Instrumentation and research methods at IFE:

Challenges with neutron diffraction under high hydrogen pressure

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Physics department, IFE

Outline

- Institute for Energy Technology and the JEEP II reactor
- Neutron diffraction and hydrogen storage materials
- Plans for neutron diffraction sample cell for measurements under high hydrogen pressure (150-200 bar)

Institute for Energy Technology - IFE

- ~ 650 employees (Kjeller and Halden)
- Annual turnover 100 Meuro (2011) (15% from gov.)
- Organized in 5 sectrors

Nuclear Technology and Physics

Energy and Environmental Technology

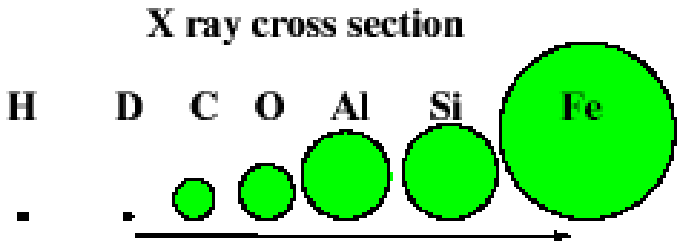
Petroleum Technlogy

Nuclear Safety and Reliability

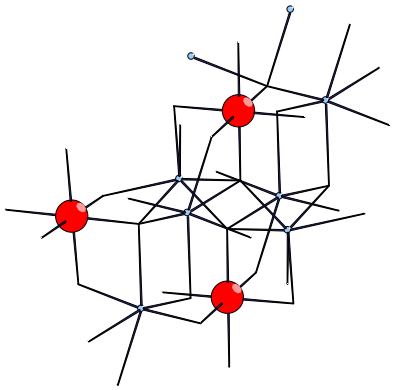
Safety Man-Technology-Organisation

The Physics Department:
"conduct basic research in materials science based on the JEEP II reactor at Kjeller".

Revealing the atomic structure

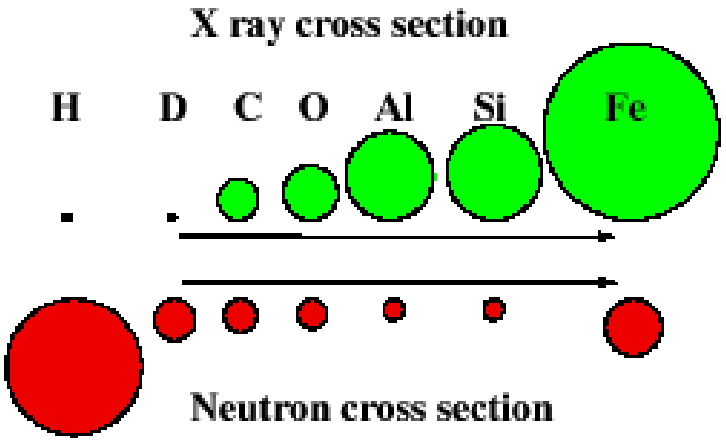


The heavier an atom is, the stronger it scatters X-rays.



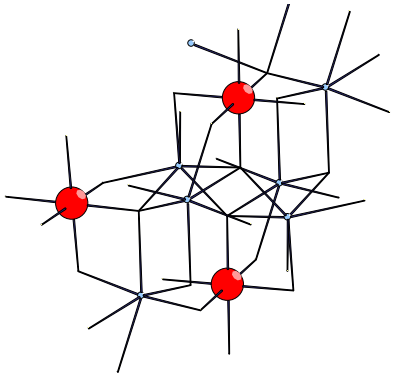
Li_3AlH_6 seen by X-rays

Revealing the atomic structure

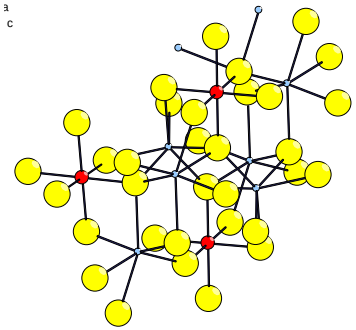


The heavier an atom is, the stronger it scatters X-rays.

… not so with neutrons!



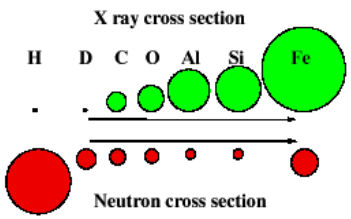
Li_3AlH_6 seen by X-rays



Li_3AlD_6 seen by neutrons

The benefits of neutrons

- There is no systematic correlation between atomic number and the scattering length.



- The neutron interacts weakly with matter.

