CHAPTER VI

Principles of Cryostat design

1. Introduction

An appropriate cryostat will have to be designed for carrying out low temperature measurements of different properties. Before embarking on the design the following questions should be answered:

(1) In what temperature range will the cryostat operate; (a) above 77 K, or (b) above 4.2 K or (c) above 1.8 K?
(2) Should the cryostat be of the bath type, continuous flow type or should it be cooled by a closed-cycle refrigerator?
(3) What is the nature of measurement to be done and the size of the sample to be used, and lastly,
(4) Would one like to perform a fixed temperature or a variable temperature measurement?

Once these questions are answered, the design of a suitable cryostat can be made with the available materials in the market and the technical expertise in the laboratory.

In this chapter certain useful principles to be followed in the design of cryostats will be discussed. Much of the material in this chapter is based on Reference 1 cited at the end of this chapter.

2. Precautions against differential contraction

The most commonly used materials in the construction of cryostats are stainless steel, copper and brass. All these materials do not become brittle at low temperatures. Stainless steel has a large strength to weight ratio and one can use thin walled tubes for construction. This reduces the weight of the cryostat. However for the sample holder and the sample chamber one uses copper. The large thermal conductivity of this material makes for a greater uniformity of temperature. Brass is also used occasionally for making vacuum cans for the sample holder. To reduce the radiation heat load, one should plate the low temperature parts with gold. This is especially recommended for very low temperature work. For electrical insulation one may use teflon in parts of the cryostat.

We see that in the construction of a cryostat one may have to use dissimilar materials. In such a case one factor that requires careful consideration is the relative thermal expansion of the different materials. The cryostat will be subjected to warming and cooling repeatedly. Differential contraction of dissimilar materials will then lead to opening of joints leading to leaks.
The cumulative thermal expansion for unit length is given by

\[ \Delta L/L_0 = \int_0^T \alpha \, dT \]  

(VI.2.1)

This data for various materials is given in Table VI.1. \( L_0 \) is the length of the sample at 0 K. The contraction of stainless steel from 300 to 10 K is less than that of copper, which in turn is less than that of brass.

### Table VI.1
Cumulative thermal expansion for unit length, \( \Delta L/L_0 \), for different materials as a function of temperature \( T \). The values have to be multiplied by \( 10^{-5} \).

<table>
<thead>
<tr>
<th>Temp [K]</th>
<th>Stainless steel</th>
<th>Brass</th>
<th>Copper</th>
<th>Aluminium</th>
<th>Nylon</th>
<th>Teflon</th>
</tr>
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<tbody>
<tr>
<td>0</td>
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<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
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<td>4</td>
<td>16</td>
<td>12</td>
<td>9</td>
<td>81</td>
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</tr>
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<td>80</td>
<td>13</td>
<td>34</td>
<td>26</td>
<td>24</td>
<td>142</td>
<td>210</td>
</tr>
<tr>
<td>100</td>
<td>32</td>
<td>58</td>
<td>45</td>
<td>46</td>
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<td>290</td>
</tr>
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<td>120</td>
<td>49</td>
<td>83</td>
<td>67</td>
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<td>301</td>
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<td>140</td>
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<td>900</td>
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<td>845</td>
<td>1110</td>
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<td>283</td>
<td>241</td>
<td>295</td>
<td>981</td>
<td>1310</td>
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<tr>
<td>260</td>
<td>240</td>
<td>320</td>
<td>273</td>
<td>339</td>
<td>1109</td>
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<tr>
<td>280</td>
<td>272</td>
<td>357</td>
<td>305</td>
<td>384</td>
<td>1284</td>
<td>1535</td>
</tr>
<tr>
<td>300</td>
<td>304</td>
<td>397</td>
<td>337</td>
<td>418</td>
<td>1450</td>
<td>1600</td>
</tr>
</tbody>
</table>
Fig. VI.1 Taking care of differential contraction effects in joints of dissimilar materials

In making a joint between such dissimilar metals one should take care to see that the material with the higher contraction is outside the material with the lower contraction as shown in Figure VI.1(a). When the joint is cooled the outer tube will contract more and squeeze the inner tube.

If the tube, made of material with a smaller contraction, is put outside a tube made of material with a larger contraction, there is a danger of solder giving way and the joint leaking at low temperatures. Similarly in using a flange of stainless steel it is better to use a brass screw to tighten the flange with a washer of material with a lower expansion coefficient than brass between the flange and the screw as shown in Figure VI.1(b). This makes the contact tighter on cool down.

