Research competencies of CoE NOMATEN Materials Research Laboratory at National Centre for Nuclear Research Poland

NOMATEN
Centre of Excellence in Multifunctional Materials for Industrial and Medical Applications

Narodowe Centrum Badań Jądrowych
National Centre for Nuclear Research
ŚWIERK

NOMATEN Centre of Excellence

The overall goal of the CoE NOMATEN is to support the Research and Development in Multifunctional Materials for Industrial and Medical Applications

CoE NOMATEN will develop a Long-term Science and Innovation Strategy in Multifunctional materials by focusing on two strategic research and innovation topics

Novel high-temperature, corrosion and radiation resistant materials for industrial applications

Novel radiopharmaceutical materials for medical applications

European Funds Smart Growth
Republic of Poland
European Union Horizon 2020 Framework Programme
European Union European Regional Development Fund

NOMATEN Centre of Excellence

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NOMATEN CoE Materials Research Groups

Complexity in Functional Materials
Functional Properties
Materials Characterization
Materials Structure, Informatics and Function

Novel high-temperature, corrosion and radiation resistant materials for industrial applications topic is focused on:

Material synthesis *– to develop key materials for High-Temperature applications and be able to connect to leading partner and other groups for providing samples and establishing collaborations*

Functional properties of materials *– to investigate materials range from heat resistance to mechanical properties as friction, resistance to chemical environments and radiation and finally to their combined multiphysics effects*

Analytics and characteriztion – *to quantify the properties of materials after synthesis and characterizing advanced functional materials important for high-end customers*

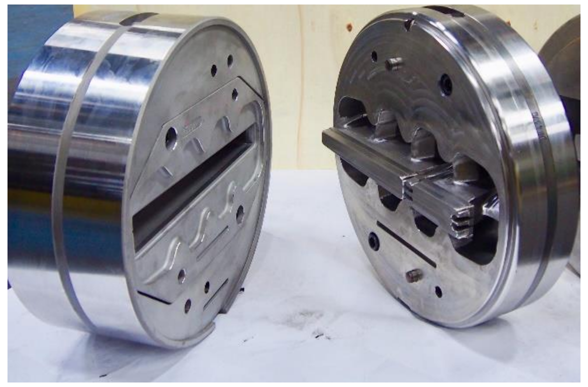
European Funds Smart Growth
Republic of Poland
European Union Horizon 2020 Framework Programme
European Union European Regional Development Fund

The CoE NOMATEN Materials Research Laboratory offers research and engineering problems solving using advanced and unique research infrastructure in following areas:

- *NDT testing of materials and welded joints with accredited procedures*
- *Macro- and microstructure characterization and metallographic analyses with LM, SEM and TEM techniques*
- *X-ray diffraction phase analysis*
- *Mechanical testing of materials with accredited procedures*
- *Nanohardness testing*
- *Thermal properties testing*
- *Chemical composition and elemental analysis*
- *Synthesis of PVD layers and coatings*
- *Analysis of thin layers and coatings with Raman spectroscopy*
- *Surface modification of materials with Ion implantation techniques*
- *Engineering consulting in the field of Materials Science, Surface Engineering, Corrosion Science, Heat Treatment Technologies of metals and alloys….*

Nuclear and Conventional Power Industry

Tooling Industry



Aerospace Industry

Biomaterials and Implantology

Automotive Industry

Oil and Gas Industry

Materials Research Competencies
CoE NOMATEN Materials Research Laboratory

Materials Research Divisions

- ***Non-Destructive Testing NDT***
- ***Mechanical Testing Division***
- ***Structure and Corrosion Research Division***
  NOMATEN SEM / TEM Laboratory
  NOMATEN XRAYLAB Laboratory
- ***Thermal Properties Testing Laboratory***

CoE NOMATEN Materials Research Laboratory
Non Destructive Testing – NDT Services

Non-Destructive Testing NDT Division – Research Infrastructure

The non-destructive testing division carries out tests with using following methods:

- Visual test method (VT)
- Penetration testing method (PT)
- Magnetic particle method (MT)
- Ultrasonic method (UT)
- Eddy current method (ET)
- Magnetic permeability testing

NDT Testing Research Infrastructure

UT Defectoscope Olympus OMNISCAN MX2

ET Defectoscope Olympus NORTEC 600D

Foerster MAGNETOSCOP 1.069

Accredited NDT Testing
Polish Centre for Accreditation
Accreditation number AB 025

PCA POLSKIE CENTRUM AKREDYTACJI BADANIA
AB 025

ilac-MRA

Financed by
PROJECT HTGR

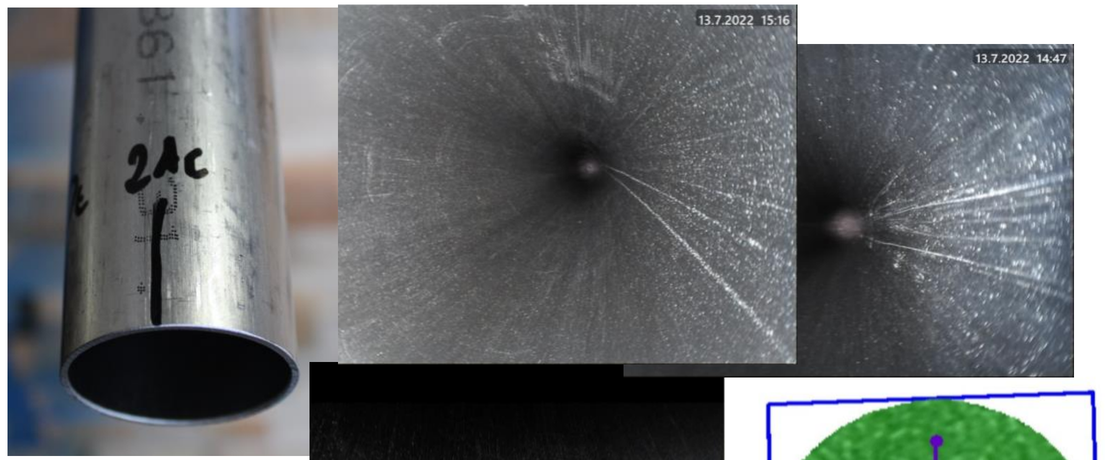
VT Flexible Videoendoscope
Mentor Visual iQ - Waygate Technologies

Penetrant method

Magnetic particle method

Non-Destructive Testing NDT Division – Research Activities

AISI 316L seamless pipes NDT testing for ITER Blanket System components (First wall panels cooling system) – commissioned by ITER's supplier BIMO TECH



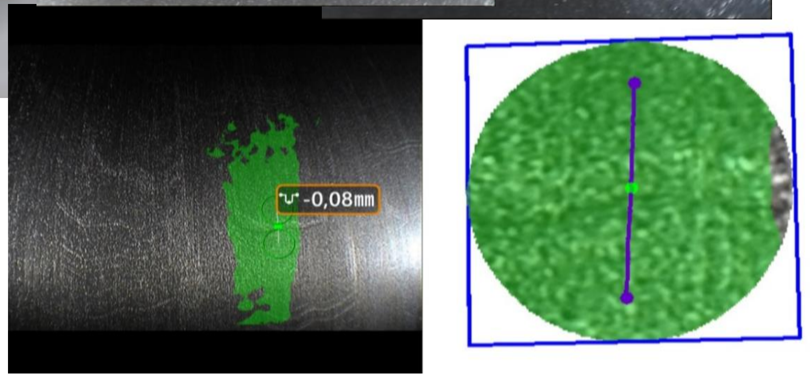
Accredited NDT tests realized according to:

FF9U2X Technical Specification X2CrNiMo17-12-2, (AISI 316L) Tube for Blanket Application

Supply of Normal Heat Flux First Wall (FW) Panels for ITER Blanket System INSPECTION NOTIFICATION

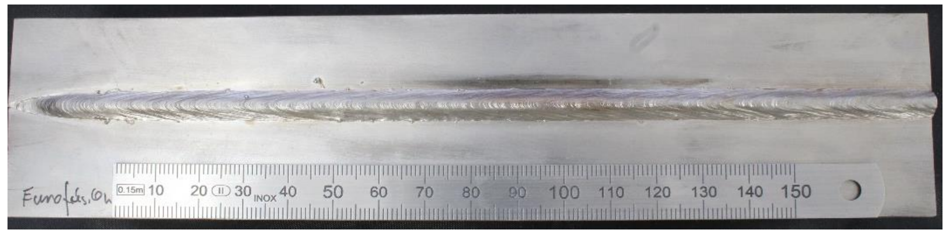
***Visual Testing VT** – visual inspection outer / inner surface*
***Ultrasound tchickness test UTT** – wall tchickness*
***Direct meaurements** – pipes dimensions*

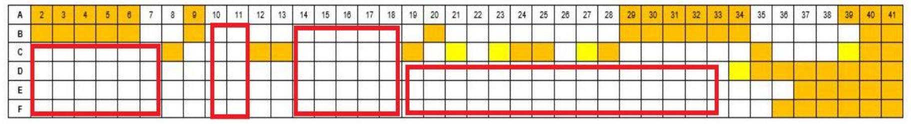
Magnetic Permeability Test according to IEC 60404-15 and ASTM A342M

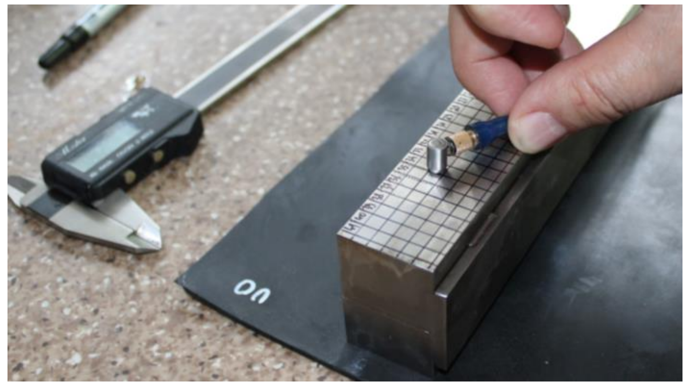


Cold drawing effects depth measurements
Analysed by VT Flexible Videoendoscope

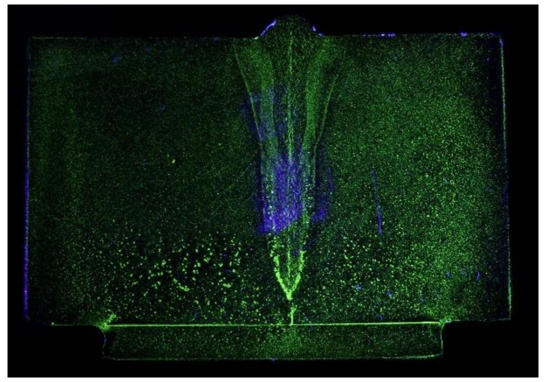
EUROFER 97 after Electron Beam Welding NDT testing in cooperation with Karlsruher Institut für Technologie







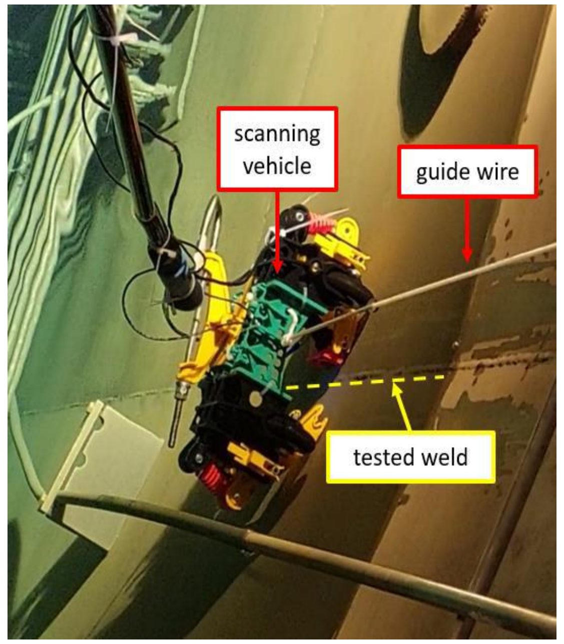
Ultrasound testing UT



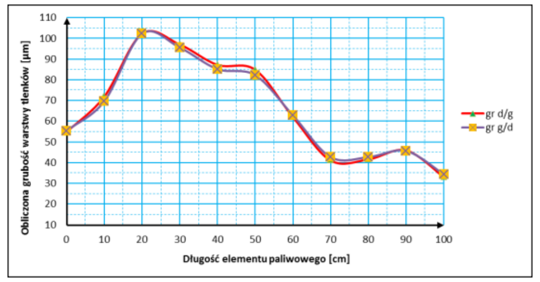
Magnetic particle inspection MT

MARIA Reactor NDT Inspections

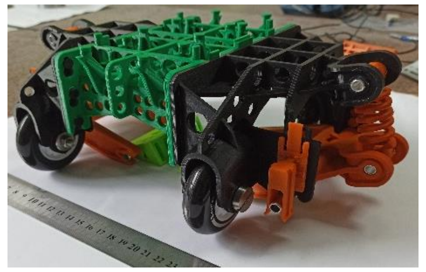
Welded joints on secondary circuit piping with UT, VT, MT



VT, UT reactor pool weld joints inspection



Thickness evaluation of Al_2O_3 layer of the fuel element shells, Eddy Current Testing



MARIA reactor weld joints UT scanning vehicle designed by Reactor and MRL engineers

Research Nuclear Reactor MARIA upgrade – Neutron Diffractometers / NDT Testing

Reaktor MARIA

New Large-Scale Scientific Instruments for the MARIA reactor….Installation already started …..ready to use in 2024

NCBJ cooperation with Helmholtz-Zentrum Berlin

HZB Helmholtz Zentrum Berlin

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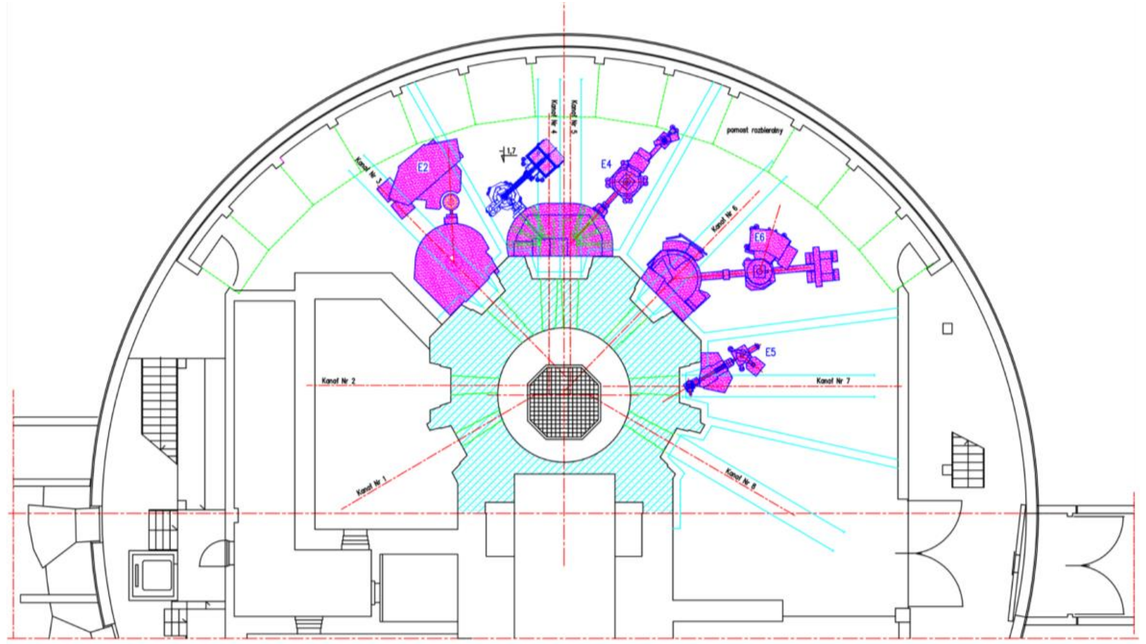
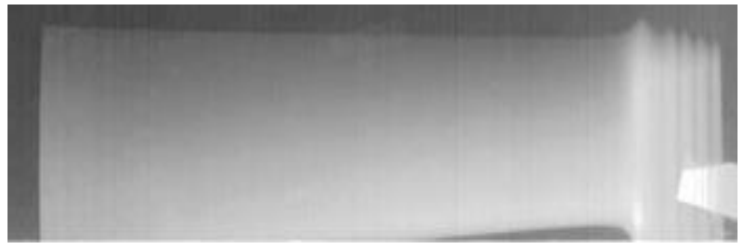


Visualization of the MARIA reactor experimental hall after upgrading



NR of Aircraft Engine Turbine Blade

Source: IAEA-TECDOC-1604

NR of Diesel Engines Nozzle

Source: IAEA-TECDOC-1604

H1	*Thermal neutron*	*Spectrometer*
H2	*Flat Cone*	*Diffractometer*
H3	*Residual*	*Stress Analysis Diffractometer*
H4	*Two-Axis*	*Diffractometer*
H5	*Four-Circle*	*Diffractometer*
H6	*Focusing*	*Diffractometer*

Materials Research with new Neutron Infrastructure

- *Neutron Radiography NR – NDT Testing of ready elements i.e. Microcracks analysis, Porosity after casting, Weldments quality control etc.*
- *Analysis of internal and residual stresses deep within a crystalline material*
- *Determination of the atomic and magnetic structure of a crystalline solids, gasses, liquids or amorphous materials.*
- *Measurements of highly-textured elements*

CoE NOMATEN Materials Research Laboratory Mechanical Testing Services

Mechanical Testing Division – Research Infrastructure

Materials hardness testing at micro- / nanoscale

Accredited Mechanical Testing
Polish Centre for Accreditation
Accreditation number AB 025

PCA Polskie Centrum Akredytacji BADANIA
AB 025

Semi-automatic Zwick/Roell DuraVision G5 hardness tester

Financed by
PROJECT HTGR

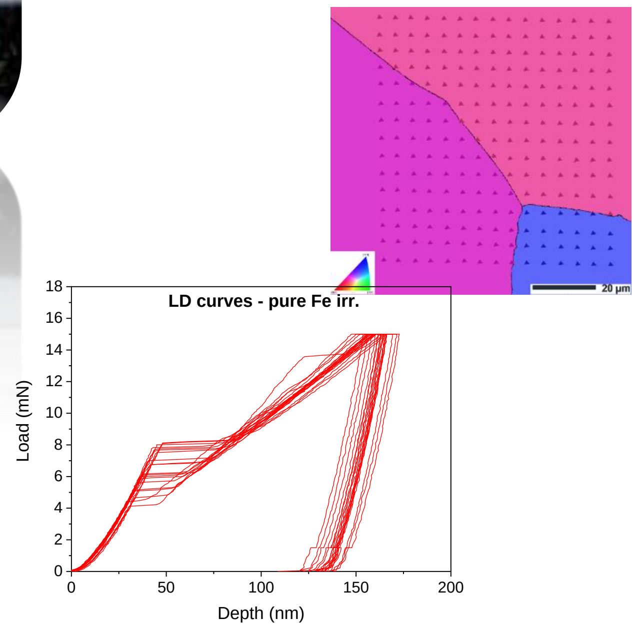
- Low-load hardness testing
- Load range 0.3-250 kg
- Brinell HB according to ISO 6506 (ASTM E10) 2.5/5 mm ball
- Vickers HV according to ISO 6507 (ASTM E-92)
- Rockwell HR.. according to ISO 6508 (ASTM E-18) - A,B,C,L,N,T scales

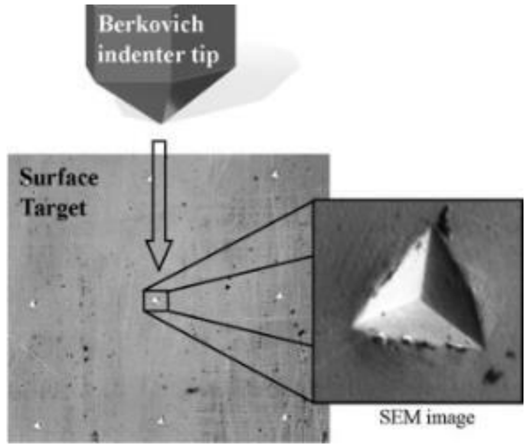
Microhardness tester HV1000 MEGA Instruments (Suzhou) Co., Ltd

- Load range 10 – 1000 G (HV0.01 – HV1)

Nanohardness tester NanoTest Vantage by Micro Materials Ltd., Wrexham UK

- **Berkovich, Vickers, Cube Corner and Conical** type indenters available for RT testing
- **HT measurements** with diamond (up to 450°C) and cBN (up to 750°C) indenter Measurements under **controlled argon atmosphere**
- **Humidity cell**
- Coupled **Atomic Force Microscope**
- Optical microscope (up to 40x mag.)
- Convers range forces from 0.1 mN to 20 N
- Load or depth-controlled mode
- **Single forces or Load Partial Unload**





Mechanical Testing Division – Research Infrastructure

Static and dynamic strength testing

INSTRON Universal Testing Machine

- Servohydraulic (static/dynamic testing)
- Load capacity ±100 kN
- Class 0.5 starting from 200 N
- Clip-on extensometers class 0.5
- AlignPRO Alignment Fixture provides full angularity and concentricity adjustment while load is applied to the specimen
- Additional 1kN load cell

Temperature test chamber with cooling module
Temperature range:
from **-150°C to +350°C**

Three-Heating zone split furnace
Nominal maximum
specimen temperature: **1000°C**

Accredited Mechanical Testing
Polish Centre for Accreditation
Accreditation number AB 025

AB 025

Mechanical Testing Division realizes:

- ***Tensile testing***
- ***Compression testing***
- ***Fracture toughness testing K_{IC}, critical CTOD, J_{IC} (CT25, SENB)***
- ***Determination of the rate of fatigue crack growth da/dN***
- ***Small Punch Test (SPT)***

All tests according to International Standards ISO, ASTM, BS…

SPT Small Punch Test:
Samples: ϕ3 x 0,25 mm discs
Punch: Ball ϕ = 1 mm
Temperature of test: ambient

Mechanical Testing Division future goals:

- Test samples miniaturization
- Testing of mm samples at HT with non-contact DIC extensometer !!!

