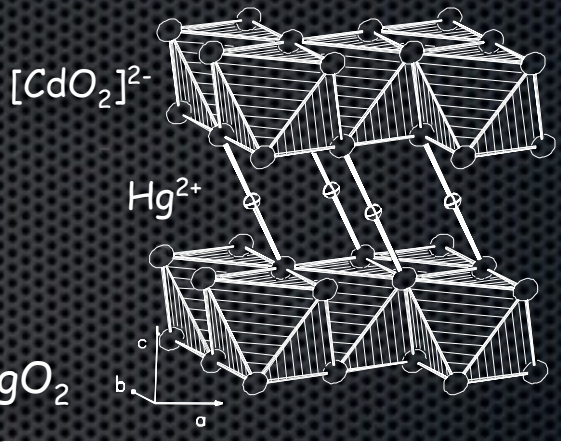
DESIGNING AND BUILDING A NEUTRON INSTRUMENT
XIV School of Neutron Scattering Francesco Paolo Ricci (SoNS)
Erice, 1-9 April 2016

Me and "my" instrument

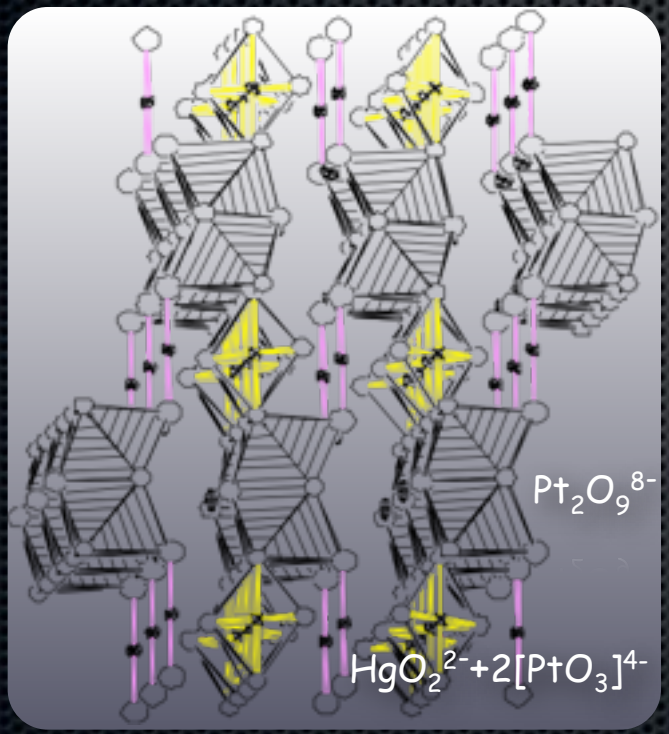
Thomas Hansen
Institut Laue-Langevin - Diffraction Group
D20 high intensity powder diffractometer responsible

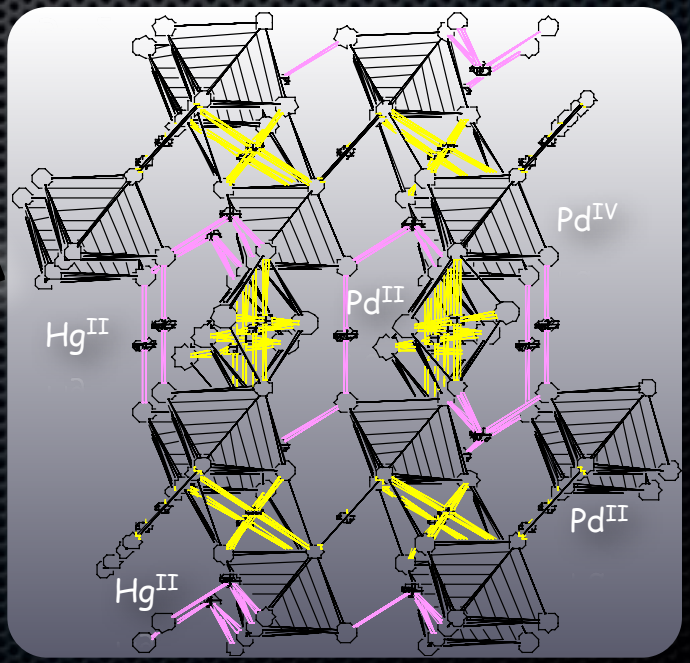
High pressure synthesis of oxomercurates

- Chemistry, Kiel, Germany
  - Hk. Müller-Buschbaum, Inorganic Chemistry
- Mercury oxides at high pressure of oxygen
  - 600°C,
  - 8 days,
  - 6 kbars O_2



