Center of Accelerators
and Nuclear Analytical Methods
(CANAM)

Neutron Physics Laboratory (NPL)

Pavel Strunz, Jan Kučera

SAC meeting, November 24, 2015

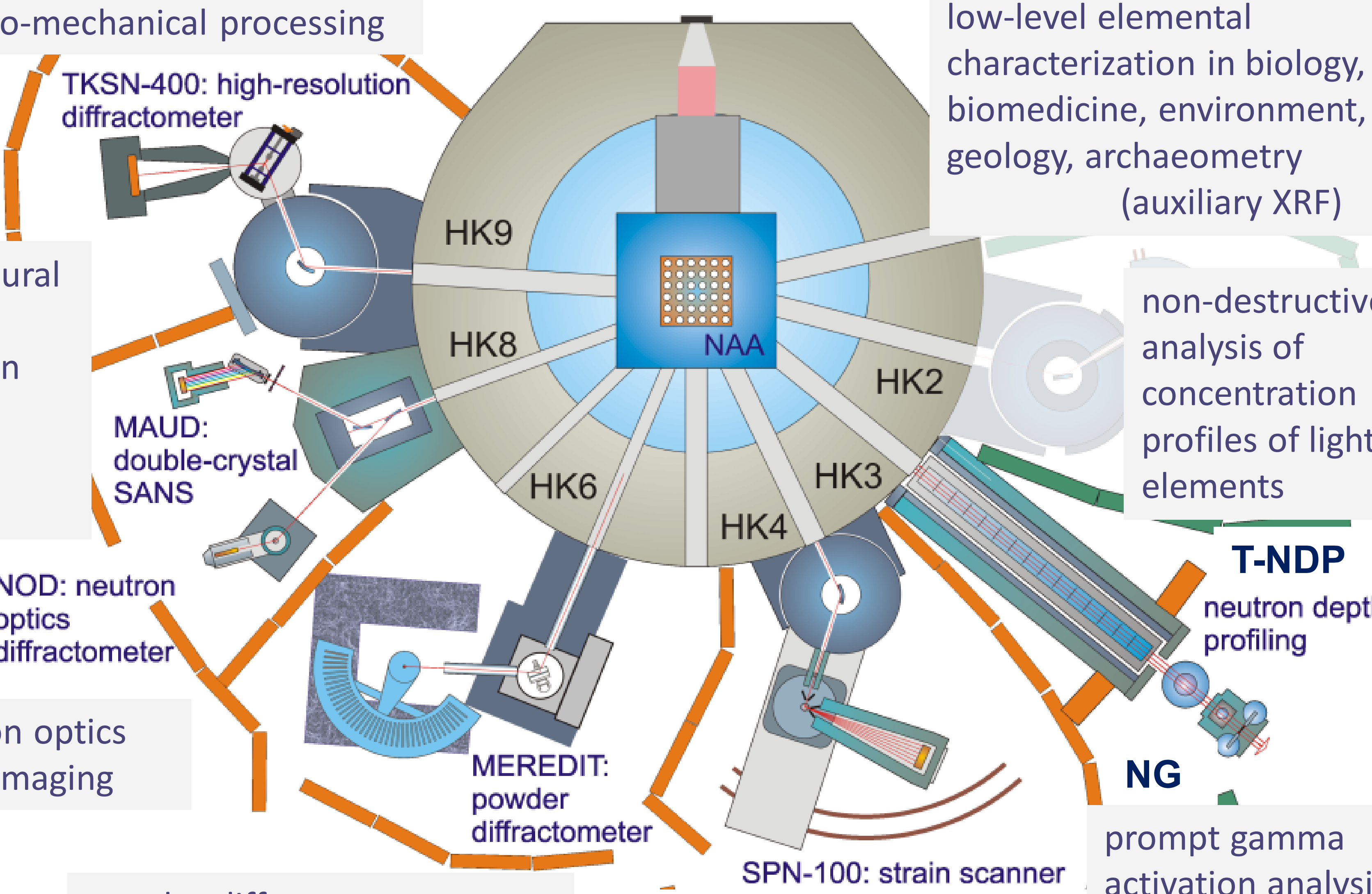
Outline

- NPL (reminder)
- technical development 2014 -2015
- technical outlook
- Examples of the experiments and results

NPL mission

- neutron-physics experiments according to the NPI research program (standard grant projects, Excellence project)
- providing the experimental facilities and research experience to external users in the **open access** mode

FACILITIES



macro- and microstrains in metals, in-situ thermo-mechanical processing

low-level elemental characterization in biology, biomedicine, environment, geology, archaeometry (auxiliary XRF)

micro-structural studies (precipitation in alloys, porosity in ceramics)

non-destructive analysis of concentration profiles of light elements

neutron optics tests, imaging

powder diffractometer – phase analysis (also magnetic), investigation of materials in-situ

macrostrain scanning in polycrystalline materials (e.g. welds)

prompt gamma activation analysis; photon–strength functions, nuclear structure

3 nuclear-analytical techniques

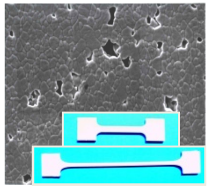
5 diffraction techniques

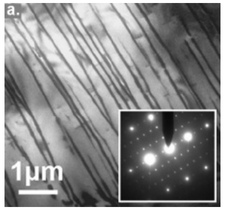
NPL facilities: Applications (generally):

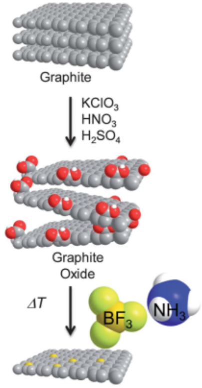
- materials research using neutron diffraction
- neutron activation analysis
- experiments in nuclear physics

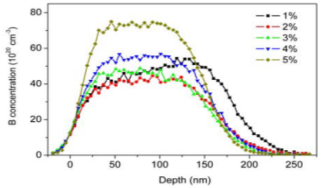
- **structure** (incl. magnetic) and **microstructure**, advanced metals and ceramics; micro- and macro-strains; porosity; **in-situ** thermo-mechanical processing; phase transformations at high- and low-temperatures; archaeological artifacts.

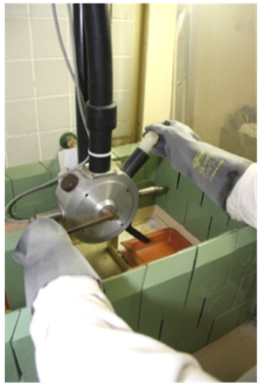
- Non-destructive analysis of concentration profiles of light elements; low-level **elemental characterization** in biology, biomedicine, environment, geology, metallurgy; prompt gamma activation analysis; nuclear structures.







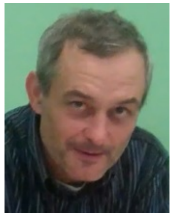




Instrument scientistsis

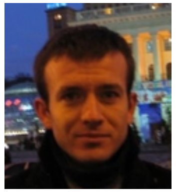
Prof. Jan Kučera
NAA

Jiří Vacík
HK3 / T-NDP



Ivo Tomandl
HK3 / NG

Přemysl Beran
HK6 / MEREDIT
Powder diffraction
structure analysis



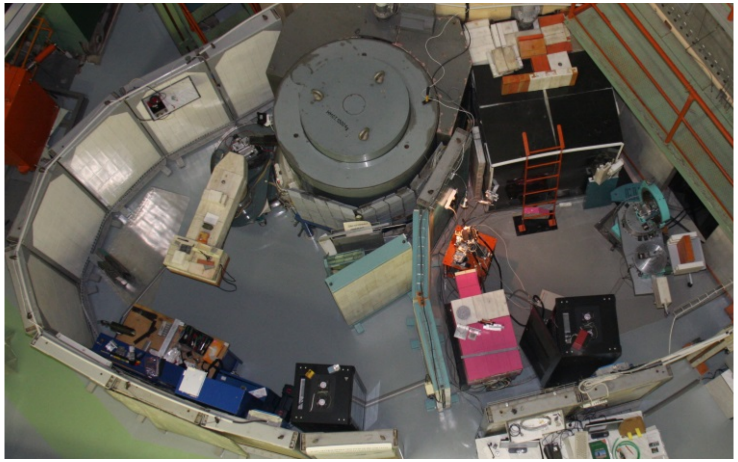
Vasyl Ryukhtin
HK8 / MAUD
Small-angle scattering

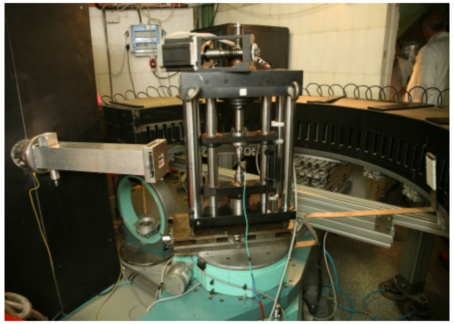
Pavol Mikula
HK8 / NOD
Neutron optics & applications

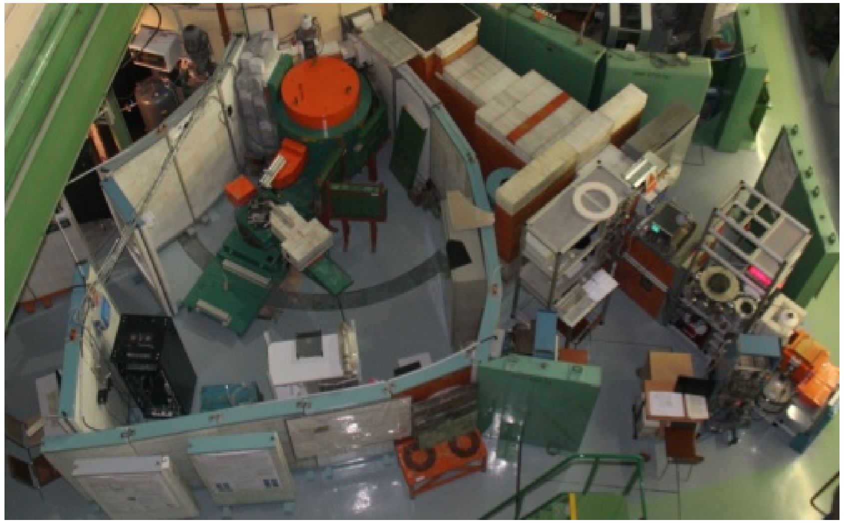
Charles Hervoches
SPN100
Residual strains

Jan Pilch
HK9 / TKSN400
Materials research
In-situ neutron diffraction

Open Access







- continuous and **fast** evaluation of proposals
- **support from in-house scientists** for external users (IR for each facility)
- eligible users from EU and associated states: **support within NMI3** project

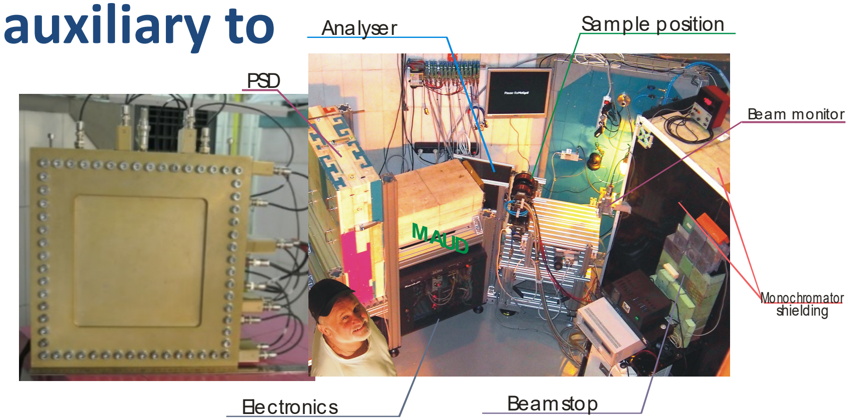
NMI3 (Integrated Infrastructure Initiative for Neutron Scattering and Muon Spectroscopy): European consortium of 18 partner organisations from 12 countries, including all major neutron-physics labs

Open access statistics since Sept. 2012 (≈ the start of the user portal):

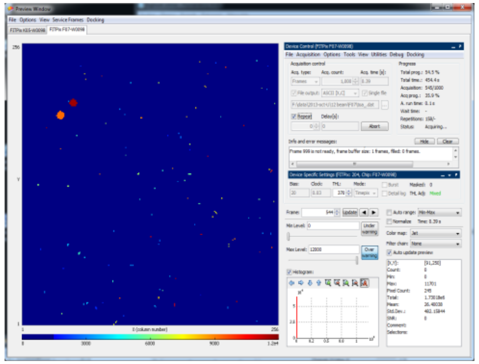
- **64** external NPL proposals accepted (**51** internal in the same period)
- **687** beamdays for external proposals (**698** beamdays for internal)
- **≈30** scientific papers **per year** (NPL, incl. internal experiments)

