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Leak Testing of Cryogenic Components – Problems and Solutions

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Abstract. A prototype of Cold Neutron Source (CNS) for Dhruva Reactor is being manufactured at Centre for Design and Manufacture (CDM), BARC, Mumbai for validating the mechanical and thermal engineering design aspects, besides checking the integrity of all joints and components at low temperature, 77K. Task of a Cold Neutron Source is to generate cold neutrons by cooling down the thermal neutrons, which are originally produced in a nuclear research reactor. The complete Cold Neutron Source system comprises a complex arrangement of moderator pot, transfer line (piping), pumps, refrigerators, storage tanks, a heat exchanger and associated controls and instrumentation. The heart of the system is moderator pot in which water (moderator) is cooled down by Liquid Nitrogen (LN2) being circulated through an annular cavity machined on the walls of the pot. Transfer lines for LN2 basically consist of two concentric Stainless Steel flexible pipes, which are joined to the inlet and outlet Aluminium tubes of the moderator pot through transition joints. Leak in any component may result in loss of liquid Nitrogen, degradation of vacuum, which in turn may affect the heat removal efficiency of the source. Hence, leak testing was considered a very important quality control tool and all joints and components were subjected to helium leak test using mass spectrometer leak detector (MSLD) at cryogenic temperature. During one of the earlier experiments, flow of LN2 through inner flexible pipe of the transfer line resulted in rise of pressure in the vacuum annulus and sweating on the outer flexible pipe. After investigations it was found that large thermal stress compounded with mechanical stress resulted in cracks in the inner pipe. Accordingly design was modified to get leak proof transfer line assembly. Further, during leak testing of thin wall moderator pot, gross leak was observed on the outer jacket welded joint. Leak was so large that even a small amount of Helium gas in the vicinity of the moderator pot was driving the MSLD out of range. Since it was very difficult to locate the leak by Tracer Probe Method, some other technique was ventured to solve the problem of leak location. Finally, it was possible to locate the leak by observing the change in Helium background reading of MSLD during masking/unmasking of the welded joints. This paper, in general describes the design and leak testing aspects of cryogenic components of Cold Neutron Source and in particular, the problems and solutions for leak testing of transfer lines and moderator pot.

1. Introduction

In a nuclear power plant the heat produced is the desired result whereas in research reactor only the neutrons are of interest. A research reactor is constructed in such a way that in addition to the neutrons needed to maintain the chain reaction as many as possible, neutrons are available for physical and
Some of the neutrons are directed to various experimental facilities by means of beam tubes. Depending upon the temperature of the moderator (graphite, water, fluid deuterium) one gets- cold, thermal and hot neutrons having properties given in ‘table 1’. Primarily, any research reactor produces thermal neutrons which may be converted to cold or hot by installing a cold or hot source respectively inside the beam line of the reactor. Hot neutrons are needed especially for scattering experiments on fluids whereas cold neutrons having large wave lengths are used for studying large objects rather than the atomic level studies. The experiments are chiefly conducted by using the triple axes, time of flight, back scattering spectrometer as well as diffractometer and neutron spin echo spectrometer.

| Source | Energy E (meV) | Temperature T (K) | Wave Length λ (Å) |
|--------|----------------|-------------------|-------------------|
| Cold   | 0.1-10         | 10-120            | 30-3              |
| Thermal| 10-100         | 120-1000          | 3-1               |
| Hot    | 100-1000       | 1000-6000         | 1-0.2             |

A cold source, that is equipped with a moderator, cools down the neutrons and converts thermal neutrons into cold neutrons. The complete Cold Neutron Source (CNS) system comprises a complex arrangement of moderator pot, transfer line (piping), pumps, refrigerators, storage tanks, a heat exchanger and associated controls and instrumentations, refer ‘figure 1’. The moderator pot, surrounded by a vacuum vessel is located inside the reactor, in line with the thermal neutrons beam, whereas other components are situated outside the reactor. Water inside the pot acts as a moderator and brings down the temperature of thermal neutrons passing through it. Water is cooled down by liquid nitrogen being circulated through annular cavity, machined on the outer wall of the pot.

