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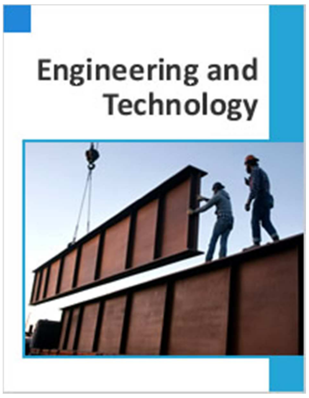

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Re-evacuation, Repair and Revival Techniques of Cryogenic Dewars

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Abstract
Cryogens like liquid nitrogen and liquid helium are the essential requirement in many areas of scientific research and technology. Liquid nitrogen has a boiling point of -196°C (77 K), liquid helium has a very low boiling point of -269°C (4.2 K) and therefore these cryogens need specialized containers for their storage and transportation. Commercial containers for storing these cryogens are of special construction. To arrest the heat leak, the containers are doubled walled annular space between the inner and outer vessel is evacuated. The heat conduction through the walls is kept a minimum with use of thin walled stainless steel material. To reduce the heat loss due to radiation, the inner tank is wrapped with multiple pairs of reflecting and insulating layers which reflects the heat radiation away. This combination of multi-layer insulation and vacuum is called super-insulation. In practice, the vacuum level of the dewars deteriorates over long period of time and needs to be revived by re-evacuation. The methods, tools, time of evacuation depend on the condition of the dewar. In-house techniques were developed and successfully re-evacuated and revived many dewars at Low Temperature Facility, Tata Institute of Fundamental Research, Mumbai. The paper throws light on the extensive experience explaining the specific techniques and methodology for the reconditioning of vacuum in the cryogenic dewars.

1. Introduction

Low Temperature Facility (LTF) of Tata Institute of Fundamental Research (TIFR) caters to the cryogenic needs for the largest number of laboratories and research users in one institution in India. The facility manages about 95 cryogenic dewars and about 65 liquid nitrogen dewars, of various capacities. Unlike other cryogenic facilities elsewhere, all these cryogenic dewars are repaired and maintained in-house at LTF. Thus the healthiness and timely availability of dewars has high importance in order to cater the user demands in an uninterrupted way. In order to maintain the “on-demand” availability of cryogens to the research laboratories, it is necessary that these non-functional dewars are re-conditioned and put back in regular usage at the shortest possible time. This paper concerns the reconditioning aspects of cryogenic dewars, with a special focus on reconditioning of lightweight aluminium alloy liquid helium dewars and liquid nitrogen dewars which do not have a provision for re-evacuate.

2. Necessity of Dewar Re-conditioning

Dewars have designed with certain evaporation rate specified by the manufacturer as Net Evaporation Rate (NER), usually it is about 1% net evaporation per day for liquid helium and about 2.5% for liquid nitrogen dewars. But, after long usage and time it tends to have larger evaporation rate than that specified by the manufacturer [1]. With time and usage the performance of the dewars deteriorate due to variety of reasons such as ageing of O-ring, degassing, micro leak in weld joints due to crack in transportation, fatigue failure due to repeated thermal cycling stress etc results in the drop in insulation vacuum of the cryogenic dewars and hence its performance. Depending on the state of deterioration, the NER increases and in worst cases, the walls of the dewar will sweat profusely due moisture condensation on the cold dewar wall. Hence, it is necessary to routinely check the performance of dewars and recondition whenever necessary.

3. Dewar Reconditioning Techniques

Normally, the dewars are reconditioned, by evacuating the double wall space and checking if it holds vacuum or not. If not, necessary repairs are carried out and the dewar is brought back into operational condition. For evacuation high vacuum pumping station comprising of two stage direct driven rotary vacuum pump with a capacity of 280lpm as backing / roughing pump along with 300lps turbo molecular pump was used. On evacuation, at the pump head it is observed with an ultimate pressure of $1x10^{-7}$ mbar, which is almost the rated value of the high vacuum pumping unit [2]. The typical evacuation setup is depicted in Figure 1.

