Neutron and X-ray Diffraction for Energy Materials

Ove Korjus, Emmanuelle Suard, Sreelakshmi Anil Kumar, S. F. Mayer
Institut Laue Langevin
korjus@ill.fr

Outline

- Introduction (scope)
- Information from powder diffraction
- Phase identification
- Le Bail and Rietveld method
  - Structure factor
  - Phase quantification
  - Microstructure analysis (size and strain)
- Neutrons vs X-rays
- Examples
  - *Operando* NPD
  - Bond-valence energy lanscapes
  - Synchrotron XRD depth profiling (ED)
  - XRD-CT

ILL

ESRF

Introduction (scope)

The lecture is designed a practical oriented guide for energy material researcher

The fundamentals are not covered:

- Generation of the flux
- Monochromatization
- Derivation of Bragg's law
- Crystallography

Also things not covered in this lecture:

- Single-crystal diffraction
- PDF (Pair Distribution Function)
- Magnetic structures

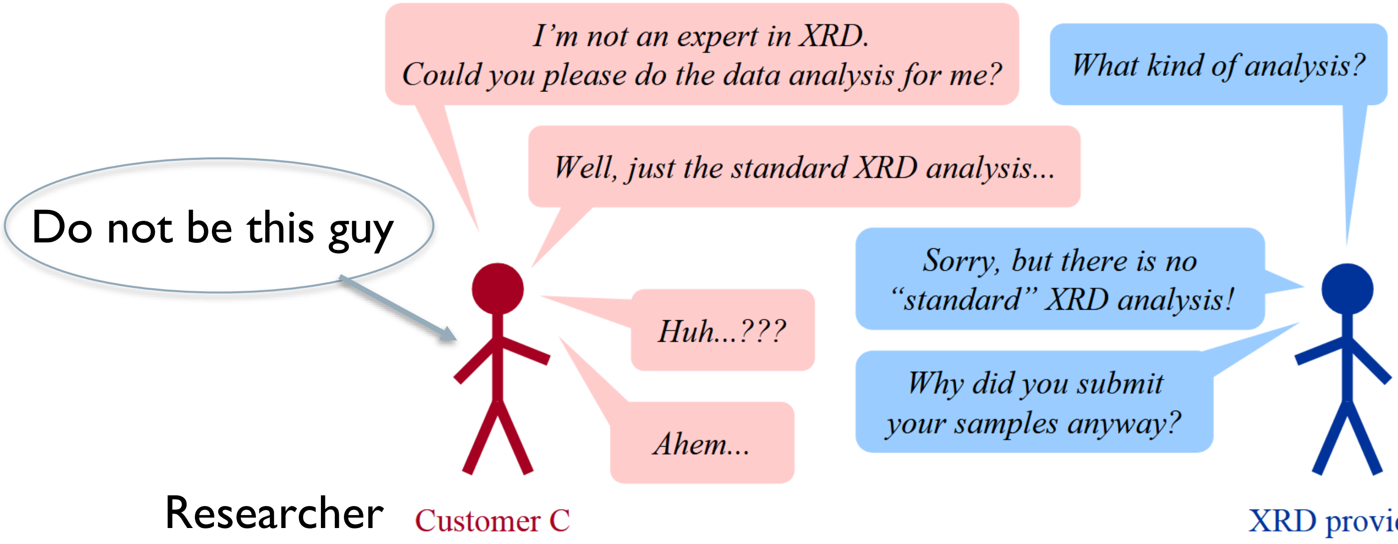
NEUTRONS FOR SOCIETY

Introduction (scope)

Scenario 4

Yet another day, customer C comes again to the XRD service provider:

NPD



Researcher
PhD student

Local contact

Frank Girgsdies, Preparation Group, Dept. AC, Fritz Haber Institute of the MPG, Berlin, Germany

NEUTRONS FOR SOCIETY

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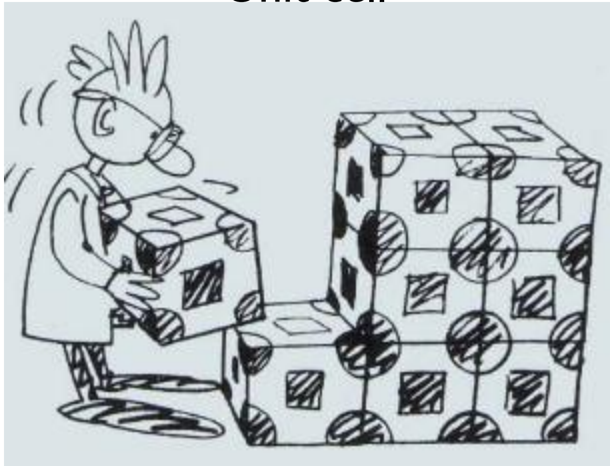
Unit cell

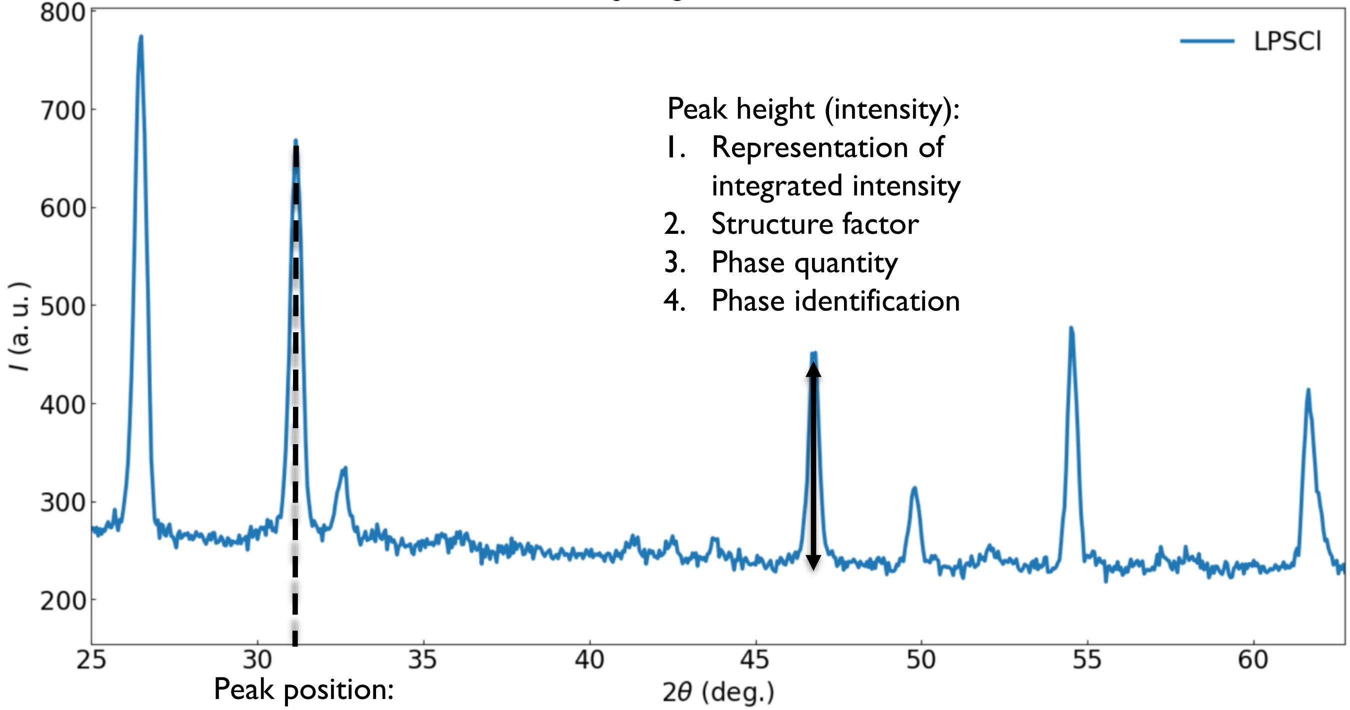


Neutron Scattering - a Primer,´ Los Alamos Sci., vol. 19, p. 33, 1990.

NEUTRONS FOR SOCIETY

Useful information from powder diffraction

Li_6PS_5Cl (LPSCl)



Peak height (intensity):

1. Representation of integrated intensity
2. Structure factor
3. Phase quantity
4. Phase identification

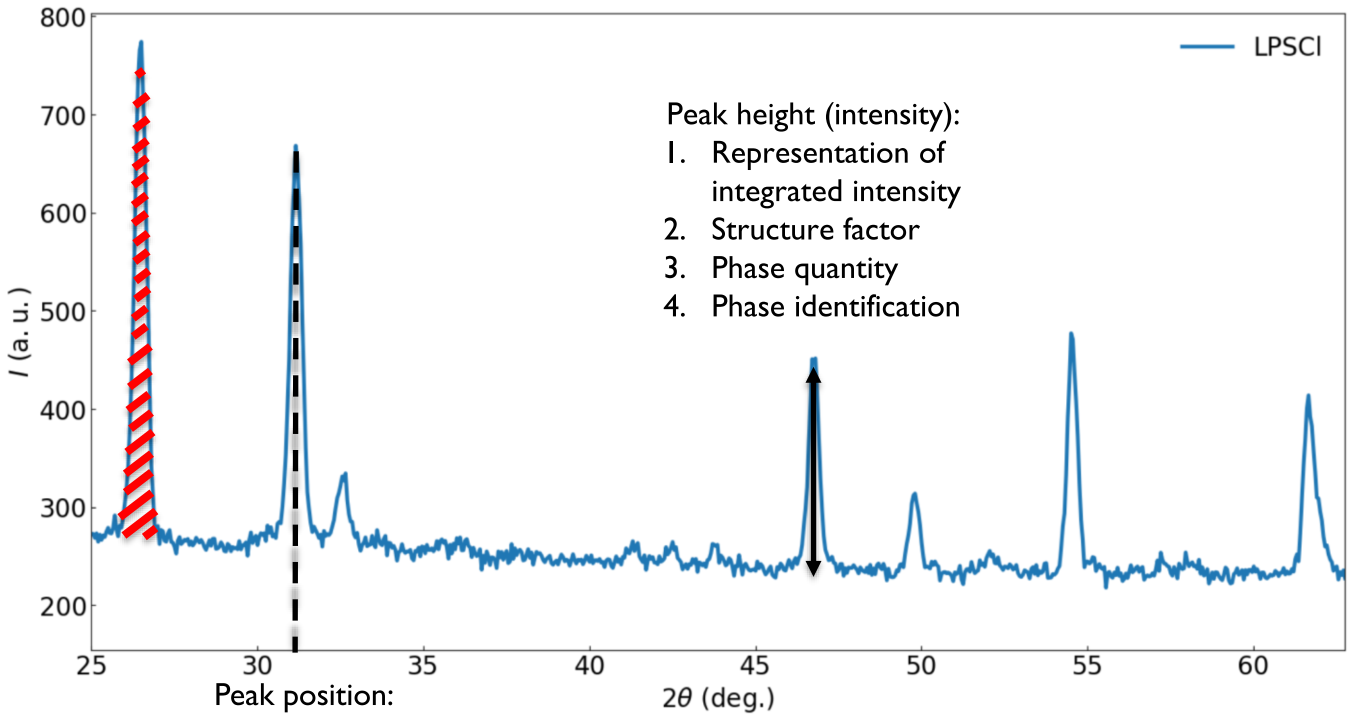
Peak position:

1. D-spacing
    1. Lattice parameters
2. Phase identification

NEUTRONS FOR SOCIETY

Useful information from powder diffraction

Peak area:
1. Structure factor
    1. Atomic occ.
    2. Atomic pos.
2. Phase quantity
3. Thermal parameters



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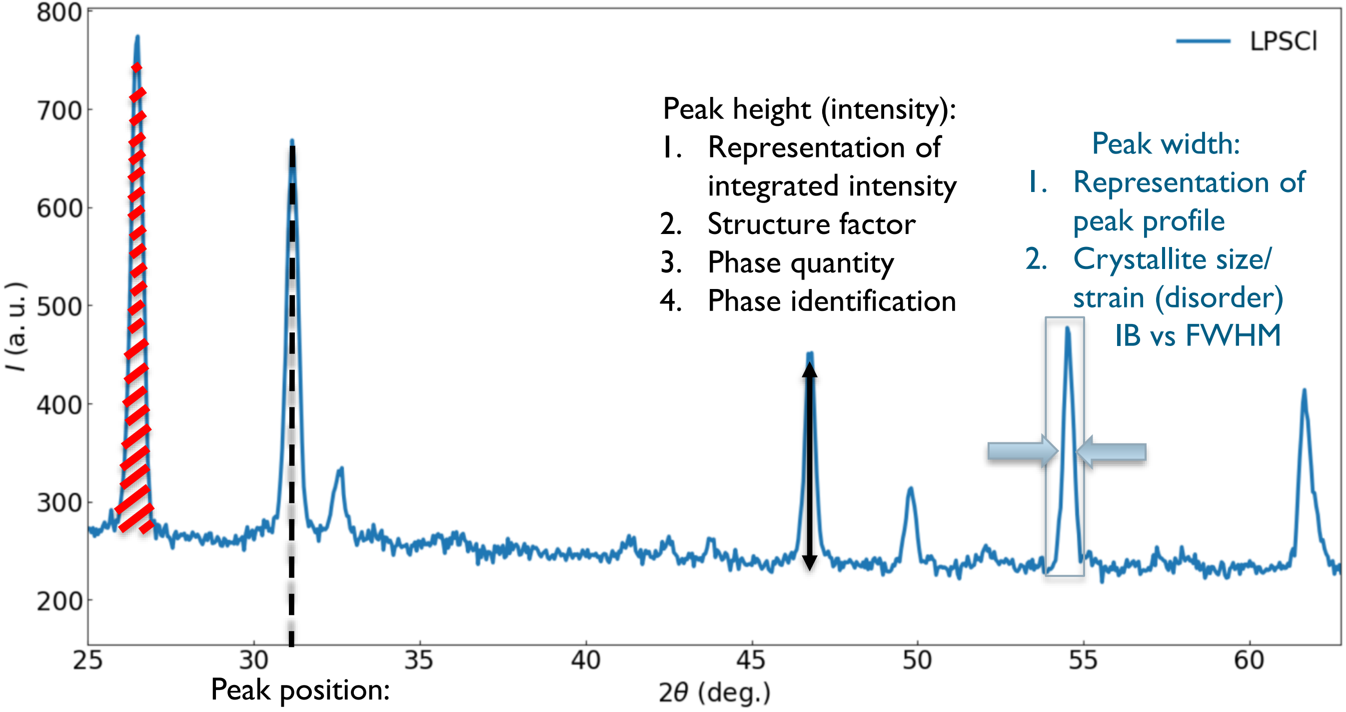


Peak position:

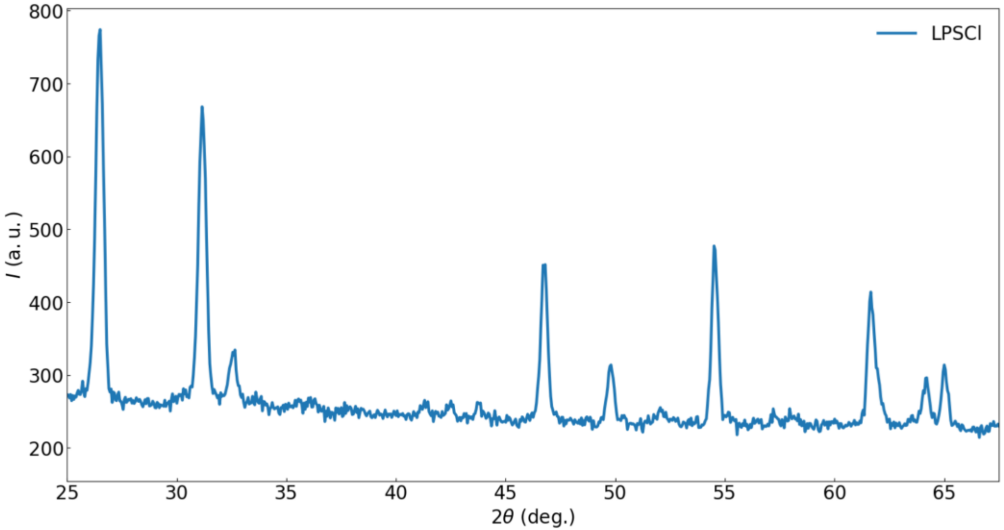
1. D-spacing
    1. Lattice parameters
2. Phase identification

NEUTRONS FOR SOCIETY

Phase identification

Important parameters:

- Peak positions
  - Correct single wavelength
- Peak intensity
  (or integrated intensity)



Databases:

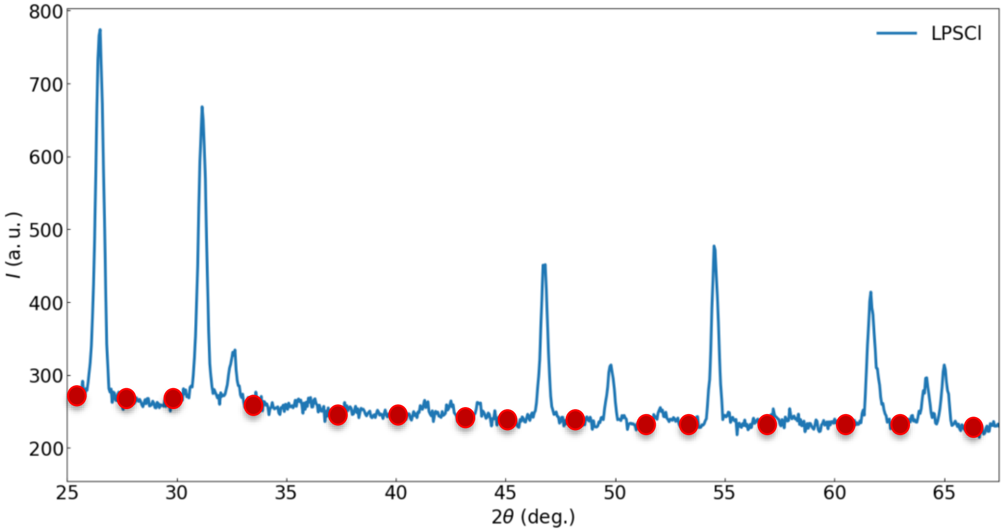
- COD (Crystallographic Open Database)
- PDF (Powder diffraction file)
- ICSD (Inorganic Crystal Structure Database)

NEUTRONS FOR SOCIETY

Phase identification

Important parameters:

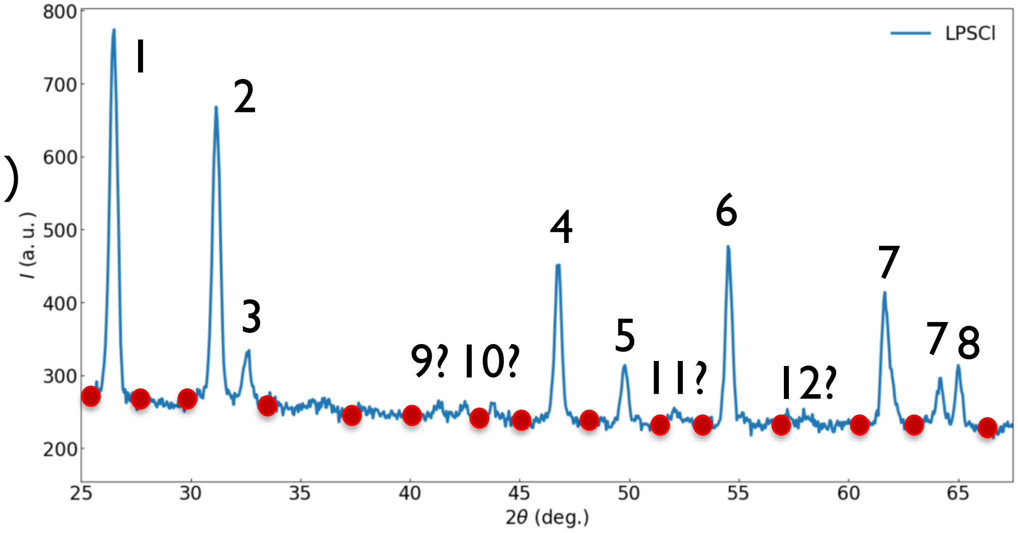
- Background



Phase identification

Important parameters:

- Background
- Label peaks (if monochr, beam)

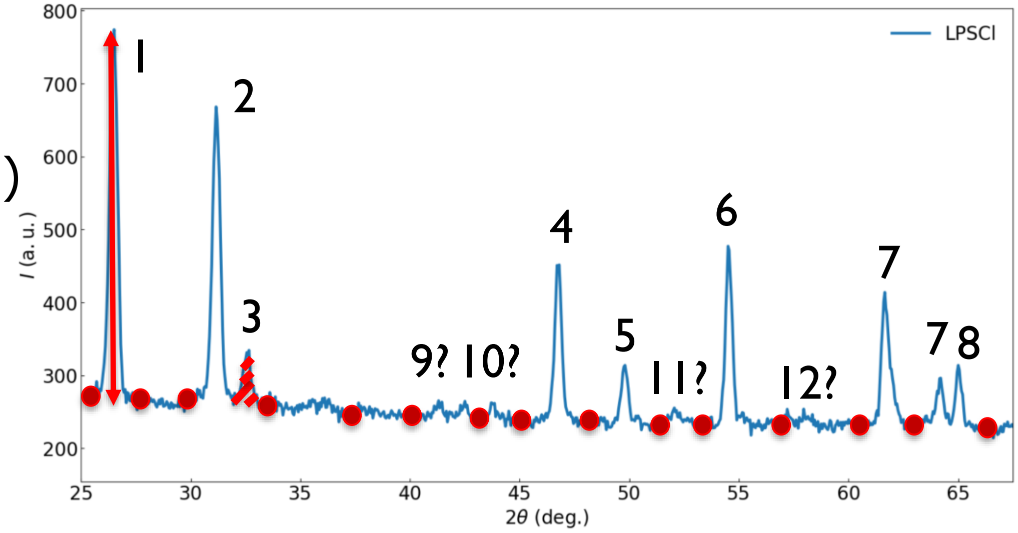


NEUTRONS FOR SOCIETY

Phase identification

Important parameters:

- Background
- Label peaks (if monochr, beam)
- Get intensities for all peaks (or areas) by fitting
- Match the profile with the database using some software

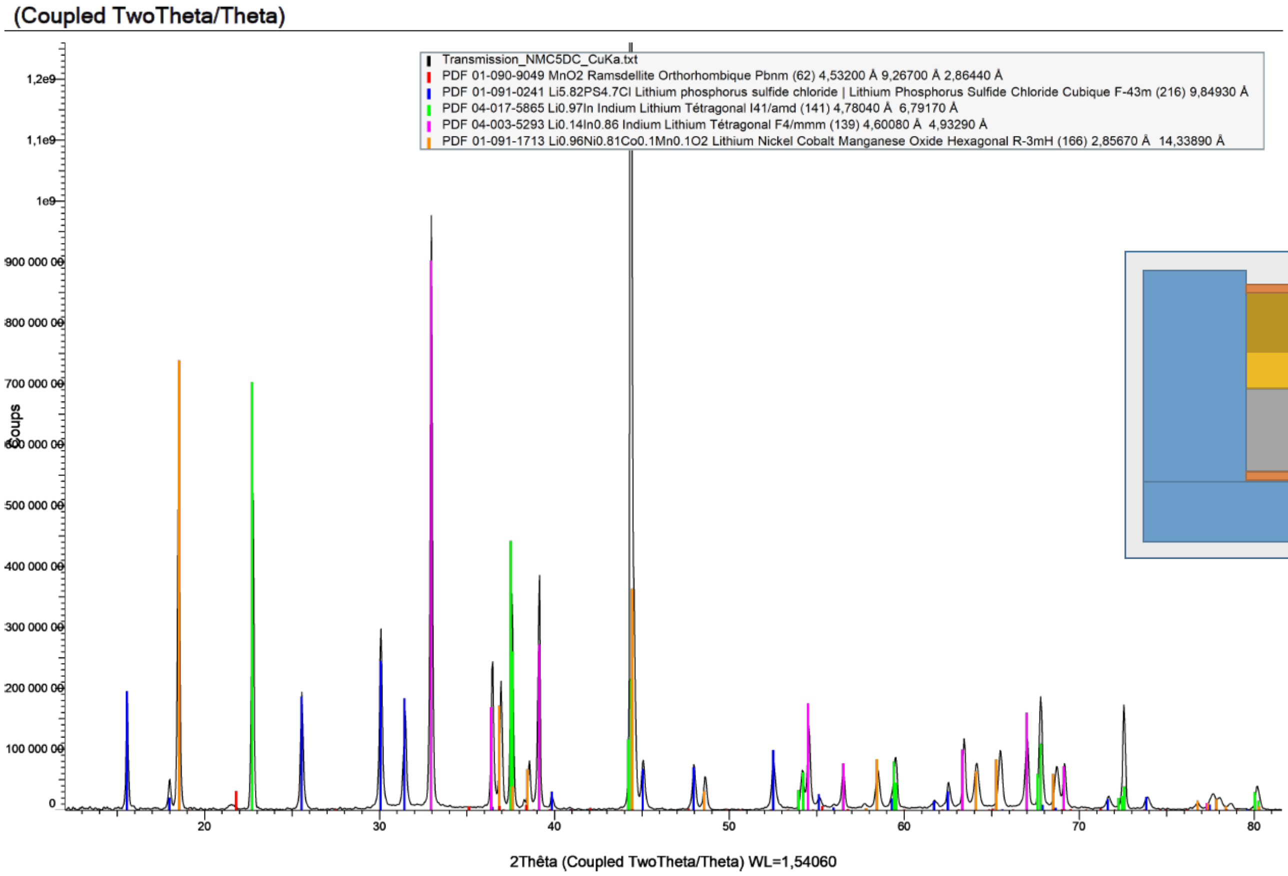


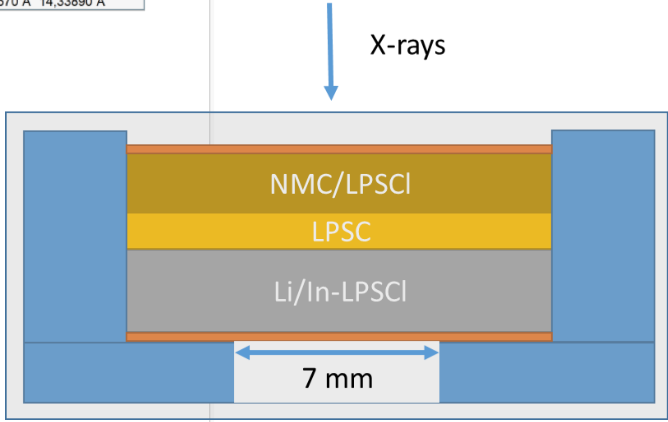
Different software:

- Match!
- Diffrac EVA

NEUTRONS FOR SOCIETY

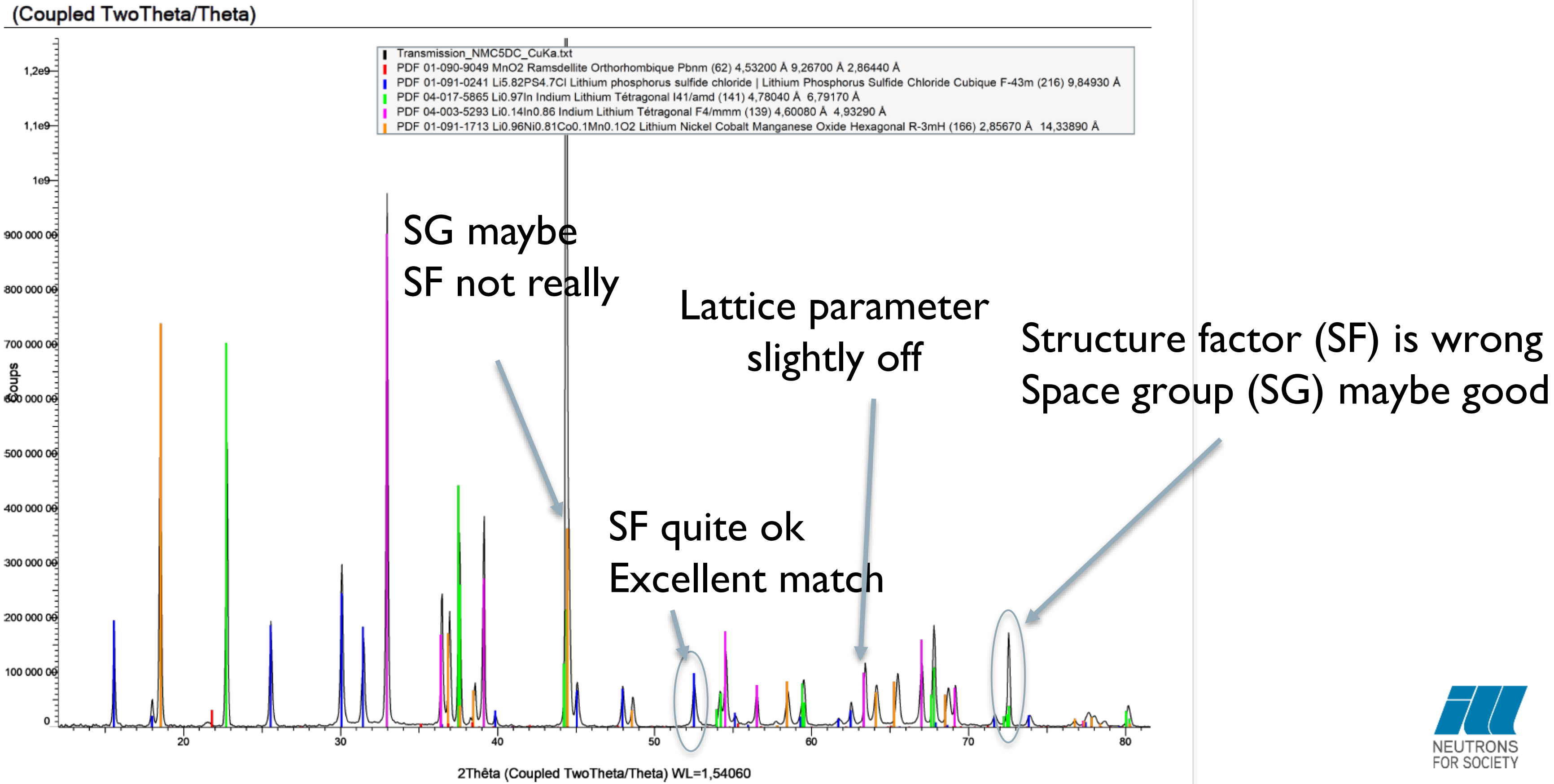
Phase identification





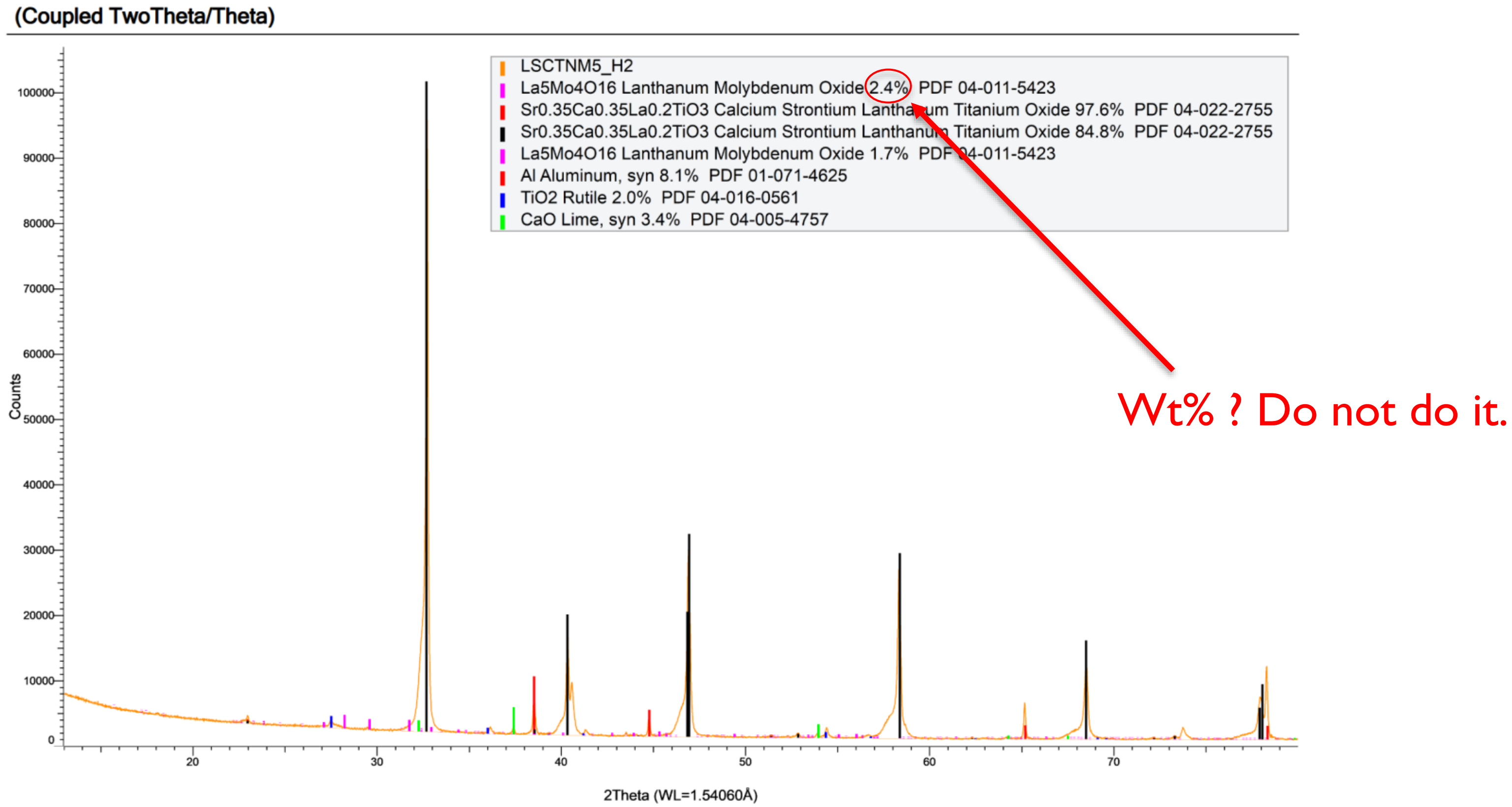
NEUTRONS FOR SOCIETY

Phase identification



NEUTRONS FOR SOCIETY

Phase identification



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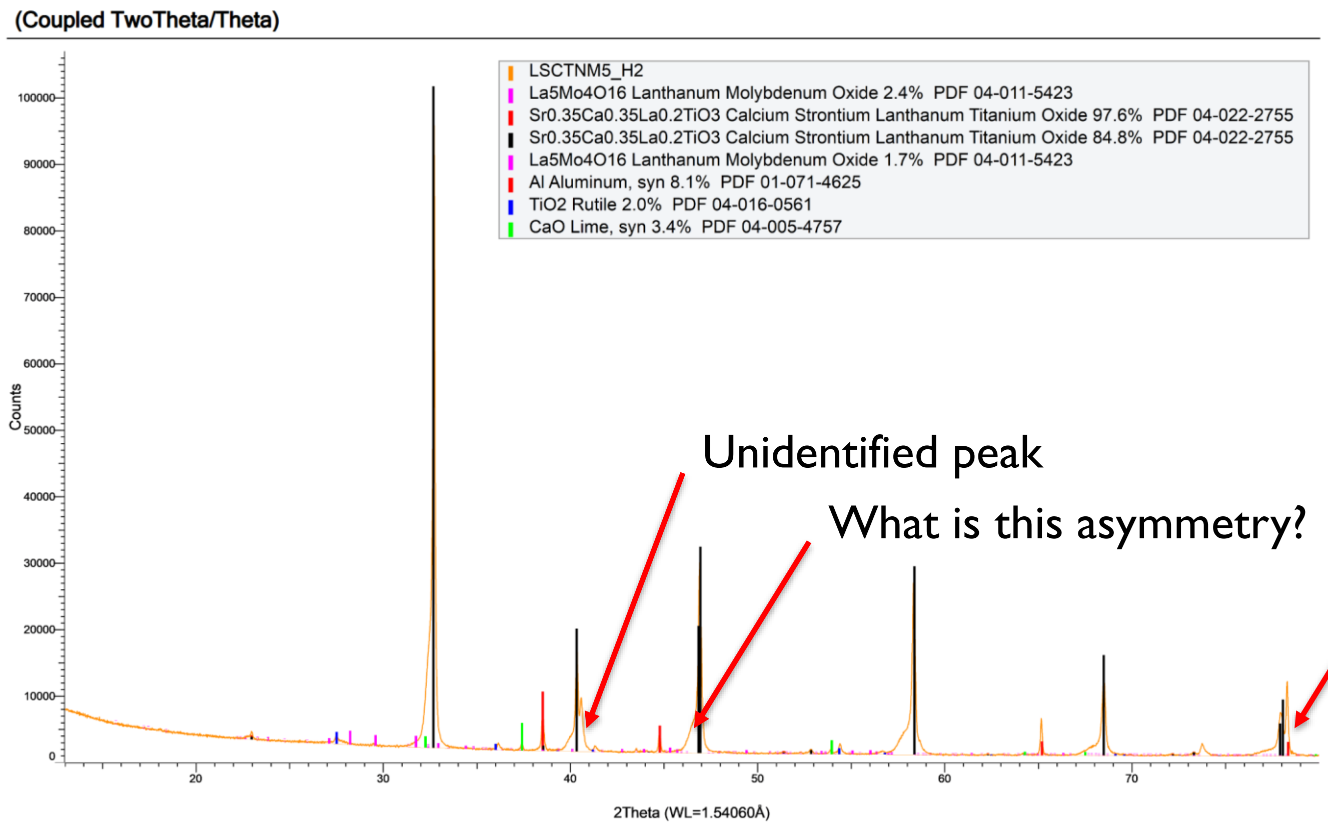
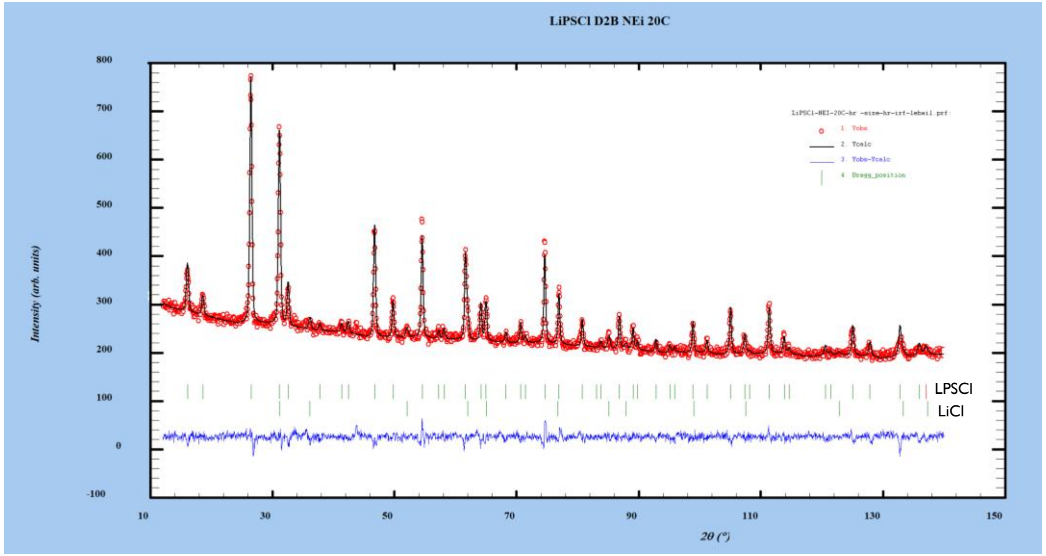