![Figure 1. Cold neutron system with fluid flow loop.](image)

Vacuum vessel (jacket) spatially isolates and thermally insulates the moderator pot and its LN₂ lines from the much warmer, atmosphere of the beam tube. Vacuum vessel also provides barrier to the invasion of water from beam tube to moderator pot and also prevents the escape of LN₂ into the beam, in case of leakage in either system. Continuous monitoring of the vacuum exhaust from the vessel permits prompt detection of any LN₂ leakage from the moderator pot and feed lines. In case of shut down and power failure vessel is filled with Helium gas which helps in achieving vacuum at faster rate as compared to air filling, at the time of restart. Helium gas also acts as an insulator and doesn’t allow LN₂ into the feed line to boil off thus CNS remains at low temperature even during shut down.
A prototype set up has been manufactured at CDM for carrying out experiments, refer ‘figure 2’. In this set up two Dewars are used for the circulation and recovery of LN₂. Nitrogen gas cylinder is connected to the input Dewar and high pressure N₂ gas forces out LN₂ from the input Dewar into the cooling loop. LN₂ is recovered when high temperature LN₂ is returned to the output Dewar. Flow of the LN₂ in the loop can be reversed simply by connecting the high pressure N₂ gas cylinder to the output Dewar and vice versa. Heat load is simulated by fixing disc type heater on the surface of the moderator pot.

**Figure 2.** Prototype set-up for cold neutron source

### 2. Design considerations

Components of CNS must withstand exceptional service conditions such as – nuclear environment, cryogenic temperature and large temperature variation. The major considerations are the need to minimize the generation of nuclear heat inside the body of the pot, removal of heat, large service life and neutron economy. The moderator pot sees the most demanding service conditions of all components of a CNS. Since its size is dictated by the size of the neutron beam line, accommodating the nuclear heating load (neutron flux = 1.8x10⁹ nm⁻² s⁻¹) within such a small volume of moderator is a major challenge. The density of construction material of the pot is a major factor in the generation of unwanted nuclear heat in the source. Since a cold source is isolated from its surroundings by a vacuum annulus to minimize heat conduction between them, the nuclear heat generated in the pot must be removed by the moderator at low temperature which in turn is cooled by cryogenic liquid such as liquid Nitrogen. Therefore, other parameters such as thermal conductivity which determines the rate of removal of heat and the thermal expansion coefficient which bears on the thermal stresses produced in the pot become important from the cryogenic point of view. From a physics point of view, the priorities are low cross sections for neutron absorption and scattering to minimize losses of neutrons within the source [1]. Further loss of ductility at low temperature, nuclear irradiation, radiation growth, swelling and compatibility with moderator medium are other important factors which have been considered while selecting Aluminium alloy 5052 as the material for the moderator pot. With regard to
manufacturing point of view Al 5052 is workable, machine-able and weld-able. Since austenitic Stainless Steel gives satisfactory performance for low power cold neutron source, other components which reside in low neutron flux area, have been manufactured out of SS 304 so that they can provide firm support to the moderator pot inside the vacuum vessel.

3. Problems and hazards associated with cryogens
The field of cryogenic involves the temperature below 93K since the normal boiling points of the so called permanent gases such as helium, hydrogen, neon, nitrogen, oxygen and normal air remain below 93K. Cryogenic liquids (known as cryogens) are liquified gases that are kept in their liquid state at very low temperature due to application of high pressure. Liquid Nitrogen is the most commonly used cryogen and is legally purchasable around the world. Liquid Helium is also commonly used for attaining the lowest possible temperatures.

Some of the typical problems and hazards associated with cryogens, which must be considered while handing them, are listed in the following paragraphs:

- Cryogenic liquids and their associated cold vapours and gases can damage living tissue (thermal burn or frost bite). The eyes are particularly vulnerable.
- As regard to mechanical properties, they embrittle the materials having high nil ductility transition temperature (NDTT).
- Since cryogenic gases are usually heavier than the air, they result in rapid displacement of air and potential for asphyxiation by reducing the concentration of Oxygen in the air, if released in confined areas.
- When exposed to the atmosphere they condense moisture in air creating a highly visible dense fog that dissipates with warming. They are capable of condensing atmospheric Oxygen causing Oxygen entrapment and unexpected Oxygen enriched atmosphere.

4. Leak testing of transfer lines
Cryogenic liquid containers and transfer lines are specifically designed to withstand rapid temperature changes and extreme differences in temperature. They are non-pressurized, vacuum jacketed vessels or pipe, refer ‘figure 3’. As per the initial design at CDM, the transfer line basically consists of two concentric Stainless Steel pipes welded together with each other at both the ends. Major part of both the pipes consists of flexible metallic hose (FMH) to accommodate any kind of bends and thermal

Figure 3. Transfer line with inner and outer flexible metal hoses/pipes.
pump through the side port for evacuating the annular space between the pipes. Outer surface of the inner pipe is completely wrapped with the foil of insulating material such as Mylar to minimize the heat loss due to conduction. Transfer lines are connected to the inlet and outlet Aluminium tubes of the moderator pot through transition joints and they facilitate transfer of LN$_2$ from Dewar to the pot and vice versa.

