International students practice 2018
Stage 3
JINR, Dubna

Joint Institute for Nuclear Research
Frank Laboratory of Neutron Physics

Precision investigation of modern crystalline material $NbC_{0.96}$ by neutron and X-ray diffraction

Students:

Mohamed Asran Mohamed Hassan

Physics Department, Faculty of Science, Sohag University, Egypt

Lkhagvajav Sarantuya

Department of Material Science, Institute of Physics and Technology, Mongolian Academy of Science, Mongolia

Pantić Tijana

Laboratory of Physics, Vinča Institute of Nuclear Sciences, Serbia

Supervisors:

Bobrikov Ivan, Kuznetsov Vitalii, Sumnikov Sergei

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Overview

Part 1:

Introduction to diffraction

Monochromatic beams vs. Time of Flight

Neutron vs. X-ray Diffraction

Part 2:

Neutron diffraction investigation of $NbC_{0.96}$

Part 3:

X-ray diffraction investigation of $NbC_{0.96}$

Aim of the project

To increase our knowledge of diffraction methods

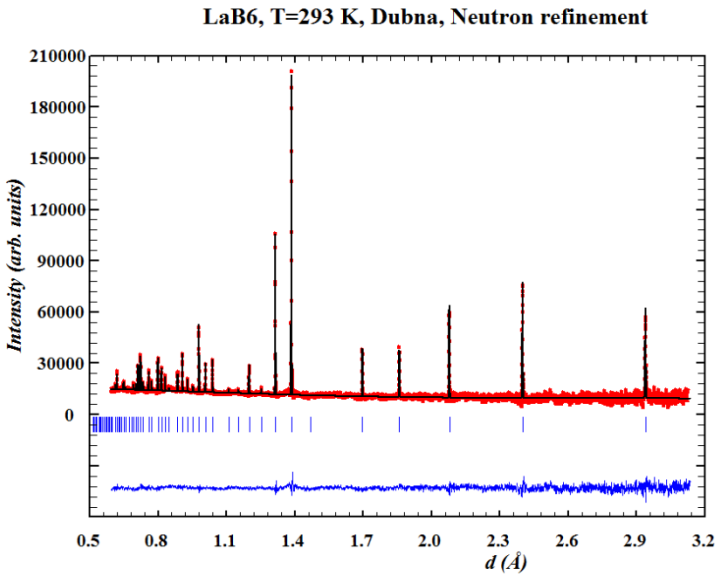
Investigation of a real sample

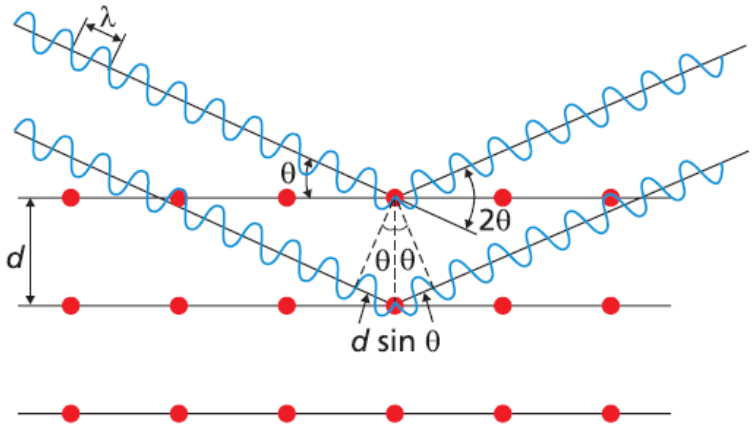
To get an experience with Rietveld refinement

To get an experience with Williamson-Hall method

PART-I
Introduction to Diffraction

Introduction to Diffraction





Diffraction methods are the most important approach to the analysis of crystalline solids

