MNI3 JRA Sample Environment Meeting

Helmholtz-Zentrum Berlin

High Pressure Task

29 -30.03.2011

Science & Technology Facilities Council

Work out a joint approach to the **Health & Safety** aspects based on the **Pressure Equipment Regulations (PED)** *Work in progress*

Inert Gas Cells *(LLB, ISIS)*

Tasks:

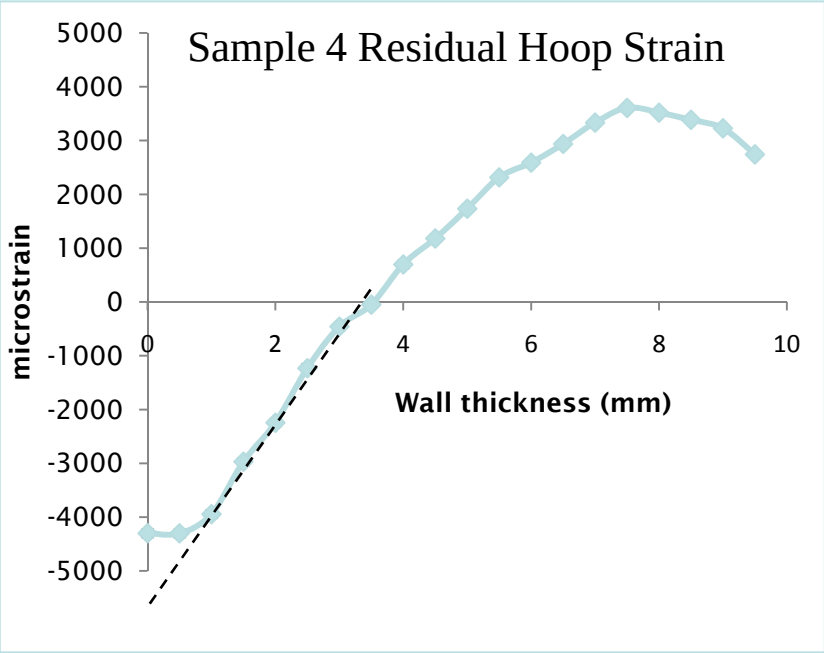
1. ***10 kbar*** *automated gas handling system for inert gases and* ***15 kbar*** *'oil' intensifier for hydraulic testing.* *Completed*
2. *Design and produce cells and test seal systems up to* ***8 kbar*** *(LT – 300 K).* *Completed*
3. *Test seal systems up to* ***10 kbar*** *and design and prototype cells for* ***10 kbar*** *300 K* *Work in progress*

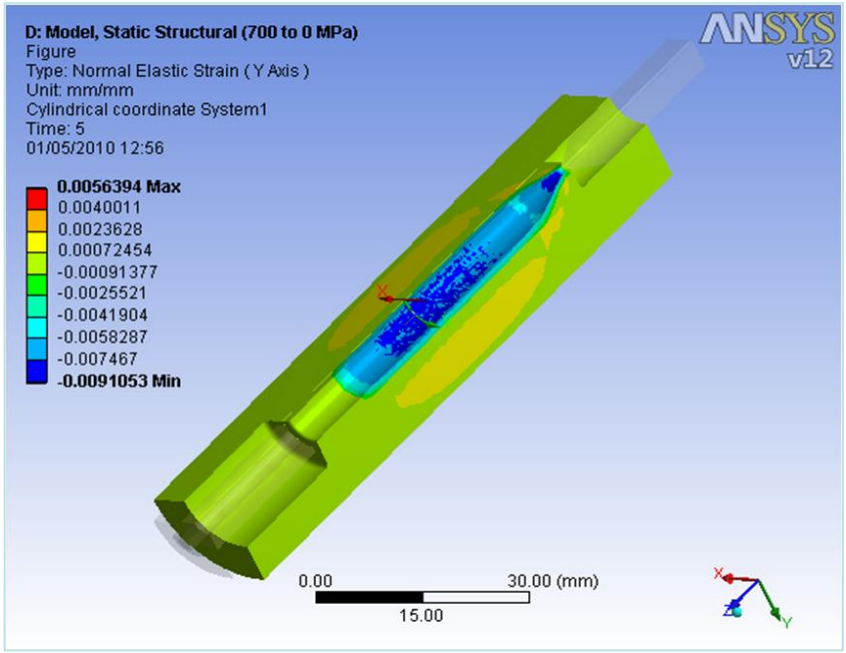
Hydrogen Cells *(ISIS, LLB and HZB)*

Tasks:

1. *Materials – H_2 compatibility/neutron transmission properties.* *Work in progress*
2. *Sourcing, assembly and commissioning of* ***10 kbar*** *H_2 intensifiers and gas handling system.* *Work in progress*
3. *Produce and test cell for* ***4 kbar*** *up to* ***700 K*** *Completed*
4. *Produce and test cell for* ***6 kbar*** *up to* ***300 K.*** *Work in progress*
5. *Design and prototype* ***8 kbar*** *cells for* ***LT – 300 K.*** *Work in progress*

Developing a prototype of 8 kbar inert gas cell.





(a) Experiment results (**ENGIN_X**)

(b) The FEA model

Experiment results and FEA model for sample 4 (**700MPa** autofrettage pressure)

We have been allocated time on beam-line ***JEEP***
I12 at Diamond Light Source

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JRA seal options and hydrogen compatible material tests

Specification

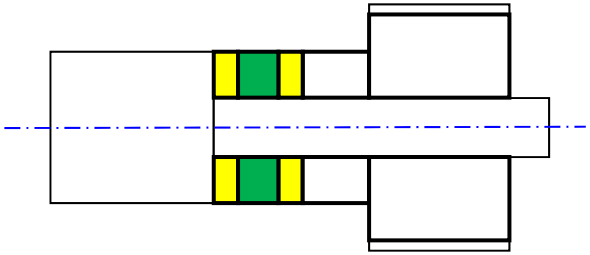
- To investigate the sample chamber seal design and material compatibility of a pressure vessel intended to operate up to ***8000 bar*** with hydrogen gas on the ISIS neutron instruments.

- The working temperature will be from ***300 K*** down to ***4 K***.

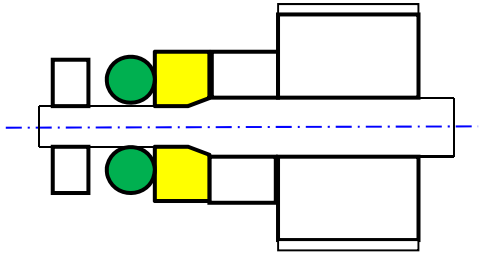
- The material of construction should be ***neutron compatible*** and able to retain the ***necessary strength*** to endure the enormous stresses likely to occur at the intended high pressures.