$CdHgO_2$



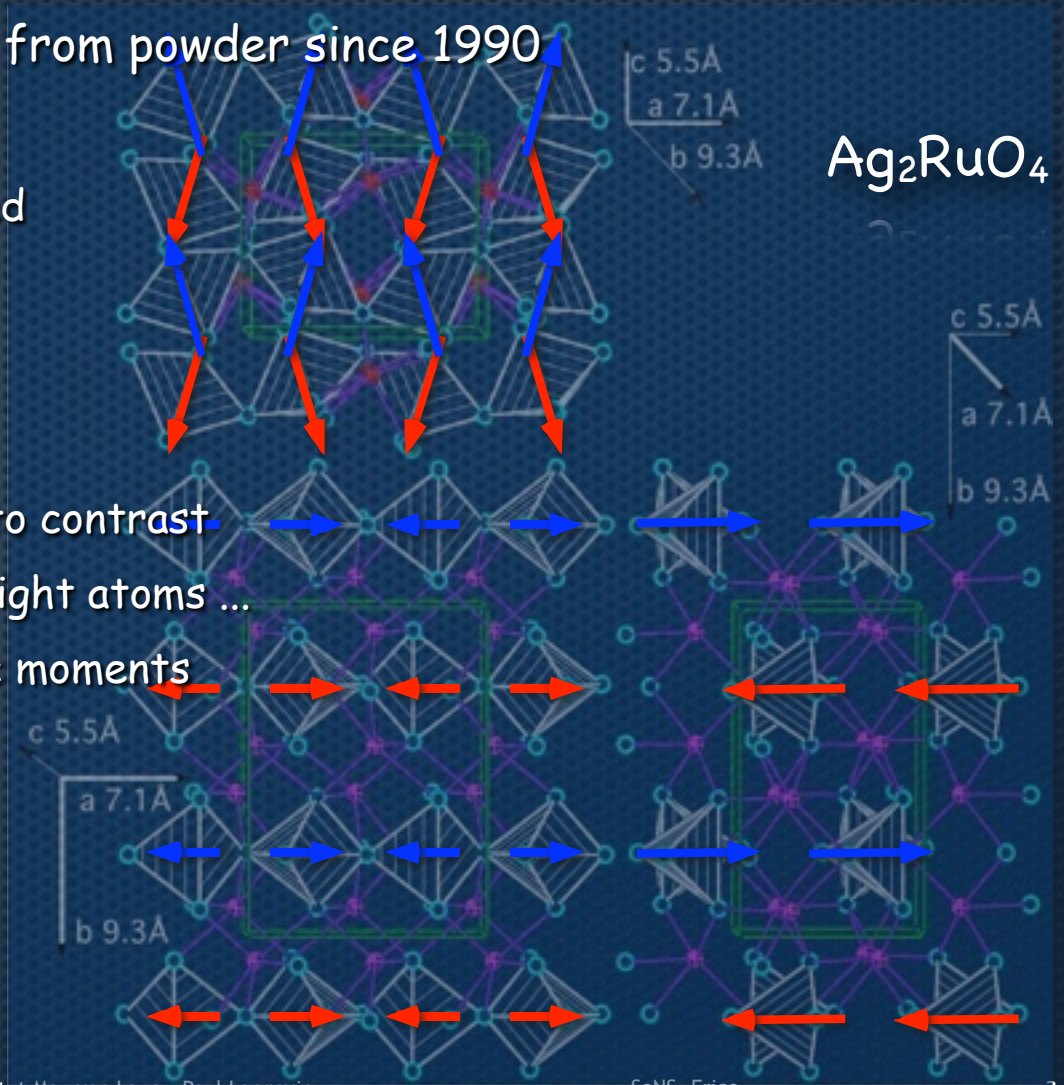


$Ba_3HgPt_4O_{11}$

$Ba_2Hg_3Pd_7O_{14}$

Structure determination from powder

- Post-Doc with A. Le Bail in Le Mans, France
  - Continuing on mercurates, starting on ruthenates, "wet" chemistry
- ab initio structure solution from powder since 1990
- Indexing problem
  - high resolution data needed
- Intensity extraction
  - overlapping reflections!?
- Direct methods
  - X-rays often better, due to contrast
  - however, neutrons locate light atoms ...
  - ... and, of course, magnetic moments



NEUTRONS FOR SCIENCE

D20

Versatility

High Intensity

4 Monochromators

5·3 take-off angles

Soller Collimators, slits

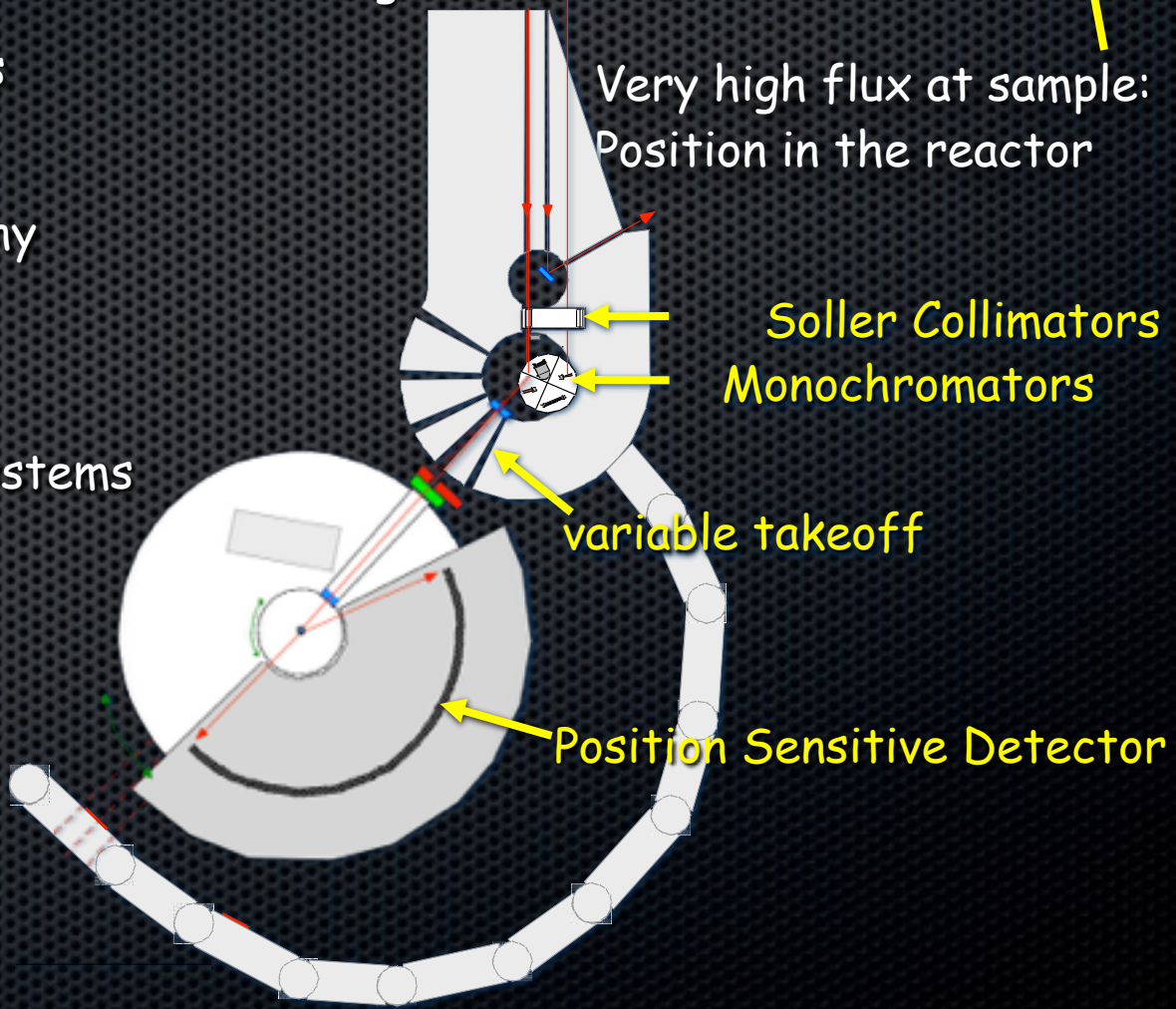
Detecting maximum of neutrons:
Large PSD, 160°, definition 0.1°

