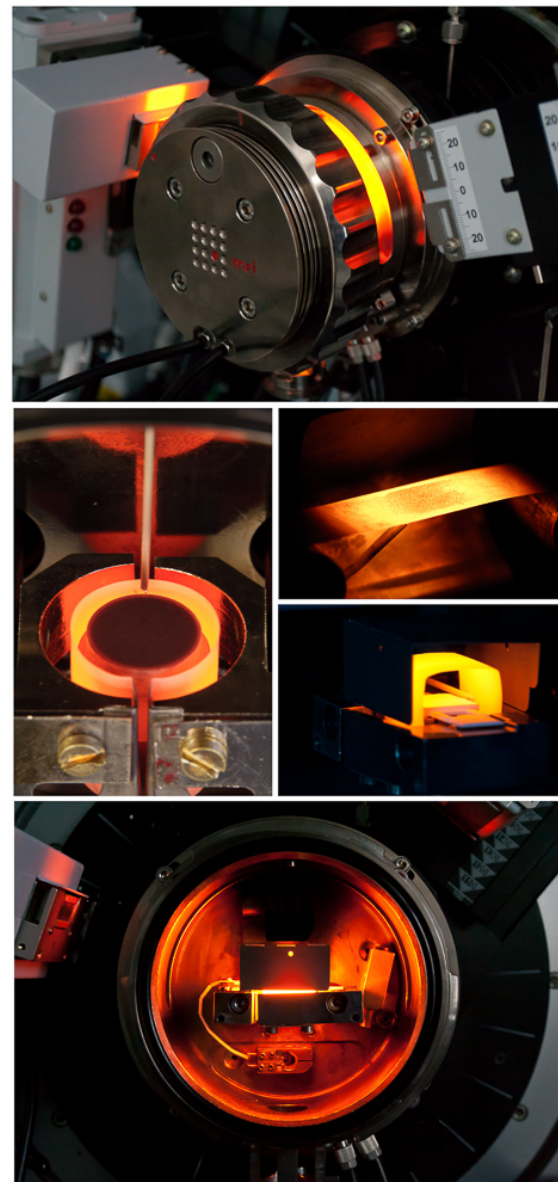# Non-Ambient XRD

Julian Tolchard

Non-ambient / in-situ

- Experiments requiring special conditions
- Fundamental material understanding
  - Phase transitions, structural distortions
- Reproduce "real" application / process conditions
  - High/Low temperature
  - High/Low pressure (mechanical or gas)
  - Reactive environments
  - Solutions
  - Electrical potential
- Collect data as a function of controlled variables
  - Temperature, pressure, potential, time…
- Ex-situ measurements not possible
 - Sometimes in-situ faster and easier

