Design, Development and Testing of Vacuum Compatible Seals at Cryogenic Temperature

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Abstract

De-mountable joints are one of the major requirements in cryogenic assembly, especially in hydraulic connection. Different methods have been adopted for this purpose. Some demountable joints are also commercially available, but not economically viable solution for laboratory experiments. Therefore, a cost effective solution have been worked out for routine laboratory experiments, so that permanent joining processes can be avoided in order to save assembly/de-assembly time as well as avoiding permanent damage to the piping. Making a demountable type joint for cryogenic experiments require special skill and technique. The technical requirements for such a de-mountable type joint is very stringent, to mention a few are; Compatibility with cryogenic temperature as per use of cryogenic fluids (Liquid helium 4.2 K, Liquid Nitrogen 77 K), Fluid position with respect to the seal (inside flowing condition, outside bath condition) vacuum compatibility and above all consistency and repeatability of making the demountable joints with high level of operational reliability. The design of such seals also require a reliable analysis as thermal stress developed across the joint, due to differential thermal contraction of dissimilar metals, may lead to fluid leakage in the system, which is not desirable.

Indium wire seals have been extensively used at cryogenic temperature for sealing vacuum Cans, mainly because of its fluid flow properties in pressure condition. However, no reference was found where such seals have been used in demountable joints with fluid flow condition in hydraulic lines. The joint developed with indium, as sealing material remains vacuum compatible at liquid nitrogen as well as at liquid helium temperature. The joints even withstood thermal shock test with ten repeated cooling to LN_2 and LHe temperature and consequent warm up to room temperature. The leak rate measurement at 77 K and 4.2 K showed no detectable leak above the helium background level in the order of 1×10^{-9} mbar- l/sec during testing. The paper will describe the details of design consideration and concept, engineering and special techniques involved as well as test results at 77 K and 4.2 K of the developed seal.

1. INTRODUCTION

There are various method of joining two mating surfaces to prevent a leak, but at low temperature the sealing between different or same materials require a skill while designing such seals, in order to access the parts of an assembly that may need service of breakable or demountable joints. The type of joints chosen depends upon the operational and service requirement. We have developed a seal, which is vacuum tight at liquid nitrogen and even at liquid helium temperatures and have tested for our electrical isolators and current lead experiments at LHe temperature.

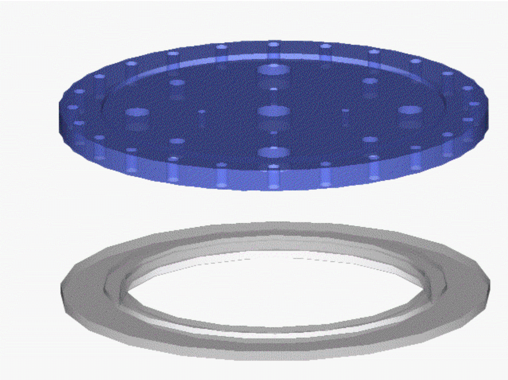
There are basically two types of seals, namely permanent seals and demountable seals. Metal parts are joined permanently by welding or brazing, glass to glass is joined permanently by fusion, glass to metal and ceramic to metal seals are constructed by using specific techniques. Table 1 represents the properties of indium seal material.

Table 1. Physical constants of pure Indium

Atomic weight	*114.82*
Density	*7.30 gm/cc*
Melting point	*156.6 C*
Boiling point	*2080 C*
Coefficient of thermal expansion	*Linear 24.8μm/m.K at 20 C*
Latent heat of fusion	*28.47 KJ/Kg*
Latent heat of vaporization	*1959.42 KJ/Kg*
Thermal conductivity	*83.7 W/m. K at 0 C*
Electrical resistivity	*84 nΩ-m at 20 C* *Super conducting at 3.38 K*
Tensile strength	*At 295 K: 1.6 Mpa* *At 76 K: 15.0 Mpa* *At 4 K: 31.9 Mpa*
Compressive strength	*2.14 Mpa*
Hardness	*0.9 HB*
Elastic modulus at 20 C	*12.74 Gpa in tension*
Poison ratio at 20 C	*0.4498*
Magnetic susceptibility	*Volumetric: 7.0x 10 E-06*

2. INDIUM SEAL DESCRIPTION

Numerous materials have been used as gasket for low temperature seals. The most consistent successful seals have been made with indium wire. It deforms easily to the general contour of the mating faces and remains relatively soft at low temperature. Normally it is fitted in a pre-machined groove on one of the flanges, the size of the groove and the diameter of the wire controls the flash width and pressure intensity on the seal face for a given clamping load. The joints ends can be made with an overlapping scrarf or butt joints, but since the end joints are usually made manually in-situ, a more accurate joint can be made by butting while angular inaccuracies in a scrarf joints can cause problem. The indium seal is not recommended between the faces which are subjected to high pulsating load, as the hammering causes the sealing flash to increase in width and decrease in thickness, thus lowering the pressure intensity on the sealing face and reducing the bolt tension.



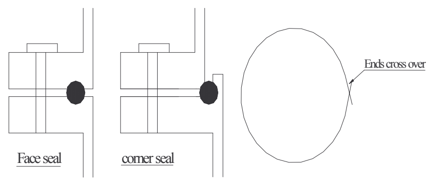


Figure 1. Schematic diagram of indium seal (different arrangement)

3.FABRICATION PROCEDURE

For the joints, which can be removed and replaced easily using a thin indium wire to form the seal. The wire is compressed to close contact with the two surfaces that are to be sealed together. We have used 0.5 mm and 2.2 mm diameter indium wire for making a seal. The seal positioned in a corner of two mating flanges, by bending one end of the wire sharply outwards and laying the other end across the corner of the bend, and by overlapping the indium wire. The wire is so soft that the joint will be compressed into a cold weld.
The manufacturing procedure of the seal is listed below.