Neutrons penetrate stuff…

5 mm Al

X-ray $\lambda = 1.54$ Å — $I/I_o = 10^{-29}$

Neutrons $\lambda = 1.0$ Å — $I/I_o = 0.996$

X-ray $\lambda = 0.2$ Å — $I/I_o = 0.02$

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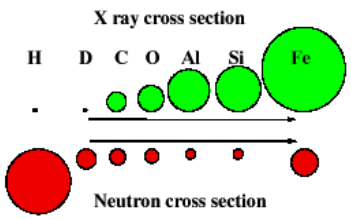

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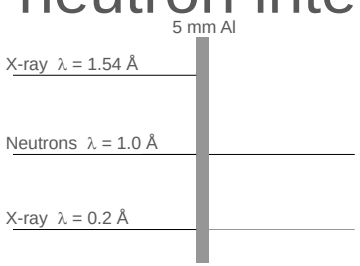
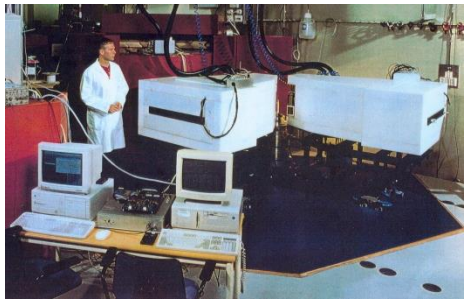


- The neutron has a magnetic moment.

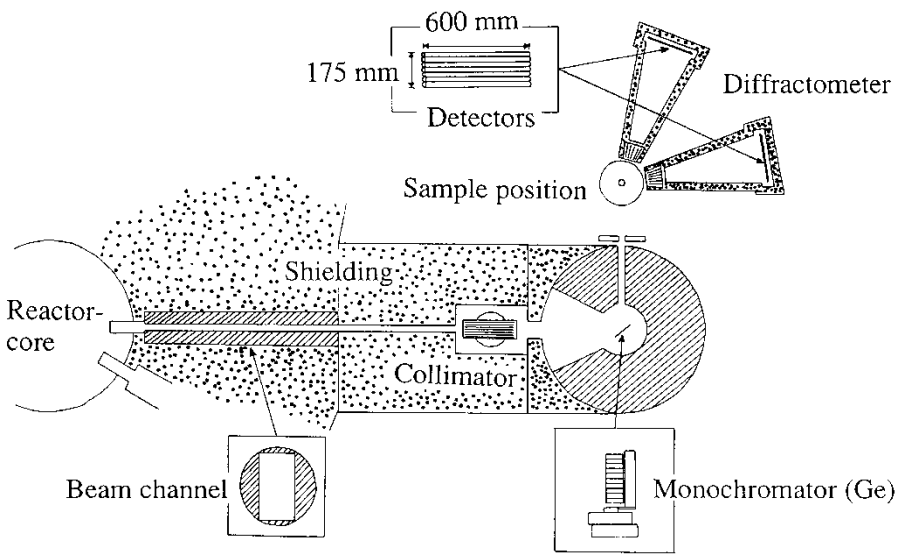
Instruments in H2FC

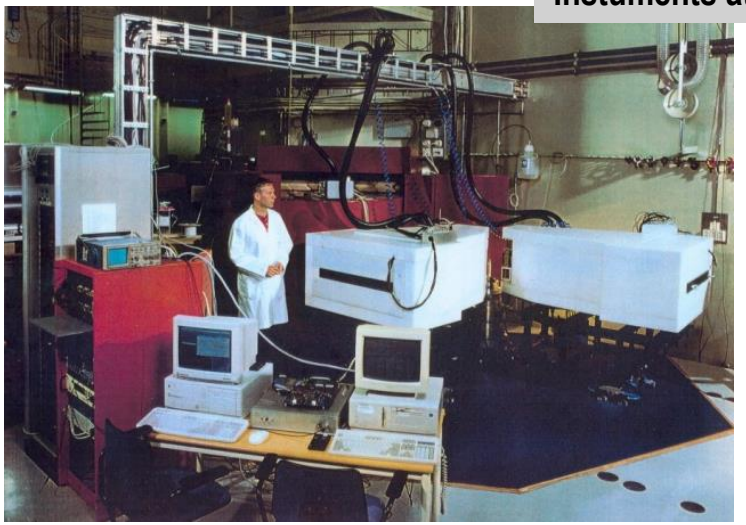


Neutron diffractometers PUS and ODIN
(60 days each for H2FC)

SANS (Small Angle Neutron Scattering)
(60 days for H2FC)

