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RECX – A Norwegian Centre for X-Ray Diffraction, Scattering and Imaging

Julian Tolchard

Overview

- Organisation
- Motivation
- Capability
  - What relevance is it to you?
  - How do you use it?

RECX Organisation

What is RECX?

- Norwegian Centre for X-ray Diffraction, Scattering and Imaging REsource Centre X-rays
  - Admittedly not a very "catchy" name
- Virtual centre across UiO and NTNU
- Funded jointly by Forskningsrådet, UiO, and NTNU
  - 25.5MNOK from Forskningsrådet
  - >20MNOK from UiO and NTNU
- New equipment / personnel
- "Donation" of existing equipment to centre
- 5 year commitment (initially)

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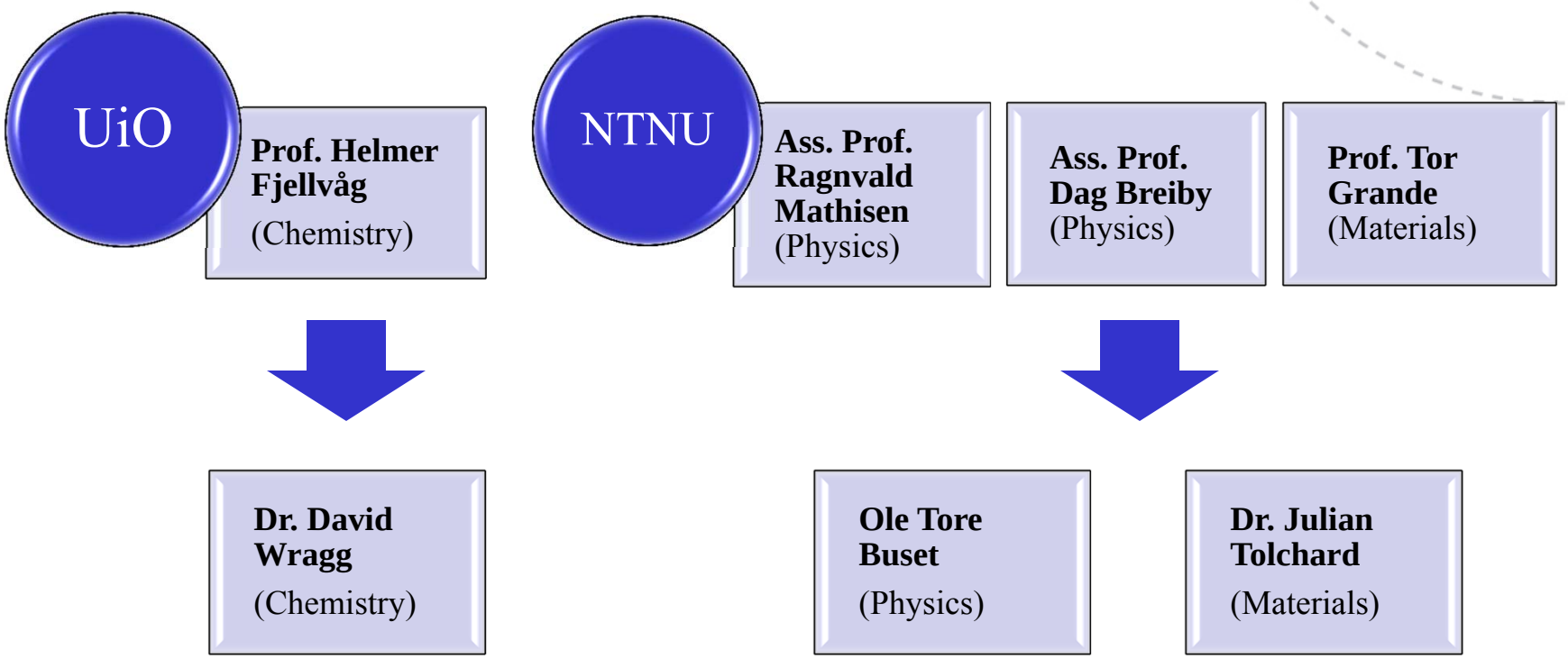
Where is RECX?

- Dept. of Chemistry, UiO
  (http://www.mn.uio.no/kjemi/english/research/about/infrastructure/xrd-lab/)
- Dept. of Physics, NTNU
  (http://www.ntnu.edu/physics/xray)
- Dept. of Materials Science and Engineering, NTNU
  (http://www.ntnu.edu/mse/powder-diffraction-lab)

Who is RECX?


UiO
Prof. Helmer Fjellvåg
(Chemistry)
NTNU
Ass. Prof. Ragnvald Mathisen
(Physics)
Ass. Prof. Dag Breiby
(Physics)
Prof. Tor Grande
(Materials)
Dr. David Wragg
(Chemistry)
Ole Tore Buset
(Physics)
Dr. Julian Tolchard
(Materials)

Why establish a National Centre?

- Maintain academic strength in x-ray scattering / imaging
- Formally link existing academic labs
  - Provide a framework for exchange of knowledge/expertise/capability
- Improve communication between industry and academia
  - Norway has a relatively high level of industrial XRD usage
- Provide "capability" to the Norwegian X-ray community
  - Access to equipment and competence

What are the goals?

