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Precision investigation of modern crystalline materials by neutron diffraction method

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Frank Laboratory of Neutron Physics (FLNP)

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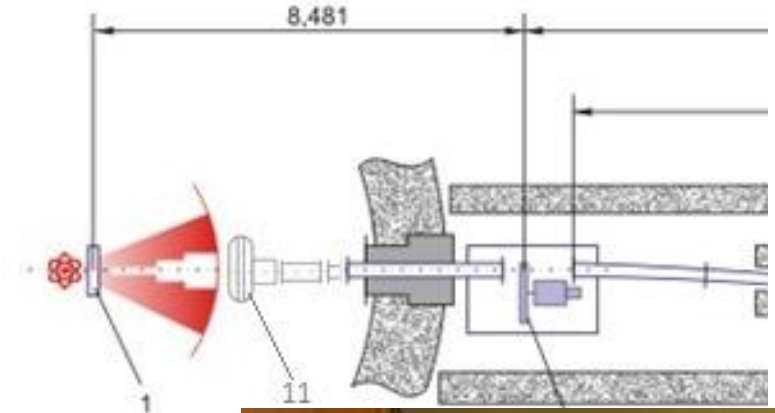
AIM

➢ **Investigation of magnetic structure of NiO by data treatment by Rietveld method**

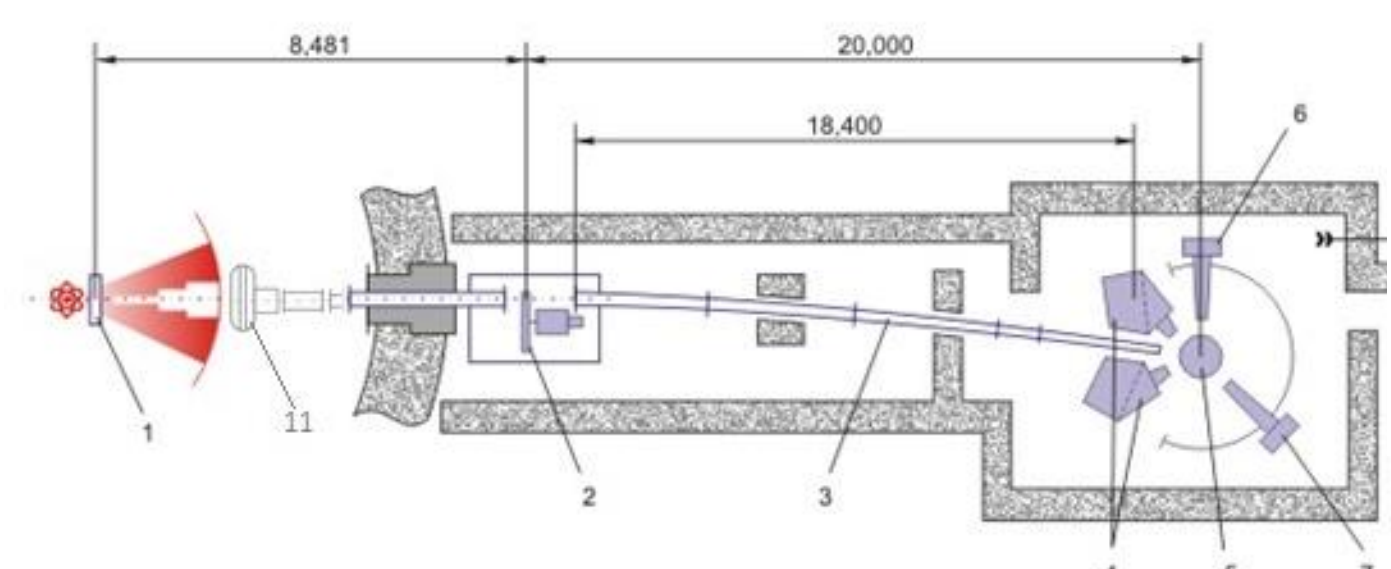
- Increase knowledge of neutron difraction method
- Practise and basic information using standard samples
- Investigation of a real sample (crystal and magnetic structure of NiO)

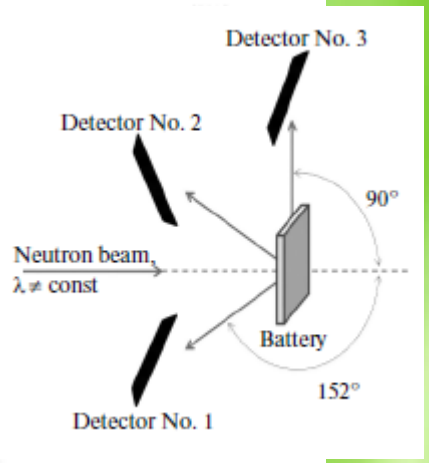
High Resolution Fourier Diffractometer (HRFD)



1 – M
2 – M
3 – C
4 – M
5 – S
6 – S
7 – M
8 – V
9 – V
10 – M
11 – M

High Resolution Fourier Diffractometer (HRFD)