PUS



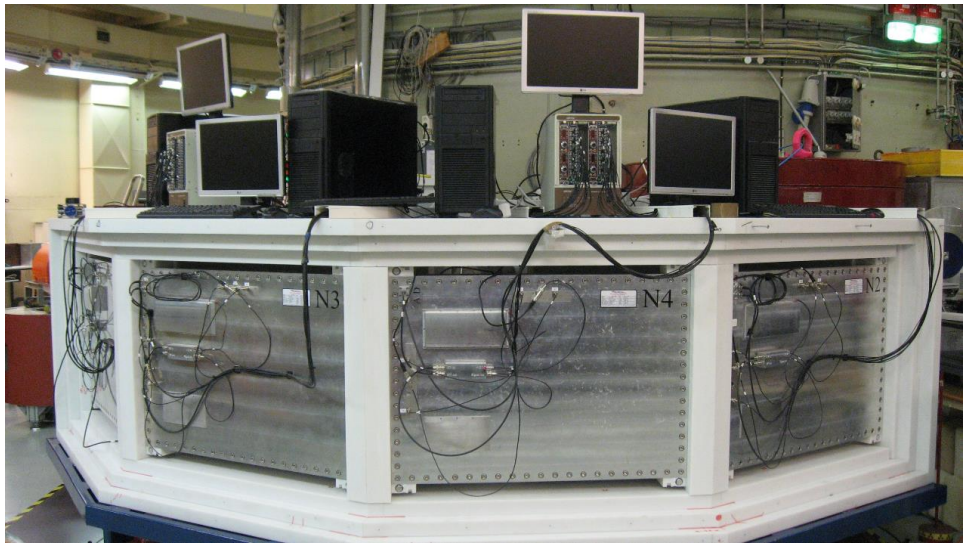


- λ = 0.75-2.60 Å
- T_{sample} = 9 – 1300 K
- P = 0 – 8 bar
- $\Delta d/d \sim 3 * 10^{-3}$
- Flux on sample ~ $1.5 * 10^5 n/cm^2 * s$
- Typical range: 2θ = 10-130°

New 150-200 bar sample holder is planned under JRA3.2.1.

ODIN

- – a new powder diffractometer

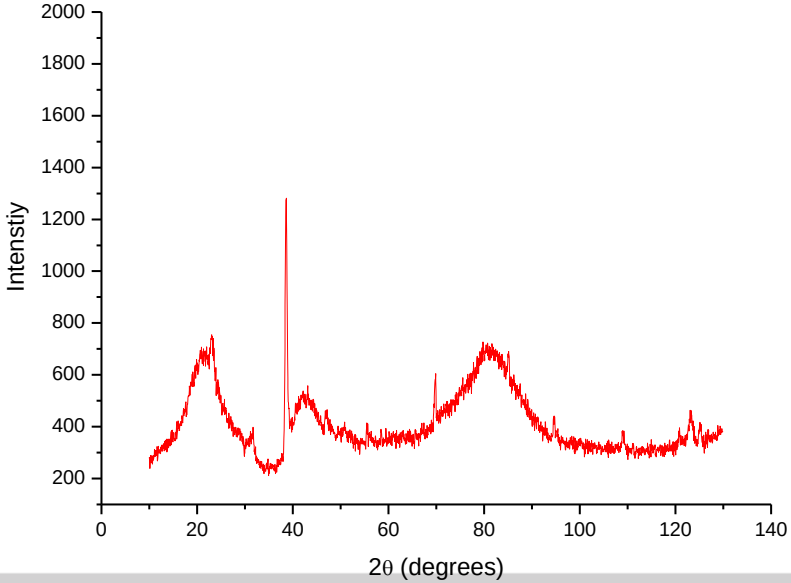


- Take-off angles 75-120°
- $\Delta d/d \sim 1.3*10^{-3}$
- Flux on sample 1.4*10^{6} n/(cm^{2}s)
- 4 detectors, each 50x80cm^{2}

Measurement under hydrogen pressure

Requirements for the sample cell:

- Sufficient mechanical strength
- Inert to hydrogen
- Low neutron absorption
- Weak scattering



Current setup (max 8 bar)

