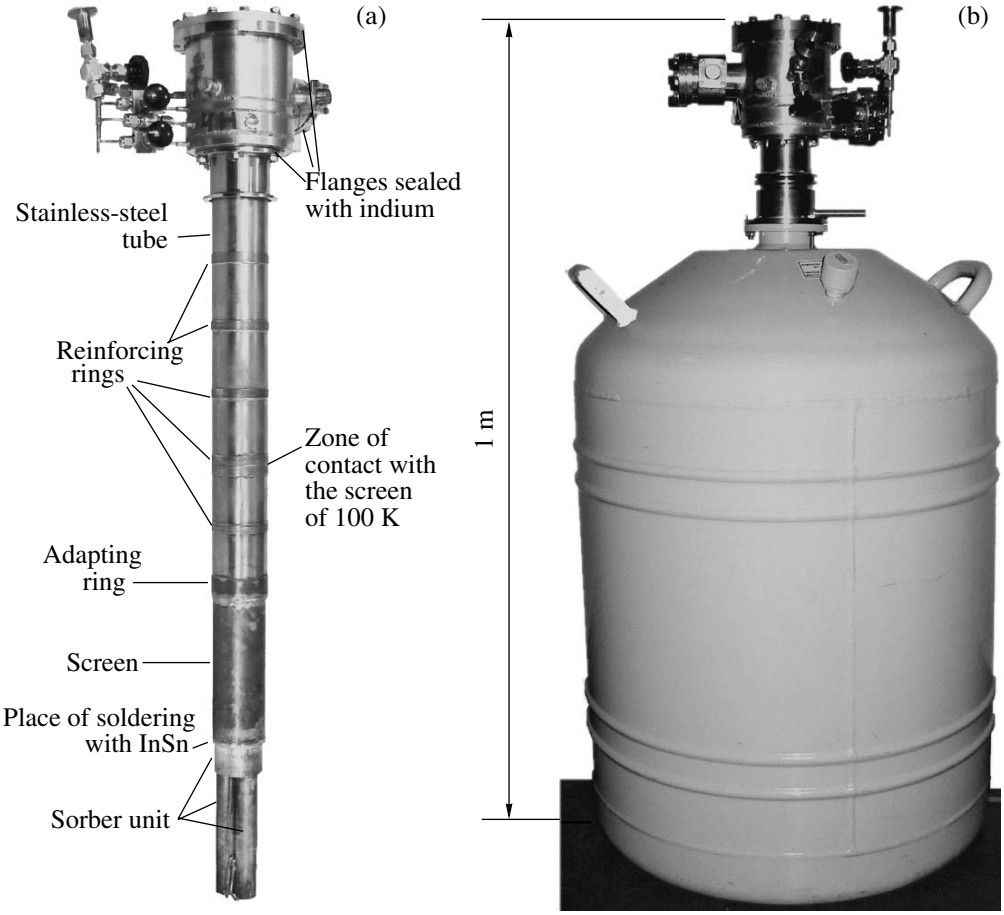

Fig. 1. (a) Appearance of the cryostat–insert mounted in (b) a portable helium cryostat with a volume of 35 l.

the screens to the ^{3}He bath, copper flange *30* (Fig. 2b) connected through three annealed copper rods *19* to sorber unit *16*, and brass sleeve *31* with leaf-type springs pressed from the inside to the thin-wall stainless-steel tube. The place of contact lies in the region of the experimentally selected area of the portable cryostat's throat, so that the temperature of this screen cooled with helium vapors is stationary at a level of about 100 K (Figs. 1 and 2b). When the flow of helium from the Dewar vessel increases during regeneration of sorbers, this temperature decreases to 70–80 K. All heat conductors were annealed in vacuum in the presence of a small (a pressure of ~1 mTorr) amount of oxygen. Owing to this, the thermal conductivity at liquid-helium temperature reached 30–35 W/(cm K).

Sample holder *3* is fastened to the ^{3}He screen through tension braces made from polymeric filaments stretched with centering screws *28* (Figs. 2a and 3). In the same manner, the screens with temperatures of 0.4 and 100 K are fastened to the screen of 4.2 K and the housing, respectively.