In making electrical feed throughs one uses an epoxy to isolate electrically the metal leads from the flange. All epoxies have a cumulative expansion several times larger than metals. As the feed-through is cooled the epoxy contracts more than the metal. If the epoxy is filled in a hole in the flange through which the electrical lead is brought out, it will crack from the wall of the flange when the lead is cooled. The correct way to make a feed through is to braze or solder a thin walled SS tube to the flange. The lead is brought out through the tube and the epoxy is applied to cover the outside of the thin walled tube. The epoxy will then squeeze the tube when cooled and this will preserve the integrity of the
seal. Often inadequate attention to such detail will lead to cold leaks and loss of valuable time in detecting such leaks.

Sometimes the sample chamber is connected by several tubes to the top flange. In low temperature cryostats the length of such tubes is usually large. If the tubes of dissimilar materials are used, one will contract more than the other and this will lead to large stresses at the joints of the tubes. One must pay particular attention to avoid such stresses.

3. Bath cryostats

Cryostats using liquid cryogens can be conveniently classified in one of two types: bath cryostats or continuous flow cryostats. In the former the entire experimental set up is immersed in a bath and is suspended from a top flange. In the latter, the liquid cryogen is sucked by a vacuum pump and flows through the parts to be cooled.

One can make a bath cryostat either out of stainless steel or out of glass. For a beginner in low temperature experimentation, a glass bath cryostat has the advantage that one can see through the walls of the cryostat. A bath type cryostat made of glass is shown in Figure VI.2. For liquid nitrogen the glass cryostat can be made of pyrex glass. The liquid cryogen is contained in a double walled glass dewar. The space between the walls can be evacuated to a high vacuum and sealed. To reduce evaporation loss from the dewar, one may also
coat the inner wall of the outer tube and the outer wall of the inner tube with silver leaving two vertical slits opposite each other for viewing.

Helium gas slowly permeates through the walls of the glass dewar and the vacuum will be spoiled with time. Lead glass has a much lower diffusion coefficient for helium gas and so glass dewars for helium are also made of lead glass. It will be better to make a glass dewar for liquid helium work with provision for re-evacuating the vacuum space periodically and resealing it with a mechanical seal.

The top of the dewar ends in a ground glass flange. The cryostat parts hang from a metal flange that will sit on the flange of the glass dewar with an O-ring seal to make it leak tight. While using glass cryostats there is a danger of the glass dewar exploding or imploding. If the glass dewar is not properly annealed there is a danger of this happening sooner than later. As a matter of safety, a glass bath cryostat should be surrounded by a thick transparent plastic shield to protect the personnel against injury in the event of an explosion.

![Figure VI.3 A general-purpose liquid helium Metal bath cryostat](image)

Stainless steel cryostats are safer to handle from the long term point of view. Figure VI.3 shows a schematic diagram of such a general-purpose metal bath cryostat for work down to 1.8 K. A temperature of 1.8 K is reached by pumping on liquid \(^{4}\)He. In the metal
cryostat shown in figure, there is a liquid nitrogen shield surrounding the helium bath and the radiation baffles in the helium dewar prevent radiation from the top of the dewar from reaching the helium bath.

The sample chamber in the dewar can be evacuated or filled with exchange gas of helium at about one mbar pressure. If one wants to measure the temperature variation of a property like electrical resistivity, then one can evacuate the sample chamber after the sample has reached the lowest temperature and then heat the sample at a slow rate. It should be noted that the electrical leads should be made of thin (SWG 40) copper wires or manganin and the wires should be anchored at a point at 4.2 K as shown. This will reduce the heat leak through the leads.

Such a bath cryostat, about a meter in height with a capacity of ten litres of liquid helium, can be fabricated at a cost of about Rupees fifty thousand. It is important to have a bursting disc and a pressure relief valve on the outer vacuum chamber. In case there is a leak of cryogen from the nitrogen or helium dewars, the liquid will evaporate rapidly, building up pressure in the outer vacuum vessel. The pressure relief valve and the bursting disc prevent the pressure from reaching such a high value as to cause the outer vessel to explode.

In the construction of helium and nitrogen vessels one uses a stainless steel sheet (SS 304) of suitable thickness. From the point of view of reducing heat leak, one should use a sheet of as small a thickness as possible. In case the helium evaporates fast the pressure in the helium dewar will build up. The wall thickness must be large enough to withstand a sufficient over-pressure as well as the reduced heat leak. One makes a compromise between the two conflicting requirements and chooses a wall thickness to withstand an overpressure of about 5 psi. To provide for safety in case the pressure crosses this value, a pressure relief valve is provided on the top of the helium dewar which will release helium gas from the dewar when the pressure exceeds 5 psi.