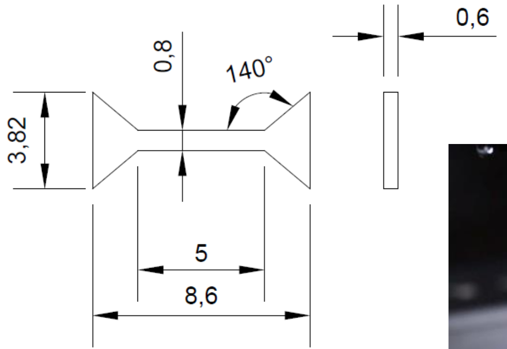
NCBJ Materials Research Laboratory – Mechanical Testing Division

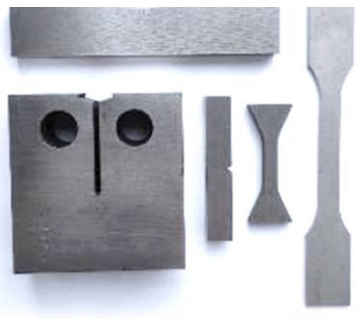
Small-scale samples preparation and testing

On-site samples machining by WEDM Electrical Discharge Machining

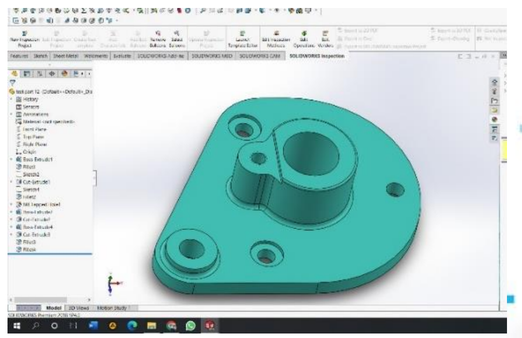
NEW WEDM Machining Center by the end of June 2023 !!!
(0.10 mm and 0.25 mm wire)

- Cuts any metallic conductive material
- **NO-FORCE PROCESS** (machining without surface effects and stresses regardless of material structure and hardness)
- **HIGH ACCURACY** machining +/- 2 µm
- **HIGH SURFACE FINISH** (by finishing passages implementation) up to Ra 0.2
- Cost-effective
- Possibility of cutting complex shapes (CAD/CAM inside)
- Possibility of cutting small and thin-walled samples





CAD/CAM Software



Financed by
PROJECT HTGR

GF Charmilles CUT E350 WEDM Machining Center

Miniaturized samples testing

Zwick/Roell Z020 AllroundLine

Static testing machine (20 kN)
Electromechanical
0.5 class starting from 20 N
Furnace up to 1000 °C
Non-contact extensometer
DIC software
Sub-sized tensile specimens
Alignment Fixture

NEW testing machines
Financed by
PROJECT HTGR

Dynamic testing machine (± 10-15 kN)
Resonance system
CT1/2”, CT1/4” and SENB
<100 mm samples
Alignment Fixture

Zwick/Roell Vibrophore 25

Mechanical Testing Division – Research Infrastructure

Charpy V Impact Testing

Accredited Mechanical Testing
Polish Centre for Accreditation
Accreditation number AB 025

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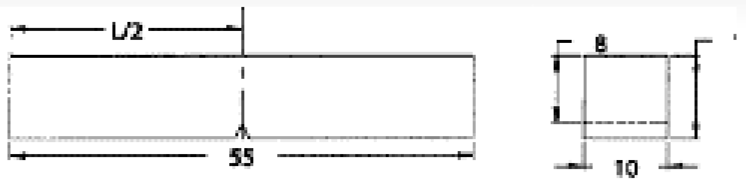
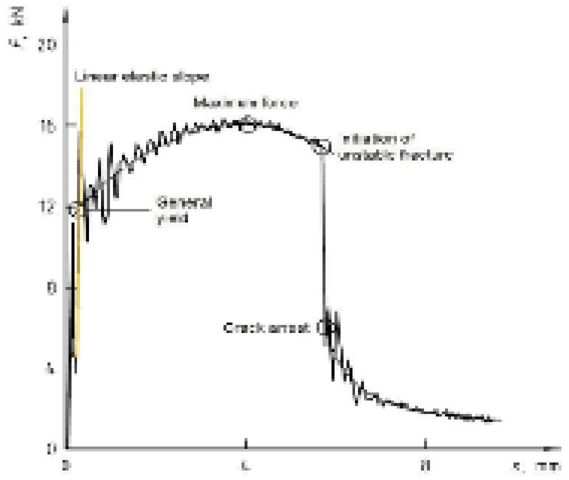
Zwick/Roell 450J Pendulum Impact Testing Machine

Standard samples 55x10x10 n2



Zwick/Roell 25J Pendulum Impact Testing Machine

- Miniaturized samples
- Instrumented (ISO 14556)



Impact tests at ambient, low (to -90°C) and elevated temperature (to 300°C)

- 2 mm striker
- According to :
  - ISO 148-1 and ASTM E23 (standard samples)
  - ASTM E2248 (miniaturized charpy V-notch specimens)
  - ISO 14556 (charpy V-notch instrumented test method – miniaturized samples)
- Dynamic fracture toughness K_{id}

Mechanical Testing Division – Research Activities

Mechanical testing of the high-speed railways in cooperation with Polish Railway Institute

70 IK years RAILWAY RESEARCH INSTITUTE

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NOMATEN Centre of Excellence in Multifunctional Materials for Industrial and Medical Applications

High-speed railways with speeds over 200 km/h, are one of the most demanding systems that must several requirements as: Safety, Durability and cost efficiency, Minimum acoustic impact

To achieve these objectives, the high-speed rail manufacturing process aims to meet the following requirements:

- Fracture mechanics – involves the initiation and growth of a crack, which can cause the material to break at a stress below its ultimate strength in the crack-free condition.
- Chemical, mechanical and structural homogeneity
- Uniformity of dimensions
- Absence of surface and internal defects
- Impact strength, wear and fatigue resistance
- Residual stresses
- Good Weldability

Accredited Mechanical Testing
Polish Centre for Accreditation
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PCA Polskie Centrum Akredytacji BADANIA
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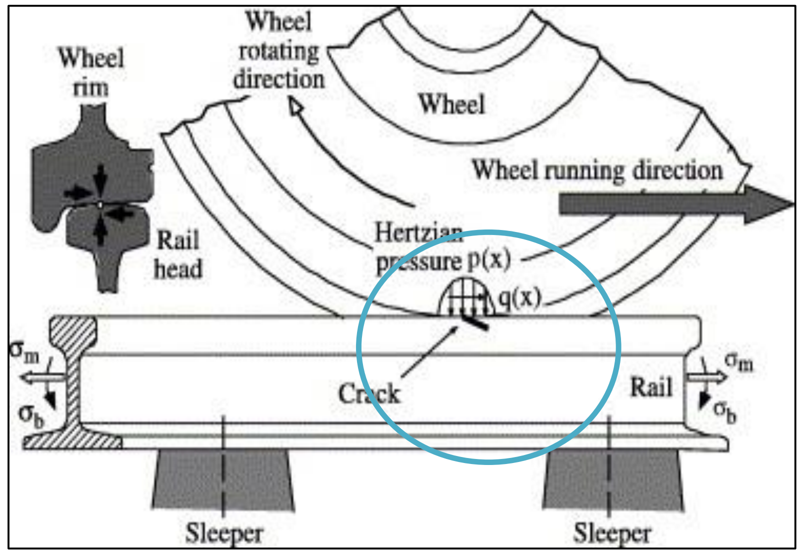
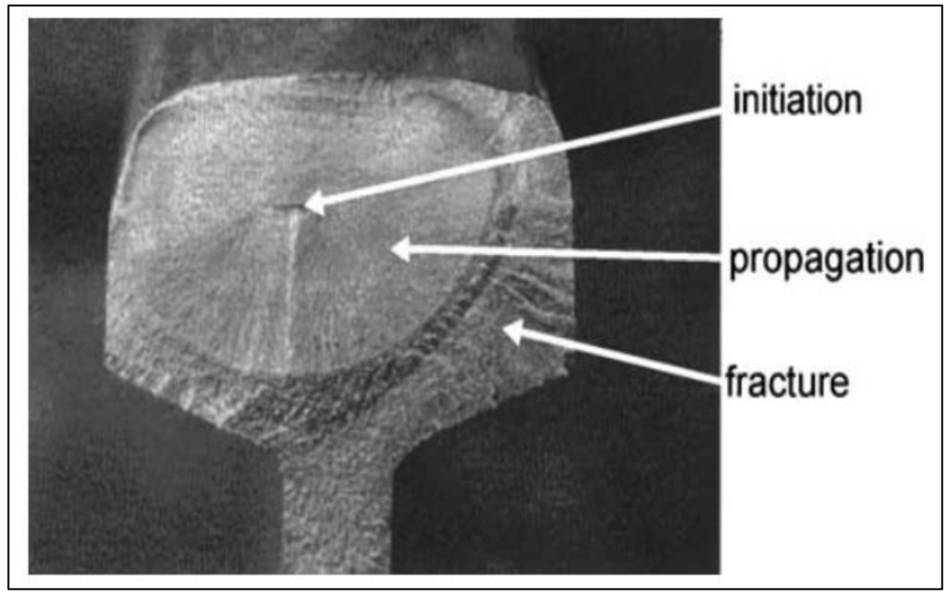


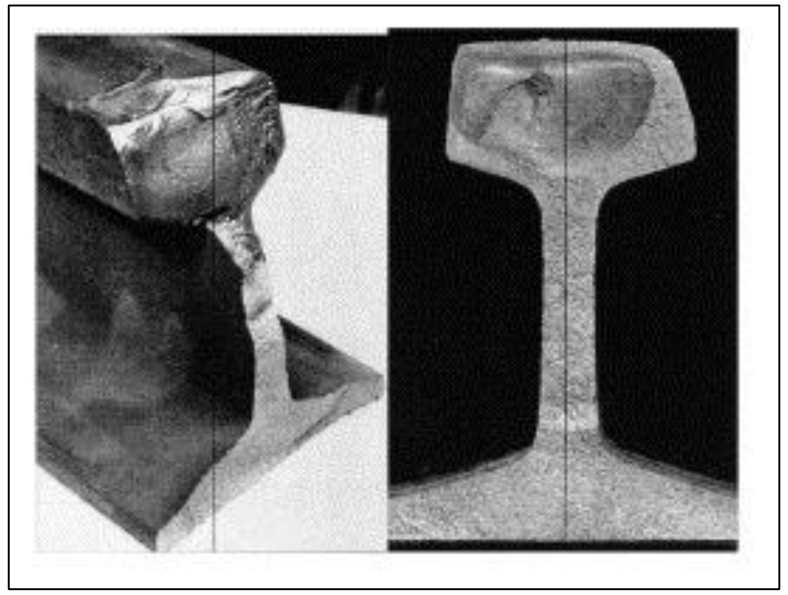
Cracking mechanism and testing of railways Stresses and Bielajew's point analysis

High-Speed ICE Pendolino train, max. speed 250 km/h

We realize the complex fracture mechanics accredited tests for railway systems since 2004…

- ✓ We measure the ***Rate of crack propagation (da/dN) – acc. ISO 12108***
- ✓ We evaluate the ***Critical stress intensity factor K_{IC} at lowered temperature – acc. ASTM E399***





Macroscopic observations of cracks and analysis of rails breakthrough

High-Speed ICE train disaster, Eschede, (Germany,1998)

Mechanical Testing Division – Research Activities

Studying effect of ion irradiation and temperature on the properties of Ferritic / Martensitic steels

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Samples: Pure Fe; Fe9%Cr; Fe9%Cr-NiSiP, Eurofer 97
Ion irradiation in HZDR up to 8MeV Fe ions, 5 dpa, temp. 300 (and 450°C)
Techniques: Nanoindentation at rT and HT; SEM+FIB/EBSD & TEM; XRD & MD simulations

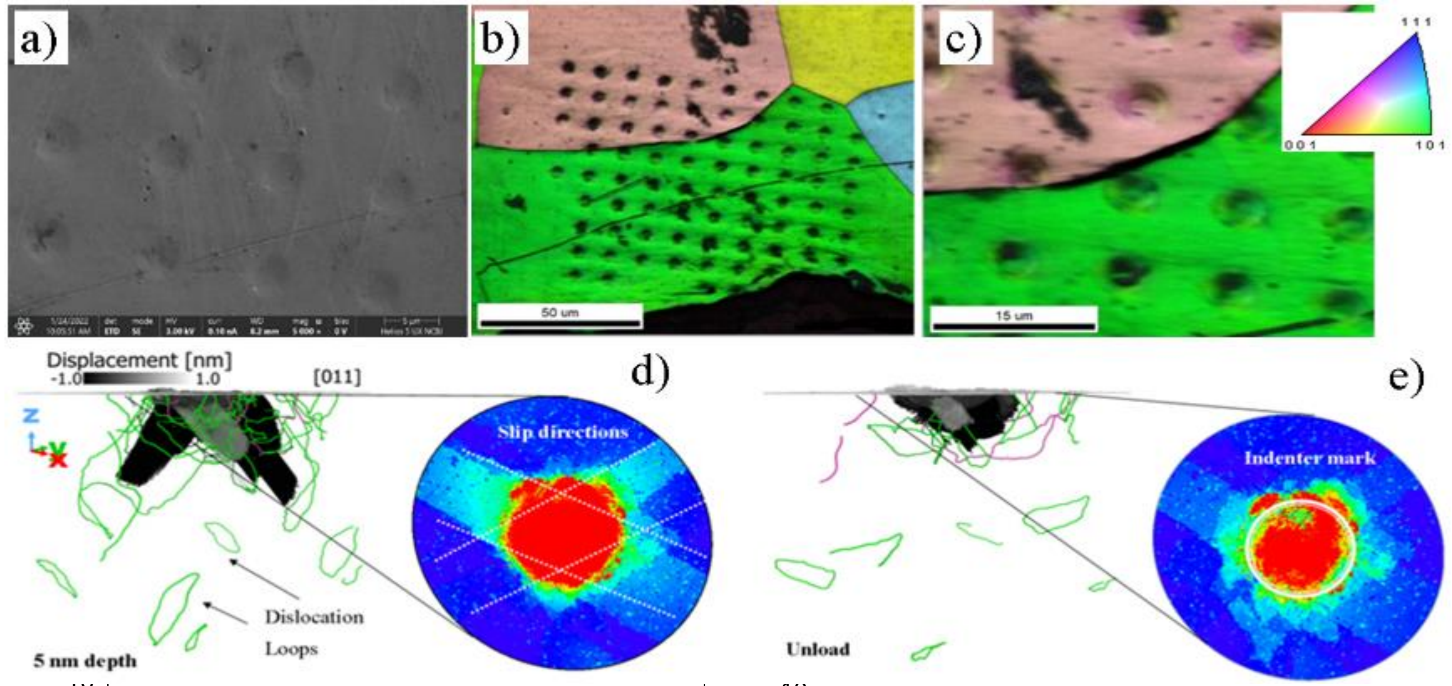
Results:

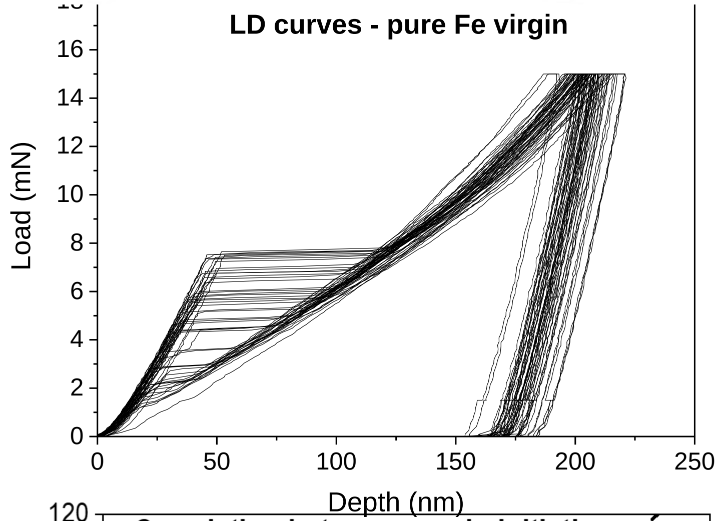
- Elastic analysis based on the Hertz revealed that the first pop-in is typically caused by plasticity initiation
- Calculated shear stress is about 3 GPa (theoretical strength)
- Interstitial atoms like C influence pop-in behaviour by blocking preexisting dislocations

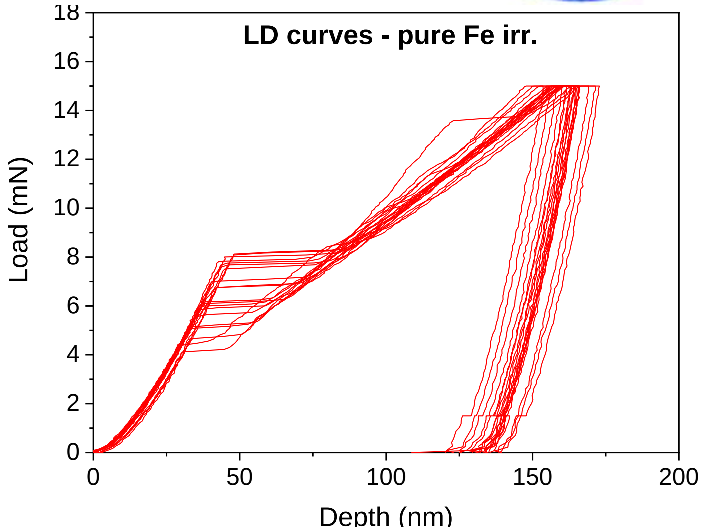
Mechanisms to consider:

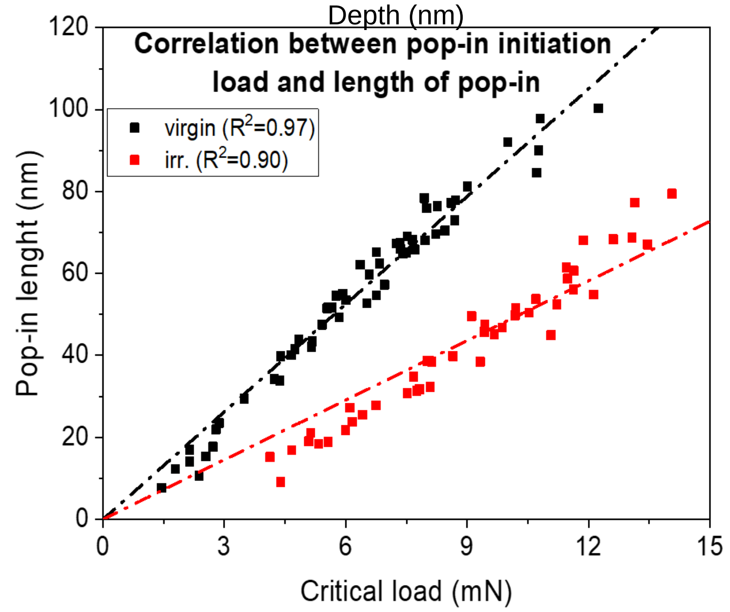
- Dislocation nucleation at neighboring grain, unlocking pinned by C atoms dislocations at grain boundaries, slip transfer?
- Do we see the impact of crystal orientation?