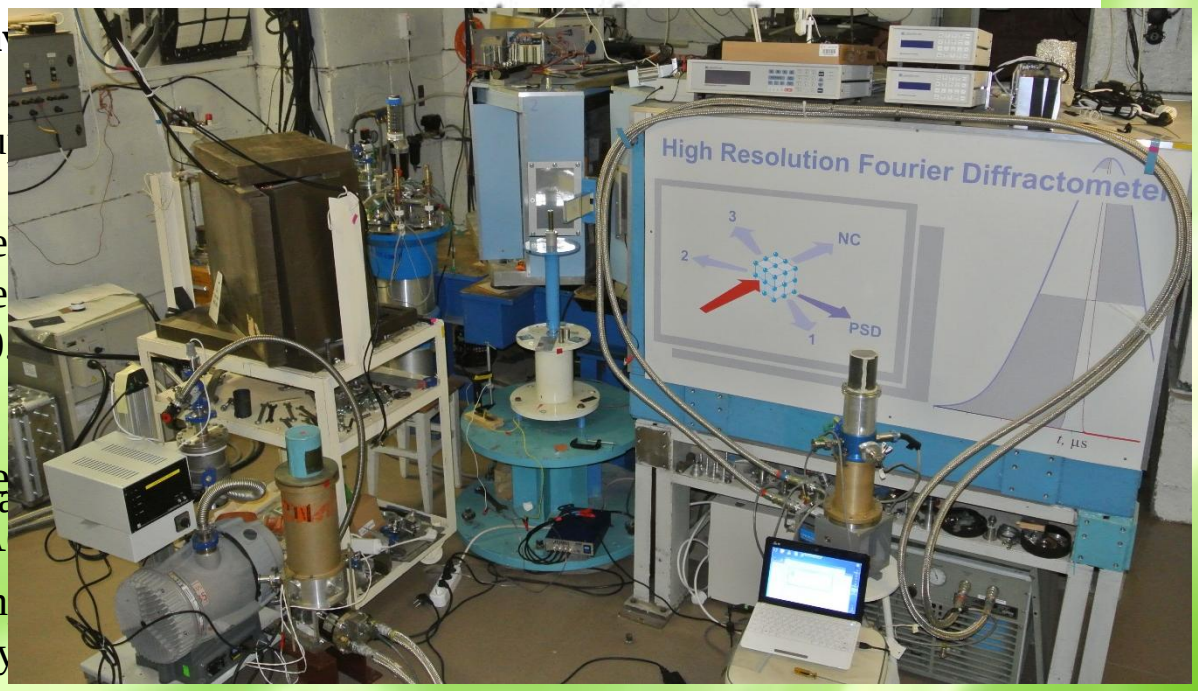


The available sample env

1 – Moderator
2 – Fourier Chopper
3 – Guide Tube
4 – Main Detector
5 – Sample Position
6 – 90 -Detector
7 – PSD Detector
8 – VME Control and Operative
9 – VME Station (OS/9) Data A
10 – EtherNet Data Transfer
11 – Background chopper

1) Air furnace (from RT u
2) Vacuum furnace (from
3) Closed-cycle helium re
4) Closed-cycle helium re
5) Electromagnet (up to 0
responsable person);
6) Goniometer GKS-100;
7) Galvanostat-potentiosta

Possibility to prepare sam
possibility to make X-Ray



Pulse neutron sources

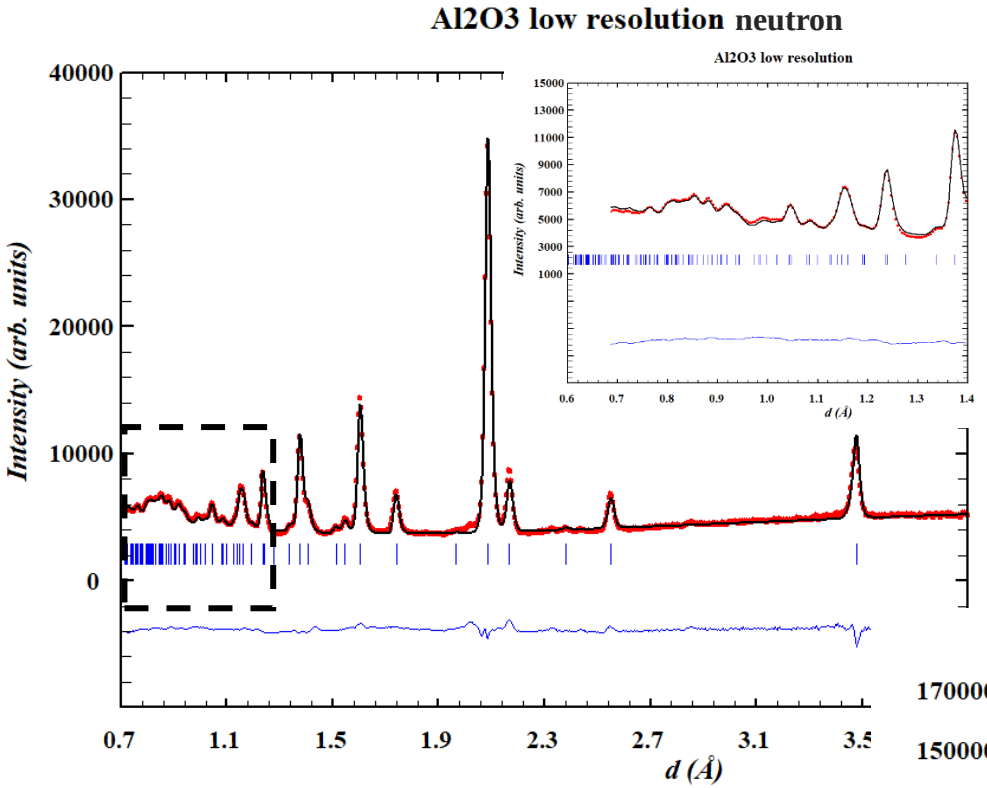
- Pulse neutron sources differ from conventional sources (fixed λ) in that the entire moderated beam with multiple wavelengths is used at fixed angle and the diffraction pattern is recorded as the function of *time of flight* of the neutrons.

