

Adapting a displex closed-cycle helium refrigerator for ultrahigh vacuum operation^{a)}

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(Received 17 April 1980; accepted for publication 1 September 1980)

A modification of a commercial closed-cycle liquid helium refrigerator is described which permits operation at pressures below 10^{-9} Torr. By using a sapphire insulator on the low-temperature stage, and incorporating vacuum-compatible heating elements, samples can be biased to ± 30 kV over a temperature range of 15–300 K.

PACS numbers: 07.20.Mc

During the development of a point-projection microscopy to image molecular contours,¹ we have found it necessary to adapt a commercial "Displex" closed-cycle helium refrigerator² for UHV operation below 10^{-9} Torr. Commercial units may not be assembled with UHV compatible brazes, and are often supplied with teflon, mylar, and fiberglass-wrapped heating elements for controlling the temperature of a sample. (The temperature of a sample is most often controlled by supplying electrical power to a heating element in order to partially defeat the cooling capacity of the refrigerator. We have investigated various braze alloys and have found that silver braze³ #603 is suitable for UHV operation. The use of this braze (or an equivalent) can be specified when the refrigerator is ordered from the manufacturer. To ensure UHV operation when the refrigerator is not operational, materials with a low outgassing rate must be used for the specimen heater. With this in mind, we have designed the simple specimen heater assembly shown in Fig. 1. The heater assembly is mounted in an OFHC adapter that screws into the OFHC end of the second stage of the refrigerator.

Since our samples must be biased to high voltage (up to 30 kV), we have also incorporated a sapphire insulator which provides an electrically insulated path of high-thermal conductivity between the sample and the grounded refrigerator. To ensure good thermal contact and provide for sample interchange, the sample is clamped into a small groove cut into the sapphire by a moveable counterelectrode of negligible thermal conductivity⁴ (not shown in Fig. 1). The sapphire insulator was machined with a 2-mm diam. shoulder at its base. It is retained in a 3-mm deep counterbore in the top of the OFHC adapter by an axially-drilled, threaded cap which captivates the shoulder in the counterbore (see Fig. 1). Since copper has a greater thermal expansion than sapphire, it will shrink around the sapphire during cool down to provide a more intimate thermal contact. A pure, dead-soft, indium gasket placed between the sapphire insulator and the bottom of the counterbore ensures maximum thermal contact between these parts. A pure indium gasket is also used to ensure maximum

thermal contact between the refrigerator and the OFHC adapter. This gasket also minimizes the possibility that these pieces will loosen during operation. (The second stage of the Displex refrigerator contains a piston assembly which transmits an axially directed mechanical vibration of ≈ 0.2 mm peak amplitude to the OFHC adapter).

The second stage of the Displex refrigerator has a cooling capacity of ≈ 3 W at 80 K. Therefore, to heat a specimen to a temperature significantly higher than 80 K, one must supply ≥ 3 W of electrical power to the second

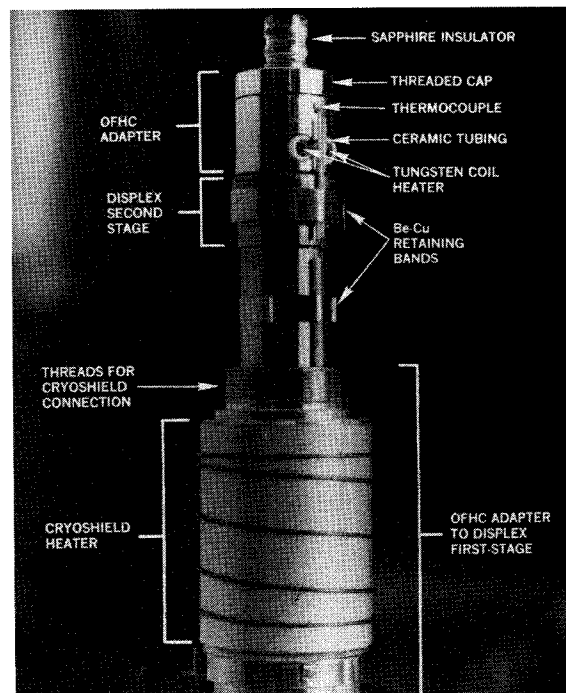


FIG. 1. A photograph of a commercial, "Displex" closed-cycle, liquid-helium refrigerator (Ref. 2). The Figure shows a first and second-stage heater assembly and temperature monitor designed for UHV operation. A sapphire insulator provides an electrically insulating path of high-thermal conductivity between a sample and the second stage of the refrigerator. In this way, the sample can be routinely cooled to <20 K while being biased to 30 KV dc.

stage heater. The heating element is constructed of two coils connected in series and wound from 0.25 mm diam. molybdenum wire. Each coil is mounted in a 3 mm ID ceramic tube which is inserted into a hole drilled into the OFHC adapter (see Fig. 1). To determine the electrical operating parameters of the heating element, the two coils (in series) are placed in vacuum and an ac current is passed through them until they barely achieve dull red heat (600 C) as determined by visual observation in a darkened room. The corresponding current and voltage is recorded, and the electrical power calculated. We have chosen to supply ≈ 10 W of power to the heating elements. If the power is less than 10 W, more turns of wire are added to each coil to increase its resistance. In this way the maximum operating temperature of each coil is always kept below 600 C. An ac filament transformer is then chosen to supply this voltage and current. A Kp/Au-0.07 at. % Fe thermocouple is soldered (using pure indium) into a shallow depression drilled into the OFHC adapter. This thermocouple controls the ac voltage to the primary of the filament transformer by means of a commercial set-point controller.⁵ In this way, the temperature of the OFHC adapter, the sapphire insulator, and the sample can be maintained to within ± 0.5 K over the temperature range of ≈ 15 –220 K.

To ensure that the lowest possible specimen temperature is reached, the entire second-stage assembly is surrounded by a highly-polished, OFHC radiation

shield. This "cryoshield" is normally maintained at 40 K (the temperature of the first stage of the refrigerator), by attaching it to a threaded, OFHC adapter in thermal contact with the first stage. If desired, the temperature of the cryoshield assembly can be raised by a heater in the first-stage adapter (similar in construction to the second-stage heater, but capable of overcoming the ≈ 7 W cooling capacity of the first stage at 77 K).

The closed-cycle system described in this note has been in use in our laboratory for over four years. The reliability of the closed-cycle refrigerator has been excellent. No difficulties have been encountered during ≈ 5000 h of almost continuous operation.

The system described in this note also acts as an integral cryopump to achieve pressures $< 5 \times 10^{-10}$ Torr. However, since In gaskets are used, bakeout temperature is limited to well below the melting point of indium (156 C).

^{a)} This work was supported by the U.S. Department of Energy under Contract DE-AC04-76-DP00789.

^{b)} A. U.S. Department of Energy facility.

¹ Abstracts of the 26th International Field-Emission Symposium, Berlin, West Germany (1979) in *Ultramicroscopy* 5, 248 (1980).

² Air Products and Chemicals, Advanced Products Department, P.O. Box 538, Allentown, Pennsylvania, Displex Closed Cycle-Refrigeration System, Model CS 202.

³ Handy and Harman, 850 Third Avenue, New York, NY.

⁴ The geometry of the sample interchange mechanism is shown in: J. A. Panitz, *Prog. Surf. Sci.*, 8(6), 224 (1978).

⁵ Reference 2, Model APD-B.