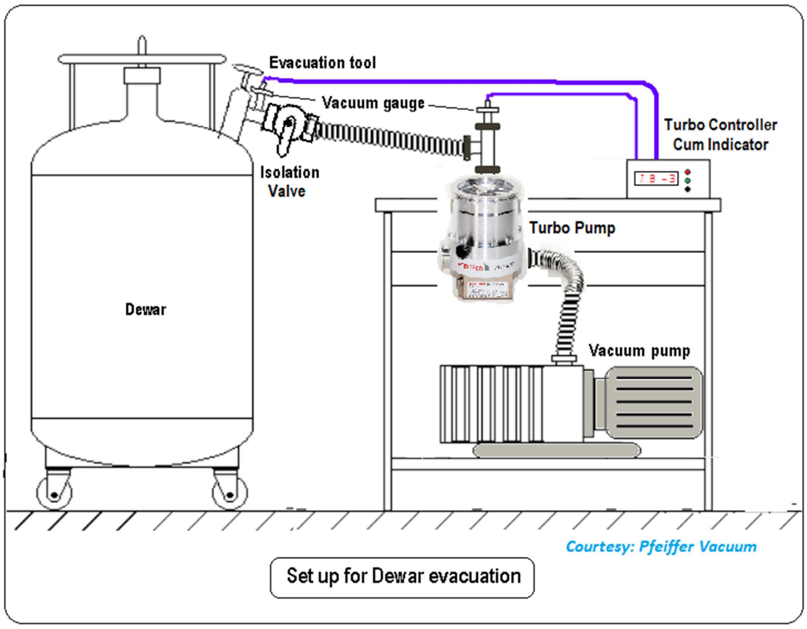

Figure 1. *Typical Evacuation Setup.*

3.1. Evacuations Tools

Dewars will be usually provided with a port for re-evacuating the insulation space. However, the type of port for evacuating purpose will vary with the manufacturers; hence the tool for evacuation will also vary. A modified plug was designed and developed for the evacuation port, so that the standard evacuation tool developed by us can be utilized [3]. Thus a standard vacuum port and vacuum plug was developed. These vacuum plugs may have one or two O-rings for the sealing. It is observed that the two O-rings ensure better reliability of the vacuum seal. A modified the evacuation port, which has two O- ring seals was used wherever required. To operate the evacuation, a universal pump-out too was developed, which will have accessibility points for flushing, charging, vacuum measuring operation along with safety fittings as shown in Figure 2. With this universal vacuum tool, it is possible for us to carry out all the required reconditioning operation conveniently, without disturbing the dewar evacuation port.

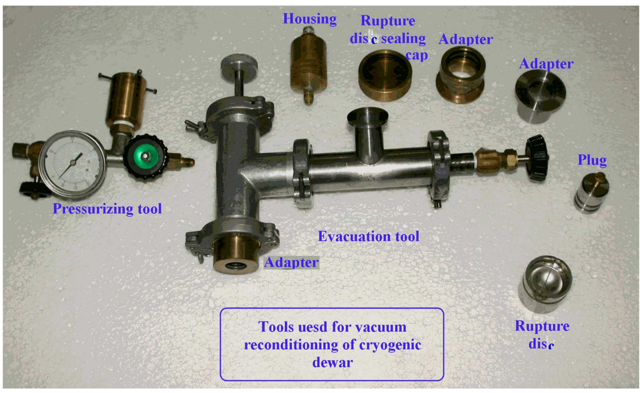

Figure 2. *Universal Evacuation Tool.*

3.2. Evacuation Procedures

In a normal condition, the vacuum level at the dewar end usually reaches a value of about $1x10^{-5}$ mbar within 3-5 hours. In some cases, the time for achieving the required insulation vacuum will be longer even after continuous pumping and in few cases; vacuum level may also stagnate around 10^{-2} mbar. Such dewars are left for two to three days and status of vacuum holding is checked. If the vacuum after four days is around $1x10^{-1}$ to $3x10^{-1}$ mbar, then such a case indicates no major leaks, but the presence of adsorbed moisture in the vacuum space. This will require flushing and purging with dry nitrogen or argon gas. Caution must be exercised in this operation as there is a danger of positive pressure building in the vacuum space. Purge gas pressure should be kept as low as 0.5 to 0.8 bar(g) with a flow control valve. Higher pressure may cause severe damage by tearing the multi-layer insulation sheets. Usually, within a three cycle of above flushing and purging the required vacuum level will be achieved. Not only cryogenic dewars, but also many cryogenic accessories like transfer siphon were successfully revived at TIFR [3]. Few snaps of the dewar and accessories evacuation process is given in the Figure 3.