NPL - Facility development 2014 and 2015

X-Ray Fluorescence (XRF) for elemental analysis (auxiliary to NAA)

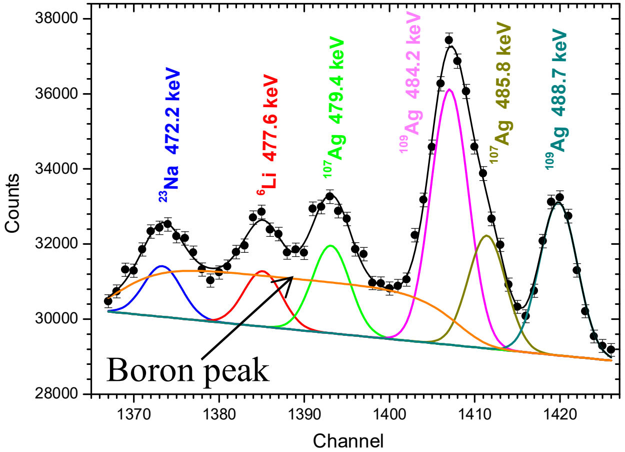


- Recent upgrades of facilities (2014 and 2015)
- 2D PSD and shielding at MAUD
- 2D PSD and shielding at SPN-100
- Multipixel detectors (TimePix) for 3D NDP
- motor-controller modernization
- separation of analyzer and sample table at MAUD
- …



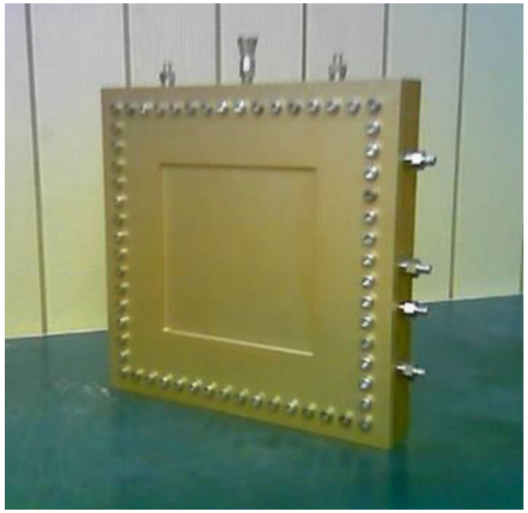
Other (auxiliary, software)

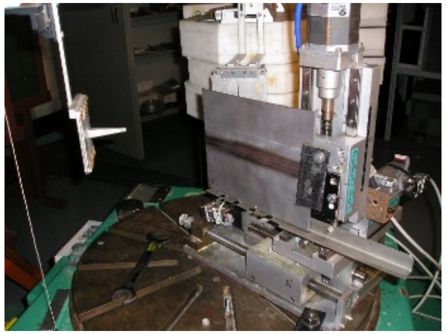
- NAA: Cryogenic mill
- NG: Developed software for correct and precise evaluation of Doppler broadened 478 keV peak (important for low signal-background ratio and interference with other gamma lines).

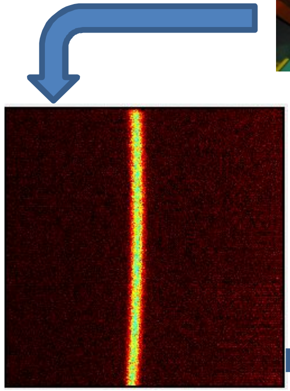


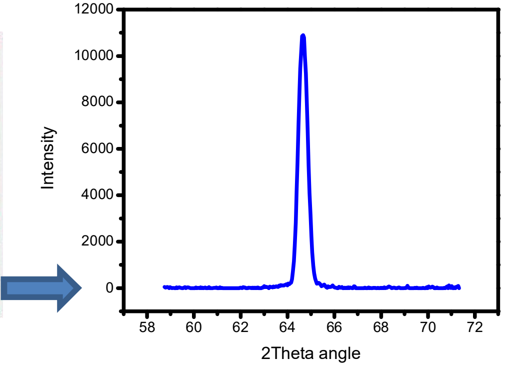
New 2D PSD detectors (SPN-100 and MAUD)

Detector type	3He, delay line
Neutron sensitive area	200x200 mm^2
Depth of detection volume	33 mm
Spatial resolution (FWHM)	2.5x2.5 mm^2
Efficiency	68% (for 1.8 Å neutrons)
Count rate	up to 500 kHz
Produced by	JINR Dubna

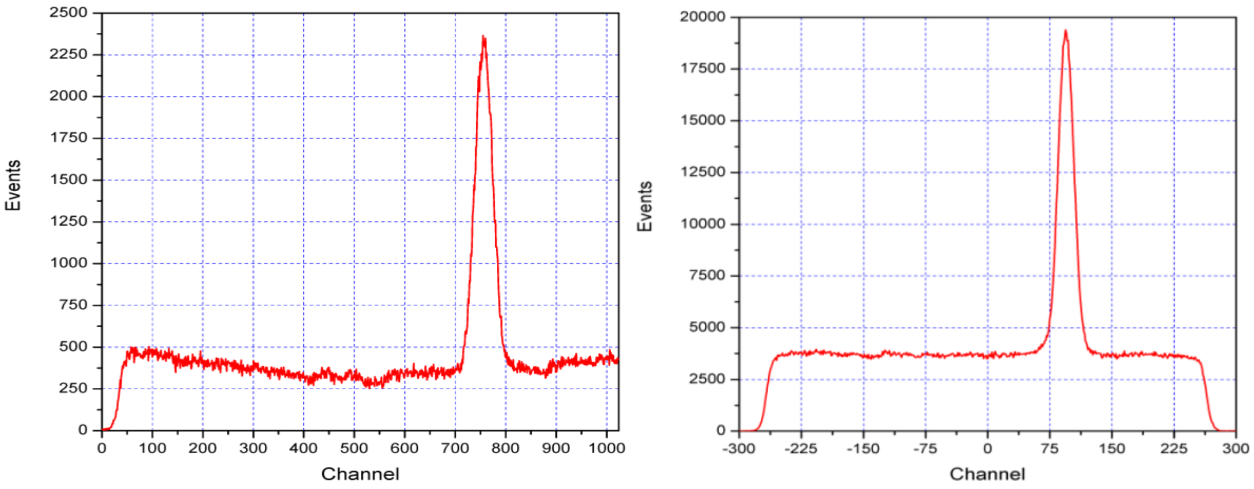








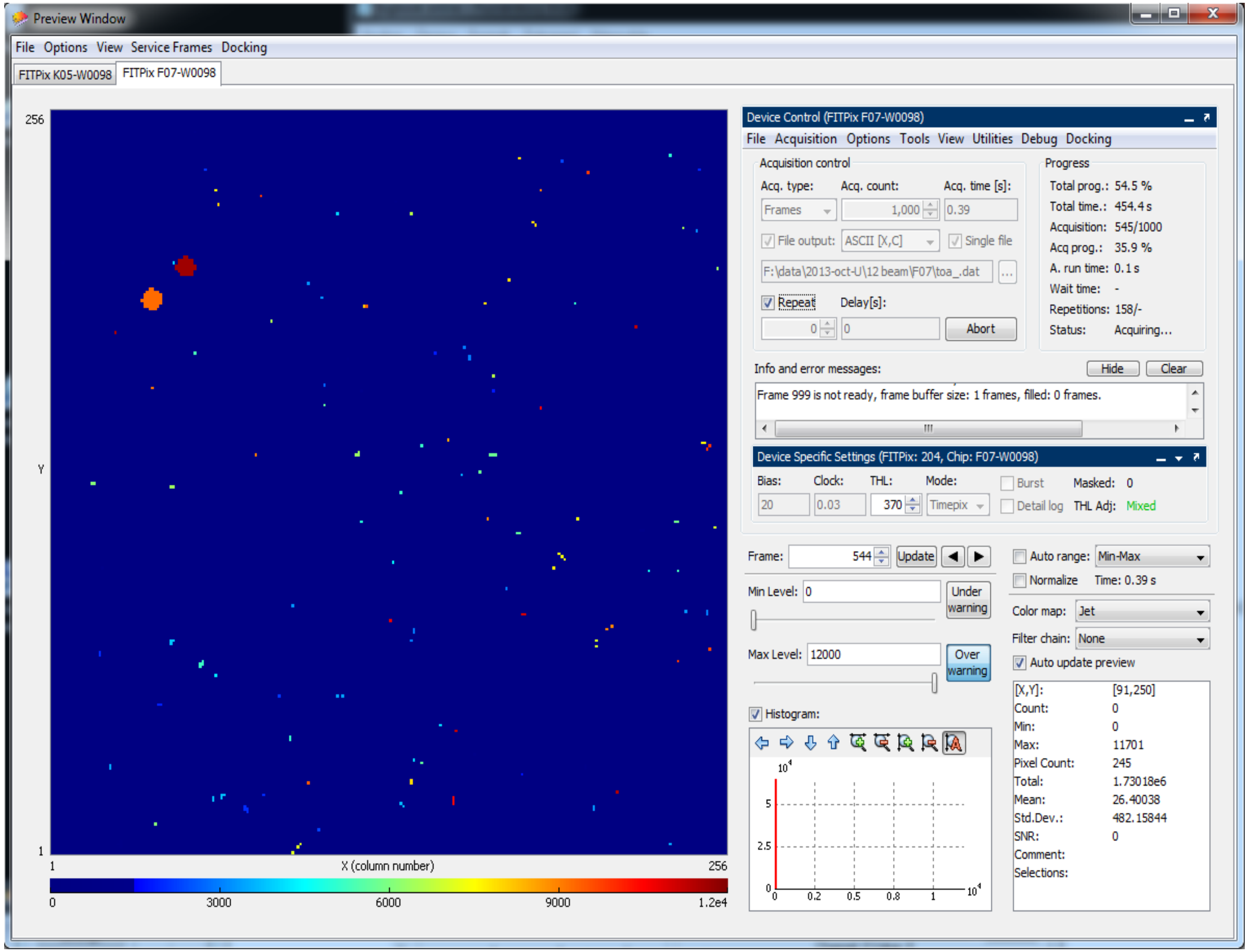
Detector image of ferrite (110) reflection (AISI 1008 steel sample), and related diffr. pattern



Peak intensity eight times higher due to the increased size of the sensitive region and higher efficiency

Multipixel detectors (TimePix) for 3D NDP

NDP equipped with TimePix detector which enables 3D reconstruction of concentration profile



Acq. type: Frames
Acq. count: 1,000
Acq. time [s]: 0.39
☑ File output: ASCII [X,C]
☑ Single file
F:\data\2013-oct-U\12 beam\F07\toa_.dat
☑ Repeat 0
Delay[s]: 0

Total prog.: 54.5 %
Total time.: 454.4 s
Acquisition: 545/1000
Acq prog.: 35.9 %
A. run time: 0.1 s
Wait time: -
Repetitions: 158/-
Status: Acquiring...

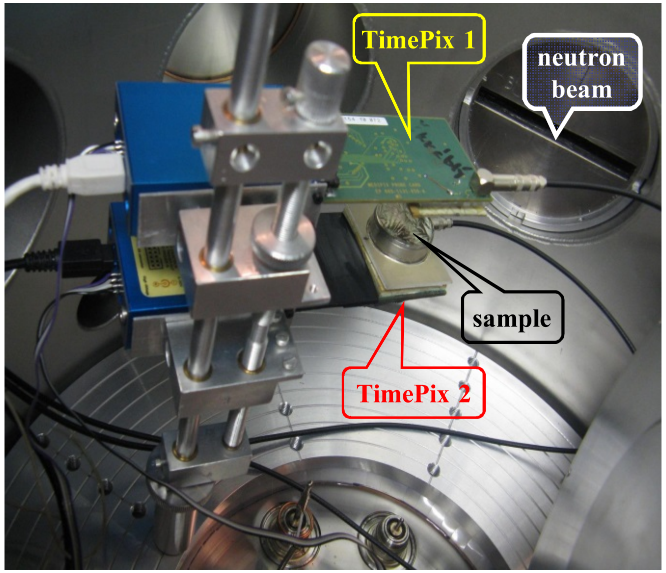
Info and error messages:
Frame 999 is not ready, frame buffer size: 1 frames, filled: 0 frames.