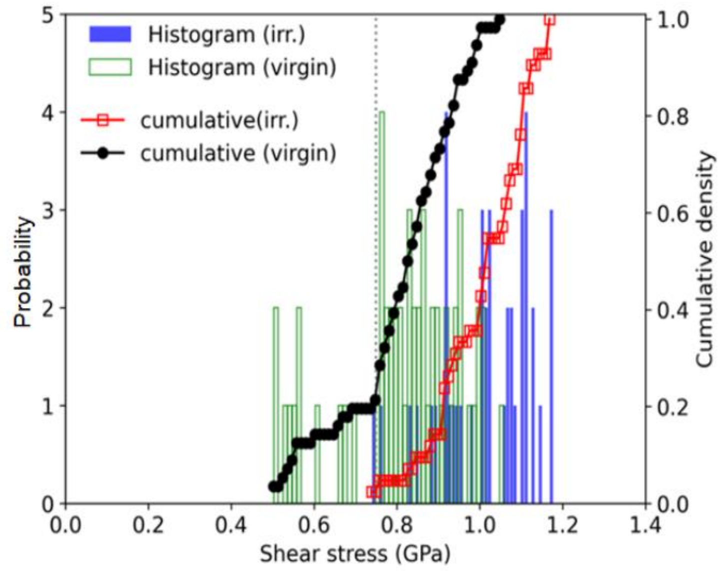
Spherical nanoindentation (R_i=25 µm)

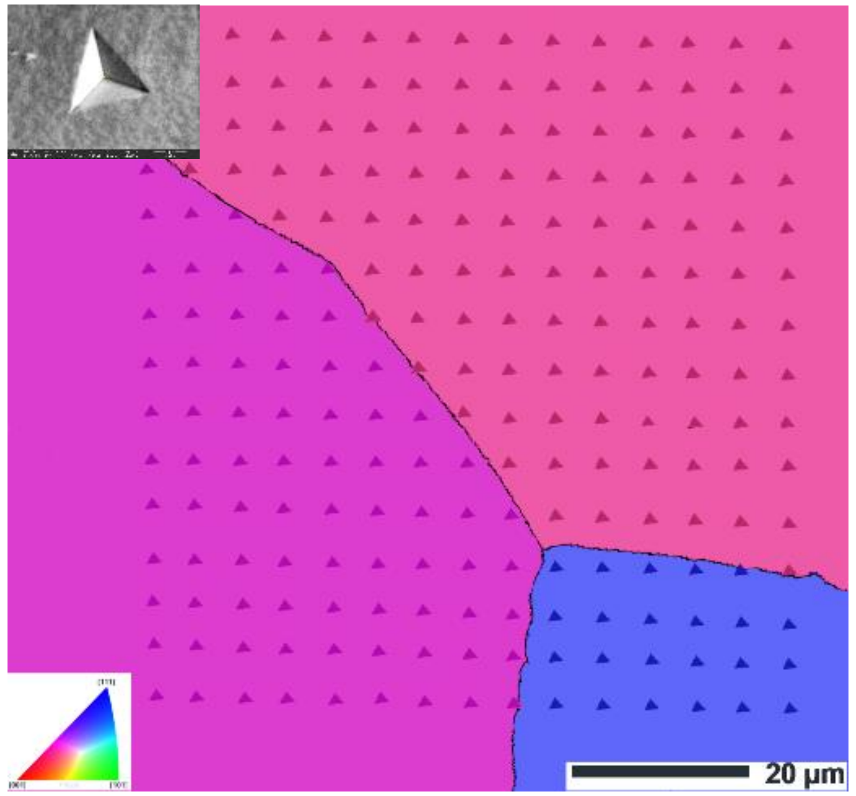


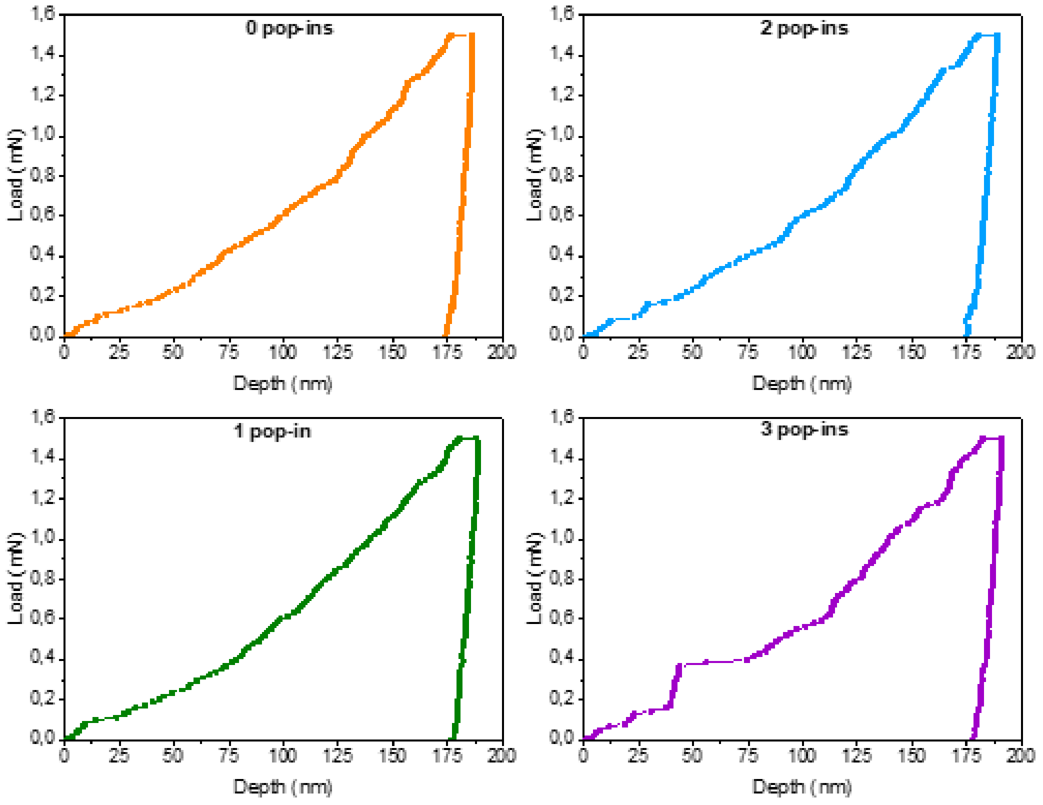












CoE NOMATEN Materials Simulation Division

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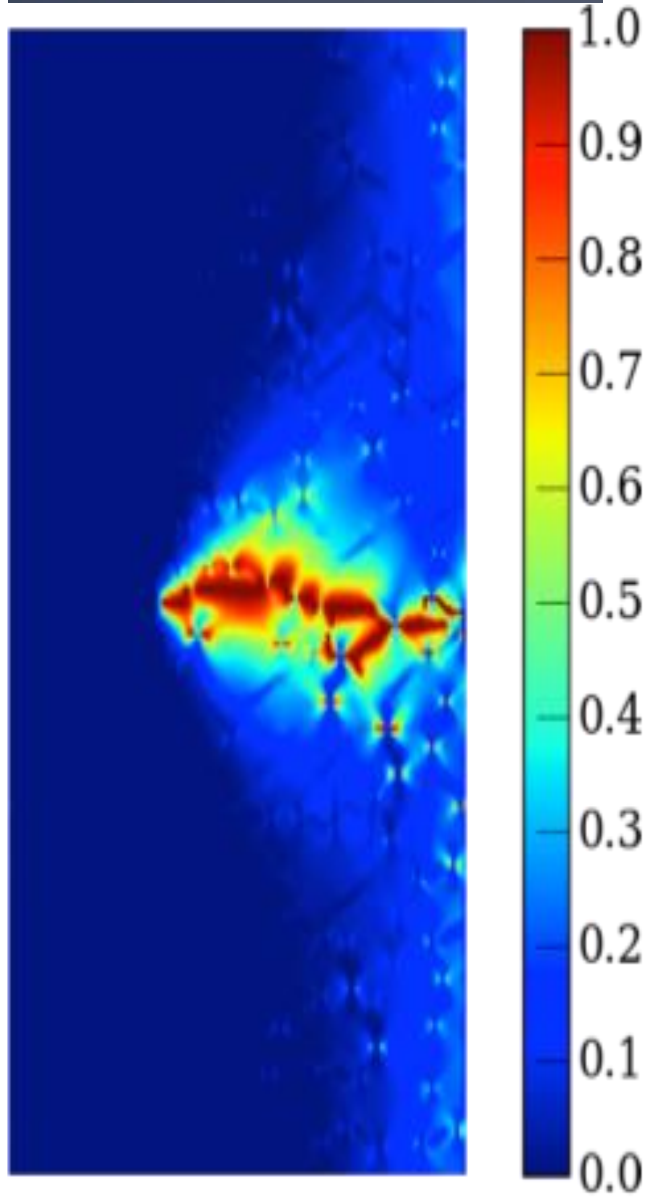
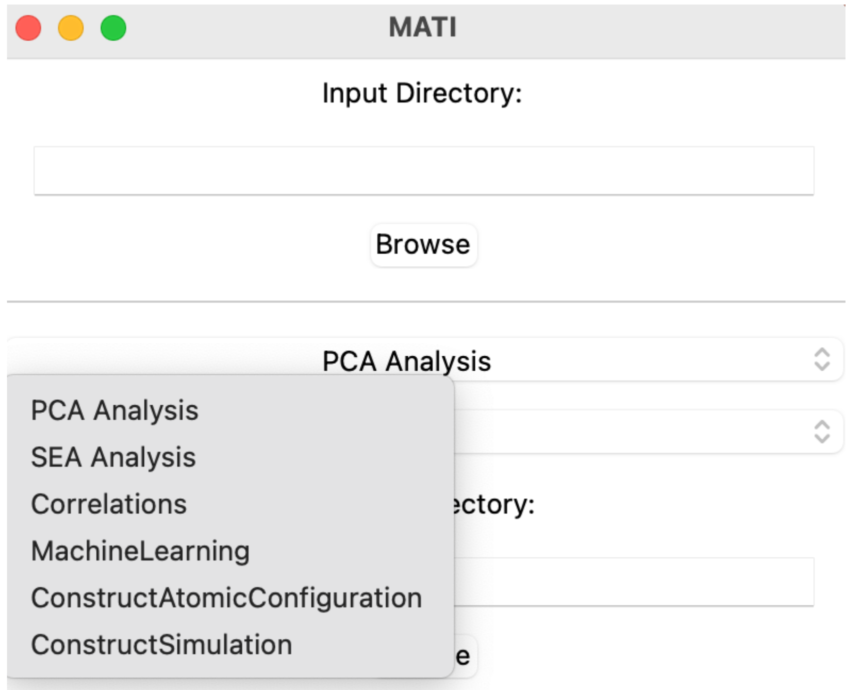
Simulations at NOMATEN CoE

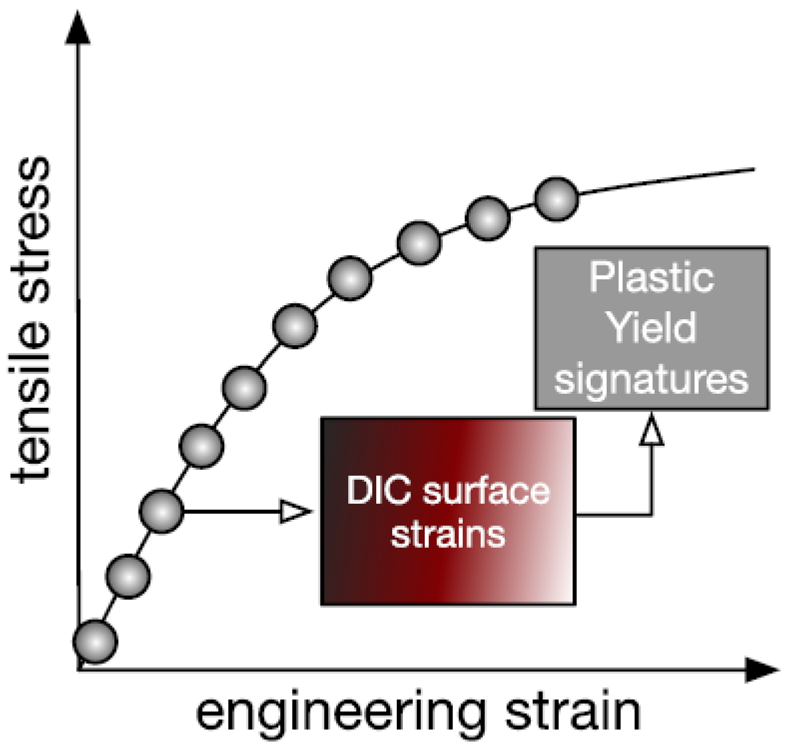
Plasticity

Analysing digital image correlation with statistical techniques and machine learning

MATi MATERIALS INFORMATICS







Predicting mechanical response and microstructure evolution during plastic deformation

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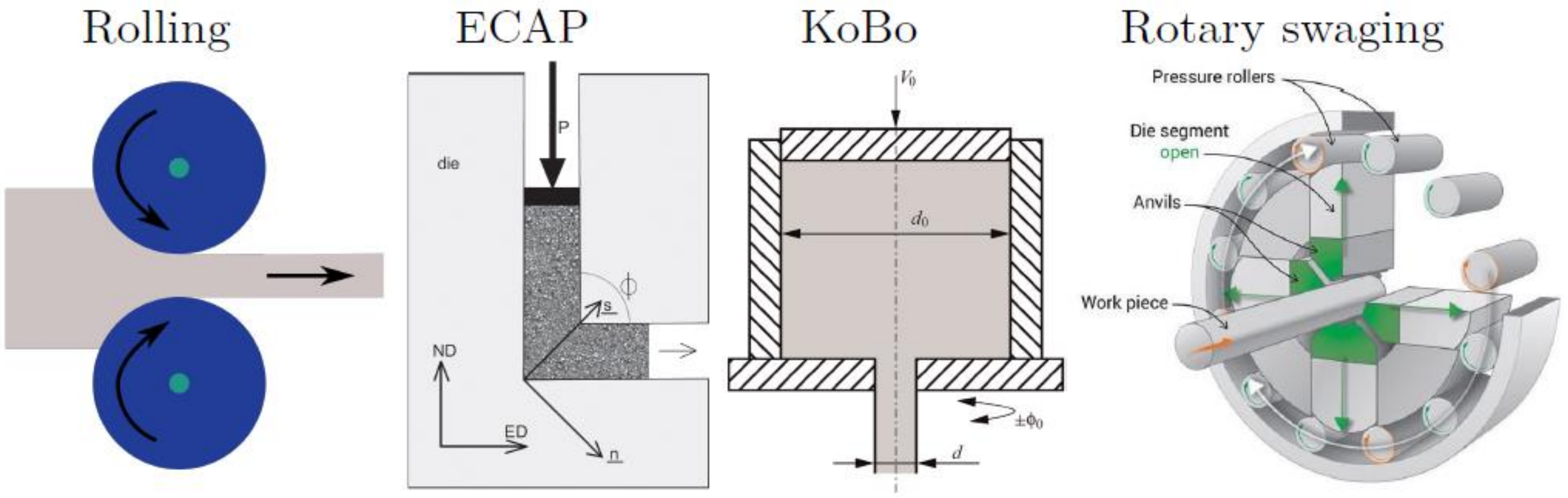
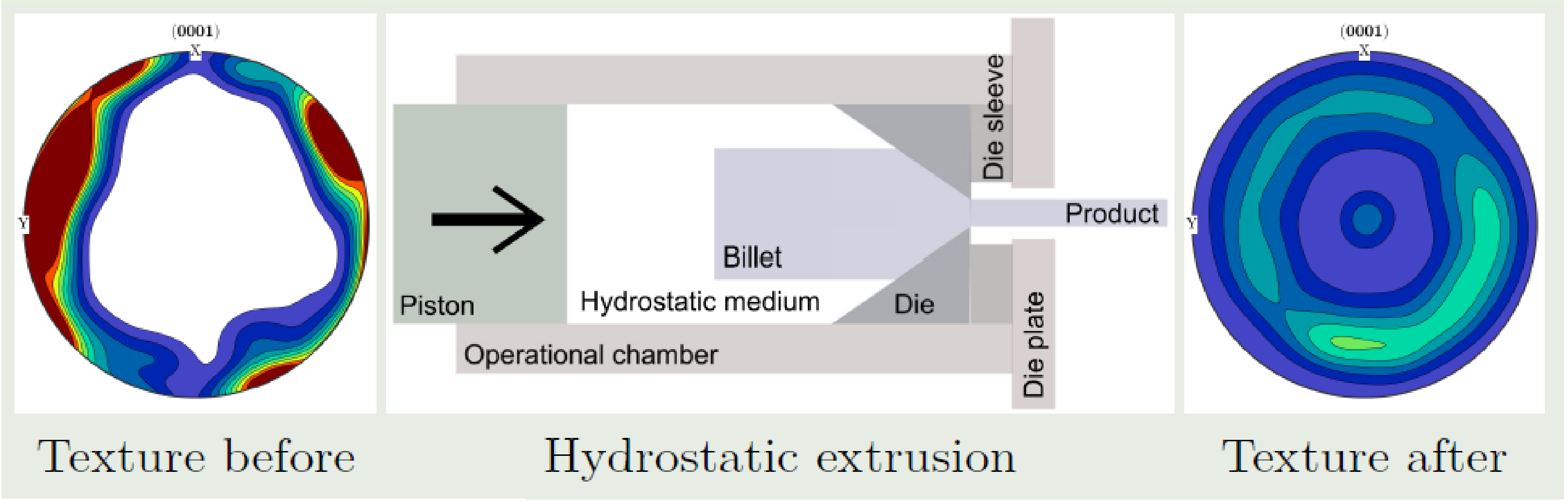
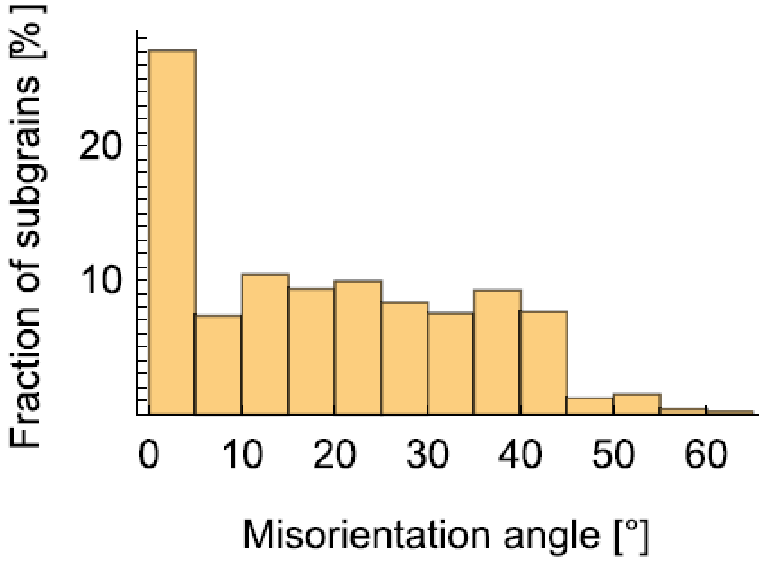
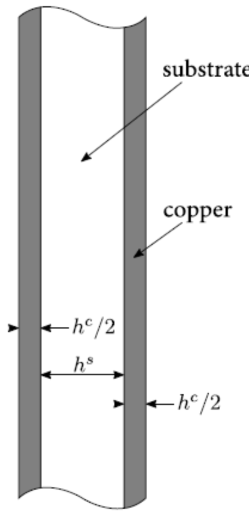
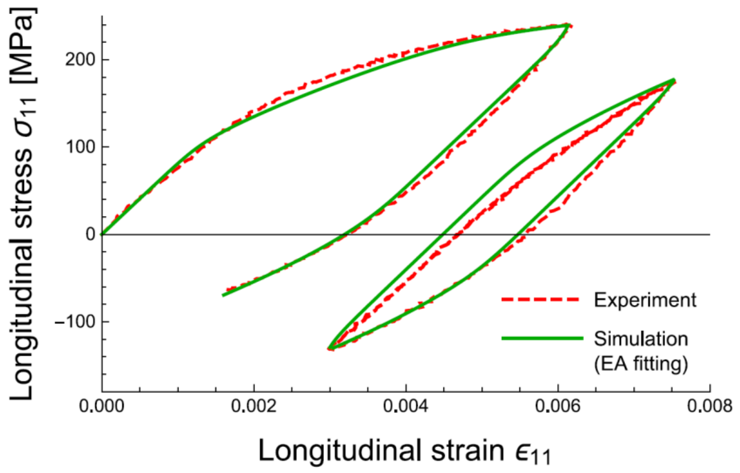