- λ=h/p p=mv v=L/t => $\lambda = \frac{ht}{Lm}$

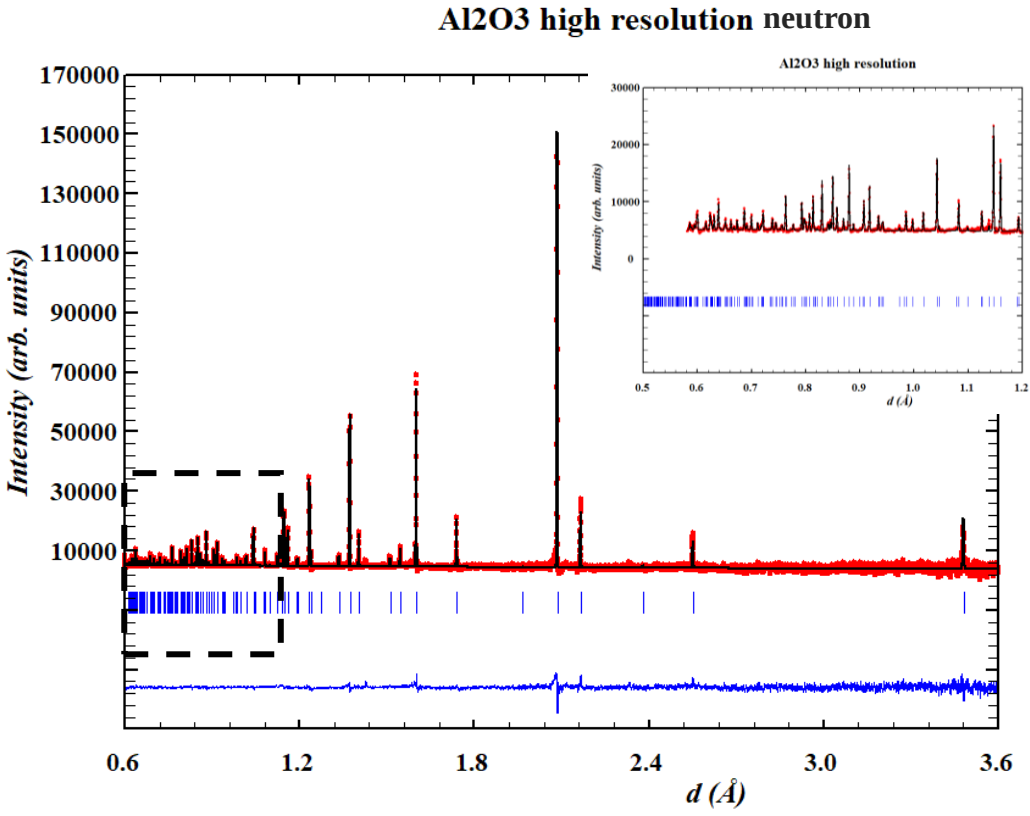
$$\mathrm{TOF} = \mathbf{Zero} + \mathbf{Dtt1}\, d + \mathbf{Dtt2}\, d^2$$