Very high flux at sample:
Position in the reactor

Resolution
Q range
Intensity
Wavelengths

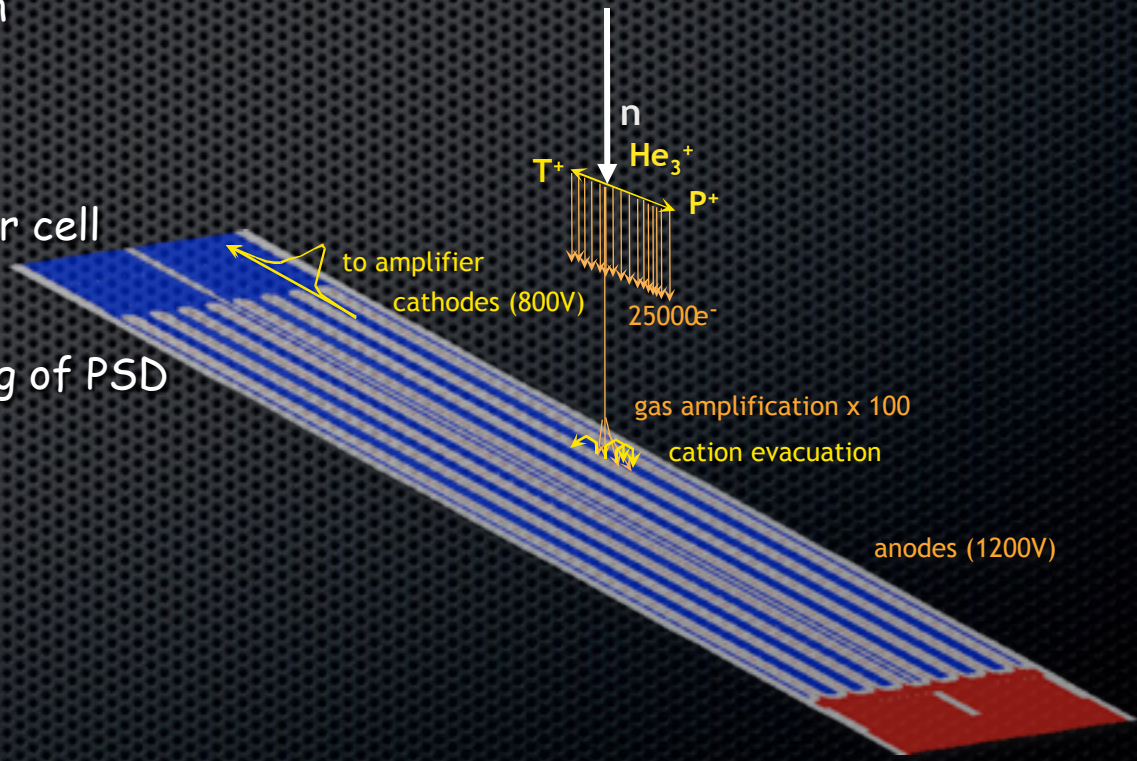
0.82 to 2.51 Å

Crystallography
Kinetics
Magnetism
Disordered systems



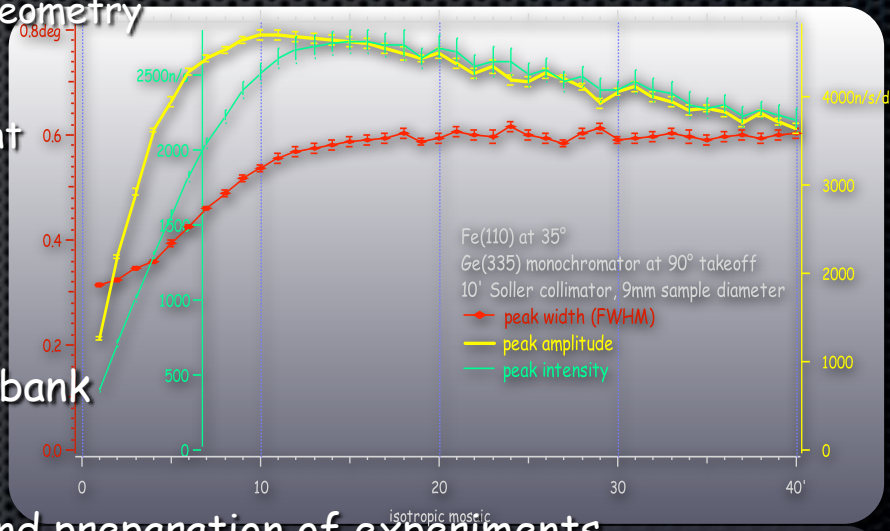
Micro-strip gas chamber PSD

- New technology:
  - Precise and stable geometry & high stability
  - High gaseous amplification with low high voltage
  - Fast cation evacuation & high counting rates
- Micro-strip plates
  - 2 anodes/cathodes per cell
  - 32 cells per plate
  - 48 plates in Al-housing of PSD