The screen of 4.2 K is centered relative to the screen of 100 K thanks to the structure stiffness. The tension braces are manufactured from Armos 100A filament (OAO Tverkhimvolokno), which is a bundle of thin filaments. Its certified parameters are as follows: a modulus of elasticity of 14 500 kgf/mm^2, a rupture strength of 25 kgf, and a fracture stress of 500 kgf/mm^2. These data allow assessment of the bundle cross section at a value of 0.05 mm^2. The integral thermal conductivity of the bundle measured from room to nitrogen temperatures is 2.0–2.5 mW cm. In accordance with the results of studying the parameters of the cryostat at low temperatures, the thermal conductivity can be considered equal to $0.07T^2$ μW cm/K^3 to an accuracy sufficient for practical application.

Electric leads connected to the sample holder run from the contacts of adapters produced from cermet packages of microcircuits (Fig. 3). Twisted pairs of 0.08-mm-diameter constantan wires running inside the screen of 0.4 K are soldered to the same contacts. Cermet parts are soldered through copper heat conductors to the screen of 0.4 K, thereby allowing removal of heat conducted through wires from warmer parts of the cryostat [1]. The wires pass from the bottom part through a guide tube soldered into the screen bottom. The elec-

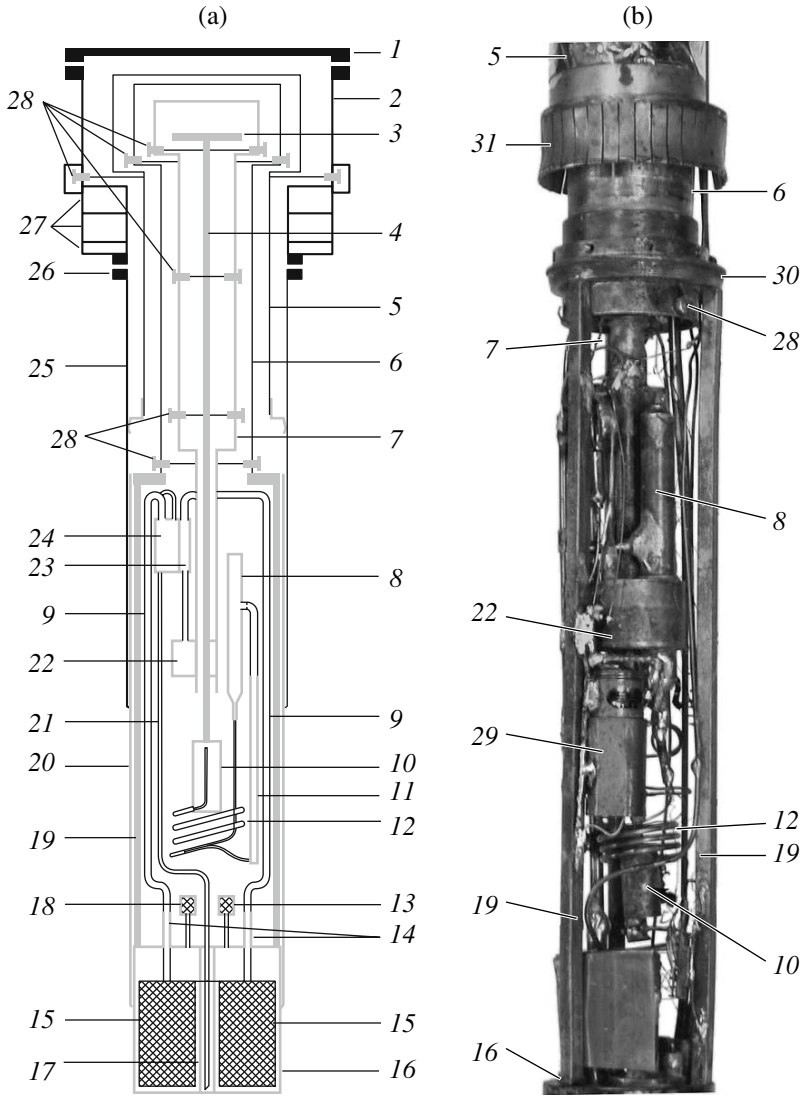

Fig. 2. (a) Simplified diagram of the cryostat–insert and (b) appearance of the low-temperature part of the cryostat with a removed screen: (*1*) indium-sealed stainless-steel cap; (*2*) upper part of the body; (*3*) sample holder; (*4*) copper heat conductor; (*5–7*) copper screens of 100.0, 4.2, and 0.4 K, respectively; (*8*) condenser of mixture vapors; (*9*) tubes for evacuating the ^{3}He (right) and ^{4}He (left) baths; (*10*) mixer; (*11*) still; (*12*) heat exchanger; (*13, 18*) sorption heat valves of the ^{3}He and ^{4}He sorbers, respectively; (*14*) copper inserts in evacuation lines; (*15*) ^{3}He (right) and ^{4}He (left) sorbers; (*16*) copper case containing sorbers; (*17*) ^{4}He