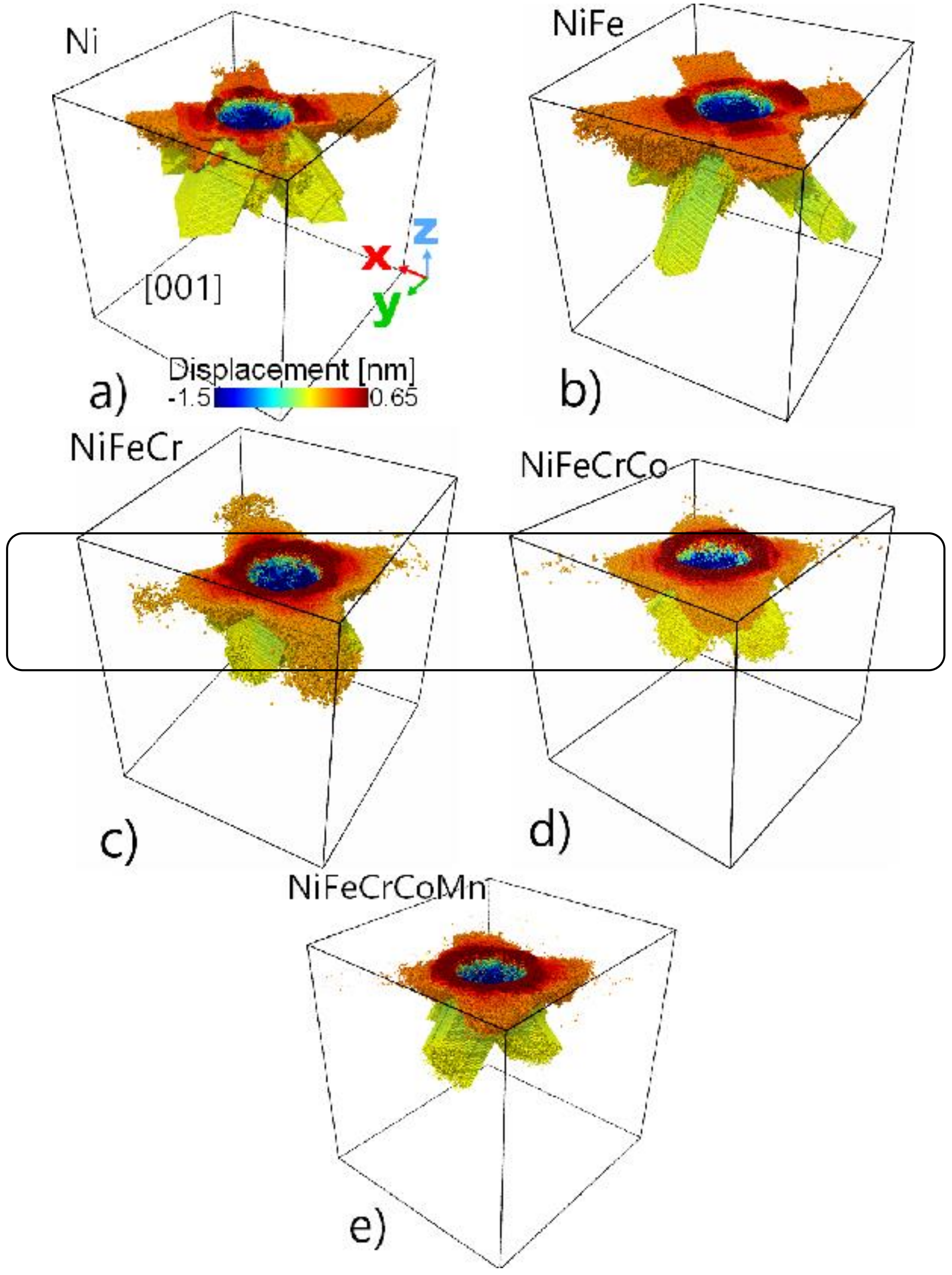




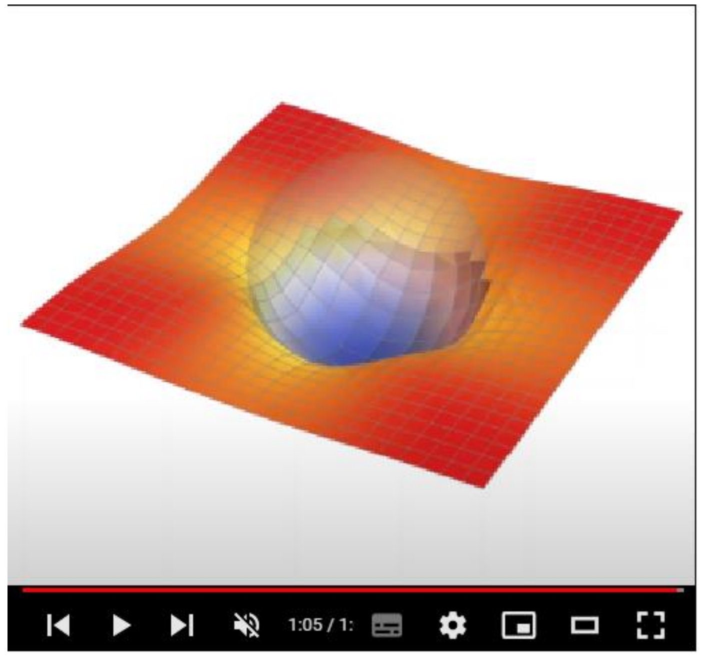
Simulations at NOMATEN CoE

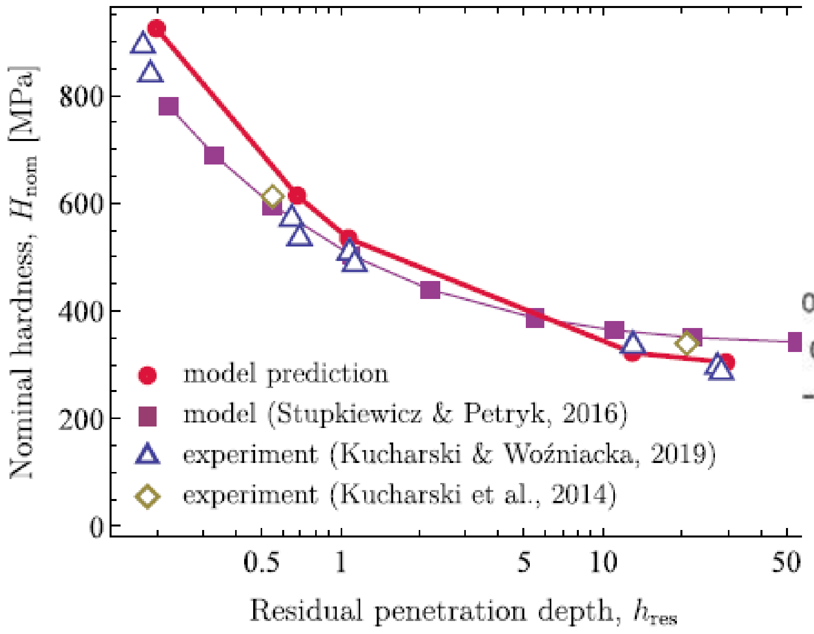
Micro- and nanoindentation

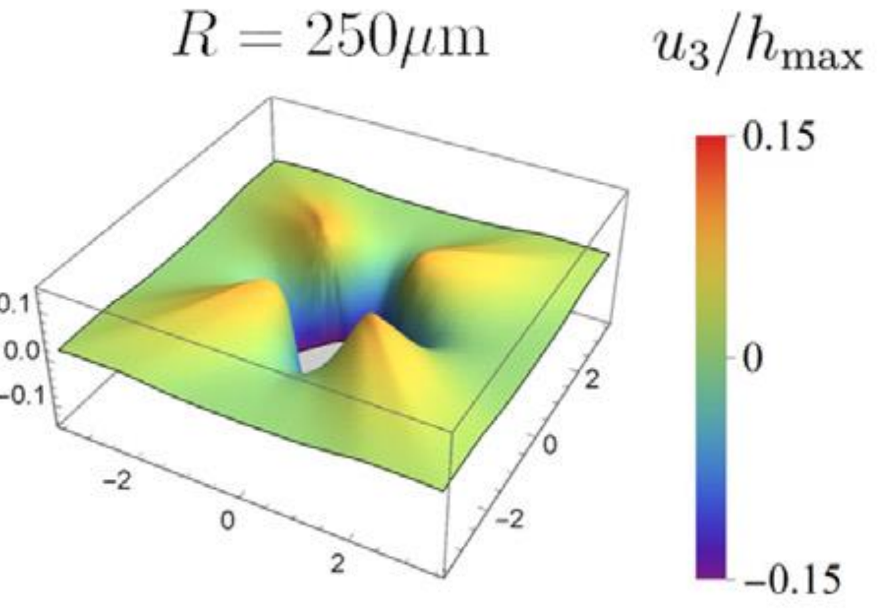
Atomistic simulations of nanoindentation



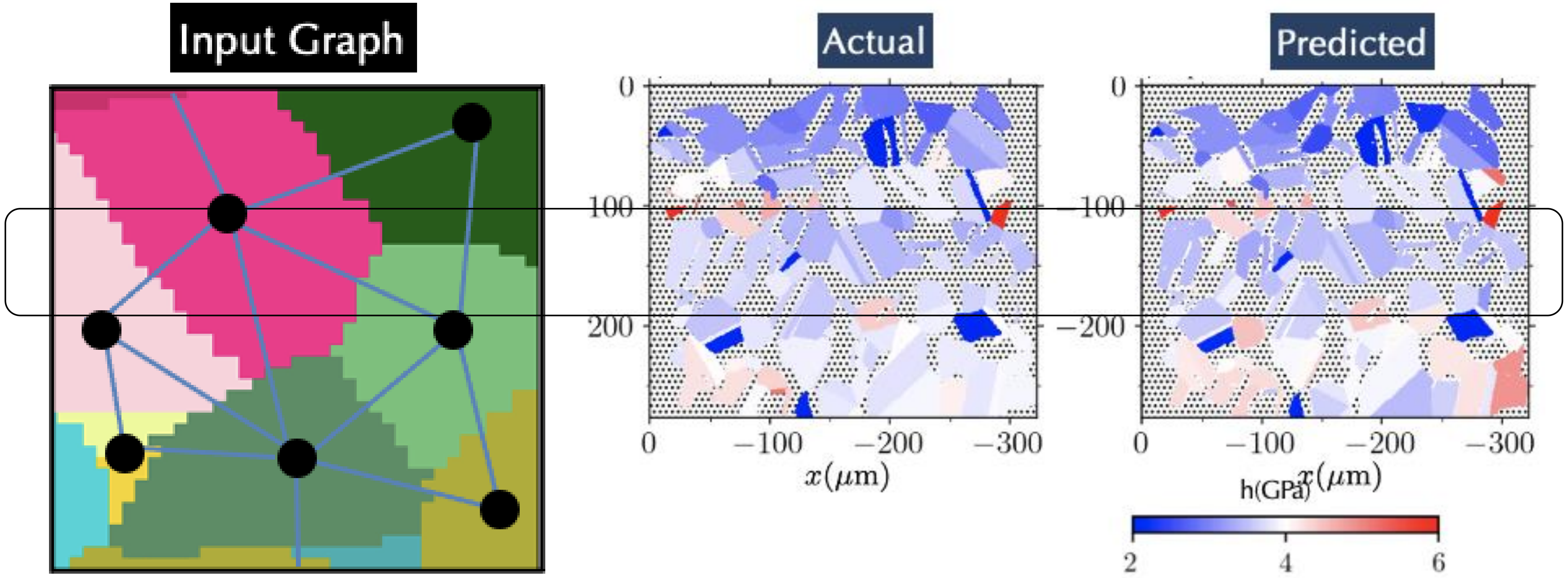
Continuum crystal plasticity simulations with the finite element method (FEM)







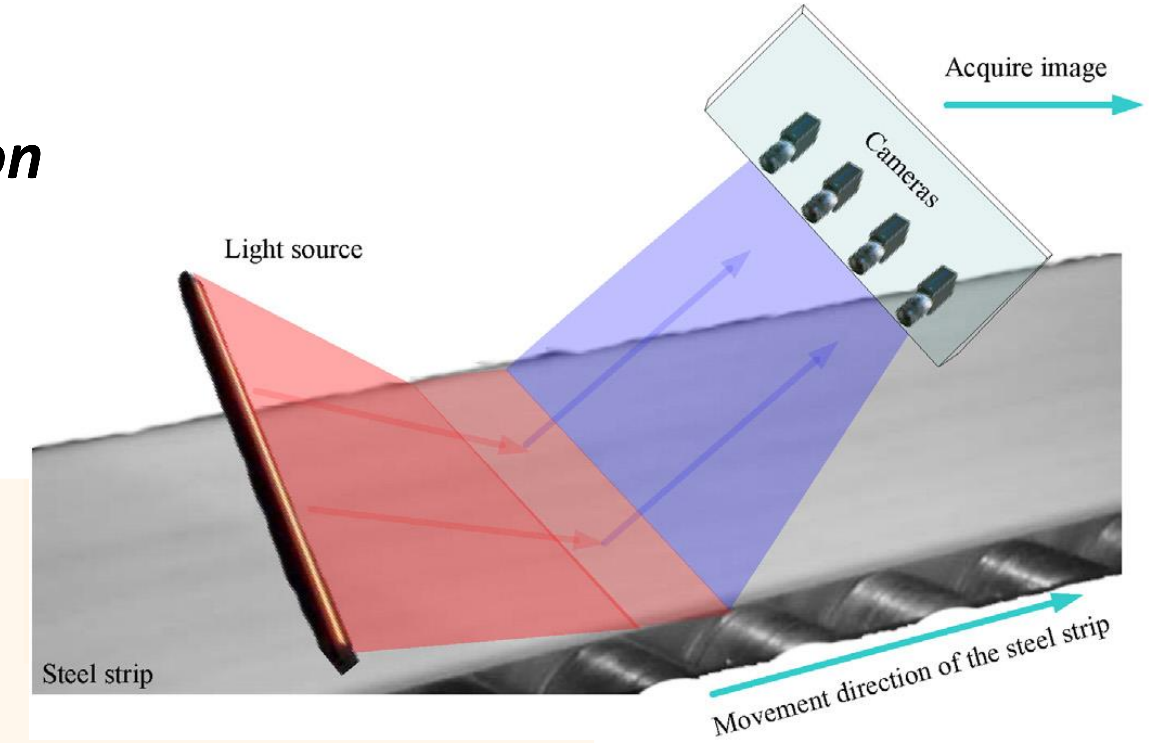
Graph neural networks for hardness/EBSD maps

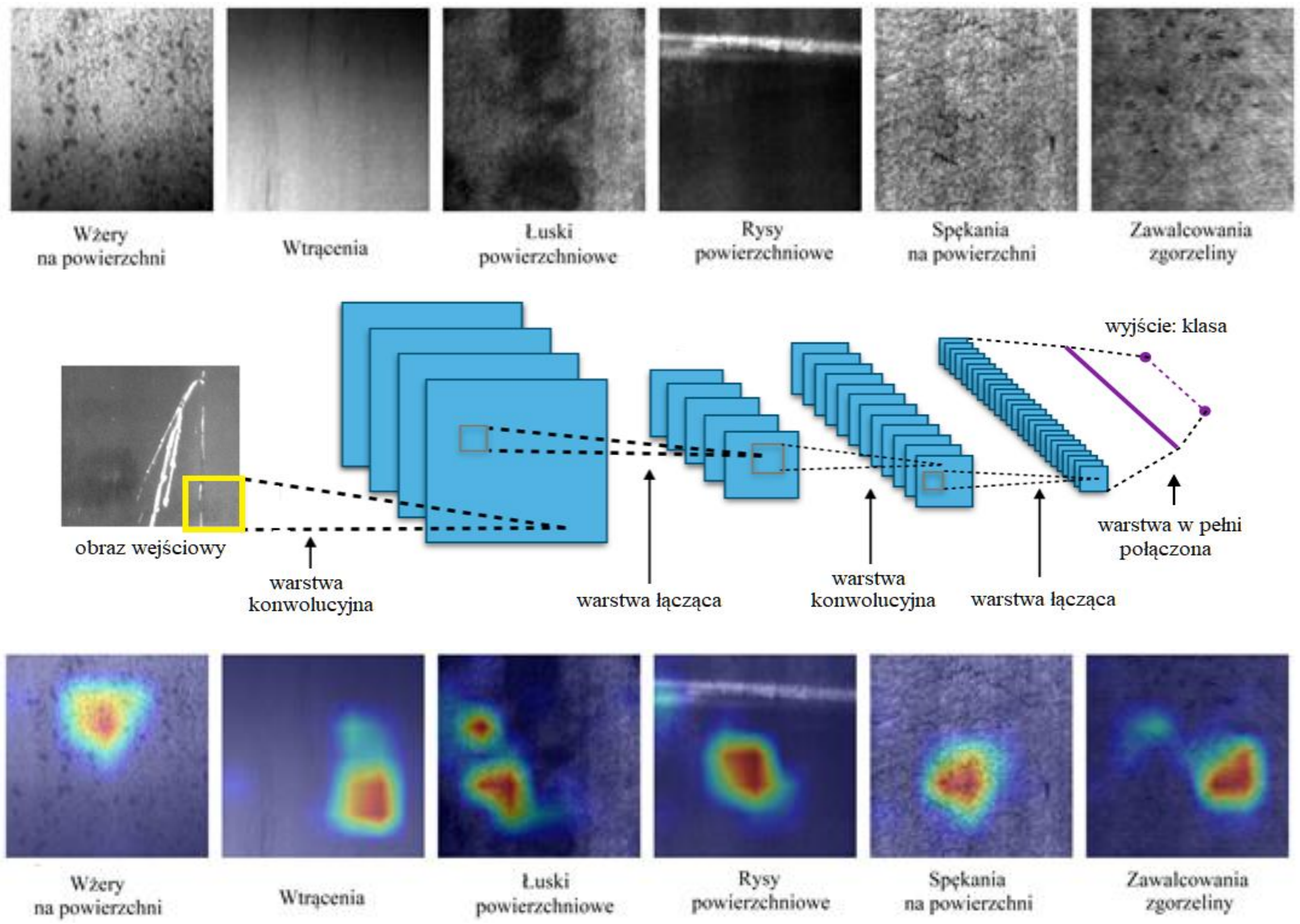


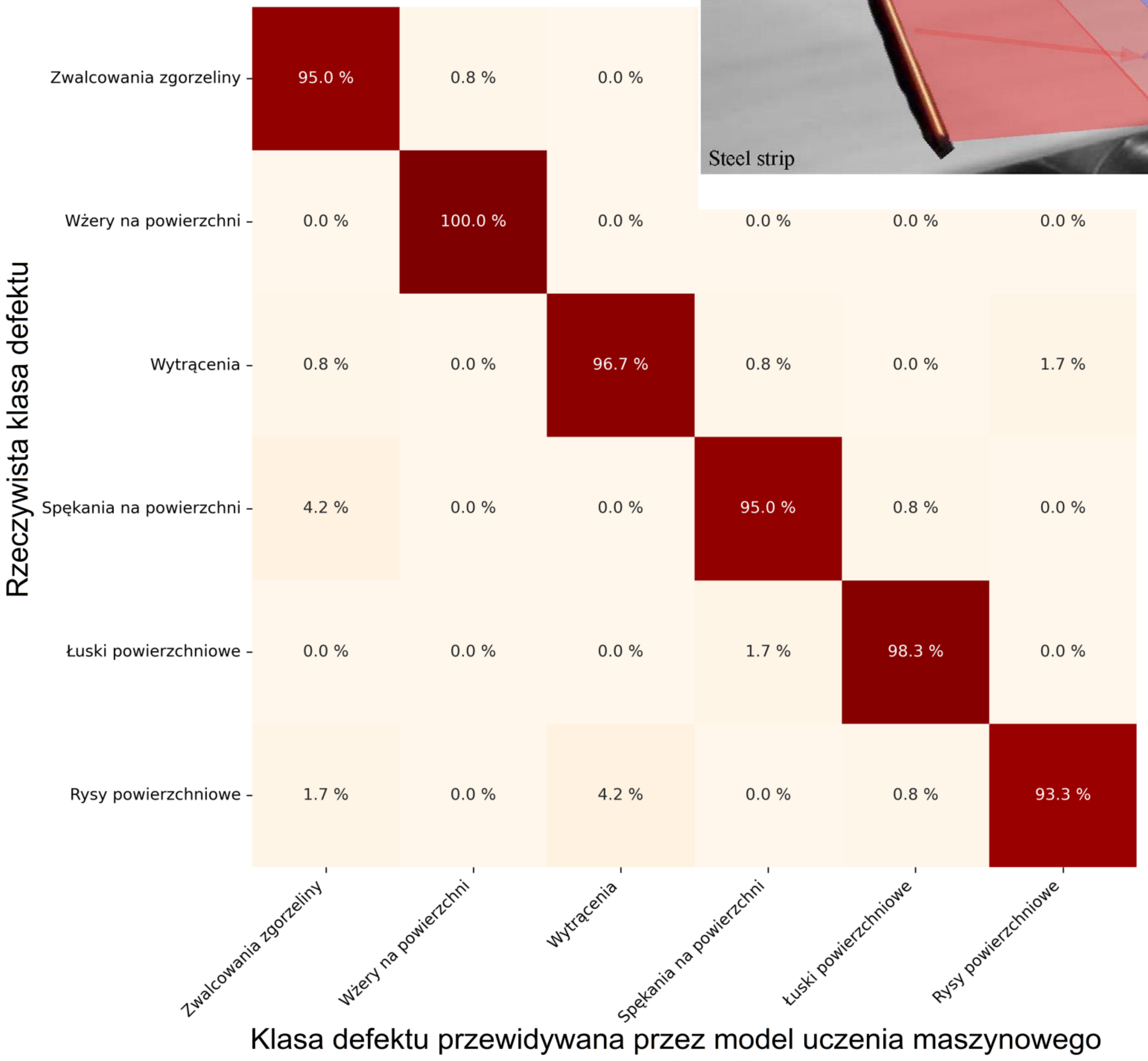
Simulations at NOMATEN CoE

Machine Learning

- ***steel defects identification***







Other applications of machine learning (including deep, convolutional or graph neural networks):

- *automatic microstructure classification and segmentation based on OM and SEM images,*
- *laser welding,*
- *natural language processing (AI for specific applications),*
- *etc.*

