Research programme 1

Physics for materials engineering

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The project Solid state physics for the 21st century – SOLID21
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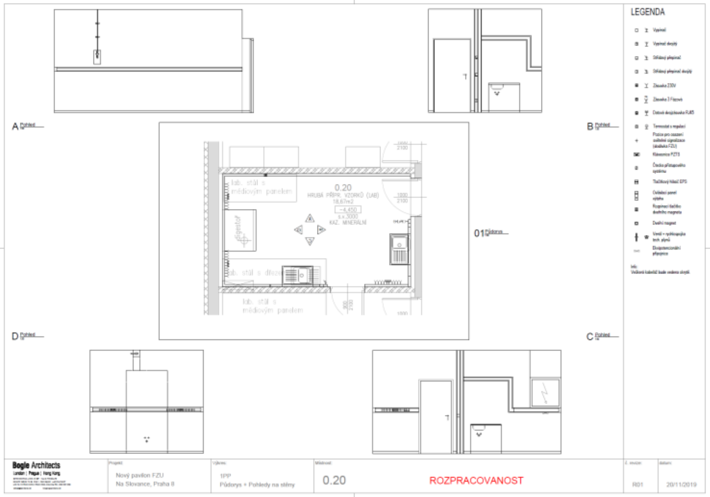
Activities and Benefits

- Excellent research – some additional funding
- New state-of-art new equipment
  - Will be mentioned for each VA
- New research facility
  - not only state-of-art but also basic facilities



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General characteristic of RP 1

The main goal is the systematic study of ferroelastic and multiferroic functional materials, such as shape memory alloys, ferromagnetic memory alloys, liquid crystals and piezoelectric materials. The programme covers physics research of modern functional materials with properties achieved through targeted manipulation of their microstructure and path to such microstructure. It includes the construction of a “Domain Engineering Atelier” for modern PhD training in the field of ferroic materials and the commissioning of a unique modern X-ray microscope allowing us to develop a unique *in situ* 3D microscopy.

Research activities of the project are divided into five collaborating key research programs reflecting interdisciplinary character of solid state physics.

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Research activities (RA)

1. Functional ferroelastic materials and layers

P. Šittner

2. Shape memory alloys NiTi for technical applications

L. Heller

3. Magnetic materials with martensitic transformation

O. Heczko

4. Physical research of piezoelectric materials

J. Hlinka

5. Multifunctional liquid crystalline materials and composites

V. Novotná

Technology and equipment

D. Šimek

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RA1&2

Functional ferroelastic materials and layers Shape memory alloys NiTi for technical applications

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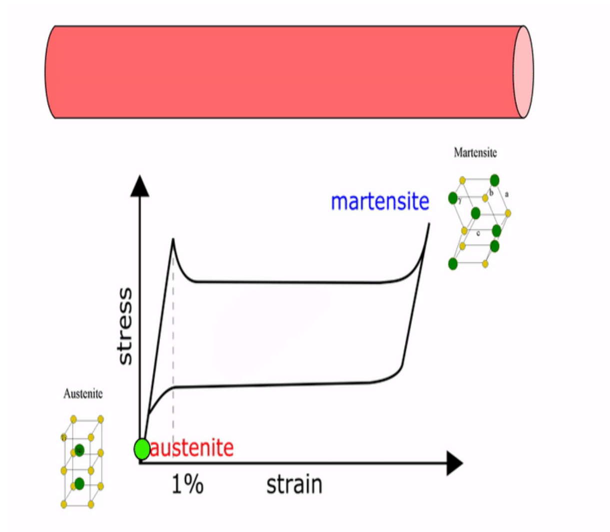
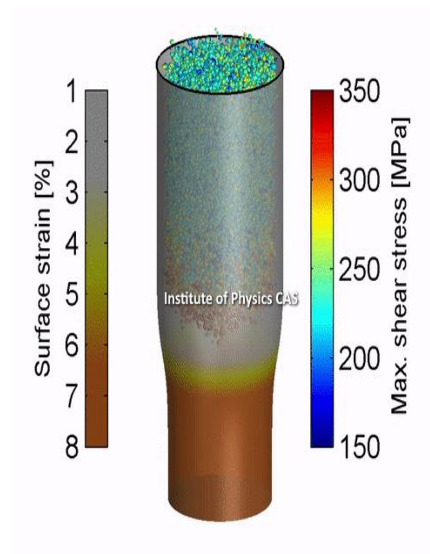
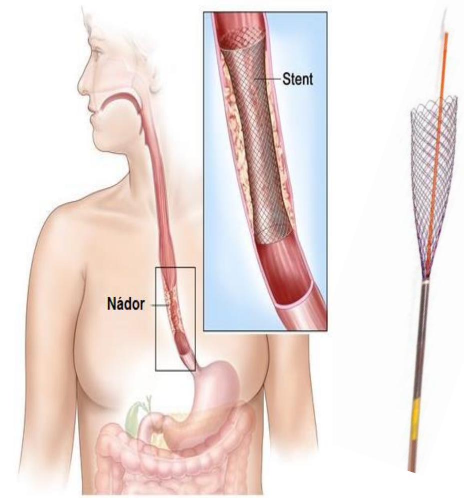
Functional ferroelastic materials and layers (RA1) and Shape memory alloys NiTi for technical applications (RA2) focus on the basic and applied research of martensitically transforming shape memory alloys.



Superelastic deformation of NiTi wire in tension



Cone shaped front of the macroscopic localized deformation band propagating in NiTi wire deformed in tension



Esophageal braided NiTi stent

Thermomechanically loaded NiTi shape memory alloys (SMA) deform solely via the intrinsically recoverable deformation processes - elastic deformation and martensitic transformation, only if the applied stresses, strains and temperatures fell within certain material specific limits. The stress-strain-temperature responses of NiTi within this range can be reliably predicted by SMA models and variety of engineering applications already exist, mainly in superelastic medical devices.