X-ray | electron | neutron

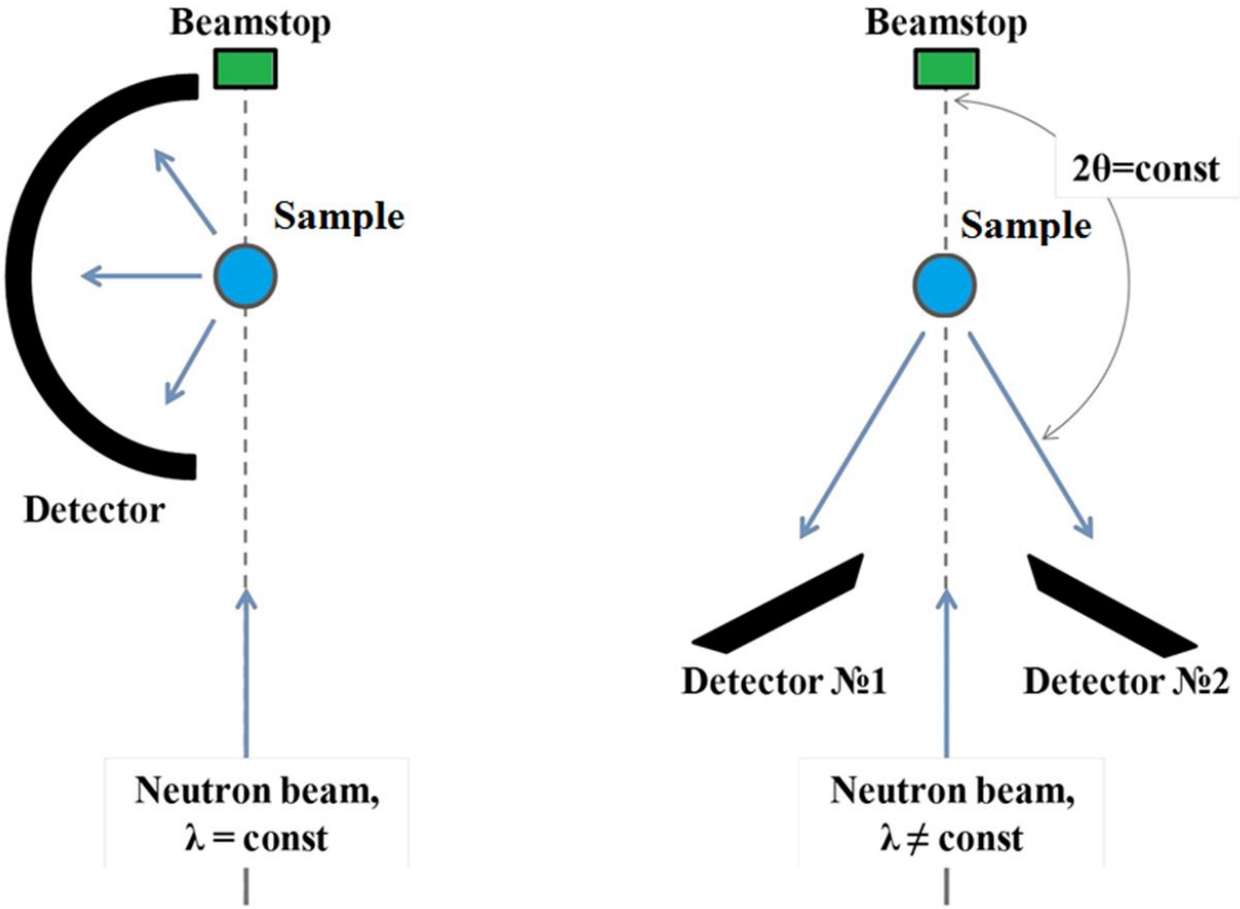
What can we obtain from the diffraction experiment?

Reflection	depends on	main information
Position	- periodic arrangement of the atoms/molecules - wavelength used	- qualitative phase analysis - lattice parameters - residual stress
Intensity	- crystal structure - wavelength used - sample preparation	- quantitative phase analysis - crystal structure - preferred orientation/texture
Profile	- lattice distortions	- micro-strain - crystallite sizes

Braggs law

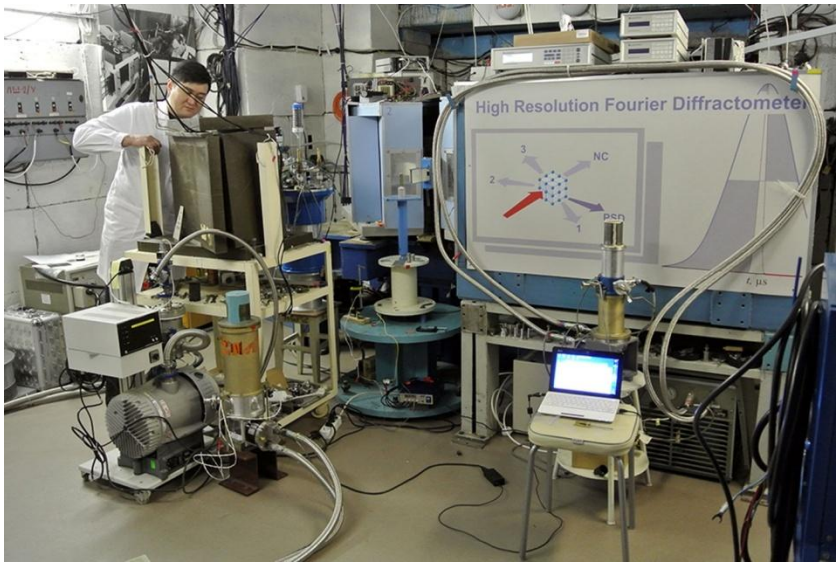
$$n\lambda = 2d\sin\theta$$

Monochromatic Beams vs. Time of Flight (TOF)



$$n\lambda = 2d\,sin\theta$$

Neutron vs. X-ray Diffraction



- scattered by magnetic moments
- neutrons interact with nucleus
- scattering power independent of 2θ
- lower absorption
- large amounts of sample needed
- light elements can be seen
- low availability (nuclear reactor)

- insensitive to magnetic moments
- X-ray photons interact with electrons
- scattering power falls off with 2θ
- stronger absorption
- lower amounts of sample needed
- light elements hard to detect
- high availability (lab instrument)