- Before making the seal the mating surfaces should be thoroughly cleaned with fine emery paper and wiped off with acetone. Old indium wire should be removed from the seal faces.
- Keep the rounded piece of indium wires on the male spigot of the flange and overlap as shown in figure1.
- Marks should be made on flange for alignment to indicate the correct orientation. Two flanges should be brought together and hold loosely while putting the bolts and should be hand tightened only. Bolts later should be tightened with a small spanner or Allen key.
- For best result larger seals (typically more than 50 mm diameter) are recommended to leave the joints for about an hour. The indium flows during this period.
- After an hour all the bolts should be tighten slightly more.
- Using jacks can separate large seals or notches provided on the flange while inserting sharp blade between the flanges can separate smaller joints.

4. TEST METHOD AND RESULTS

We have developed an indium seal joint for our cryogenic experiments mainly for electrical isolators testing at 4.2 K and for current lead test.
The indium seal joints have undergone for the helium leak test under following conditions.

- Room temperature leak test of the indium seal joints
- Helium leak test after repeated thermal shock test at liquid nitrogen temperature
- Helium leak test of indium seal at Liquid nitrogen temperature in flow condition.
- Helium leak test of indium seal at Liquid helium temperature in flow condition.

4.1 ROOM TEMPERATURE LEAK TEST OF THE INDIUM SEAL JOINTS

We have tested about twenty numbers of indium seal joints at room temperature in order to check the helium leak rate from the seal joint. The average measured leak rate of all seals of order better than 4.0×10^{-9} mbar-l/s by the helium leak detector. For testing the seals, silicon vacuum compatible tubes have been used for vacuum connection to avoid welding etc.

Figure 2 a. complete views of Indium seal joints (end connection KF type)

Figure2 b. complete views of Indium seal joints (end connection tube type)

4.2 HELIUM LEAK TEST AFTER REPEATED THERMAL SHOCK TEST AT LIQUID NITROGEN TEMPERATURE

The same indium seals underwent 4 temperature cycles at liquid nitrogen temperature, by immersing into liquid nitrogen bath and warming up at room temperature then seals have been checked for helium leak rate, no leak was observed in any seal joints. The measured average helium leak rate was observed of the order 2.0×10^{-9} mbar-l/s.

4.3 HELIUM LEAK TEST AT LIQUID NITROGEN TEMPERATURE FLOW CONDITION:

The same seal underwent 5-temperature cycles at liquid nitrogen temperature, as LN_2 was passed through the seal and helium background was better than 10^{-9} mbar-l/s. The helium leak detector was installed online to check helium background at 77 K. Figure 3 shows the test scenario of indium seal joint in the isolators test assembly.

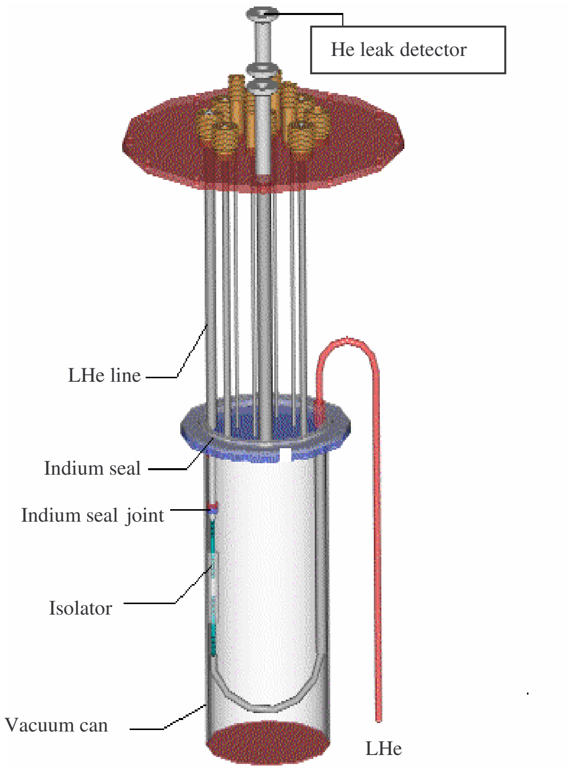


Figure 3. LN_2/LHe test assembly for indium seal joint testing

4.4 HELIUM LEAK TEST AT LIQUID HELIUM TEMPERATURE FLOW CONDITION:

The same indium seal has been tested in same test assembly set up as shown in figure 3 for more than 10 cycles at liquid helium temperature. The seal is connected in line with the isolators set up loop. The indium joint was also tested at 4.2 K during isolator test and helium background was better than 10^{-9} mbar-l/s.

5. CONCLUSION

The experimental results of testing ten demountable, cryogenic indium seals after two complete mounting and demounting cycles and 10 cryogenic temperature cycles, five of which were at 4.2 K and five of which were at 77 K, showed that the measured helium leak rate of the seals was better than 10^{-9} mbar-l/s. The indium seal joints meet the requirements and can be used in any LHe & LN_2 transfer lines or low temperature assembly testing.

6. REFERENCES

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