condenser; (*19*) supporting rods–heat conductors; (*20*) copper screen; (*21*) tube for transferring ^{4}He; (*22*) ^{3}He bath; (*23*) ^{3}He condenser; (*24*) ^{4}He bath; (*25*) stainless-steel tube; (*26*) indium-sealed lower flange; (*27*) containers of the mixture, ^{3}He, and ^{4}He; (*28*) screws for stretching polymeric centering filaments; (*29*) magnetic thermal switch for preliminary cooling of the mixer and ^{3}He system; (*30*) adapting ring of the screen of 4.2 K; and (*31*) leaflike springs.

tric leads for feeding the sample holder and servicing the operation of the cryostat are connected to hermetic multipin connectors mounted on the top of the body in a unit welded to its side (Fig. 1). This unit has an indium-sealed cover that facilitates access to the contacts of the connectors from the side of the vacuum cavity.

The refrigerator unit installed in the apparatus operates periodically. Its designed is described in detail in [1] and contains the following functional units placed in a common vacuum volume (Fig. 2):

(i) A ^{4}He loop incorporating chamber *24* with $T \sim$ 1 K (1-K chamber) and ^{3}He condensation chamber *23* that compose a common unit, ampoule *17* for ^{4}He condensation, capillary *21* for transferring ^{4}He and sorption pump *15* that evacuates helium vapors through stainless-steel tube *9* with a ~5-cm-long copper insert *14*; the lower part of the insert is soldered through the cap of housing *16*; sorption thermal valve *18* serves to control the heat exchange between sorber *15* and its container, housing *16*.

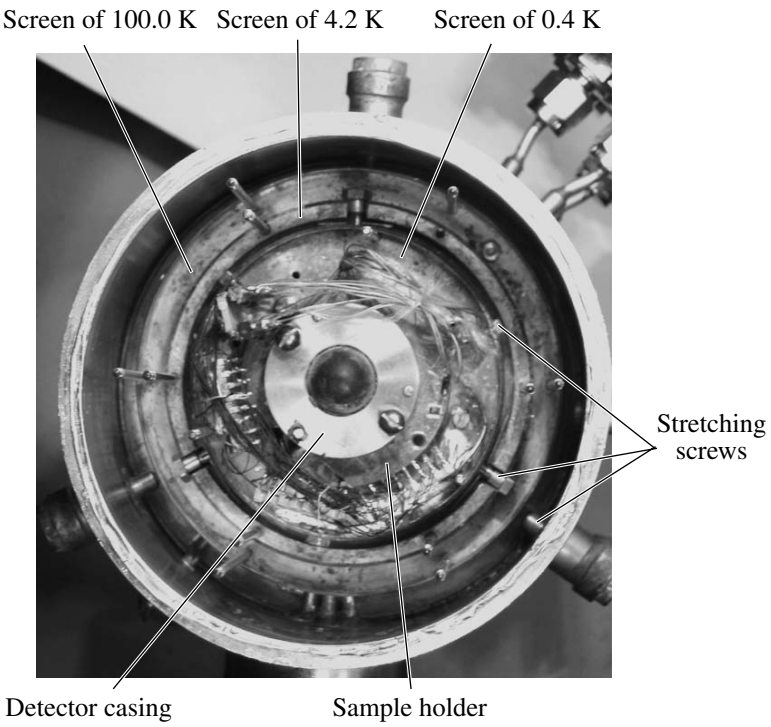

Fig. 3. Apparatus with the removed upper parts of the screens and the cap (top view).

(ii) The main ^{3}He loop containing ^{3}He bath *22* and condenser *8* of ^{3}He–^{4}He mixture vapors; bath *22* is cooled by evacuation of liquid-^{3}He vapors through tube *9* by sorption pump *15* equipped with thermal valve *13*.

(iii) The dilution loop containing mixer *10*, tubular heat exchanger *12*, still *11*, and aforementioned condenser *8* installed in bath *22*.

