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Multiple Sample Cryostat for the Determination of Superconductor Properties

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A Multiple Sample Cryostat for the Determination of Superconductor Properties

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Abstract

Cryostats which are currently used to characterize the properties of superconductors between 4 K and 100 K are primarily single sample devices. The purpose of this paper is to present an instrument design which can hold up to five (5) one cm. diameter samples at a stable temperature (± 0.1 K) within the above range specified while measurements of the sample properties are made.

Introduction

The characterization of potential superconducting materials requires an instrument which can maintain a stable temperature within the superconductor's electromagnetic transition range (usually between 4 K and 77 K). Such devices, called Cryostats, use liquid helium, LHe, and/or liquid nitrogen, LN_2, to provide a cold reservoir above which the sample temperature is varied. Commercial Cryostats, while providing excellent temperature stability, are usually single sample devices which cost between $10,000 and $20,000 (Van Sciver, 1986). The object of this project is to design and build a multi-sample Cryostat for superconductor evaluation for a total cost of $5,000.

The design of the Cryostat began with a survey of the pertinent literature regarding the design and construction of commercial cryostats, from both practical and theoretical considerations. The various methods of varying temperature were explored with the additional constraints of producing an economical accessible multi-sample device.

Design

Heat Load and Vacuum Considerations.--The Cryostat is composed of two concentric dewers (Fig. 1), a liquid helium, LHe, reservoir which is surrounded by a reservoir of liquid nitrogen, LN_2. The LN_2, at 77 K, reduces the radiational heat load from room temperature into the more expensive liquid helium by three orders of magnitude since this load is proportional to the fourth power of the absolute temperature (White, 1968). The relation is given by Stephan-Boltzmann's law and is of the form

$$Q_r = \sigma (T_2^4 - T_1^4)/V,$$

where σ is the Stephan-Boltzmann constant ($5.67 \times 10^{-4} W/m^2 \cdot K^4$), and V is the view factor which relates the areas and emmisivities of surface two as seen by surface one.

To further reduce heat loss, the volume external to each of these dewers is evacuated to decrease the conductive heat loss of the gases and to reduce the losses due to external condensation on the LN_2 dewer. By lowering the pressure, the distance a molecule travels before colliding with another (called the mean free path) increases to the point where the dominant method of thermal energy exchange is due to collision between the remaining gas molecules and the dewer walls. Pressure reductions of 10^{-4} torr correspond with a mean free path of 100 cm (Rose-Innes, 1973), which is much less than the interwall distance of the dewers. Because the pressure in these areas is determined by the statistical interactions of the molecules (molecular flow), the ports to the external diffusion pump must be made as large as possible. This provides a large cross-sectional area through which the particles may pass and be trapped.

The location of welds and venting of blind threaded holes also becomes important when considering molecular flow. If a weld between two plates is on the pressurized side of a vacuum-pressure interface, as shown in Fig. 2, the molecules within the seam will only 'see' the face of the seam to pass through and the time to pump down to sufficient vacuum increases (Green, 1986).

The diameter of the tubes which are used to fill the cryogens into their respective dewers are important in that if the tube is sufficiently small (less than 2.5 cm) and is long (over 30 cm) the tube may develop powerful low frequency vibration due to the thermosiphoning of vapor from the dewer to the atmosphere (Green, 1986). Fill tubes are made as short as possible to eliminate these problems.