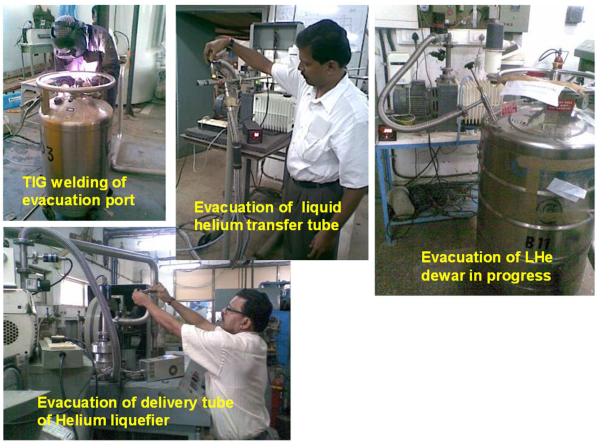

Figure 3. *Evacuation of dewar & accessories.*

For higher degrees of moisture contamination, and to degas the adsorber, outer body of the dewar is wrapped with a blanket heater. This blanket heater increases the dewar body temperature to about 80 to 90 degrees centigrade. An electrical blanket heater (size 72" x 12" x 2", 500 Watts, 230VAC) was used for this purpose. To enhance the quality, purge gas is circulated within in the vacuum space while the outer vessel is being heated by the blanket heater. If the problem of achieving the required insulation vacuum level still persists, then it indicates the possibilities of leak or crack in a weld joint of the dewar either internally or externally. To confirm the internal leakage of the dewar, the liquid space of the dewar can be filled with a purge gas well within the safety valve pressure rating, while the dewar insulation vacuum is going ON. In case of any sudden deterioration in the insulation vacuum, this indicates the possibility of purge gas from the dewar liquid space entering into the insulation space. This should also be verified by connecting the mass spectrometer leak detector (MSLD) in vacuum mode, to the dewar insulation space and small quantity of helium gas shall be sprayed inside the dewar liquid space and also the external part of the dewar such as weld joints for cracks and other suspected areas. MSLD will precisely indicate the leak if so.

4. Lightweight Aluminium Alloy Liquid Helium Dewars

The inner and outer containers of the dewars are made of aluminium alloy, hence, extremely lightweight. The typical tare weight of a 100 liter capacity liquid helium, aluminium alloy dewars would be just 35 kilograms, compared to 95kgs of similar capacity steel dewars. The inter connection joint between the inner vessel of the aluminium alloy dewars and the outer vessel is by a fiberglass (very low thermal conductivity composite material) tube, which is fixed with the help of an adhesive. The space between the inner and outer vessel is evacuated, so as to restrict the conduction and convection heat in leaks along with the multilayer insulation to restrict the radiation heat in leaks [4][5][6]. Typical construction of a lightweight aluminium alloy liquid helium dewar is depicted in Figure 4.

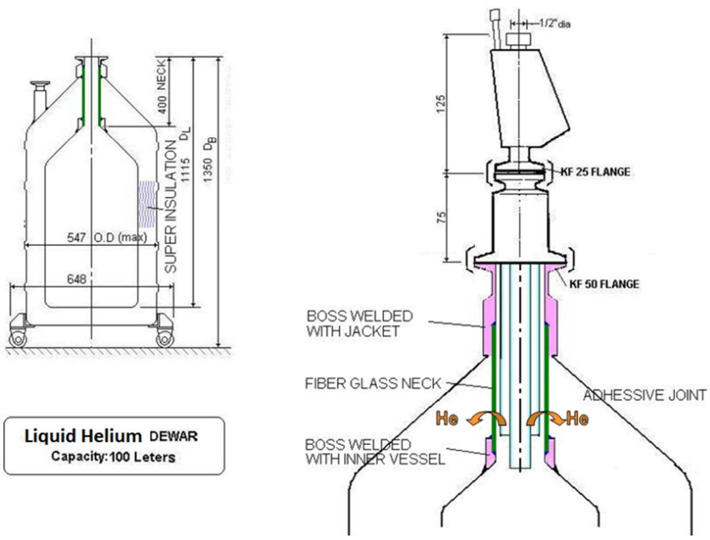

Figure 4. *Construction of lightweight dewar.*

These dewar will also have the adsorber (usually activated charcoal) material inside the insulation space to adsorb the traces of left out gases at low temperature.