JRA seal options

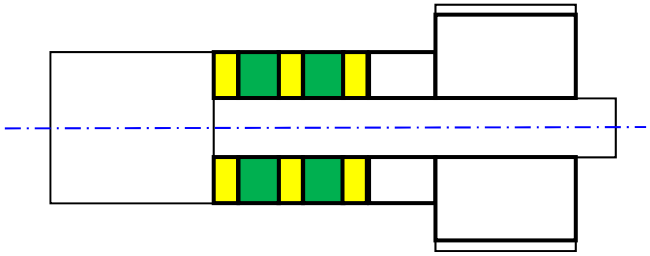
Standard Bridgman seal



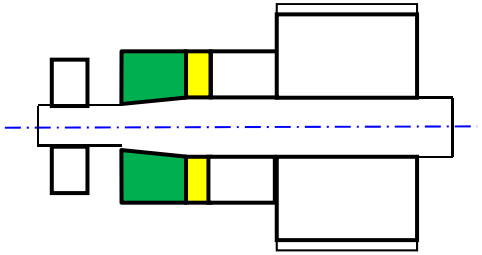
MSE version



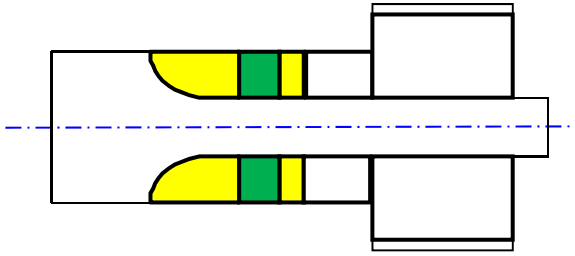
Double Bridgman seal



Tapered version



LLB version



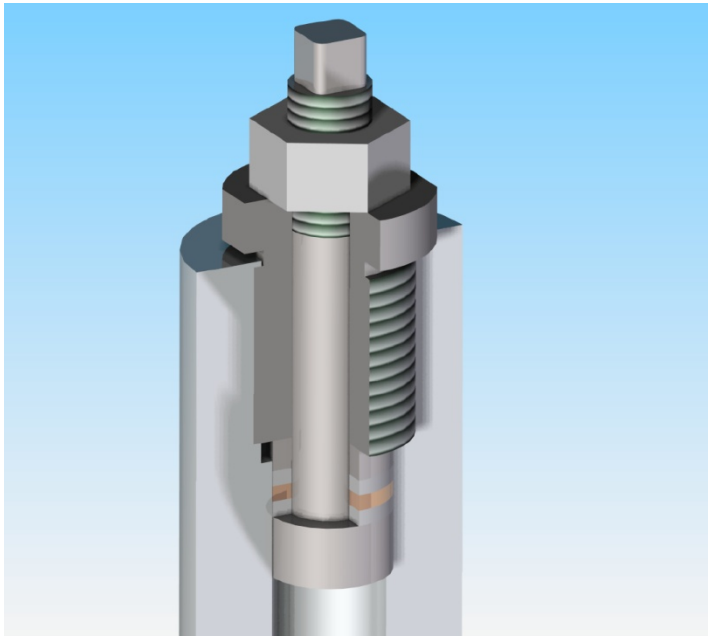
JRA seal options

Double Bridgman seal prior to pressurisation.

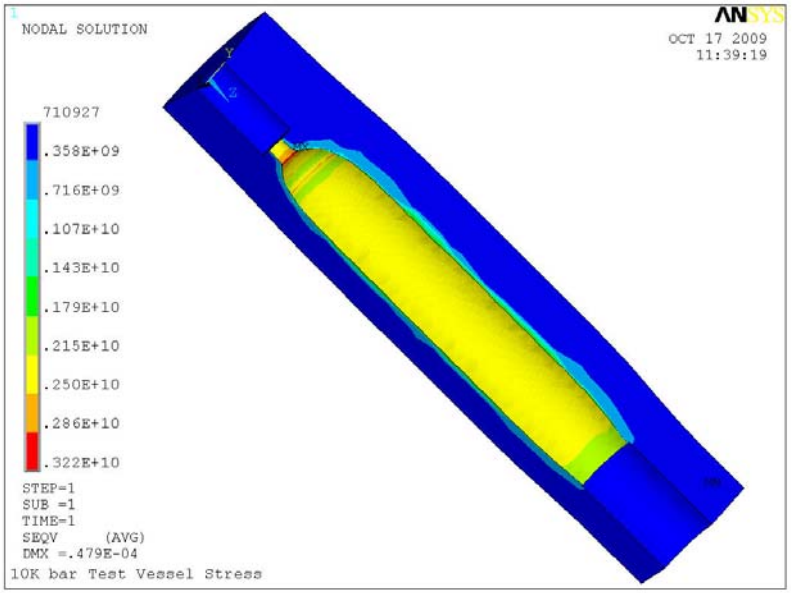
Double Bridgman seal after pressurisation

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

10 kbar seal-test containment vessel



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



Axial FEA results of the seal-test containment vessel.

JRA Bridgman seal test results

Seal Configuration	Room Temp Leak Tight at 2Kbar	Room Temp Leak Tight at 10Kbar	Liquid Nitrogen Leak Tight at 2Kbar	Liquid Nitrogen Leak Test at 10Kbar
Pb\Cu\Pb\Cu\Pb (Copper seals lead plated)	10 mins hold time ✓	3hrs hold time ✓	10mins RT hold time then immersed in N_2 10mins hold time ✓	Pressurised to 10Kbar in N_2 3hrs hold time ✓
Al\Cu\Al\Cu\Al (Copper seals lead plated)	10 mins hold time ✓	Left over night hold time ✓	10mins RT hold time then immersed in N_2 10mins hold time (slight leak observed) ✓	Pressurised to 10Kbar in N_2 (leak sealed) 3hrs hold time ✓
Al\Cu\Al\Cu\Al (Copper seals *not* lead plated)	Seals struggled to seal but finally sealed at 2Kbar ✓	Seal remained leak tight until 7.6Kbar then failed and never resealed during attempt to obtain 10Kbar ✗	Test not performed ✗	Test not performed ✗