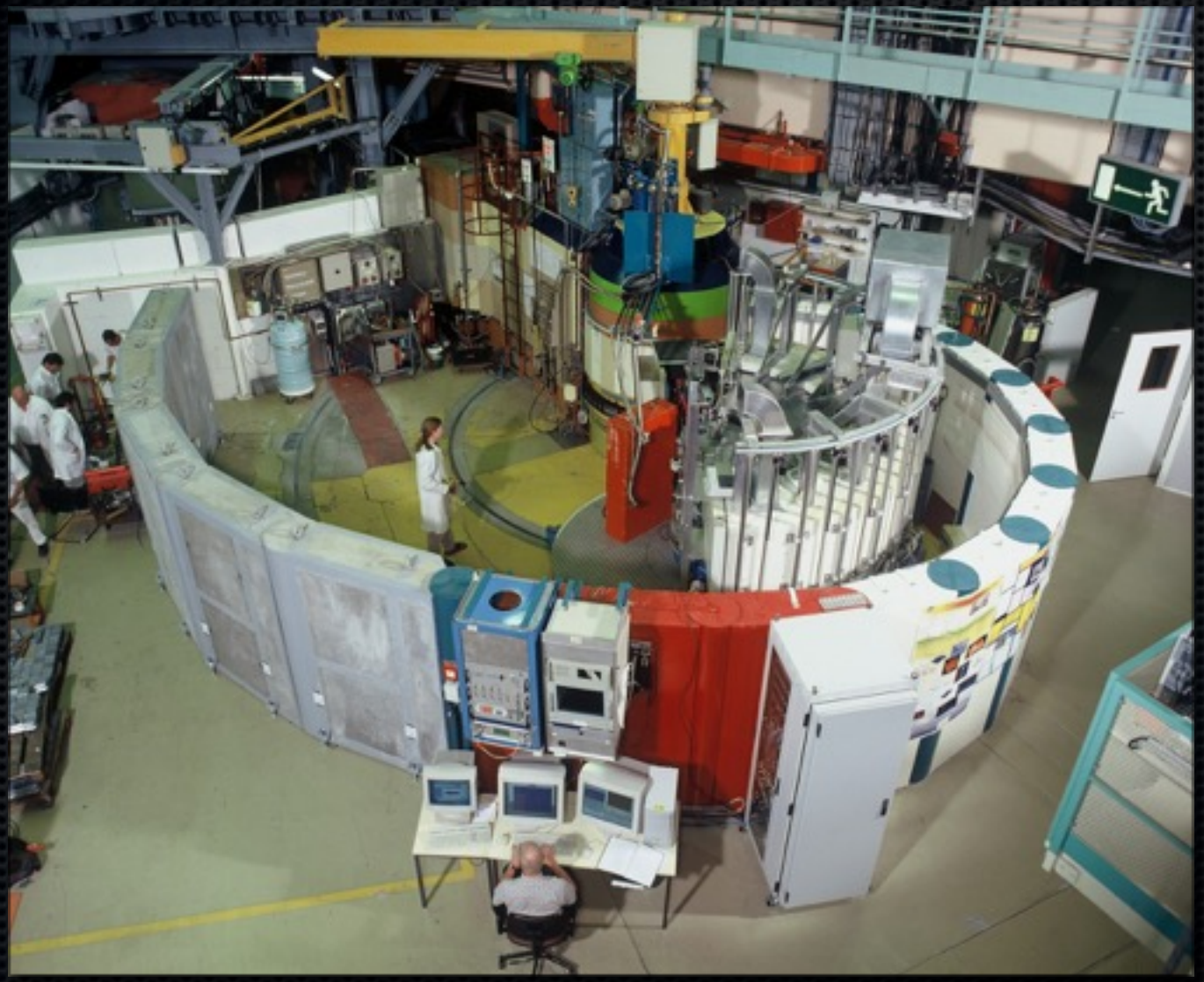


Monte-Carlo instrument simulation

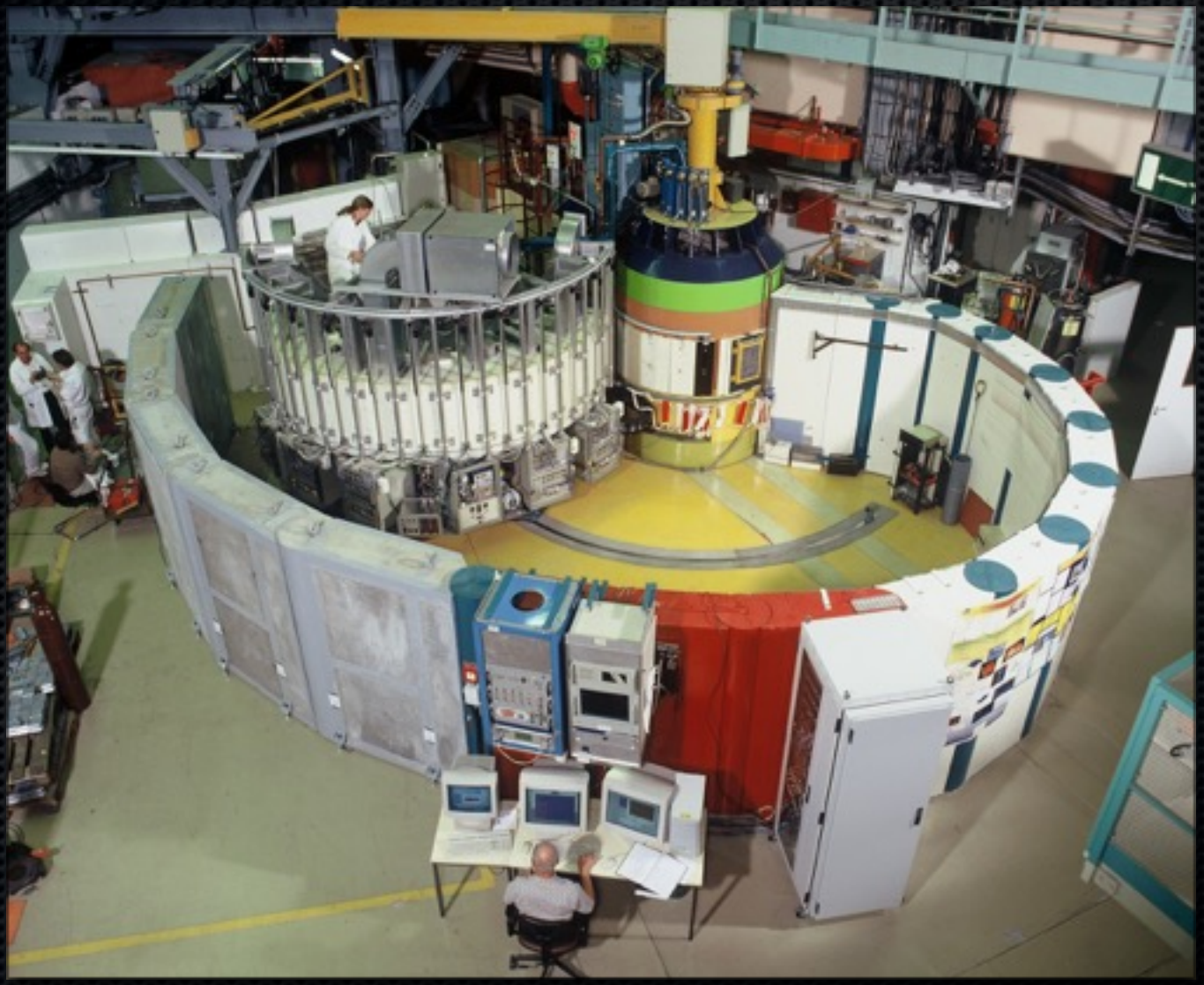
- McStas on D20 … or any other 2-axis diffractometer
  - Quantitative description of the source and incident optics
  - Realistic treatment of monochromator crystals
    - Finite thickness: reflectivity from structure factor
    - Multiple scattering: asymmetrical shift of neutron beam
    - Monochromators in transmission geometry
  - Complete description of sample
    - Contribution of sample environment
    - Multiple scattering
    - Incoherent scattering
  - Radial oscillating collimator
  - Gas chamber PSD, multi-detector bank
- What's the use?
  - Optimisation of new components and preparation of experiments
  - Observation of inaccessible information about the neutron beam