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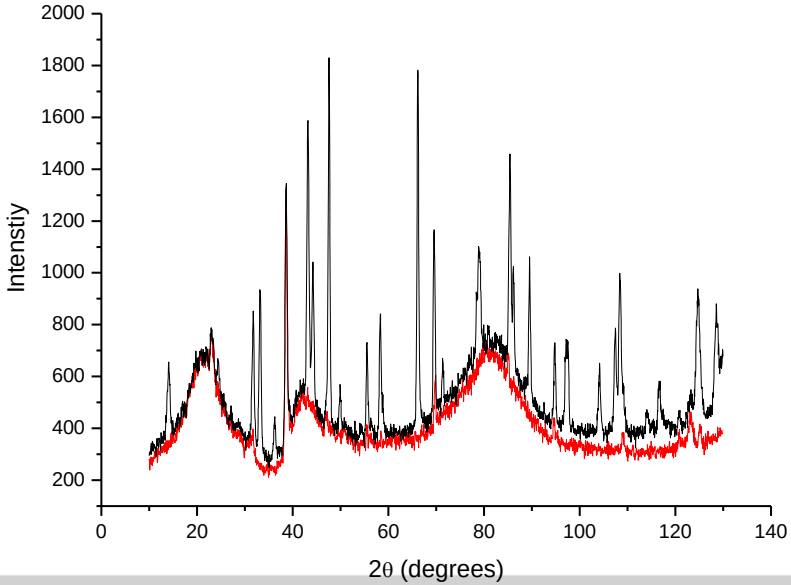
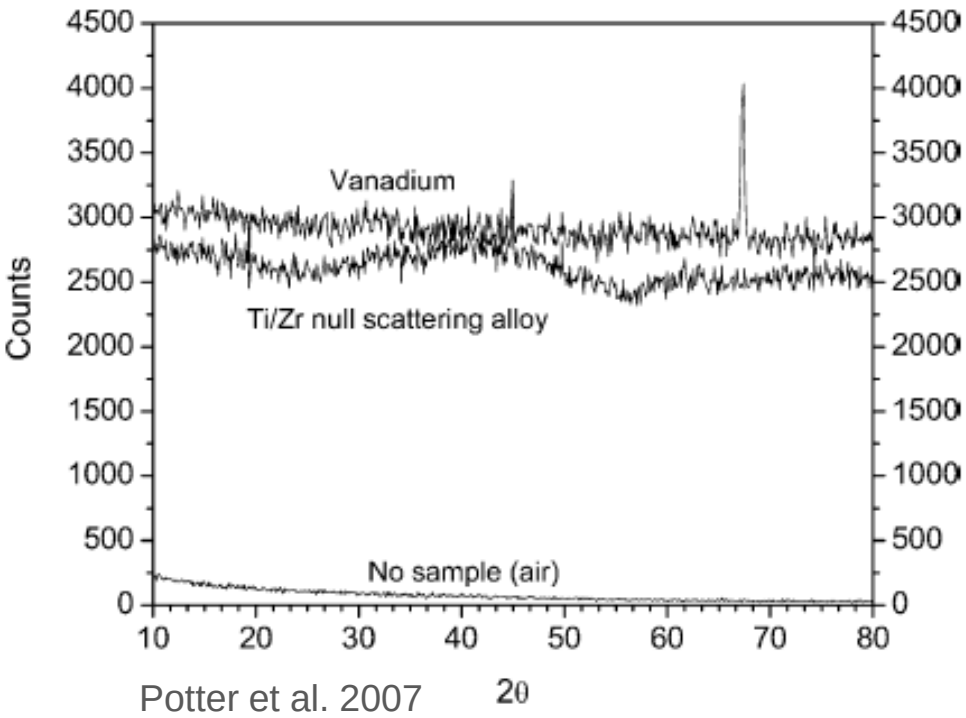


Current setup (max 8 bar)

Some candiate materials for sample cells

Vanadium or «zero-scattering» alloys (e.g $Ti_{68}Zr_{32}$)



Potter et al. 2007

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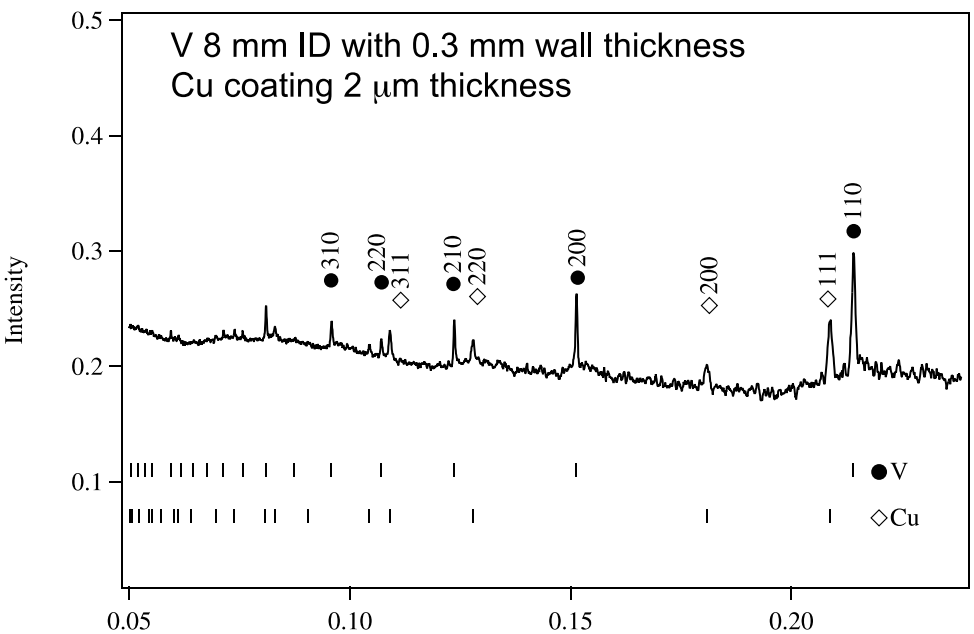
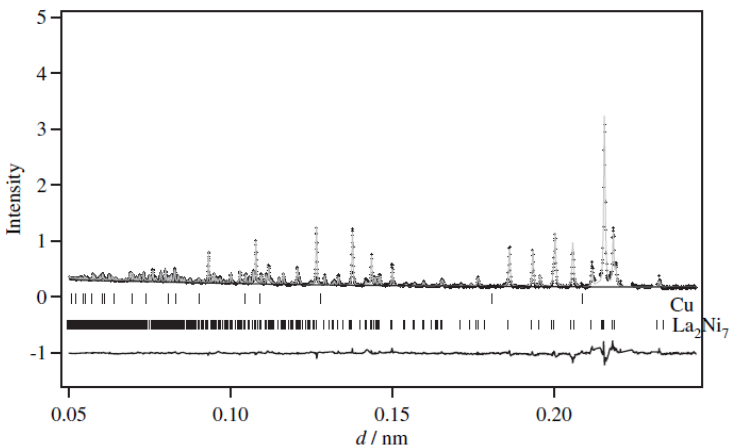