- Improve competence in x-ray scattering/imaging techniques
- Maintain and expand academic use of x-ray methods
  - Both at lab scale and at major central facilities
- Direct use of the infrastructure by external users:
  - 4 universitities and colleges
  - 3 research institutes
  - **8 Norwegian companies**
- To establish RECX as a meeting place for x-ray based research and analysis in Norway
- Keep growing beyond our 5 year mandate

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RECX roadmap

- We are up and running now
  - Three established, well equipped labs
- Equipment tenders in process
  - Decision Nov/Dec 2012
  - Installation spring/summer 2013
- Website early 2013
- Be fully operational by 2014
- Workshop early 2014
  - Show off our full capabilities

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RECX capability

Equipment overview

UiO

- 2 x Siemens D5005
- Huber powder XRD
- Bruker D8 DaVinci (Cu Ka1)
- Powder XRD (Cu Ka1)
- Powder XRD (Mo, Cu)
- Bruker D8 Apex single xtal
- Single Crystal XRD
- SAXS
- Thin Film XRD

NTNU Physics

- SAXS
- Bespoke Diffractometer
- X-Ray Micro Tomograph
- Pilatus detector for SNBL
- High flux source
- Energy dispersive detector

NTNU Materials

- Siemens D5005 (Cu Ka1)
- Bruker D8 Advance (Cu Ka1,2)
- Bruker D8 Focus (Cu Ka1,2)
- Powder XRD (Cu Ka1,2)
- Powder XRD (Mo, Cu)

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Equipment overview

- Numerous temperature stages
  - Both at NTNU and UiO
- In-situ Li ion batteries
- Sample stages and holders for every occasion
  - Micrograms of powder to lumps of steel, air sensitive samples
- State of the art analysis software and databases

Capability

- Powder Diffraction
  - High throughput, high resolution
  - Cr, Cu, Mo radiation / reflection and transmission geometry
  - Multi temperature (80K-1800K), High pressure gas (1000C, 20bar)
- Single Crystal Diffraction
  - Structure evaluation and solution
- Pair Distribution Function analysis (local structure)
- SAXS (particle size/shape)
- Thin film analysis (grazing incidence and reflectometry)
- Micron resolution tomography
- Design your own experiment…