Device Specific Settings (FITPix: 204, Chip: F07-W0098)
Bias: 20
Clock: 0.03
THL: 370
Mode: Timepix
☐ Burst
☐ Detail log
Masked: 0
THL Adj: Mixed

Frame: 544
Min Level: 0
Max Level: 12000
☑ Histogram:
☐ Auto range: Min-Max
☐ Normalize
Time: 0.39 s
Color map: Jet
Filter chain: None
☑ Auto update preview

[X,Y]: [91,250]
Count: 0
Min: 0
Max: 11701
Pixel Count: 245
Total: 1.73018e6
Mean: 26.40038
Std.Dev.: 482.15844
SNR: 0
Comment:
Selections:

- 256x256 pixels, size 55 µm
- Signal pulse processing electronics provide simultaneously fast and noise-free image

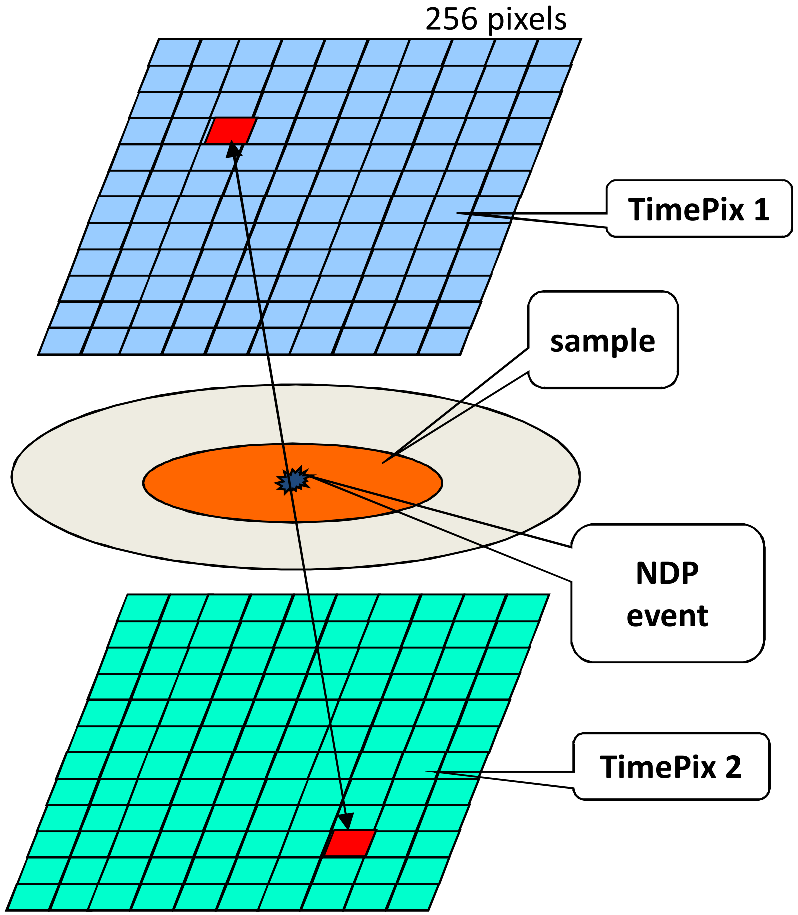


- *D. Vavrik, M. Holik, J. Jakubek, M. Jakubek, V. Kraus, F. Krejci, P. Soukup, D. Turecek, J. Vacik and J. Zemlicka, Journal of Instrumentation 9 (2014) C06006*

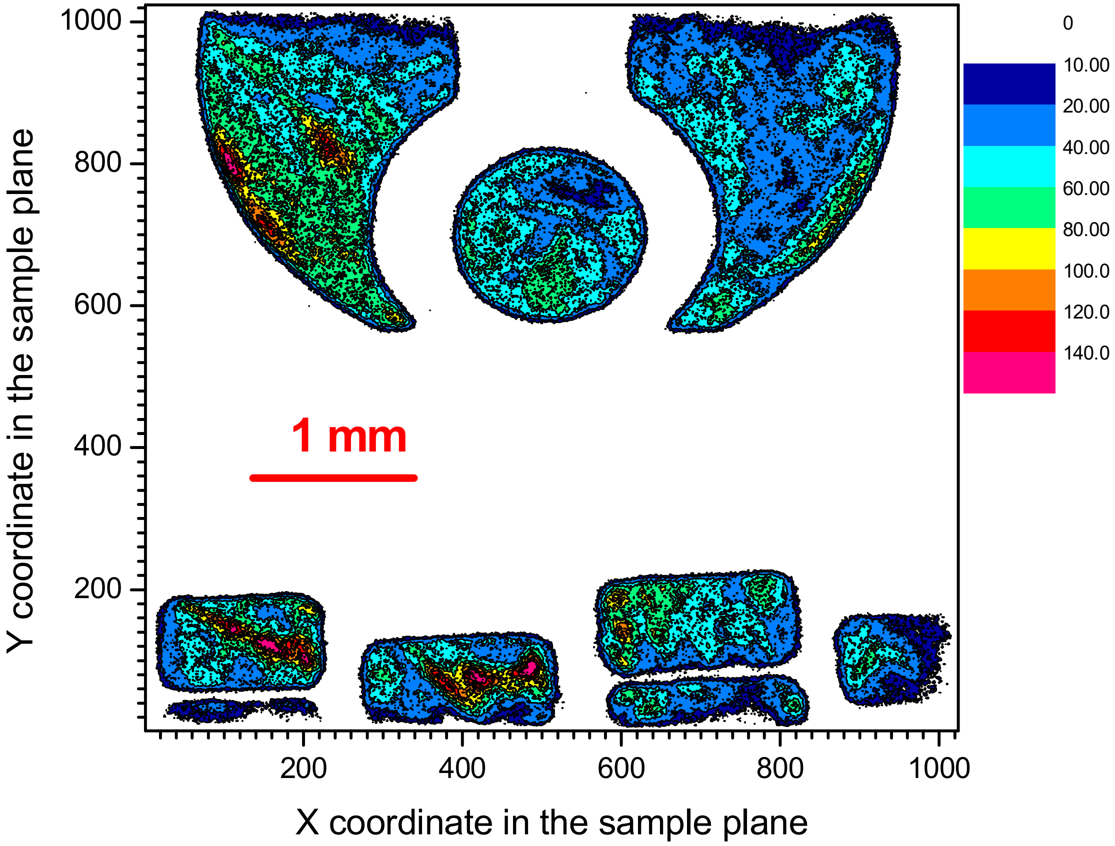
Large angle spectroscopy with TimePix detectors (NDP)

- Another option how to achieve 3D reconstruction of the conc. profiles:
- detection of coincidence events
- => Lateral resolution improvement

Principle two TimePix detectors in coincidence



Test: 2D Lateral reconstruction of the thin ^{6}Li structure deposited on 1 mm PET

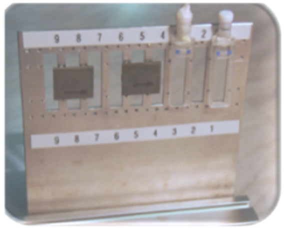


Edge line (lateral) resolution ~ 4 mm

Additionally possible standard depth resolution ~ 15 nm

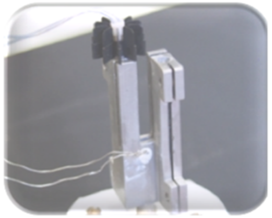
- *C. Granja, V. Kraus, Y. Kopatch, S.A. Teleznikov, J. Vacik, I. Tomandl, M. Platkevic, S Pospíšil, EPJ 21 (2012) 10004-1 – 10004-5.*
- *I. Tomandl, Y. Mora Sierra, C. Granja, J. Vacík, submitted to Nucl, Instr. Methods B 2015.*

Sample environment for in-situ experiments with neutrons


Sample changer with linear positioning, 9x quartz cells


Sample changer with linear positioning and regulated heating (up to 420 K)

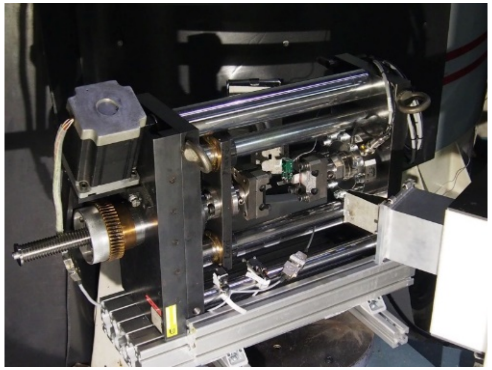
- **before 2014**
  - Samples positioning systems
  - Close cycle cryostat (10 - 298 K)
  - vacuum furnace for powder diffraction (<1000ºC)
  - Mirror furnace (up to 1000ºC)
  - vacuum furnace SANS
  - deformation rig 20kN (with joule heating)


Sample holder with heating regulation (up to 500K). Can be installed in magnet

vacuum furnace for powder diffraction

closed-cycle cryostat

20 kN deformation rig with Joule heating

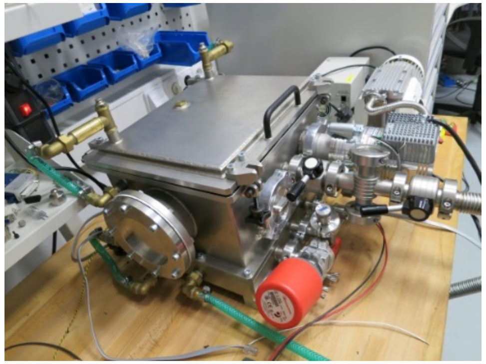


vacuum furnace for SANS

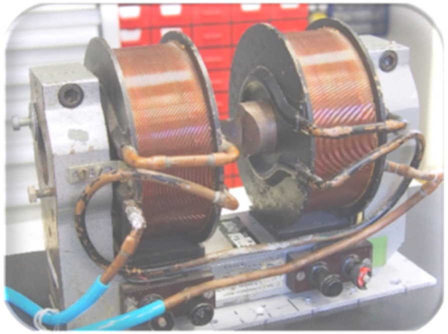
Sample environment for in-situ experiments with neutrons

- **acquired in 2014 and 2015**
  - Robotic arm at SPN-100
  - New deformation rig (60kN) (with Joule heating) at TKSN-400
  - furnace SANS - vacuum and controlled gas
  - Electromagnet at MAUD
  - Permanent magnet for feromagnetic samples

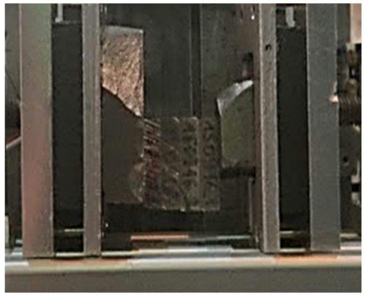
vacuum furnace for SANS



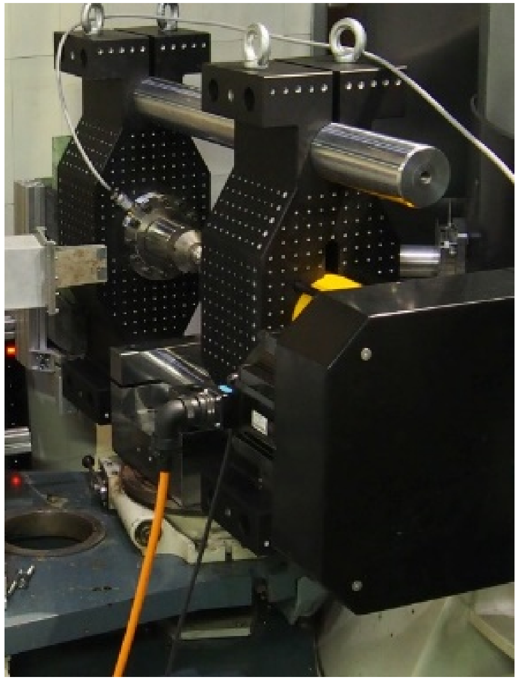
Electromagnet (0.5 – 1.2T, max. field depends on sample size)