Case Studies

Case study 1 – LaFeO$_3$

- 11 day data collection
  - RT-1450°C, 71 scans, 15-140° 2θ
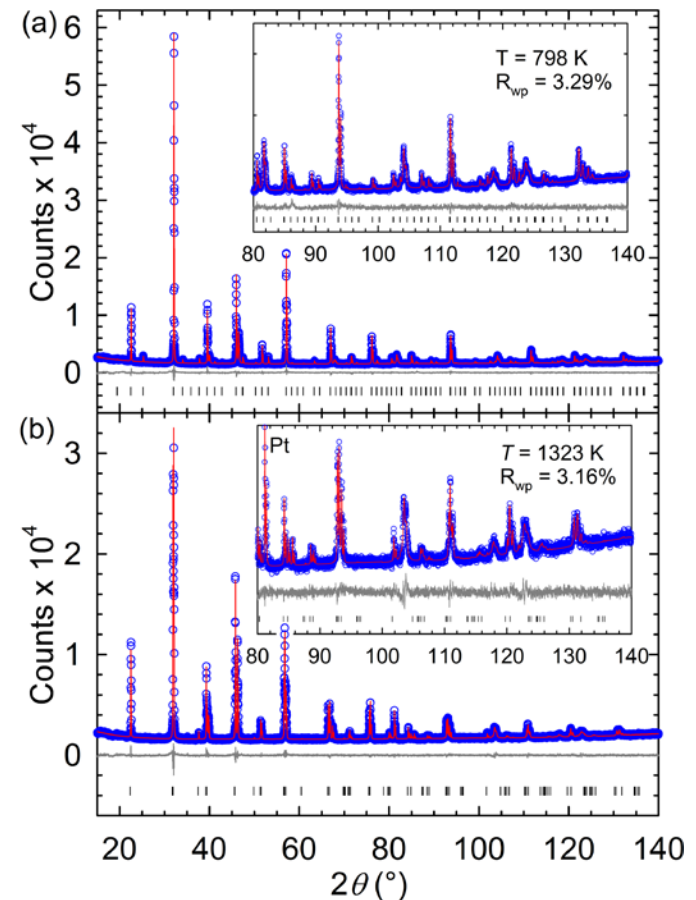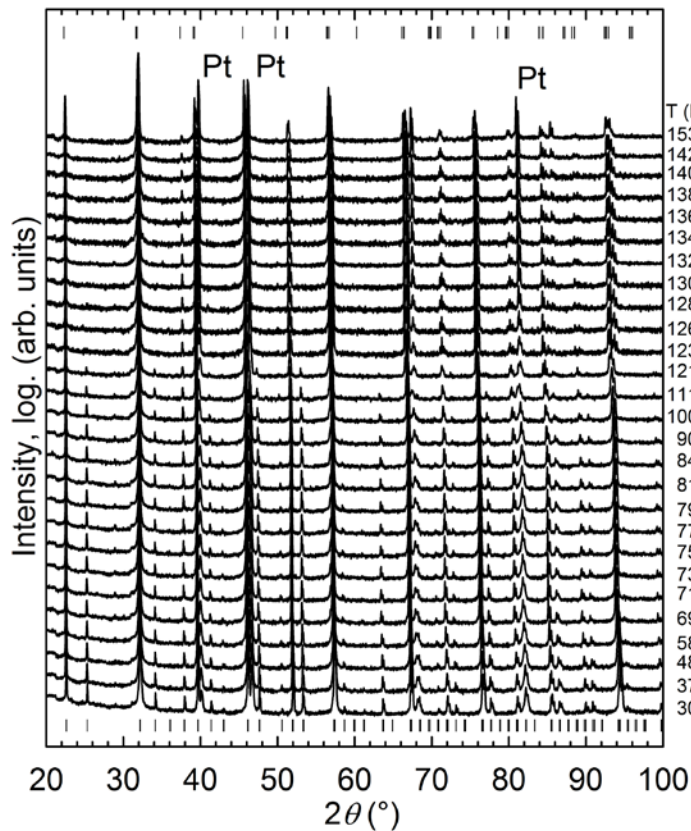
Case study 1 – LaFeO$_3$

- ## Phase transition
  - Pbnm to R-3c

- ## Non-isotropic expansion
  - Two cell axis crossovers
  - Unit cell metrically "tetragonal"

- ## Cell distortion around T$_N$
  - Octahedral distortions
  - Magnetostriction

S.M. Selbach et. al. J Solid State Chem. 196, (2012), 249
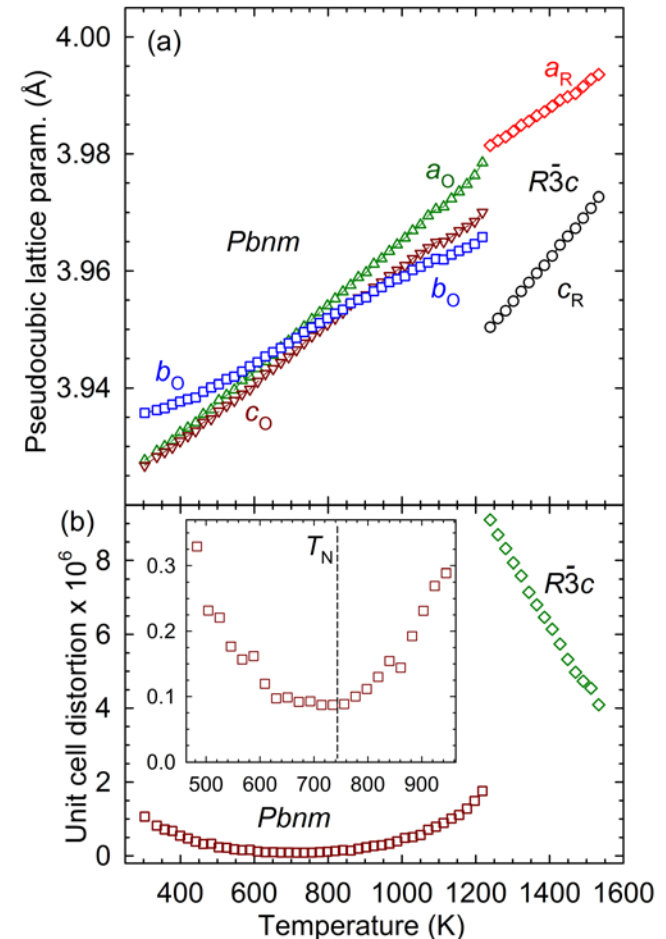
Case study 2 – setting cements