Iwase et al. 2011



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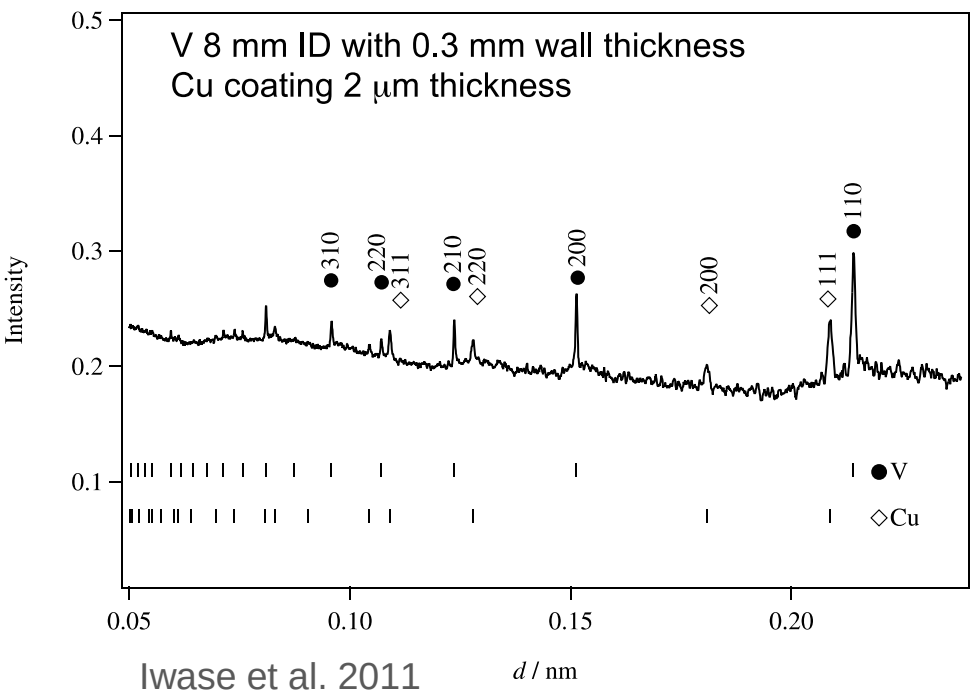

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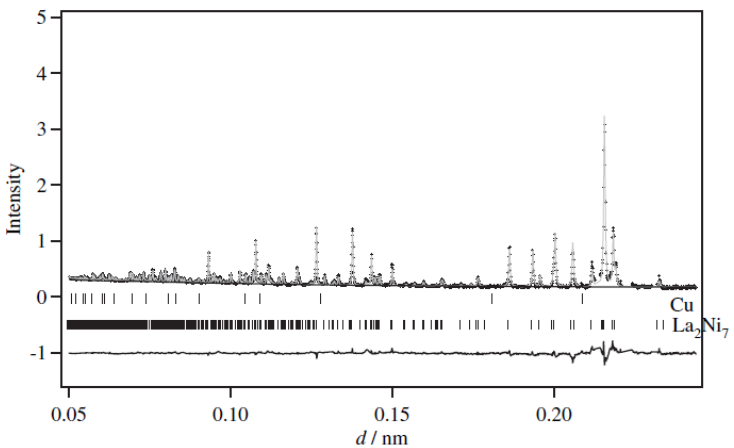