Permanent magnet system for feromagnetic samples



60 kN deformation rig with heating



robotic arm for sample positioning

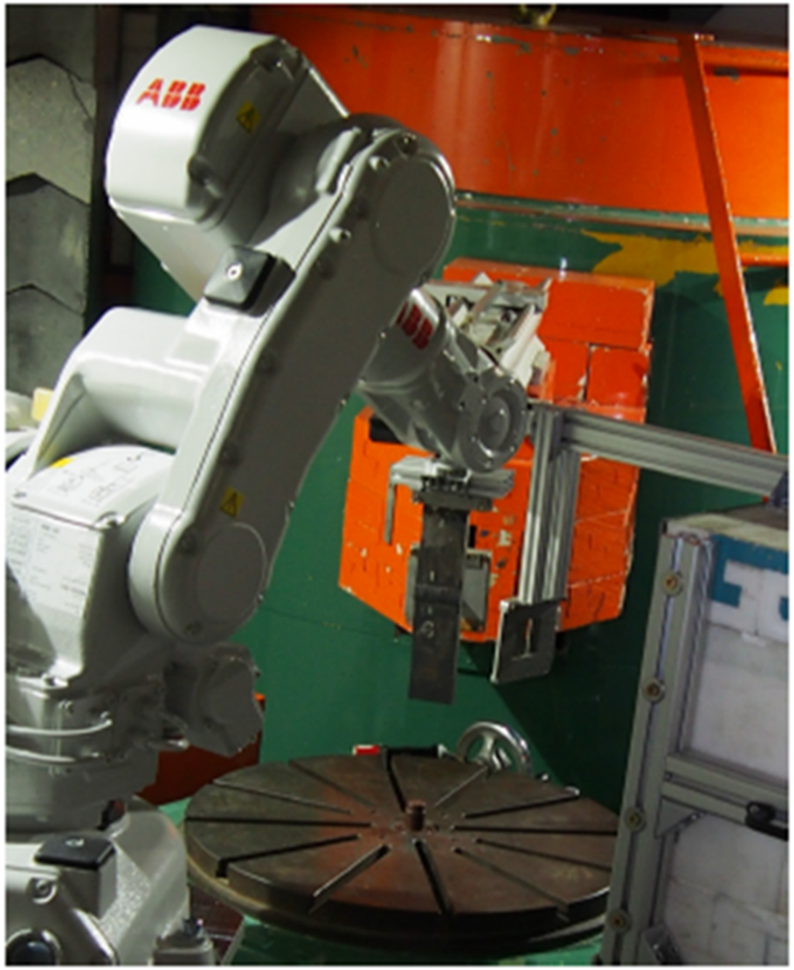
Robotic arm on channel HK4 (SPN-100 strain scanner)

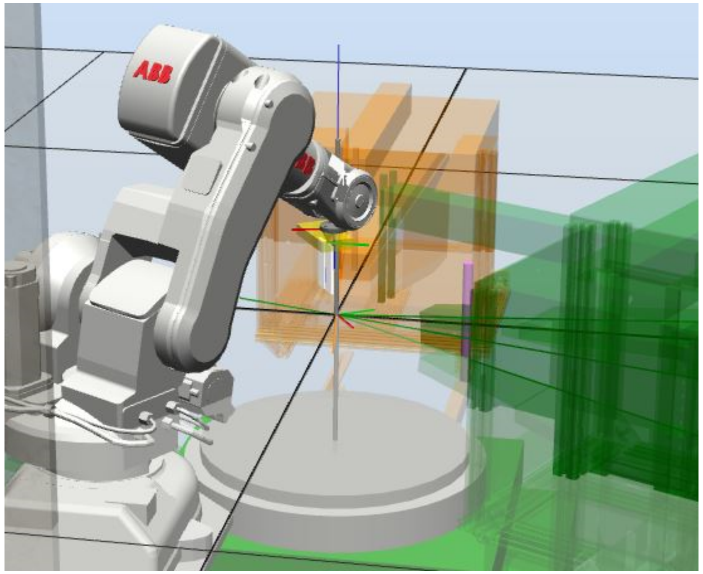
for sample manipulation, to complement the xyz stage

- More flexibility in sample manipulation
- Optimization of neutron beam time used

ABB IRB-140 robot

maximum load 6 kg



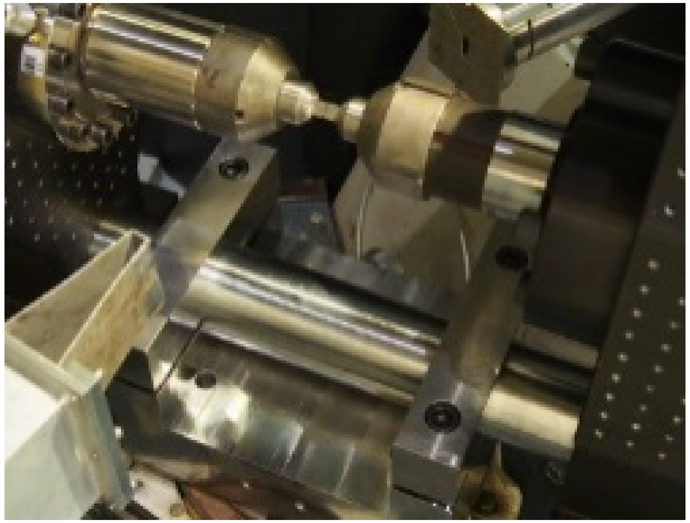


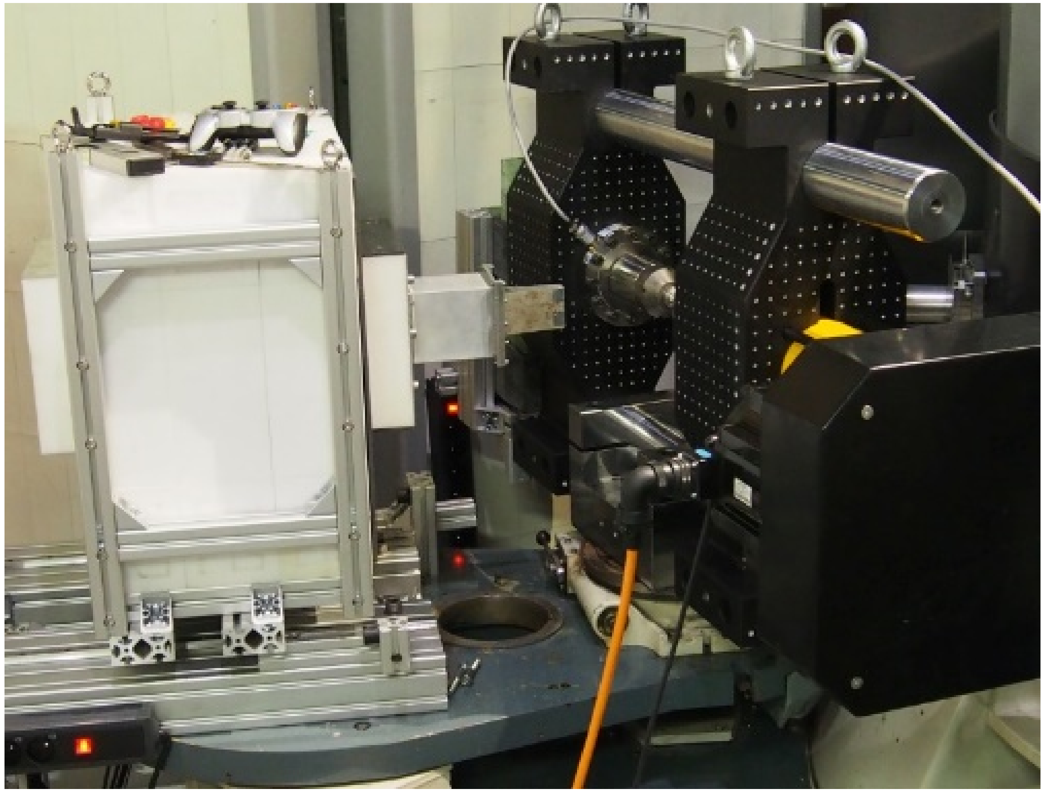
3D virtual environment model

- Advanced planning (simulation) of neutron strain scanning experiments (define measurement points and orientation)

60 kN deformation rig with heating

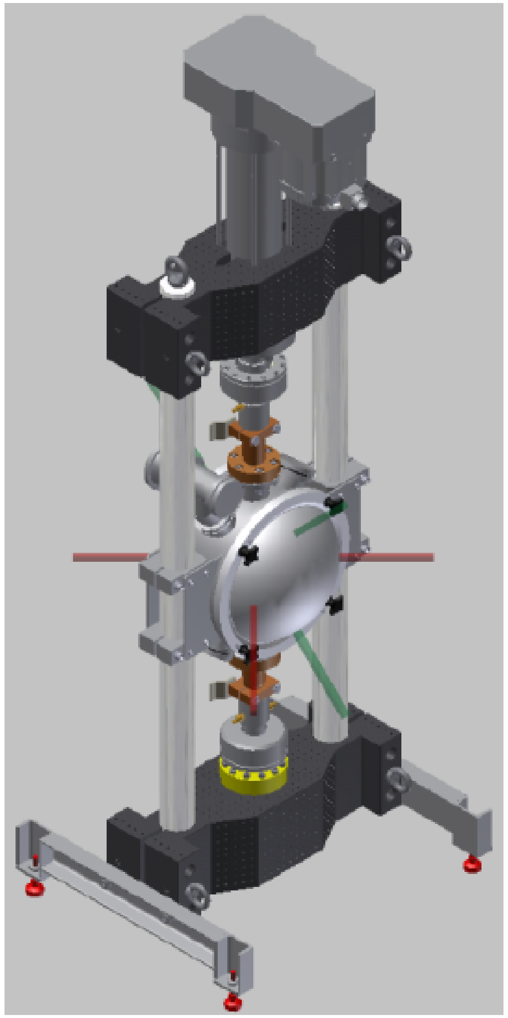
- Max. force ± 60kN
- Electrically isolated grips
- Cold/hot grips design
- Joule heating



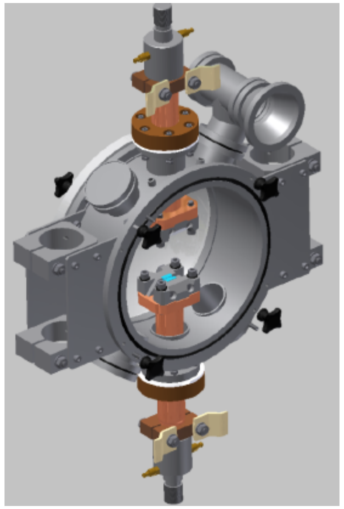


future: gradual upgrade of facilities, auxiliary methods and sample environment

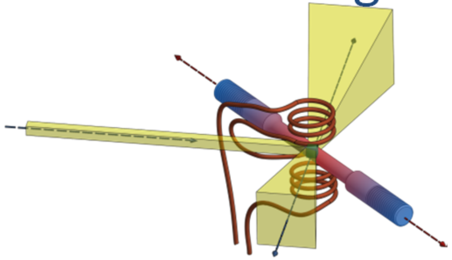
- wish: Positioning system for TKSN-400 (for heavy equipment)

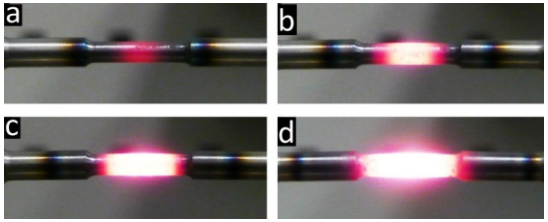


- Vacuum or air or gas atmosphere furnace (chamber) for deformation rig, large angular opening, heating rates (conductive samples) > 500°C/s, max. temperature > 1800°C



- Induction heating + combination of heating techniques

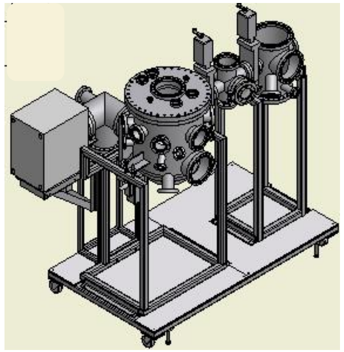




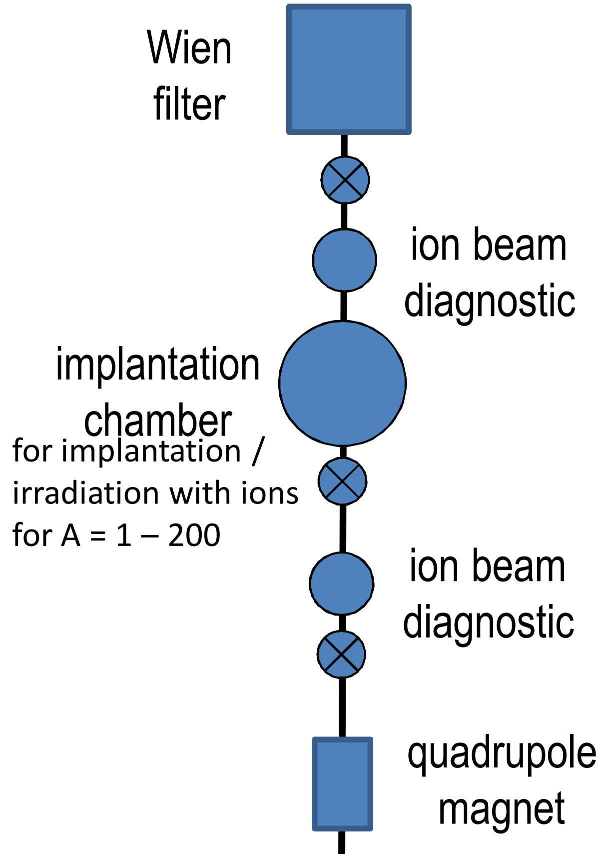
future: gradual upgrade of facilities, auxiliary methods and sample environment