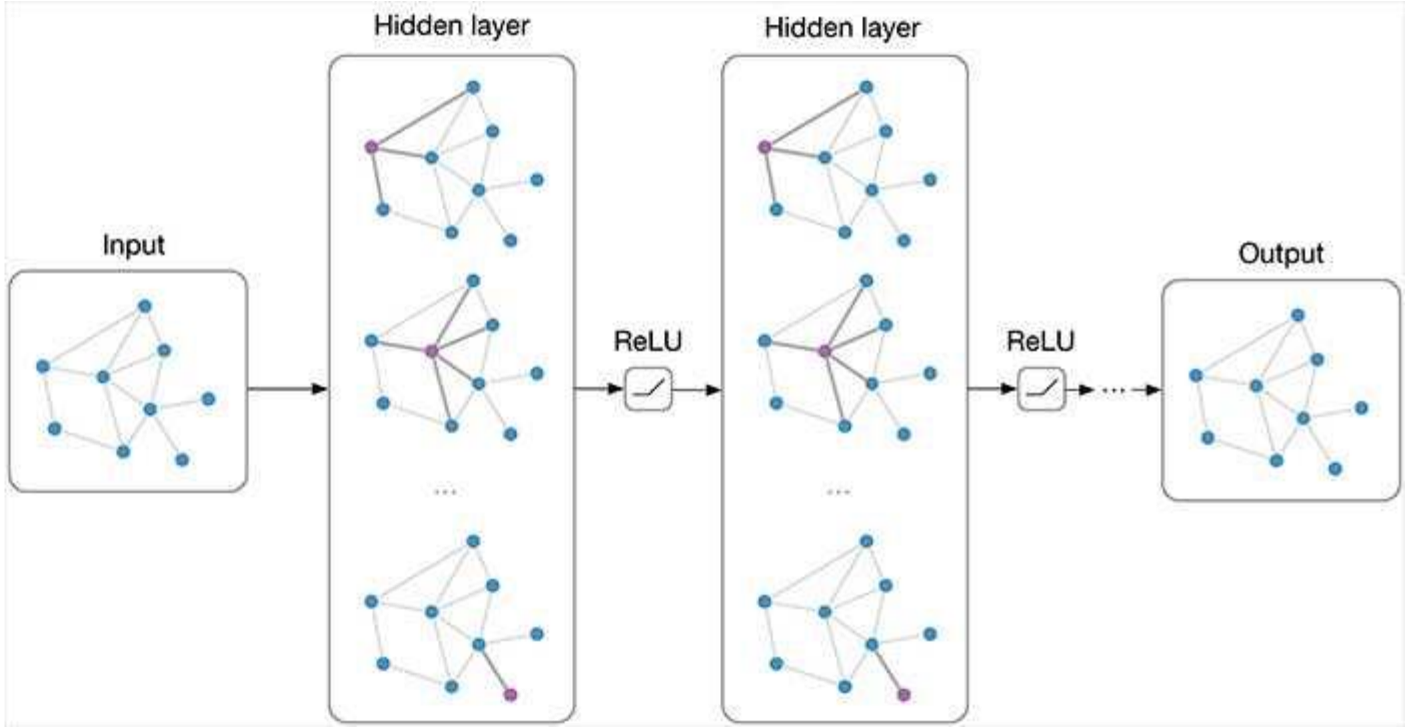
Simulations at NOMATEN CoE

Hydrogen related research

Hydrogen energy storage

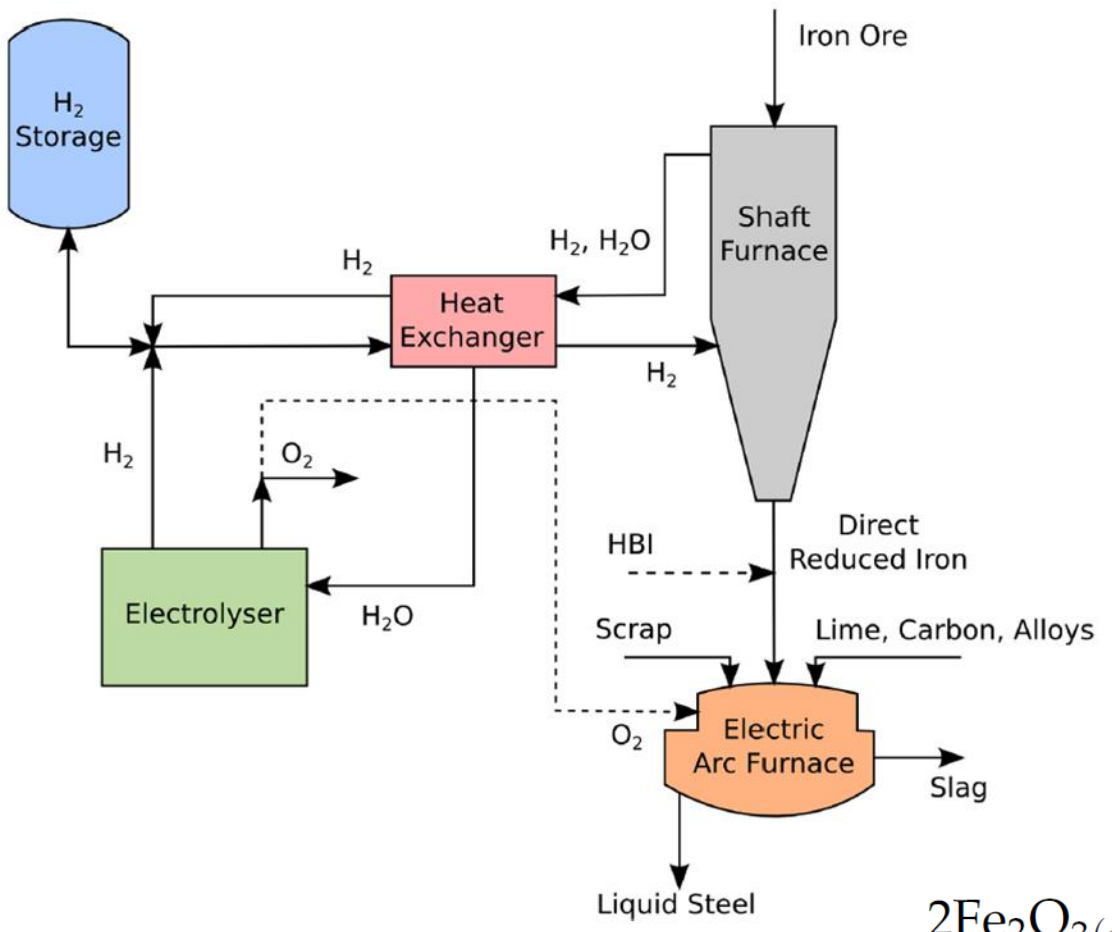
Ab initio simulations with machine learning interatomic potentials

Graph neural network



Serch for optimum intermetallic compounds as catalysts for:

- hydrogen evolution reaction (HER)
- oxygren evolution reaction (OER)



Is this method applicable also to hematite reduction?

Can we find a catalyst for:

$$2Fe_2O_{3(s)} + H_{2(g)} \rightarrow 2Fe_3O_{4(s)} + H_2O_{(g)}$$

$$Fe_3O_{4(s)} + \frac{16}{19}H_{2(g)} \rightarrow \frac{60}{19}Fe_{0.95}O_{(s)} + \frac{16}{19}H_2O_{(g)}$$

$$Fe_{0.95}O_{(s)} + H_{2(g)} \rightarrow 0.95Fe_{(s)} + H_2O_{(g)}$$

$$2Fe_2O_{3(s)} + CO_{(g)} \rightarrow 2Fe_3O_{4(s)} + CO_{2(g)}$$

$$Fe_3O_{4(s)} + \frac{16}{19}CO_{(g)} \rightarrow \frac{60}{19}Fe_{0.95}O_{(s)} + \frac{16}{19}CO_{2(g)}$$

$$Fe_{0.95}O_{(s)} + CO_{(g)} \rightarrow 0.95Fe_{(s)} + CO_{2(g)}$$

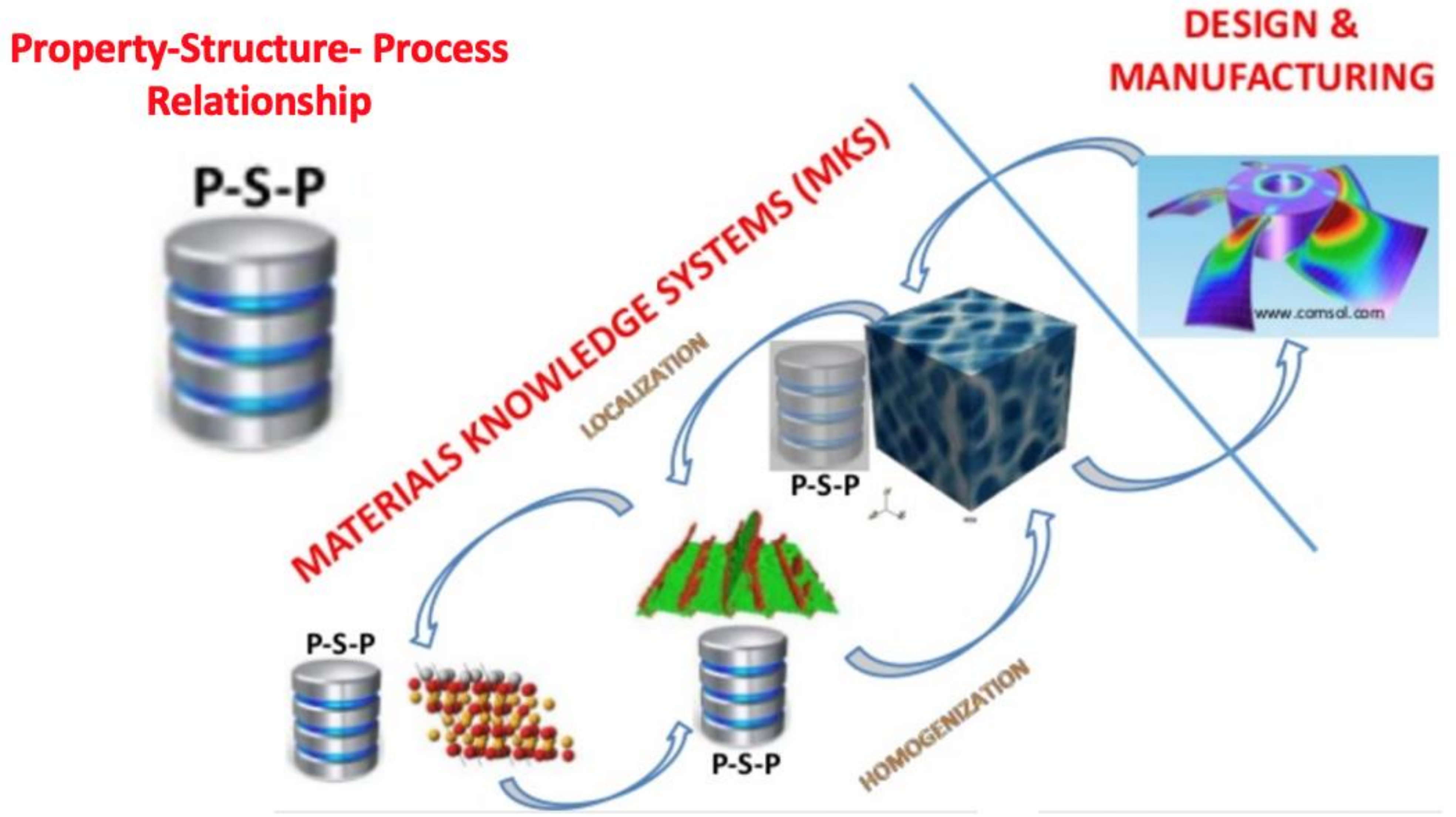
?

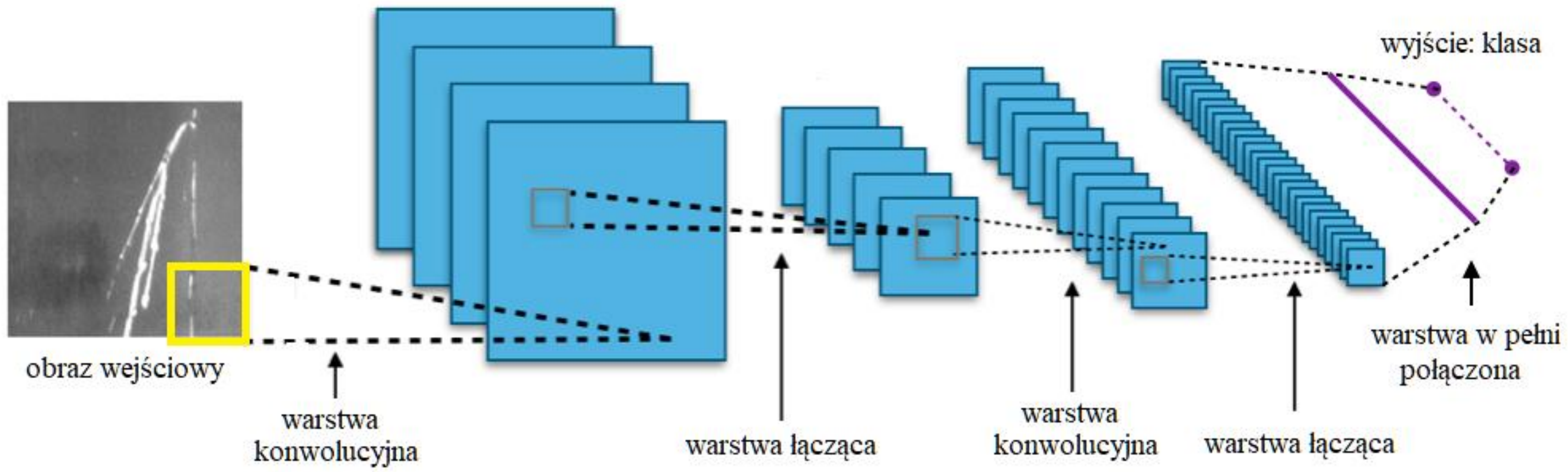
Simulations at NOMATEN CoE

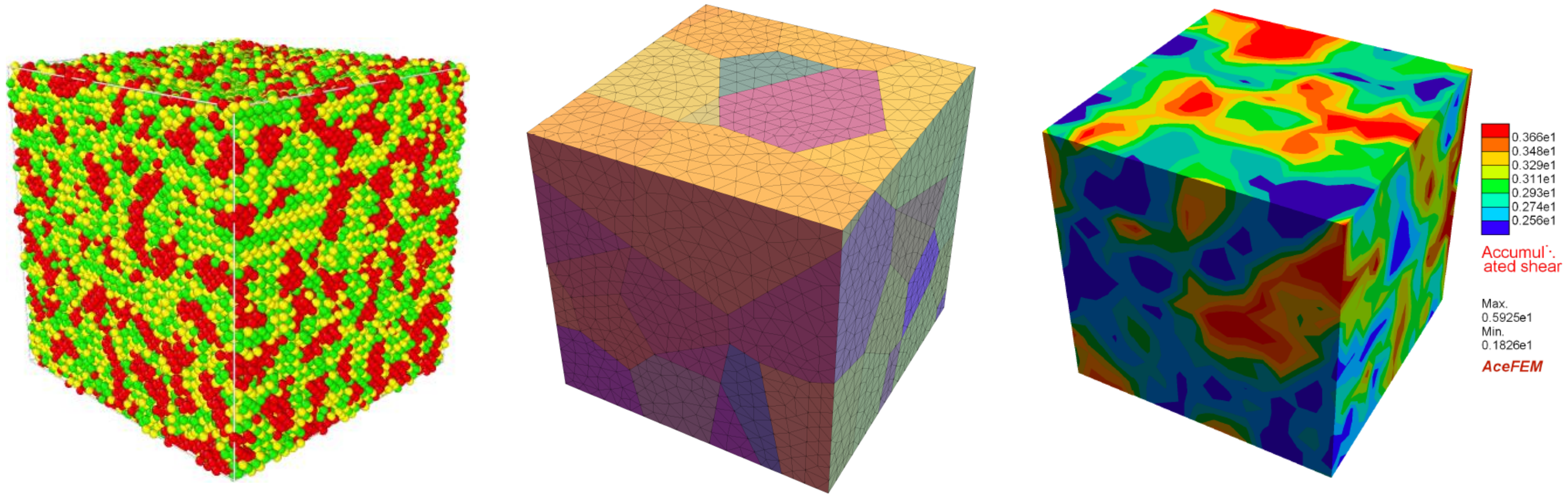
Conclusions

- **Multiscale simulations capabilities:** density functional theory, molecular dynamics, discrete dislocation dynamics, crystal plasticity and macroscopic plasticity,
- **Artificial intelligence at every level:** machine vision, natural language processing, optimization, machine learning interatomic potentials,

Tell us what you need – it is highly probable we will be able to compute it ☺.







NATIONAL CENTRE FOR NUCLEAR RESEARCH ŚWIERK

NOMATEN Centre of Excellence in Multifunctional Materials for Industrial and Medical Applications

CoE NOMATEN Materials Research Laboratory
Structure Analysis and Corrosion Research Services

Structure and Corrosion Research Division – Research Infrastructure

Metallographic samples preparation and analysis

Metallographic sample prepartion section

- Cut-off machines (precision cutting)
- Manual or automatic grinder / polisher
- Manual or automatic, electrochemical (0-100V) and vibropolishing (60 - 120 Hz)
- Electrochemical polishing and etching (0-25V) / possibility of electrolytic polishing in cool temperature mode
- Hot Mounting Press

QATM QUALITY ASSURED

QATM Opal 410 press

QATM Saphir Vibro polisher

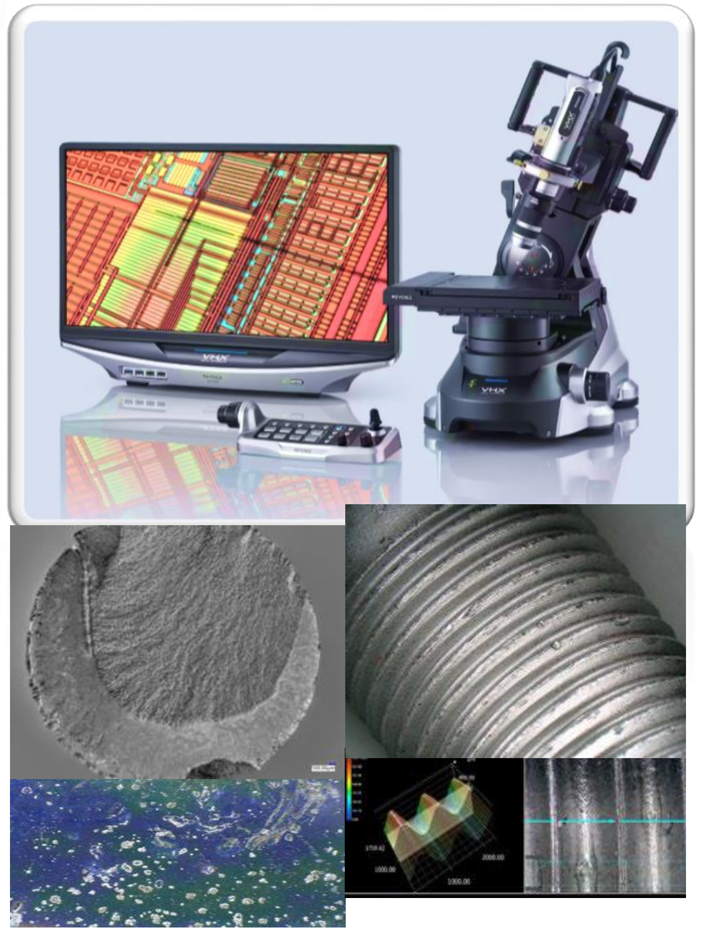
STRUERS – LectroPol electrochemical polishing / etching system

QATM Saphir 250 M2 automatic grinder / polisher

Microstructure characterization – Light Microscopy

- Leica DM IL Inverted Metallurgical Microscope
- Olympus BX53M Metallurgical Microscope
- Keyence VHX-700 Optical Microscope
- ✓ Light microscopy contrast methods such as Brightfield BF, Darkfield DF, Polarized light POL, and Differential Interference Contrast DIC
- ✓ Olympus licensed software for determining average grain size according to international standards (i.a. ASTM E112, ISO 643) and phase analysis

Olympus BX53M Metallurgical Microscope



KEYENCE VHX-7000 Optical Microscope

Structure and Corrosion Research Division – Research Infrastructure

Samples preparation and microstructure analysis

SEM/TEM Laboratory financed by NOMATEN Centre of Excellence in Multifunctional Materials for Industrial and Medical Applications

SEM microscope Helios 5 UX DualBeam (Thermo Fisher Scientific)

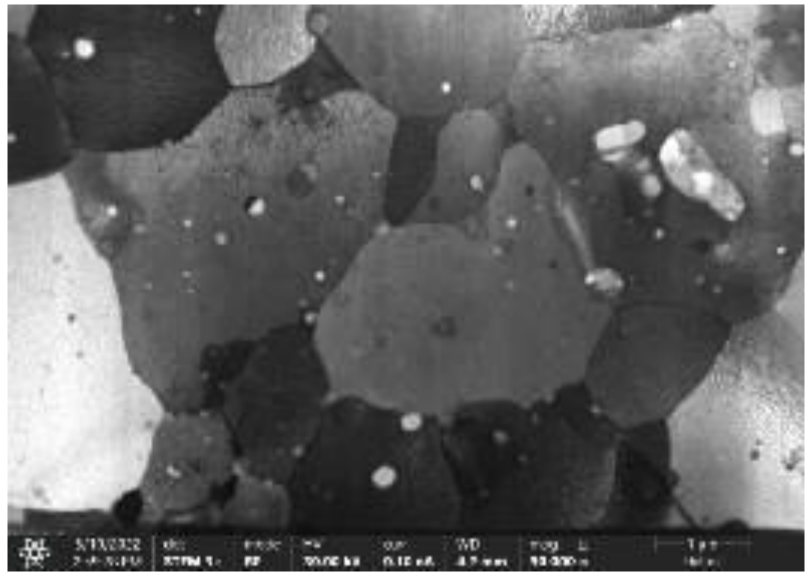
The Extreme High Resolution (XHR) Field Emission Scanning Electron Microscope (FE SEM) equipped with:

- FIB (Focused Ion Beam) technology
- EDS (Energy Dispersive X-ray Spectroscopy)
- EBSD (Electron Backscatter Diffraction)

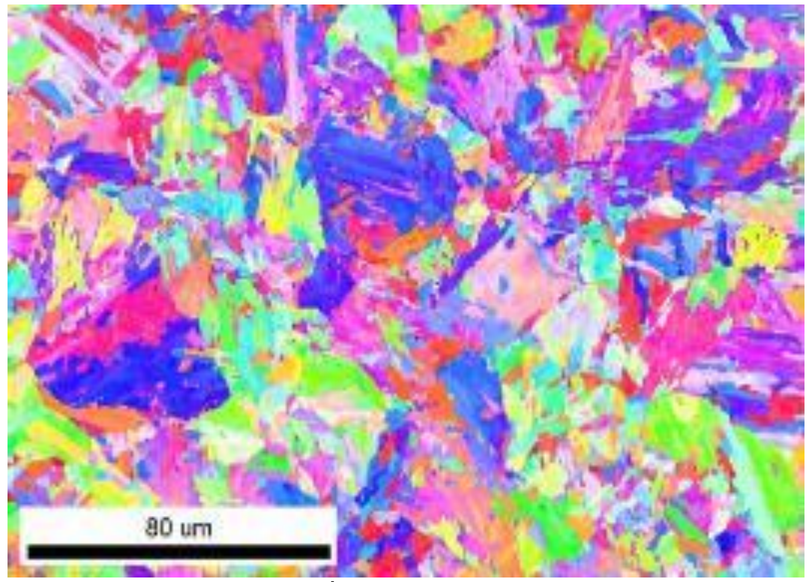
Ion Beam Precision Etching System

The PECS II (Gatan) is used to polish surfaces and remove without damage with two broad argon beams. This method is powerful for producing high-quality samples:

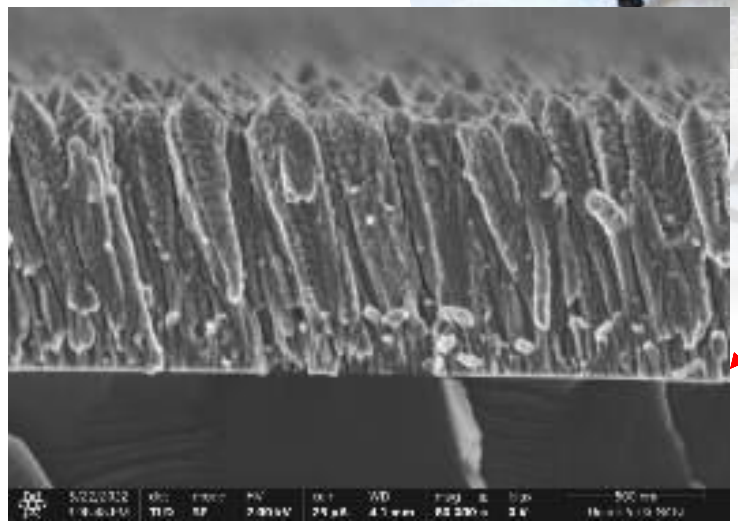
- for scanning electron microscope (SEM) observations
- for SEM imaging and EDS mapping
- for EBSD analysis,
- for STEM, TEM observation etc.



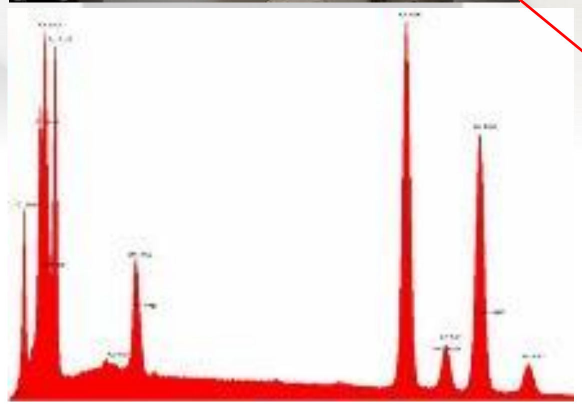
STEM imaging
Recractable STEM 3+ detector



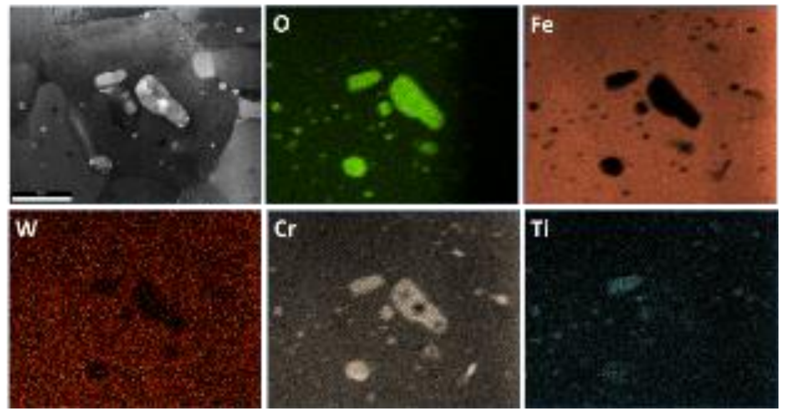
Crystal orientation mapping
Hikari Super EBSD Camera, 1400 fps
Operation down to 100 pA/5kV



High resolution SEM imaging
Acceleration voltage: 350V – 30kV
Resolution: 0.6 nm (2 - 15kV), 0.7 nm (1 kV)
Detectors: ETD, TLD, ICD, MD, ICE



EDS Chemical composition analysis
Octane Elite Plus EDS System
SSD detector, area: 30mm2, resolution: 125eV, Si3N4



Structure and Corrosion Research Divisi

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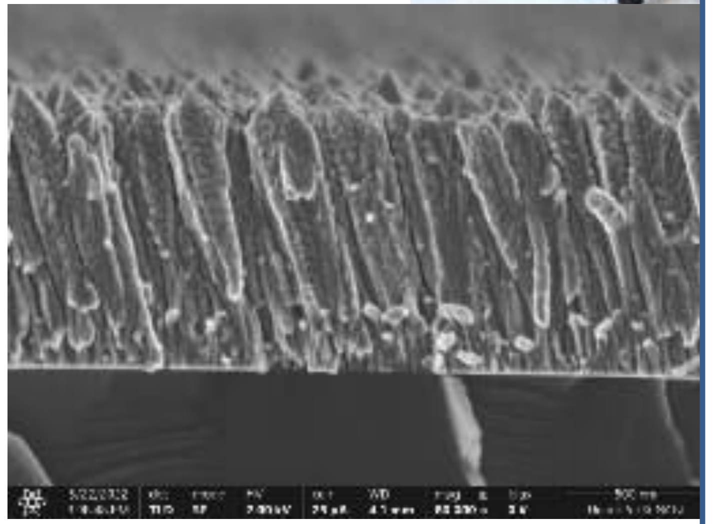


High resolution SEM imaging
Acceleration voltage: 350V – 30kV
Resolution: 0.6 nm (2 - 15kV), 0.7 nm (1 kV)
Detectors: ETD, TLD, ICD, MD, ICE

TEM Microscope

Transmission Electron Microscopy TEM – JOEL F200 Microscope

TEM with STEM, HAADF, EDS, BEI, BF and ABF detectors
Equipped with in-situ tensile and HT annealing up to 1000°C holders

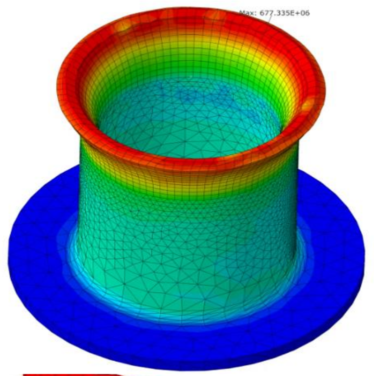
Structure and Corrosion Research Division – Research Activities

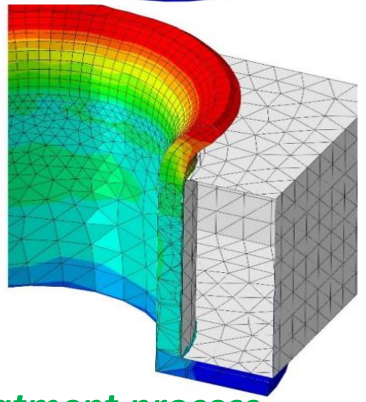
Materials fatigue analysis for Automotive Indsutry

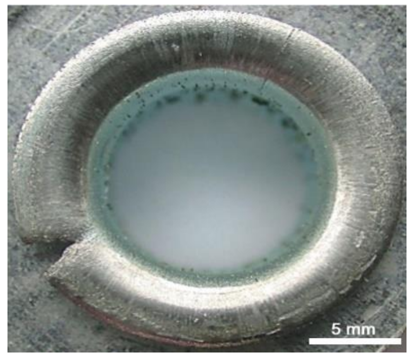
Problem to solve: Cracking of tubular rivets for clamping Knee-Airbag modules

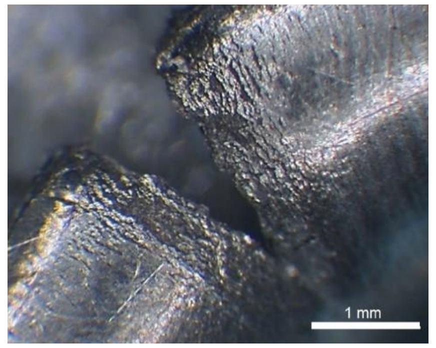
- We realized metallographic analysis of the low-carbon steel, macroscopic, LM and SEM observations and EDS analysis
- We showed that material cooling after heat treatment was realized wrongly – tertiary cementite in the grain boundaries
- We finally selected the proper heat treatment parameters of the rivets and estimated the optimal clamping force with FEM analysis

FEM analysis of riveting process – clamping force



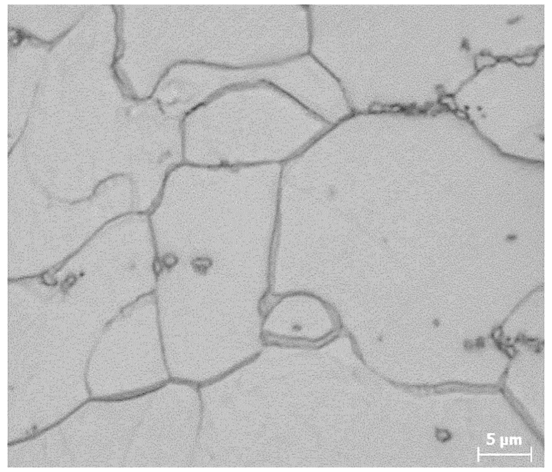


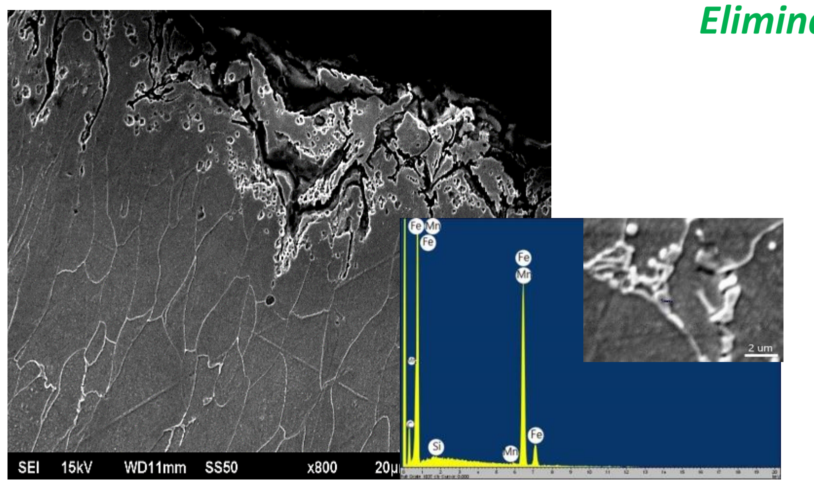




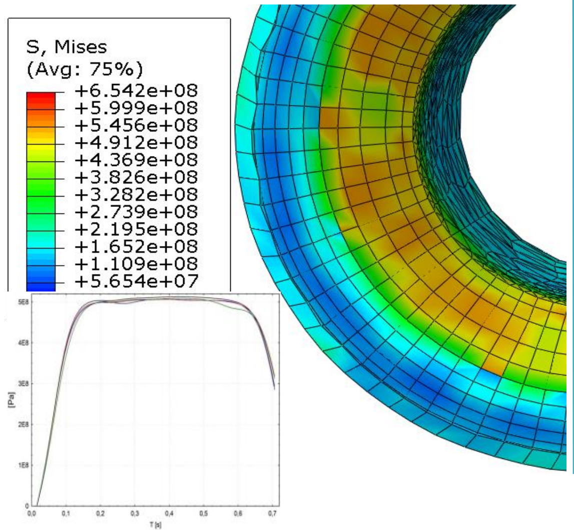
Macroscopic observation of the cracking area of the rivets

Result: Heat treatment process improvement
Elimination of production losses



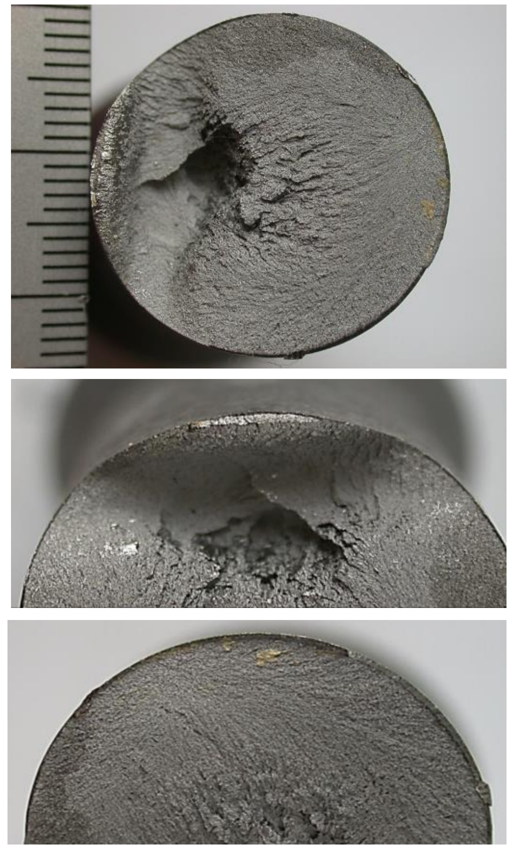


Microstructure analysis of the low-carbon steel – tertiary cementite in the grain boundaries



Problem to solve: Induction hardening optimization
Cracking of the drive shafts during straightening at quality control stage

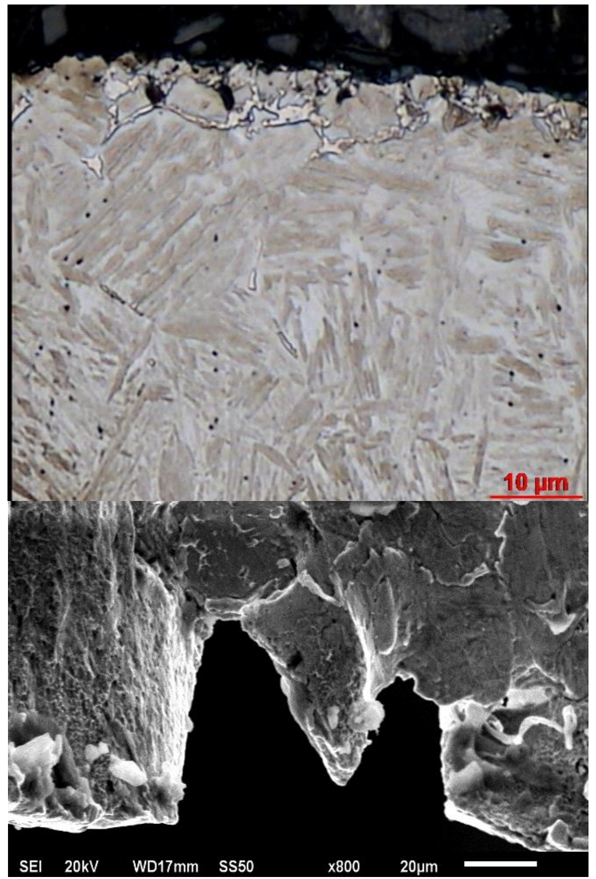
- We analyzed the material after each production stage (rod delivery state > machining > induction hardening)
- We realized metallographic analysis of the low-alloy steel (macroscopic observations of breakthrough, LM and SEM observations, hardened case depth analysis)
- We showed that material after induction hardening tends to crack at the surface
- We optimzed heat treatment with adding stress relief stage after machining and set proper induction hardening parameters incl. low-tempering after hardening



Macroscopic observation of breakthrough

Microstructure analysis of low-alloy steel – surface ckracking effects after induction hardening

Result: Full eilimination of the cracks
Quality control report:
100% Drive shafts OK



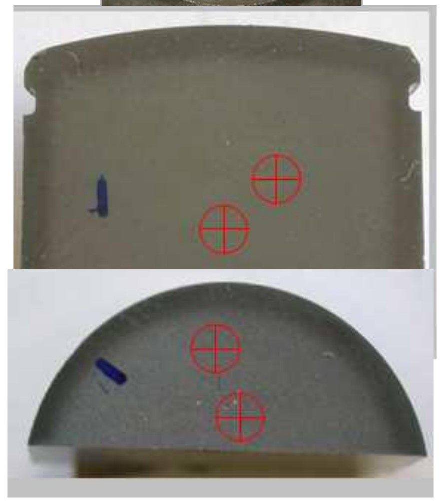
Structure and Corrosion Research Division – Research Activities

Chemical Heat Treatment optimization

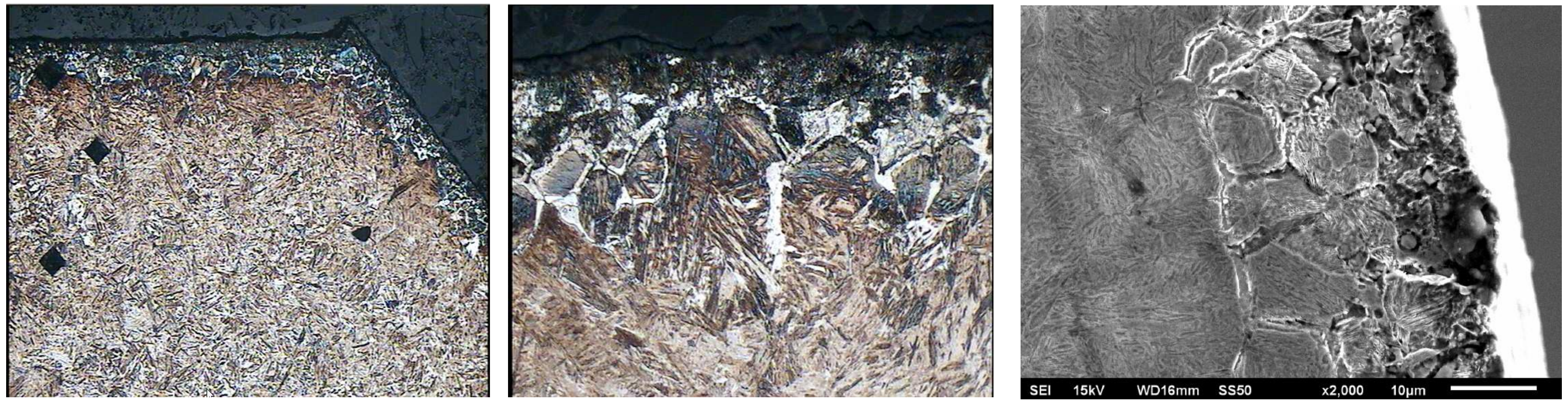
Problem to solve: Elimination of errors in the carburising process of drive shaft cross joints

- We analyzed the carburizing parameters of the drive shaft cross
- We realized metallographic analysis of the 20MnCr5 steel after carburizing / macroscopic, LM and SEM observations
- We confirmed that material was wrongly carburized (too slow subcooling during process) – ferrite net through the grain boundaries and bainitic-martensitic islands at surface
- We selected the proper carburizing parameters and heating and cooling speed during heat treatment – cross met requirements od dynami testing / no-pitting effect observed

Result: Carburizing improvement – no pitting effect



Macroscopic observation of the drive shaft cross after dynamic testing according with manufacturer quality control system – visible pitting effects