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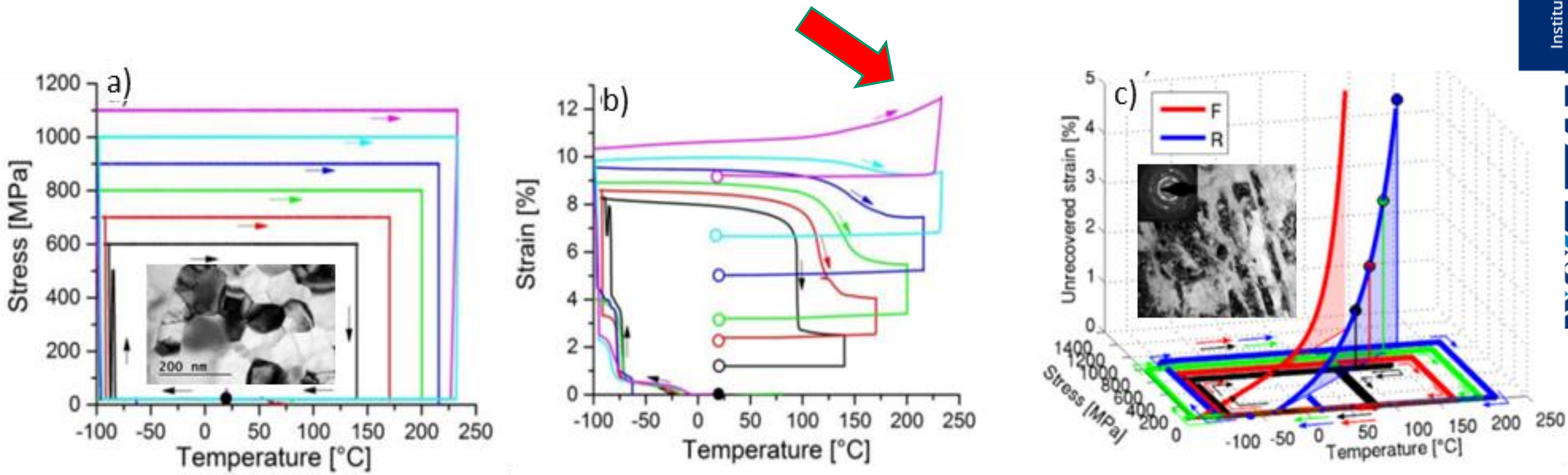
Unique thermomechanical loading responses of NiTi wire in tensile tests at elevated temperatures and stresses



Stress strain-temperature responses of NiTi SMA wire with nanograined microstructure subjected to 6 thermomechanical loading tensile tests [1]. a) prescribed stress-temperature paths, b) recorded strain-temperature responses, c) unrecovered strains recorded in individual tests are generated upon the reverse martensitic transformation under large stress. TEM micrographs show microstructures in wires deformed at lowest (a) and highest (c) tensile stresses. **When the reverse martensitic transformation proceeds upon heating under highest applied stress 1100 MPa, tensile stress increases instead of decreasing and austenite twins appear in deformed microstructure**, since the reverse martensitic transformation proceeds into twinned and slipped austenite as explained in [1]. This represents a TRIP like deformation mechanism involving coupled martensitic transformation and plasticity [1,2].

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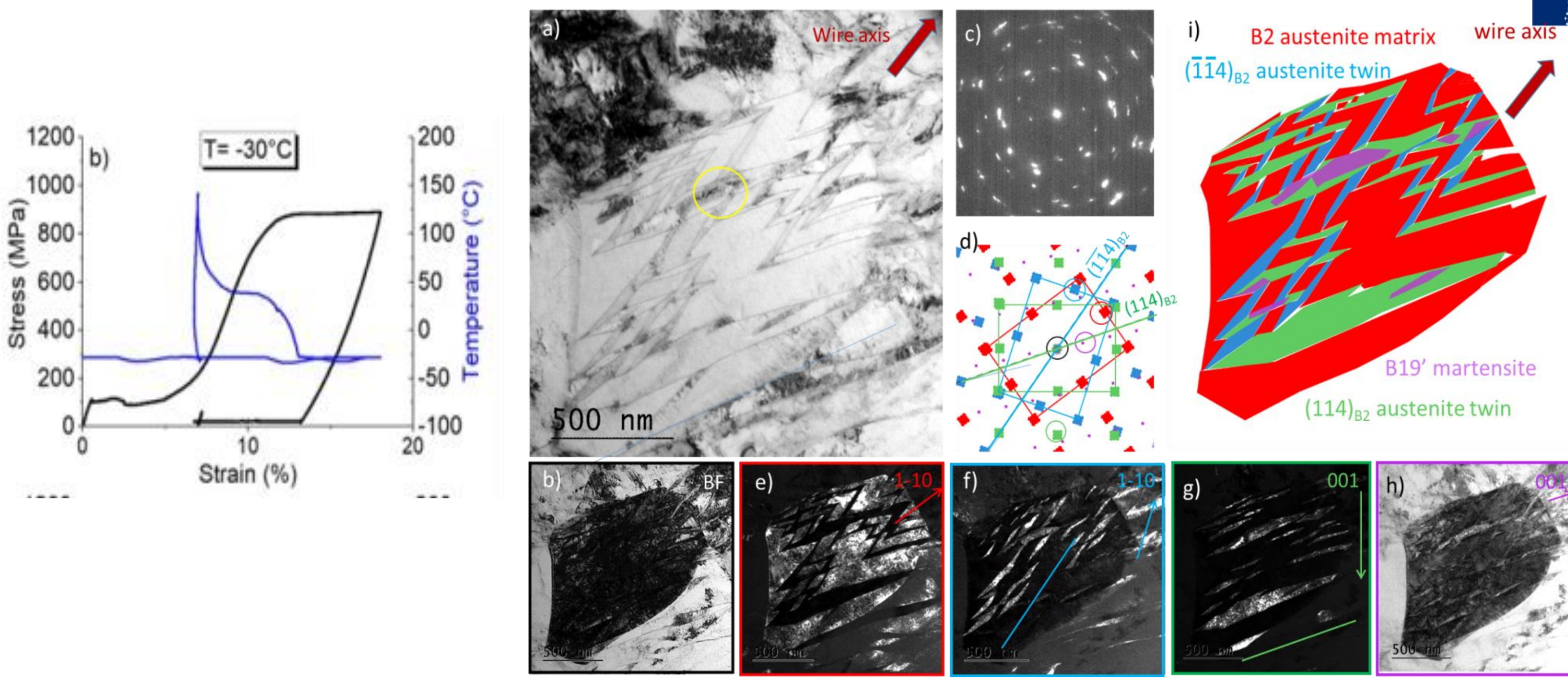
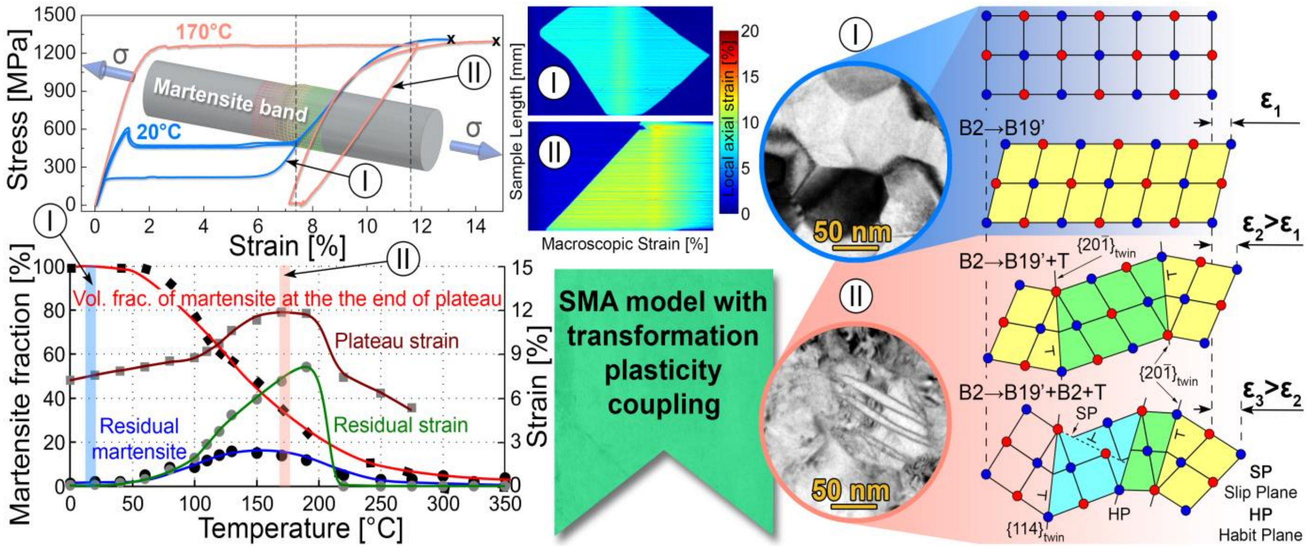
{114} austenite twins created by the tensile deformation beyond the crystallographic limits of the transformation



