NMI3 JRA Sample Environment Meeting

Helmholtz-Zentrum Berlin (Wannsee)

High Pressure Task

22.11.2012

ISIS

Activities, Deliverables & Milestones

Gas handling and pressure Cells for Inert Gases

- ***D21.02*** Report on current inert gas pressure cell technology (STFC) *Completed*
- ***D21.06*** 13-15 kbar hydraulic intensifier for cell testing assembly (STFC) *Completed*
- ***D21.07*** Procurement of LLB cryogenic system for pressure measurements (LLB) *Completed*
- ***D21.15*** Procurement **10 kbar** automated gas handling system (STFC) *Completed*
- ***D21.18*** Manufacture a cell for up to 8 kbar (LLB) *Completed*
- ***D21.26*** Procure 10 kbar automated gas handling system (LLB) *Completed*
- ***D21.29*** Manufacture and test prototype cell up to 10 kbar (LLB) *Prototype has been manufactured; awaiting for test*

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Activities, Deliverables & Milestones

Gas handling and pressure Cells for Hydrogen

- ***D21.08*** Manufacture and test prototype cell for 4kbar and up to 700K (STFC) *Completed*
- ***D21.12*** Sourcing, assembly and commissioning of 8-10 kbar H_2 intensifier and gas handling system (STFC) *Work in progress*
- ***M21.2.2.1*** Cell material and seal design review *Completed*
- ***D21.16*** Report on material research: H_2 and neutron compatibility . *Completed*
- ***M21.2.2.2*** Design plan review for H_2 gas pressure cell for 8 kbar at 4-300K *Completed*
- ***D21.24*** Manufacture and test prototype cell for 6kbar and up to 300K (STFC) *Completed*
- ***D21.29*** Manufacture and test prototype cell for 8 kbar 4 – 300K (STFC) *Prototype has been manufactured; awaiting for test*

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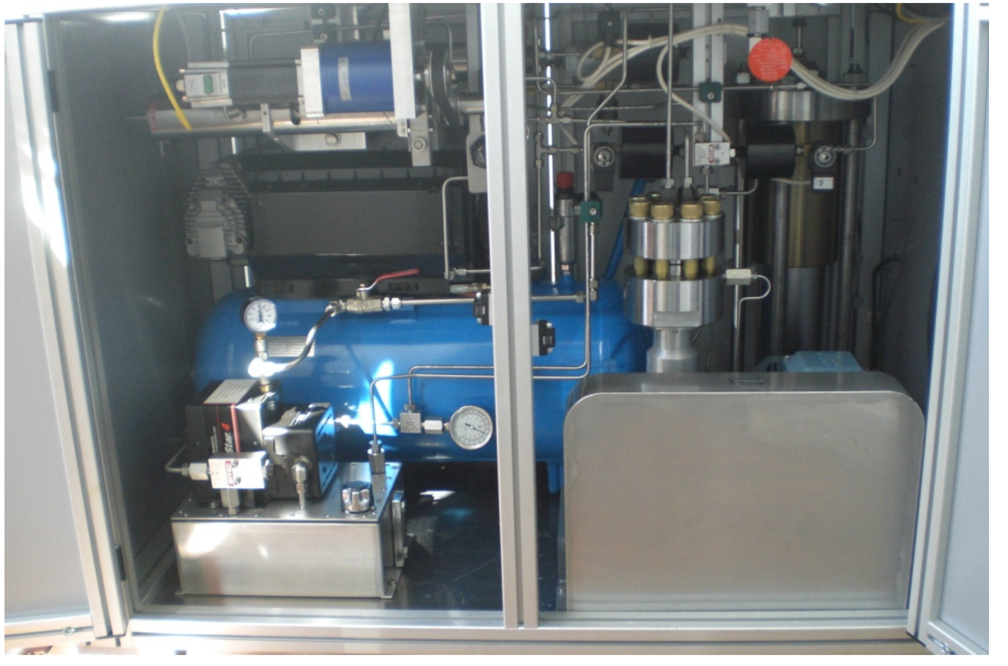
D21.15 **Procurement 10 kbar automated gas handling system**