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RECX capability - examples

Powder/Single Xtal Diffraction

- Evaluation/determination of a crystal structure

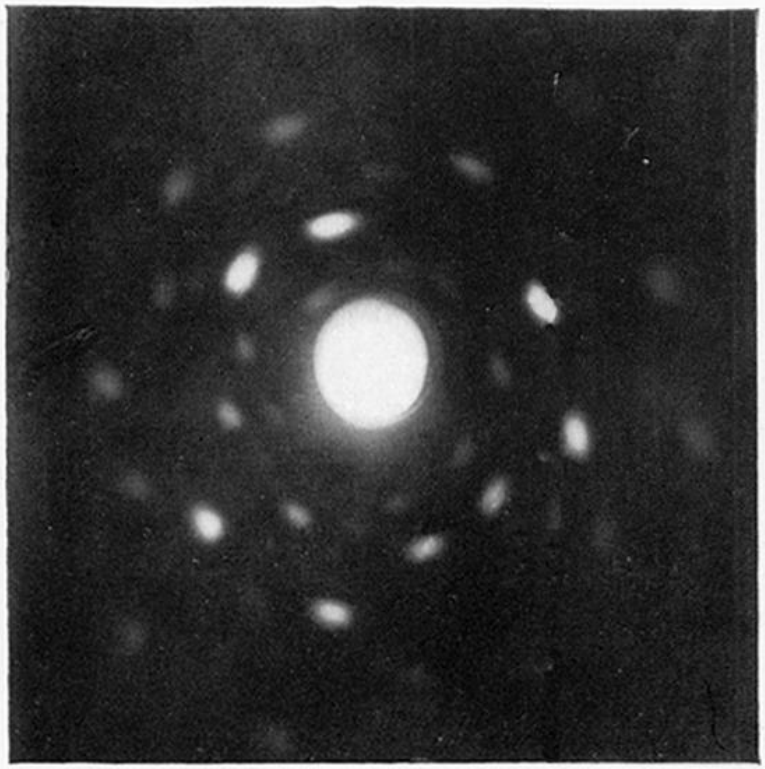


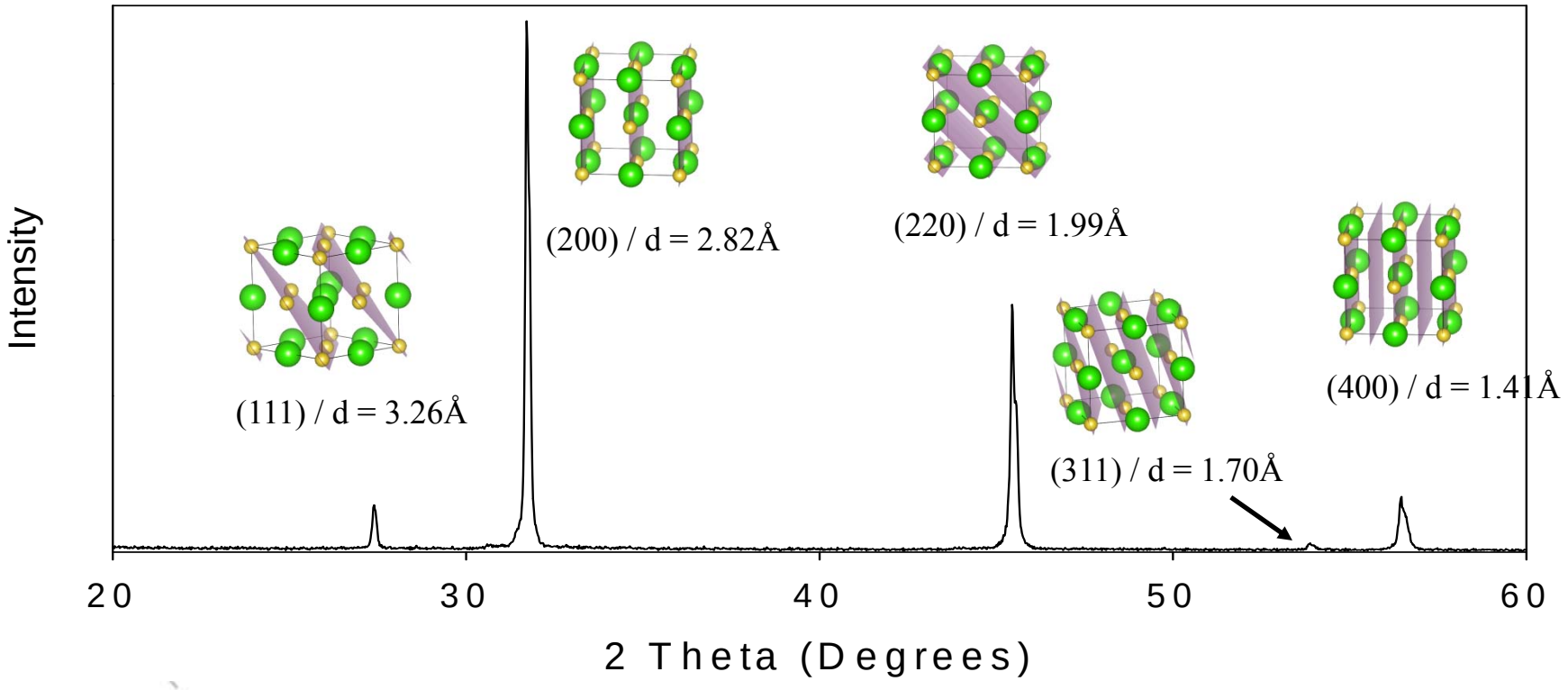
FIG. 15.—Rock-salt.

FIG. 8.—Rock-salt, 2·5 mm. thick.

W.L. Bragg, Roy. Soc. Proc. A, 89, (1913), 248

Powder/Single Xtal Diffraction

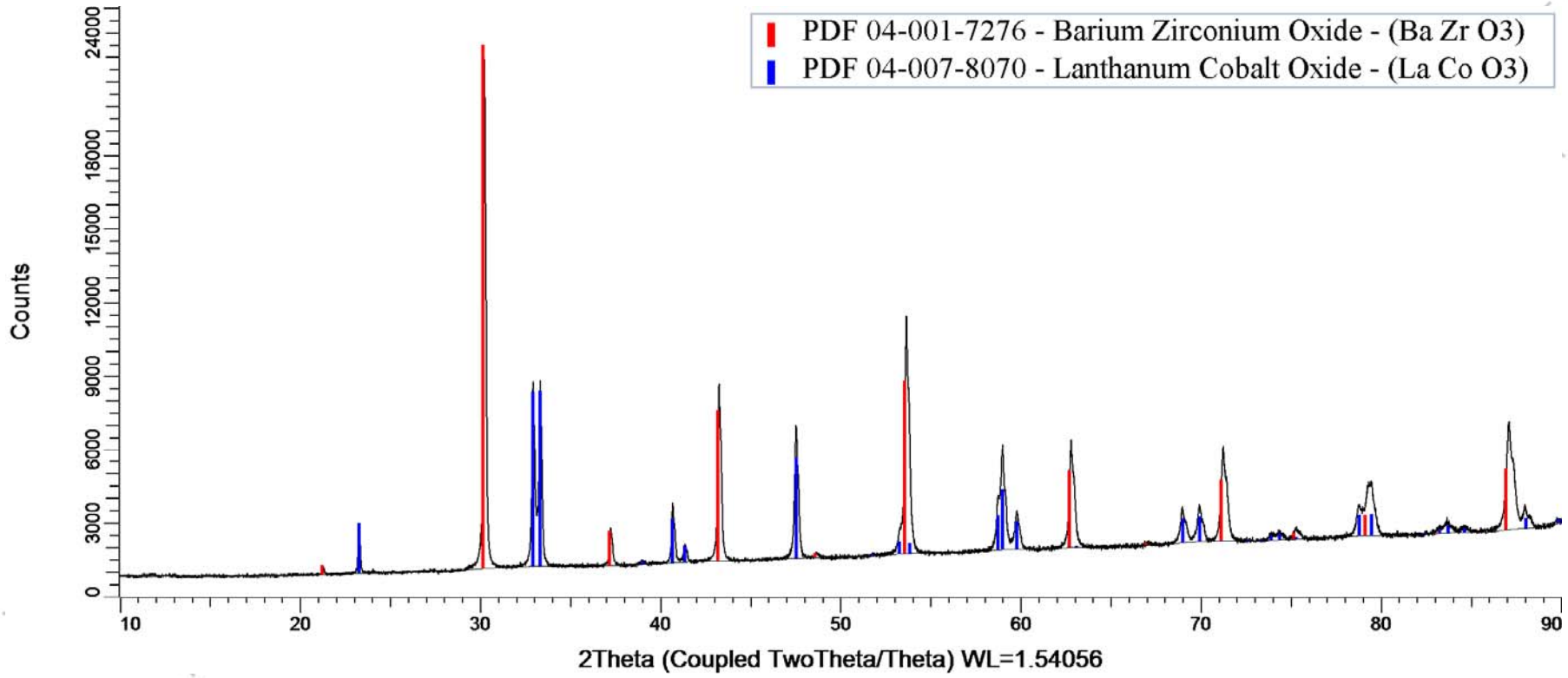
- Evaluation/determination of a crystal structure