When the pressure inside the dewar is more than the pressure in the outer chamber the wall of the dewar experiences a tensile force. On the other hand in the reverse situation, the wall of the dewar will experience a compressive force. For the same thickness, the wall of the dewar can withstand a higher tensile force than a compressive force. This implies that the above dewar may not withstand even a differential pressure of one atmosphere under compression. If the helium dewar is evacuated while the outer vessel is at atmospheric pressure the inner dewar will crumple. This is known as buckling. Before evacuating the inner helium dewar one should make sure that the outer vessel is evacuated. This should be taken care of during the leak checking on the dewar.

The wall thickness for the cylinders and the dished ends of the dewar both for bursting as well as buckling can be calculated using the ASME pressure vessel codes. Section VIII. (Unfired Pressure Vessels). The details are presented in Appendix and the references therein.

3. 1 Cooldown of a bath cryostat
The helium dewar must be first cooled to near 77 K before starting to fill liquid helium in it. The liquid nitrogen dewar is filled first and the helium dewar is allowed to cool by radiation. This will take several hours but will not contaminate the helium dewar.

For a more rapid cool down one may fill the helium dewar with some liquid nitrogen. Once the helium dewar is cooled to 80 K, the liquid nitrogen in the dewar should be completely boiled off before one starts cool-down of the inner dewar with liquid helium. This can be done by putting a heater inside the helium dewar and switching it on. If liquid nitrogen is left in the dewar before helium filling is started, a lot of liquid helium will be used for cool down to 4.2 K. This is because the thermal capacity of liquid and solid nitrogen is high compared to the thermal capacity of the material of the dewar.

Another point needs to be made about the cool-down with helium. One may use only the latent heat of vaporisation of helium for the cool-down or one may use the complete enthalpy of helium from 4.2 K to 300 K. For liquid helium the latent heat of vaporisation at 4.2 K is smaller by a factor of 40 than the total enthalpy from 4.2 to 300 K. If one uses a rapid cool down of the cryostat with liquid helium a lot of cold helium vapour will exit the dewar and the consumption of cryogen will be excessive. If the total enthalpy of cooling is used, the exiting helium gas will be near 300 K and the consumption of cryogen will be minimal. Though the cooling will take a longer time to achieve, it is preferable to use a slow cool down using the total enthalpy of helium. Table VI.2 gives the amount of cryogen required to cool down 1 kg of different materials.

**Table VI.2**

<table>
<thead>
<tr>
<th>Cryogen</th>
<th>From (K)</th>
<th>To (K)</th>
<th>Aluminum</th>
<th>Copper</th>
<th>Stainless Steel</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>a</td>
<td>b</td>
<td>a</td>
</tr>
<tr>
<td>Nitrogen</td>
<td>300</td>
<td>77</td>
<td>1.0</td>
<td>0.63</td>
<td>0.46</td>
</tr>
<tr>
<td>Helium</td>
<td>77</td>
<td>4.2</td>
<td>3.2</td>
<td>0.20</td>
<td>2.20</td>
</tr>
<tr>
<td>Helium</td>
<td>300</td>
<td>4.2</td>
<td>66</td>
<td>1.60</td>
<td>32.0</td>
</tr>
</tbody>
</table>

A bath cryostat has the disadvantage that it can only be used in the vertical orientation. The consumption of cryogen is more the higher the temperature at which an experiment has to be done.
4. Continuous flow cryostats

When a sample in a small volume has to be cooled and when the experiments have to be done at variable temperature, the continuous flow cryostat is eminently suitable. Figure VI.4 shows a general purpose continuous flow cryostat.

Figure VI.4  A schematic diagram of a general-purpose continuous flow cryostat
The continuous flow cryostat can be mounted in any orientation. The continuous flow cryostat consumes less cryogenic fluid, the higher its temperature of operation. The temperature can be easily controlled by controlling the flow. However, its temperature stability is good only when the flow through the cryostat is a single-phase flow. When the flow is two-phase flow then large temperature fluctuations occur. For example, with liquid helium, it is difficult to achieve stable temperatures in the range 4.2 to 15 K.