$$\sigma^2 = (Sig2 + GSIZ)d^4 + (Sig1 + DST)d^2 + Sig0$$

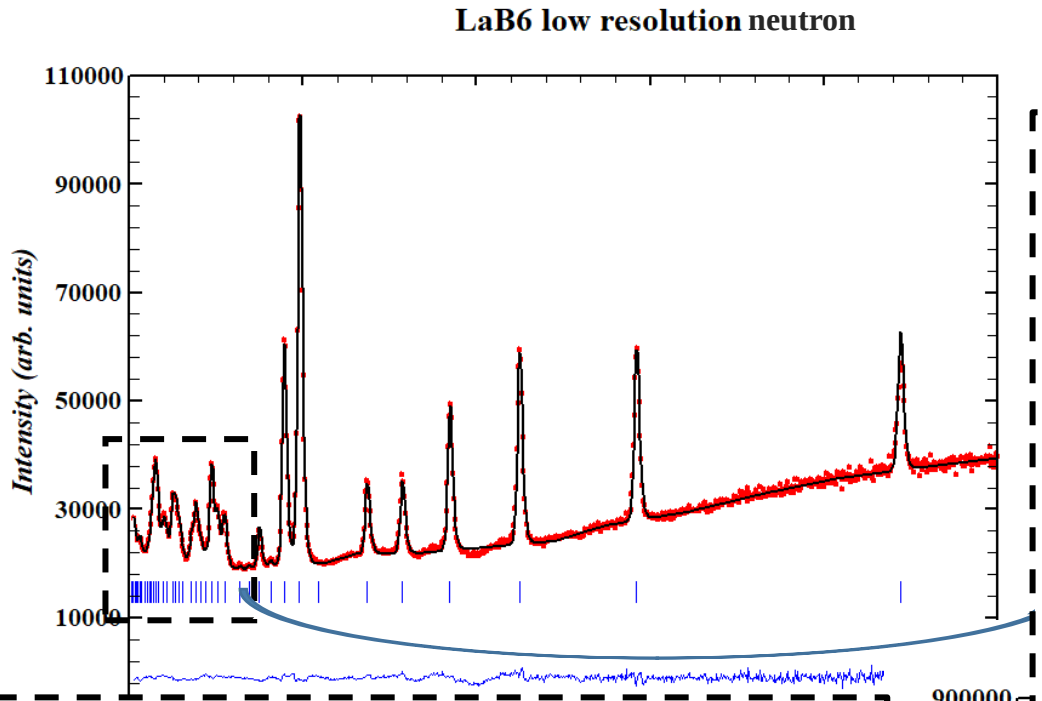
Standard sample – Al_2O_3

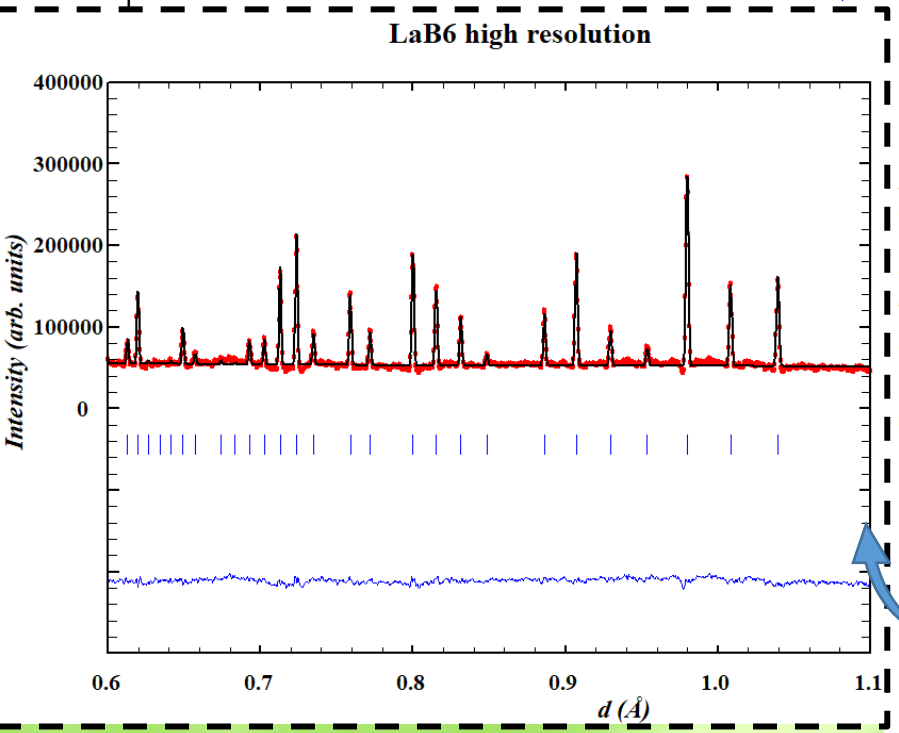


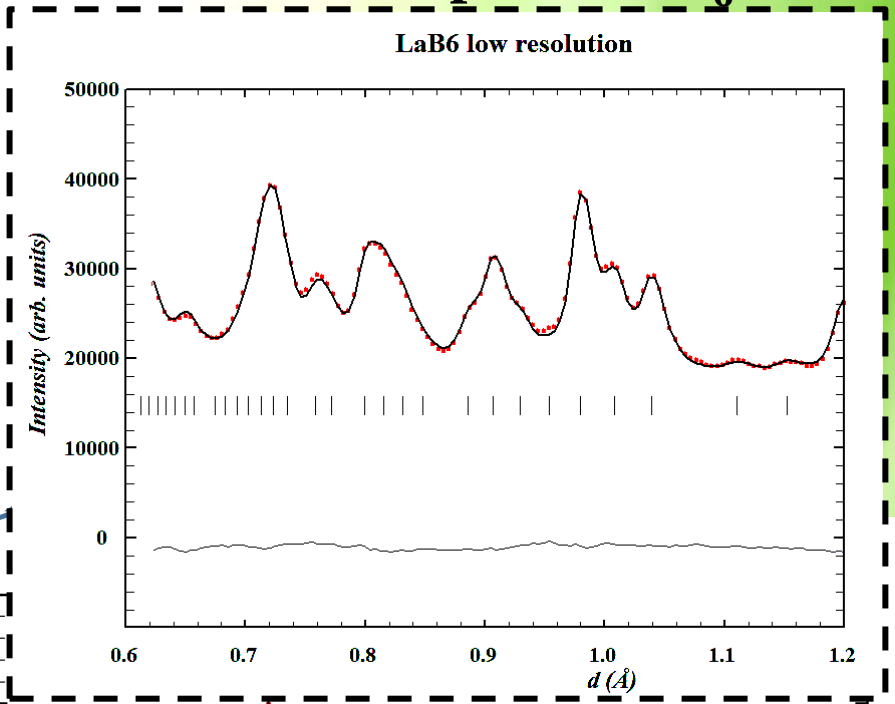
	Al_2O_3 low res.	Al_2O_3 high res.
Space group	R -3 c	
Zero	48±8	/
Dtt1	14585±4	10364.58±0.03
Sig0	9480±459	11±1
Sig 1	/	29.3±0.7