- wish: New neutron guide with an elliptical guide monolith at NG and NDP
- Upgrade of PGAA electronics by digital modules
- Upgrade of AFM – new hybrid controllers and UHV head
- Molecular Beam Epitaxy

(synthesis of (ultra)thin epitaxial films)



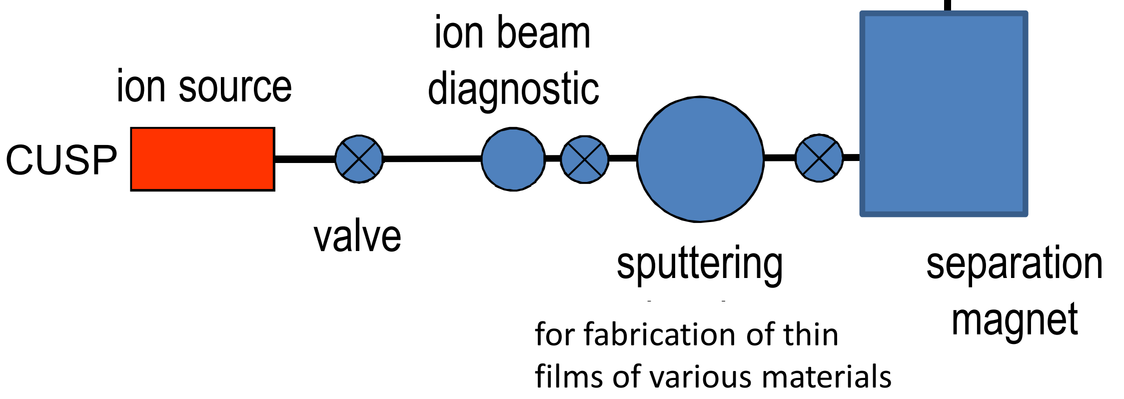
- Low Energy Ion System (LEIS) in new building (100 eV – 35 keV with possibility to upgrade the ion beam line up to 100 keV)



for implantation / irradiation with ions for A = 1 – 200

LEIS

system for fabrication and modification of thin films by low energy ions

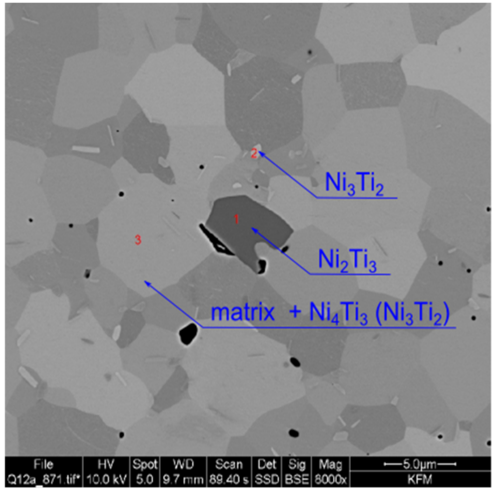


for fabrication of thin films of various materials

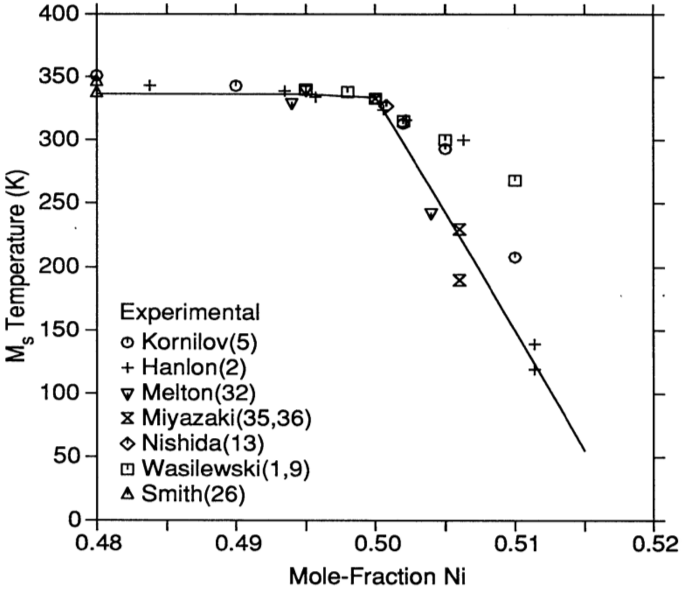
Experiments - examples

Temperature and stress induced transformations in Ni-rich NiTi shape memory alloys

- Ni-rich NiTi
- significant changes in transformation temperatures due to precipitation of secondary phases (Ni_4Ti_3, Ni_3Ti_2, Ni_3Ti, Ni_2Ti_3)



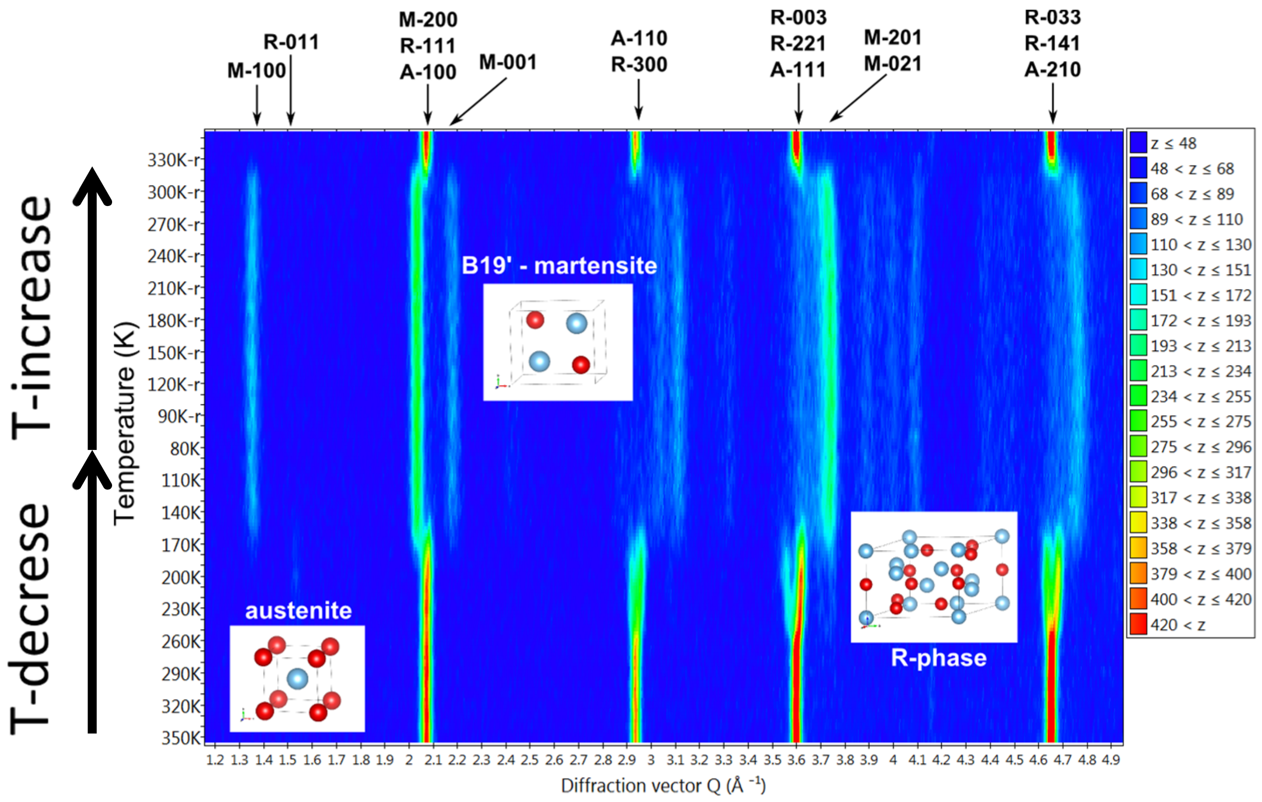
Varying the nickel content between 51 and 50 [at.%] changes the transformation temperature from -150 to 70°C

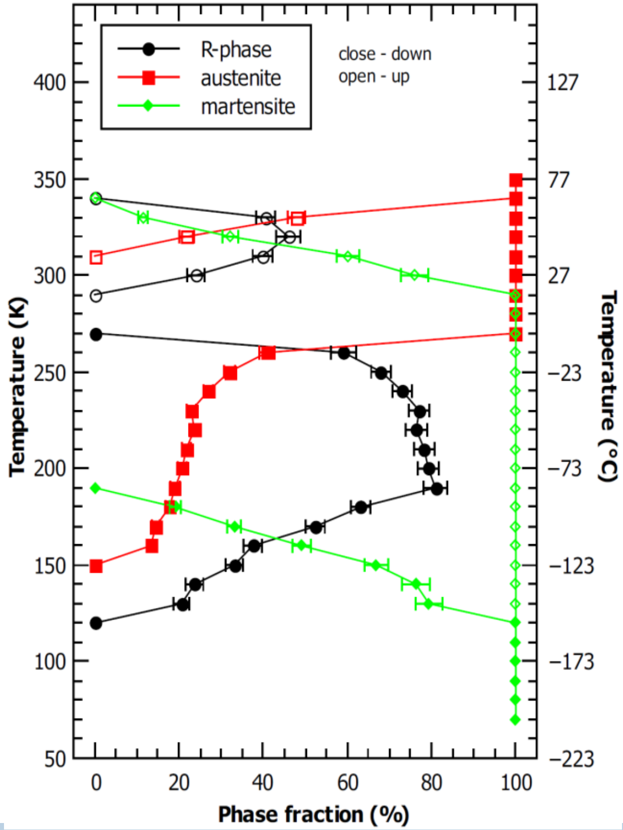


- heat treating cold worked Ni-rich NiTi (medical grade alloys most attractive for industry) causes both recovery and precipitation
- Annealing time plays significant role (precipitation kinetics relatively slow and temperature dependent)
- => modification of the alloy microstructure and thus the transformation temperatures

Temperature induced transformations in cold-worked and annealed Ni-rich NiTi

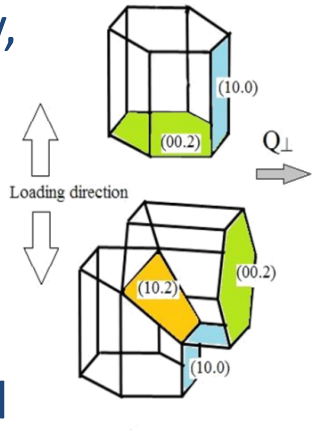
- typically characterized by DSC and X-ray diffraction methods
- But: DSC recognizes only start and/or end of the martensitic transformation
- Lab X-rays - structural information only from surface layer (can be misleading)
- high energy synchrotron X-rays do not see very important 111 reflexion
- => in-situ neutron diffraction, phase transformations in Ni-rich NiTi, in bulk
- example: the cold worked and annealed alloy - martensitic transform., 70 – 350 K





MEREDIT, experiment with Institute of Physics

Magnesium alloys and composites

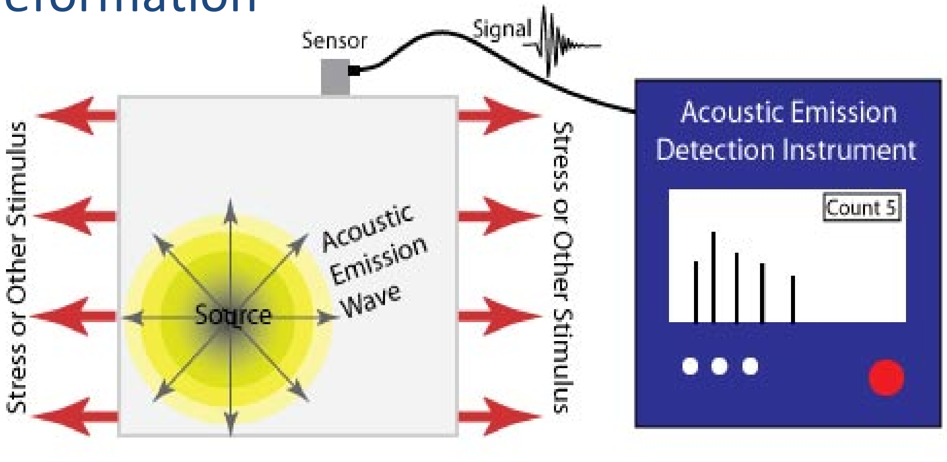


a) Tension

- The lightest structural material, applications in electronics, aeroindustry, car industry
- Alloying elements – great effect on mechanical properties
- Composites – reinforcing phase, carries the load, better mechanical properties
- deformation mechanisms (mainly **twinning**, slip system acitvity, internal strains and dislocation density), **temperature dependent**
- Neutron diffraction (ND):
  bulk information about twin volume