NEUTRONS FOR SOCIETY

Le Bail refinement

Developed by Armel Le Bail around 1988



Starting parameters:

- unit cell parameters (r)
- space group
- Background
- peak profile parameters (r)
  - Instrumental (r)
  - Peak shape (r)
- This method **does not** extract infromation from **intensity**:
  - Structure (occupancies, atomic positions)
  - wt%
  - Thermal parameters

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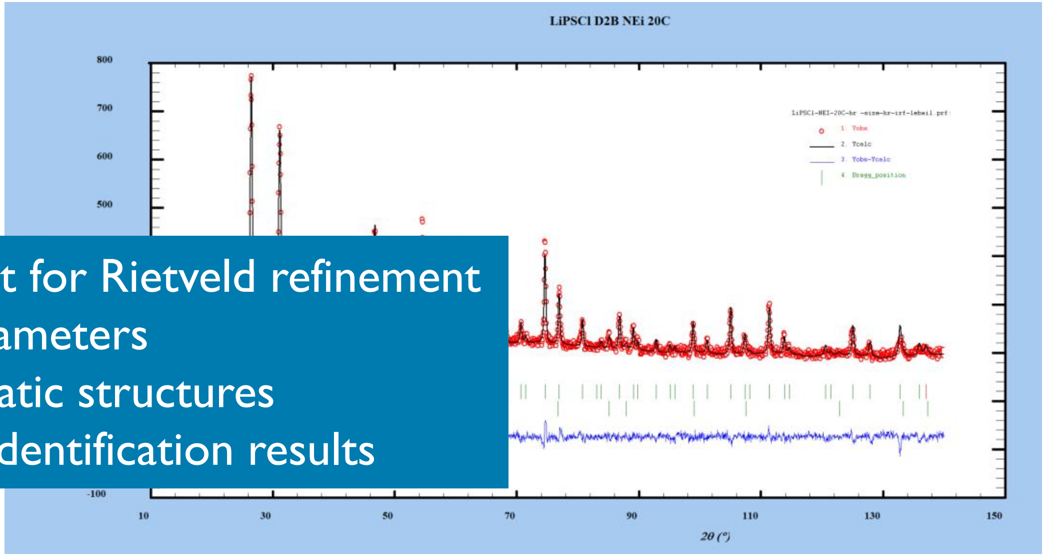

Good starting point for Rietveld refinement
Refining lattice parameters
Unknown/problematic structures
Confirming phase identification results

NEUTRONS FOR SOCIETY

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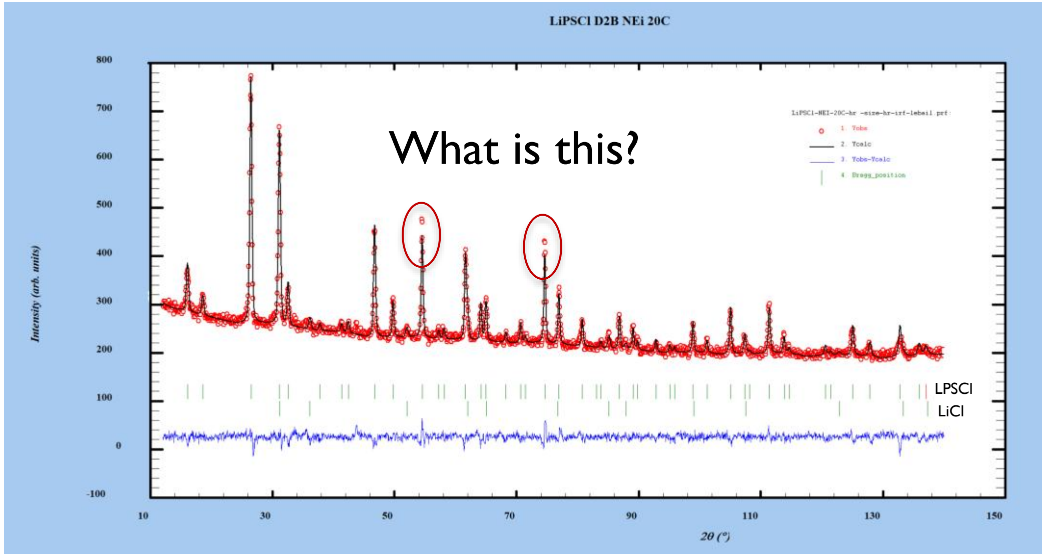

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NEUTRONS FOR SOCIETY

Rietveld refinement

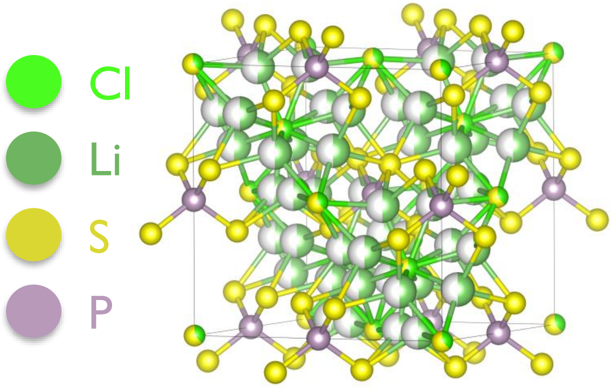
Developed by Hugo Rietveld around 1967

- Background
- Space group
- lattice parameters
- peak profile:
  - microstructure (strain, crystallite size)
  - Instrumental

Like Le Bail

- magnetic structure (for neutron diffraction)
- weight precentages of phases
- Structure factor (database):
  - atomic coordinates
  - occupancies
  - thermal parameter(s) (atomic displacement)

Integrated intensity



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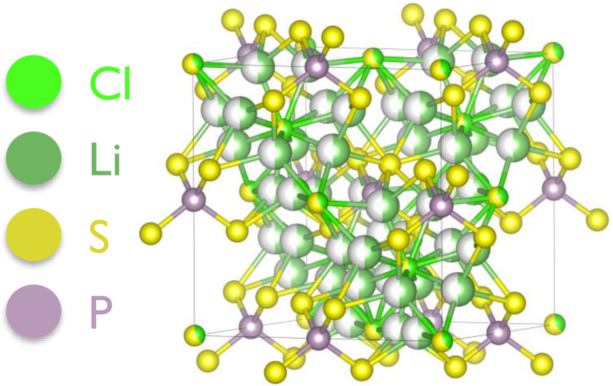
  - occupancies
  - thermal parameter(s) (atomic displacement)

Integrated intensity

```
F -4 3 M                <--Space group symbol
!Atom   Typ       X        Y        Z       Biso       Occ      In Fin N_t Spc /Codes
P1      P      0.00000  0.00000  0.50000  2.80412   0.04166   0   0   0    0
                  0.00     0.00     0.00   111.00      0.00
Li1     Li     0.31732  0.02912  0.68267  2.41177   0.25000   0   0   0    0
                 81.00    91.00   -81.00   101.00      0.00
S1      S      0.25000  0.25000  0.75000  1.51958   0.02021   0   0   0    0
                  0.00     0.00     0.00   121.00    131.00
Cl1     Cl     0.25000  0.25000  0.75000  1.51958   0.02145   0   0   0    0
                  0.00     0.00     0.00   121.00   -131.00
S2      S      0.12000 -0.12000  0.62000  2.29082   0.16667   0   0   0    0
                  0.00     0.00     0.00    31.00      0.00
S3      S      0.00000  0.00000  1.00000  1.00000   0.02145   0   0   0    0
                  0.00     0.00     0.00     0.00   -131.00
Cl3     Cl     0.00000  0.00000  1.00000  1.00000   0.02021   0   0   0    0
                  0.00     0.00     0.00     0.00    131.00
```



NEUTRONS FOR SOCIETY

Calculated intensity of one phase

$$y_i^{calc.}(2\theta_i) = s \sum_k L_k |F_k|^2 P_k A_k \phi(2\theta_i - 2\theta_k) + y_i^{background}$$

Sum over all
Bragg peaks

NEUTRONS FOR SOCIETY

Calculated intensity of one phase

Structure factor:

- Atomic positions
- Atomic occ.
- Thermal param.
- Atomic scattering factors

Scale factor:

- Amount of phase
- Flux/sensitivity

$$y_i^{calc.}(2\theta_i) = \boxed{s} \sum_k L_k \boxed{|F_k|^2} P_k A_k \boxed{\phi(2\theta_i - 2\theta_k)} + y_i^{background}$$

Peak profile function:

- Instrumental
- microstructure

Calculated intensity of one phase

Structure factor:

- Atomic positions
- Atomic occ.
- Thermal param.
- Atomic scattering factors

Scale factor:

- Amount of phase
- Flux/sensitivity

Preferred orientation
(alignment in certain direction)

Absorption

$$y_i^{calc.}(2\theta_i) = s \sum_k L_k |F_k|^2 P_k A_k \phi(2\theta_i - 2\theta_k) + y_i^{background}$$

Lorentz-polaization factor:
monochromator
polarization (0 for neutrons)

Peak profile function:

- Instrumental
- microstructure

Amorphous part
Air scattering
Instr. noise
Incoherent scat.

NEUTRONS FOR SOCIETY

Phase fraction in Fullprof

$$W_\phi = \frac{S_\phi (ZMV)_\phi . f_\phi^2 / \tau_\phi}{\sum_{i=1}^{N_\phi} S_i . (ZMV)_i . f_i^2 / \tau_i} = \frac{S_\phi ATZ_\phi . V_\phi}{\sum_{i=1}^{N_\phi} S_i ATZ_i . V_i}$$

W = weight fraction of the phase
S = scale factor
Z = number of formula units of phase
M = molecular weight of phase
V = unit cell volume of a phase
t = Brindley coefficient (Micro-absorp.)
f = value to transform the site multipl. To its real value

$$ATZ_i = Z_i M_i f_i^2 / \tau_i$$

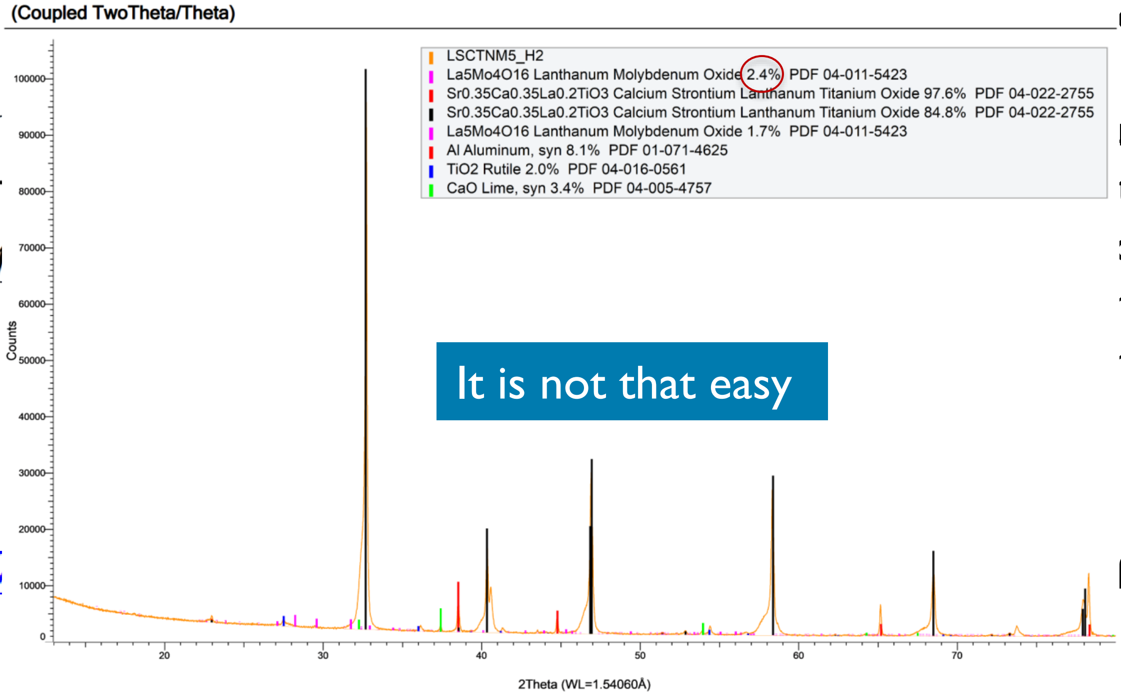
ATZ calculated automatically (if you put 0)

Only crystalline phases!

Phase fraction in Fullprof

$$W_\phi = \frac{S_\phi(ZMV}{\sum_{i=1}^{N_\phi} S_i.(ZM}$$

ATZ



It is not that easy

of the phase

ıla units of phase

t of phase

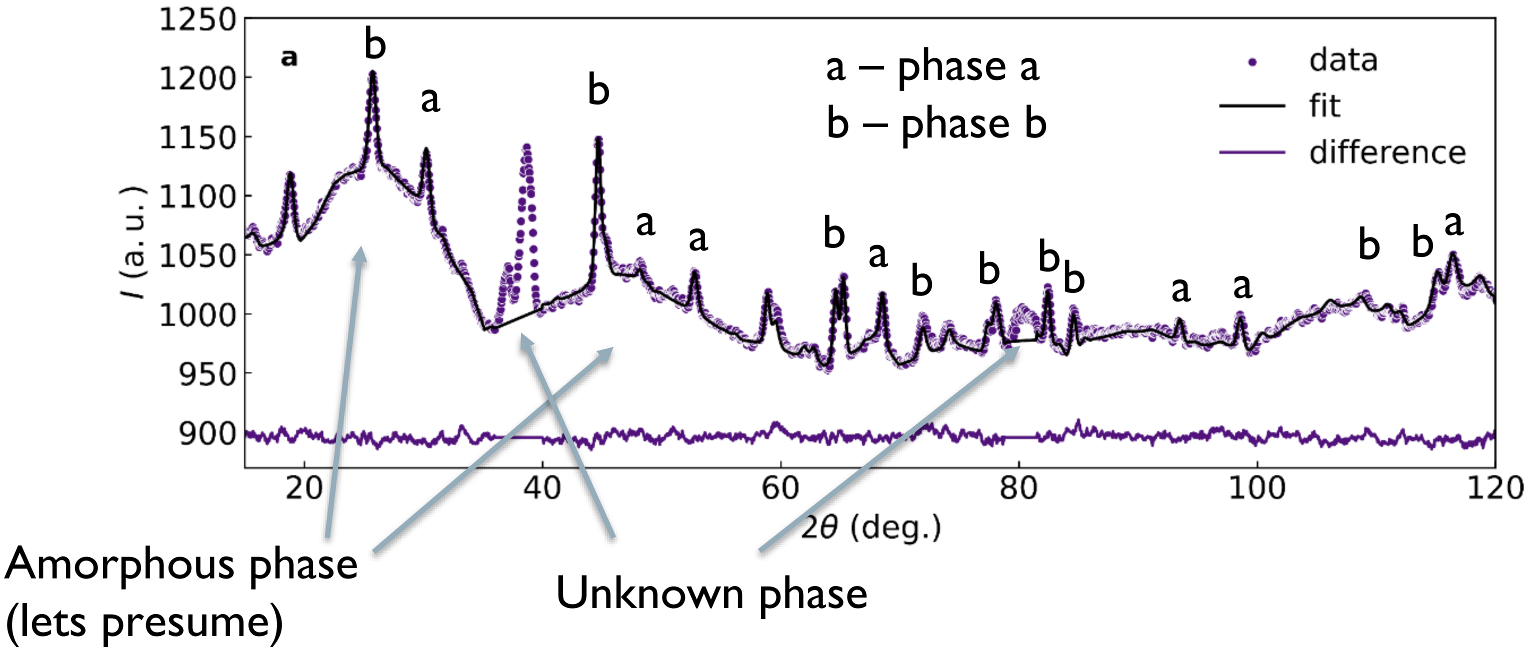
of a phase

nt (Micro-absorp.)

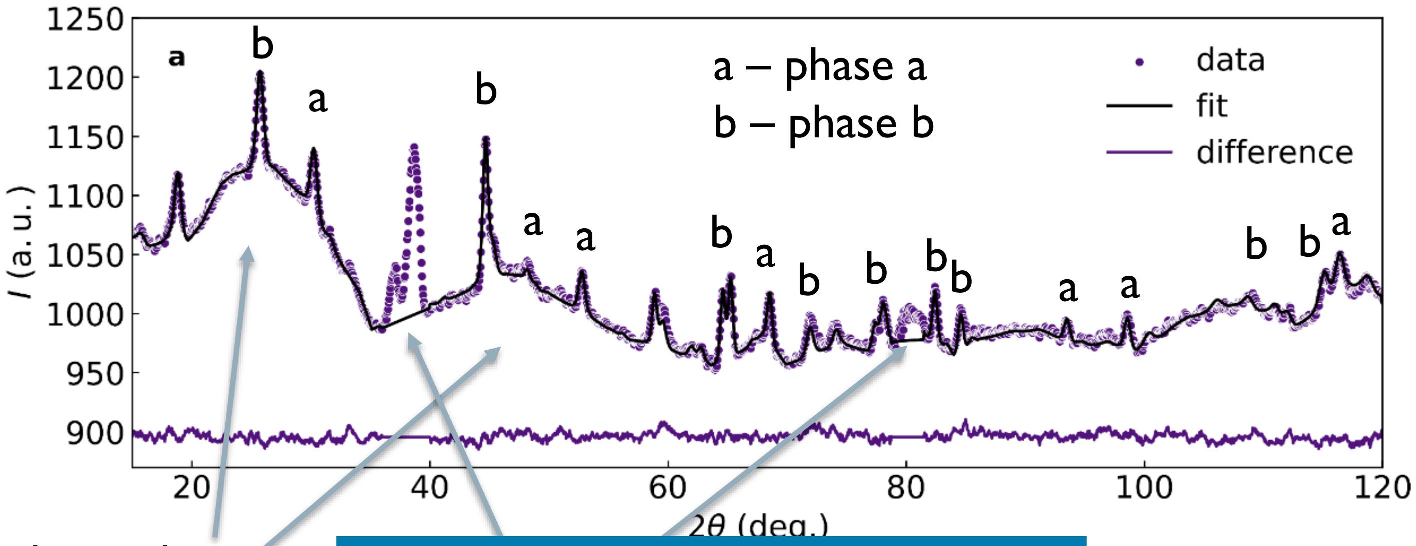
n the site multipl.

f you put 0)

Quantitative analysis



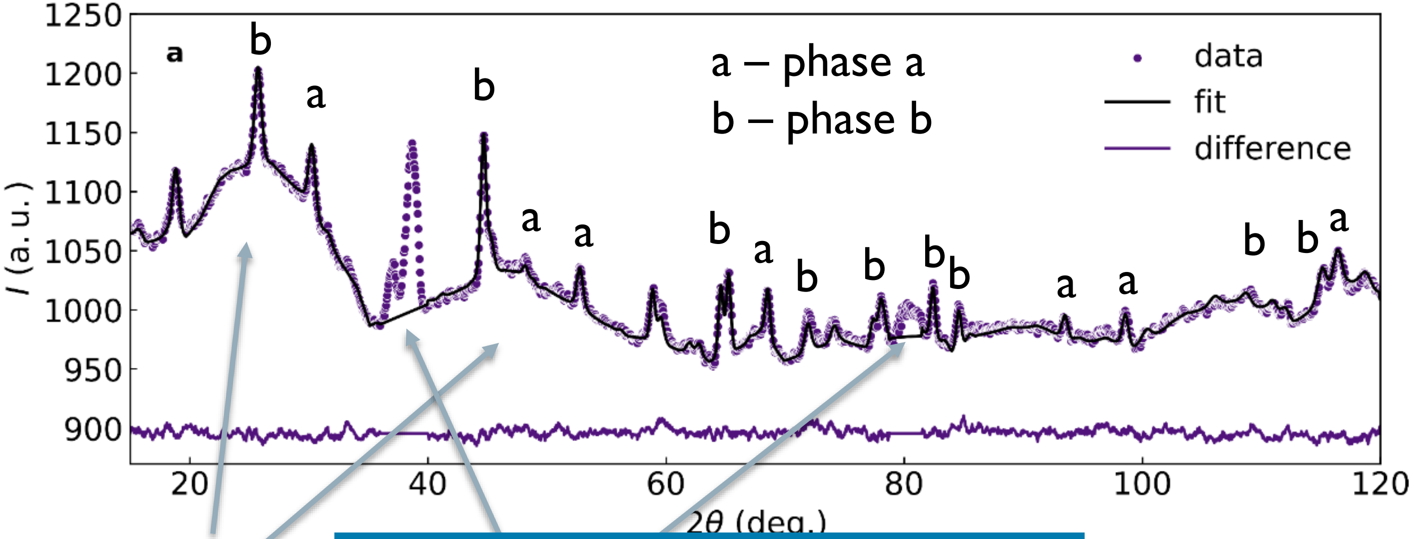
Quantitative analysis excercise



Amorphous phase
(lets presume)

Can we get absolute phase % of a/b?