4.1. Helium Permeability Through Neck

Helium gas being the lighter, the helium vapor tends to permeable through the composite fiber glass neck tube whenever the dewar becomes warm, thus the insulation vacuum deteriorates. Hence these aluminium alloy dewars are much fragile compared to that of a stainless steel type cryogenic dewars. To avoid this helium permeability into the insulation space, it is essential to keep the dewar always in the liquid helium temperature. Since, it may not be always possible to keep it cold, usually the liquid space is evacuated (about 50 mbar) and keep it in the under pressure, till the dewar is refilled again. This will avoid the helium gas permeation into the insulation space through neck tube.

4.2. Re-conditioning of Aluminium Dewars

The insulation vacuum of the dewars may deteriorates over longtime and needs to be strengthened by re-evacuation. After visual checking for any external damage, the dewar insulation space is evacuated using the required evacuation tool (pump out tool). After about 8 hours of continuous evacuation, the port is isolated, by which usually$1.4x10^{-5}$ mbar is achieved near the evacuation tool end / dewar end. Many such light weight dewars were successfully revived and put back into operation. The developed techniques iincludes, modified the evacuation plug from the single O-ring sealing as given by the manufacturer, to the double O-ring conical plug for better sealing and enhanced reliability in leak tightness. Few photos of the light weight dewar revival process and the modified evacuation plug are given as Figure 5.

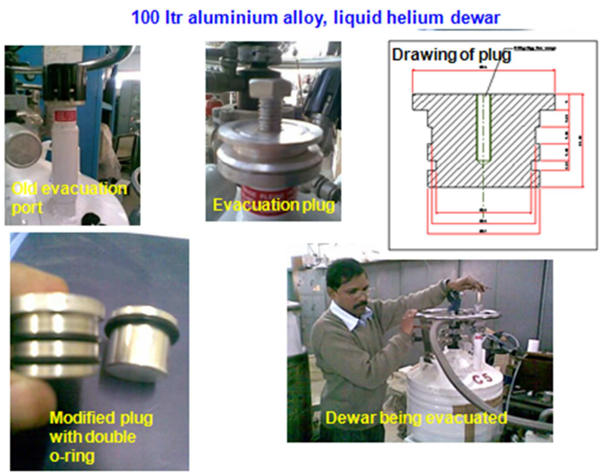

Figure 5. *Modified evacuation plug.*

4.3. Rectification of Internal Leaks

If the leaks are traced externally to the dewar, weld cracks if any, is rectified by TIG welding and the above evacuation procedures are carried out to recondition the dewar. However, in case of MSLD confirm the existence of dewar internal leaks, the remedial actions for its rectifications are very difficult as the parts are beyond the accessibility for any traditional repair process like re-welding etc. It may also be noted that almost all the internal leaks are seen either at the joint of dewar neck tube with outer vessel or at the joint of neck tube and internal vessel. The first location is comparatively of easier access to that of other location. These internal leaks can be managed by applying the standard epoxy adhesive at these internal joints. The epoxy adhesive in a molten form is sparingly applied on the suspected internal joints, while the insulation vacuum is going on. The adhesive is expected to penetrate into the porosity holes and gets trapped due to the vacuum on the other end. The adhesive after sufficient curing time will become harder and plug the leaks. This process of applying the adhesive may be repeated for two more times, to ensure the better leak tightness. Araldite®, a standard epoxy adhesive (resin & hardener) was used. However, this method will work effectively for dewar having porosity leaks/ hair crack / cold leaks only. Being, this method of dewar revival depends on many factors, such as the location leakage area, size of the leakage area, quality of the adhesive, its consistency, insulation vacuum level the results may not be consistent, but is a method of attempt. By this technique two dewars having internal leakages were successfully revived at TIFR.

4.4. Cold Leak Repair by Removing the Outer Shell

An attempt to rectify the cold leak of lightweight aluminium alloy, liquid helium dewar was made recently. The dewar was observed to have cold leak, which was beyond retrieval by the regular evacuation techniques. The dewar outer vessel was cut open the dewar for its further analyzing and repairing. Upon pressure test and MSLD leak test of the dewar, the leak (in the order of 10^{-6} mbar.lit/sec) was observed at the fiberglass neck tube joint. The leak was plugged using STYCAST® cryogenic Epoxy compound. The leak was successfully plugged and leak tightness was ensured with MSLD leak detector. The dewar is currently being repacked and welded for its testing. The figure 6, shows the repair process of lightweight aluminium alloy dewar.