PART-II
Neutron diffraction (ND) investigation of $NbC_{0.96}$

Niobium Carbide

Sample used $NbC_{0.96}$

Milling times:
5, 10 and 15 h

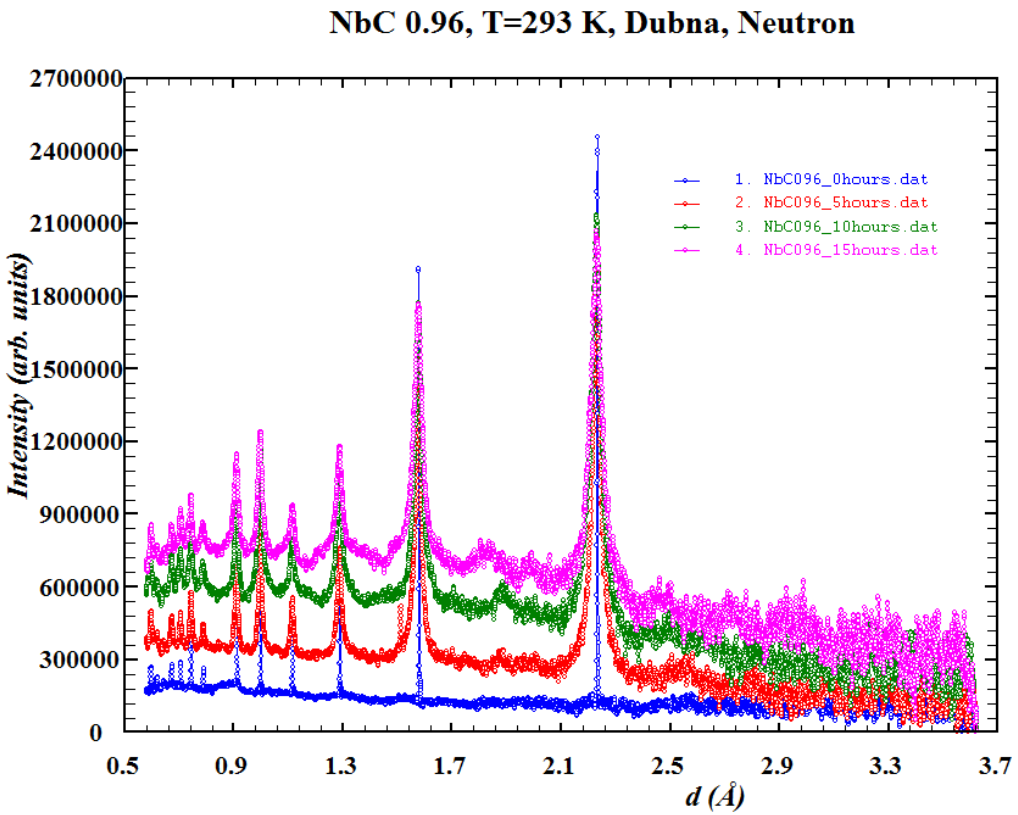
Properties

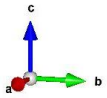
- ceramic refractory material
- extreme hardness
- highly corrosion resistant
- high melting point