Lattice defects observed by TEM in the microstructure of NiTi wire with recrystallized microstructure deformed to 18% strain at -30 °C, unloaded and heated to 150 °C and cooled back to room temperature . a) BF image of twinned microstructure, b) stress-strain-temperature record of the test, electron diffraction pattern (c) taken from the yellow circle in (a) associated with 3 austenitic and one martensitic lattice (d), sketch of the deformed microstructure (e) showing constituents of the microstructure associated to individual lattices by colours. **As explained in [3], the austenite twins formed by deformation twinning in the B19' martensite upon tensile loading followed by B19'→$B2^T$ reverse martensitic transformation on unloading and heating.**

TRANSFORMATION – PLASTICITY COUPLING IN NITI



References

1. Heller et al.,*International Journal of Plasticity* **116:** 232-264, 2019.
2. Šittner et al., *Progress in Materials Science* **98**:249-298, 2018.
3. Chen et al *Acta Materialia* **180**:243-259, 2019.
4. Šittner et al., *Shape Memory and Superelasticity*, **5**:383-396, 2019.
5. Sedmák et al., *Science* **353**:559-562, 2016.
6. Sedlak et al ., *International Journal of Plasticity* **39**, 132-151, 2012.

Deformation behaviour of NiTi shape memory alloys at elevated temperatures [1]. While the stress induced B2-B19' martensitic transformation in NiTi wire deformed in tension at room temperature 20 °C (case I) gives rise to reversible superelasticity and leaves no defects in the microstructure of the deformed wire, **the B2→B19'→B2^T transformation proceeding at high temperature 170 °C (case II) yields large inhomogenous tensile strains ($e_3 > e_2 > e_1$) not recoverable on unloading and heating and leaves behind austenite twins and slip dislocations [1]**. The tensile deformation localized in cone shaped Luders like band fronts [5] is considerably larger and the plateau length attains maximum at test temperature specific for given wire microstructure [4]. Based on the obtained results, we have implemented this TRIP like deformation mechanism into an earlier developed macroscopic constitutive model [6]. Numerical simulations using the updated model [1] reproduce the observed unusual thermomechanical responses due to the TRIP like deformation at elevated temperatures and stresses (Fig. 1).

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RA3
Magnetic materials with martensitic transformation

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Introduction

Ni2MnGa Heusler alloy is ordered L21 ferromagnetic compound exhibiting martensitic transformation

Ni-Mn-Ga compound is considered as one example of magnetoelastic multiferroics combining ferromagnetism and ferroelasticity

The essential condition for appearance of elastic multiferroicity is martensitic transformation

Transformation of ferromagnetic cubic phase to low symmetry phase results in ferroelastic order.

In Mn excess the martensitic transformation is above room temperature

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Electronic structure of martensite (and austenite)