Hydrogen compatible material tests

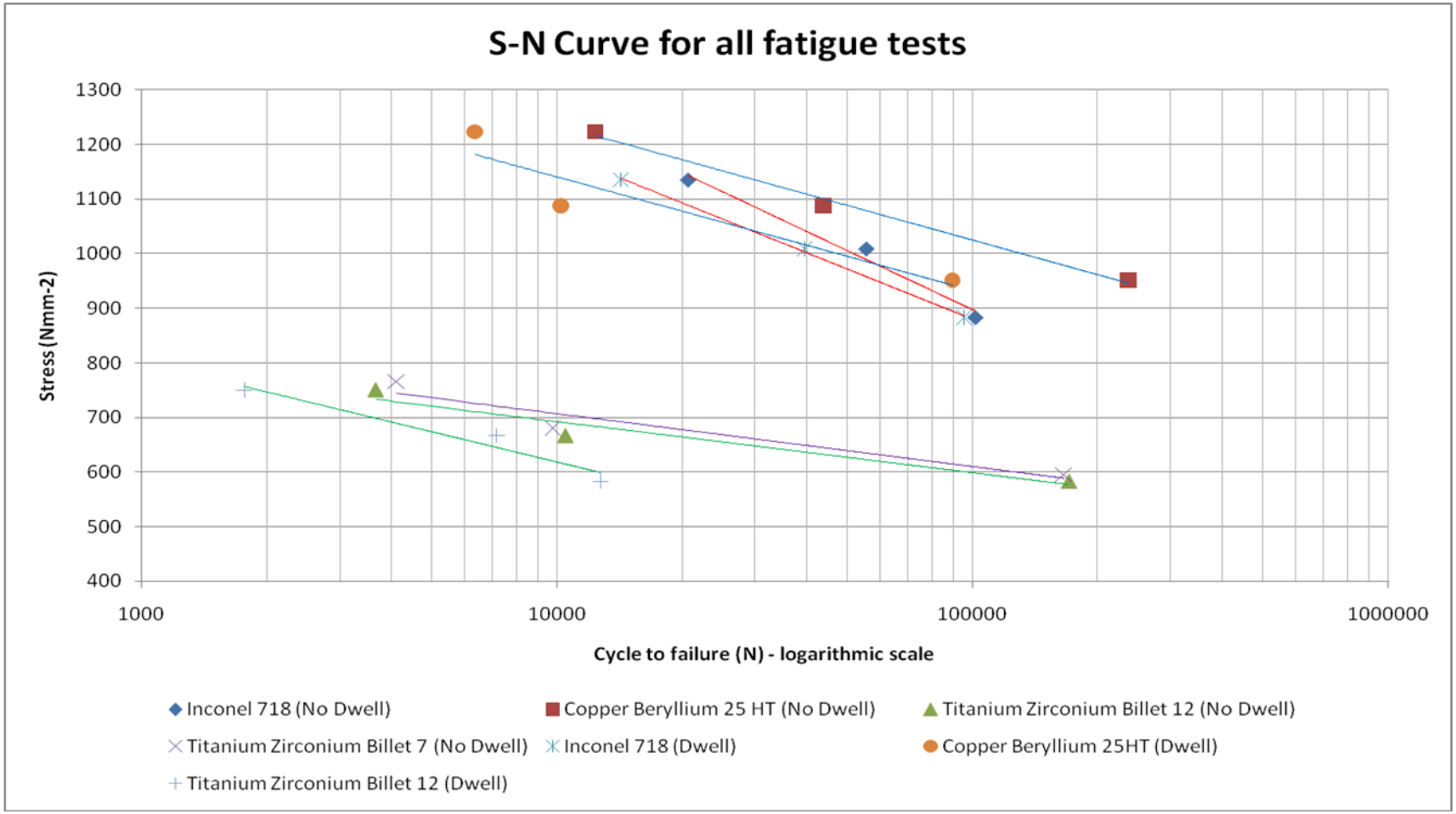
Joint project with Imperial College, the University of London

Hydrogen embritlement is a process where certain materials become brittle following exposure to hydrogen. High-strength steels, titanium alloys and aluminium alloys seem particularly vulnerable to this.