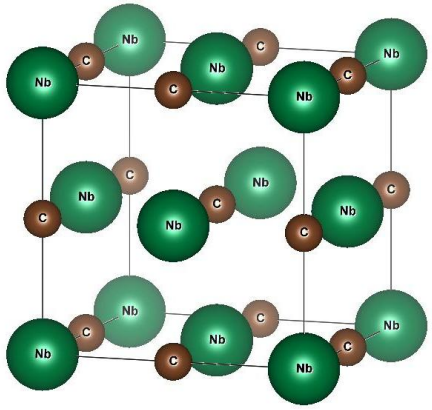
Application

- metallurgy industry
- aerospace industry
- catalysis

Neutron diffraction investigation of $NbC_{0.96}$ HRFD



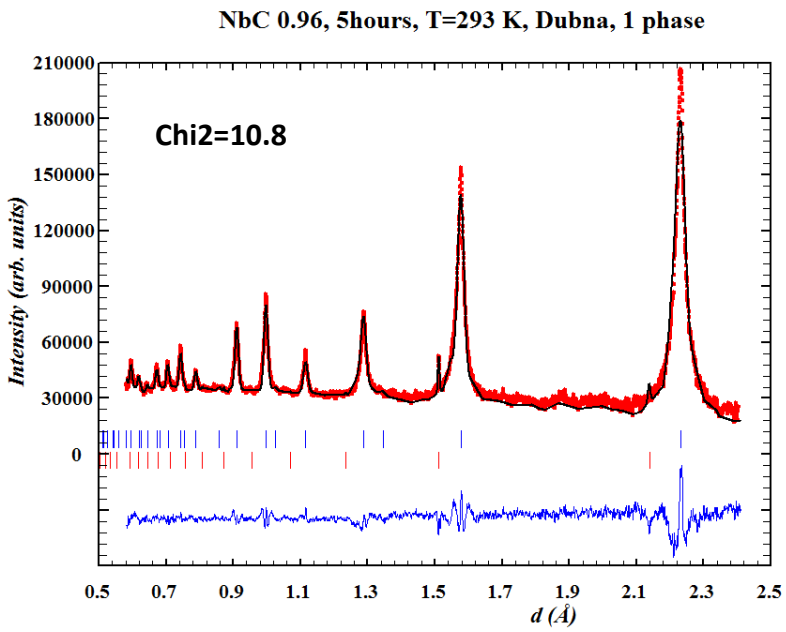


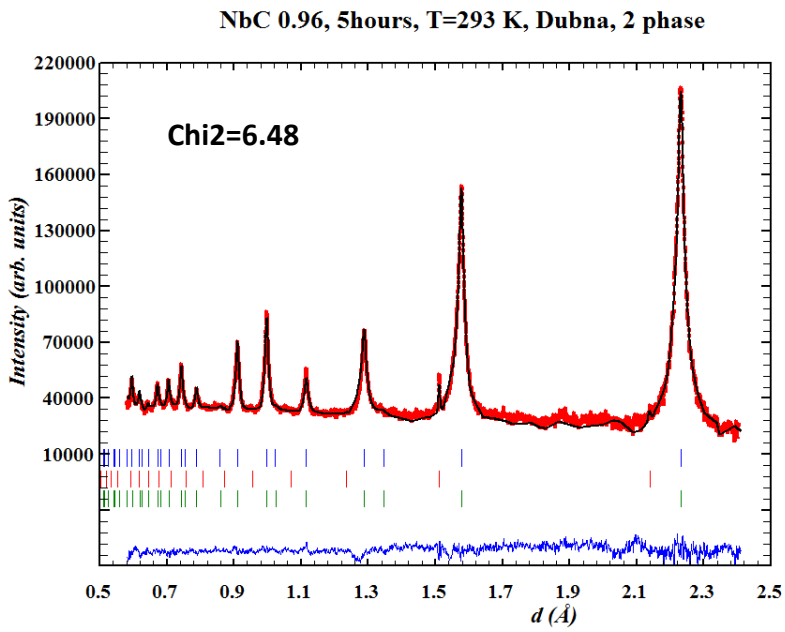


NbC (Niobium Carbide) powder

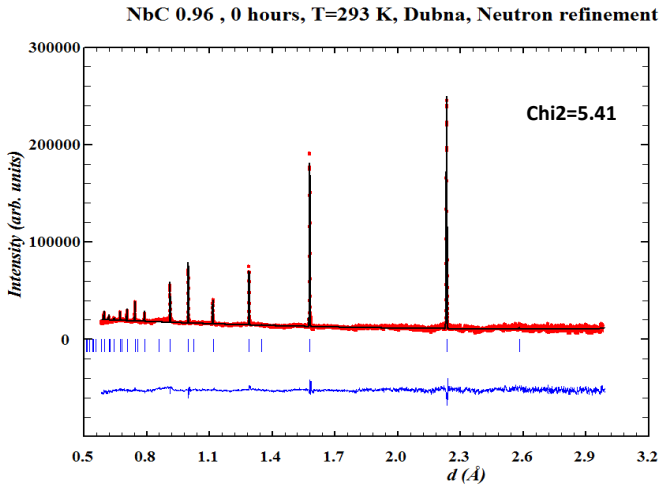
- NbC_x , x=0.96
- HRFD (high-resolution Fourier diffractometer), IBR-2
- Face-centered cubic symmetry (space group Fm3m)