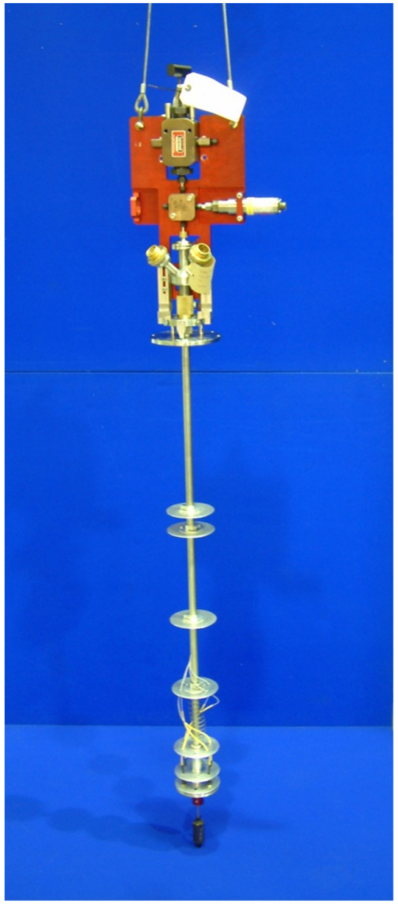
The automated inert gas handling system specification:

- Operating from 0 to 10,000bar
- Transducer accuracy of 0.3%
- Full scale and pressure changes in steps of 40 bar
- This system is able to be controlled by instrument computer

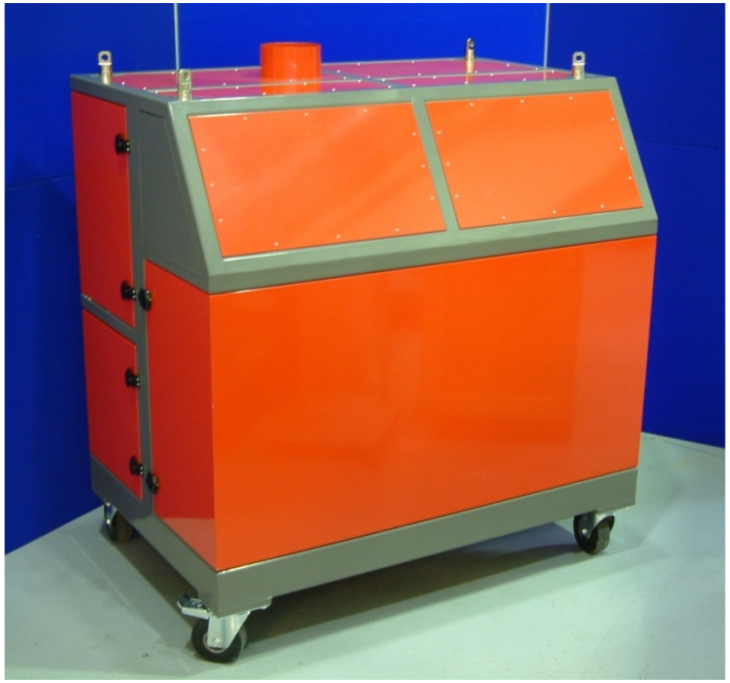
The system is released to ISIS User Program

D21.15 **Sourcing, assembly and commissioning of 10 kbar H_2** *Intensifier and gas handling system*

Due to financial restraints the 10Kbar Hydrogen Intensifier is to be assembled and tested at ISIS by the Pressure & Furnace department. Components have been purchased and assembly work is in progress . ***ISIS immensely benefitted from HZB experience***!