The design of the main units was also described in detail in [1], where, in addition, a device for forced cooling was described. This device is based on bellows pressing thermal contacts when a gas is supplied into them. Unfortunately, the bellows used in our device were unreliable and leaks appeared in their walls after several heating–cooling cycles in the cryostat. Therefore, we replaced the bellows with electromagnetic clamp *29*, which pressed the contacts connected to mixer *10*, ^{3}He bath *22*, and heat-conducting rod *19* at a current of 0.1–0.2 A through an electromagnet winding. To reduce the consumption of liquid helium, the winding is made from a NbTi superconducting wire in a copper matrix.

OPERATION OF THE CRYOSTAT

After assembling and evacuation of the vacuum jacket to a pressure of ~1 mTorr, the apparatus is immersed into liquid nitrogen. In the night period, its internal parts are cooled to 90–100 K. Subsequently, the cryostat is transferred to a portable cryostat filled with liquid helium. In this case, liquid helium intensely evaporates into the atmosphere; during slow immersion of the cryostat into a Dewar vessel (~5–10 min), approximately 2 l of liquid He are lost. The corresponding gas losses are not so high to make it reasonable to manufacture complex and expensive locking devices. This is especially so for laboratories in which helium is not collected.

After the cryostat is immersed into helium and the winding of the magnetic switch is cooled (this occurs in several minutes), forced cooling is switched on and, 4–6 h later, all cooled parts reach a temperature <10 K. Then, the operations of regeneration of the sorbers and condensation of the liquids (described in [1], requiring a time of ~1 h, and resulting in a loss of 0.15–0.20 l of liquid He from the dewar) are performed, and the ^{3}He evacuation and the still's heater are switched on. At a heating power of 200–300 μW, the mixer is cooled to 0.1 K within 30–40 min (Fig. 4) and the total heating power of the still can be reduced to 30–40 μW. With this heat supply, the low temperature is maintained for 10–13 h until the entire ^{3}He evaporates. After that, the cycle can be repeated.

Figure 4 shows that cooling is a complex process. After the heating power of the still is switched (within ~1 h), the temperatures of the mixer and still change nonmonotonically. This is due to the fact that the state of the system is determined, not only by the temperatures of its main units, but also by the probability that the initial stratification of the mixture may occur not only in the mixer, but also at other places, e.g., in the heat exchanger. This process is also affected by the heat transfer over a superfluid film, the thermomechanical

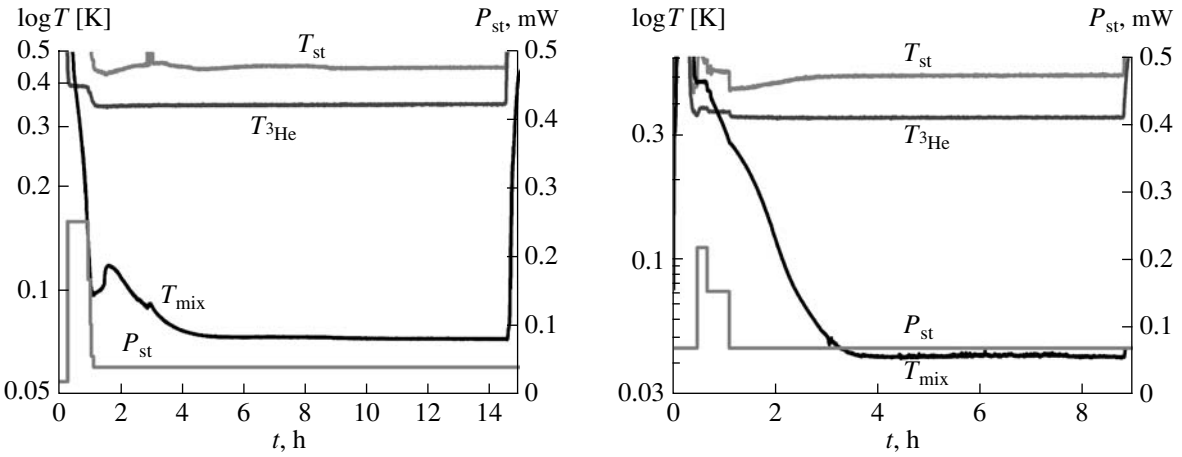

Fig. 4. Examples of time dependences of the mixer, ^{3}He bath, and still temperatures and the power supplied to the still for two experiments.

effect, and thermoosmosis. Thus, the cooling process depends on many initial parameters, including the prehistory. Therefore, the amounts of heat supplied to the still (and in that way, to the ^{3}He bath) in different experiments may differ significantly. Therefore, the time of maintenance of the low temperature slightly changes from experiment to experiment.