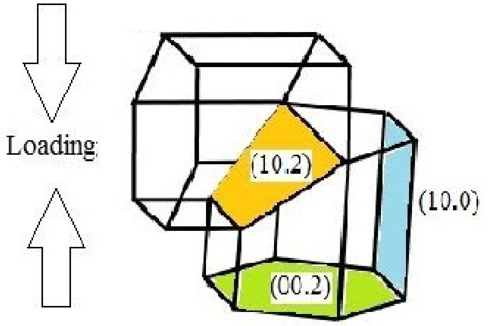
- EBSD (large grain)

- Combination with Acoustic emission (AE): in-situ monitoring of number of twins during the deformation

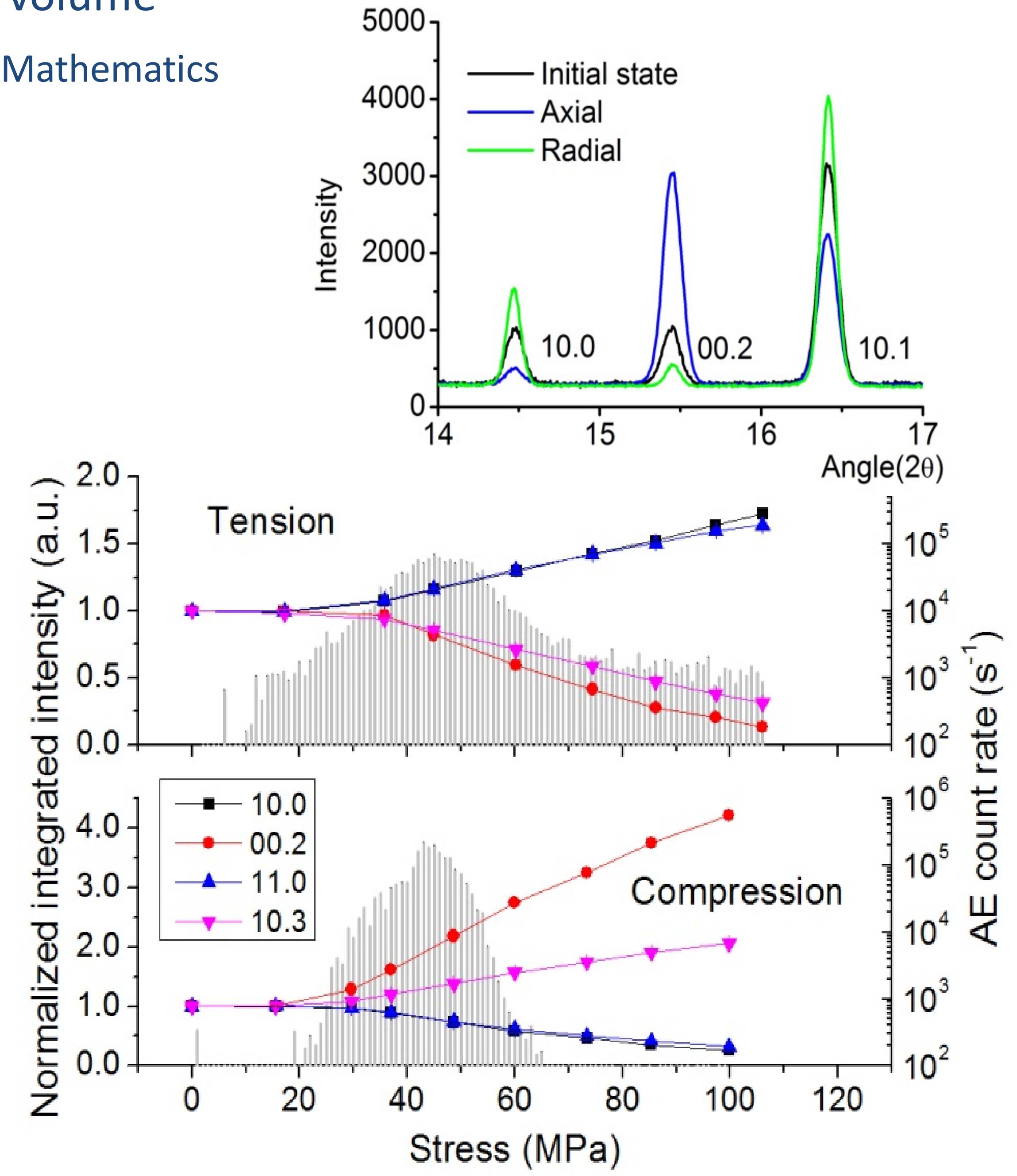


Magnesium alloys and composites

- ND: in situ information about twin volume
- TKSN-400, cooperation with Faculty of Mathematics and Physics, Charles University



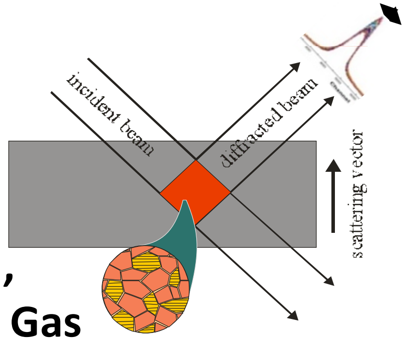
- Combination of ND, AE brings a complex info
- Acoustic emission: number of twins during the deformation
- => a large number of twins initiated first above some threshold, only afterwards their growth
- Different for compression



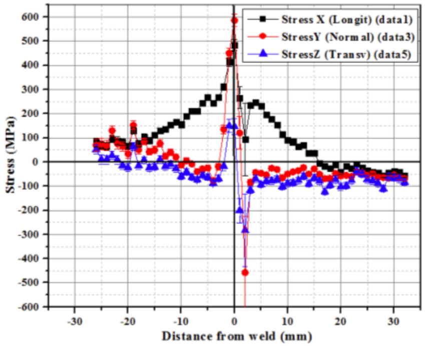
Čapek et al.

Residual stresses (SPN-100)

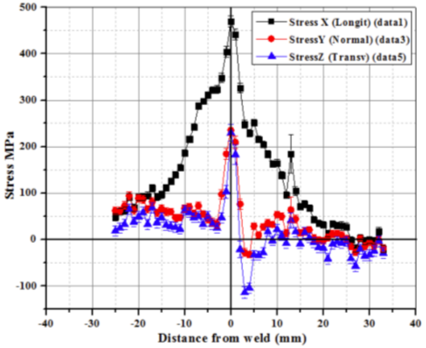
Applicability of magnetic techniques - Magnetic Barkhausen Noise (MBN) and quasi-dc permeability - to the evaluation of residual stresses



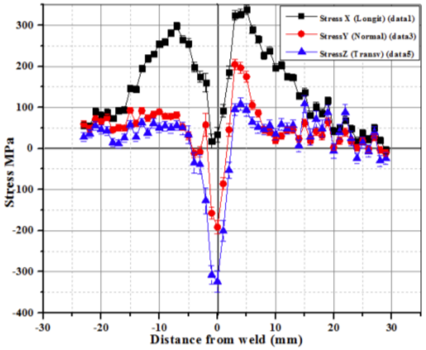
AISI 1008 steel, electron-beam welding



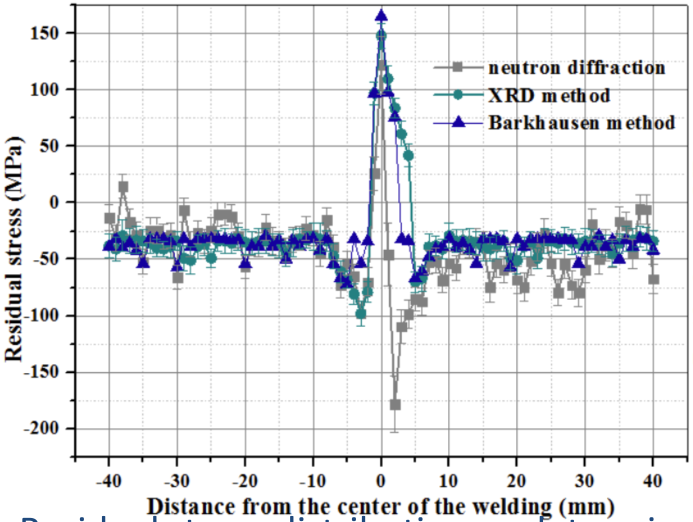
AISI 4130 steel, electron-beam welding



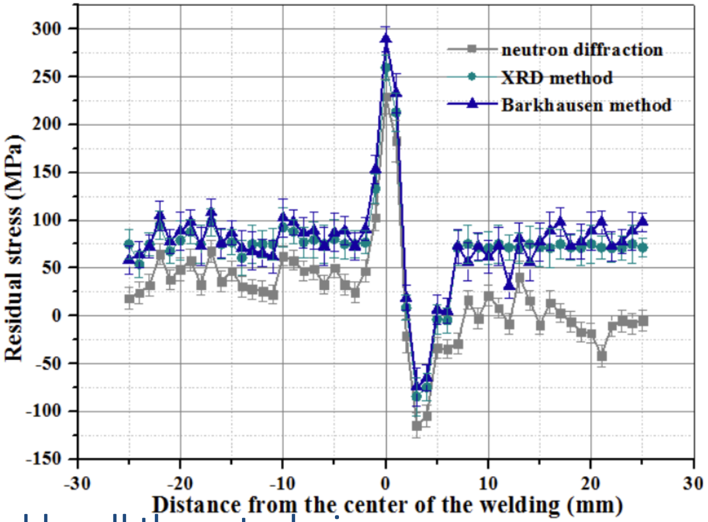
AISI 4130 steel, Tungsten Inert Gas



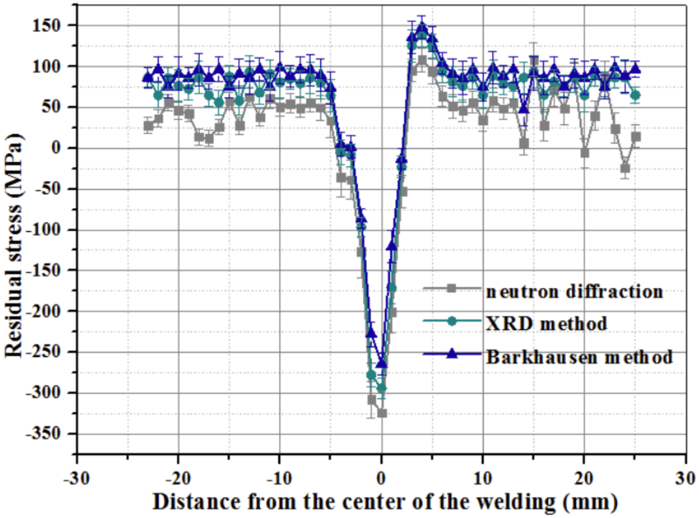
Electron Beam Welded AISI 1008 sample



Electron Beam Welded AISI 4130 sample



Tungsten Inert Gas Welded AISI 4130 sample



Residual stress distribution as determined by all three techniques.

Residual stresses determined by the MBN method are in good agreement with the XRD and ND results.