17-4PH tensile test sample

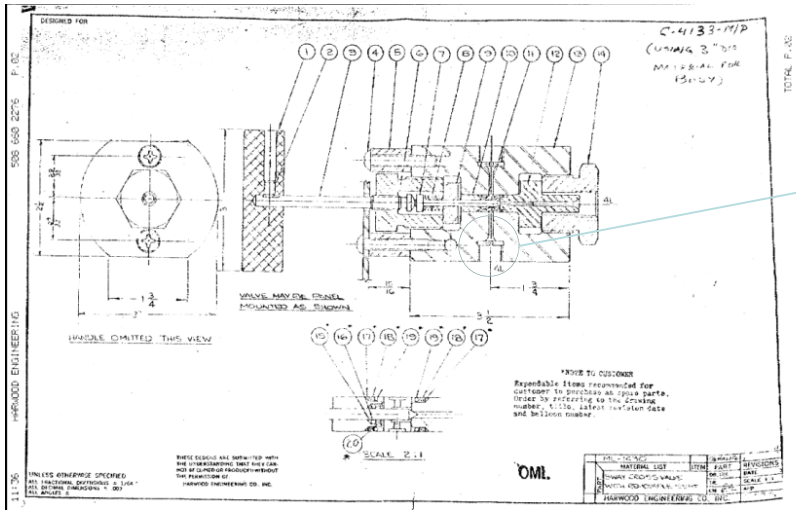
17-4PH tensile test sample

Fatigue results of various vessel materials

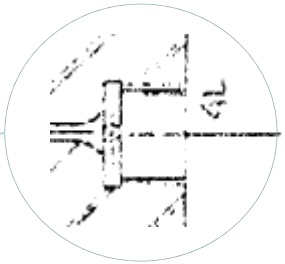


JRA 10Kbar Centre-Stick & H2 Valve

- The JRA 10Kbar Centre-Stick has been assembled but we have encountered a problem due to a Harwood 10Kbar Be\Cu Valve.
- The valve was manufactured with 4L fittings and supplied with collars that enable the use of 10Kbar 3/16” tubing. However the female taper in the 4L fitting is too large for the 3/16” tubing and leaked at 7Kbar.
- This issues have been discussed with our Harwood supplier and a mutual solution to rectify the issue currently in progress.