At a higher heating power of the still and, correspondingly, more intense ^{3}He circulation, a lower temperature is reached (the lower part of Fig. 4). However, the time for which it is maintained naturally shortens.

The temperature of the sample holder coincides with the mixer temperature to an accuracy of its measurement with RuO_2 thermometers (several millikelvins). The main contribution to the error is made by the use of the mean temperature-dependent resistance obtained for three resistor specimens instead of an individual calibration during the resistance-to-temperature conversion [1]. Note that the mixer temperature is even several millikelvins higher than the holder temperature because radiation from the thermal valve heated to ~15 K and ensuring the cooling of the ^{3}He sorber is incident on the thermometer. When the valve is switched off for a few minutes, the mixer temperature decreases and approaches the holder temperature. However, because of impaired heat-exchange conditions, the sorber temperature begins to rise, the temperatures of the ^{3}He bath and the still follow this tendency, and it becomes necessary to heat the thermal valve again.

Figure 5 shows the time dependences of the mixer and holder temperatures at different powers supplied to the holder. To demonstrate the curves more clearly, they were plotted with introduction of a small correction to the readings of the mixer thermometer to bring into coincidence the moments at which the supplied power is zero. When heat is supplied to the holder at the minimum temperature (≈0.07 K) reached in this experiment, the mixer temperature changes insignificantly and the holder temperature increases by several hundredths of a degree within characteristic times of several seconds. The inset in Fig. 5 shows that overheating rapidly increases as the temperature decreases; this increase is appreciably quicker than that following a linear law characteristic of a normal metal (copper). The contribution of the copper heat conductor at T = 0.05 K extrapolated from the region of higher temperatures is no higher than 6 mK/μW. In this temperature region, overheating is caused mainly by the exponentially increasing thermal resistance of the superconducting InSb-soldered seam joining the heat conductor to the mixer. Hence, if necessary, overheating can be reduced by using another, more complex technology for joining these units. However, a heat inflow of even 0.1 μW to radiation detectors is unacceptable and hinders their normal operation (the cryostat is designed for this purpose), and the above drawback is insignificant.

Note that overheating of the holder with a very low power almost does not affect the temperature and the mixer operating mode, occurs very quickly, and may be useful if it is necessary to stabilize the temperature. As is seen in Fig. 4, when a steady-state mode is reached, the temperature is quite stable, its variations are within 1 mK, and heating at a level of 0.1 μW is sufficient for maintaining a stable temperature. It is obvious that, for a cryostat in which the continuously operating refrigerator unit described in [1] is used, electron stabilization allows maintenance of the detector temperature to an accuracy much better than 1 mK for several days.

With the use of the described cryostat, we have begun to study superconductor–insulator–normal metal tunneling structures (SIN structures) as detectors for the terahertz frequency range. Figure 6 shows an example of the recorded current–voltage characteristics at different temperatures. As is seen, a temperature decrease actually leads to significant changes in these