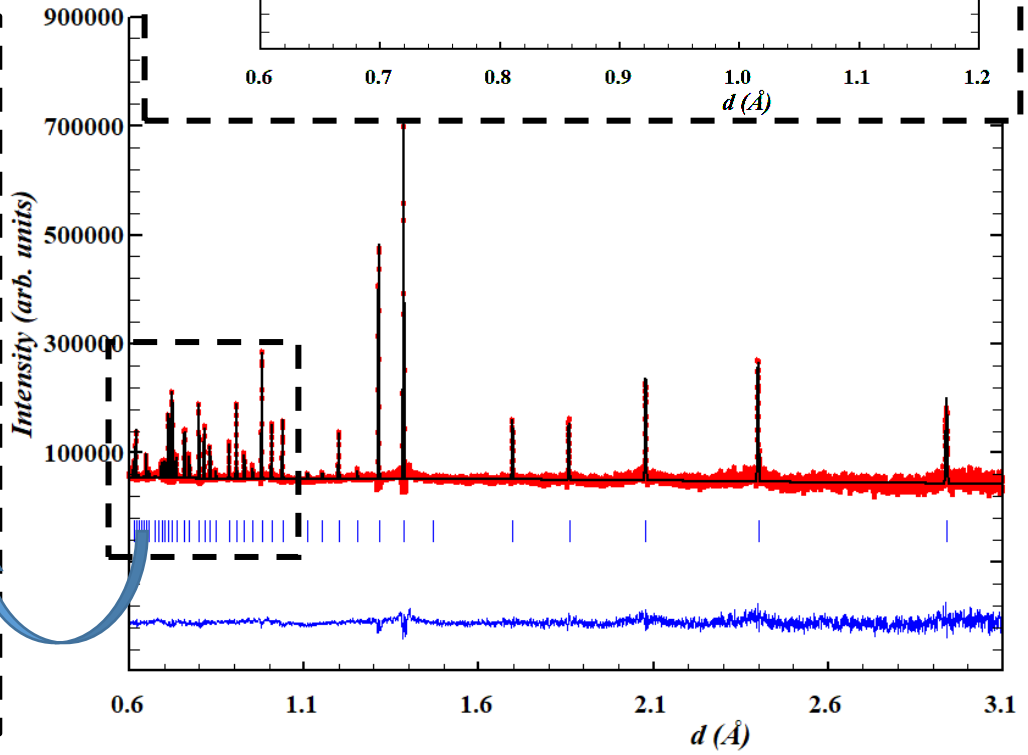


Standard sample – LaB_6

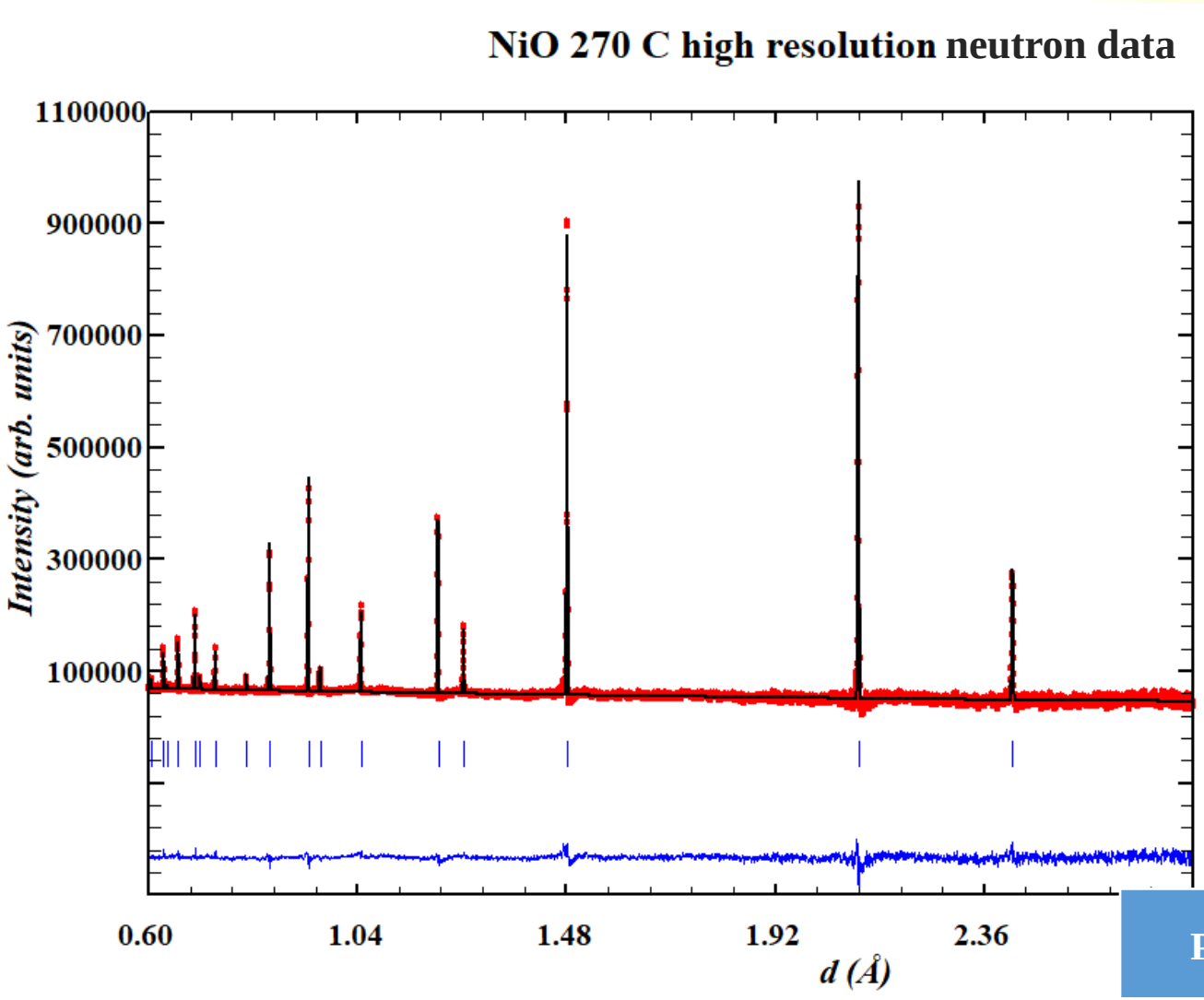


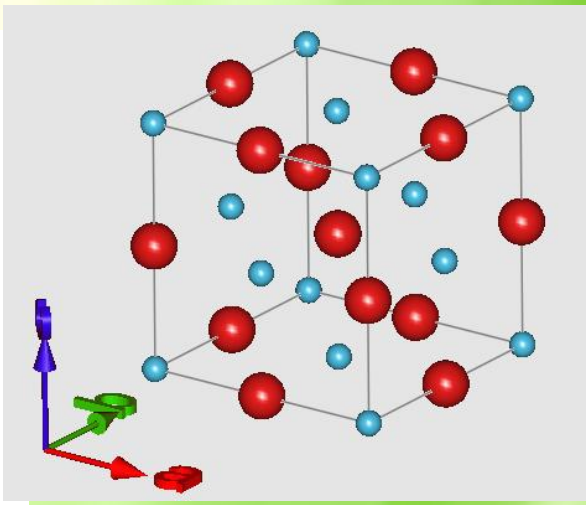






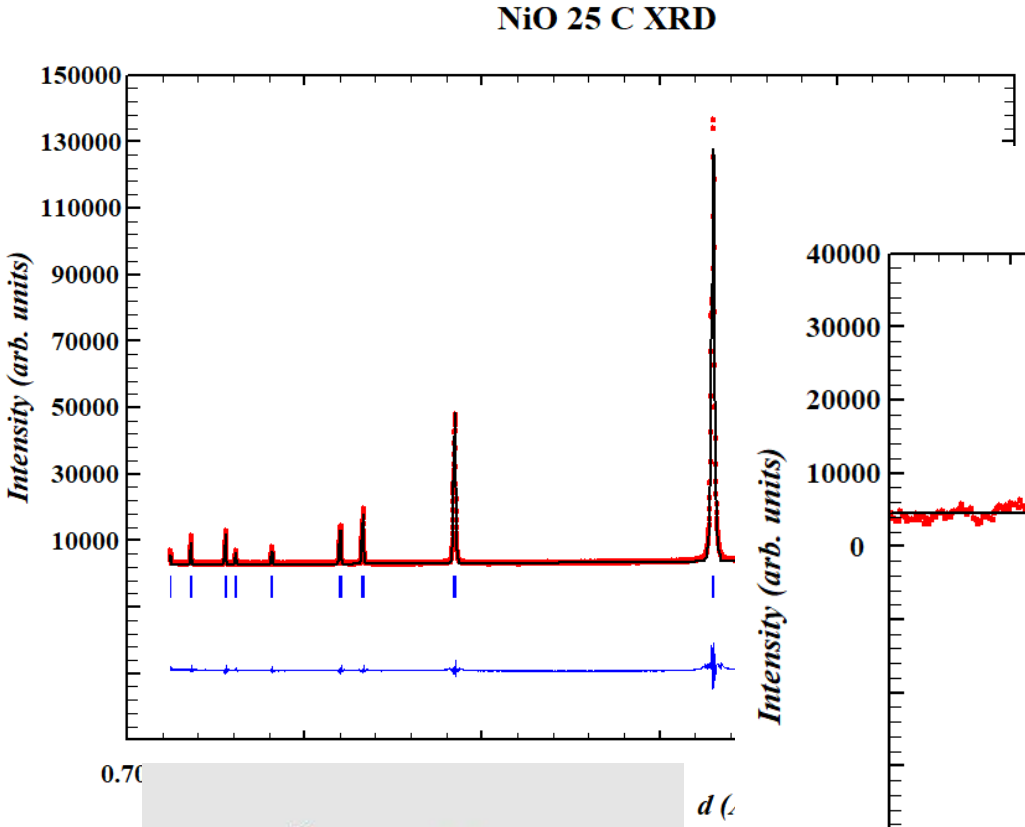
Sample – NiO at 270 C

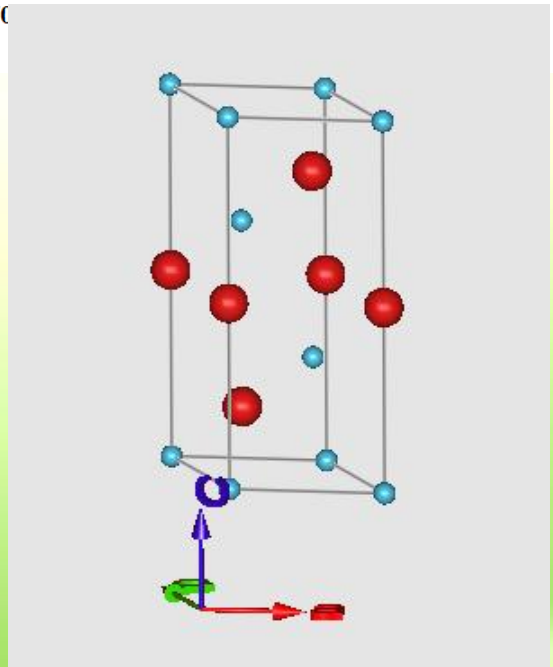


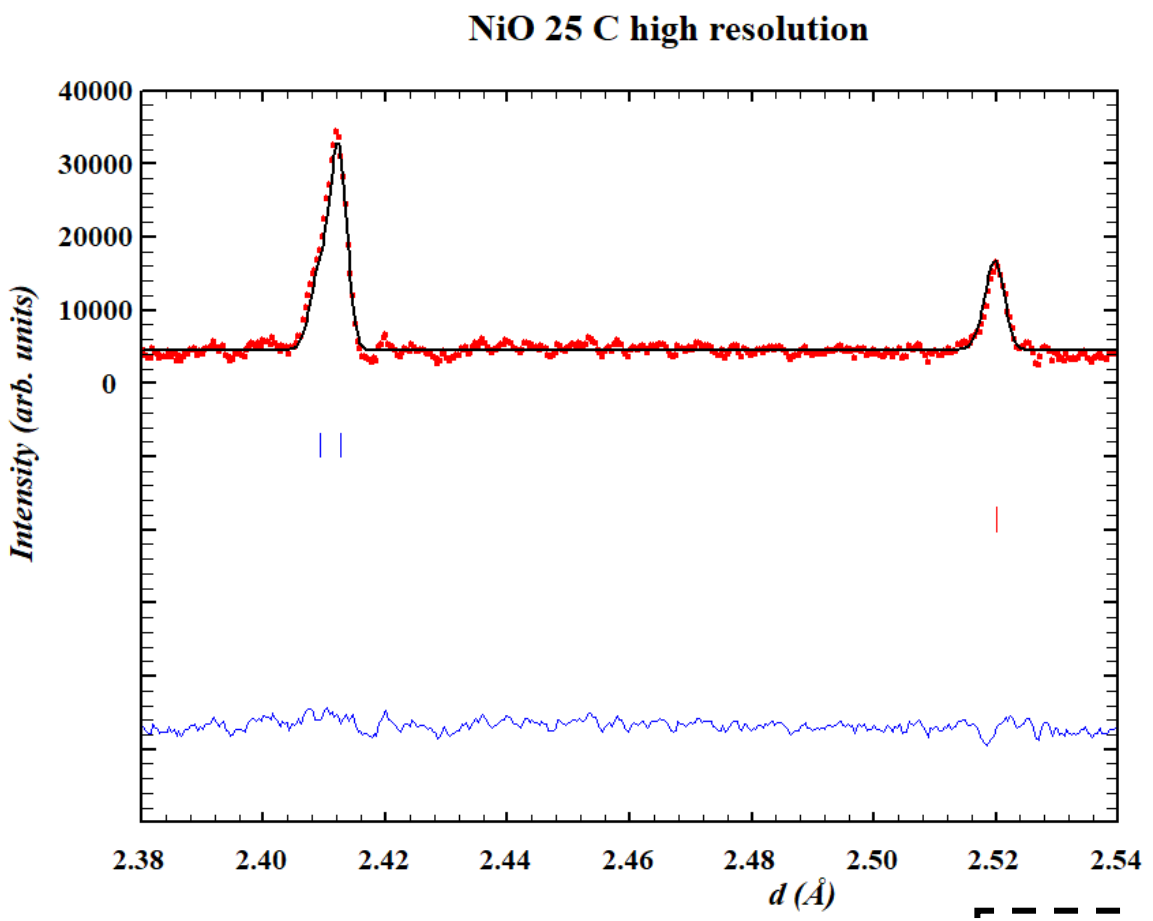


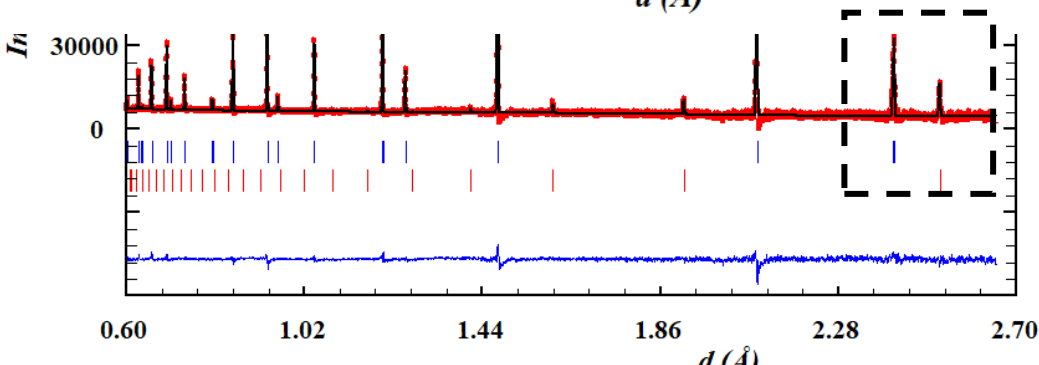
Parameters	Calculated value
Space group	F m 3 m