W.L. Bragg, Roy. Soc. Proc. A, 89, (1913), 248

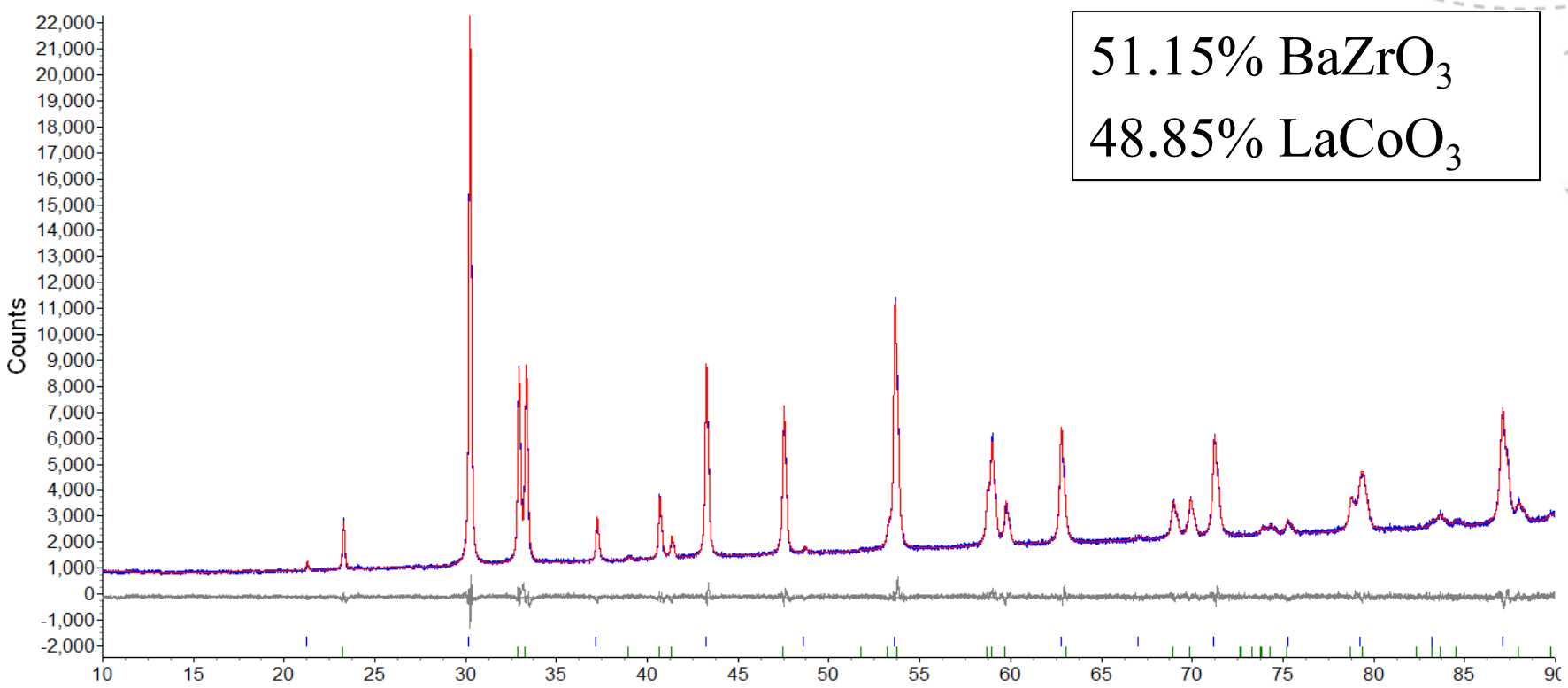
Multi-phase analysis (PXRD)

- "Fingerprinting"



Multi-phase analysis (PXRD)