Quantitative analysis excercise

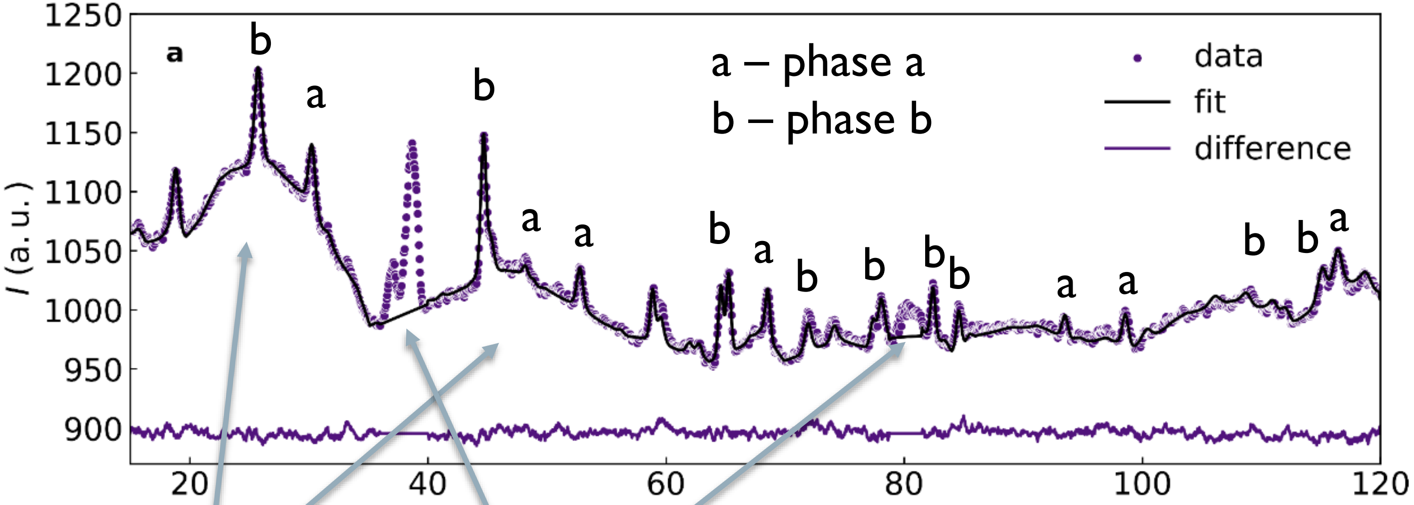


Amorphous phase
(lets presume)

Can we get absolute phase % of a/b?
Can we get their relative phase %?

NEUTRONS FOR SOCIETY

Quantitative analysis excercise

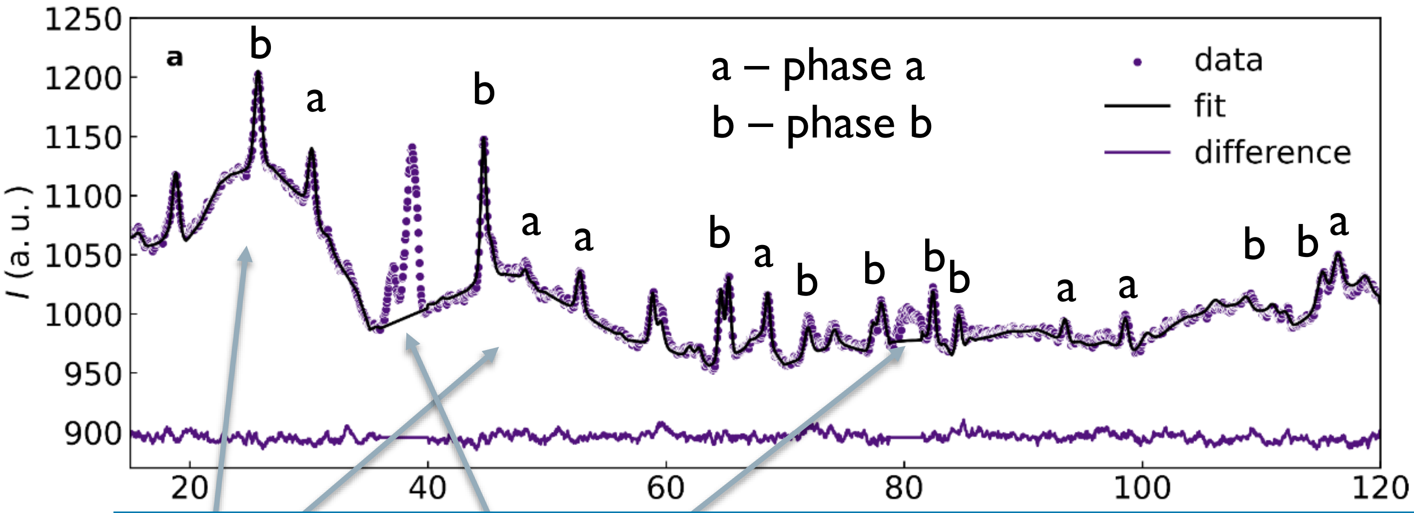


Amorphous phas
(lets presume)

Can we get absolute phase % of a/b?
Can we get their relative phase %?
Is there a way to get absolute phase wt% of a/b?
Is there any way to give wt% to amorphous phase?

NEUTRONS FOR SOCIETY

Quantitative analysis excercise



Amorphous

(lets presun

Can we get absolute phase % of a/b? No

Can we get their relative phase %? Yes

Is there a way to get absolute phase wt% of a/b? Internal standard

Is there any way to give wt% to amorphous phase? No

NEUTRONS FOR SOCIETY

Crystallite size/strain analysis

- Peak broadening:
  - Instrumental broadening
  - Size/strain

Instrumental broadening needs to be taken into account when doing size/strain analysis

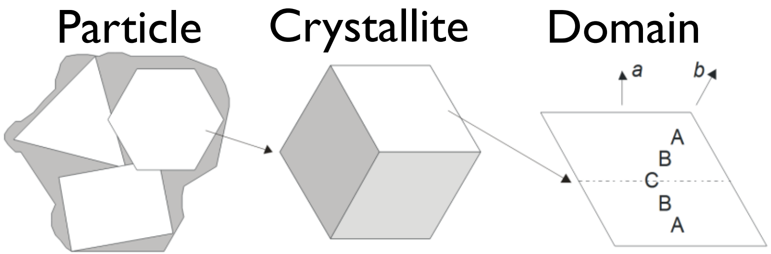
NEUTRONS FOR SOCIETY

Crystallite size/strain analysis

- Peak broadening:
  - Instrumental broadening
  - Size/strain

Instrumental broadening needs to be taken into account when doing size/strain analysis

- Crystallite size = the size of a single coherent crystal domain that scatters X-rays or neutrons coherently.
  - Perfect Bragg diffraction exists only with infinite crystals, but since that is not the case there is peak broadening
- Crystallite strain = Instead of all lattice planes having exactly the same spacing d, some regions are slightly compressed or slightly stretched.



Topas technical reference

NEUTRONS FOR SOCIETY

Crystallite size analysis

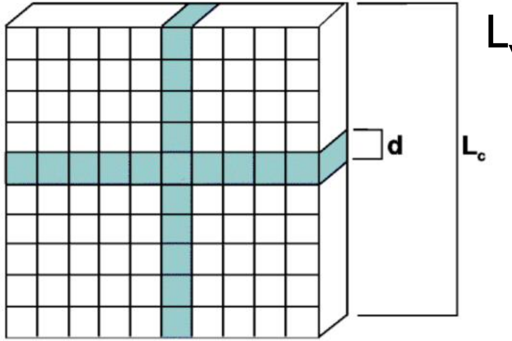
- Scherrer equation (1918)

$$\beta = \frac{\lambda}{\varepsilon \cos\theta} \qquad \varepsilon = \frac{l_{vol}}{k}$$

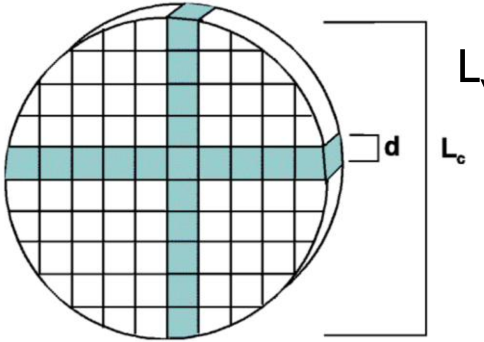
  - β – FWHM (irf corrected)
  - λ – wavelength
  - e – crystallite apparent size
  - l_{vol} – column size (weighted mean column length)
  - k – scherrer constant (depends on crystallite shape)

$$\beta = \frac{k\lambda}{l_{vol}\ \cos\theta}$$

- Laue (1926)
- $l_{vol} = \dfrac{\lambda}{\beta_{ib}\cos\theta}$
- β_{ib}=Integral breadth (IRF corrected)



$L_{vol}=L_0$



$L_{vol}=4/3L_0$

NEUTRONS FOR SOCIETY

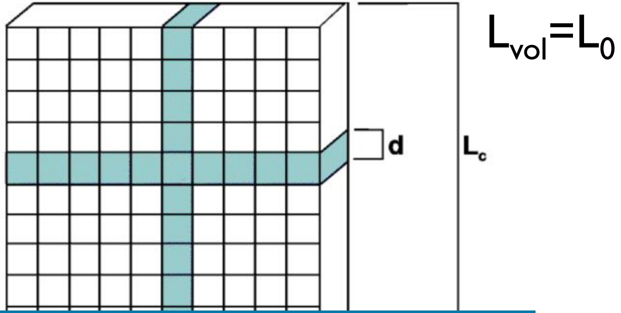
Crystallite size analysis

- Scherrer equation (1918)

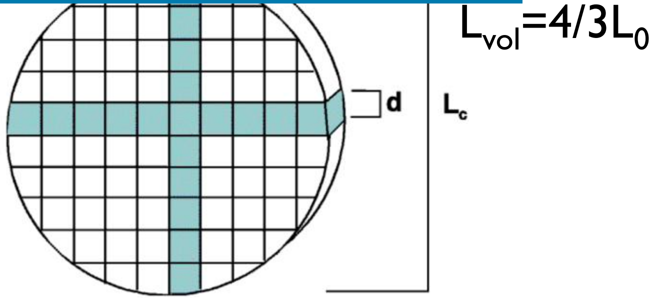
$$\beta = \frac{\lambda}{\varepsilon \cos\theta} \qquad \varepsilon = \frac{l_{vol}}{k}$$

  - β – FWHM (irf corrected)
  - λ – wavelength
  - e – crystallite apparent size
  - l_{v}
  - k

$$\beta = \frac{k\lambda}{l_{vol}\cos\theta}$$



Not the best method (although still widely used)!
Multi peak/whole pattern analysis would give information on anisotropy/strain.



- Laue (1926)
- $l_{vol} = \dfrac{\lambda}{\beta_{ib}\cos\theta}$
- β_{ib}=Integral breadth (IRF corrected)

NEUTRONS FOR SOCIETY

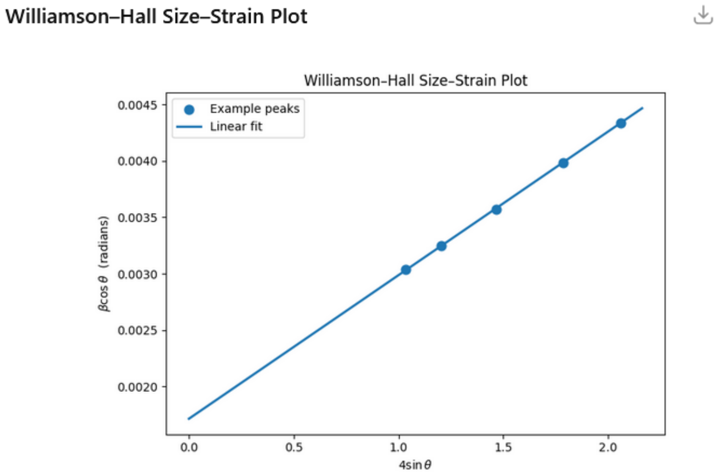
Crystallite strain analysis

- Williamson and hall (1953)
- Assumption: both size and shape are Lorentzian
  - Can add $\beta = \beta_{size} + \beta_{strain}$
  - βcos(θ) vs 4sin(θ) : slope – strain, intercept – CS (sometime 2sin(θ) is used, depending on strain definition)

$$\beta_{size} = \frac{\lambda}{l_{vol}\,\cos\theta}$$

$$\beta_{strain} = 4\varepsilon\tan\theta$$

$$\beta\cos\theta = \frac{\lambda}{l_{vol}} + 4\varepsilon\sin\theta$$



AI generated plot

NEUTRONS FOR SOCIETY

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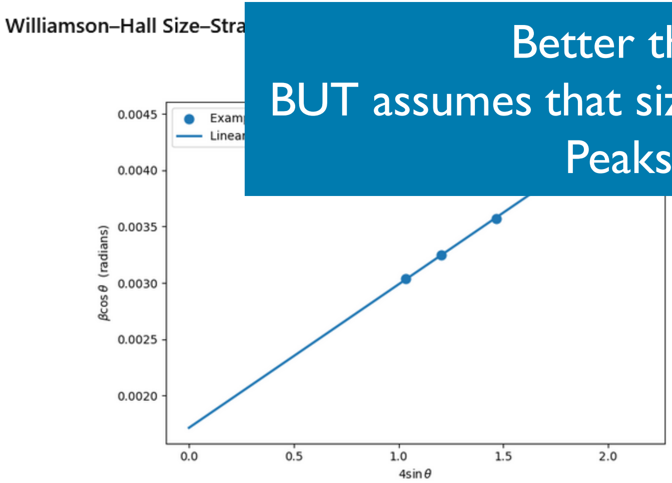
- Assumption: both size and shape are Lorentzian
  - Can add $\beta = \beta_{size} + \beta_{strain}$
  - $\beta\cos(\theta)$ vs 4sin(θ): slope – strain, intercept - CS

(sometime 2sin(θ) is used, depending on strain definition)

$$\beta_{size} = \frac{\lambda}{l_{vol} \cos\theta}$$

$$\beta_{strain} = 4\varepsilon \tan\theta$$



Better than Scherrer method,
BUT assumes that size and strain are purely Lorentzian
Peaks must be separated

AI generated plot

Crystallite size/strain analysis (profile fitting)

- Double Voigt approach
  - Size broadening ->Voigt function
  - Strain Broadening ->Voigt function
  - Uses full profile (not just width)
- Voigt function – convolution of Lorentzian and Gaussian
- Applied both in Fullprof and Topas
  - Instrumental broadening
    - Topas – FP peak type
    - Fullprof – IRF file
  - Fullprof peak type 7 (Thompson-Cox-Hastings formula)

Allows for more reliable determination of crystallite size and microstrain than simple width-based methods.
Do not trust the absolute values.

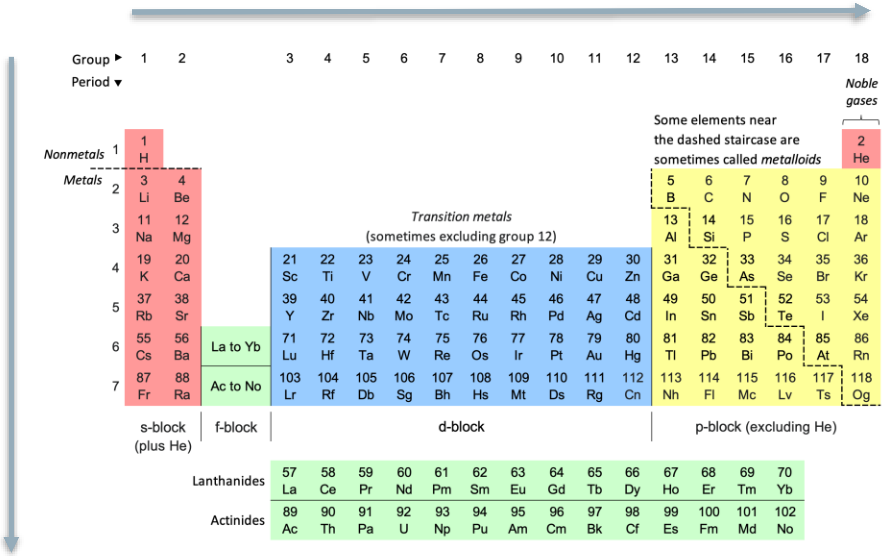
Voigt-function model in diffraction line-broadening analysis, Balzar (2003)

NEUTRONS FOR SOCIETY

Neutrons vs x-rays

X-rays:

- Interact with the electrons.
- Scattered by the electron cloud.
- X-ray form factor (scattering factor) has atomic number dependence.
- „Small penetration depth"
  (depends on energy)
  CuKα ~ 10 – 20 μm
  75 keV (ID31 ESRF) ~ 5 – 20 mm
  (depends on materials)



Wikimedia commons

NEUTRONS FOR SOCIETY

Neutrons vs x-rays

X-rays:

- Interact with the electrons.
- Scattered by the electron cloud.
- X-ray form factor (scattering factor) has atomic number dependence.
  - not so easy to distinguish between „neighbors“
- „Small penetration depth“ (depends on energy)
  CuKα ~ 10 – 20 μm
  75 keV (ID31 ESRF) ~ 5 – 20 mm
  (depends on materials)

neutrons:

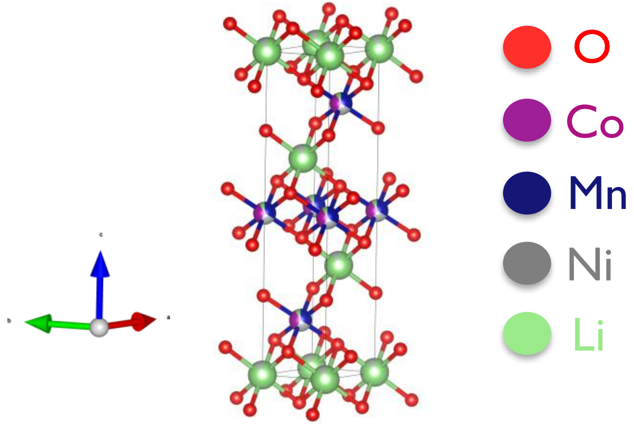
- Interact with nuclei (strong nuclear force).
- Point scattering from nuclei.
  - Strong reflections also at high angle.
- Scattering length denisty is „random“.
- Deep penetration depth.
- Isotope sensitive.
- Magnetic structure.
  - Neutrons have a spin (1/2), an additional scattering due to dipole-diploe interaction.