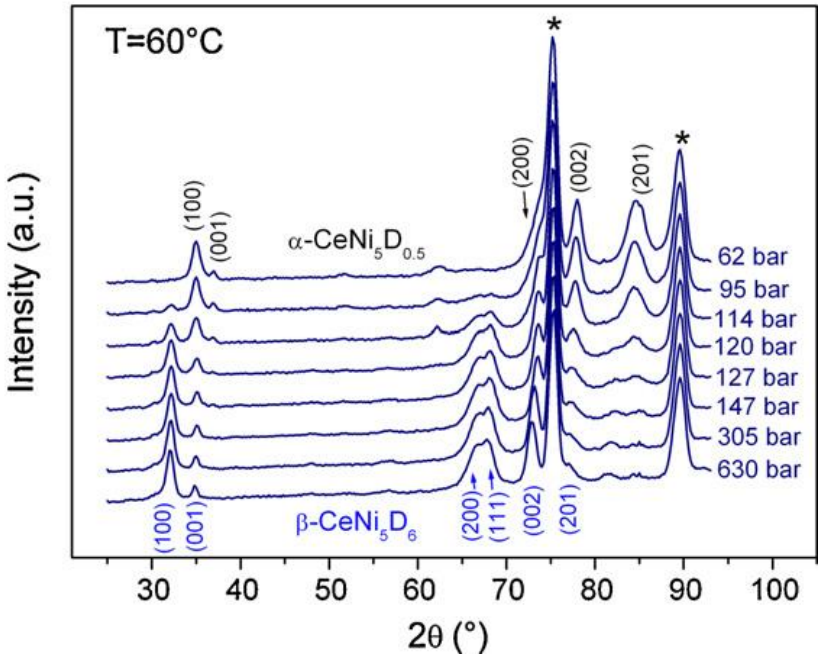
P_{max} ~ 50 bar

Requirements for the sample cell:

- Sufficient mechanical strength
- Inert to hydrogen
- Low neutron absorption
- Weak scattering

Some candiate materials for sample cells

Steel or inconel alloy



Yartys et al. 2011

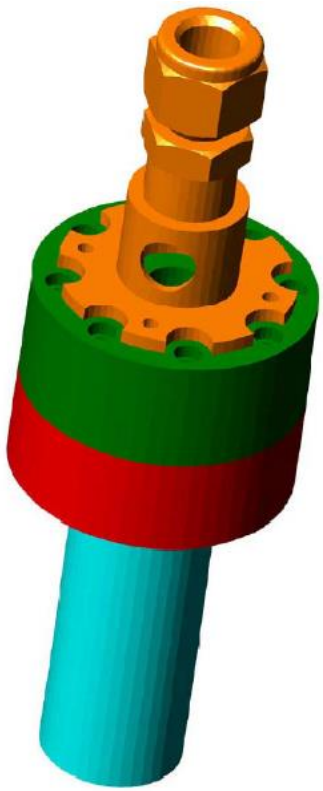
* - sample container

Requirements for the sample cell:

- Sufficient mechanical strength
- Inert to hydrogen
- Low neutron absorption
- Weak scattering

Some candiate materials for sample cells

Single crystal sapphire tube



Walker et al. 2010

Can be oriented so that Bragg spots from the single crystal are avioded in the direction of the detector.

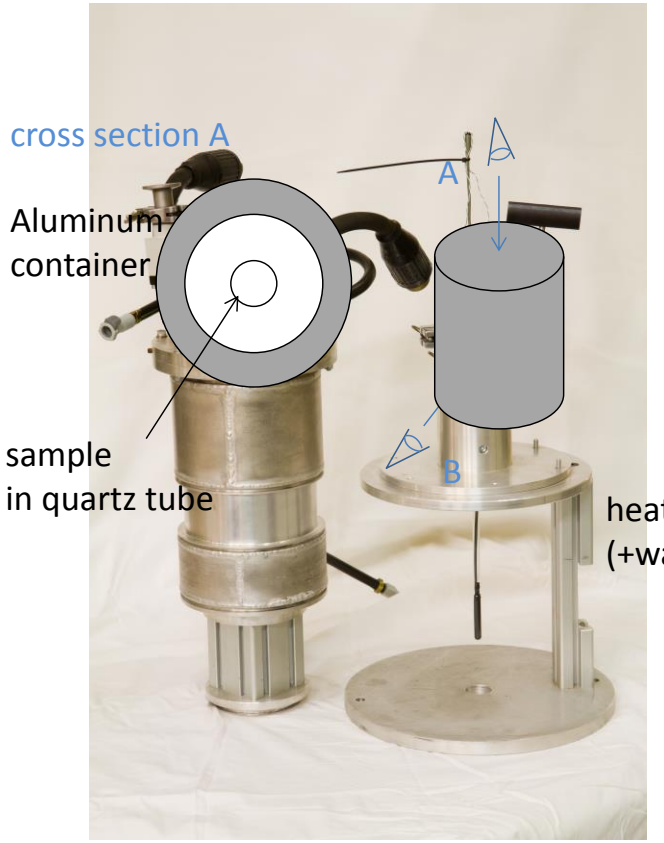
Requirements for the sample cell:

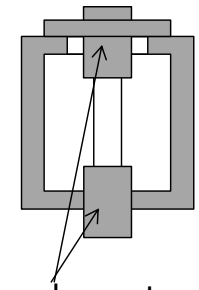
- Sufficient mechanical strength
- Inert to hydrogen
- Low neutron absorption
- Weak scattering

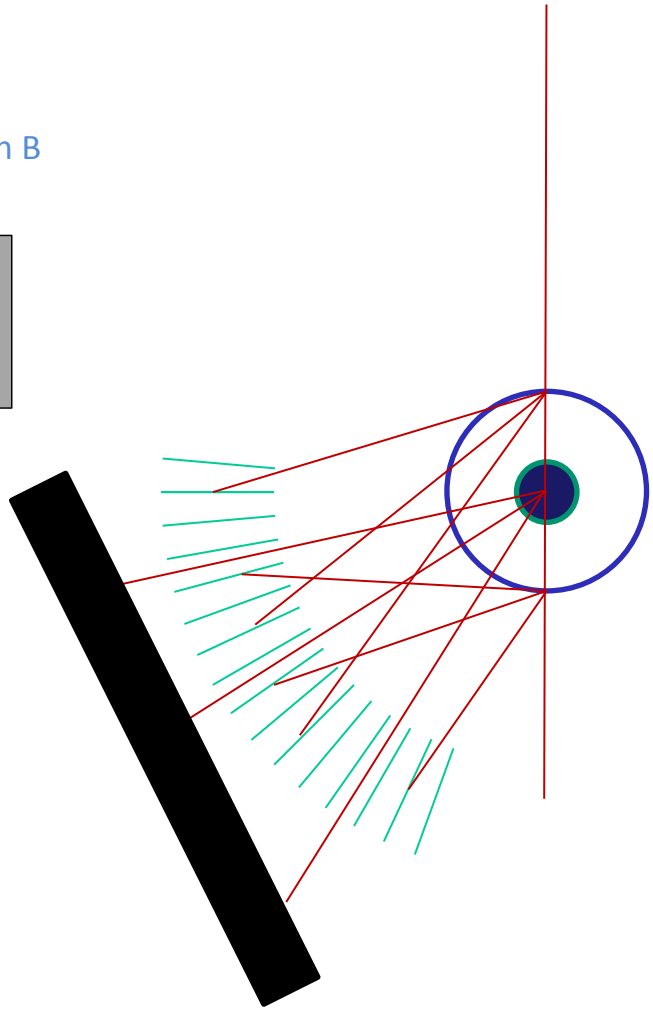
Some candiate materials for sample cells

Material	T / °C	P / bar	pros	cons
Al	< 100		-Low background	-Primary and multiple scattering -Temperature limit
Quartz	320	5	-No Bragg peak	-Fragile -Amorphous background -React with molten comples hydrides -Thermal isolator
Inconel	<200	70		-Primary and multiple scattering
Cu-coated V	< 400	< 40	-Minor Bragg peaks	-Defect in coating
TiZr alloy with a thin stainless steel inner liner	300	1210	-Expands the range in p, T	-Primary and multiple scattering
Sapphire	80 (500)	100 (1500)	-Diffraction spots can be avoided	-Brittle -React with molten complex hydrides -Thermal isolator

Idea!

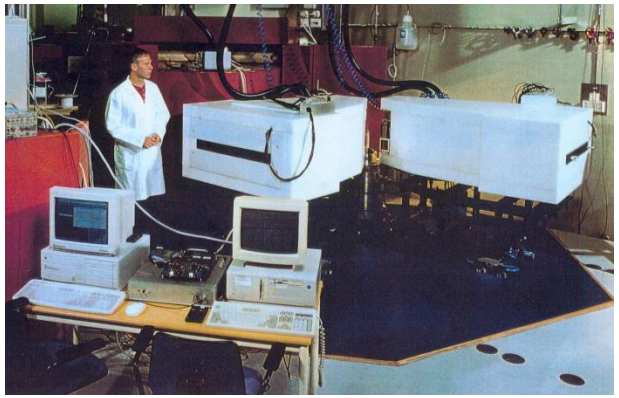

cross section A
Aluminum container
sample in quartz tube
A
B


cross section B
heating element (+water cooling?)