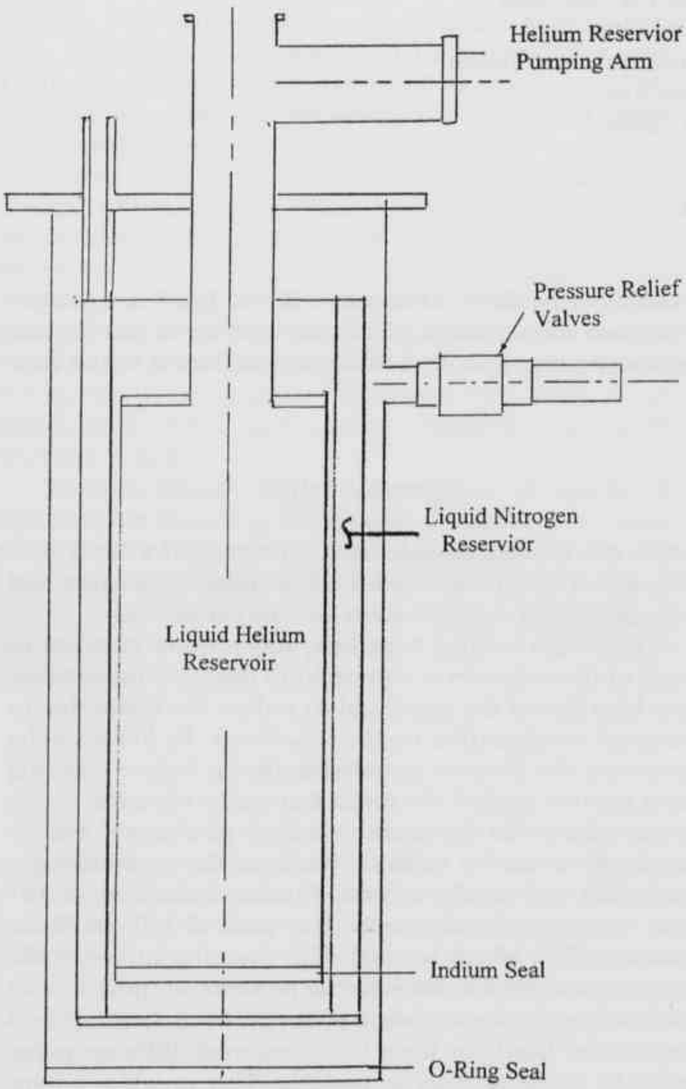

Fig. 1. Cryostat Dewer

A safety pressure relief valve is mounted on the outer shell of the Cryostat in case of a leak of one of the cryogens into the vacuum jacket. Upon contact with the room temperature wall of the jacket, the cryogen would expand and increase the pressure which might destroy the Cryostat.

Stainless steel was chosen as the material from which the Cryostat would be constructed since it does not outgass in a vacuum, has low thermal conductivity (16 W/m-K), high yield strength (130 kpsi), and is readily welded. Where a stainless steel plate is to be welded to a length of tube to form a dewer, a groove is cut into the plate to half of its thickness, as shown in Fig. 2. This reduces the stress on the weld by creating a much smaller area across which thermal shrinkage occurs during dewer cooling as the pure tensile weld stress is reduced by the cantelever thus created (Barron, 1966). Additionally, a seating groove is cut into the tube to minimalize shear stresses on the weld.

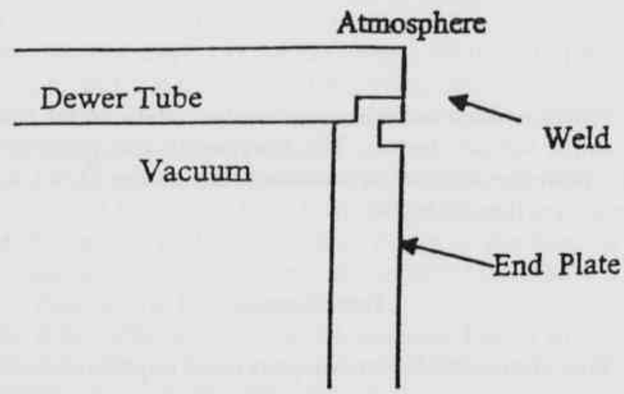

Fig. 2. Welded connection between dewer tube and end plate showing improper weld location.

Temperature Variability.--Superconducting samples may be placed directly in contact with either the LN_2 or the LHe to determine their properties at those temperatures, although the energy involved in measuring the resistance of the material will tend to raise its temperature above that of the surrounding cryogen. If the sample holder is made to be in good thermal contact with the cryogen, then any heat added to the holder will quickly be passed to the liquidfied gas and will not significantly raise the temperature of the sample. To vary the temperature of the sample then, the sample must first be cooled to cryogenic temperatures, and then the sample should be thermally isolated from the cryogen. Any heat which is subsequently added will primarily raise the temperature of the sample and not be wasted in boiling the liquified gas (Holma, 1981).