Evangelos Hristoforou (National TU of Athens)

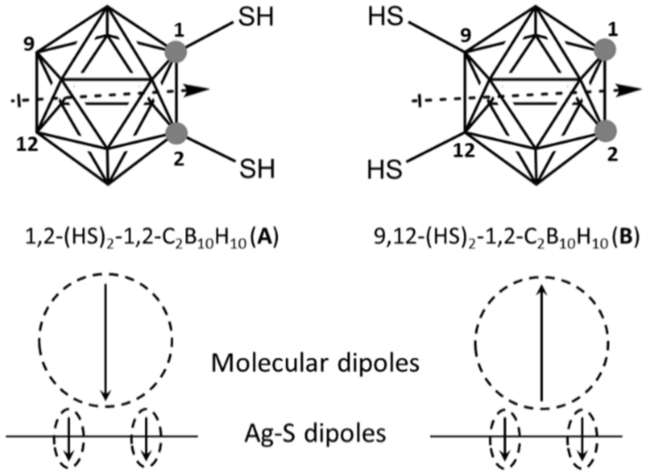
Study of adsorption of oriented carborane dipoles upon silver flat surface

NG (n,γ) instrument at LVR-15 (Prompt Gamma Activation Analysis option)

Understanding metal-organic junctions is essential for their applications in electronic devices (organic light-emitting diodes, field-effect transistors …)

General aim of the study:

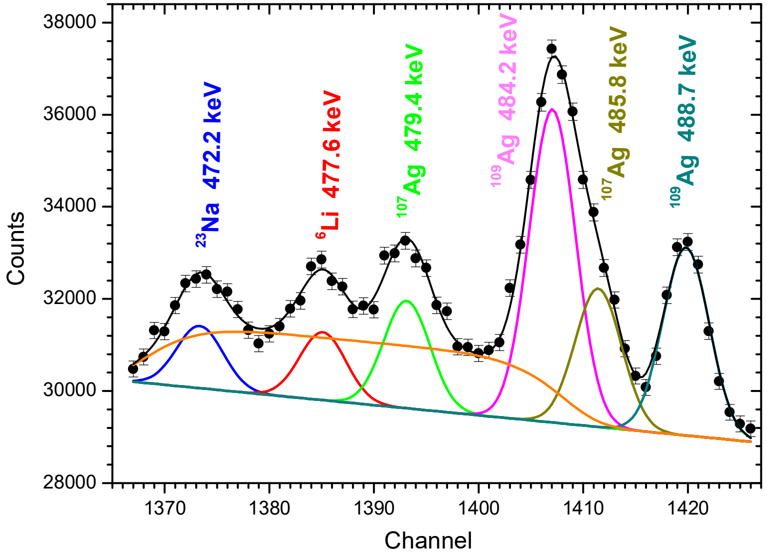
- to adjust the surface potential of a metallic substrate by adsorption of molecules with dipole moments,
- to probe the fundamental properties such as electronic structure and orientation of molecules on the surface



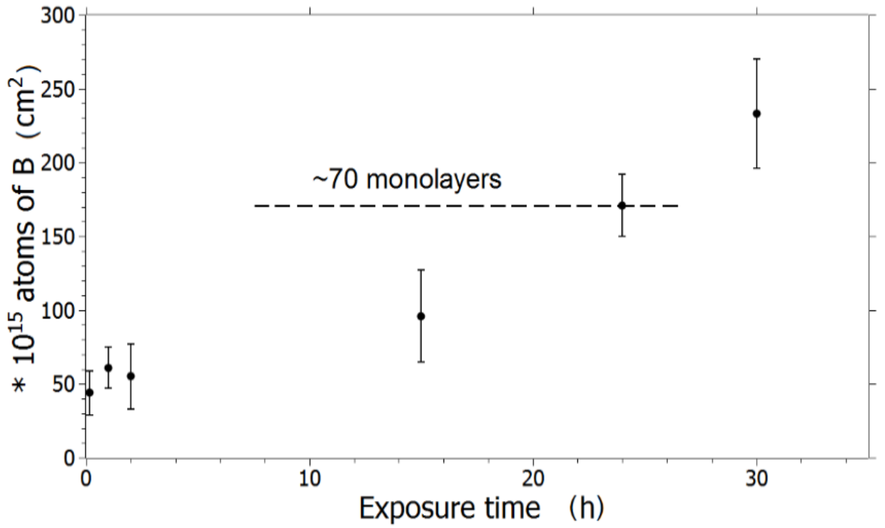
1,2-$(HS)_2$-1,2-$C_2B_{10}H_{10}$ **(A)**

9,12-$(HS)_2$-1,2-$C_2B_{10}H_{10}$ **(B)**

PGAA measurement and fit



PGAA provides the concentration of boron atoms on silver film exposed to carborane for various periods. Converted to number of self-assembled monolayers.



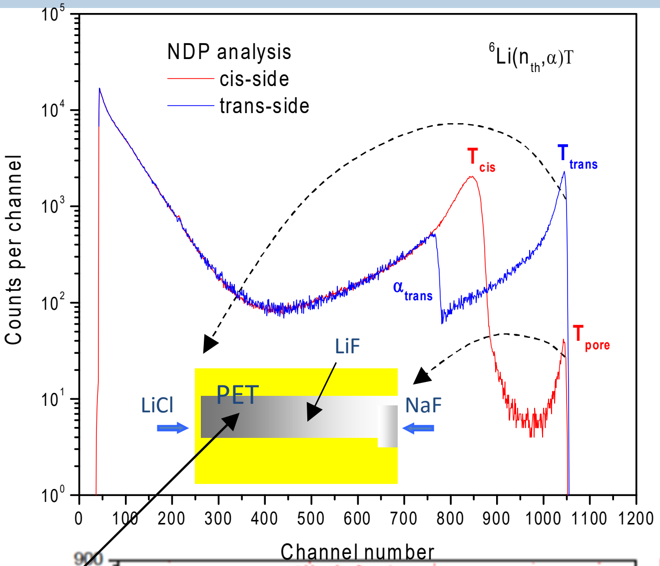
Important for tuning the surface potential of the metal

Adsorption of oriented carborane dipoles on a silver flat surface, *Aliaksei Vetushka, Laetitia Bernard, Olga Guseva, Zdeněk Bastl, Jiří Plocek, Ivo Tomandl, Antonín Fejfar, Tomáš Baše, Patrik Schmutz*, submitted to *ACS Applied Materials and Interfaces*

3D NDP: Distribution of pores in biosensors

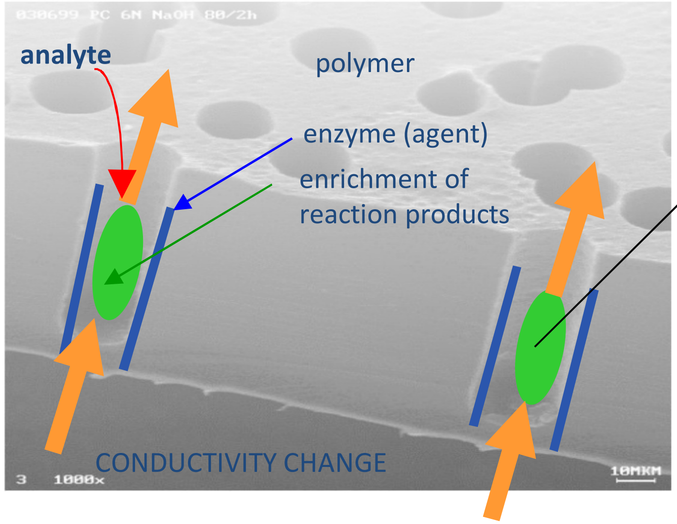
The biosensors (based on ion-irradiated polymers) properly work only if the pores have a certain (conical or cylindrical) shape, and the distribution of pores is homogeneous.

Because of a complex process of chemical etching, the pores may acquire improper shapes and become non-homogeneous.



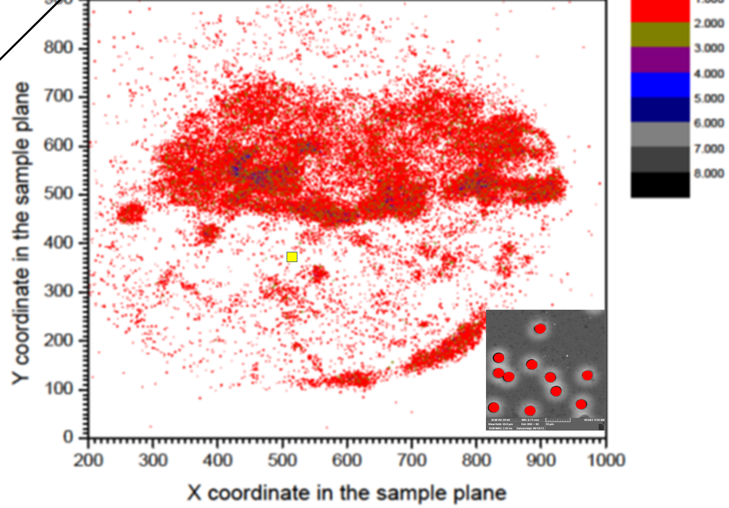
Simulation of enzymatic reaction

$\mathbf{NaF_{soluble} + LiCl_{soluble} \rightarrow}$
$\mathbf{\rightarrow\ LiF_{insoluble} + NaCl_{soluble}}$



The homogeneity of distribution can be analyzed by NDP with the new 3D profiling technique utilizing multipixel spectroscopic detectors – TimePix

3D imaging method: several micrometers lateral resolution, tens of nanometers of depth resolution



CONCLUSION

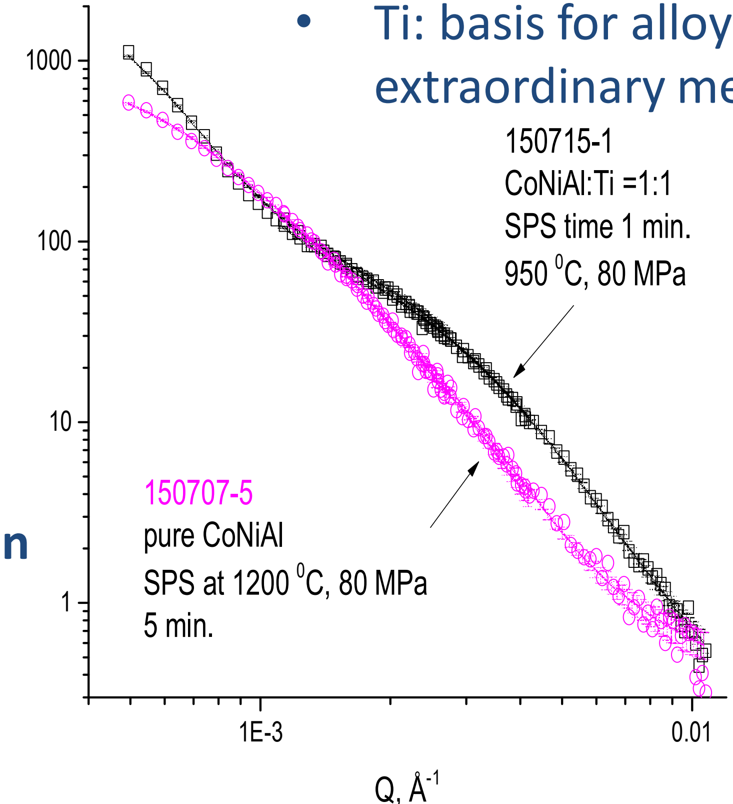
method can be used for analysis of pore distribution and understanding of their shapes

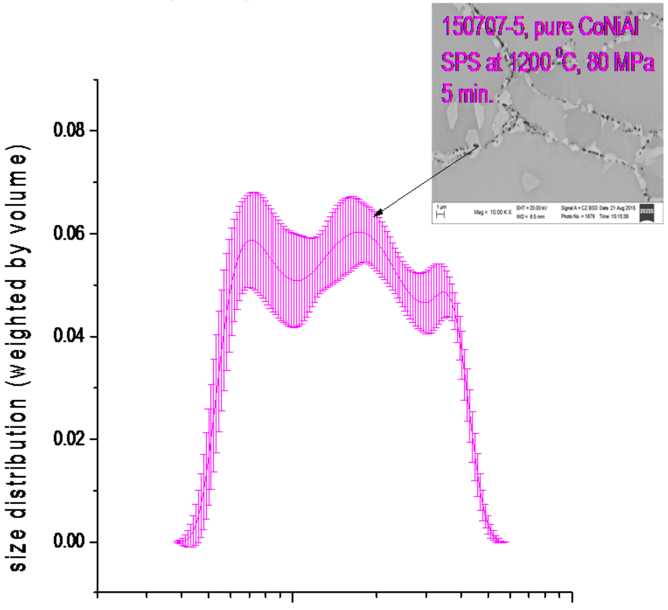
J. Vacik, I. Tomandl, V. Havranek, V. Hnatowicz, Y. Mora, 2015 IEEE Nuclear Science Symposium @ Medical Imaging Conference, San Diego