NEUTRONS FOR SOCIETY

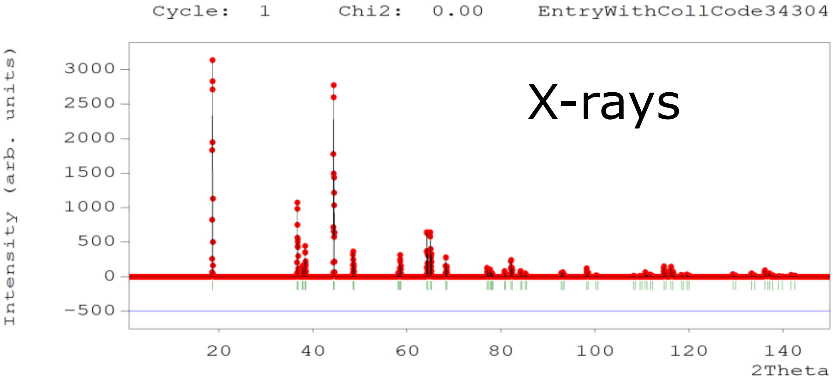
Neutrons vs x-rays

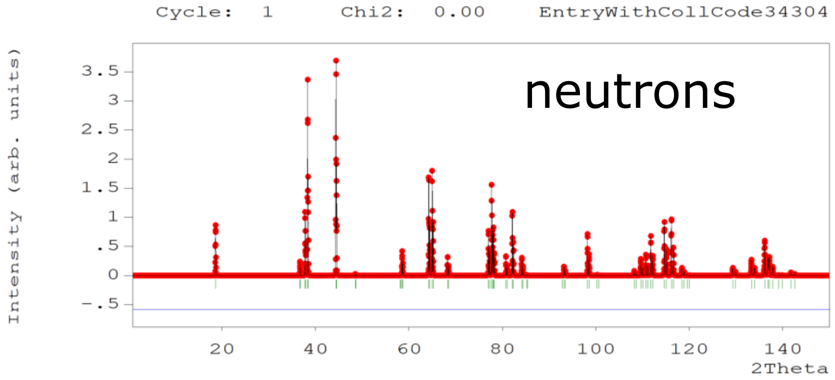
$LiNi_{0.33}Mn_{0.33}Co_{0.33}O_2$(NMC111)

```
_atom_site_occupancy
Li1  Li1+  3  a  0  0  0  0.025  0.92(4)
Co1  Co3+  3  b  0  0  0.5  0.015(9)  0.3333
Ni1  Ni3+  3  b  0  0  0.5  0.015(9)  0.26(4)
Mn1  Mn3+  3  b  0  0  0.5  0.015(9)  0.3333
O1  O2-  6  c  0  0  0.245(4)  0.017(2)  1
Li2  Li1+  3  b  0  0  0.5  0.015(9)  0.08(4)
Ni2  Ni3+  3  a  0  0  0  0.025  0.08(4)
```



Simulated patterns



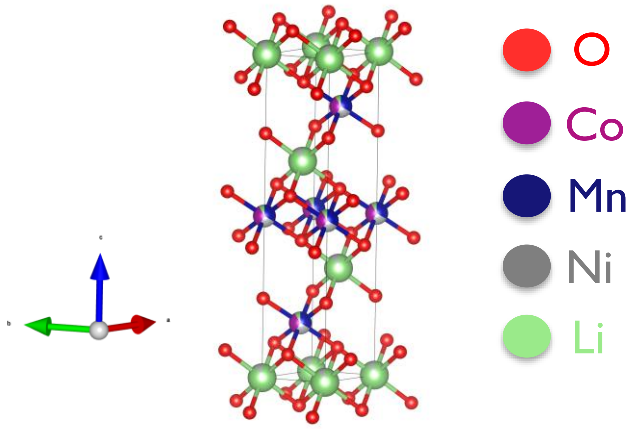


Neutrons vs x-rays

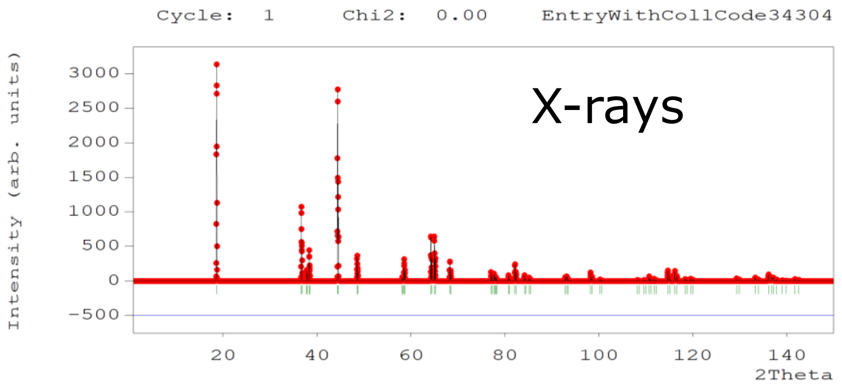
$LiNi_{0.33}Mn_{0.33}Co_{0.33}O_2$(NMC111)

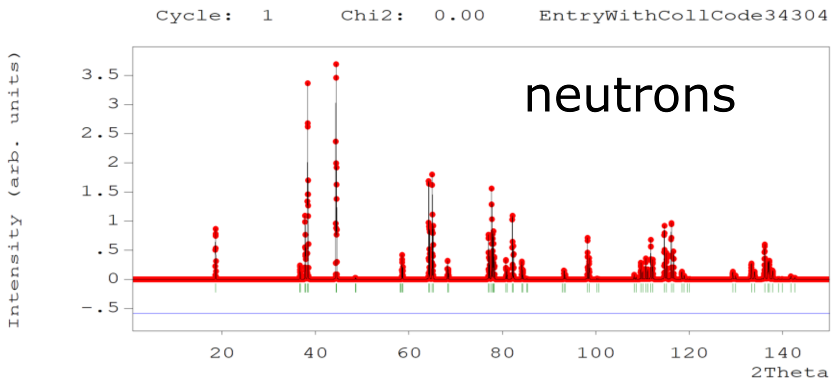
	Mn	Co	Ni
Neutron cross-sec	-3.75	2.490	10.30
X-rays z	25	27	28

https://www.ncnr.nist.gov/resources/activation/scattering_table.html#mn



Simulated patterns

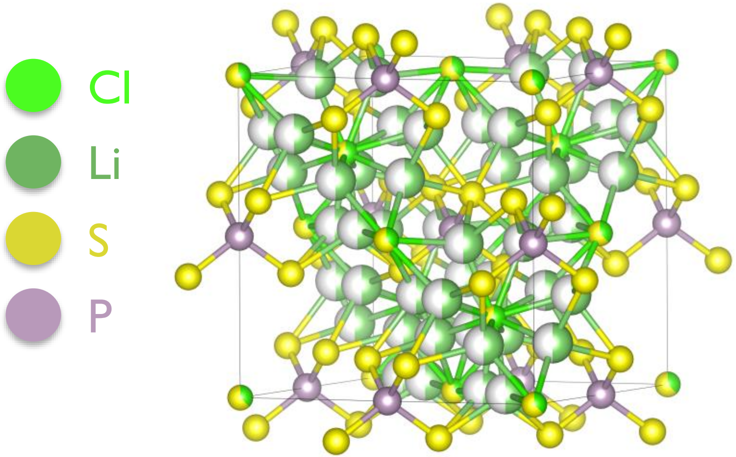


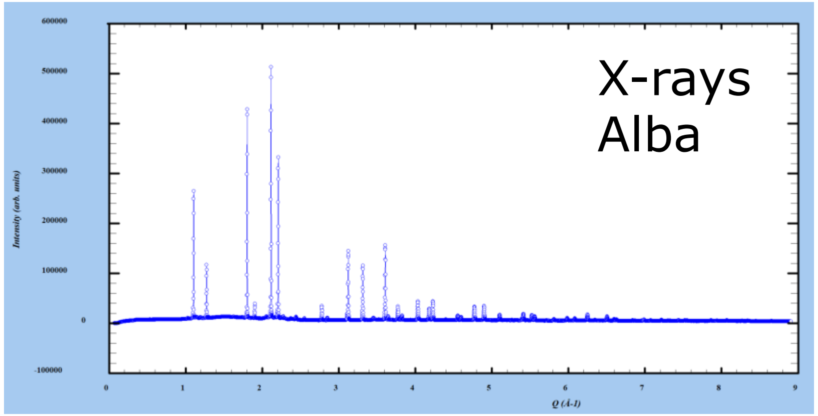


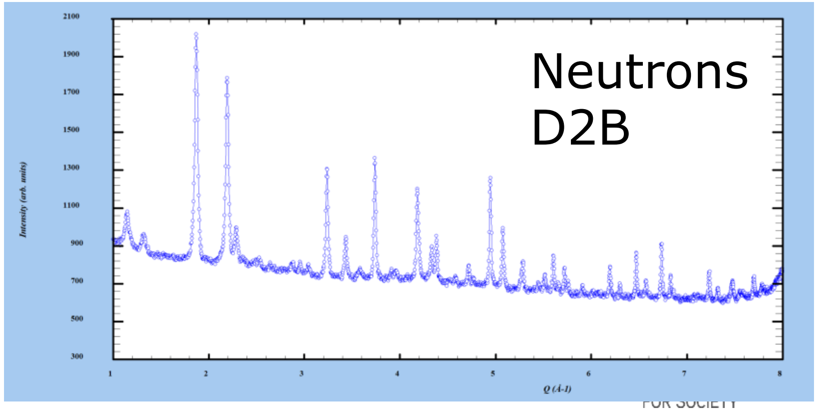
Neutrons vs x-rays

Li_6PS_5Cl (MSE)

```
Li1 Li1+ 48 h 0.3202 0.0183 0.6798 5.5 0.5
Cl1 Cl1- 4 a 0 0 1 3.94(9) 0.385
Cl2 Cl1- 4 d 0.25 0.25 0.75 2.96(8) 0.615
P1 P5+ 4 b 0 0 0.5 2.49(3) 1
S1 S2- 4 d 0.25 0.25 0.75 2.96(8) 0.385
S2 S2- 16 e 0.1200(2) -0.1200(2) 0.6200(2) 4.48(5) 1
S3 S2- 4 a 0 0 1 3.94(9) 0.615
```