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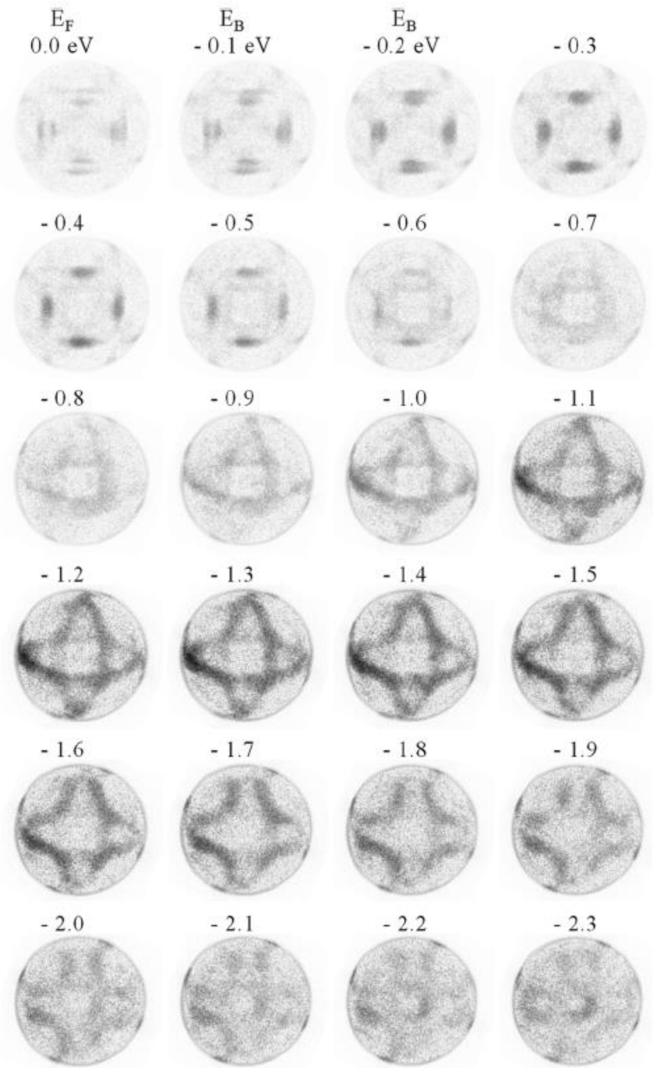
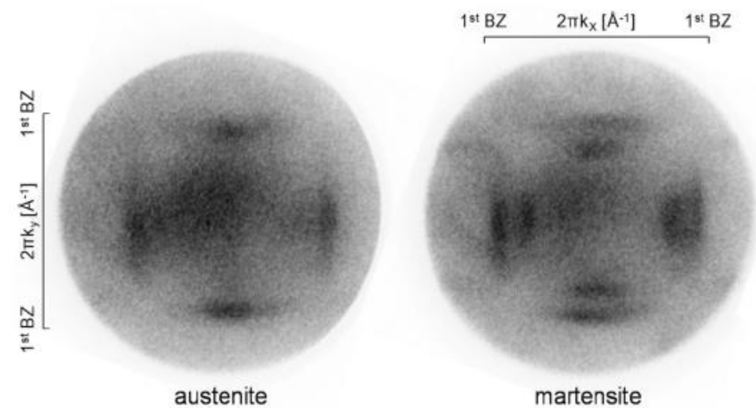
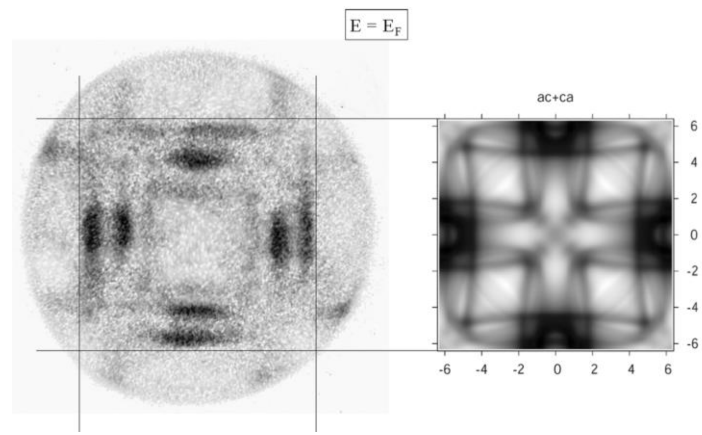
In cooperation with RP4



Sequence of selected PEEM images of equi-energetic cutsof martensite



Comparison of measured Fermi surface of twinned martensite and austenite of Ni-Mn-Ga



Comparison of experiment and theoretical calculation of Fermi surface of twinned martensite

O. Heczko et al. Electronic structure in the twinned 10M martensite phase of the Ni49.7Mn29.1Ga21.2 Heusler alloy: Experiment and theory, **Phys. Rev. B** 98, 184407 (2018)

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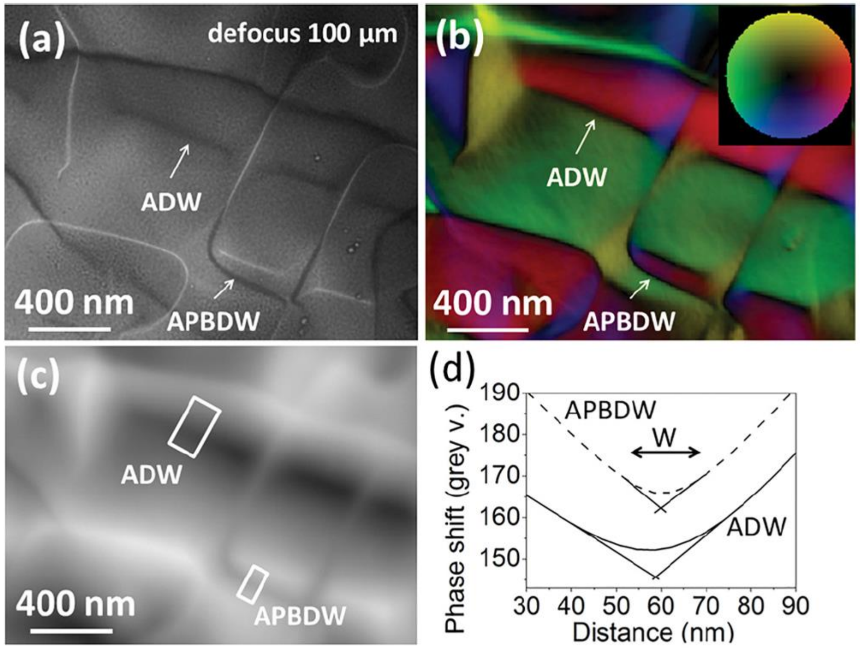
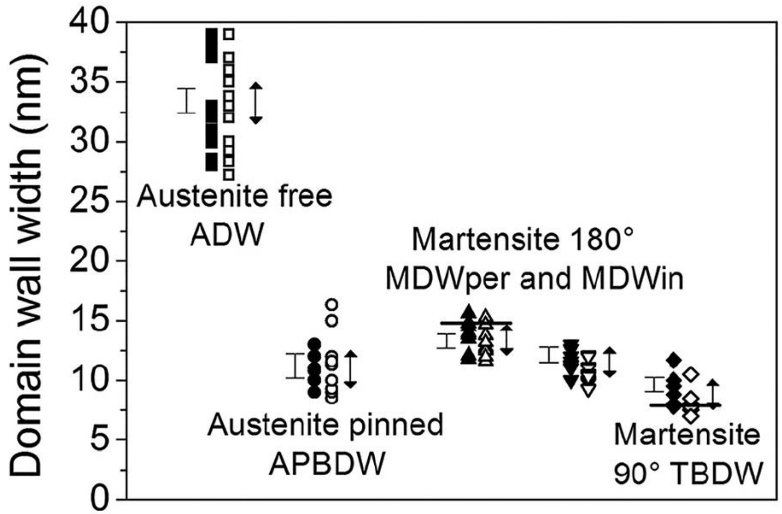
Magnetic domains and antiphase boundaries

Magneto-elastic and thus **transformation properties** can be strongly influenced by the density of antiphase boundaries, i.e. planar defects occurring in ordered L21 Ni-Mn-Ga phase in interaction with magnetic domains.



