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Abstract. High-pressure research is one of the fastest-growing areas of natural science, and one that attracts as diverse communities as those of physics, bio-physics, chemistry, materials science and earth science. In condensed matter physics there are a number of highly topical areas, such as quantum criticality, pressure-induced superconductivity or non-Fermi liquid behaviour, where pressure is a fundamental parameter. Reliable, safe and user-friendly high pressure gas handling systems with gas pressures up to 1GPa should make a significant impact on the range of science possible. The ISIS facility is participating in the NMI3 FP7 sample environment project supported by the European Commission which includes high pressure gas cell development. In this paper the progress in designing, manufacturing and testing a new generation of high pressure gas cells for neutron scattering experiments is discussed.

1. Introduction

The combination of high pressure techniques with neutron scattering proves to be a powerful tool for studying the phase transitions and physical properties of solids in terms of inter-atomic distances. In condensed matter physics there are a number of highly topical areas, such as quantum criticality, pressure-induced superconductivity or non-Fermi liquid behaviour, where pressure is a fundamental parameter. The high pressure technique based on a gas medium compression is one of the most popular in neutron scattering experiments [1, 2]. Currently this technique covers the pressure range up to ~0.7 GPa and typically uses compressed helium gas as the pressure medium. An increase in the range of available gas pressures up to 1 GPa (10 kbar) should make a significant impact on the range of science possible. However, high pressure gas sample cells require thick cell walls which may lead to an unacceptable neutron background and thus the choice of materials and geometries is critical to improving the quality of the data. Reliable, safe and user-friendly high pressure gas handling systems are also an essential part of the development. ISIS facility participates in the NMI3 FP7 sample environment project supported by the European Commission which includes a high pressure gas cell development task.

This paper will discuss the sample chamber seal design of a pressure vessel intended to operate up to 1 GPa (10 kbar) with inert gas as well as 0.8 GPa (8 kbar) hydrogen gas on ISIS neutron instruments. The working temperature will be from 300K down to 4K. The material of construction of the pressure vessel should be neutron compatible and able to retain the necessary strength to endure the enormous stresses likely to occur at the intended high pressures. In addition, it should be completely resistant to hydrogen embrittlement.

2. Seal test arrangements

A standard high-pressure seal for this type of pressure regime would be an unsupported area seal arrangement also known as a Bridgman seal [3]. Any such seal arrangement would generally consist of three components: two soft sacrificial seals, normally made from lead, and a slightly harder principle seal, normally made from annealed copper (Fig. 1 and Fig. 2).

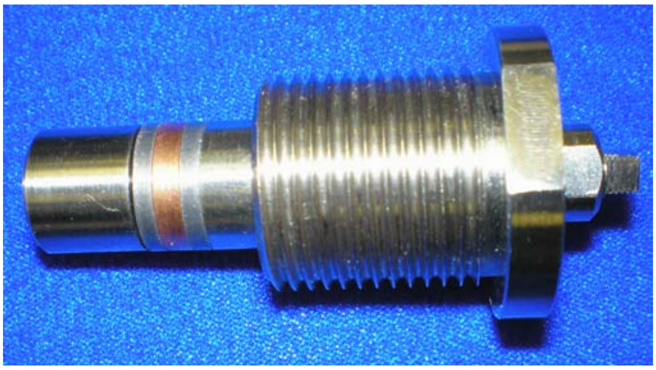

Fig. 1 Typical Bridgman seal arrangement showing a three part seal assembly and the supporting backing nut.

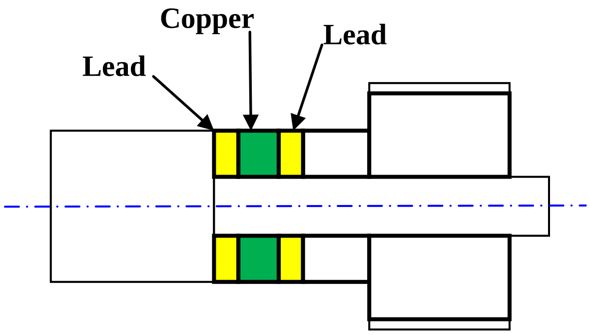

Fig. 2 Typical Bridgman seal arrangement.

All pressure testing at ISIS is carried out in a special purpose test bunker made from concrete, which has camera viewing only during the test procedure. All valve operations are made externally from an intensifier control panel thus protecting personnel in the event of a failure of the equipment inside the test bunker. The extremely high pressures needed for the test were generated using a multi-stage hydraulic oil intensifier which is rated to 1.38 GPa (13.8 kbar) and is operated by a combination of manual pumps taking an initial low inlet pressure up to the system maximum. The seal test vessel is shown in Fig. 3 and is registered as an ISIS pressure vessel.