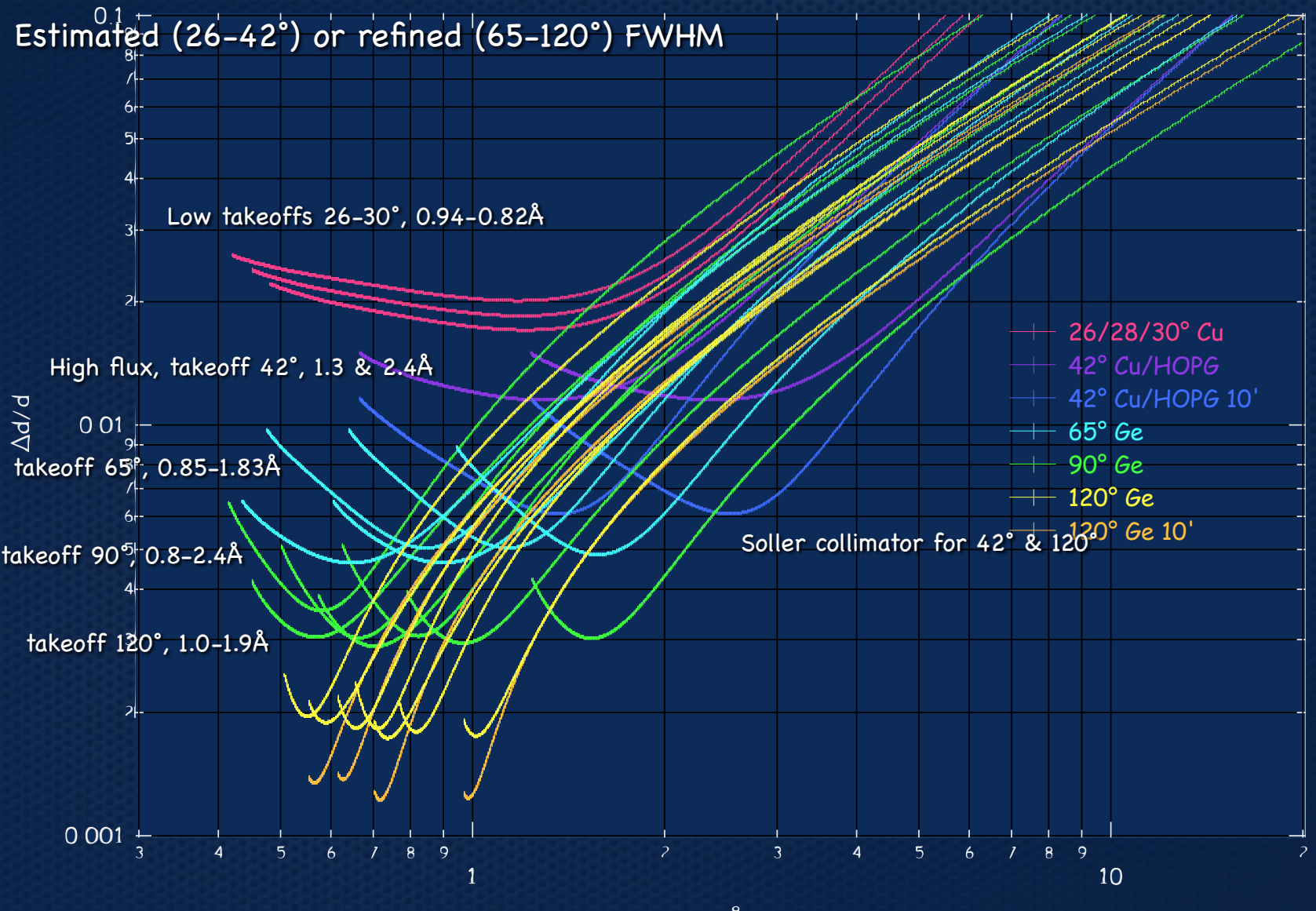
26°



120°



Available resolution and d-spacing

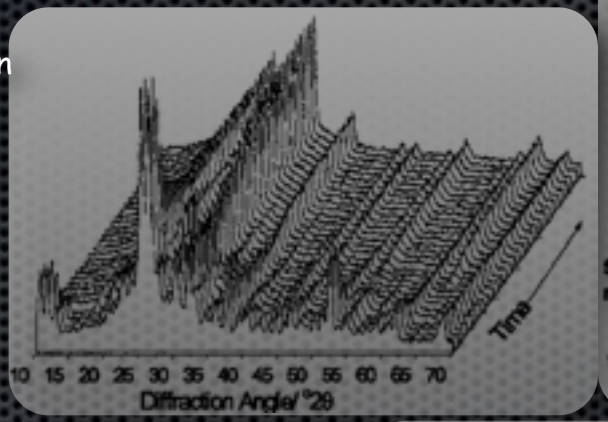


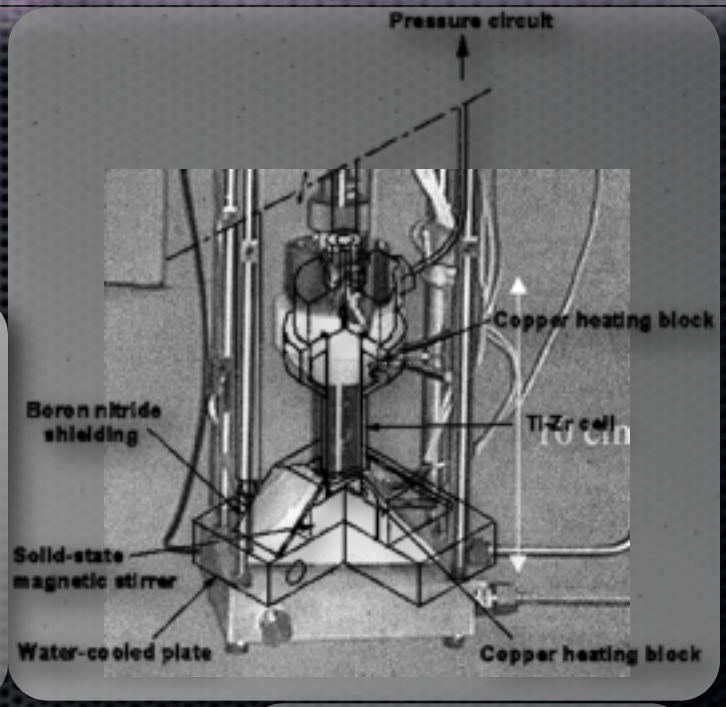
High intensity powder diffraction!

- **Parametric** diffraction (in situ)
  - **time**-resolved diffraction: I(2θ,t)
  - variation of temperature: **thermodiffractometry**: I(2θ,T)
  - variation of **pressure**: I(2θ,p), magnetic **field**: I(2θ,H), etc.
  - variation of **stoichiometry**: I(2θ,x) (many samples)
  - texture: many sample orientations: I(2θ,χ,φ)
- Small samples or **small signal** from sample
  - realization of **extreme conditions**:
    - **high pressure** or homogenous high temperature
  - limited availability:
    - expensive isotopes for isotope exchange experiments
    - difficult (reproducible) synthesis, e.g. in high pressure cells
    - biomaterials (bones)
  - high absorption (boron, hydrogen, cadmium, europium, gadolinium, ...)
- **Precise intensity**
  - **differential** experiments: weak peak intensity in magnetism or physisorption
  - **disordered** systems: liquids and amorphous materials

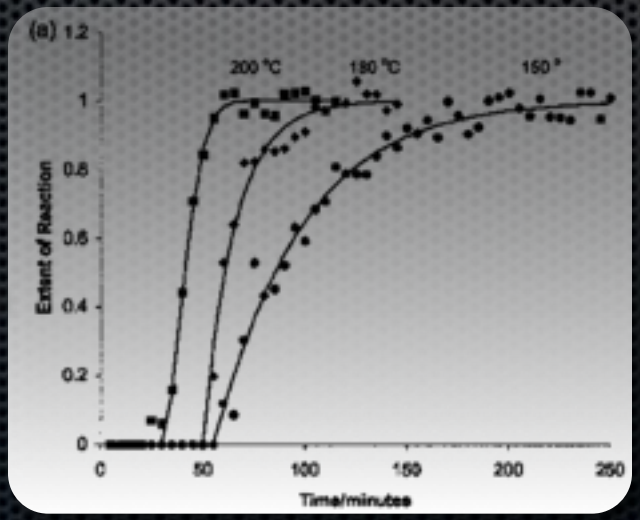
Hydrothermal Crystallization of $BaTiO_3$

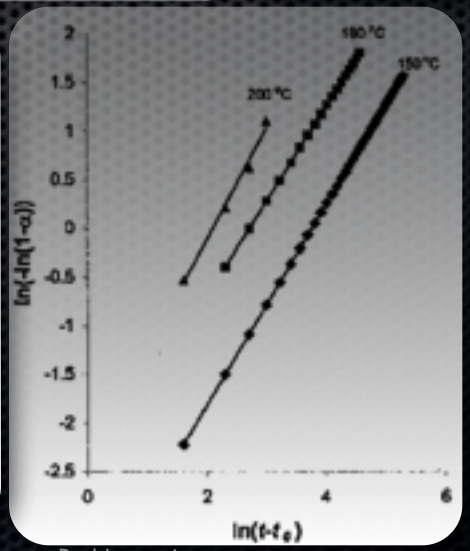
- $BaCl_2$+TiO_2 in NaOD/D_2O solution
- Avrami-Erofe'ev expression
  - Sharp-Hancock ln[-ln(1-a)]=n ln(t-t0)+n ln(k)
    - n ≈ 1
      - Rate: nucleation site formation
    - k: Arrhenius plot
      - 55 kJ/mol