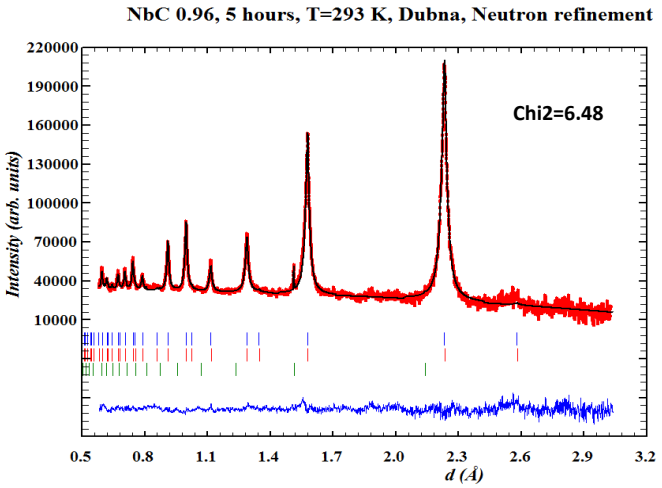
Neutron spectra refinement

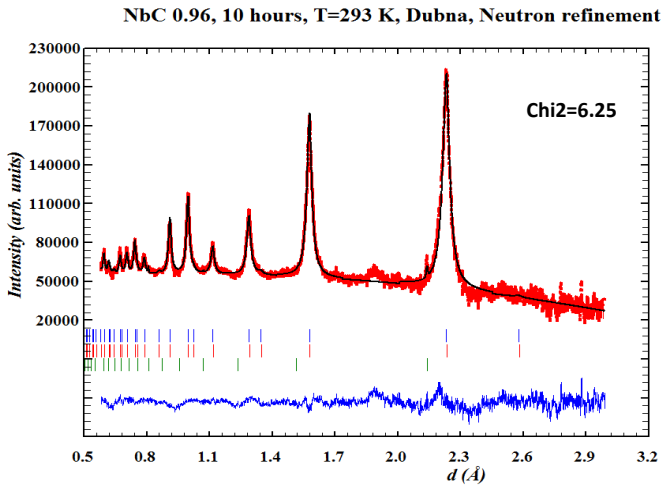


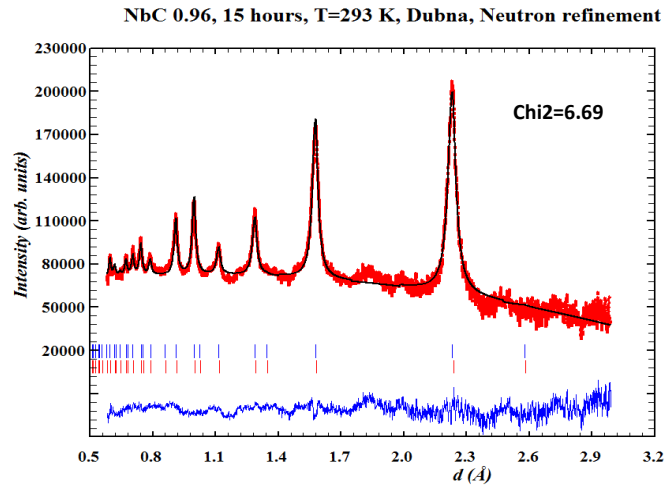


Neutron spectra refinement



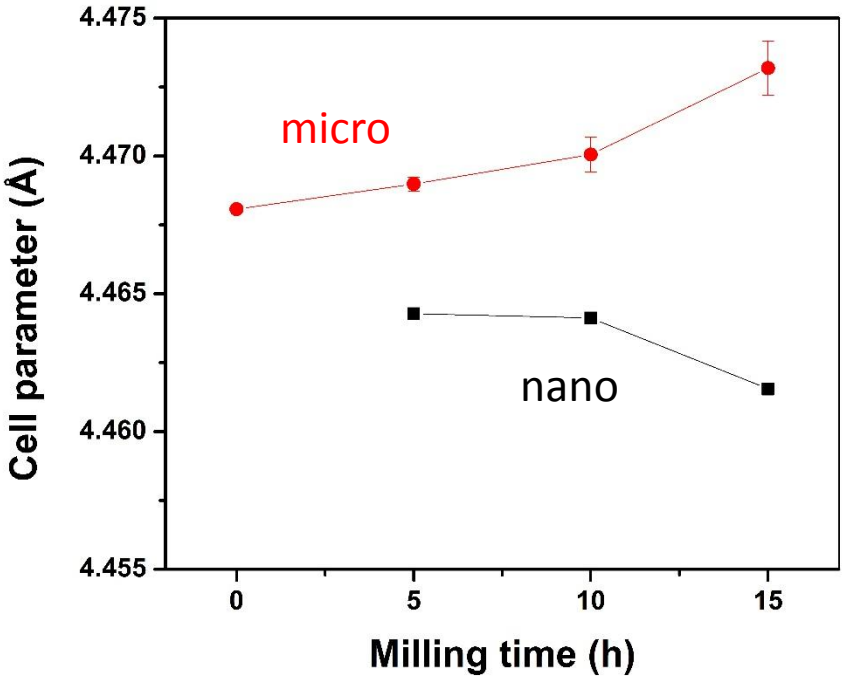




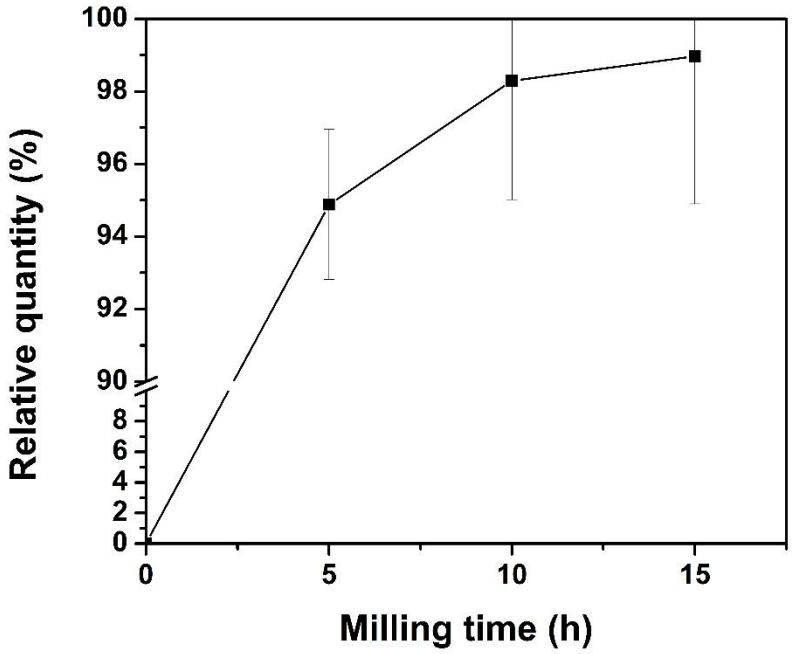


Results of Neutron spectra refinement

Dependence of the lattice parameters vs. milling time