Fig. 3 Seal test vessel.

3. Material selection

The material selection for the proposed seal combinations was based on both the specification requirements of pressure and temperature, along with previous experience of high-pressure Bridgman seal designs. Materials were chosen that are able to withstand the enormous loadings created at the working pressures, along with the ability to retain ductility at cryogenic temperatures. The materials also had to be inert to the effects of hydrogen gas [4]. The materials selected include pure aluminium, pure copper and pure lead, with backing-ring supports made from either stainless steel or beryllium copper. Two examples of seal material arrangements are shown: an aluminium/copper seal combination and a lead/copper seal combination in Fig. 4.

Fig. 4 Aluminium/copper seal combination (left); lead/copper seal combination (right).

4. Seal test results and discussion

The double Bridgman seal combination shown in Fig. 5 and Fig. 6, using five seal components of lead and lead-coated copper, was chosen as a seal likely to be leak tight at these pressures. By adding an additional sacrificial and principle seal, it was thought that any failure of the active combinations during pressurisation would always have a supporting backup seal to prevent overall breakdown. It did seal successfully, with the lead seals extruding in the process. As a sacrificial seal this is to be expected due to the high ductility of the metal and the presence of a slight clearance between the vessel bore and the seal piston. The 1 GPa (10 kbar) pressure was held for 60 minutes with no apparent leakage. An interesting finding is that a short while after release of the pressure the remaining lead seals expand, causing a clear rippling effect in the assembly (Fig. 7). It is thought that this is possibly caused by residual stresses in the lead relaxing over time creating a reverse-yielding situation allowing the seal to expand. It could also be caused by retained pressure within the structure of the metal forcing the weaker external surface outwards.

The aim of the seal test programme is to examine the quality of the seals at both room temperature and cryogenic conditions. A purpose-built cryostat centre-stick is required for the cryogenic tests and all high-pressure fittings used need to withstand both the 1 GPa (10 kbar) pressures as well as hydrogen corrosion. In many cases such fittings are not commercially available and so have been designed in-house to suit. The longer term requirements of the test programme will require an intensifier to reach over 0.8 GPa (8 kbar) with hydrogen. Room temperature tests have begun using an existing helium intensifier.

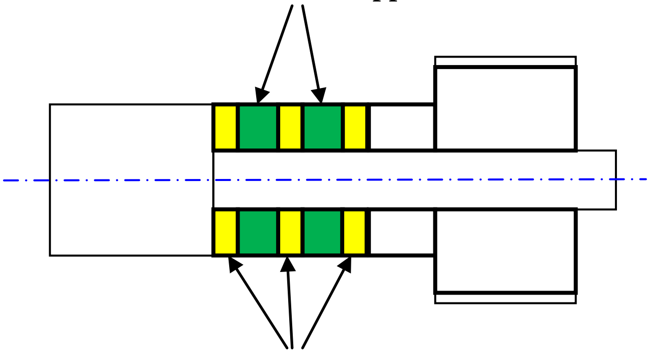

Fig. 5 Double Bridgman seal arrangement.

Fig. 6 Double Bridgman seal prior to pressurisation.

Fig. 7 Double Bridgman after pressurisation, showing the ripple effect of the lead seals.

5. Conclusion and further work

To date, the double Bridgman seal combinations of lead/lead-coated copper, and lead/aluminium, have been successfully tested up to 1 GPa (10 kbar). Seal materials were chosen that are able to withstand the corrosive effects of hydrogen gas and the enormous loadings caused by the high working pressures.

The need to optimise neutron transmission through pressure cells restricts the choice of construction materials available. This limited choice is reduced further when the gas medium required is hydrogen. An important part of this project has thus been the investigation of cell design, hydrogen embrittlement and material suitability.

A literature review of hydrogen embrittlement and material resistance has been undertaken to increase understanding of the issue. Tensile and fatigue testing has been carried out by Imperial College on behalf of ISIS on material samples, with and without hydrogen charging. The fatigue test results are particularly important as the pressure cells will experience repeated pressurisation cycles during experimental use and it is thought that hydrogen embrittlement has a greater effect on the fatigue characteristics of materials due to the nature of the corrosion mechanisms occurring.

The technical aspects of cell design have been investigated, in particular the technique of autofrettaging (a mechanical strengthening process by which pressurised cells are deliberately subjected to an internal force greater than the limiting elastic strength of the cell wall material). Experimental investigations using both neutron (ISIS) and x-ray (Diamond) instruments have been undertaken to confirm, or allow revision of, empirical and FEA modelling of pressure cell designs.

A more detailed account of this work is beyond the scope of this paper and will be covered in future publications.

6. Acknowledgments

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