To ensure the complete leak tightness at cryogenic temperature, leak testing of transfer lines are carried out at following three stages:

- **Leak testing of individual pipe/bellow-** All the pipes/bellows supplied as free issue materials were tested individually to ascertain their leak tightness. This assures elimination of any leaky component before it is welded to the assembly.

- **Leak testing of welded joints-** After welding of inner pipe and outer pipe, each welded joint and repair welding, if any, are subjected to leak testing.

- **Leak testing at cryogenic temperature –** Once the complete assembly of the transfer line is found acceptable during preliminary leak testing, transfer line assembly is leak tested at cryogenic temperature. For this the inner pipe is filled with LN$_2$ and the annular space is connected to MSLD through the side port. Helium is sprayed externally on all outer surfaces of the outer pipe including welded joints to check the failure of pipe/joint due to thermal stresses developed as a result of large temperature difference. Any drop in the vacuum of annular space indicates the failure of inner tube.

### 4.1 Failure of transfer line at cryogenic temperature

After final leak testing a few of the acceptable transfer lines were used for carrying out experiments in which liquid Nitrogen was passed through the inner pipe (12 mm diameter) to study the flow pattern, temperature profile, quantity of LN$_2$ required and other physical parameters. During experiment, sweating was observed on the outer surface of the external pipe, indicating leak in the inner bellow pipe, in one of the transfer lines. Condensation of moisture on the outer bellow pipe could have been due to low temperature N$_2$ gas leaking out from the inner bellow pipe to the annular space. Prima facie it appeared that crack must have developed due to the ductile to brittle transition at low temperature, compounded with the stress as a result of cold forming of the bellow pipe. As an immediate solution next lot of transfer lines were heat treated to remove the stresses. However, even after stress relieving one of the transfer lines failed giving similar type of indications. To investigate the matter further failed transfer line was removed from the assembly and first of all Helium leak testing was carried out to find out the exact location of the leak or crack. In this test, initially inner bellow pipe was connected to the MSLD with its one end plugged. Vacuum could not be achieved until the side port of the outer pipe was closed. Similarly when annular space was evacuated, in order to achieve vacuum, both the ends of inner bellow pipe had to be closed. These two leak tests confirmed the existence of leak in the inner pipe hence it was removed by cutting the welded joints at both ends. Since in the earlier leak testing, vacuum could not be achieved, leak was located by simply passing the compressed air inside the inner bellow pipe. Further, liquid penetrant test and radiographic test were carried out to find out the exact shape, size and numbers of cracks in the bellow pipe, refer ‘figure 4’.

![Figure 4. Inner flexible metallic hose/pipe of transfer line with an enlarged view of the defective portion showing cracks detected by liquid penetrant test.](image-url)
4.2 Failure analysis and design modification
Investigations revealed that during experiment, this particular transfer line was bent in U shape to facilitate the pouring of LN$_2$. During the passage of LN$_2$, due to very low temperature, inner bellow pipe contracts and exerts force on the outer bellow pipe since both are welded together. In free condition any expansion or contraction of inner bellow pipe can be accommodated by the outer bellow because of its flexibility. However in case of U shape the outer bellow pipe is stretched to its maximum length preventing any contraction of inner bellow pipe and very high thermal stresses resulted cracks at the end of inner bellow pipe. Accordingly, design was modified to solve the problem of crack resulting from high thermal stress. In the modified design, single piece of SS pipe, bent at 90° is used instead of full length bellow. Outer jacket consists of SS pipes, large diameter elbow (to facilitate entry of inner bent pipe) and two small bellows (for accommodating contraction or expansion of inner pipe) all welded together to match the shape of the inner pipe. Transfer lines with modified configuration have been successfully used during the mock up trial without any problem of leakage.

5. Leak testing of moderator pot
Aluminium moderator pot having overall size of 194 mm diameter, 54 mm height and 2 mm wall thickness consists of an inner chamber and an outer jacket. Inner chamber is divided into three compartments by two heat sinks in the shape of disc. For the purpose of extracting heat from the interior, heat sinks are welded from inside to the wall of the pot. Water in the central region is expelled out through the central holes taking care of any increase in the volume of water after the formation of ice. A cylindrical jacket is welded to close the annular cavity, machined on the outer side of the wall of the pot. This welded joint was found leaking during Helium leak test by vacuum method.

Normally, prior to high sensitivity leak testing method such as Helium leak test (HLT), soap bubble test, a low sensitivity method is preferred, for components expected to have gross leakage. These components may degrade the vacuum of spectrometer of MSLD which operates in the pressure range less than 10$^{-3}$ mbar. However with the advent of new MSLD equipped with contra or twin flow mode, now it is possible to check components having gross leak even in the range of 10$^{-2}$ mbar l s$^{-1}$. Hence moderator pot was not subjected to soap bubble test which suffers from the inherent limitation of not being able to quantify the leak.