Comparison of different types of magnetic domain walls in Ni-Mn-Ga austenite showing domain walls free from APB [austenite domain wall (ADW)] and pinned on APBs (APBDW).



Comparison of domain wall widths in different phases and positions by both measurement methods. Solid symbols indicate measurements by phase reconstructed
maps and open symbols by the linear extrapolation method. Lines in the martensitic phase indicate the predicted values.

M. Vronka , O. Heczko , and M. De Graef, Appl. Phys. Lett. **115**, 032401 (2019

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Domain structure in NiMnGa martensite

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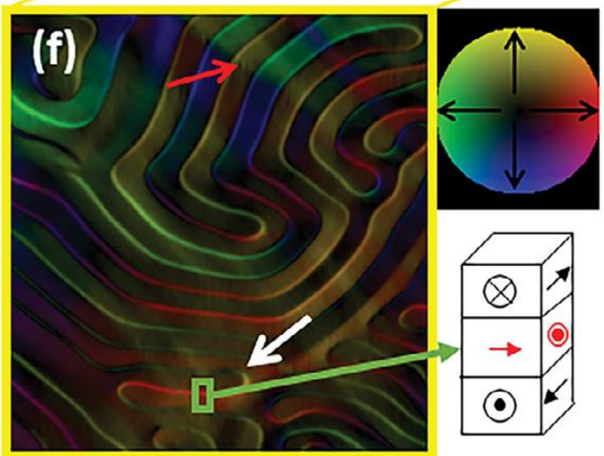
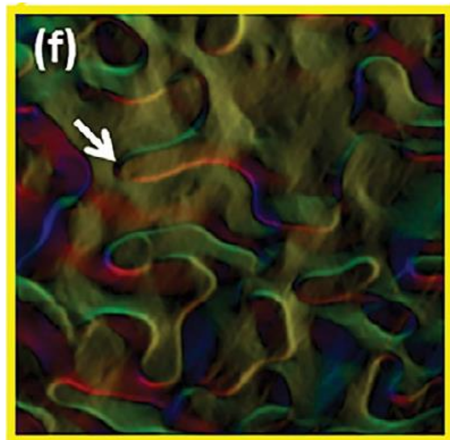
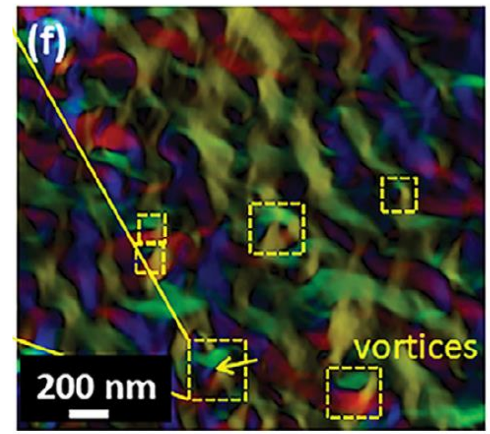
Magnetic domain structures in foils with different density of antiphase boundaries



slowly cooled sample

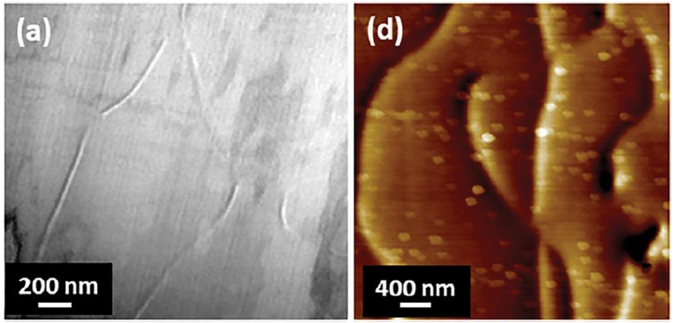


air quenched sample



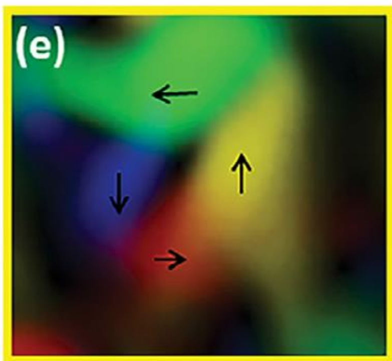
water quenched sample

presence of vortices.



APB visualized by magnetic contrast

M. Vronka, L. Straka and M. De Graef, O. Heczko, Antiphase boundaries, magnetic domains, and magnetic vortices in Ni–Mn–Ga single crystals, *Acta Materialia 184, 179–186 (2020)*



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RA4

Physical research of piezoelectric materials

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New Equipment

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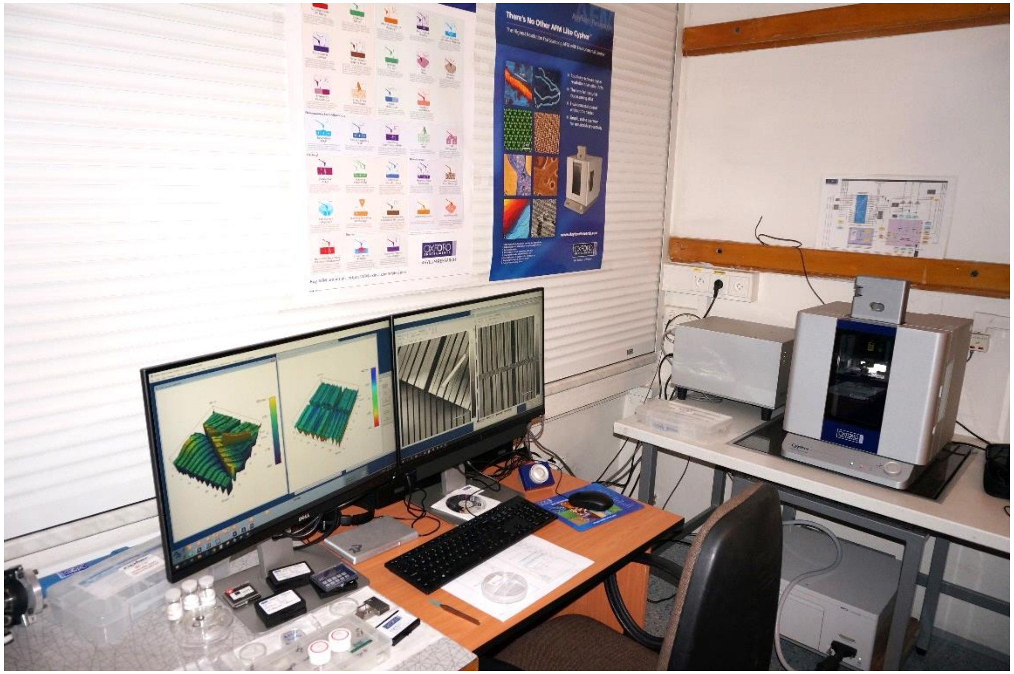
The group purchased and tested new equipment for multifrequency piezoforce microscopy (PFM) primarily allows to study ferroelectric domain structures.