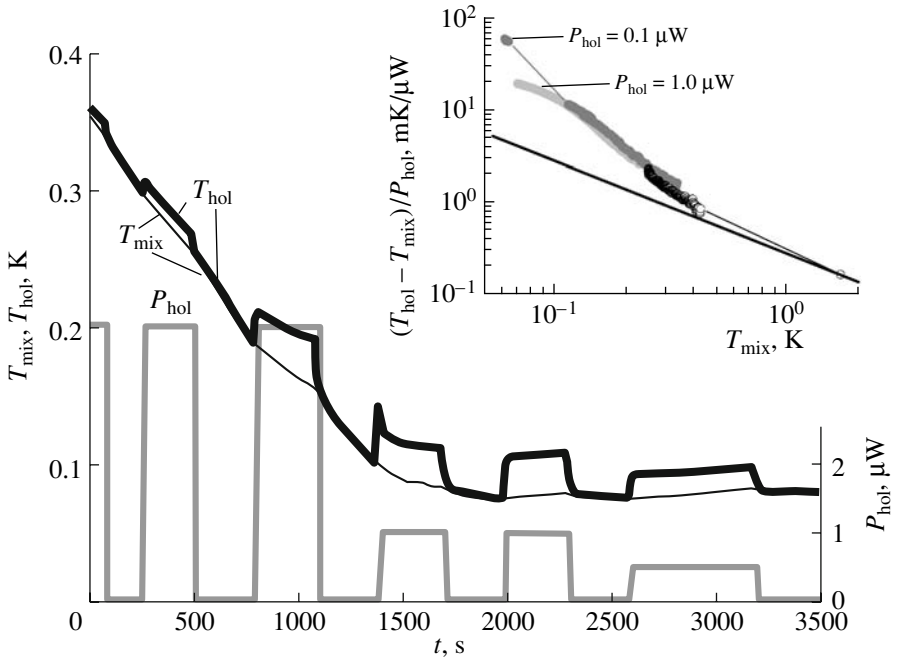

Fig. 5. Time dependences of the mixer and sample-holder temperatures at different levels of power supplied to the holder. The inset shows the overheating of the holder divided by the power supplied to it as a function of the mixer temperature (different dots correspond to different experiments); the straight line corresponds to the thermal resistance of the normal metal.

characteristics: the length of the linear region increases, and the slope of the linear region appreciably decreases.

PARAMETERS OF THE CRYOSTAT

Minimum registered temperature of the sample holder, 0.04 K.

Time of maintenance of a temperature <0.1 K, 10–13 h.

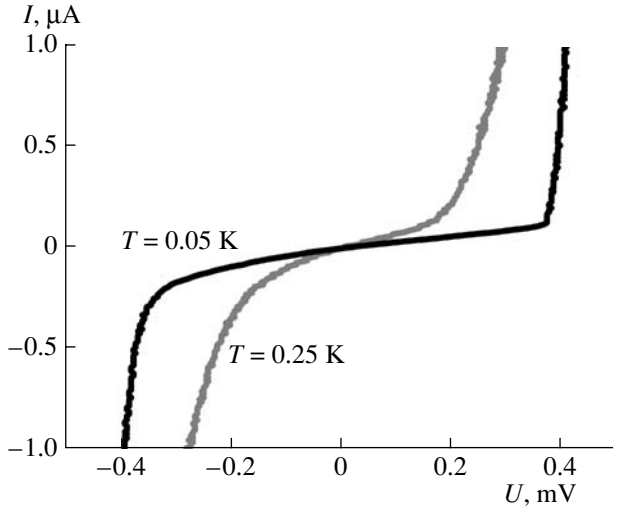

Fig. 6. Current–voltage characteristics of a tunneling SIN structure at temperatures of 0.05 and 0.25 K.

Stable operation at a power supplied to the still of 40–400 μW (at a circulation rate of ~1.5–15.0 μmol/s).

At a circulation rate of 1.5 μmol/s and a heat power of 0.5 μW supplied to the sample holder, its temperature is no higher than 0.1 K.

Time of mixer cooling from 1.0 to 0.1 K, ~30–40 min (depending on circulation rate).

Heat inflows to the still from the screen of 4.2 K through the filaments fastening the ^{3}He unit to it, 5–7 μW, and to the still and ^{3}He bath over the superfluid film in a mixture-transferring capillary, 20–25 μW.

Minimum temperatures of the ^{3}He bath, 0.34 K, and the ^{4}He bath during ^{3}He regeneration, 0.9 K.

Temperature of the screen of 100 K, 80–100 K.

Time of preliminary cooling to nitrogen temperature in liquid nitrogen, the night duration.

Time of cooling to a level of 4–5 K, the magnetic switch being switched on, 4–6 h.

Helium loss during preliminary cooling, ~5 l.

Time for preparing the initiation of a circulation cycle, 1.0–1.5 h.

Time of operation with one portable cryostat with helium, 6 days. The amounts of gases for filling the cryostat: 0.22 mol ^{4}He, 0.11 mol ^{3}He, and 0.05 mol of a mixture of 40% ^{4}He + 60% ^{4}He.

Pressures of gases in the warm filled apparatus: 50 atm (^{4}He), 25 atm (^{3}He), and 50 atm (mixture).

Masses of the insert and the Dewar vessel filled with liquid helium and containing the insert, 7.5 and at most 35.0 kg, respectively.

ACKNOWLEDGMENTS

The author is grateful to A.F. Andreev and G.D. Bogomolov for support of this study; R. Herrmann, I.N. Khlyustikov, and A.V. Ofitserov for participating in this work at the initial stage; and M.A. Tarasov for arrangement of measurements with the SIN structure.

This study was supported in part by the Russian Foundation for Basic Research, project no. 07-02-00515.

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