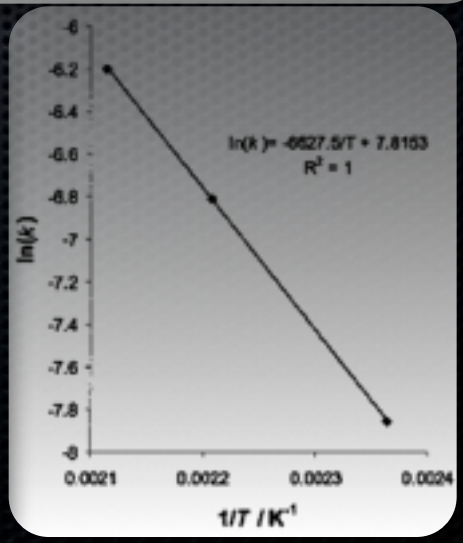




R.I. Walton, F. Millange, R.I. Smith, T.C. Hansen, D. O'Hare, *JACS* **123** (2001) 12547-12555.

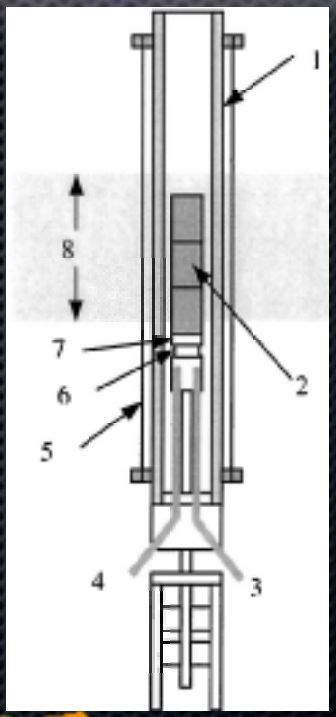


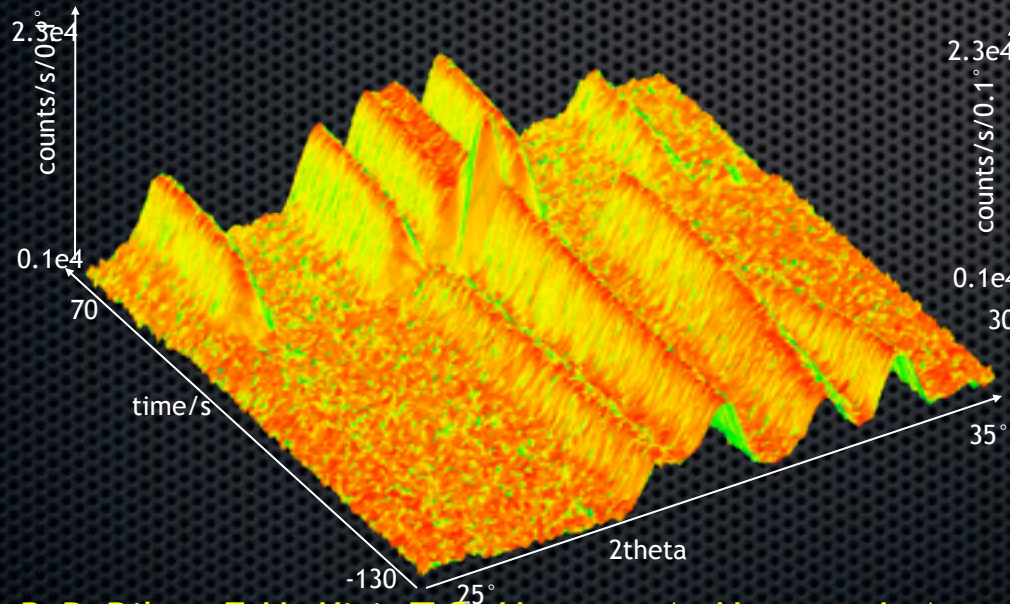


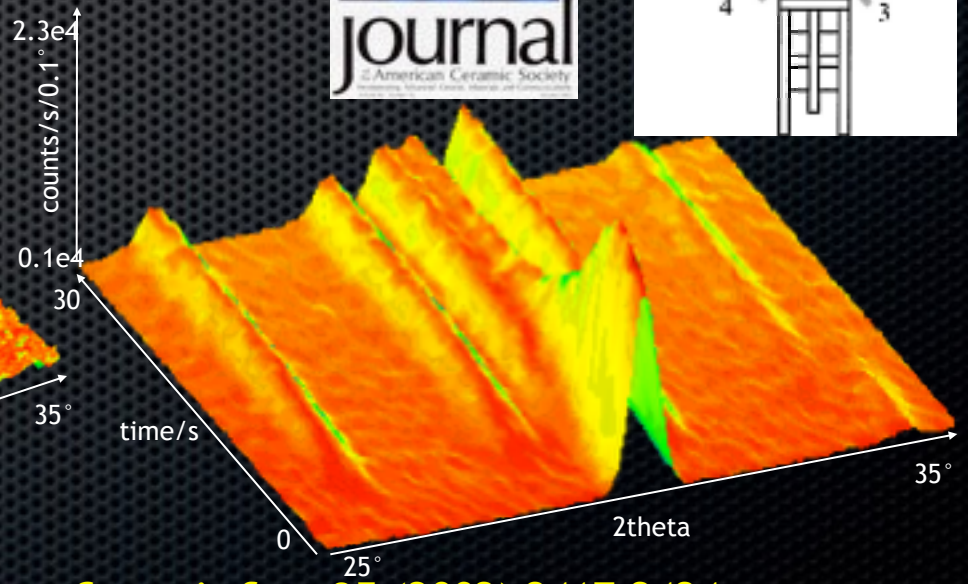


Self-propagating High-T Synthesis (SHS)

- Titanium silicon carbide Ti_3SiC_2
- Self-propagating High-temperature Synthesis (SHS)
  - Riley, Kisi et al.: 3 Ti : 1 Si : 2 C, 20 g pellet in furnace
  - Heating from 850 C to 1050 C at 100 K/min
  - Acquisition time 500 ms (300 ms)
- Hot isostatic pressing expensive