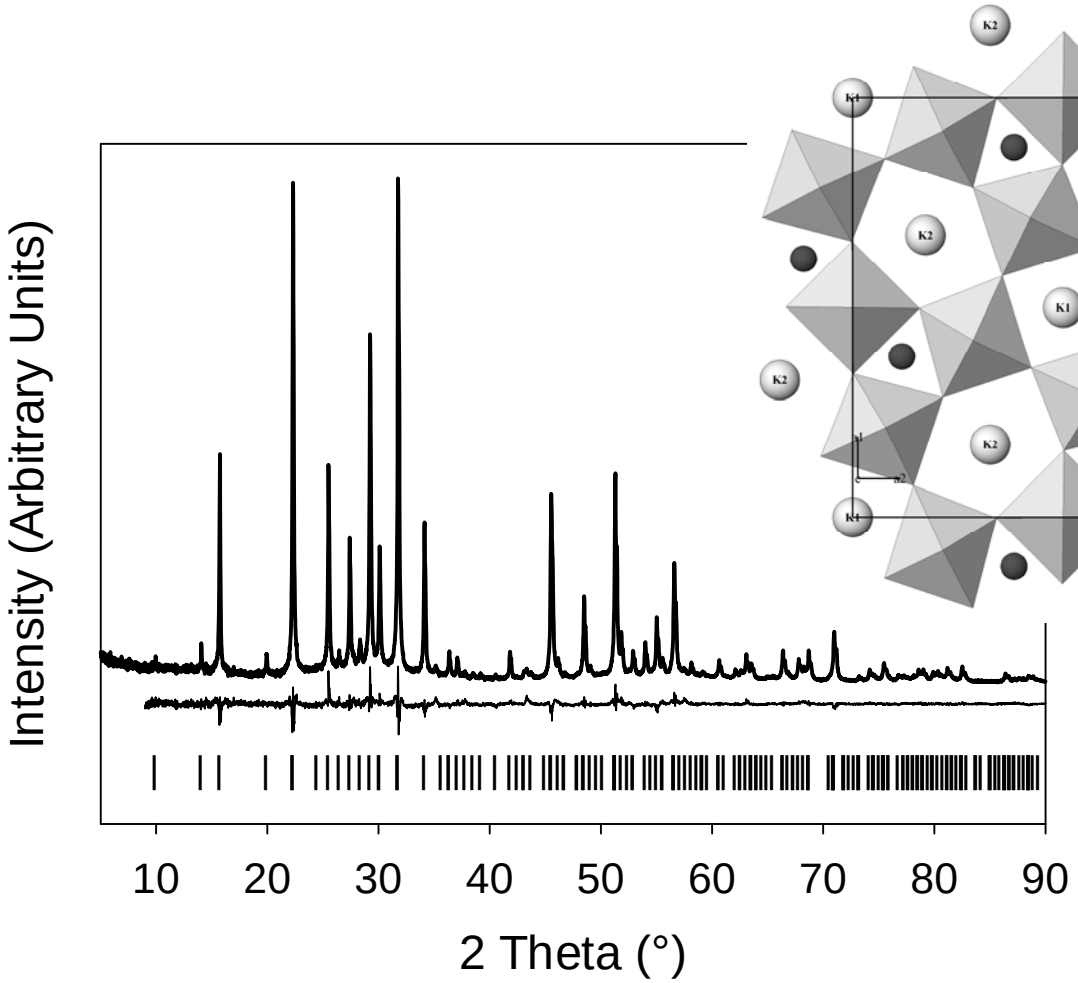
- Quantification

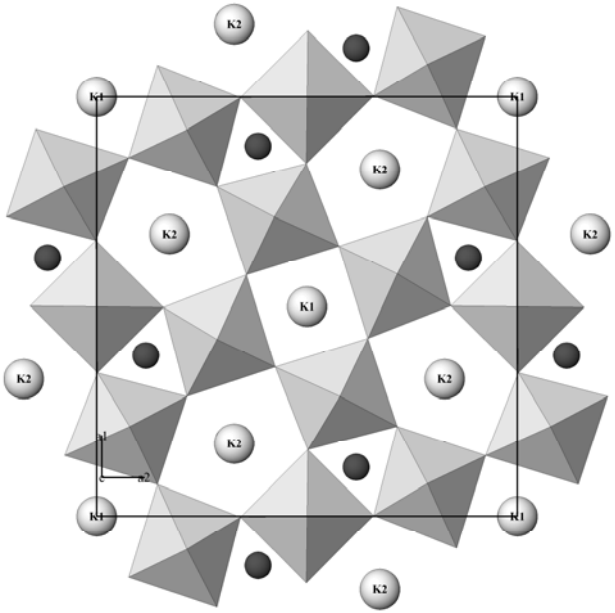


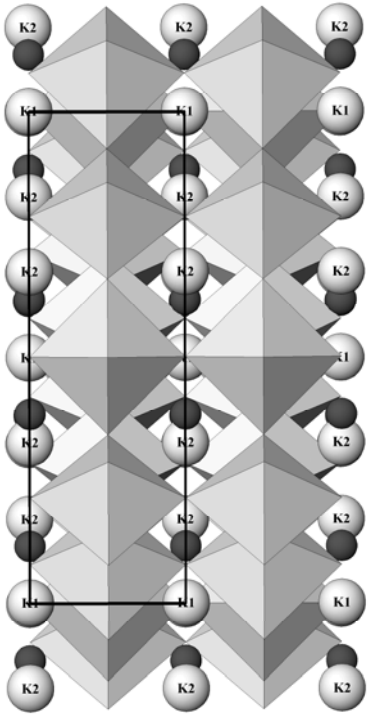
51.15% $BaZrO_3$

48.85% $LaCoO_3$

Structure analysis - $K_2Nb_4O_{11}$



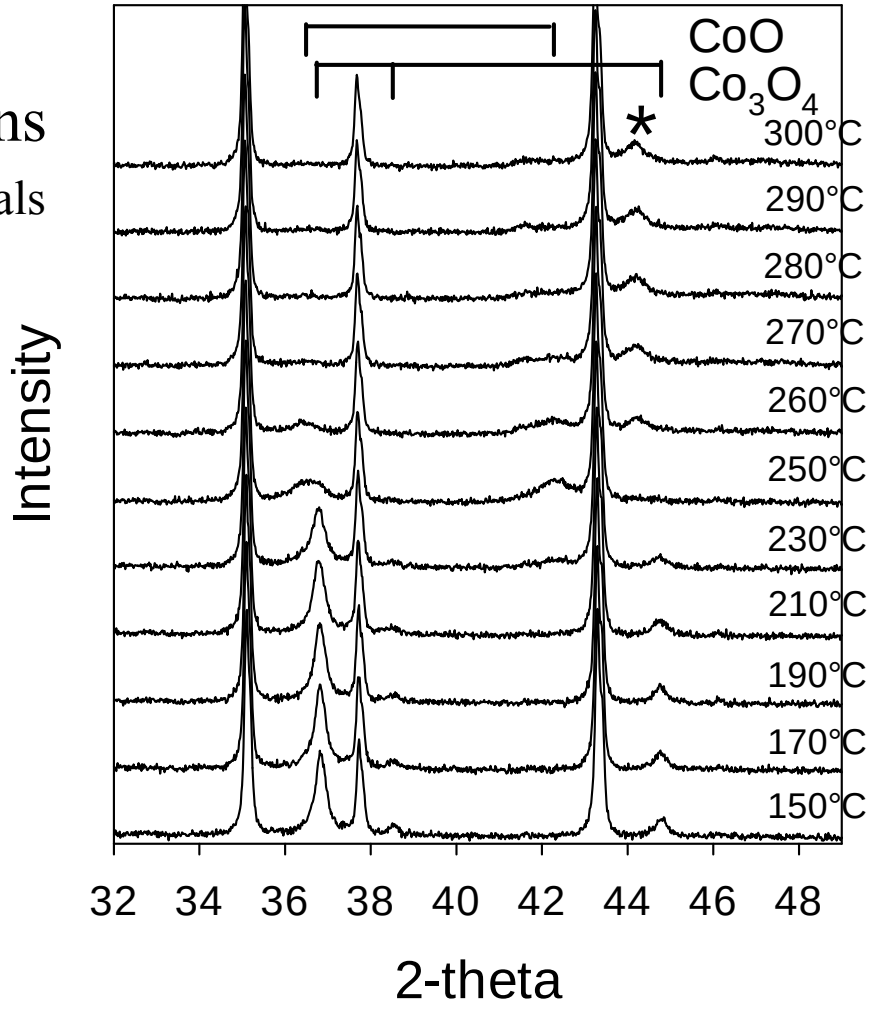




Madaro et. al. Cryst. Eng. Comm. 13, 1304 2011

In-situ measurements

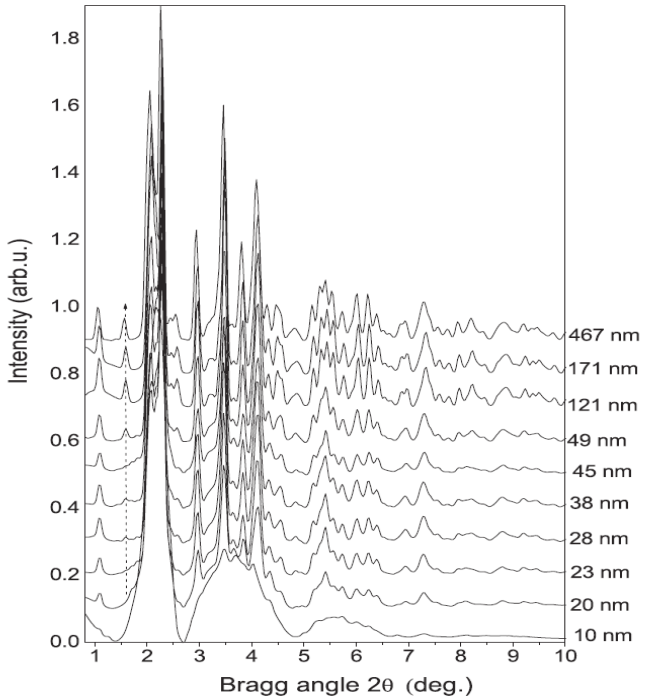
- Replicate application conditions
  - Temperatures, atmospheres, potentials
- Fischer-Tropsch catalyst