High pressure hydrogen stick



High pressure hydrogen “red“ panel
9.5 kbar with Helium has been achieved

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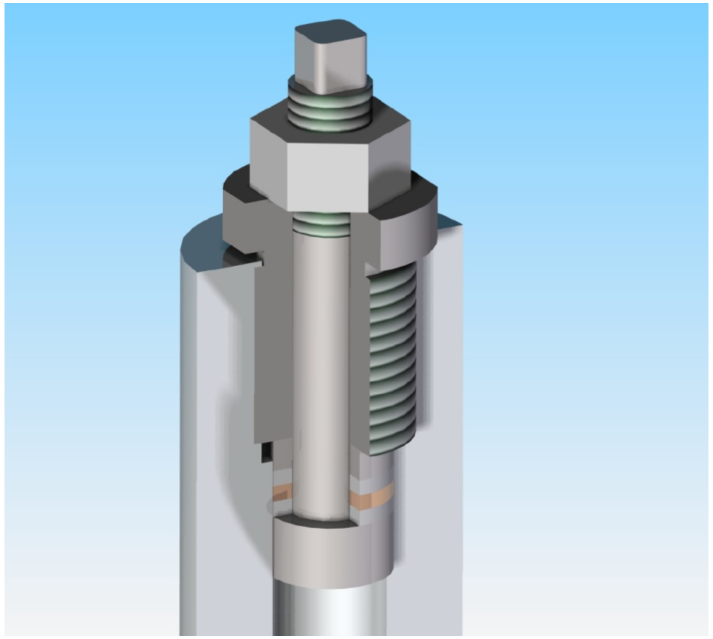
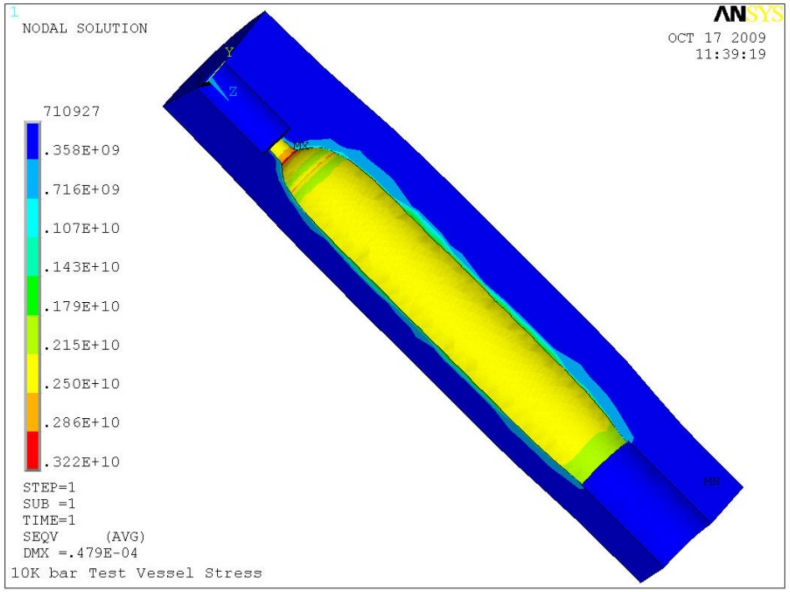
M21.2.2.1 **Cell material and seal design review**

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.

Yanling Ma et. al. *International Journal of High Pressure Research* **32** (2012) 364

M21.2.2.1 Cell material and seal design review

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 ✗

Aluminium/copper seal combination (left); Lead/copper seal combination (right).