Figure VI.5 shows how the continuous flow cryostat can be used. A vacuum pump sucks the cryogen from the storage dewar through the vertical transfer tube. The cold vapour from the storage dewar passes through the coil wound on the sample chamber and cools the chamber. A radiation shield surrounds the sample chamber and is cooled by the cold vapour flowing through a coil attached to it. The coil wound round the radiation shield is made of larger diameter tubing than the coil wound on the sample chamber to take care of the increase in specific volume of the vapour on heating. The vapour leaves the shield and reaches the vacuum pump. The rate of flow of the cryogen can be adjusted by a needle valve at the inlet to the pump to ensure that the full enthalpy of the vapour is used in cooling the sample chamber. By finely controlling the flow with an electrodynamic valve, the temperature of the specimen chamber can be controlled to an accuracy of 10 mK or better.

The sample is mounted in the sample chamber which can be filled with exchange gas at low pressure to maintain uniformity of temperature. To change the sample, the flow of the cryogen is shut off, and after the sample chamber has warmed up to room temperature, the old sample is removed and the fresh sample loaded from the top into the sample chamber.
The consumption of cryogen as a function of temperature of operation of the sample chamber is shown in Figure VI.6. The higher the temperature of the sample chamber, the smaller the consumption of cryogen. The continuous flow cryostat enables one to perform variable temperature experiments with economic consumption of cryogen.

Fig. VI. 6: Schematic showing the consumption of cryogen as a function of temperature in a continuous flow cryostat

Since the continuous flow cryostat can be operated at any orientation they have been built for a variety of applications such as cooling a sample for electron microscopic, X-ray diffraction, optical and magnetic studies.

5. Closed cycle refrigerator cryostat

Where handling of the liquid nitrogen and liquid helium is not desired or obtaining these liquids is difficult, a mechanical (closed cycle) refrigerator can be used to cool down the samples. The most common commercial refrigerators make use of a two-stage GM cycle using high purity helium gas as the working fluid. The cryocoolers are discussed in Chapter III.
Figure VI.7 shows the cold head of a closed cycle refrigerator (with mechanically driven pistons), to which is attached a vacuum jacket and an adapter containing the electrical feed throughs and a pressure relief valve. The vacuum jacket surrounds the cold head. With a two stage cryocooler, the second stage cold head acts as a sample mount, while the first stage is used for mounting a radiation shield surrounding the sample. The cold finger can be oriented in any direction in the case of a GM or Stirling type Cryorefrigerators. However, for the Pulse Tube based systems, the lowest temperature would be reached when the sample mount is at the lowest position.
A typical system will reach the lowest temperature in about one hour. This will be affected by the sample size and the associated holder. Some units may exhibit a small temperature fluctuation at the lowest temperature, which can be eliminated by using an automatic temperature controller to operate at a slightly higher temperature.

6. Seals at low temperature

A seal serves to (a) close the smallest roughness in the mating surfaces, (b) to remove any unevenness that may occur on tightening the mating surfaces, (c) to distribute the pressure uniformly on the mating surfaces and (d) to maintain leak tightness under all operating conditions.

Many commonly used sealing materials at room temperature become brittle at low temperatures. Teflon gaskets can be used for sealing down to 77 K. Metals like copper and aluminium require thick mating flanges to maintain the necessary pressure for sealing. They are not therefore suitable for use at low temperatures.

The most useful sealing material at low temperature is the soft metal indium. This metal forms a good sealant because it deforms plastically to fill the O-ring groove completely. When making an indium seal it will be preferable to have the mating flanges made of the same material say stainless steel. Figure VI.8(a) to (c) shows various possible groove profiles for indium seals. The profiles are in increasing order of complexity. Figure VI.8(d) shows a simple indium O ring seal for joining two tubes. This can be easily fabricated and assembled.

![Fig.VI.8(d) A simple Indium seal](image)
A total metal contraction seal is shown in Figure VI.9. The sealing action arises here because aluminium shrinks more than copper on cooling. This type of sealing is useful if one has to have demountable windows on the cold inner dewar of a bath cryostat.

7. Optical windows

For optical experiments light from an external source will have to pass through the cold sample in a dewar. Light will enter through two windows, one on the outer vacuum shield at room temperature and the other on the cold wall of the helium dewar. Light will also exit through similar windows. While the first window at room temperature can be fixed with a conventional viton O-ring seal, the second window requires special construction.

Figure VI.10(a) shows one method of fixing the optical window using an indium seal. The indium metal ring on the front of the window serves to distribute the pressure uniformly on the window material when the bolts are tightened on the flange. It does not prevent leaks. It is the indium O-ring seal behind the window material, which prevents leaks. In this arrangement the window material is subjected to uniaxial pressure. This is unacceptable if polarised light is to be used for the studies.