  - Co_3O_4/Al_2O_3
- Active catalyst forms in-situ
  - Nano Cobalt
- Assess effect of promoters etc
- Many applications
  - Phase transitions
  - Multiphase quantification
  - Reaction kinetics

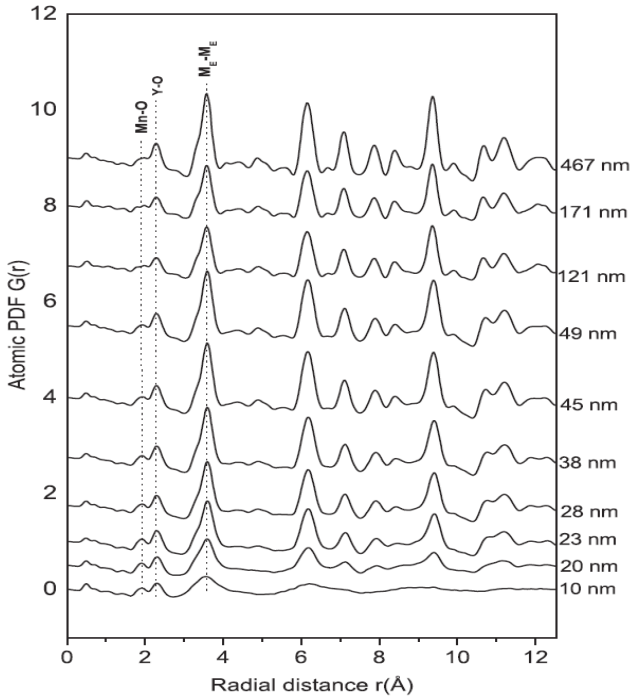


Roya Deghan-Niri, PhD Thesis, 2012

Pair Distribution Functions

- "Recalculation" of diffraction data in terms of local structure
  - Takes account of non-bragg scattering
  - Powerful nano analysis / local structure technique