M21.2.2.1 **Cell material and seal design review**

D21.16 **Report on material research: H_2 and neutron compatibility**

Hydrogen compatible material tests

Joint project with ***Imperial College, the University of London***

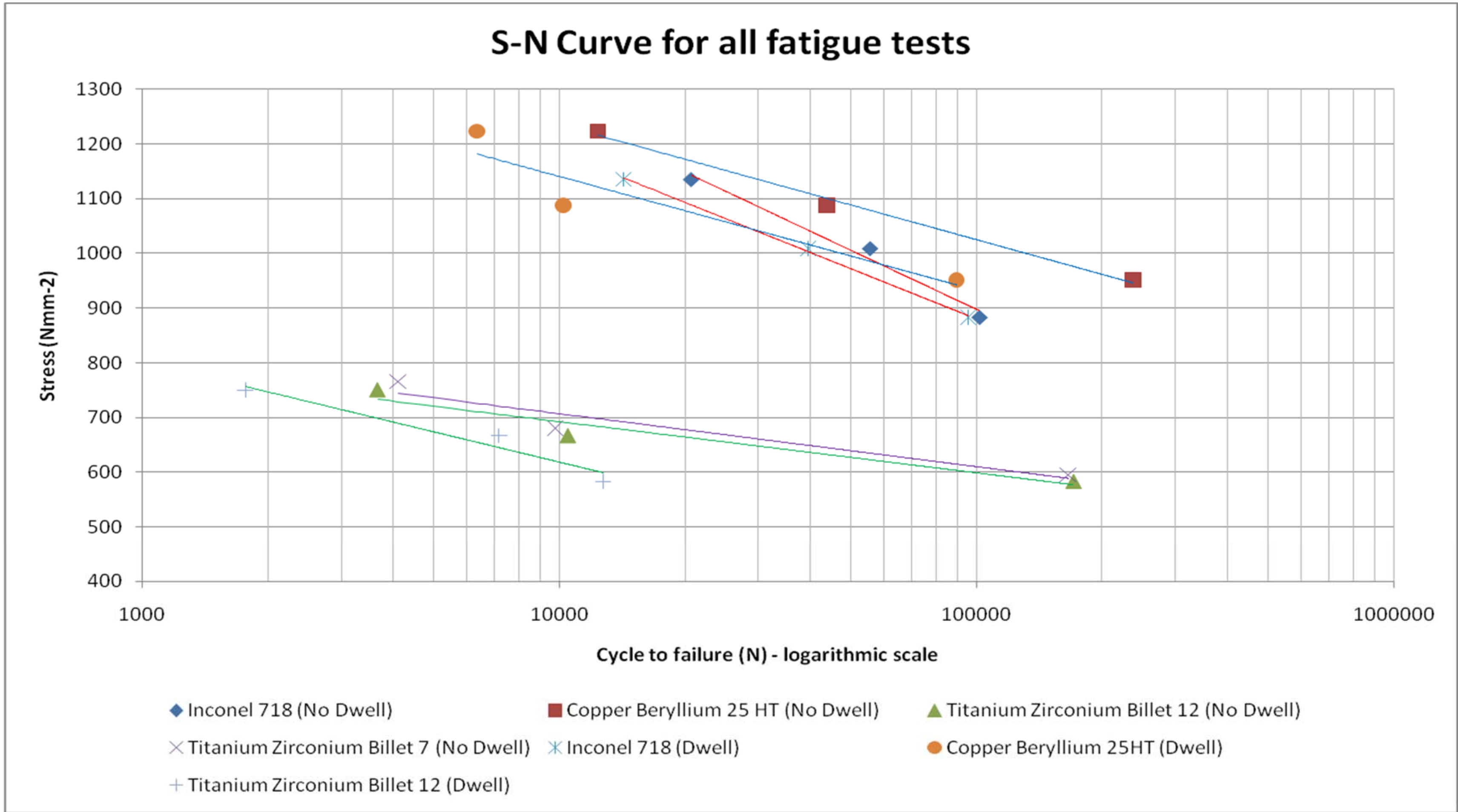
Hydrogen embritlement is a process where certain materials become brittle following exposure to hydrogen. High-strength steels, titanium alloys and aluminium alloys seem particularly vulnerable to this.