5.1 Leak testing problems and their solutions
During HLT of pot, even in contra-flow mode leak location became very difficult because MSLD was going out of range as soon as tracer probe was brought near the welded joint for spraying the helium, refer ‘figure 5’. This was an indication of gross leak or combination of more than one leaks through which large amount of Helium was going into the spectrometer. In order to solve the problem a new

![Figure 5. Helium leak testing arrangement for moderator pot and an enlarged view showing a through leak (encircled) on the weld.](image-url)
technique ‘leak location by masking’ was developed in which suspected area of leak is completely masked using small segment of cello tape.

During unmasking, removal of tape from any small segment, followed by an increase in the background indicates the presence of leak in that particular segment. In case of moderator pot initial background of $8.0 \times 10^{-8}$ mbar l s$^{-1}$ changed to of $9.8 \times 10^{-11}$ mbar l s$^{-1}$ after the weld was completely masked using a cello tape. While removing tape, at one location background increased to $2.6 \times 10^{-8}$ mbar l s$^{-1}$ indicating a leak in that particular segment. After restoring the mask, other areas were unmasked one by one and one more leak was observed, which changed the background from of $9.8 \times 10^{-11}$ to $6.7 \times 10^{-8}$ mbar l s$^{-1}$. Thus leaks were located and pot was sent for repair and re-welding.

6. Leak testing of transition joint
In any mechanical system, sometimes situation demands for a transition joint, consisting of two dissimilar materials, which offers entirely different physical properties and mechanical strength on each side of the joint. In CNS, components residing inside the beam line are of Aluminium, whereas outside components are fabricated out of SS 304, for the reasons explained in the preceding paragraphs. Both SS and Aluminium in the form of tubes are interfaced together with the help of a transition joint. Because of difference in melting temperature and thermal conductivity it is difficult to join them together by using a conventional welding process. Hence diffusion bonding and friction welding, in which joining is carried out below the melting temperature of the materials involved, were ventured to solve the problem. In diffusion bonding, two pieces are joined together under the application of high pressure, temperature and vacuum [2]. Friction welding is a process in which two mating parts are pressed against each other while one of them is rotating at very high speed [3].

Two cylindrical pieces of Aluminium and Steel having same diameter (30 mm or 16 mm), refer ‘figure 6’, were joined at the flat faces by the process of diffusion bonding and integrity of the joint was checked by ultrasonic normal beam contact test method. In spite of having shown adequate mechanical strength in tensile test, the joint was showing interface echo equivalent to lack of fusion, during ultrasonic test. This interface signal was attributed to the metallic layer used at the interface in diffusion bonding process. Due to process limitation of UT, leak test was suggested for the transition joint to satisfy the requirement of functional test. Since the joint was not amicable to HLT, a 10 mm diameter hole, slightly deeper than the interface was drilled from either side, at the center of the flat face for conducting HLT. Leak tightness was checked by connecting the flat face to the MSLD (vacuum mode) and helium was sprayed externally on the interface. Since the performance of diffusion bonding was not proven at low temperature, transition joint was subjected to thermal cycles, in which component was kept at ambient temperature for 5 minutes after dipping it into LN$_2$ for 5 minutes and the process was repeated for 5 times. After 5 thermal cycles it was again leak tested and sent for machining, if found acceptable. Though after thermal cycle none of the joints failed, initially during development of bonding process few joints were found unacceptable in HLT. As per the feed back bonding parameters were modified which lead to leak tight transition joint suitable for cryogenic application.

Figure 6. Transition joints, welded in the LN$_2$ lines.
7. Conclusion
Cold neutron source installed in the beam line of a nuclear research reactor uses cryogenic liquid to shift the energy spectrum of neutrons from thermal region to cold region. To check the integrity of all joints, components of prototype CNS were subjected to Helium leak test at cryogenic temperature. Failure analysis carried out for one of the transfer lines revealed that flexible metallic hoses in fully stretched condition were not able to sustain the thermal stresses due to large temperature difference at cryogenic temperature. Accordingly design was modified in which flexible metallic hoses were replaced with SS pipe having required bends and a small portion of bellow for flexibility to accommodate thermal expansion. Further, gross leakages in the welded joint of thin walled Aluminium moderator pot, were posing problems during Helium leak test by Tracer Probe Technique. A suitable technique for the leak location was developed in which initially weld is masked using cello tape and any increase in the helium back ground reading of MSLD during unmasking of the weld, indicates the leak location. Diffusion bonding and friction welding processes have been used for developing transition joints between dissimilar materials like SS and aluminium for cryogenic application. During mock up on prototype assembly, integrity of all the components was found satisfactory.

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