- No heating, no gas flow.
  - Just add water, close the system and watch
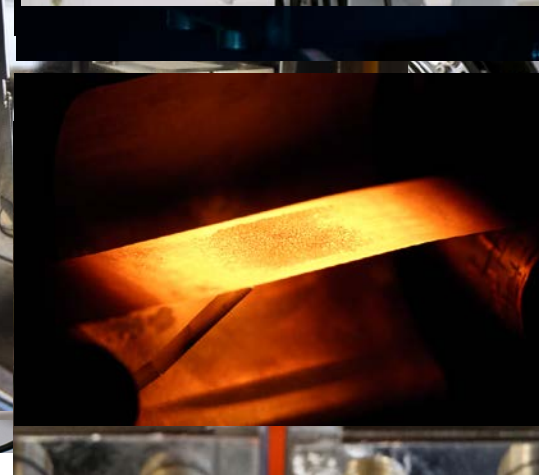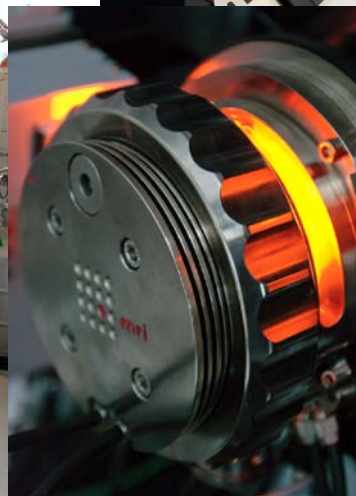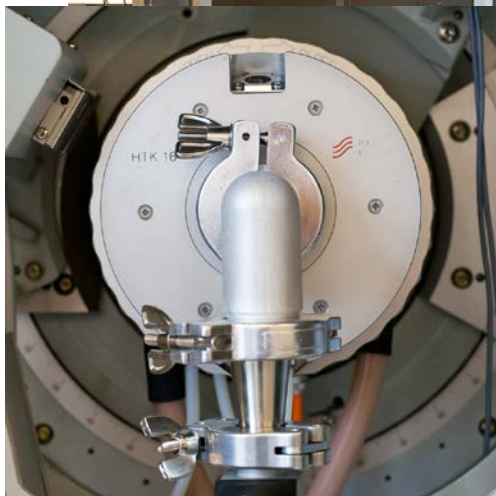
Tobias Danner, PhD 2013
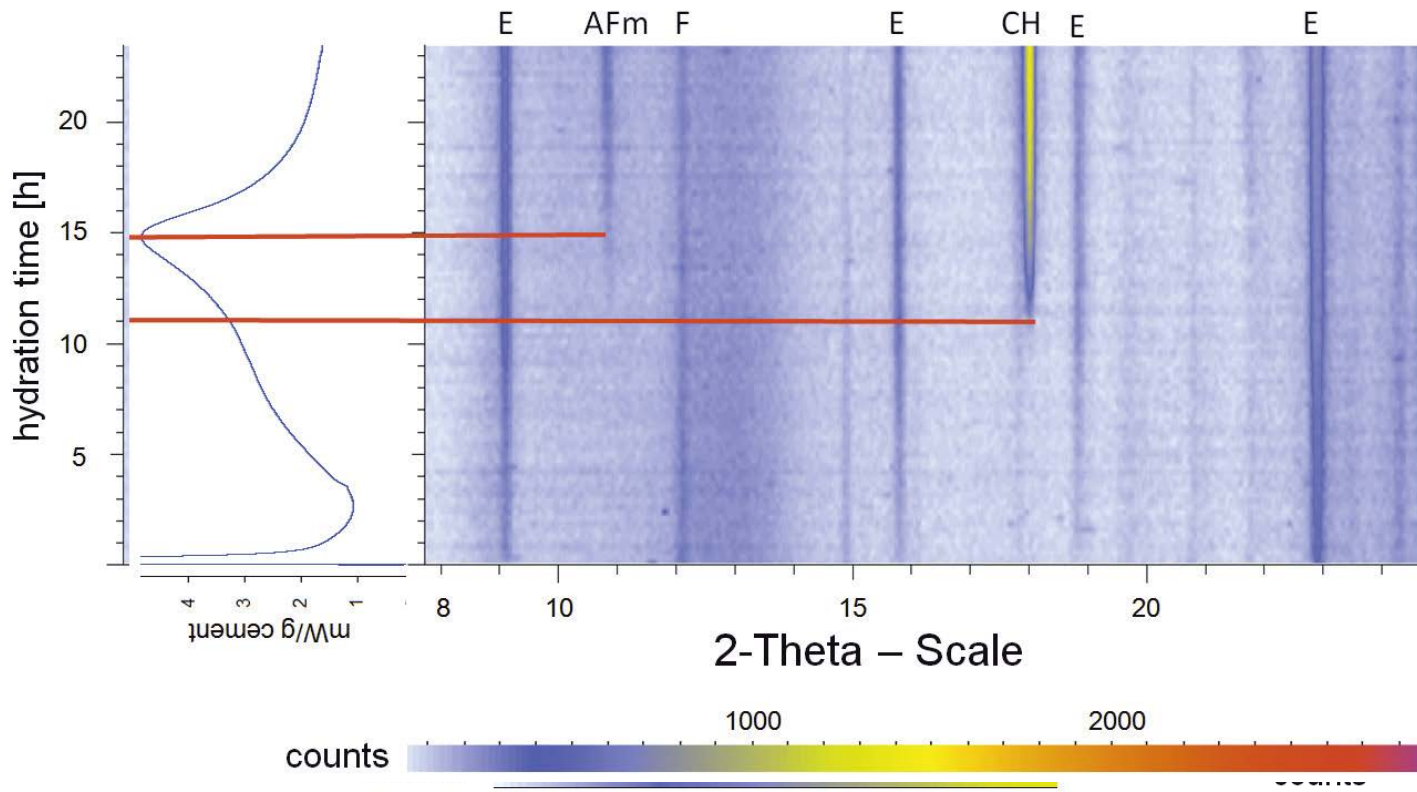
Facilities