New instrument dedicated for SOLID21: now fully operational, under test regime

(Oxford Instruments Asylum Research Cypher S AFM system)

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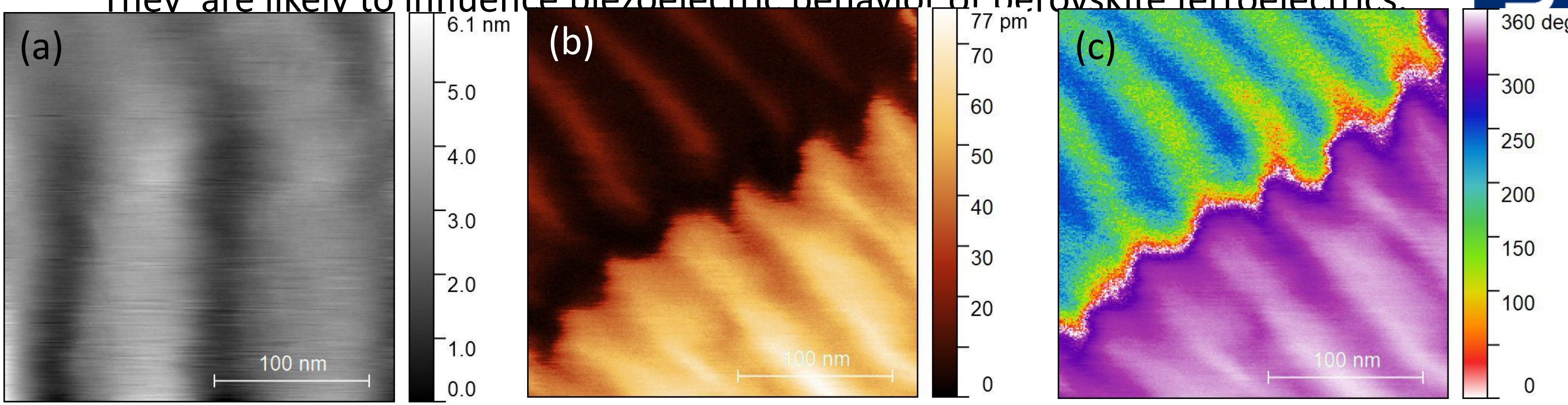
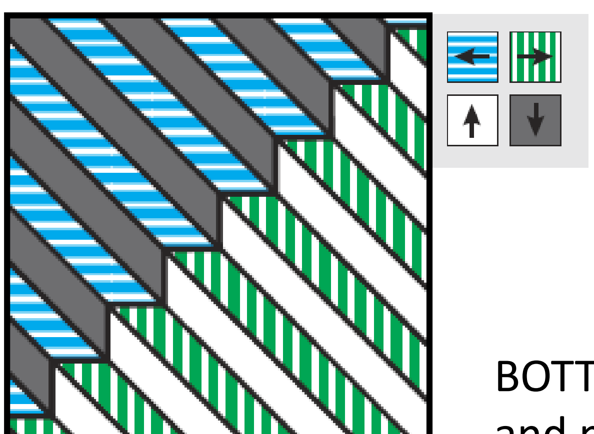
Research with new equipment

The sensitivity and resolution of the new Cypher S AFM allows to reveal details of the zig-zag structure of the interfaces between nanotwinned ferroelastic superdomains. They are likely to influence piezoelectric behavior of perovskite ferroelectrics.





TOP: $a_1/a_2/a_1/a_2$ domain structure in ferroelectric PTO thin films evidenced by the instrument: topography (a), PFM amplitude (b) and PFM phase (c) images.

BOTTOM: Identified domain arrangements with electrically and mechanically compatible walls (schematical)

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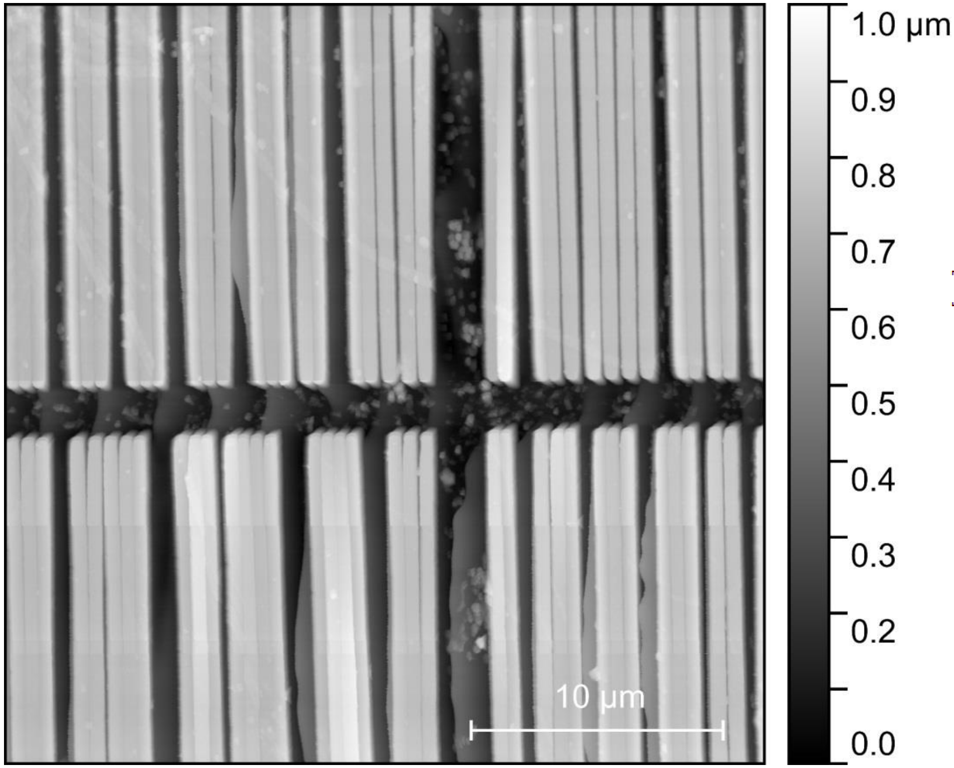
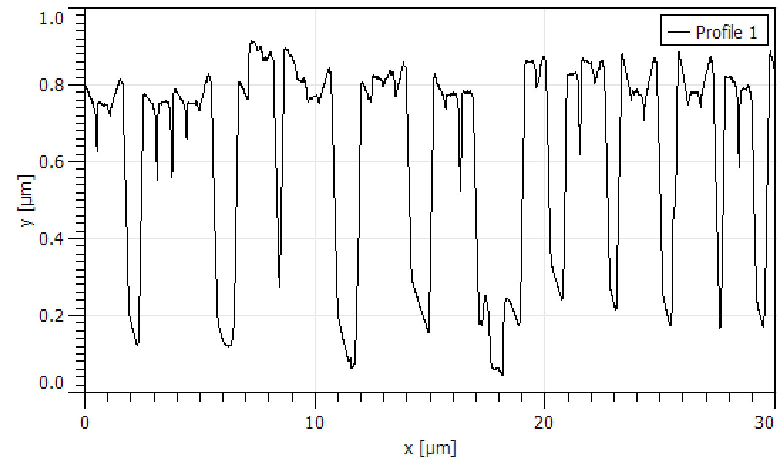
Research