Relative quantity of $NbC_{0.96}$ nano phase



Analysis of the widths of the NbC diffraction peaks

- Williamson-Hall method

$$W^2 = C_1 + C_2 d^2 + C_3 d^2 + C_4 d^4 \quad (1)$$

W- FWHM obtained from the single peak fitting of the diffraction data

d- peak position

C_1, C_2- characteristics of the diffractometer

$$C_3 = 4\boldsymbol{\varepsilon}^2 \quad (2)$$

$\boldsymbol{\varepsilon}$ - microstrain

$$C_4 = \left(\frac{1}{L}\right)^2 \quad (3)$$

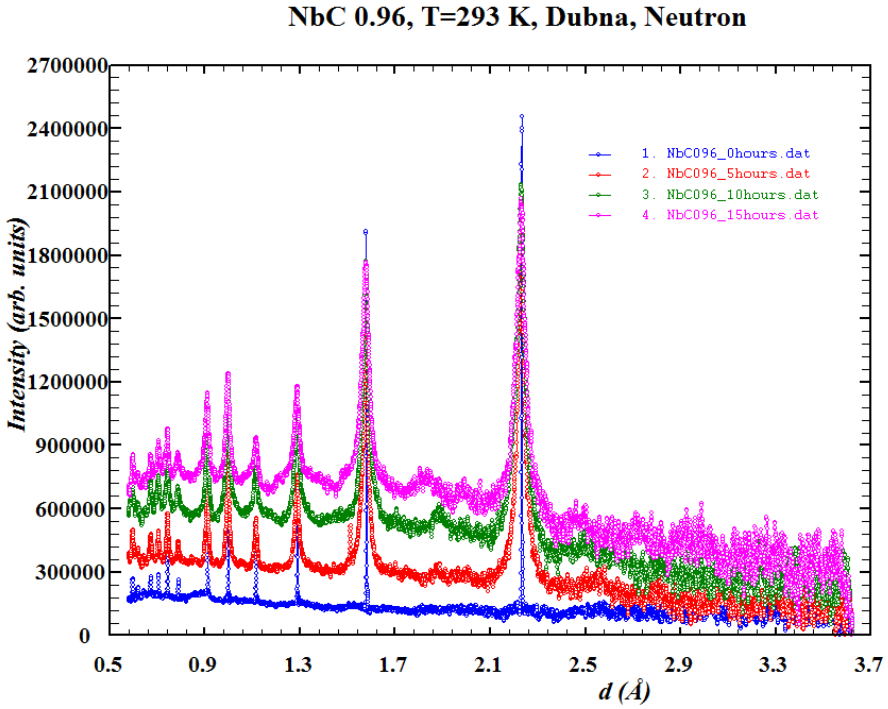
L- size of the grains

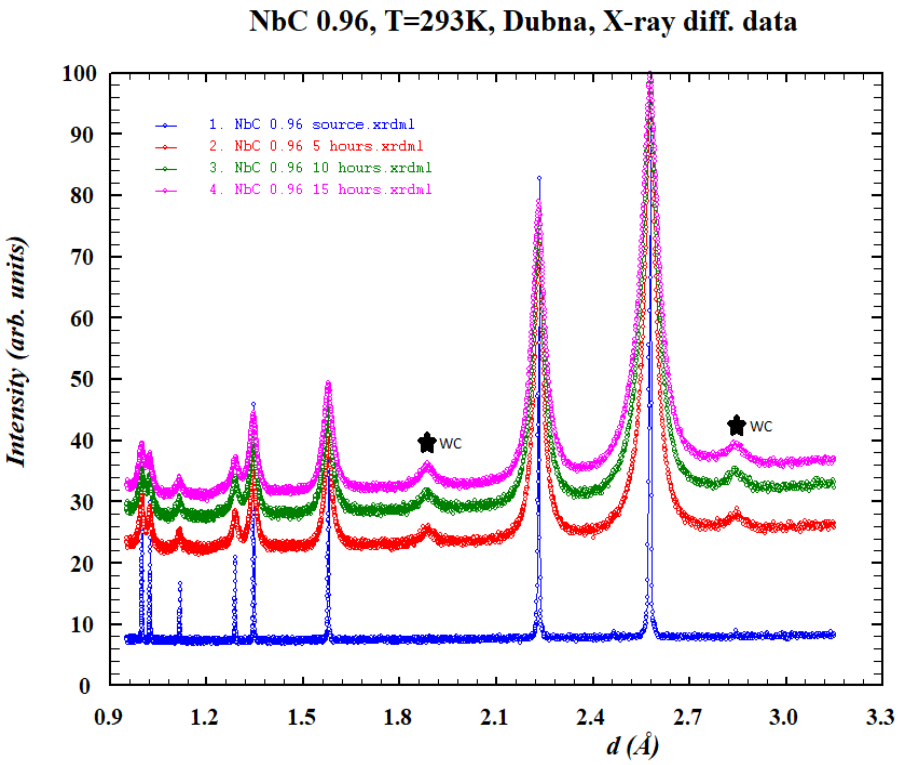
Average crystallite size - 20 nm

Average microstrain – 1.5 %

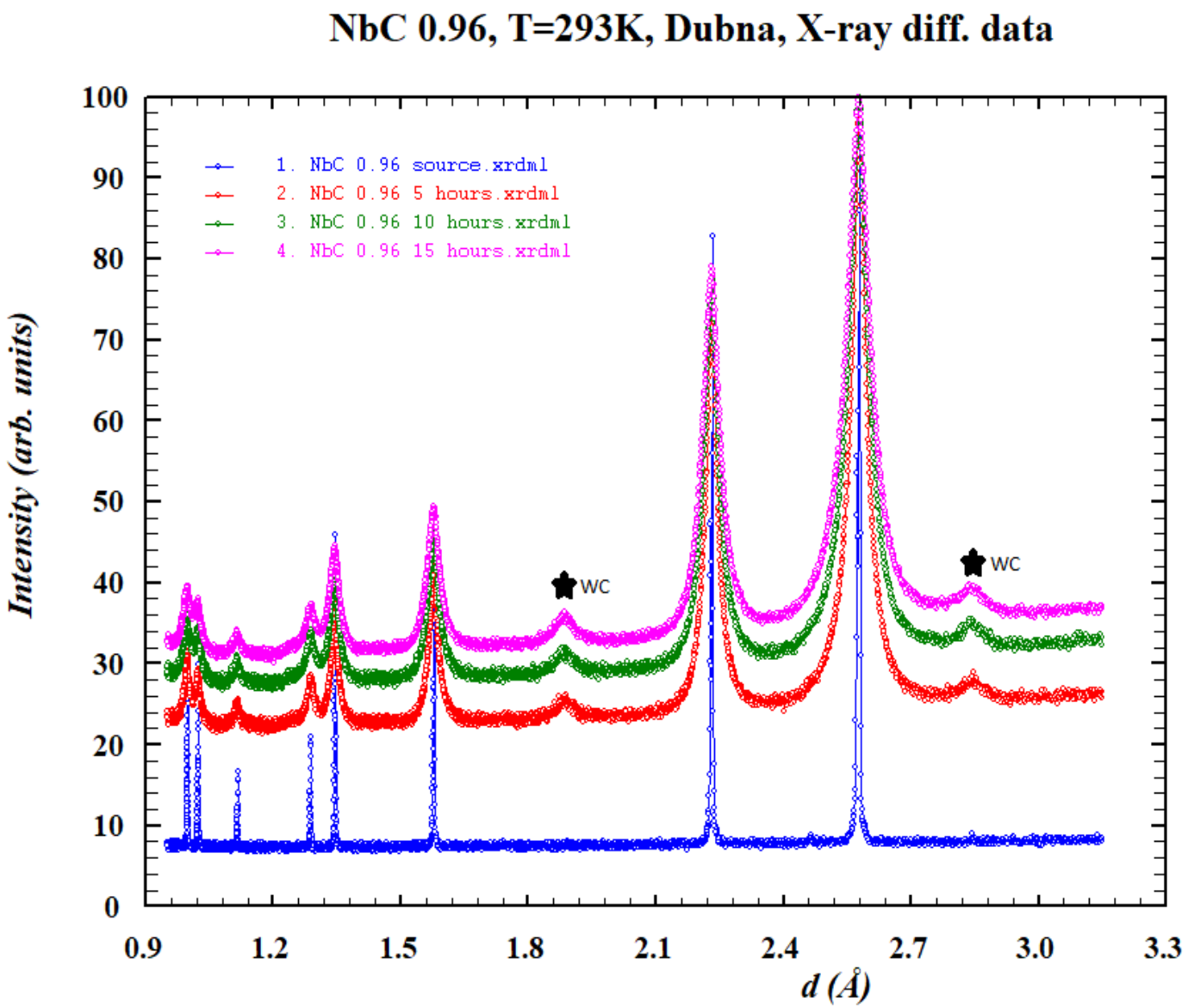
PART-III
X-ray diffraction (XRD) investigation of $NbC_{0.96}$