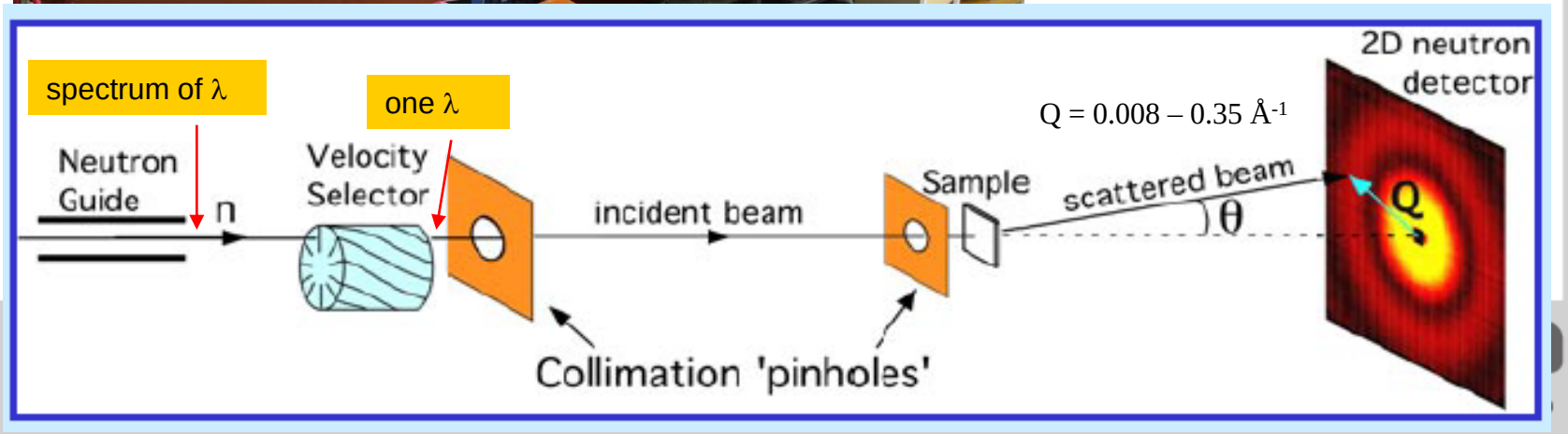
What do we need for a measurment?

- Sample in powdered form
- Fairly large amount of sample, at least 1 cm^3
- Deuterated samples are much preferred
- All sample handling and measurement can be done under inert atomsphere.

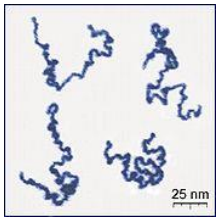


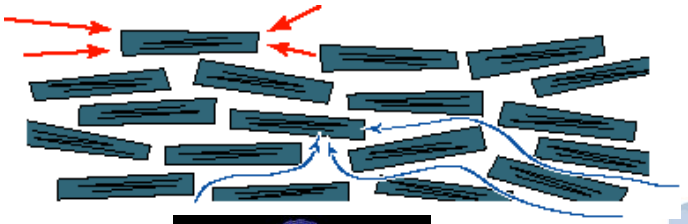
SANS
(Small Angle Neutron Scattering)

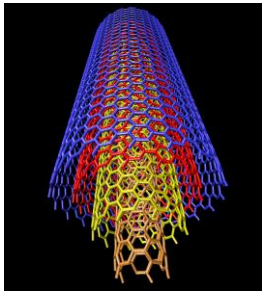
Uses small scattering angles and neutrons with "long" wavelength (5-10 Å) to see "large" things.

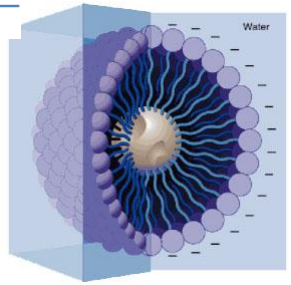


Materials investigated by SANS

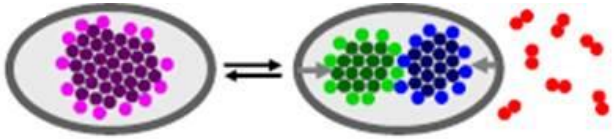








- Polymers
- Clay
- Nano-carbon
- Micelles
- Hydrogen storage materials in nanoscopic pores



- What do we need for a measurment?
  - Sample in powdered or liquid form
  - Fairly large amout of sample, about 0.5 cm^3

Conclusion

- IFE offers Powder Neutron Diffraction and Small Angle Neutron Scattering to the H2FC infrastructure.
- Improved facilities for diffraction under high hydrogen pressure is under planning.