An exchange gas method (Obert et al., 1982) for temperature variation was chosen to modify the temperature of the sample, as shown in Fig. 3. As seen in this figure, the gas exchange insert fits into the Cryostat helium dewer fill tube. The temperature of the sample is varied by indirectly coupling the sample to the LHe by varying the pressure of helium gas in the sample compartment. When the pressure of the gas is high (760 torr), the thermal link between the LHe and the sample is good and the sample approaches 4 K. As the pressure in the sample compartment is reduced, it becomes thermally isolated from the cryogen and the sample temperature can be raised by a small heater imbedded in the sample mounting block. A thermocouple mounted on the sample block is used to determine the average block temperature.

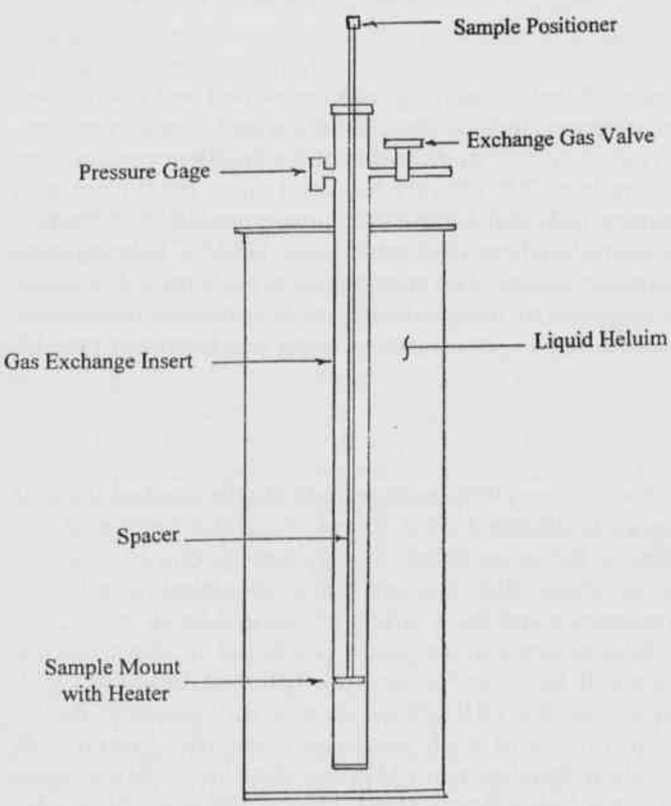

Fig. 3. Gas Exchange Insert for Cryostat

Cryostat Operation.--Initial loading of the Cryostat begins with purging of the device with nitrogen gas, which flushes the room air and concomitant moisture from the walls of the instrument. The evacuation valve is then attached to a rotary pump and the pressure is 'roughed in'. Once the pressure is reduced to approximately 10^{-2} torr, the diffusion pump is started and the pressure is reduced to 10^{-4} torr. The evacuation valve is closed and the LN_2 is the introduced into the dewer. This brings the temperature of the device down to 77K, whereupon the LHe is siphoned from a storage dewer. The LHe cryopumps the pressure in the vacuum jacket to 10^{-6} torr which provides an ideal storage environment. Estimates on the heat load indicate that the instrument will require 15 liters of LN_2 for initial cooling.

Multiple samples which are to be introduced into the Cryostat are prewired and screwed to the sample mount. The samples are cleaned to remove any oils which would outgas under reduced pressure, preventing the attainment of a sufficient vacuum. Next, the samples are mounted in the gas exchange insert, which is flooded with room temperature helium gas. The entire insert is precooled in the nitrogen dewer and then the insert is placed in the Cryostat LHe fill tube where it cools to 4K. After equilibrating, measurements on the samples are taken to determine if any of the samples are superconducting. The insert pressure is then lowered by pumping which thermally isolates the sample mount from the Cryostat sufficiently to begin heating the sample block for temperature manipulation.

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