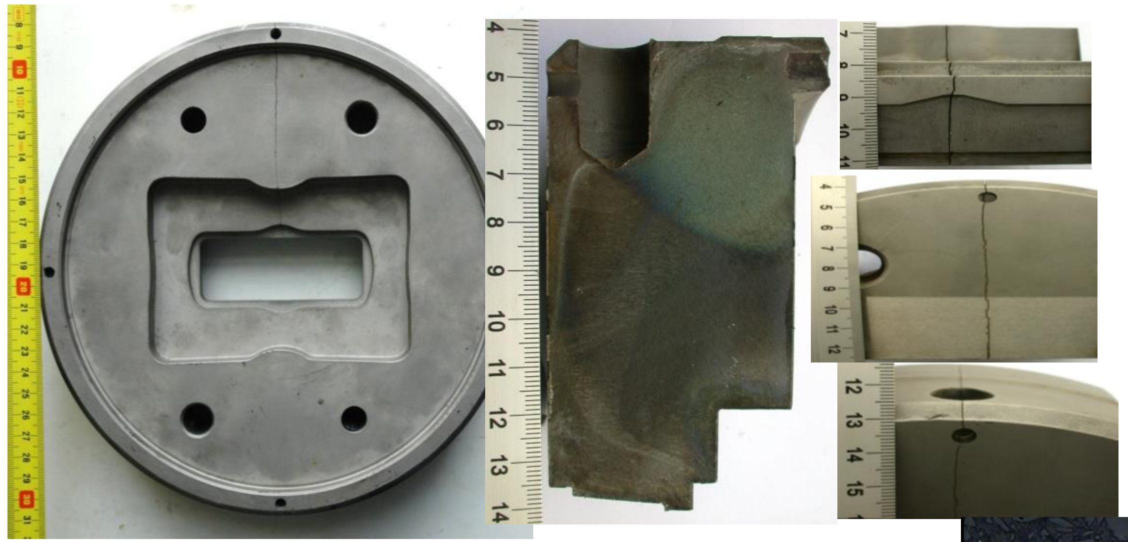


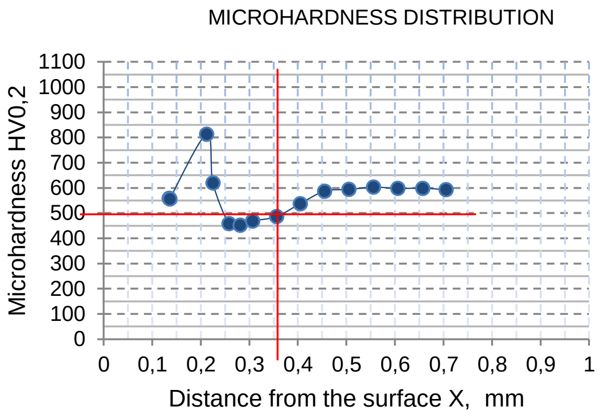
Micrsotructure analysis of the 20Cr5 low-alloy steel – visible faults after carburizing process / ferrite net at the top surface of the cross / wrongly selected subcooling speed during carburizing

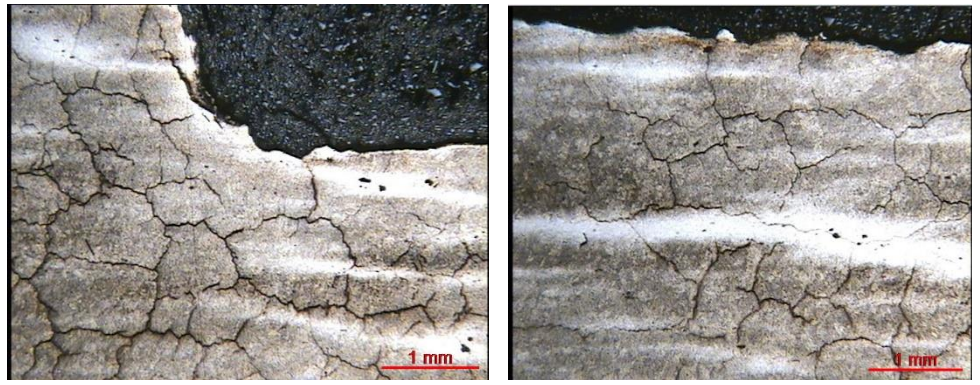
Heat Treatment and extension of tools operating time

Problem to solve: Elimination of aluminium extrusion dies cracking after short operating time

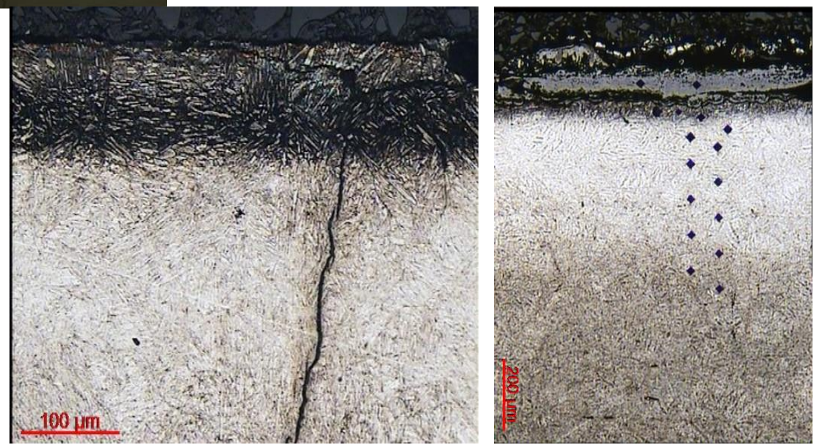
- We analyzed the causes of aluminium processing dies cracking after short operating time
- We realized metallographic analysis of the X35CrMoV5-1 hot-work tool before na after operation, Macroscopic and LM observations
- We showed that dies are were not properly pre-heated fore the extrusion process and crack beacuse of thermal shocks during extrusion (Δt surface / core of the die)
- We set the conditions for pre-heating of the dies before starting operation to equalize temperature in the whole tool
- We set the die operation thermal parameters during the extrusion process so that critical temperatures are not exceeded (temp. depends on the Al alloy)







Macroscopic observation – visible blue brittleness > ca. 350 C°



Microstructure analysis of X35CrMoV5-1 steel after operation

NATIONAL CENTRE FOR NUCLEAR RESEARCH ŚWIERK

NOMATEN Centre of Excellence in Multifunctional Materials for Industrial and Medical Applications

CoE NOMATEN Materials Research Laboratory
Phase Analysis and Chemical Composition
Research Services

Phase analysis Laboratory NOMATEN XRAYLAB – Research Infrastructure

X-Ray Laboratory financed by
NOMATEN
Centre of Excellence in Multifunctional Materials for Industrial and Medical Applications

X-ray diffraction phase analysis

Key X-ray research abilities:

X-ray powder diffraction (XRPD)

- Identification of crystalline and amorphous phases and determination of specimen purity
- Quantitative analysis of both crystalline and amorphous phases in multi-phase mixtures
- Microstructure analysis (crystallite size, microstrain, disorder…)
- Bulk residual stress resulting from thermal treatment or machining in manufactured components
- Texture (preferred orientation) analysis
- Indexing, ab-initio crystal structure determination and crystal structure refinement

Analysis of amorphous, poorly crystalline, nano-crystalline or nano-structured materials

- Phase identification
- Structure determination and refinement
- Nano particle size and shape

Thin Films and Coatings high quality analyses

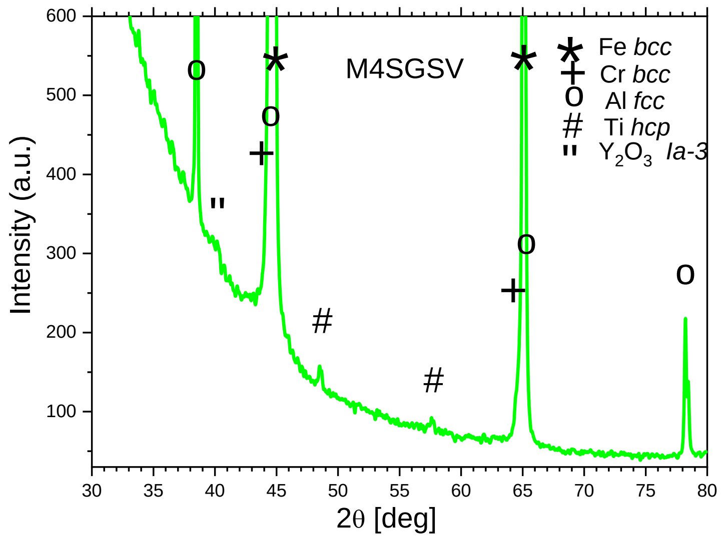
- Grazing incidence diffraction
- X-Ray Reflectometry
- High resolution X-ray diffraction
- Reciprocal space mapping

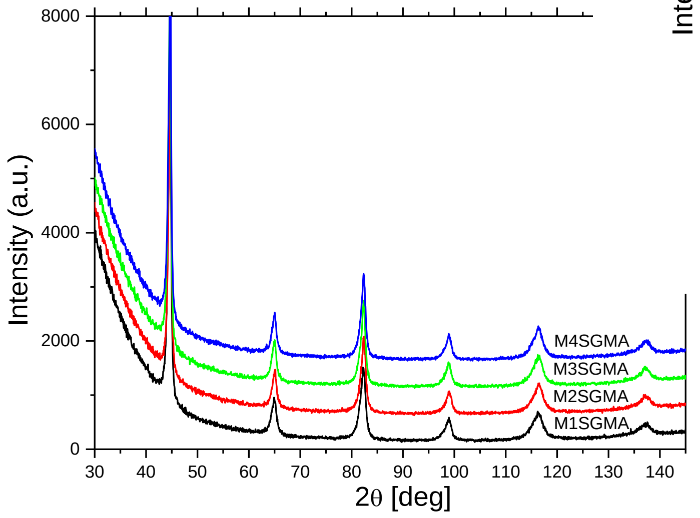
BRUKER **D8 ADVANCE**

- equipped with a sealed Cu X-Ray tube, TWIN-TWIN optics and LYNXEYE XE-T strip detector
- Cu radiation, $\lambda_{K\alpha1}$ = 1.540562 Å
- Energy Resolution < 380 eV at 8 keV
- B-B/GID geometries

High-temperature stage - Anton Paar HTK 1200 N

- temperature up to 1200°C
- operates Under Vacuum or Selected Gas Environment
- specimen Stage with Rotation (Rocking)





FeCrAl-ODS alloys powders analysis

Sample	Fe-rich solid solution (bcc)				Cr-rich solid solution (bcc)		
	Lattice constant [Å]	Crystallite Size [nm]	Strain parameter	Phase content	Lattice constant [Å]	Crystallite Size [nm]	Strain parameter
M1SGMA	2.868	28	0.0049	74	2.891	14	0.0057
M2SGMA	2.866	23	0.0048	87	2.889	11	0.0051
M3SGMA	2.869	23	0.0047	79	2.891	14	0.0053
M4SGMA	2.868	23	0.0047	87	2.891	14	0.0057

Structure and Corrosion Research Division – Research Infrastructure

Spectroscopic phase and chemical composition analysis

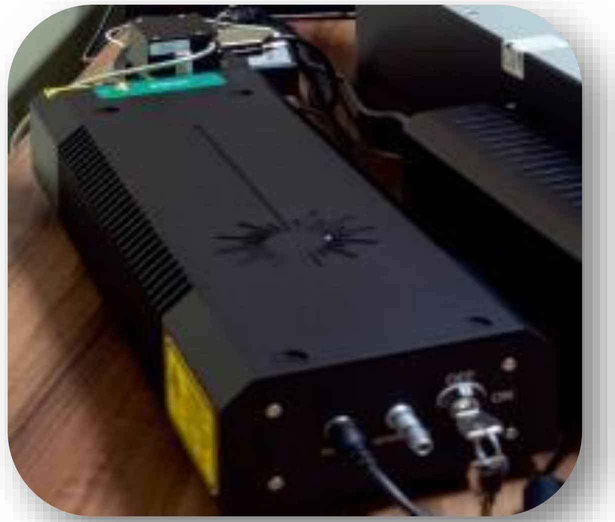
Raman Spectroscopy

Research Features

- Obtaining qualitative to semi-quantitative information on material phase composition (Raman imaging)
- Determination of stress distribution
- Examination of phase transition and corrosion of materials
- Observations of structural changes after ion implantation - defects type and amount determination

WITec focus innovations

Alpha 300R
Raman Spectrometer



Ultra-high throughput spectrometer (UHTS), for high speed and high resolution Raman imaging.

High temperature stage (up to 1000 C)

Optical microscope:
Zeiss Neofluar objectives
magnification x10, x50, x100

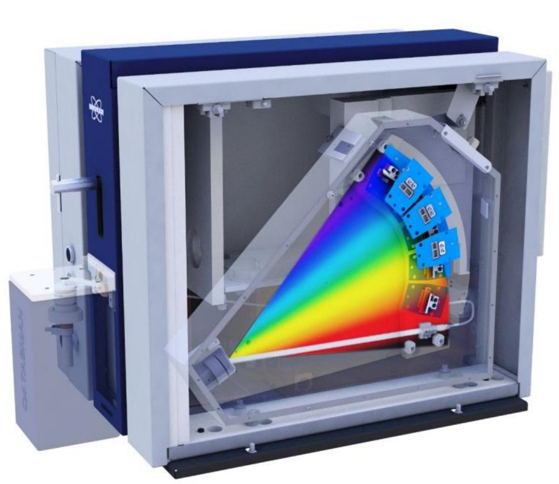
Spark Optical Emission Spectroscopy OES

BRUKER Q4 TASMAN Series 2

Research features:

- Quantitative elemental analysis
- Improved precision and stability
- High accuracy and sensitivity levels, full capabilities including C, P, S, Sb, Te
- Digital Spark Source delivers improved analytical precision and shorter time-to-result.
- Dual optics concept with robust Paschen Runge mount, multi-chip systems with temperature stabilization

PCA Polskie Centrum Akredytacji BADANIA AB 025 — ilac-MRA



Accreditation procedure in progress !!!

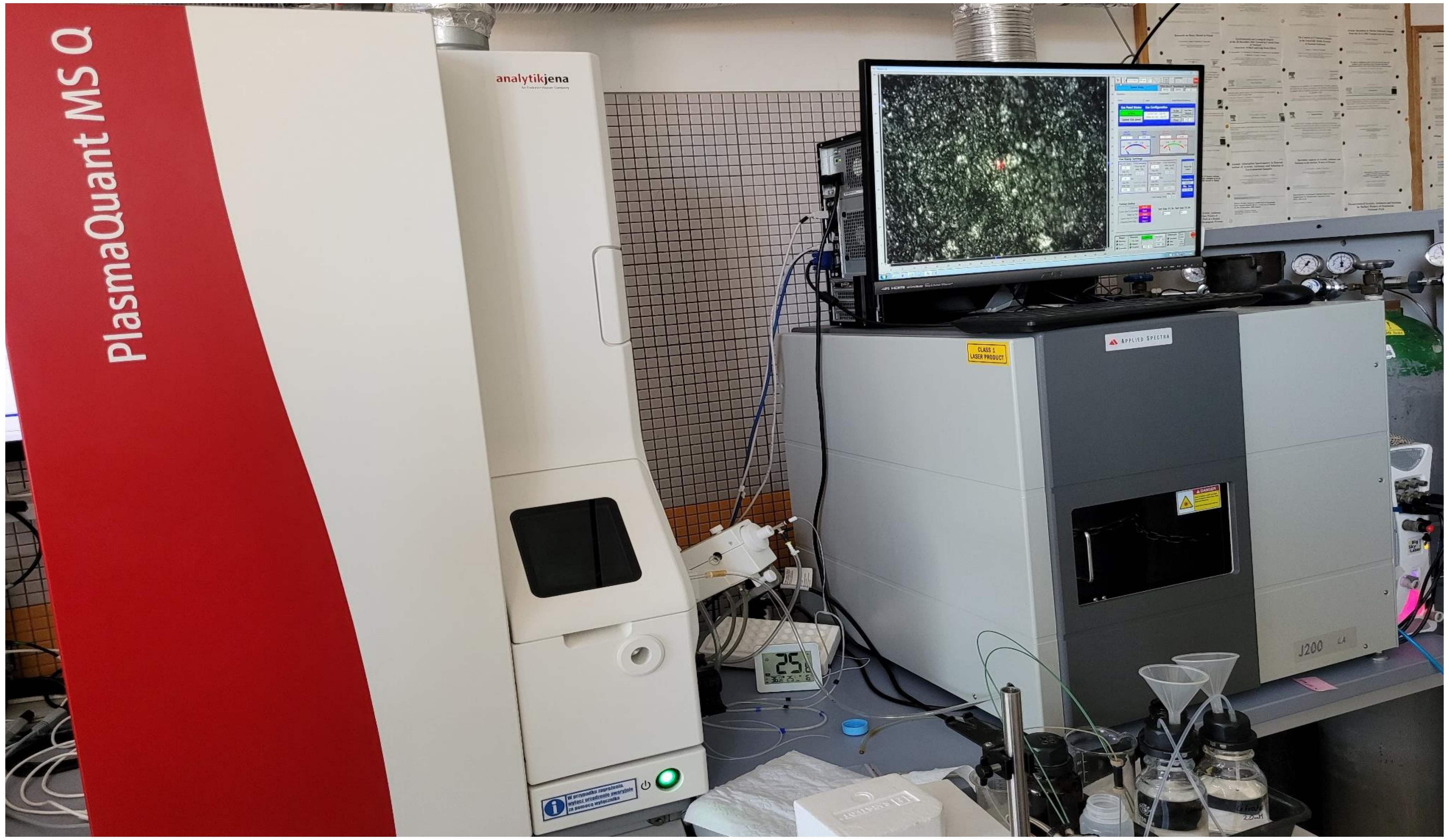
4 Analytical Bases Fe, Al, Ni and Ti alloys

30 CRM materials with ISO 17034 Standard

Structure and Corrosion Research Division – Research Infrastructure

ICP-MS integrated with Laser Ablation LA and LIBS spectrometer

Analytik Jena Plasma Quant MS Q **<> Applied Spectra J200 LA system with LIBS**



Financed by
PROJECT HTGR

Chemical analysis at .ppm and .ppb level of high purity graphite in accordance with IAEA regulations

Other research plans:

- solid samples inpurities analysis
- C,H,O,N detection in materials (LIBS)
- MARIA reactor water analysis

TOYO TANSO

■ Impurity Analysis Example

Unit: mass ppm

Element	Content: Ultra High Purity Graphite	Content: High Purity Graphite	Content: Regular Graphite	Measurement Method
Li	<0.001	<0.001	<0.03	ICP-MS
B	0.10	0.15	3	ICP-MS
Na	<0.002	<0.002	<0.5	ICP-MS
Mg	<0.001	0.004	0.2	ICP-MS
Al	<0.001	0.012	14	ICP-MS
Si	<0.1	<0.1	2	UV
K	<0.03	0.04	2	FL-AAS
Ca	<0.01	0.08	6	FL-AAS
Ti	<0.001	<0.001	33	ICP-MS

Element	Content: Ultra High Purity Graphite	Content: High Purity Graphite	Content: Regular Graphite	Measurement Method
V	<0.001	0.018	40	ICP-MS
Cr	<0.004	0.006	<0.3	ICP-MS
Mn	<0.001	<0.001	<0.2	ICP-MS
Fe	<0.02	0.06	26	ICP-MS
Co	<0.001	<0.001	<0.3	ICP-MS
Ni	<0.001	0.006	4	ICP-MS
Cu	<0.002	<0.002	<1	ICP-MS
Zn	<0.002	<0.002	<0.6	ICP-MS
Pb	<0.001	<0.001	<1	ICP-MS

Spark Optical Emission Spectroscopy OES

SMAN Series 2

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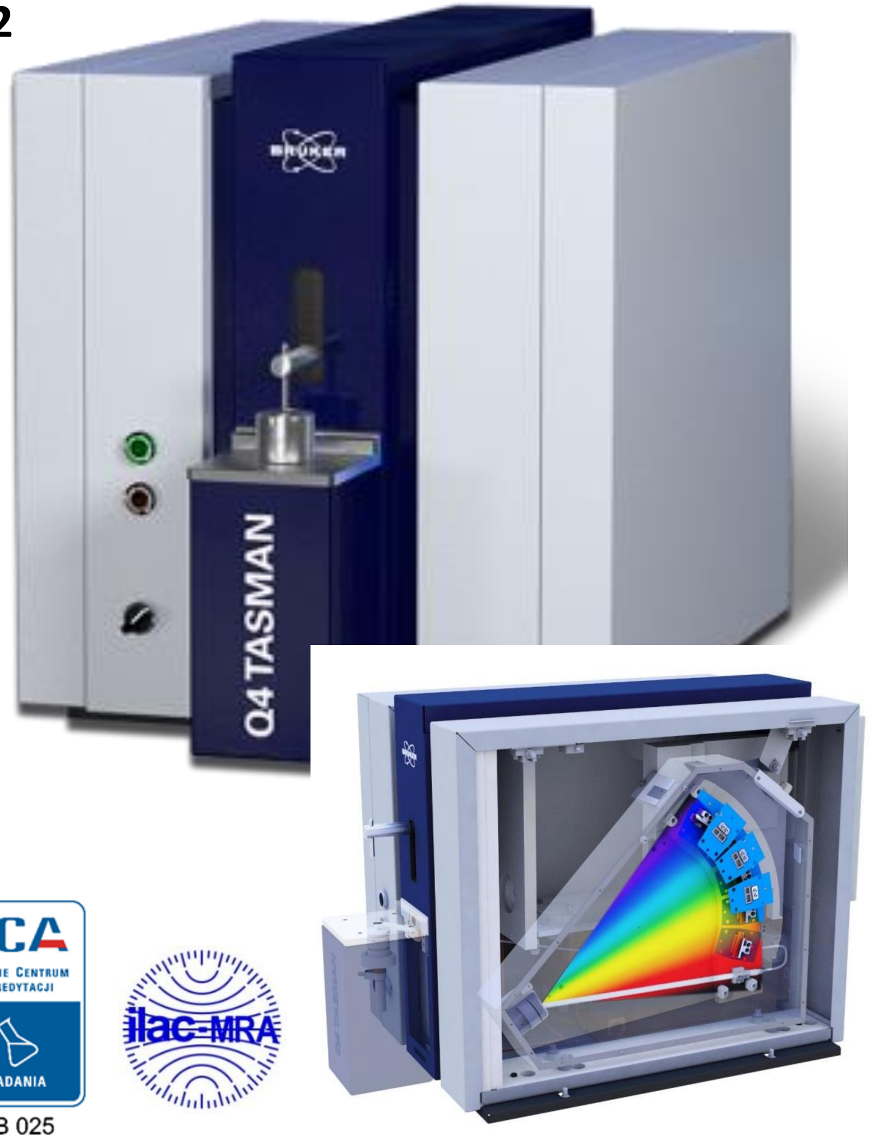

PCA Polskie Centrum Akredytacji BADANIA AB 025 ilac-MRA

Accreditation procedure in progress !!!