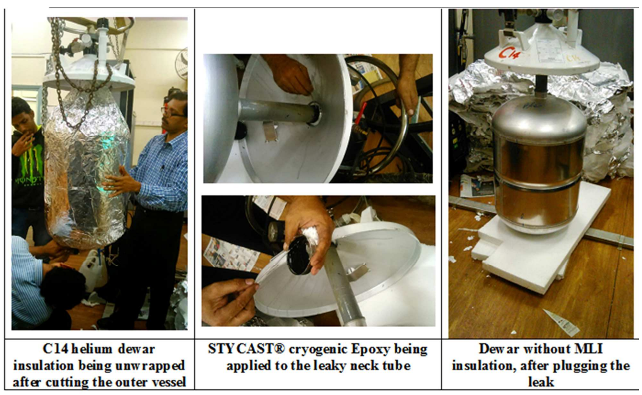

Figure 6. *Cold leak repair of aluminium Dewar.*

5. Dewars Without the Evacuation Port

There are many liquid nitrogen dewars which do not have a provision to evacuate it, as the vacuum sealing are with pinched off port. The old port had to be cut off and a new standard port is welded in the same place, so that the dewar can be re-evacuated. Enough precautions are take care, while doing removing the pinch off port. A small cut is made in the copper pinch tube to release its vacuum and to prevent pressure build up during de-brazing. Then the pinched-off copper tube seal is cut-off manually with a baby saw and surface near the opening is filed and polished using emery paper. A new SS evacuation port is TIG welded in the above opening [7]. Enough care and precautions are taken in protecting the inner vessel by providing local cooling at during the welding to avoid the damage to the multi-layer insulation around the weld area. The welding of evacuation port and the process of dewar revival is depicted as Figure 7. About eight dewars of pinched vacuum seal type were successfully reconditioned with the standard re-evacuation ports.

Figure 7. Revival process of a nitrogen dewar.

6. Testing of Repaired Dewars

It is highly essential that the dewars after repair needs to be tested for its satisfactory performance complying to the stipulated Net Evaporation Rate (NER), quantifying the cryogen boil-off rate per day in terms of its total liquid capacity. Typically the laboratory liquid nitrogen dewars are in the range of 2 to 2.5% NER and the liquid helium dewars of 60 to 100 liters capacity should be around 0.8 to 1% NER. This test bench is designed to check the NER of the dewars by measuring the quantity of boil-off gas through an appropriate gas flow integrator. Typical test setup made by us for the testing of any cryogenic dewars is schematically represented as Figure 8.

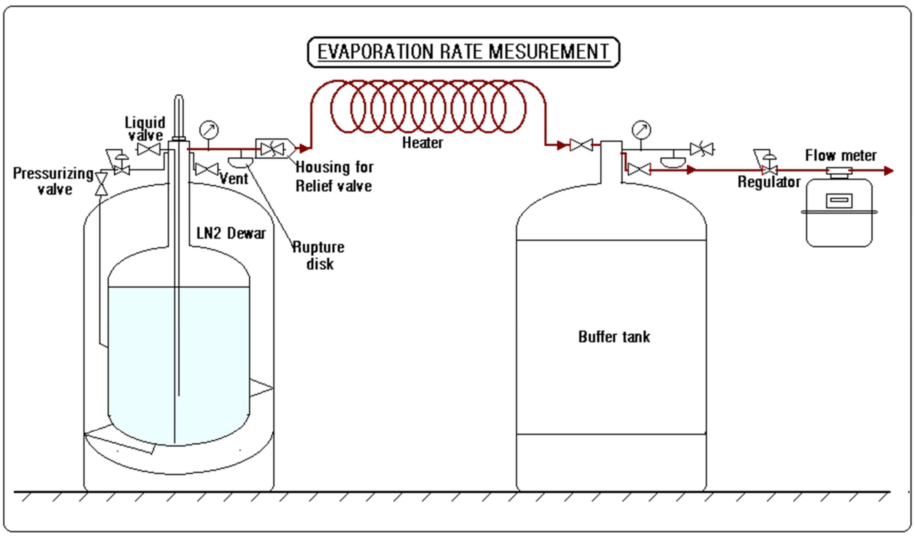

Figure 8. Test setup to check the dewar NER.

7. Conclusion

Using the above reconditioning methods and procedures, more than 47 dewars were successfully revived and reconditioned in a period of 8 years. Many of the dewars were as old as 20 years old, were reconditioned. All these dewars are in regular service without any major problems. This techniques of evacuating are now being extended to not only cryogenic dewars but also to various other accessories of such as liquid helium transfer siphon, liquid helium co-axial transfer tube of helium liquefier, liquid helium transfer pumps etc.

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