Neutrons vs x-rays

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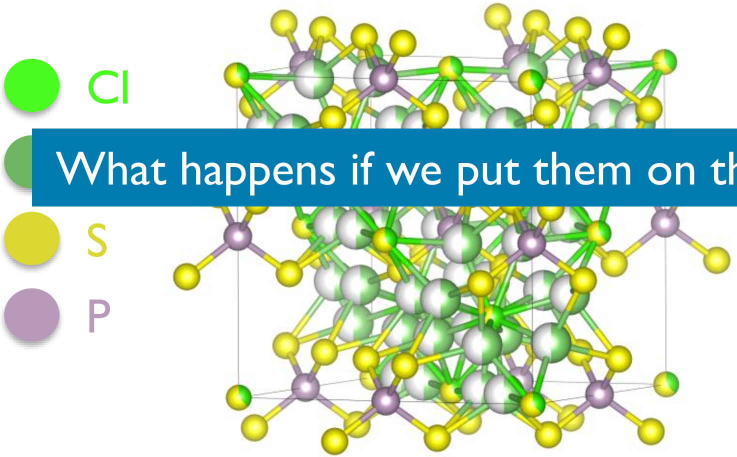
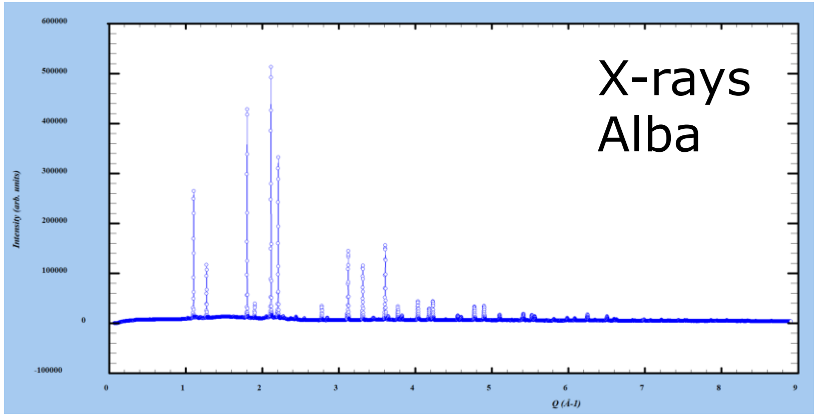
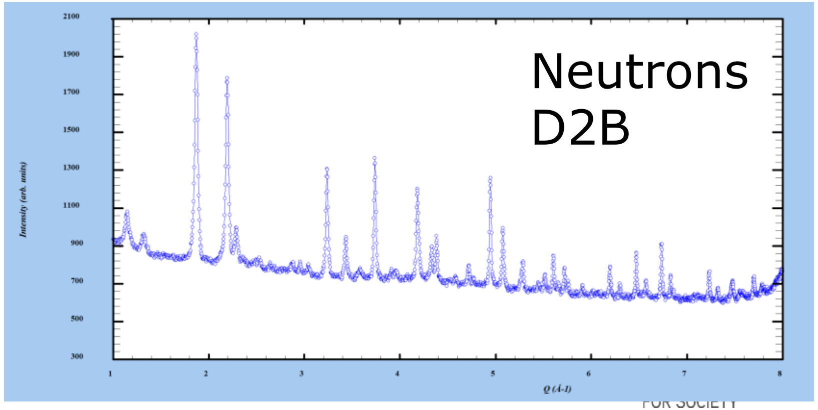
```



What happens if we put them on the same graph?





Neutrons vs x-rays (in general)

X-rays (synchrotron):

- Possibility of lab-scale
- Super high flux
- Fast measurements
- Lower penetration
  - Less choice for sample cells
- Sensitive to heavy elements
- No magnetic structure
- Possibility of profiling, and special methods (XRD-CT)

neutrons:

- No lab-scale
- Good to determine atomic positions, isothermic factors
- Slower measurements
- Higher penetration
  - Sample cell versatility (no hydrogen containing plastic)
- Sensitive to light elements
- Magnetic structure

Microstructure

NEUTRONS FOR SOCIETY

Neutrons vs x-rays (in general)

X-rays (synchrotron):

- Possibility of lab-scale
- Super high flux
- Fast measurements
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neutrons:

- No lab-scale
- Good to determine atomic positions, isothermic factors
- easurements
- Higher penetration
  - Sample cell versatility (no hydrogen containing plastic)
- Sensitive to light elements
- Magnetic structure

Why not use both ?

Microstructure

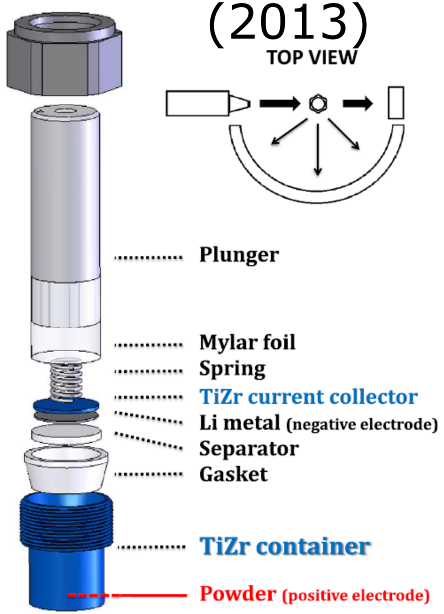
NEUTRONS FOR SOCIETY

Neutron *operando* cells for EC

Neutrons can have **negative** scattering lengths (wave is shifted in phase by π (180°) relative to the incident wave)

Bianchini et al., J. Electrochem. Soc., **160** (11) A2176-A2183 (2013)

ILLBAT#1 (2013)



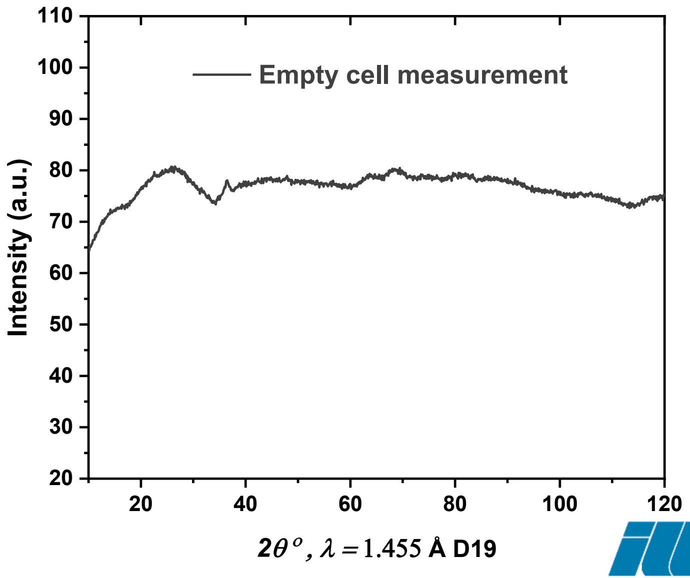
$Ti_{2.08}Zr$

Null-matrix Alloy

b_{Ti} = -3.44 fm

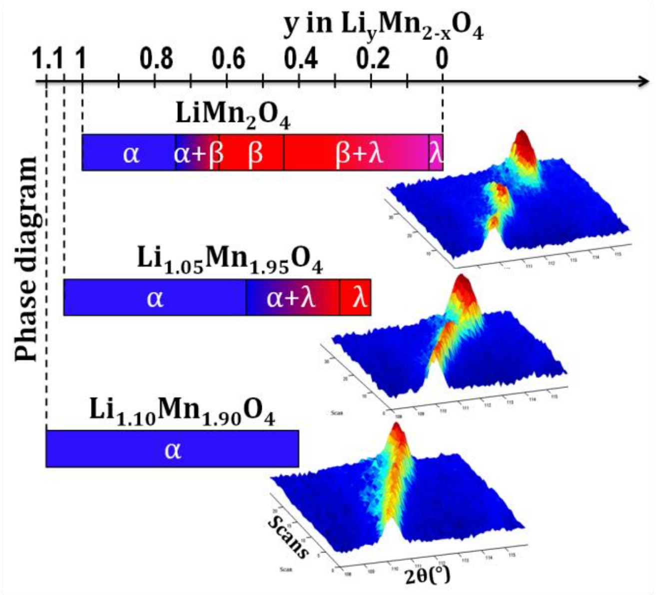
b_{Zr} = 7.16 fm

Prof. Matteo Bianchini



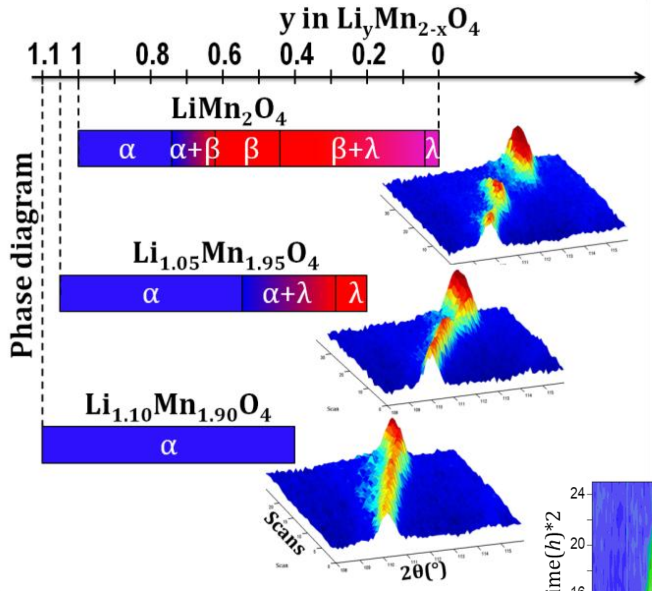
NEUTRONS FOR SOCIETY

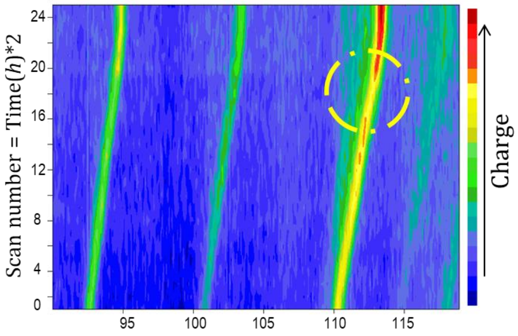
$Li_{1+x}Mn_{2-x}O_4$ *operando* cycling



J. Phys. Chem. C 2014, 118, 45, 25947–25955

$Li_{1+x}Mn_{2-x}O_4$ *operando* cycling





J. Phys. Chem. C 2014, 118, 45, 25947–25955

NEUTRONS FOR SOCIETY

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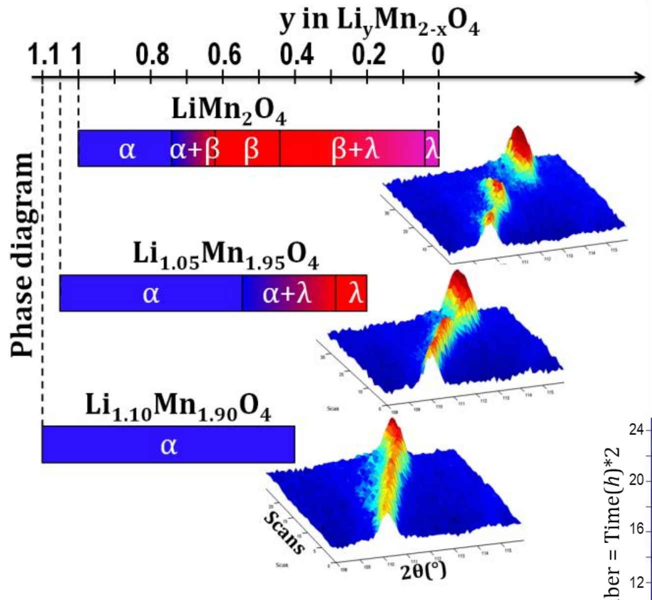

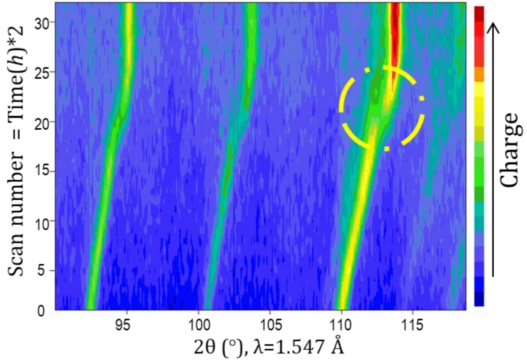

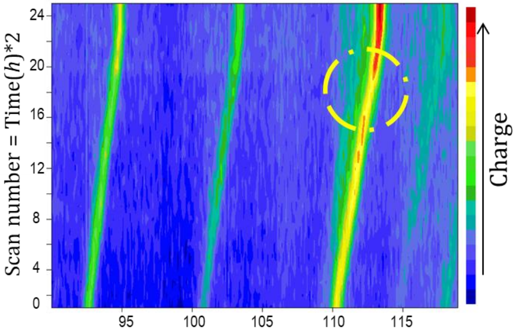

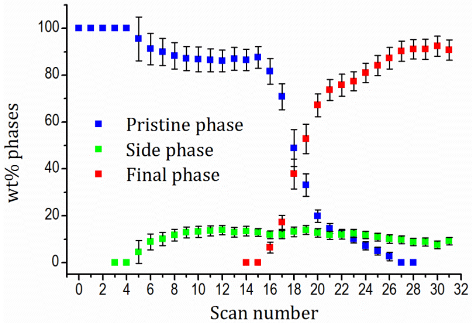

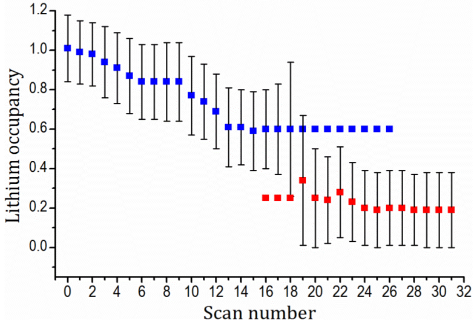

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NEUTRONS FOR SOCIETY