4 Analytical Bases Fe, Al, Ni and Ti alloys

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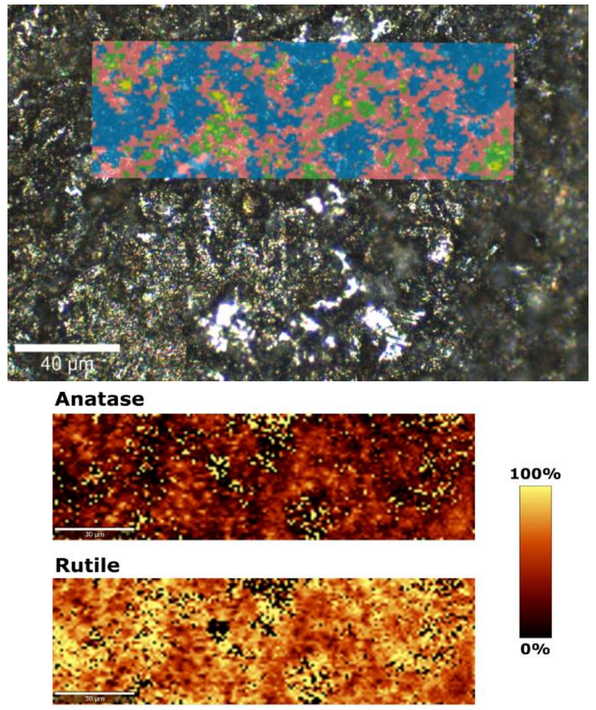
Structure and Corrosion Research Division – Research Activities

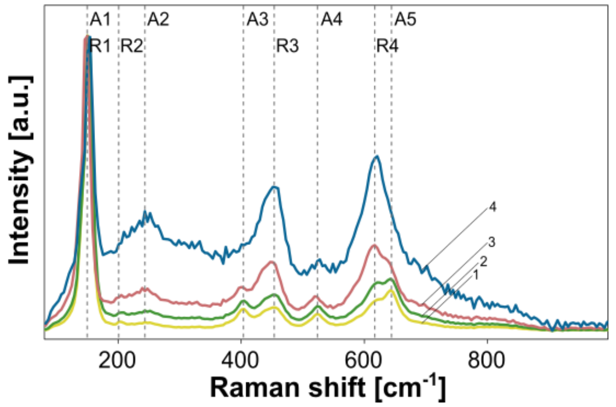
Phase analysis of TiO_2 thin layers by Raman spectroscopy imaging for Biomedical Indsutry

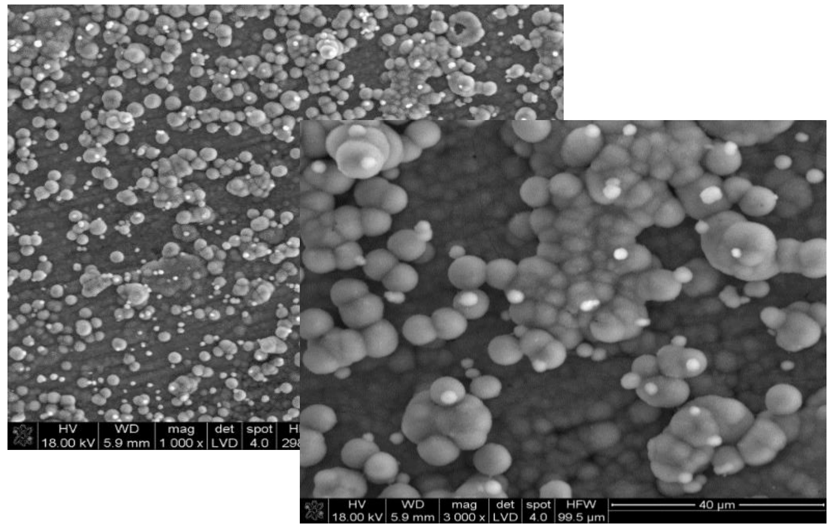
Problem to solve: Bioactivity of the Titanium surface and ossointegration improvement of the dental implants

- We designed the Titanium Grade 2 oxidation technique
- We made qualitative and quantitative analysis of TiO_2 oxides phase concentration
- We showed that Rutile and anatase phase mixture has beneficial properties to create a permanent tissue-implant connection
- We applied Raman spectroscopy imaging to determine phase distribution and estimated rutile/anatase concentration in TiO2 thin layers.

Oxidized Titanium surface analysis with Raman imaging/mapping

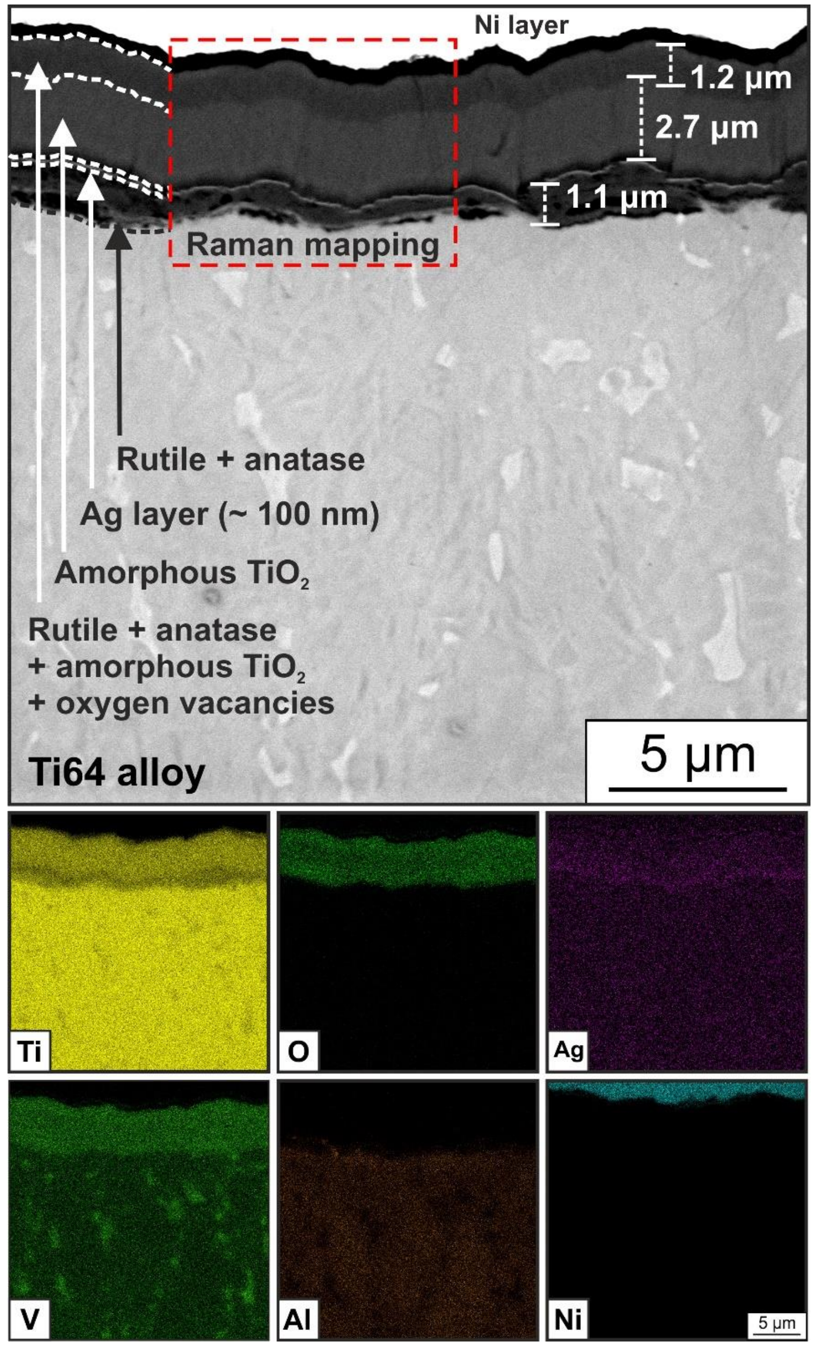




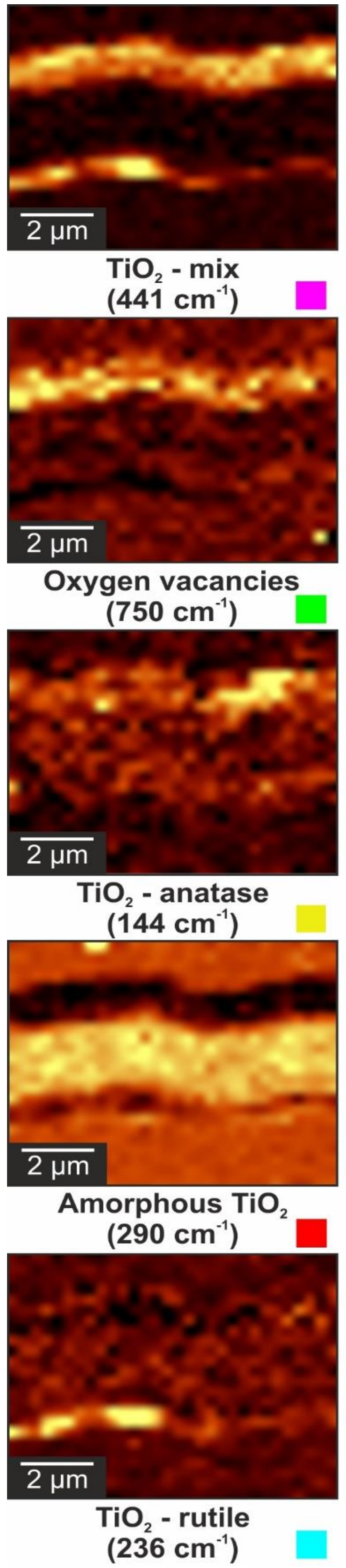


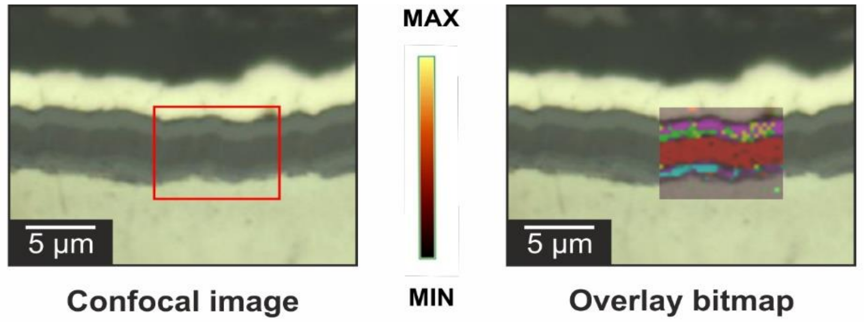
Result: Improved osseintegration of dental implants

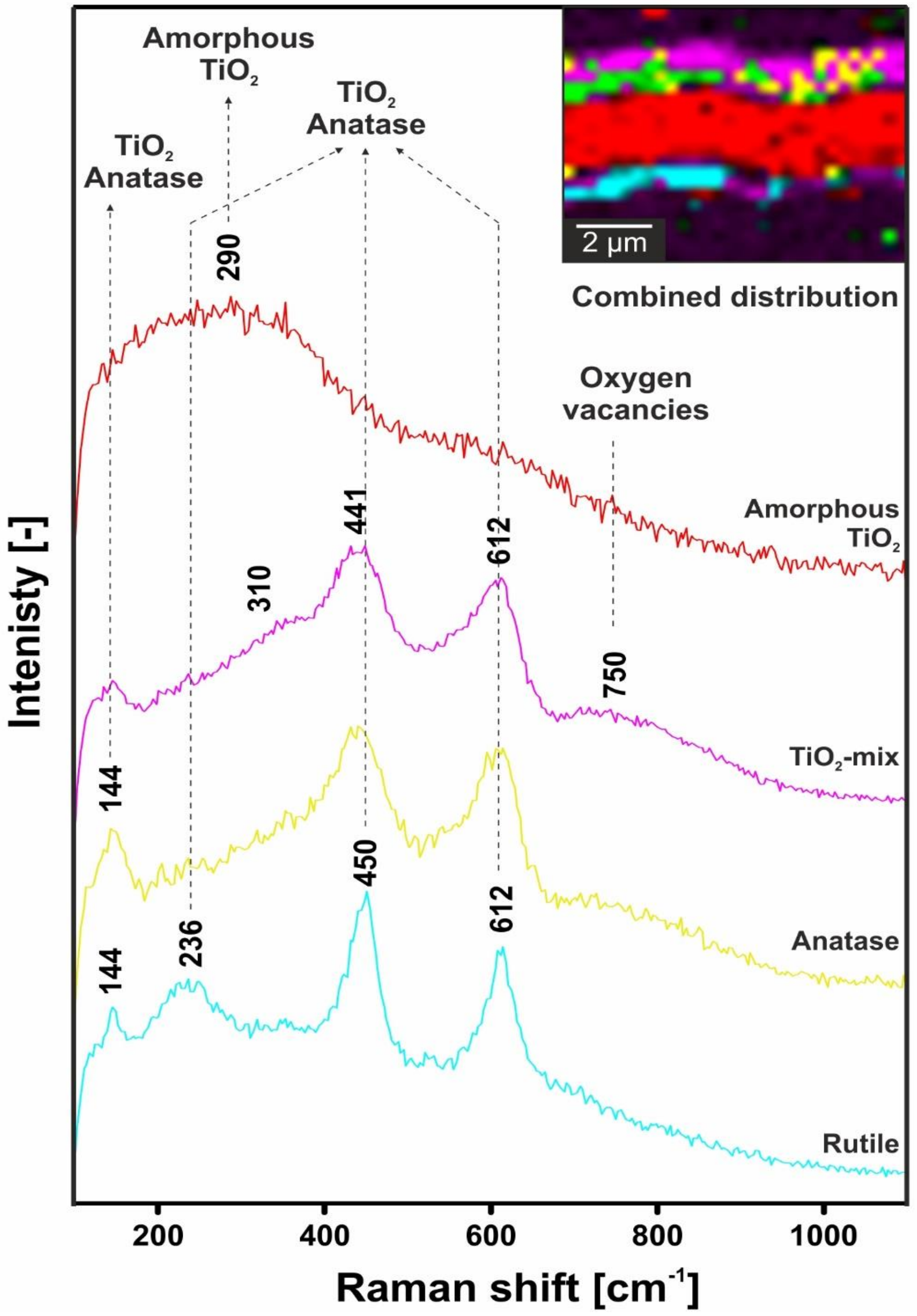
Cross-section of TiO_2 layer
SEM image with EDS elemental maps



Raman imaging of TiO_2 layers cross-section







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NOMATEN Centre of Excellence in Multifunctional Materials for Industrial and Medical Applications

CoE NOMATEN Materials Research Laboratory Thermal Properties Analysis Services

Thermal Properties Testing Laboratory – Research Infrastructure

Thermal Research Laboratory
Financed by Project HTGR

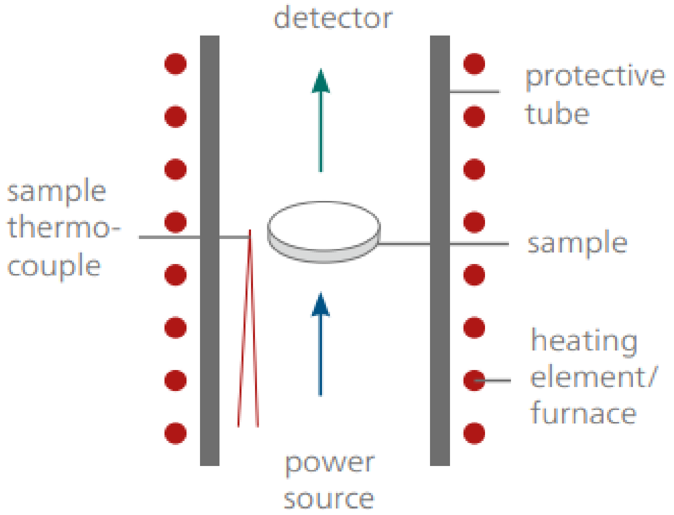
PROJECT HTGR

The Thermal Laboratory enables full characterization of the thermal properties of advanced materials

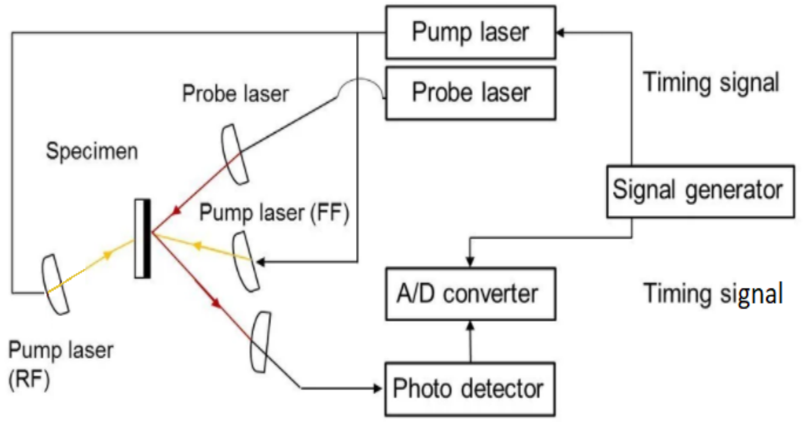
The laboratory equipment includes:

(I) high-temperature dilatometer
(II) device for measuring of thermal diffusivity of volumetric materials,
(III) device for measuring of thermal diffusivity of thin films,
(IV) a set for simultaneous thermal analysis
(V) a thermal mass spectrometer.

High-temperature Dilatometer Netzsch DIL402
Operates in horizontal mode within the temperature range from RT to 1600°C.
The load on the sample is in the range from 50mN to 3N, with measurement of cylindrical samples and cuboidal samples with an accuracy of 1 nm and in the range of measuring 10 mm.



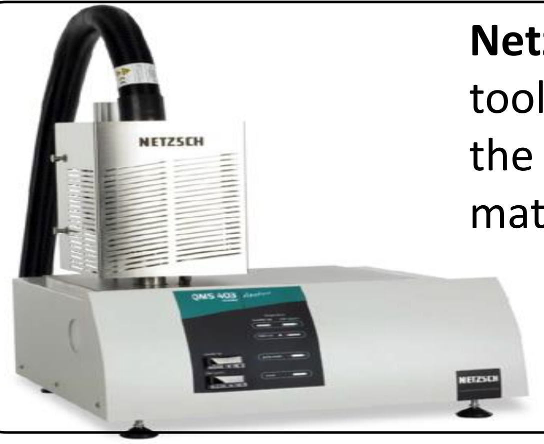
Netzsch LFA 467 *HT HyperFlash*® allows for measurement of **thermal diffusivity and thermal conductivity between RT and 1250°C with Xenon Flash**



NanoTR enables measurements of thermal diffusivity of metallic, ceramic and composite layers in the range from 0.01 to 1000 mm^2/s with an accuracy of 5%.

Netzsch STA 449 F3 Jupiter®
STA instrument combines two measuring techniques: Thermogravimetry (TG) and Differential Scanning Calorimetry (DSC) for a single sample.
The device includes two high-temperature furnaces:

- High-temperature furnace enabling operation in a protective atmosphere (in the range of RT to 1600°C)
- High-temperature furnace enabling operation in a water vapour atmosphere (in the RT to 1250°C range, at a relative humidity in the range of 5-90%.).



Netzsch Mass Spectrometer QMS 403 Aëolos Quadro useful tool for obtaining the chemical and analytical information about the products causing the weight changes of the different materials during heat treatment.

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Final Conclusions

- **We have a research laboratories with high-end infrastructure,** which will be fully equipped and operational by the end of the year 2023
- **We have a management system** under PN-EN ISO/IEC 17025 norm and we can realize **accredited testing** in line with **international research and materials standards ISO, ASTM, BS**…
- **We have a young Staff of Engineers** who continue to expand and develop their competencies…

We invite you to cooperate with us…!!!

Acknowledgements for cooperation to all Partners in

NOMATEN
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Mazovia. heart of Poland

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Ministry of Education and Science

Ministry of Climate and Environment

THANK YOU FOR YOUR ATTENTION

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