Harwood C-4133 Drawing



Enlarged detail of 4L female taper

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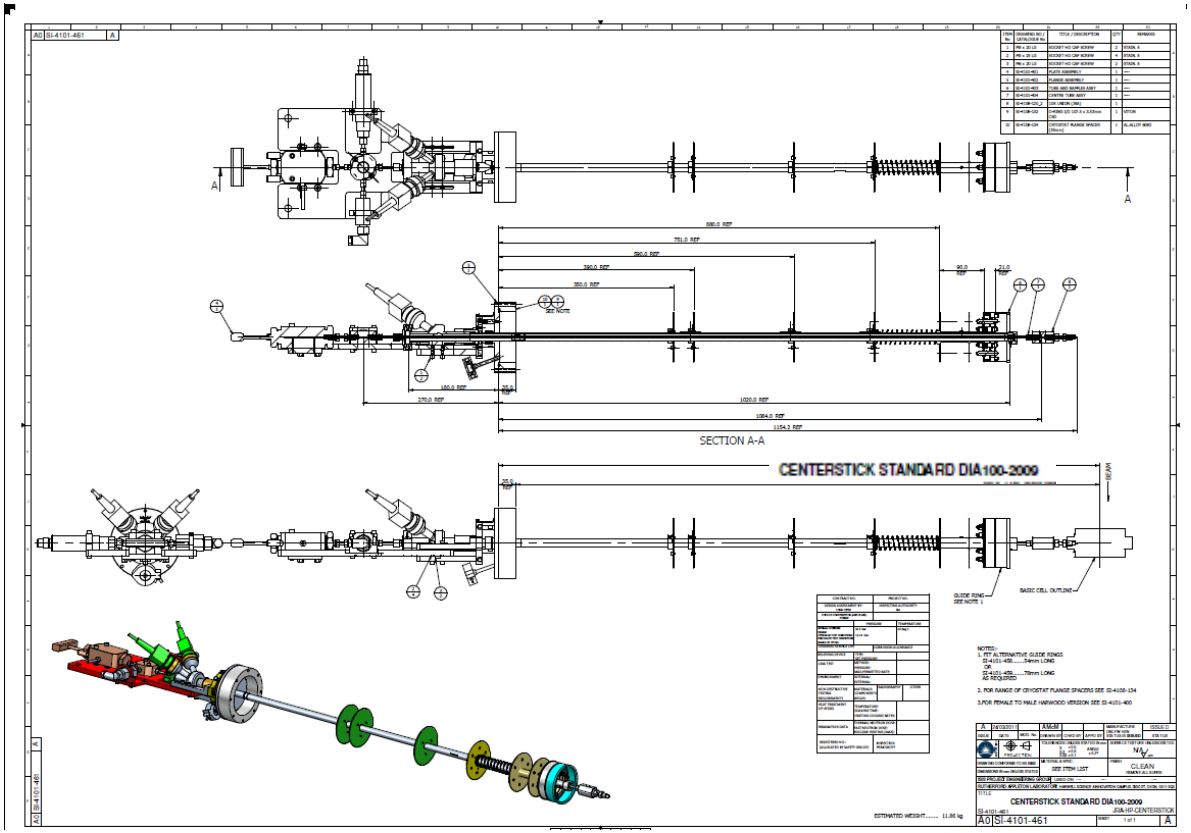
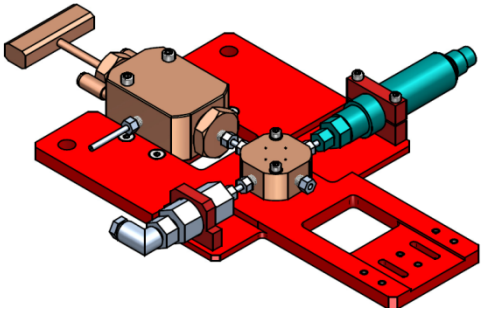
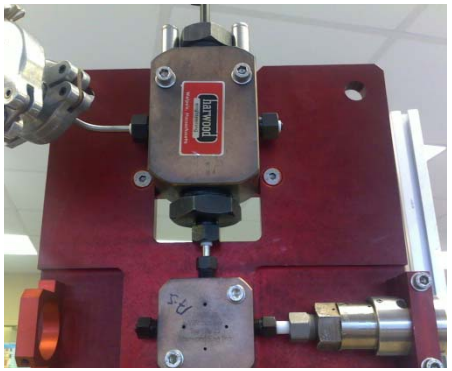
JRA 10Kbar Centre-Stick & H2 Valve



JRA 10Kbar Centre-Stick Drawing





Harwood C-4133 H2 10Kbar valve assembly on Centre-Stick

JRA Intensifiers

10Kbar Inert Automated Gas Handling System (Intensifier)

- The 10Kbar inert system has been run up to 9800bar. Some required changes were identified and Hi-Pro our supplier have offered to make modifications to the system at their cost to make it a more usable system.

10Kbar Hydrogen Gas Handling System (Intensifier)

- Due to financial restraints the 10Kbar Hydrogen Intensifier is to be assembled and tested at ISIS by the Pressure & Furnace department. Components have been sourced and several of them purchased and the initial assembly work is scheduled for September 2011.

Conclusions

- From the seal tests we have carried out to date it is apparent that the *Pb\Cu\Pb\Cu\Pb* seals are the best but the lead does extrude extensively. The next set of seal tests will include a similar design but will incorporate an anti-extrusion ring to prevent the lead from extruding.

- We continue hydrogen compatible material tests. We have completed tests of Copper Beryllium, Inconel and Titanium Zirconium samples. Now we are trying different techniques to hydrogenate the samples and planning to perform similar fatigue tests with hydrogenated samples.

- The Harwood 10Kbar Be\Cu Valve has been return for modification to incorporate 2 adaptors and a replacement valve seat assembly which will terminate in 3M fittings. This will rectify the leak issue and standardise the fitting connection.