The instrument will also serve to support activities of VP2.





Profile of the topography image

Topography image 30x30 µm of the GaAs nanobars obtained using the Cypher S AFM microscope allows estimating the distribution of the nanobars and their height and thickness, important feedback for THz spectroscopy experiments designed within VP2.

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RA5

Multifunctional liquid crystalline materials and composites

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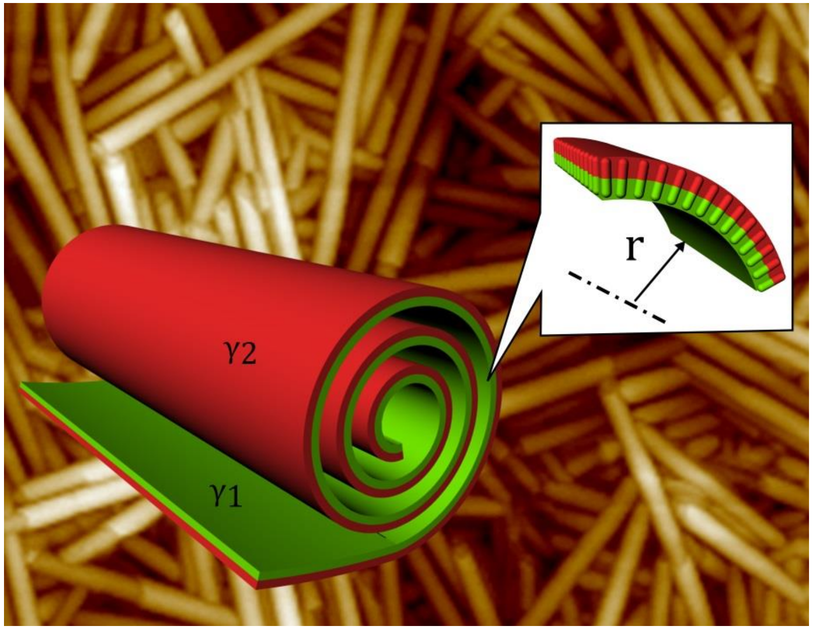
Research



- We prepared and studied new self-assembling systems, focusing on identifying new effects and understanding the structure-properties relations.
- We studied photosensitive liquid crystalline compounds, in which the illumination of light with specific wavelength induced change of the molecular shape and thus the molecular ordering in a mesophase.
- We found a facile route how to prepare the nanotubes from organic mesogenic molecules dissolved in a typical solvent. For selected types of chiral rod-like molecules, both enantiomers as well as the racemic mixtures formed the nanotubes by slow evaporation from a solution.

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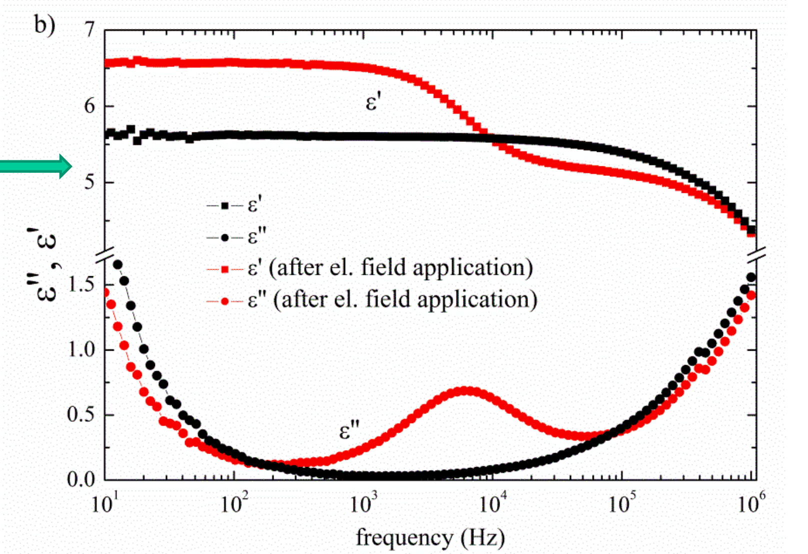
Equipment

In 2019 we purchased the **impedance analyser Solartron1260A**.

This equipment allows us to perform **broadband dielectric spectroscopy** and to obtain more information about electro-optical parameters of studied compounds. This type of **multifunctional equipment** is working in a broad frequency range, provides low noise, good resolution, possibility to measure in a bias voltage. We started to measure dielectric properties of new liquid crystalline compounds or composites.