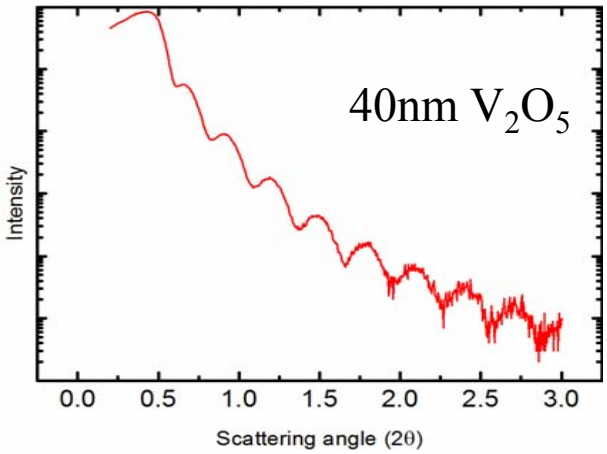
Tripathi et al., Phys. Rev. B 86, 2012, 094101

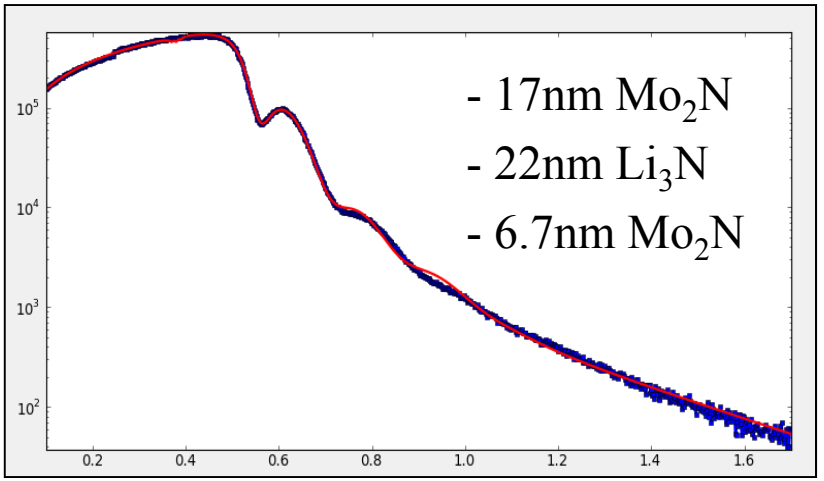
Thin Film/Texture analysis

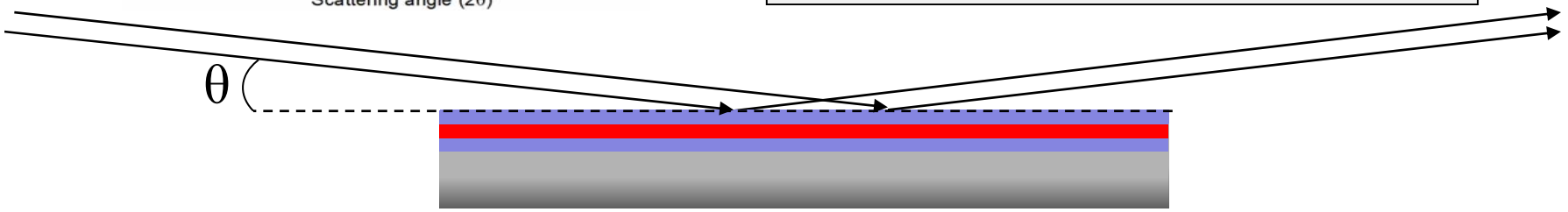
- Nanometer thick films/multilayers on substrates
  - Thickness/smoothness/orientation of single and multilayer films
- Dimensionally oriented samples (texture analysis)
  - Grain orientation in metals/ceramics etc
- Tailoring and enhancement of properties
  - Electrical, magnetic, mechanical
- Important industrial applications
  - Semiconductors, electronics, optics etc
  - Metallurgy and engineering

Thin Film Analysis

- Film thickness/smoothness (Reflectometry)