NTNU Node

- Dedicated high speed instrument (HT-XRD)
  - D8 Advance with Våntec-1 PSD (12° detector opening)
 - Reflection mode, Cu-K$_\alpha$

NTNU Node

- Lithium Battery Cycling
    - Bespoke reflection mode cell (Cu or Mo)
    - Coffee bag cells for transmission measurements (Mo)
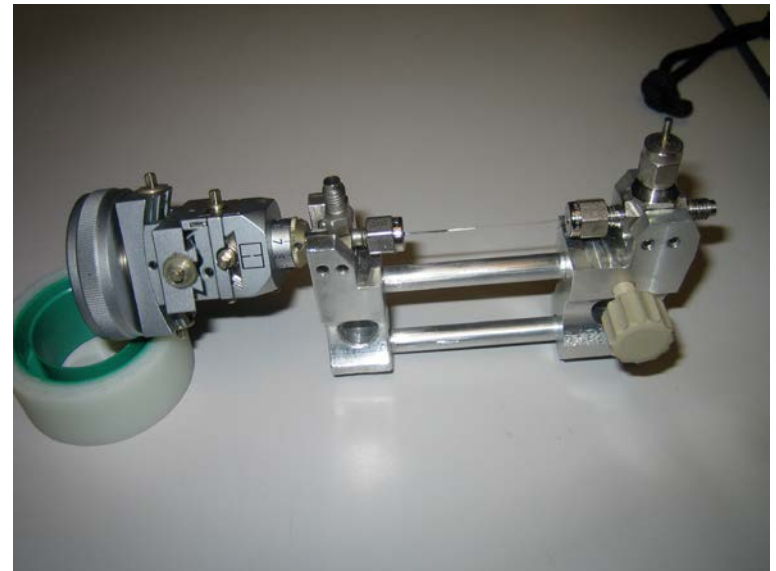
UiO Node