17-4PH tensile test sample

17-4PH tensile test sample

D21.16 Report on material research: H_2 and neutron compatibility

Fatigue results of various vessel materials



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Hydrogen gas cells

D21.24 **6kbar** and **7kbar** Hydrogen gas cells for **LT – 300 K**

D21.29 Prototype cell for **8 kbar** of Hydrogen **LT – 300 K**

High magnetic field + high pressure

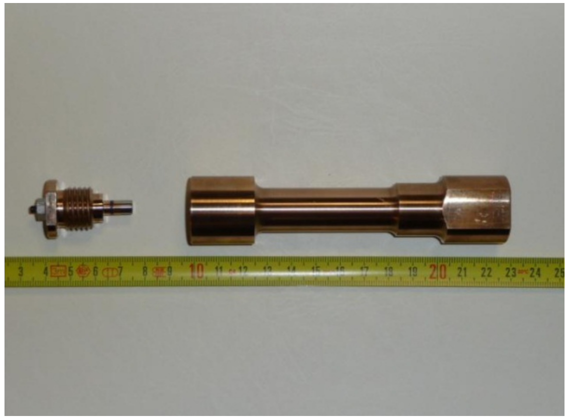
Free energy landscape of hydrogenated La(Fe,Si)13

K G Sandeman, Z G Gercsi, Imperial College London, UK

O Gutfleisch, IFW-Dresden, Germany

A. Barcza et al., "Giant Magnetoelastic Coupling in a Metallic Helical Metamagnet" Phys. Rev. Lett. 104 247202 (2010) [HRPD + theory]

7.5T magnet



LLB High pressure cells for inert gases 8kbar

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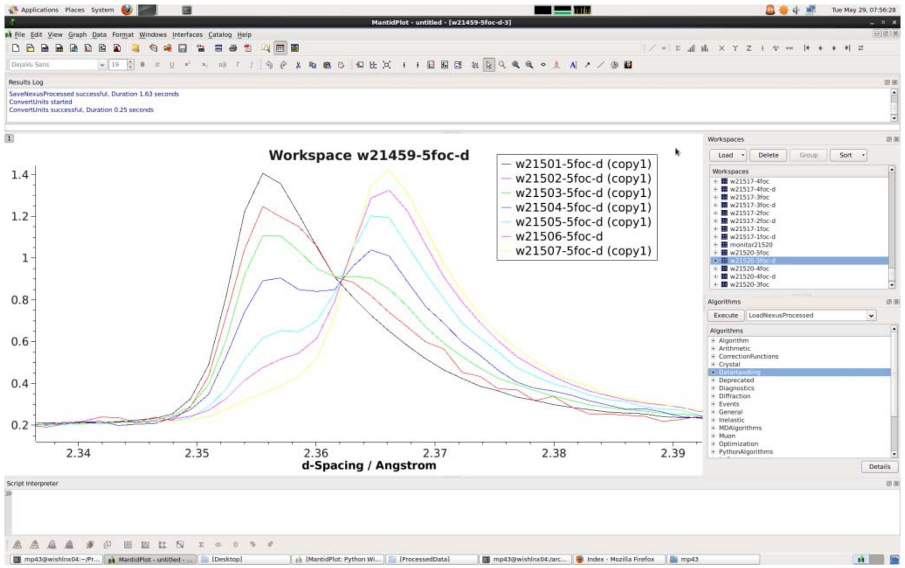
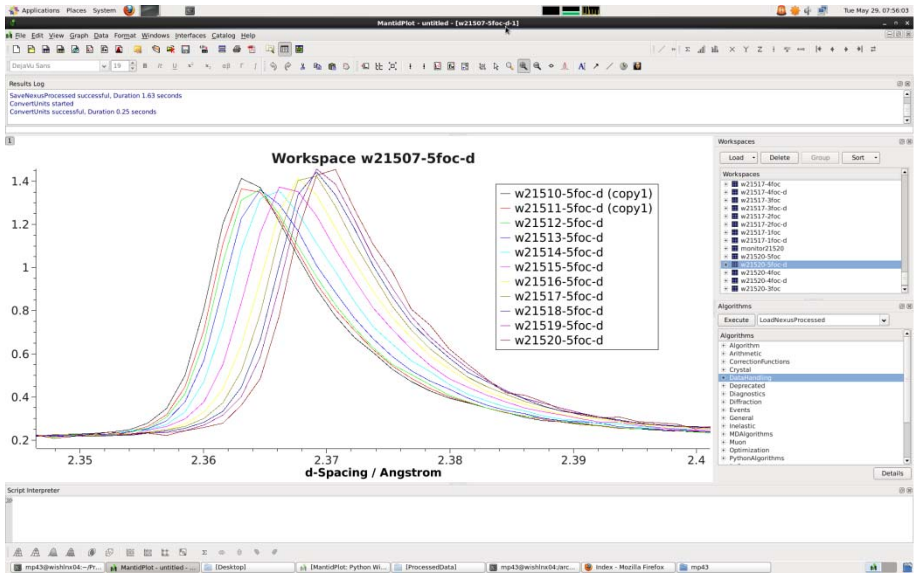
High magnetic field + high pressure