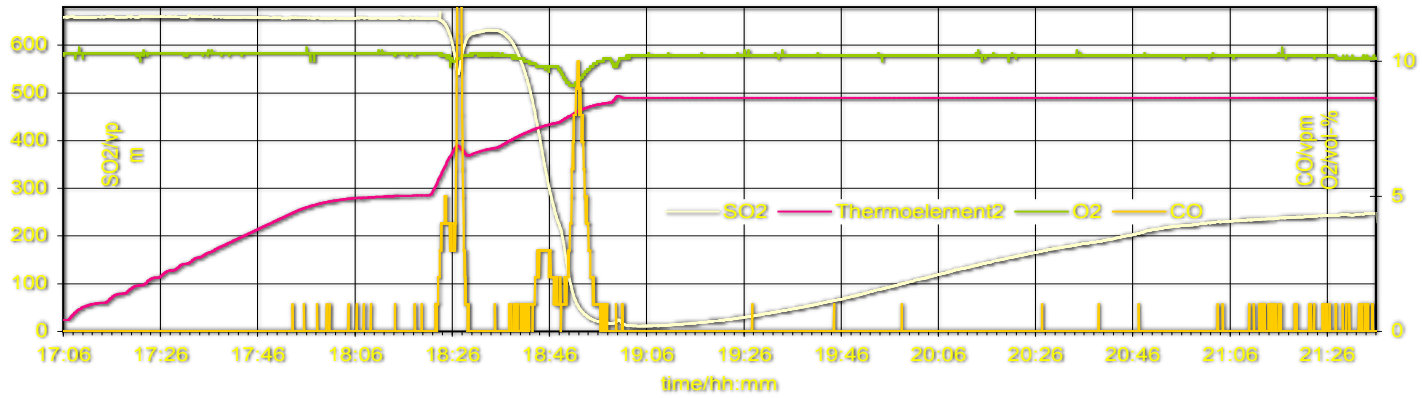


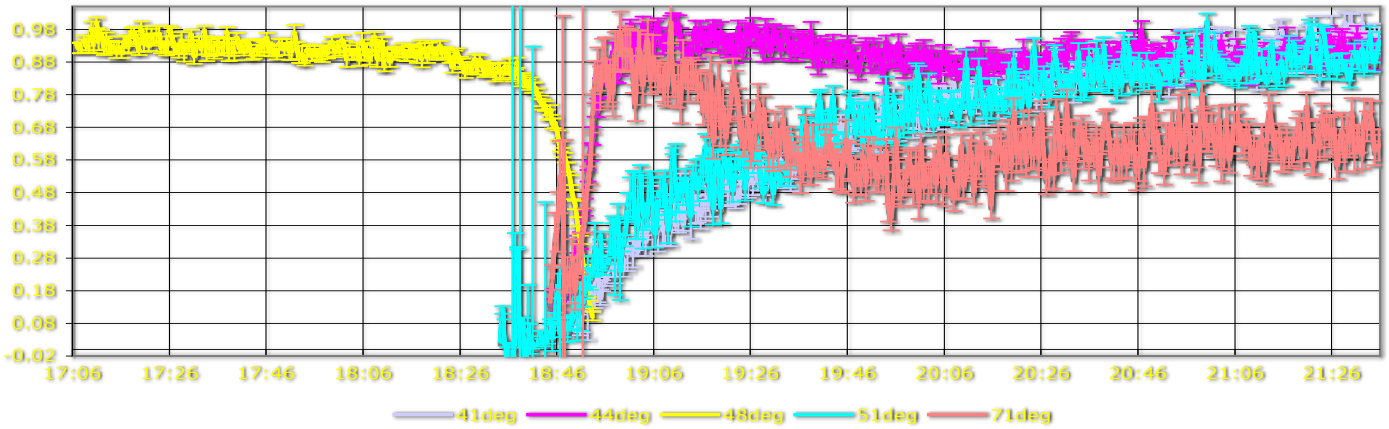


D.P. Riley, E.H. Kisi, T.C. Hansen, A. Hewat, *J. Am. Ceramic Soc.* **85** (2002) 2417-2424.

Industrial processes

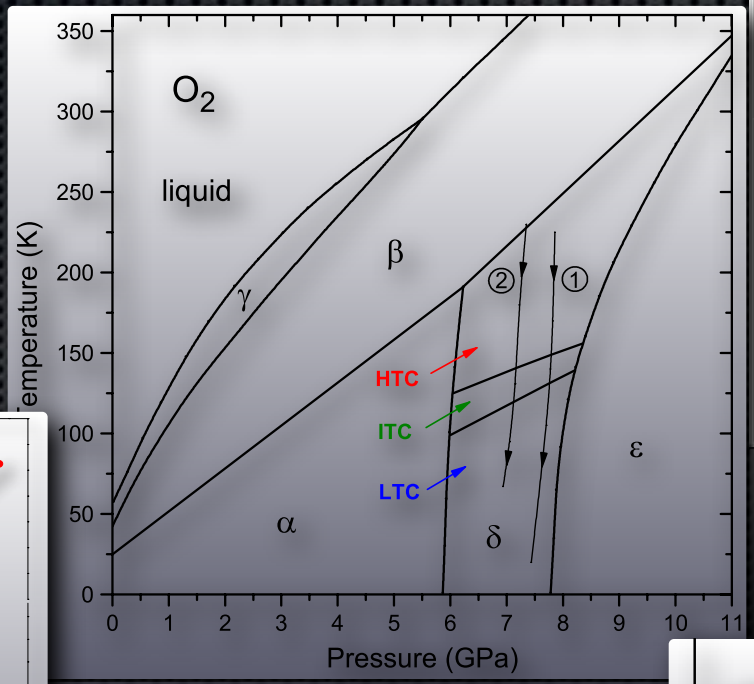
- flue gas desulphurisation & magnetic roasting