XRD data vs. ND data

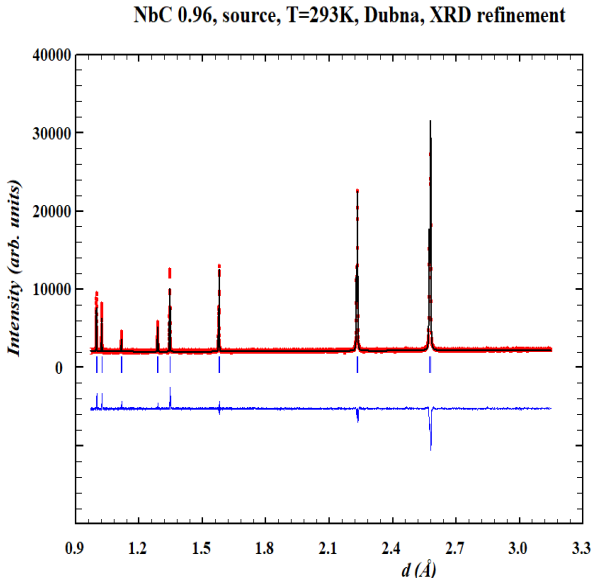


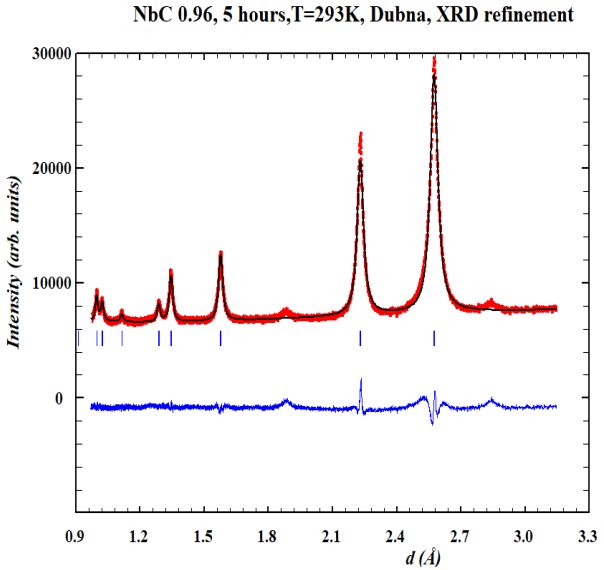


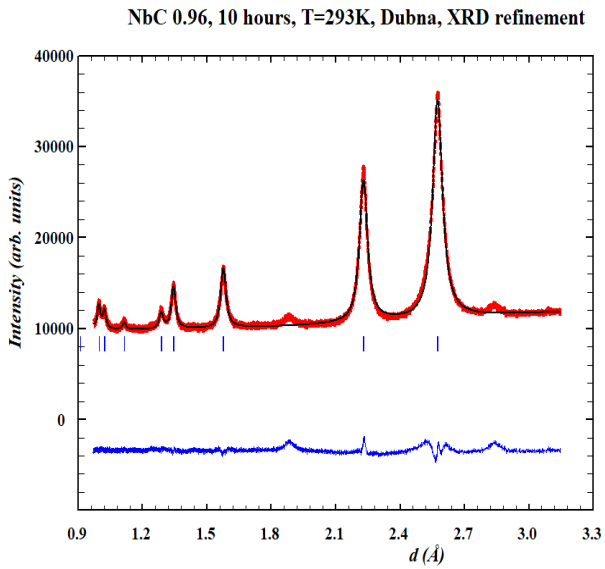
XRD investigation of $NbC_{0.96}$

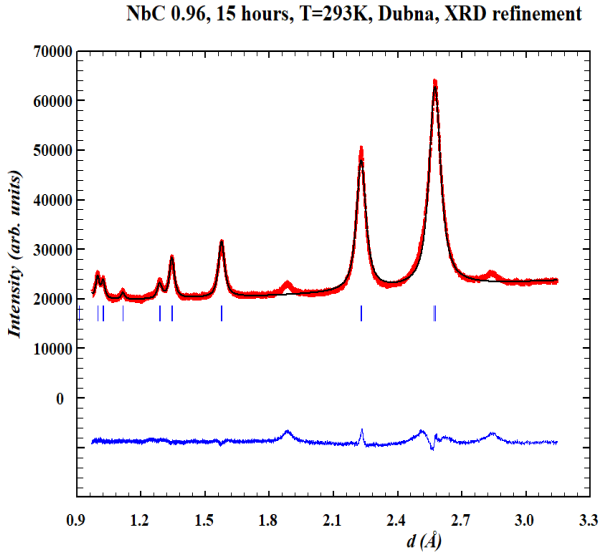


Refinement of $NbC_{0.96}$ XRD data

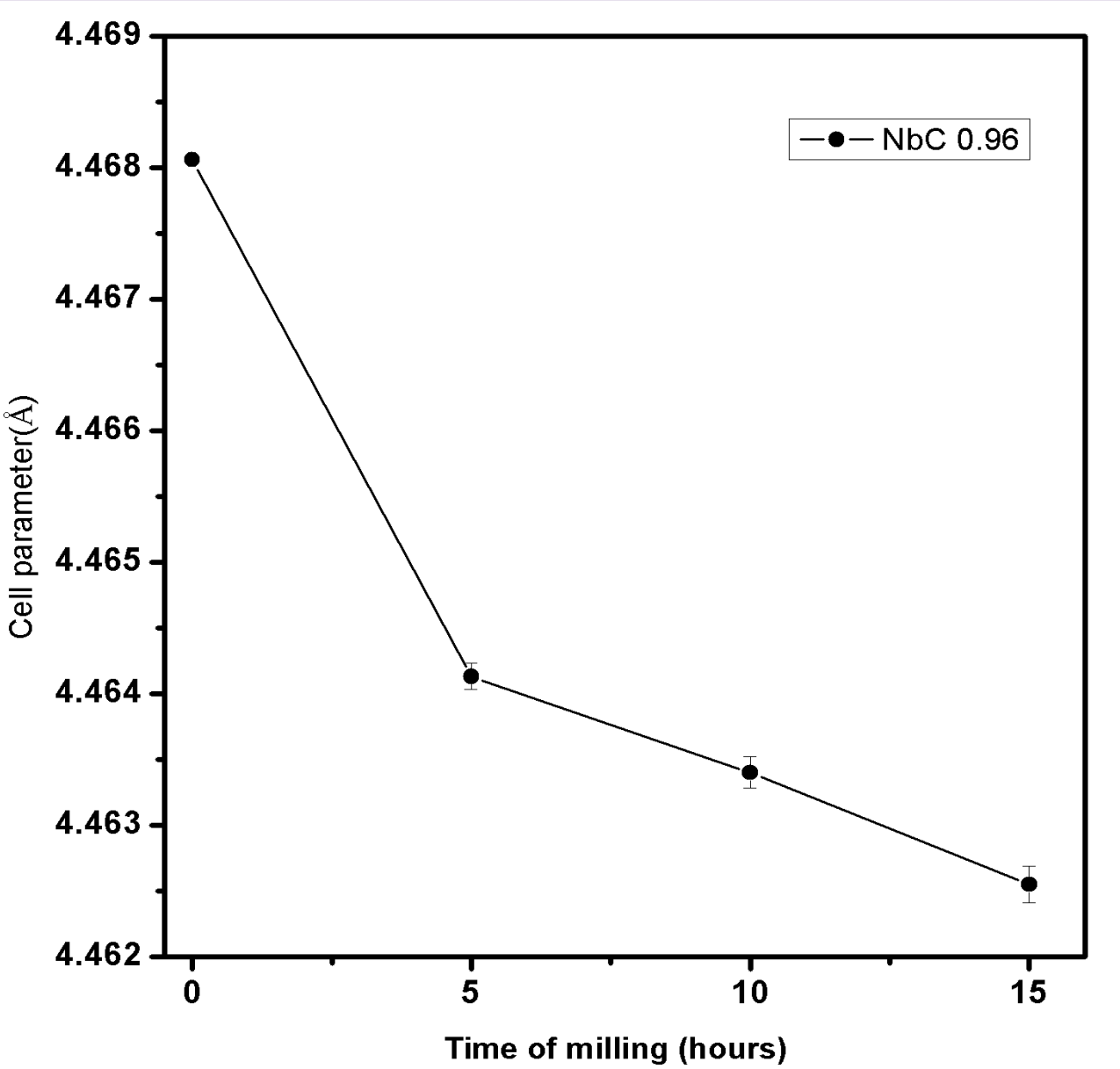








Cell parameter vs. Time of milling



Summary

- There is a difference in crystal structure and crystalline size between the source sample of $NbC_{0.96}$ and the milled samples.
- Using HRFD we were able to identify two fractions with different crystallite size in the sample, while we couldn't do it using our X-ray diffractometer.
- The crystallite size for nanofraction doesn't change within the error with increasing the milling time.

Thank you for your attention