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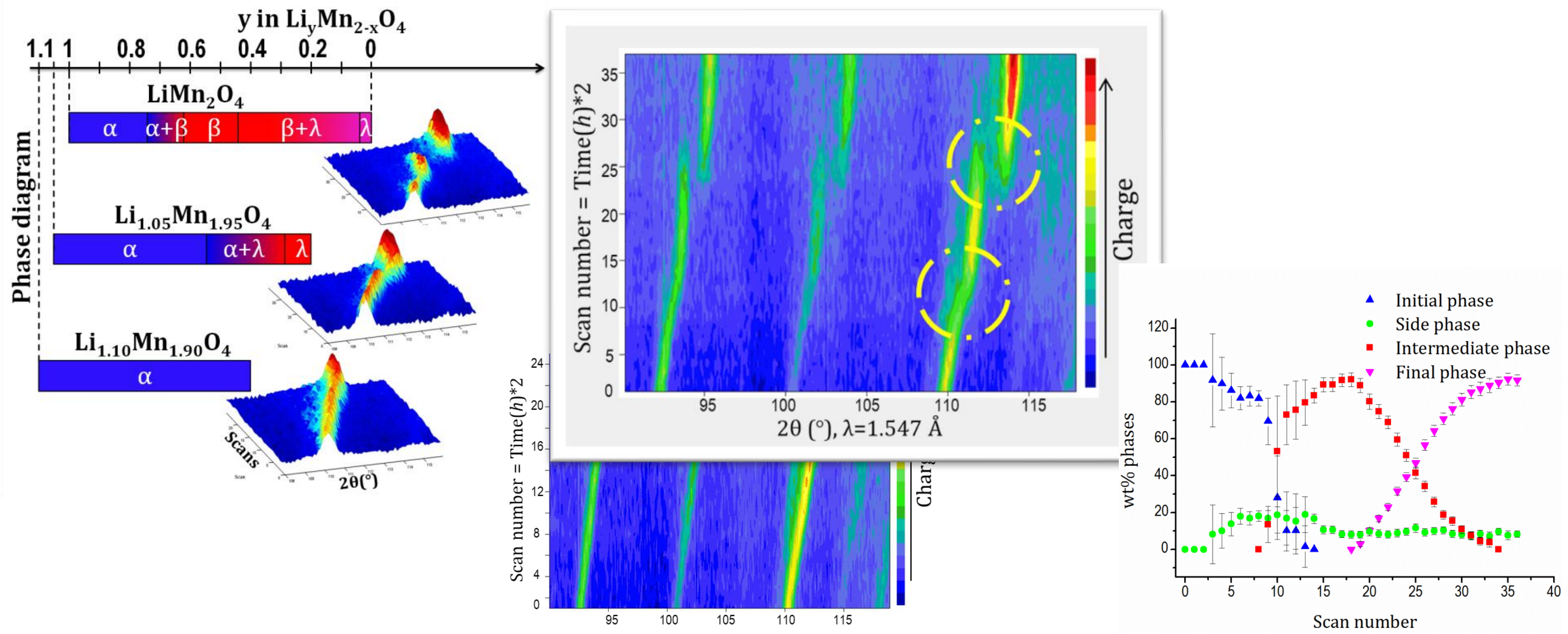
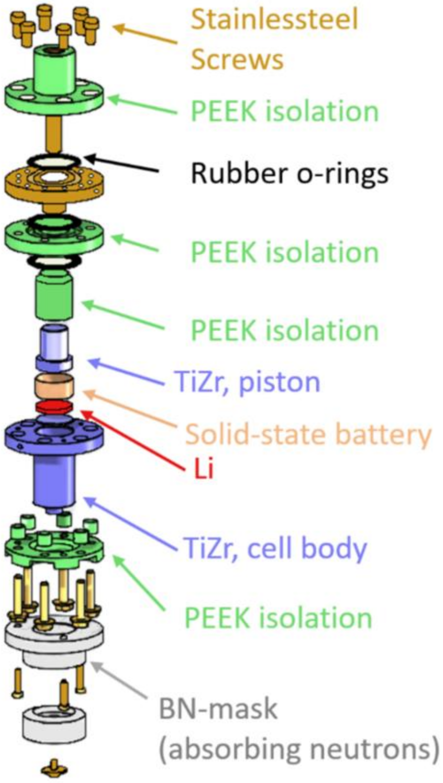
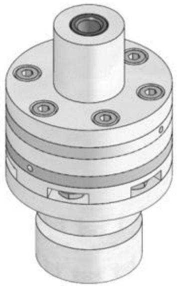


NEUTRONS FOR SOCIETY

Cycling of solid-state batteries





Korjus et al. ACS Materials Lett. 2025, 7, 8, 2725–2731

NEUTRONS FOR SOCIETY

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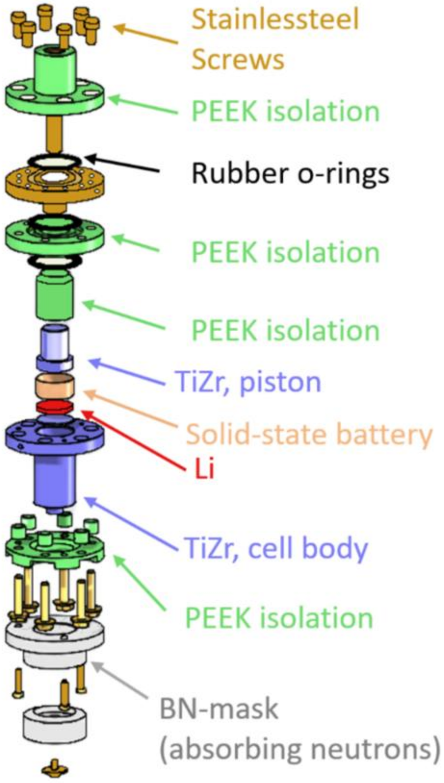
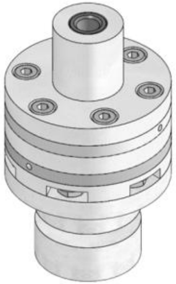
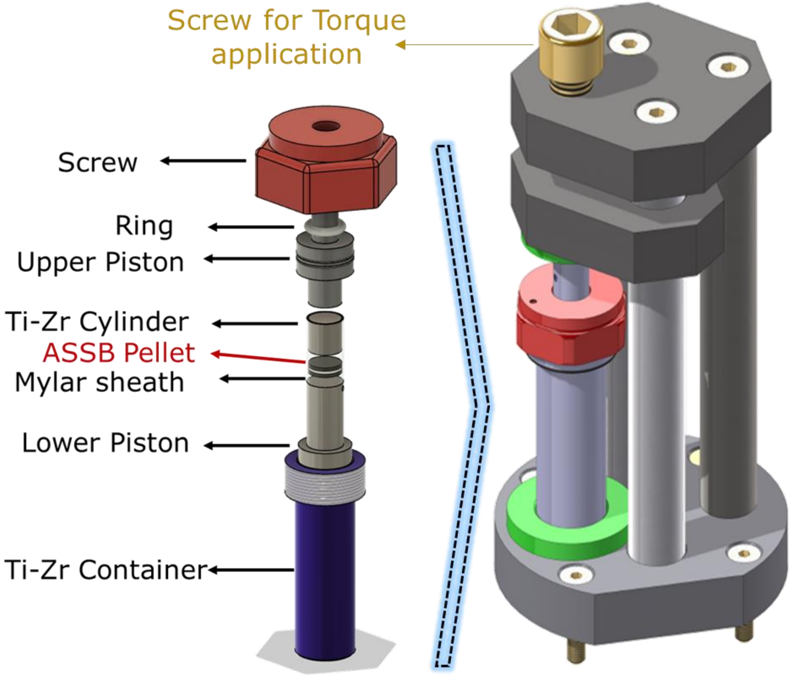
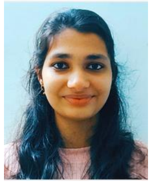




Korjus et al. ACS Materials Lett. 2025, 7, 8, 2725–2731



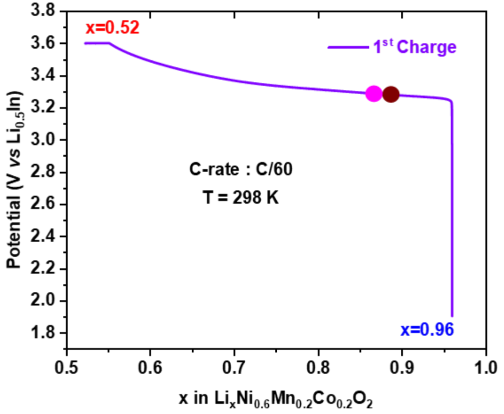
S.A.Kumar et al. Advanced Energy Materials (2026)



Dr. Anil Kumar

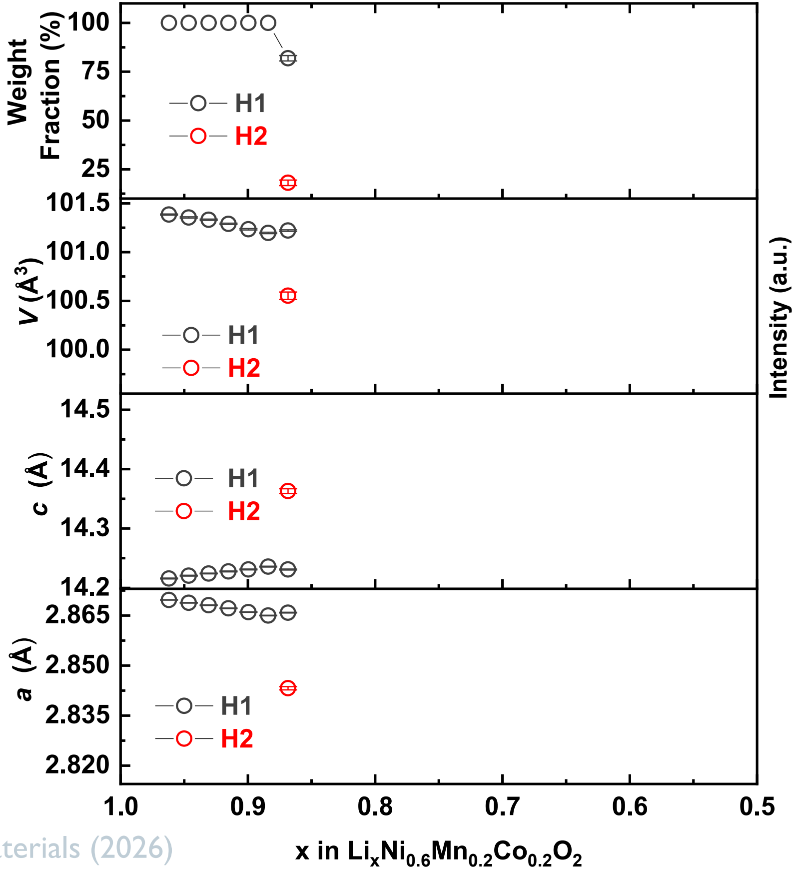
NEUTRONS
FOR SOCIETY

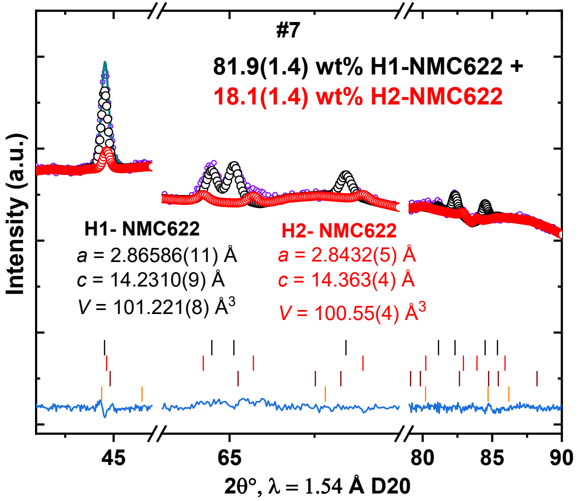
Cycling of solid-state batteries



Solid solution mechanism until: $Li_{0.88}$NMC622 structure

In **biphasic** regime:: H1 phase → $Li_{0.88}$NMC622 structure

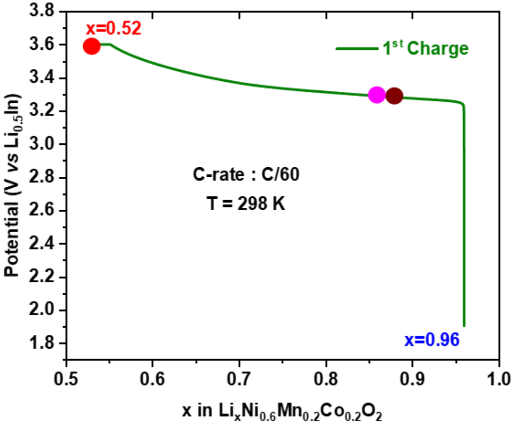


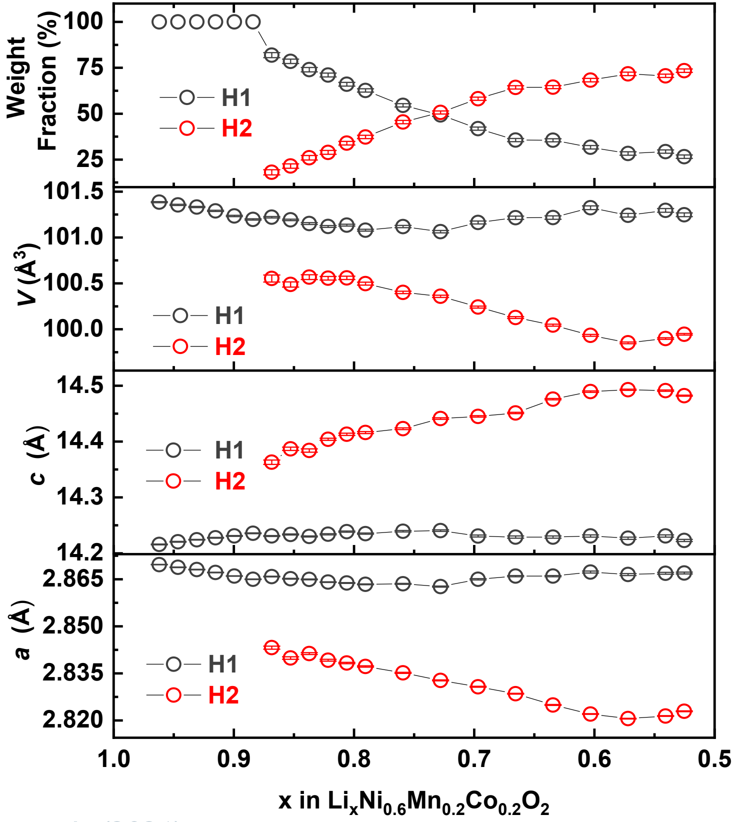


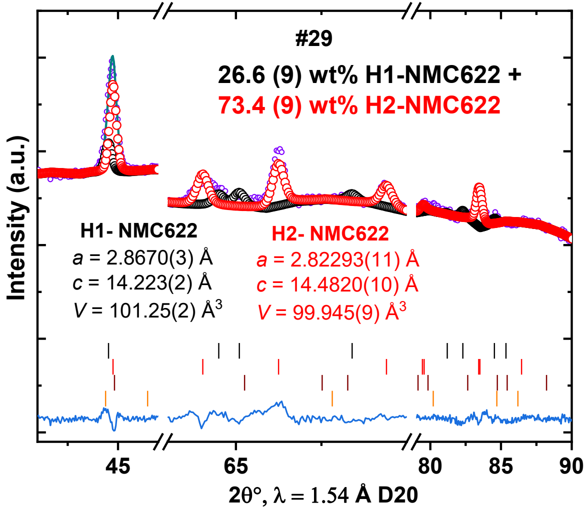
S.A.Kumar et al. Advanced Energy Materials (2026)

NEUTRONS FOR SOCIETY

Cycling of solid-state batteries





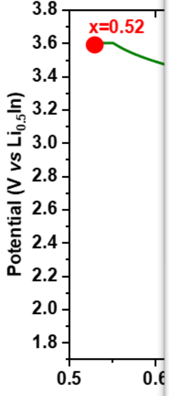


In **biphasic** regime:
H1 NMC remains stable while **H2 NMC** continues to evolve in structure and composition

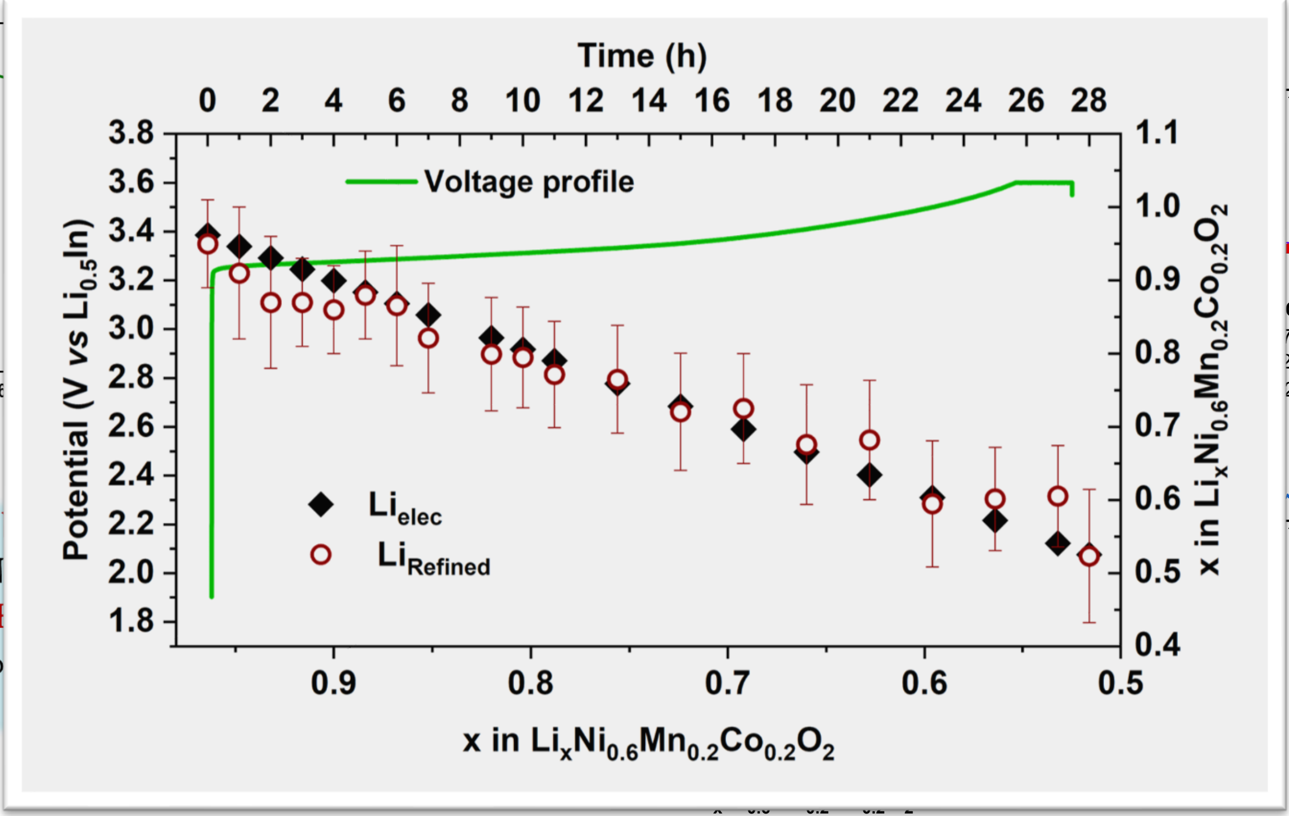
S. A.Kumar et al. Advanced Energy Materials (2026)

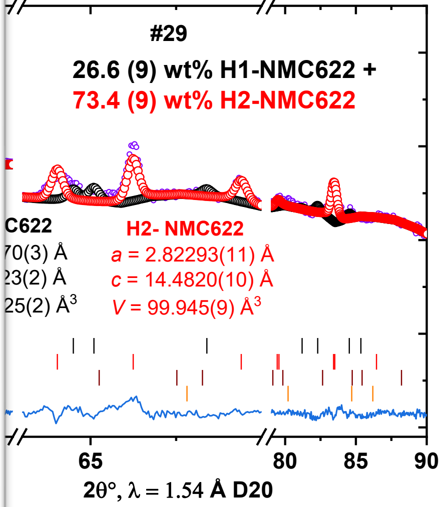
NEUTRONS FOR SOCIETY

Cycling of solid-state batteries



In
H1 N
while **I**
to evo





S.A.Kumar et al. Advanced Energy Materials (2026)

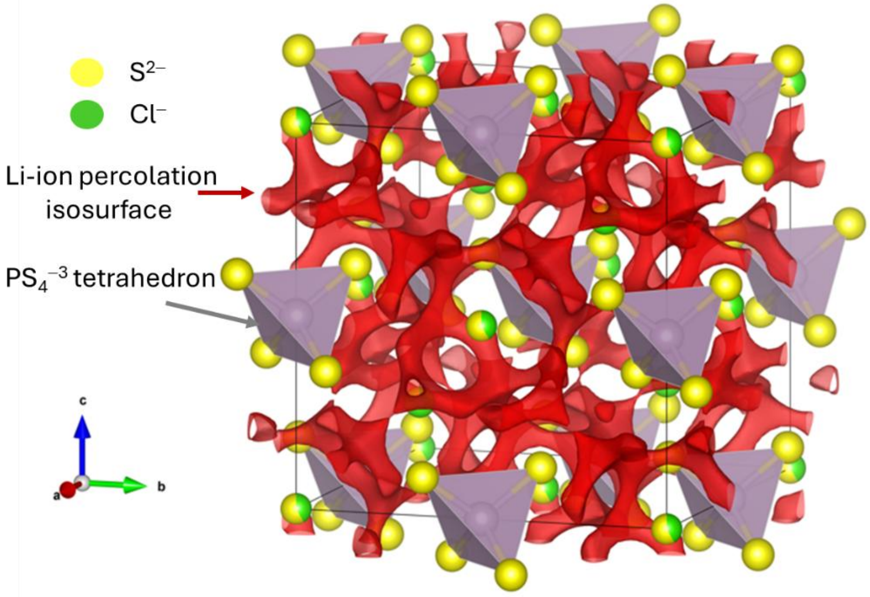
NEUTRONS FOR SOCIETY

Bond valence energy landscapes

BVEL is a semi-empirical method based on the bond valence model.

It estimates the energy of placing an ion in the structure using bond valence mismatch with surrounding atoms.

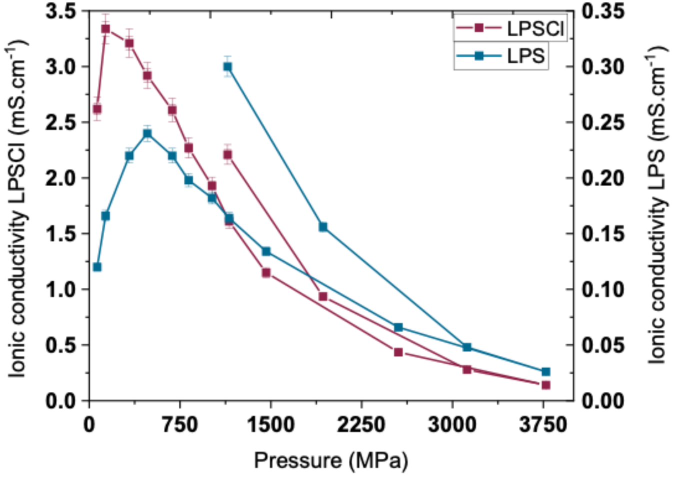
Li_6PS_5Cl



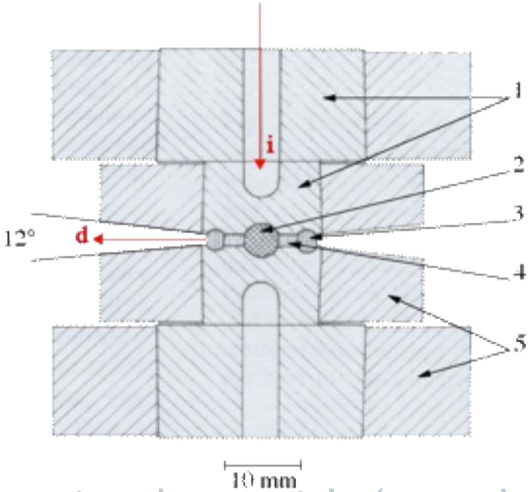
Workflow:

- Refine structure (Fullprof)
- Export CIF
- Remove mobile ions
- Run BVEL calculation
- Visualize pathways

High pressure application on LPSCl



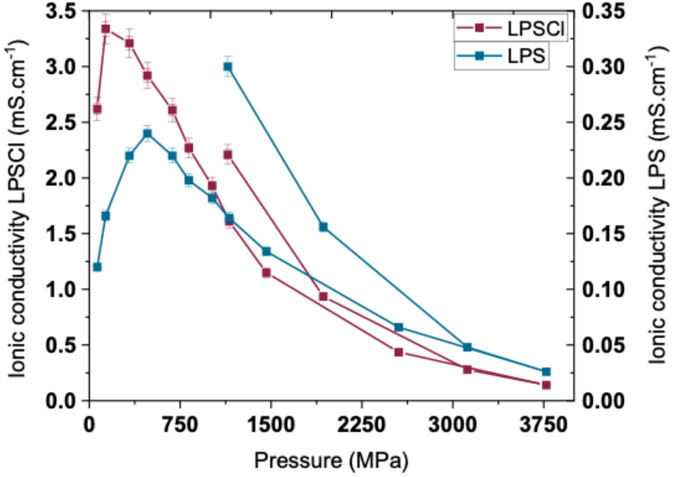
10 GPa maximum
Ø3 x 0.6mm < 10 GPa
Ø6 x 0.6mm < 5 GPa

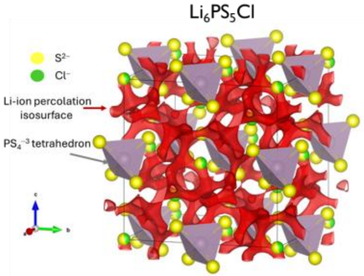


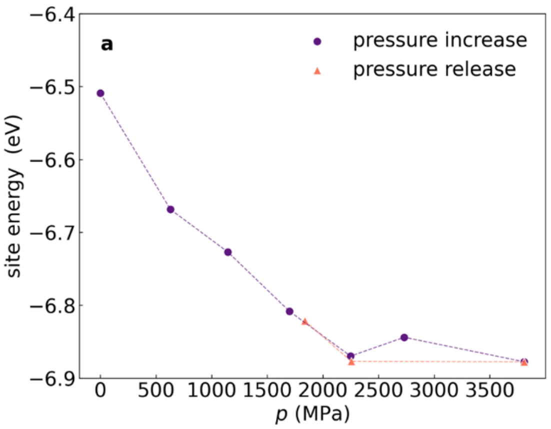
Thompson, Korjus, Villevieille et al. Adv. Functional materials (soon, hopefully)