One should then have a window with no load on it. Such an arrangement is shown in Figure VI.10(b). The window is fused to a tubular fixture using a glass to metal seal. This tubular fixture is joined to a concentric thin walled tube, which is then brazed on to the connecting tube on the flange. In this arrangement tightening the bolts on the flange does not cause any stress on the window material.
8. Mounts for thermometers

A resistance thermometer is fixed to the body of the sample holder by an epoxy, which is electrically insulating but thermally conducting. Any heat leaking into the thermometer from a higher temperature will warm up the thermometer. The reading of the thermometer will be erroneous.

To avoid such a heat leak the thermometer leads are put in good thermal contact with the body of the sample holder before they are led out. This ensures that the thermometer and sample holders are at the same temperature. Epoxy has a high emissivity and will absorb radiation from the high temperature parts of the cryostat to which it is exposed.

To prevent such heat from reaching the thermometer, the thermometer is put in a groove in the body of the sample holder and covered with epoxy. A radiation shield is fixed on the epoxy and anchored to the sample holder. This is shown in Figure VI.11(a).
Another way of mounting a thermometer is to put it in a copper tube, which is evacuated and filled with helium exchange gas at 1 mbar pressure. This tube is soldered to a copper plate, which is tightly screwed to the body of the sample holder. This is shown in Figure VI.11(b).

Figure VI.11 (a) and (b) Thermometers mounting Arrangements

In case one uses a vapour pressure thermometer a possible arrangement is shown in Figure VI.12. Here a small hole is bored in the wall of the sample chamber or sample holder, as the case may be, to hold a few cm3 of the liquid used for vapour pressure thermometry.
This hole is connected by a narrow stainless steel capillary to the room temperature flange. The tube can be evacuated, purged and filled with the pure gas to be used in the vapour pressure thermometer. One should use a fairly long length of capillary so that the heat leak into the sample holder is small.

![Figure VI.12 Vapour pressure bulb and capillary (Drawing not to scale)](image)

9. Different types of commercial cryostats

Commerically, experimental cryostats are available for different applications. M/s Janis Research Company Inc. is one of the major suppliers of such cryostats and the figures of some of the cryostats are shown below.

Figure VI.13(a), (b), and (c) show a detachable tailed dewar with liquid nitrogen reservoir for liquid helium experiments, an optical exchange gas cryostat, a variable temperature magnet cryostat respectively. For further details, the reader should refer to the technical literature available from the company.
Figure VI.13 (a) A detachable tailed dewar with liquid nitrogen shield for liquid helium experiments.
Figure VI.13(b) An optical Cryostat for liquid helium experiments.
Figure VI.13(c) A Superconducting magnet Cryostat for variable temperature and liquid helium experiments.
10. Transfer Siphons

One can make a simple transfer tube for transferring liquid cryogen from the storage dewar to the bath cryostat. Figure VI.5.14 shows the construction of such a U shaped transfer tube. One SS tube is put inside another and both are bent to form a U. The space between the tubes will be evacuated and sealed. Both the tubes should be sufficiently thin walled to reduce the heat leak into the storage dewar and bath cryostat.

When bending such thin walled tubes there is a danger of kinking of the tubes at the bends. To avoid this, the tubes are filled with water and the water is frozen by the use of liquid nitrogen. The tubes are then bent to the required shape when cold. On warming the ice will melt and the water can be removed.

When the cryogenic liquid flows through the transfer tube the inner tube will contract relative to the outer tube. To provide for this contraction, the inner tube is mounted slightly above the axis of the outer tube so that it has a longer length. On cooling it will contract and move down. To prevent the inner tube from touching the outer tube spacers are used.

Figure VI.14 A simple U shaped transfer tube
11. Conclusion

When one uses a closed cycle refrigerator, the sample holder should be attached in good thermal contact with the cold head of the cryocooler. One can design different sample holders for different measurements. The radiation shield for the sample holder is usually anchored to the first stage of the cryocooler. Since the space available within the outer vacuum mantle of a cryocooler is limited, the size of the sample holder becomes a constraint. Since most of the cryocoolers can be mounted in any orientation, one can design suitable sample holders for different measurements, optical, magnetic, Mossbauer etc.

The vibrations in a closed cycle refrigerator will vitiate measurements, for example in a Mossbauer set up or in a vibrating sample magnetometer. Suitable precautions will have to be taken to reduce the vibration level.

In this chapter, some general principles of cryostat design have been mentioned. Many cryostats for different measurements are described in the literature. It is not possible to describe such cryostats in detail within the purview of the book. The reader should consult the literature and decide on a suitable design of a cryostat for his studies.

REFERENCES