Unit cell parameters, a (Å)	4.1916 ± 0.0002

Sample – NiO at 25 C









	NiO low res. 25 C	NiO (XRD) 25 C	NiO high res. 25 C
Space group	R -3 m		
Unit cell parameters, a (Å)	2.95277 ± 0.00011	2.95470 ± 0.00008	2.95530 ± 0.00011
Unit cell parameters, c (Å)	7.22402 ± 0.00206	7.23017 ± 0.00026	7.227742 ± 0.00018
Magnetic moment	1.696 ± 0,06	NO	2.044 ± 0.05

$$a_r \approx \frac{a_c}{\sqrt{2}} \qquad \alpha \approx 60.07$$

Thank you for your attention!

Basic Parameters

Neutron beam cross-section at sample position	15 × 100 mm
Moderator - sample distance	~ 29.6 m
Chopper - sample distance	21.14 m
Fourier-chopper (disk-type)	Al-alloy
- outer diameter	540 mm
- slit width, number of slits	0.7 mm, 1024
- max speed of rotation	6000 rpm
- max modulation frequency	102.4 kHz
- effective pulse width	≈ 10 μs
Main detectors at 2θ = 90° and 2θ =152°	^{6}Li, time-focusing
Detector for large d_{hkl}	^{3}He, PSD, Δx ≈ 1.8 ммм, 2θ ≈ 30°
Aperture of the main detectors:	0.16 sr (2θ = 152°), 0.04 sr (2θ = 90°)
Wavelength range	0.9 - 8 Å
d_{hkl} range;	
- high resolution	0.7 - 4 Å
- medium resolution	1 - 16 Å
Neutron flux at sample position	1.3×10^{7} n/cm^2/s
Standard sample volume	~ 1 cm^3
Resolution (Δd/d) for 2θ = 152°, d = 2 Å	~ 0.001

$$\frac{\Delta d}{d} = 0.1\%$$