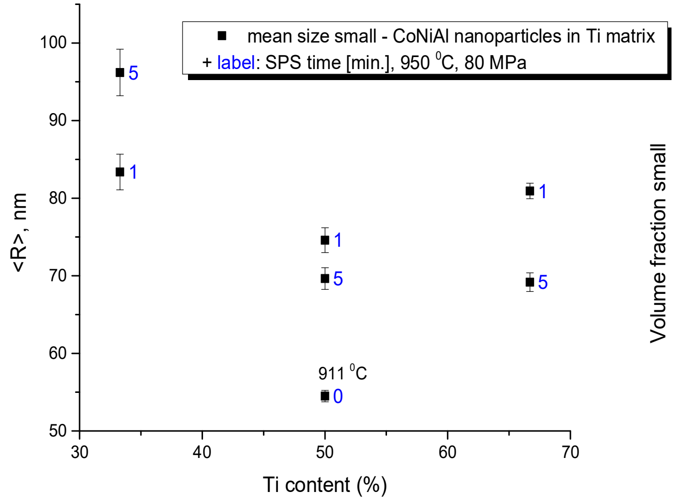
Ti+CoNiAl system prepared by spark plasma sintering (SPS) technique

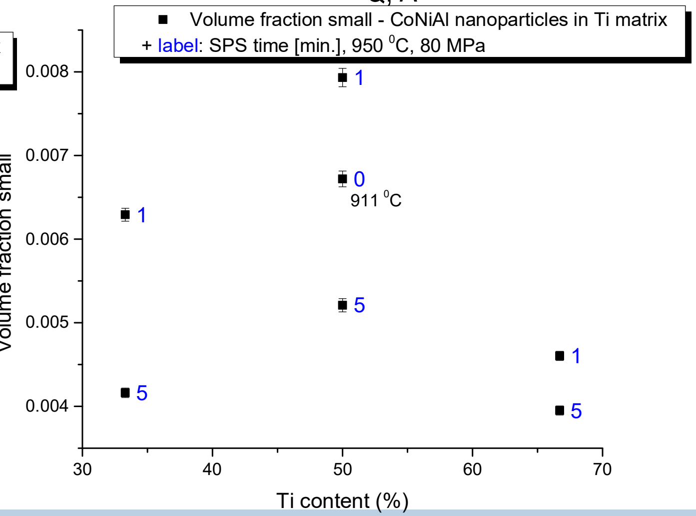
- Experimental composite material from phases with significantly different functional characteristics
- step towards tailored functional materials
- **joining by SPS**
- **microstructure investigation**

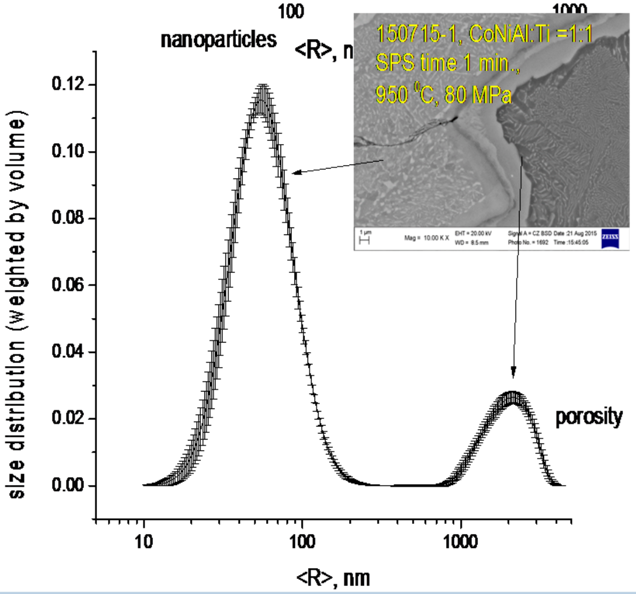
- CoNiAl: ferromagnetic shape memory
- Ti: basis for alloys exhibiting extraordinary mechanical properties







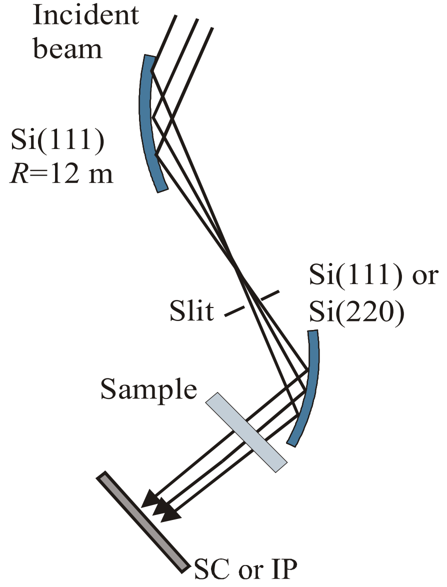




Institute of Plasma Physics; IP; MFF UK, CZ MAUD (SANS diffractometer)

Neutron optics diffractometr (NOD)

- primarily designed for testing neutron diffraction optics (neutron monochromators and analyzers
- Tailoring of thermal neutron beams by diffraction on elastically deformed single crystals



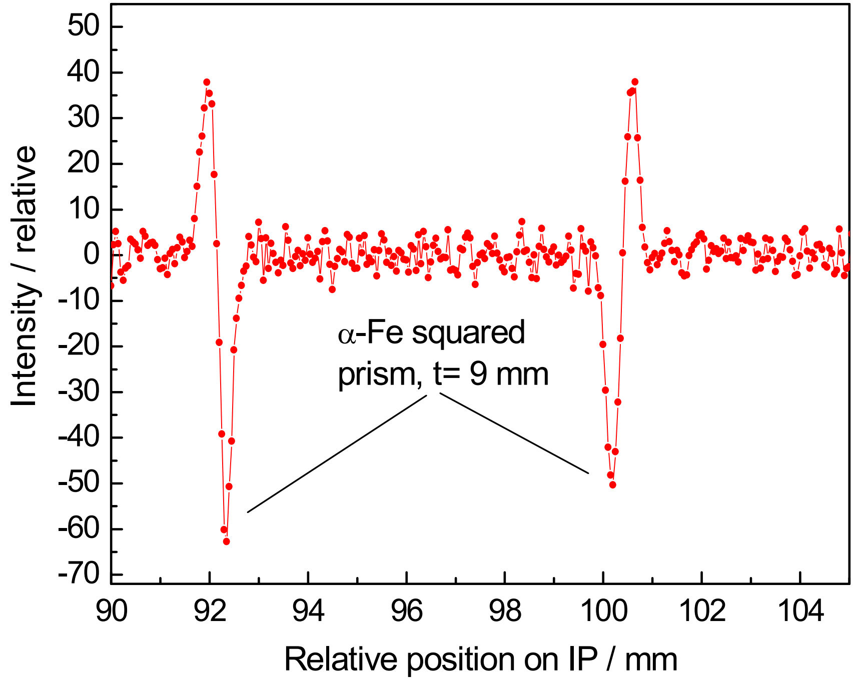
Applications

Focusing neutron monochromators

- Neutron imaging

- Powder diffraction

Example: quasi-planar neutron wave:
20 mm beam with 1’ divergence for imaging refraction effect.
This proves the **extremely high monochromaticity and collimation** of the beam, permitting the investigation of refraction effects at sharp edges.



Proposal: W. Woo, KAERI Daejeon

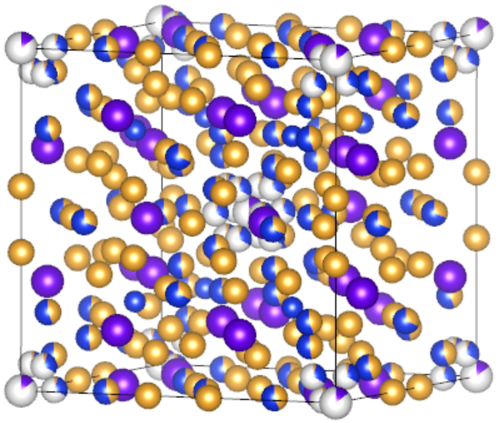
P. Mikula, M. Vrána, J. Pilch, B. S. Seong, W. Woo, V. Em, *J. Appl. Cryst.* 47 (2014) 599-605.

P. Mikula, M. Vrána, J. Šaroun, F. Krejčí, B. S. Seong, W. Woo, M. Furusaka, *J. Appl. Cryst*. 46 (2013) 128

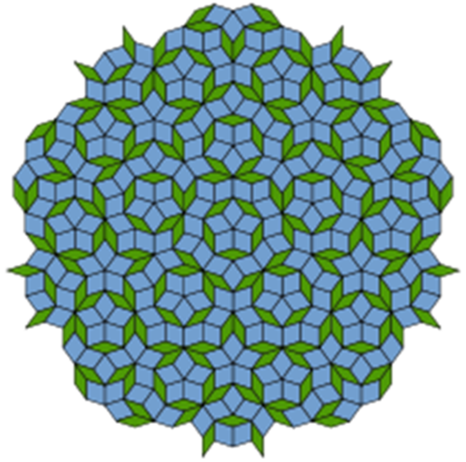
P. Mikula, M. Vrána, J. Šaroun, V. Davydov, V. Em, B. S. Seong, *J. Appl. Cryst*. 45 (2012) 98-105.

Quasicrystal approximants

Tb-Au-Si

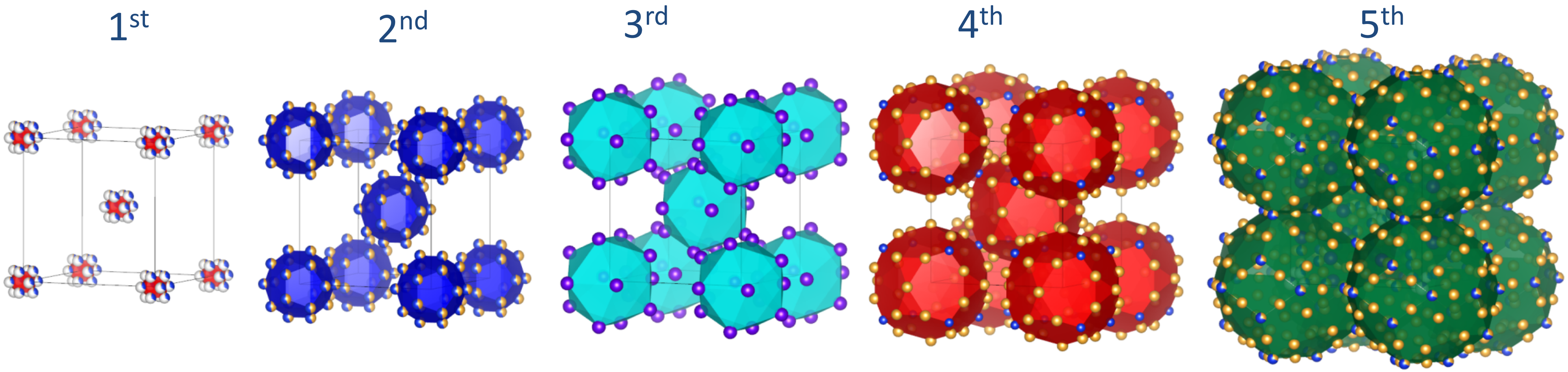


quasicrystal:
- symmetric
- aperiodic



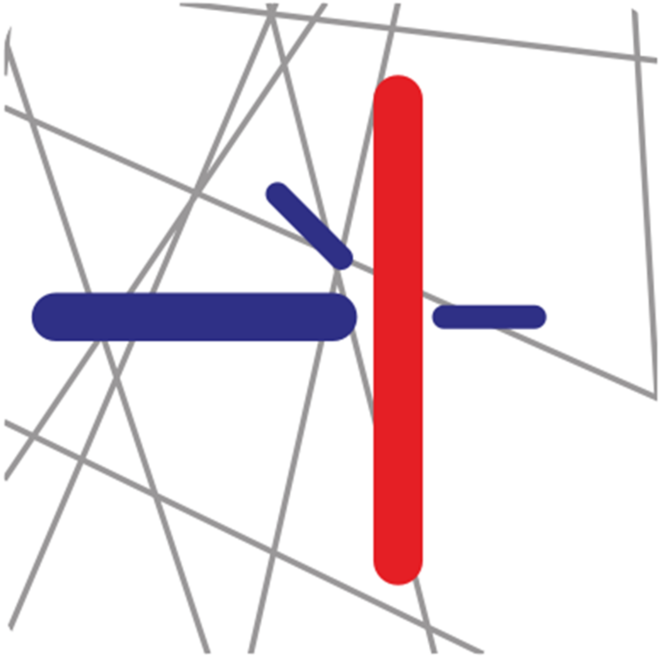
quasicrystal approximant:
- regular crystal with a complex unit cell
- composition similar to a real QC
- contains motives with QA symmetry

coordination spheres:



1st determination of magnetic structure in a quasicrystal approximant was done at NPI

G. Gebresenbut, M. S. Andersson, P. Beran, P. Manuel, P. Nordblad, M. Sahlberg and C. P. Gomez, *J. Phys.: Condens. Matter* 26 (2014) 322202.



Thank you for your attantion.

presented by Pavel Strunz
Head of Neutron Physics Laboratory