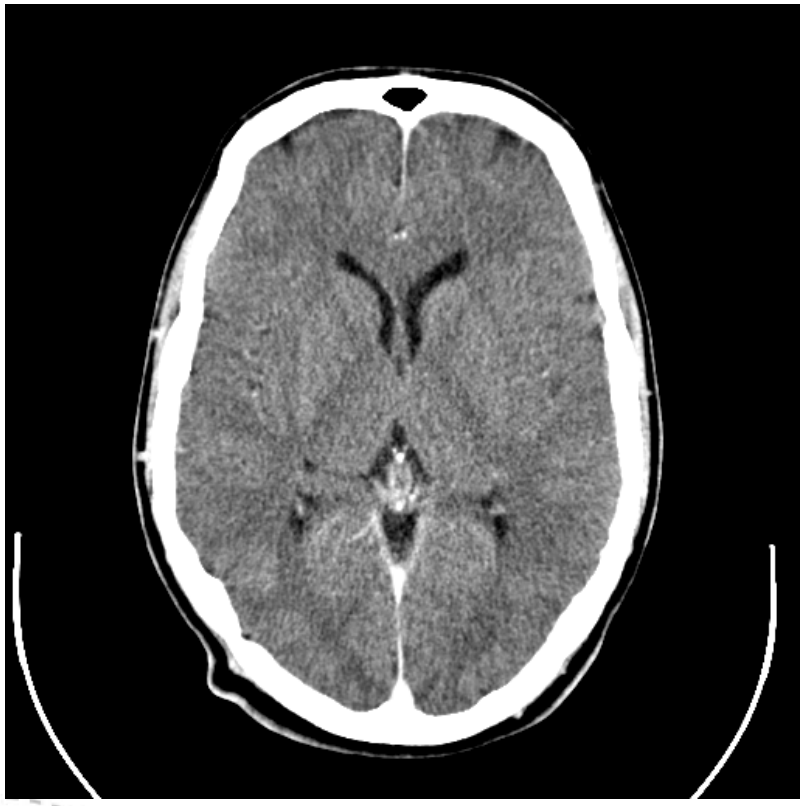


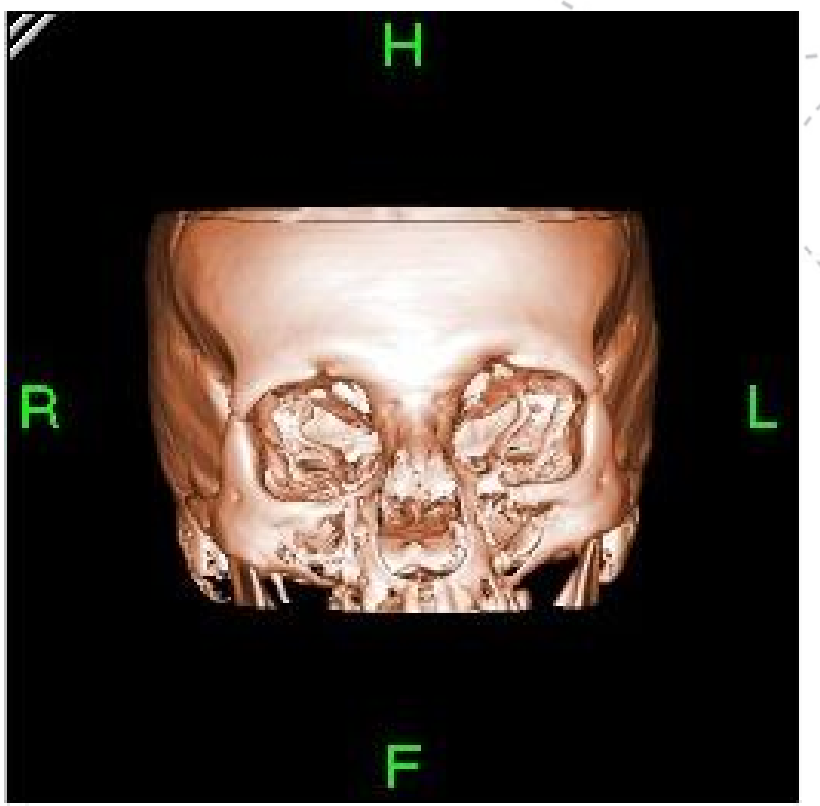
Bratvold et al. In preparation

SAXS

- Low angle x-ray scattering
  - Access to extremely long d-spacing values (10-100nm)
- Scattering related to longer length scales
  - Particle size, shape and distribution
  - Colloids, dispersions. Agglomeration
  - Porosity, pore spacing
- Look at solids, liquids, gels, sheets, fibres…
- Even thin films
  - Grazing incidence geometry
  - Nano-particles in a layer/matrix

Computed Tomography





Images from Wikimedia commons

Computed Tomography

- Three dimensional reconstruction of a object using x-ray attentuation
- Non-destructive
- Widely known medical uses
- Industrial and research applications also
  - Failure analysis, quality control
  - Mineral distributions in rock
  - Porosity / channel structures
  - Composites

Images from Wikimedia commons

So what does RECX offer?

- All of the above, and more…
- Data collection / experiments not possible in smaller labs
  - Higher resolution instruments, Tomography, Single Crystal XRD
  - In-situ / high temperature measurements
  - Large routine sample batches difficult for us due to manpower
- Focus on education and competence
  - Assistance with data collection, data analysis, problem solving
- Joint projects
- Networking and communication
- What do you want?

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Accessing RECX

- To start, no formal system
  - We don't really know what the demand will be
- Directly contact the support staff:
  - Julian Tolchard (julian.tolchard@material.ntnu.no)
  - David Wragg (david.wragg@smn.uio.no)
- We need to know what you want to do
  - We can arrange which site / instrumentation to use
  - Plan experiment with you
  - Organise who collects the data, does the analysis, give training if needed
- It is not free
  - Costs will be reasonable and negotiable

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Any Questions?