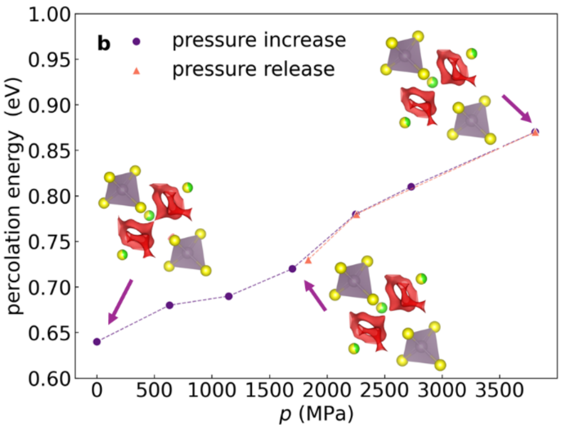
NEUTRONS FOR SOCIETY

High pressure application on LPSCl





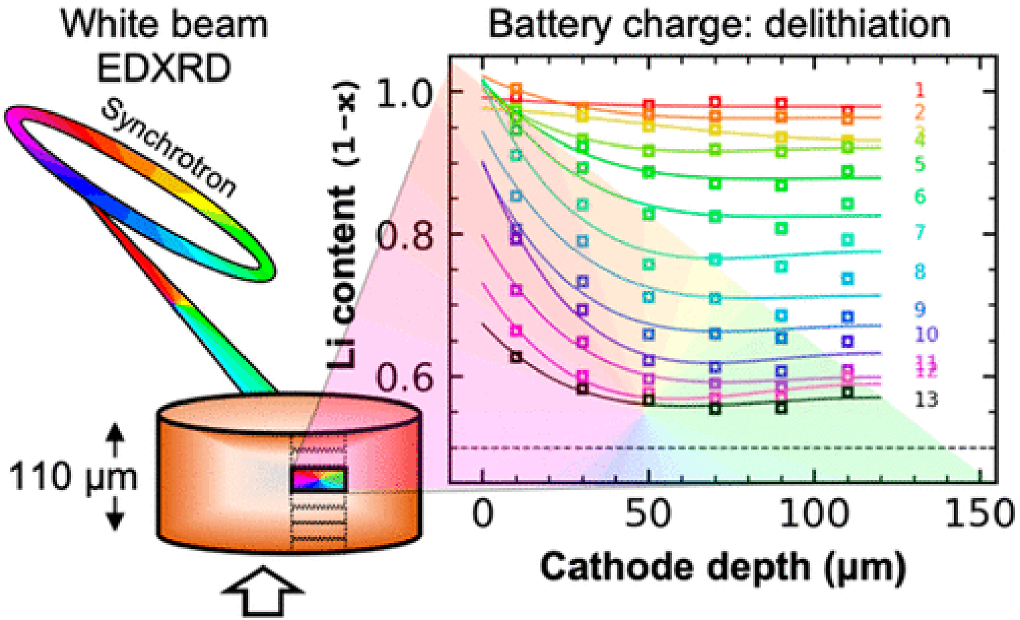




Thompson, Korjus, Villevieille et al. Adv. Functional materials (soon, hopefully)

NEUTRONS FOR SOCIETY

Depth profiling using at synchrotron

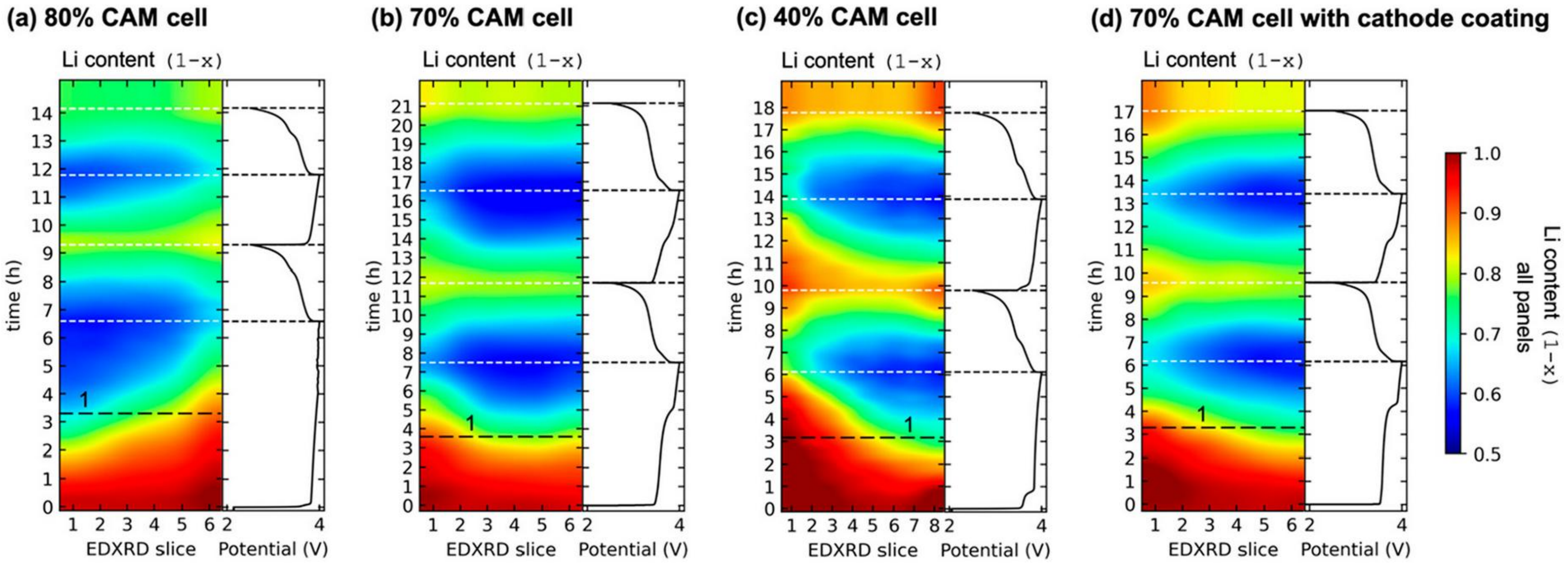


$Li_{(1\text{-}x)}Ni_{1/3}Mn_{1/3}Co_{1/3}O_2 + Li_6PS_5Cl$ cathode

Stavola et al., ACS Energy letters. 2025

NEUTRONS FOR SOCIETY

Depth profiling using at synchrotron



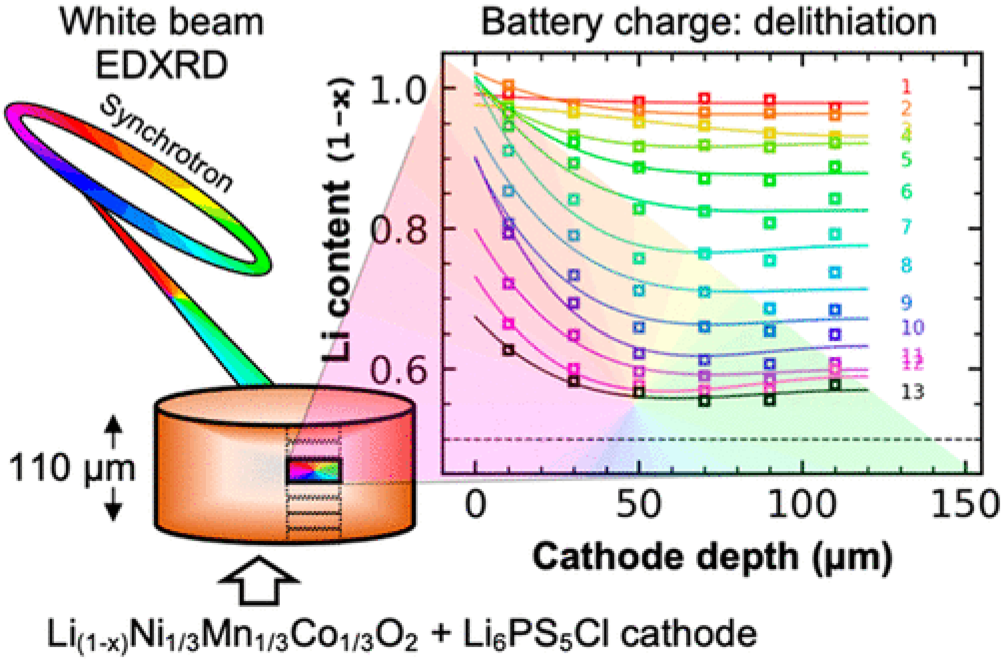
I slice 20 um

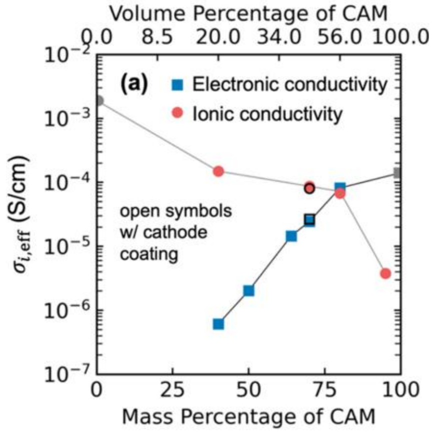
Stavola et al., ACS Energy letters. 2025

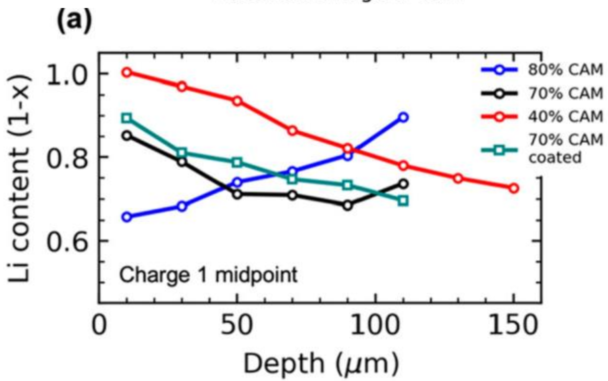
Li content estimated from LP (3 peak fit)

NEUTRONS FOR SOCIETY

Depth profiling using at synchrotron





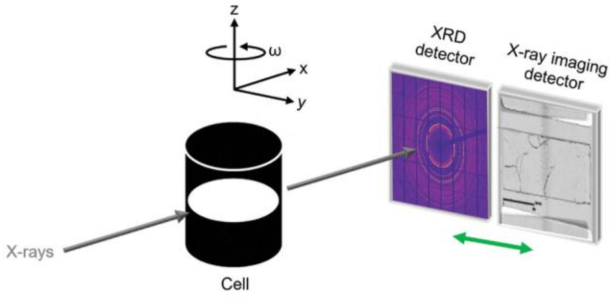


Stavola et al., ACS Energy letters. 2025

Li content estimated from LP (3 peak fit)

NEUTRONS FOR SOCIETY

XRD-CT

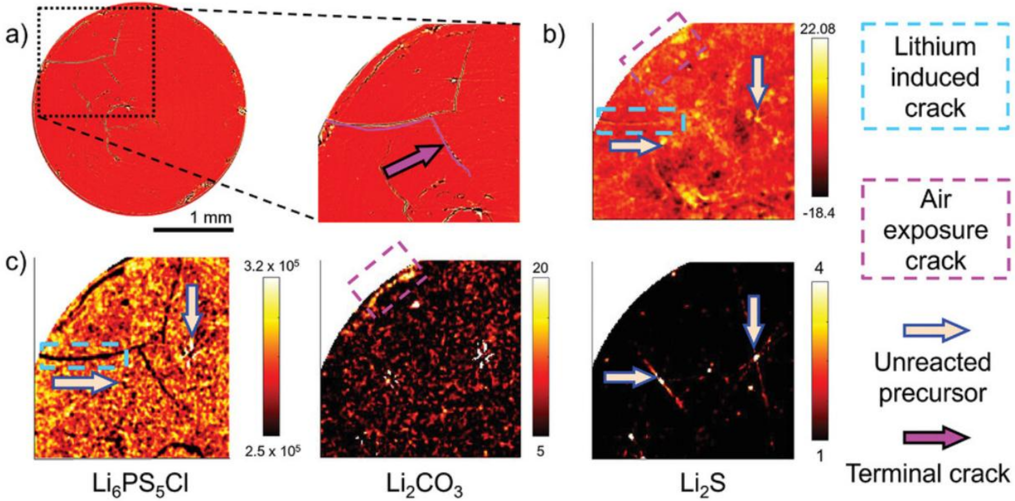


$$\mathscr{E} = \frac{a^3_{\mathrm{Li_6PS_5Cl}} - a^3_{\mathrm{mean}}}{a^3_{\mathrm{mean}}}$$

a_{mean} – LP at bulk

LPSCl degradation $Li|Li_6PS_5Cl|Li$

Stress (volume change)



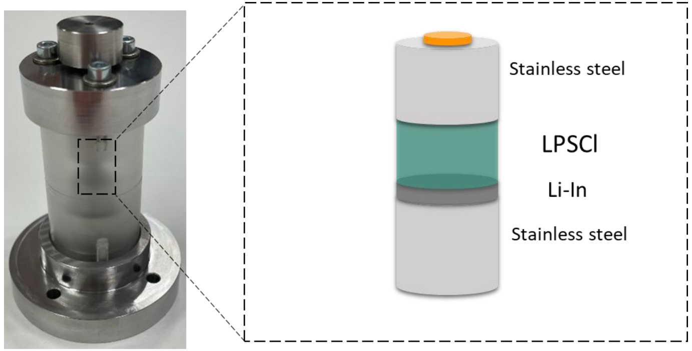
Rietveld refinement(topas7): (wt%, LP)

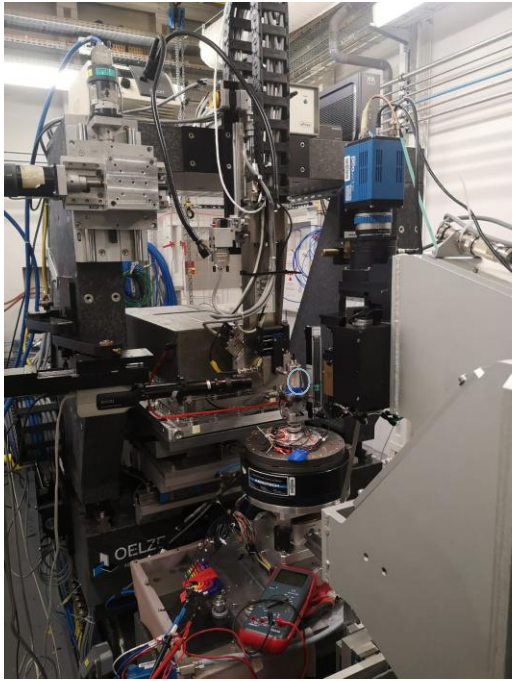
Hu et al. Adv. Energy Mater.2025

NEUTRONS FOR SOCIETY

XRD-CT

0 – 2 V vs Li-In
Li-In | LPSCl | Stainless steel





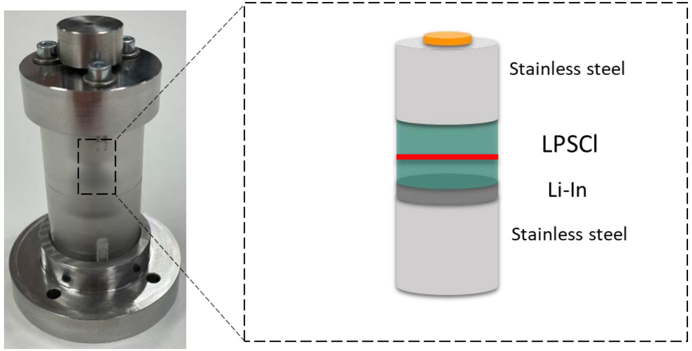
Oskar Thompson

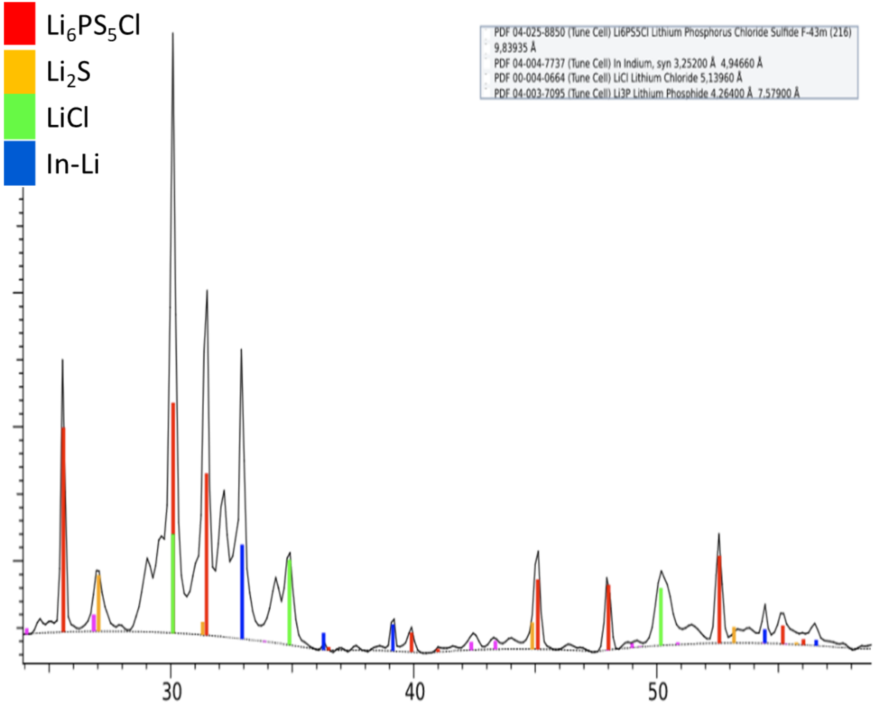
Villevieille et al. 2024, Research Square

XRD-CT

0 – 2 V vs Li-In

Li-In | LPSCl | Stainless steel





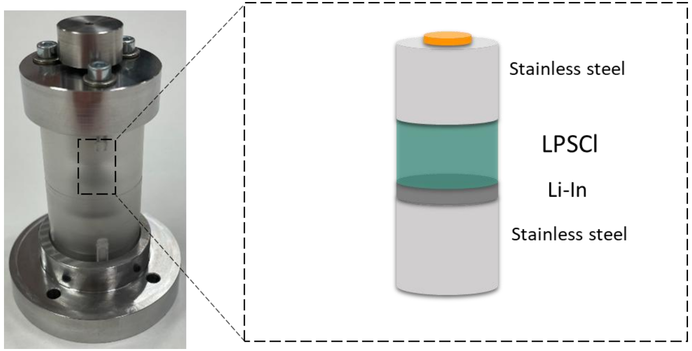
Villevieille et al. 2024, Research Square

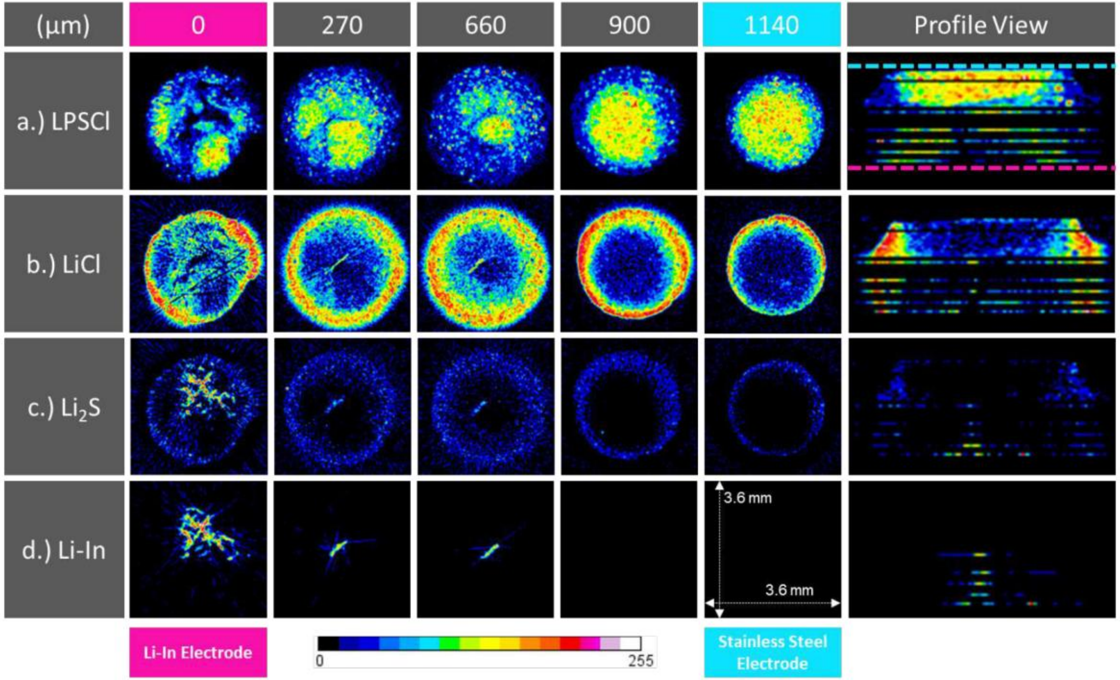
NEUTRONS FOR SOCIETY

XRD-CT

0 – 2 V vs Li-In

Li-In | LPSCl | Stainless steel





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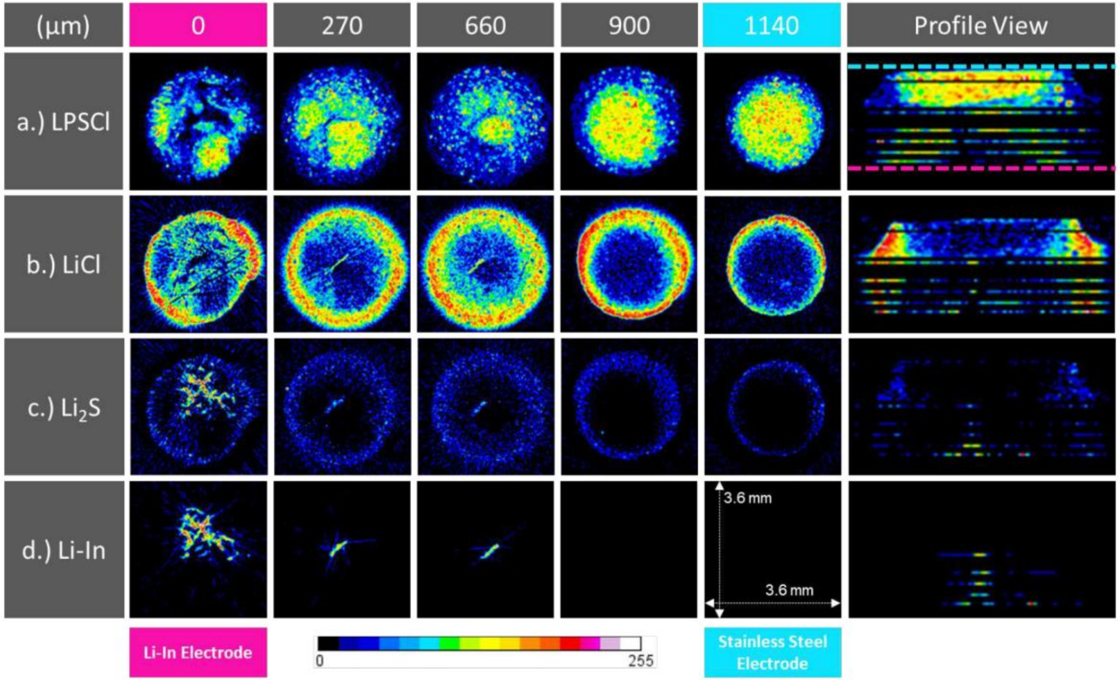
XRD-CT



- LPSCl/LiCl signal inversely proportional
- Porosity is greater around the perimeter of the cell
- Perimeter porosity appears to be correlated to degradation

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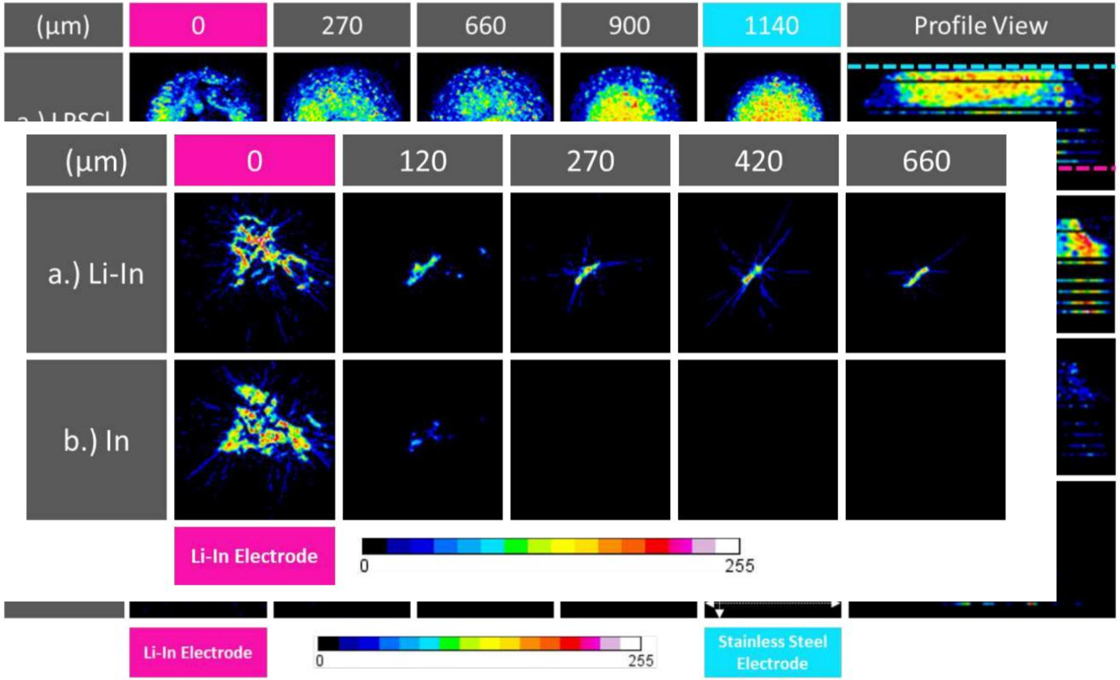
XRD-CT

- Li/In-phases with different Li-content



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Thank You

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