Apparatus

Dielectric data measured for new studied compound



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Technology

Equipment purchase

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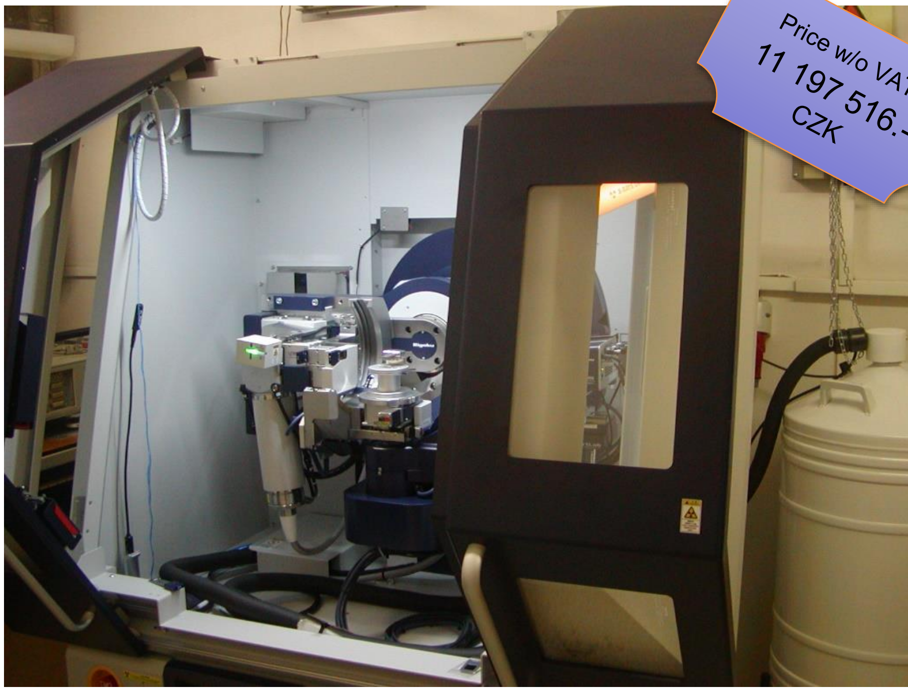
Texture diffractometer



Price w/o VAT:
11 197 516.-
CZK

- Designed as universal (convertible) diffractometer focused on in-situ (temperature) texture measurements
- Allows to quantify phase transformations and reorientation of functional materials with temperature or other load (not a part of the solution).
- Measurement of surface residual strain possible with ~ 0,1 mm position precision on the sample.
- Standard powder (polycrystalline) operational modes with fast data acquisition possible:
  - Phase identification
  - Phase transformations
  - Structure refinement for powder samples

…currently under commissioning in temporary premises...

Rigaku SmartLab

•Cu/Mo sealed tubes alternatively
•Bragg-Brentano, parallel beam geometry or low resolution collimated beam for both tubes
•High resolution collimated beam for Cu radiation
•Non ambient chamber -180 to +500 ºC
•Eulerian cradle / out of plane detector arm
•Area detector, smallest beam spot of 150 µm

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X-ray microscope

For delivery into the new pavilion (second quarter 2021 soonest), 2 prospective solutions:

Zeiss Xradia Versa

ThermoFisher Scientific Heliscan micro CT

Diffraction contrast with limited capabilities

W/O DCT so far (in negotiation)

- Capabilities of laboratory Diffraction Contrast Tomography are still under investigation and evaluation with regards to scientific goals of the project.
- The scope of vendors of micro CT (X-Ray microscopes) without DCT is much broader (e.g. Rigaku as well) offering perspective to save money for other instrument with more reliable outcomes.

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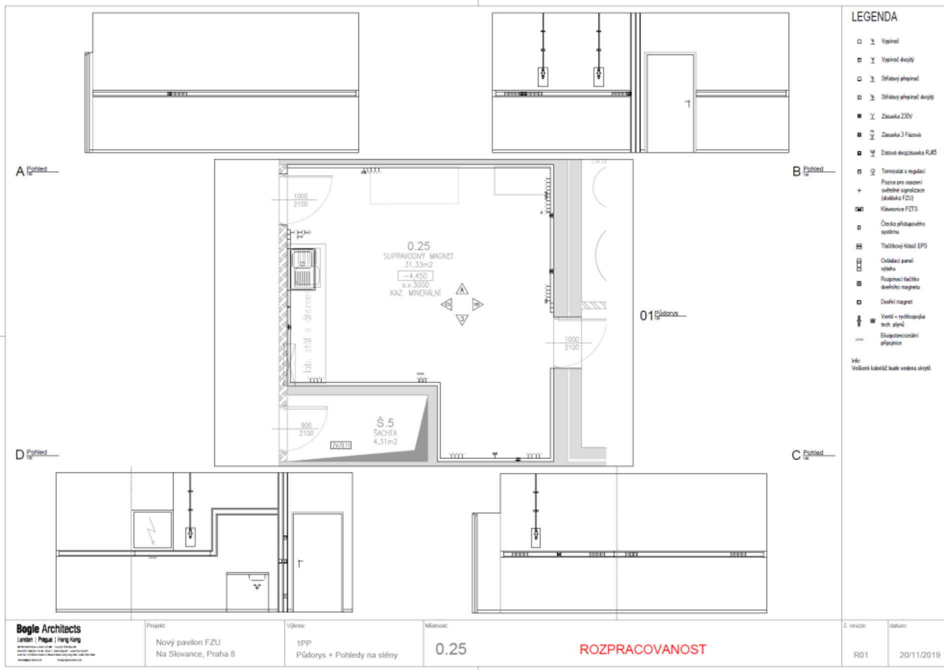
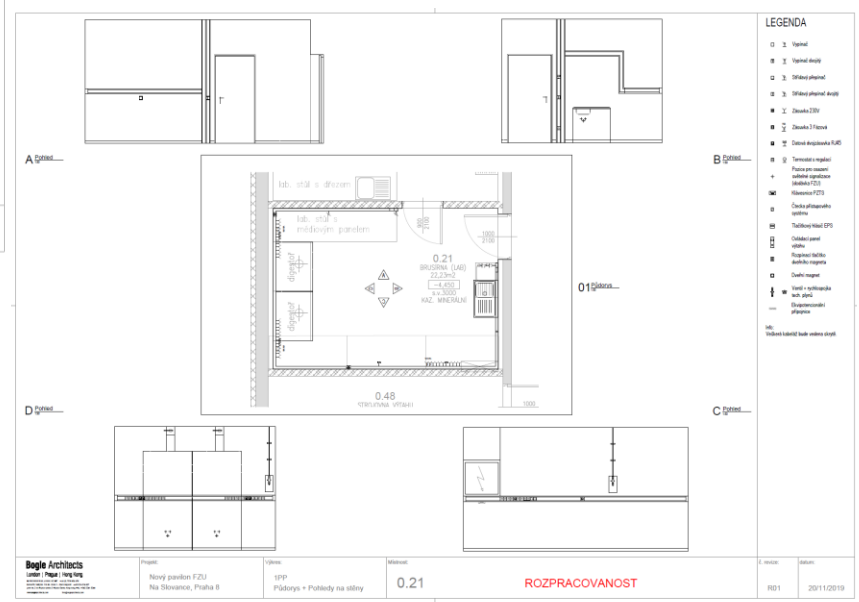
Laboratory arrangement (example)





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The End

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