We seem to be observing the influence of the pressure-temperature-field-induced critical point (i.e. first order changing to second order transition in a single material, depending on the exact experimental conditions).





Screenshot 1 P=7kbar, 1st order transition at 20 K above "Tc" with increasing field (B=4.5 to 5.5T @235K)

Screenshot 2 P=0, 2nd order transition at 15K above Curie temperature with increasing field (B= 0-5T@285K)

By courtesy of Dr. Karl Sandeman, Imperial College, University of London

The University of Edinburgh
School of Engineering CSEC
Konstantin Kamenev's Group

"SPUTNIK" Cell:
- up to 6 Gpa (60 kbar)
- 0.05 – 300K

SPUTNIK - 3

JRA SE High Pressure Task technical publications:

1. Yanling Ma et. al. ***International Journal of High Pressure Research*** **32** (2012) 364
2. O. Kirichek et. al. ***Journal of Physics: Conference Series*** **340** (2012) 012008
3. R. Done et.al. High pressure gas vessels for neutron scattering experiments ***ArXiv*** (2010) 1007.3135
4. R. Done et.al. ***NMI3 FP7 JRA Report*** on current inert gas pressure cell technology (2010)

Experiments which directly benefited from JRA SE project:

1. E. Gray, C.J. Webb In-situ diffraction techniques for studying hydrogen storage materials under high hydrogen pressure ***International Journal of Hydrogen Energy*** **37**, (2012) 10182
2. M. T. F. Telling et. al. ***Phys. Rev. B*** **85** (2012) 184416
3. T. Lancaster et. al. ***Phys. Rev. B*** **84** (2011) 092405
4. M Enomoto et. al. ***Polyhedron*** **30**, (2011), 3137
5. F K Katsaros et al ***Journal of Physics: Conference Series*** **340** (2012) 012046

Experiments which benefited from technologies and equipment developed by JRA SE project:

Sihai Yang et. al. ***Selectivity and Direct Visualization of Carbon Dioxide and Sulfur dioxide in a Decorated Porous Host*** *Nature Chemistry* (2012) doi:10.1038/nchem.1457

Kazuya Kamazawa et. al. ***In-Operando Neutron Diffraction Studies of Transition Metal Hydrogen Storage Materials*** *Advanced Energy Materials* (2012) 10.1002/aenm.201200390

Acknowledgements

We are grateful to our partners from ILL, HZB, LLB, PSI and FRM II for really fruitful collaboration in NMI3 FP7 JRA SE framework supported by the European Commission.

We also really appreciate great opportunity of knowledge and experience sharing with our colleagues from neutron facilities all over the world.

Thank you very much for your attention!