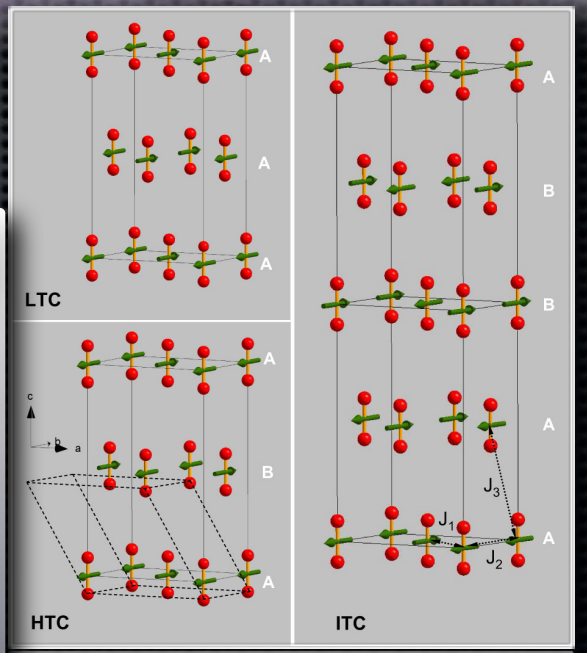


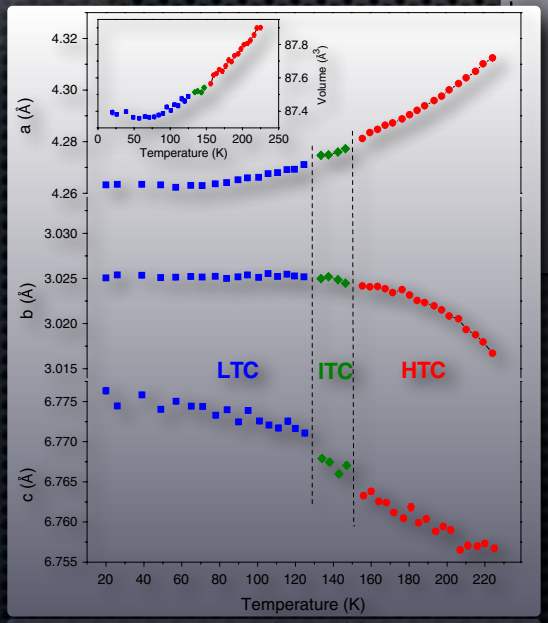


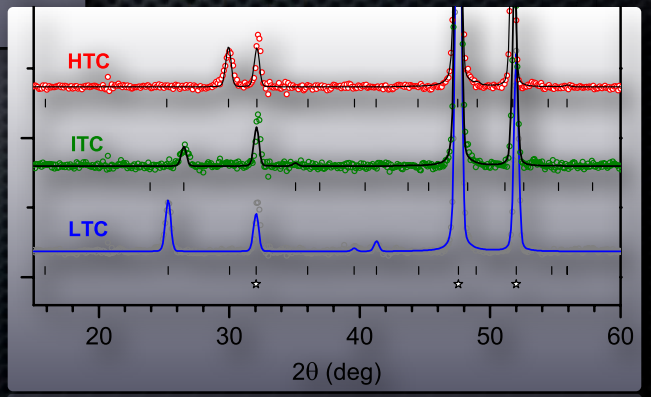
High Pressures at D20

- Three new magnetic phases of δ-oxygen
  - solid O_2 phases NOT "spin-controlled"

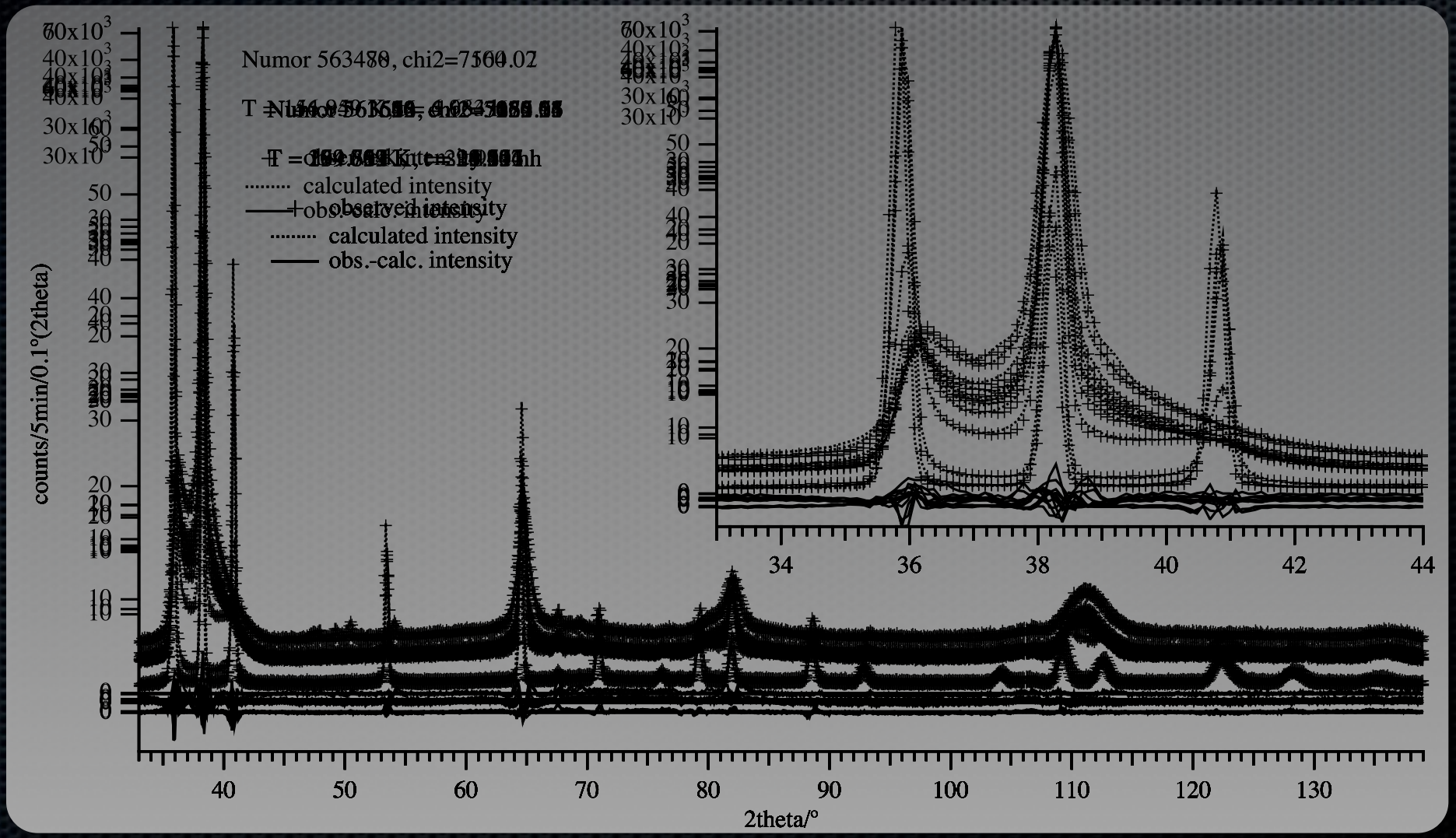








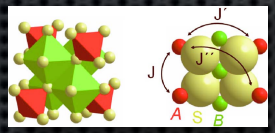
Ice Ic$_V$ annealing: end of each T-stage

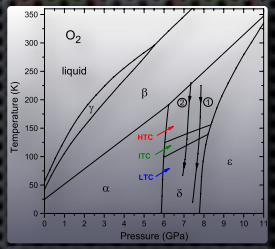


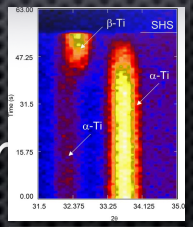
Scientific Output: blockbusters, …

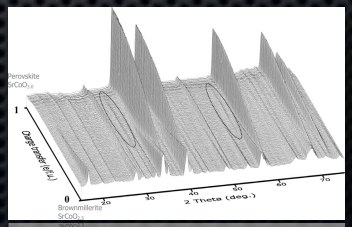
Fields by a quick look at the most cited papers 2005-10

- Magnetism
  - low-dimensional systems
  - frustrated systems
  - mesoporous systems
  - nano-particles
  - perovskites
  - pnictides
  - high pressure phases
- Solid state chemistry
  - SHS
  - Electrochemical oxidation
  - Lithium ion conductors

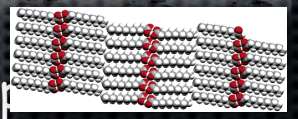


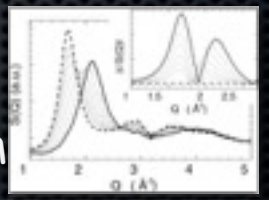






- Physical chemistry
  - Carbon nanotubes
  - physisorption
  - amorphous polymorp
  - confined systems
  - metallic melts
  - correlations in polym





- Geosciences
  - contaminant uptake of water
  - gas-hydrates
- Materials science
  - fatigue processes
  - shape-memory alloys

A workhorse instrument ...

- A workhorse does a lot of quite different heavy duty things ...
- ... but one thing it is not, it is ... **not sexy at all!**