- Paar HTK 1200 stage for PXRD
  - Radiant Al_2O_3 heater
  - RT-1200 °C
  - Air or vacuum
  - Flat plate or capillary samples
- Paar DHS1100 Thin film heater
  - AlN radiant heater
  - RT-1100 °C
  - Air or vacuum
- Hot air blowers
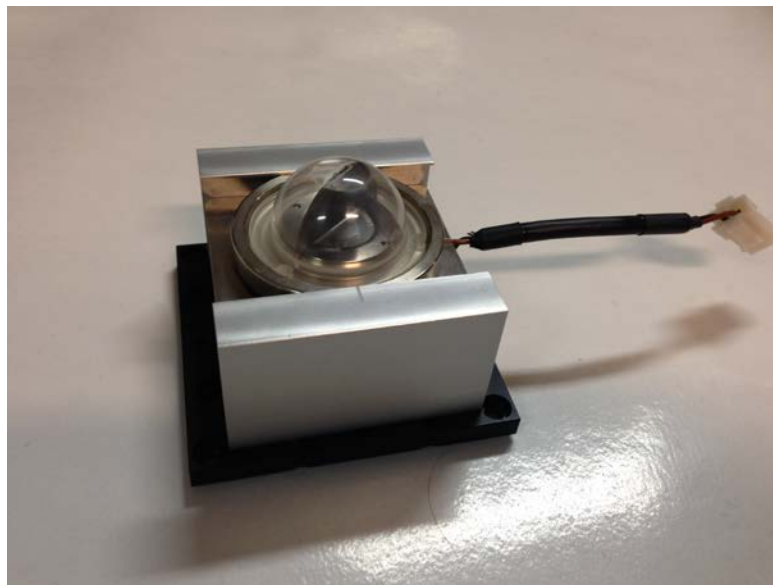 - Heating to 700 °C
  - Capillary samples
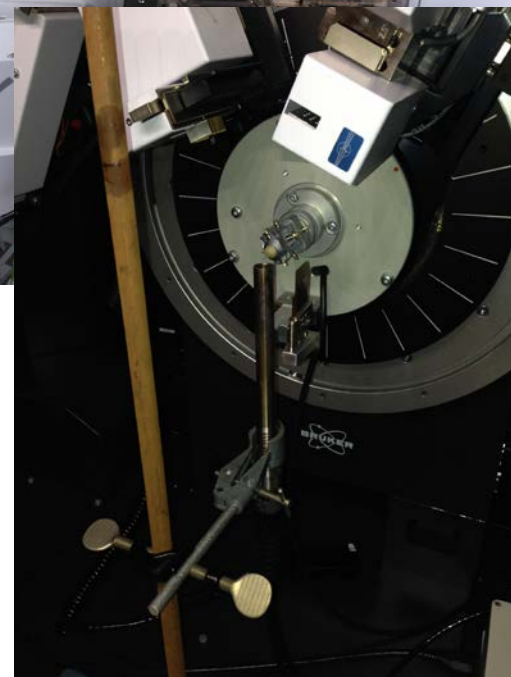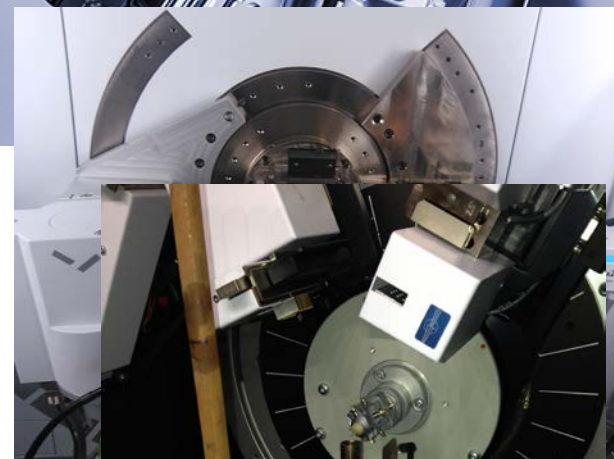
UiO Node

- Capillary flow cell
  - Heated by blower
  - Extensively tested at SNBL
  - Spatial resolution possible with xzy stage
- Huber Image plate XRD
  - 10K-RT
  - RT-900 °C
  - RT-1700C (Laser heater)

UiO Node

- Bruker coin cell battery testing system
- Transmission mode film based coin cell system

Both Nodes

- Expertise
  - We've been doing this a while…
- Software and databases
  - Access and training
- Bespoke solutions
  - We quite enjoy problem solving ☺

Thank you for not sleeping…