FACULTY OF MATHEMATICS AND PHYSICS
Charles University

Nuclear Physics Institute

2021
MECASENS
Prague, November 25-27, 2021

10th International Conference on Mechanical Stress Evaluation by Neutron and Synchrotron Radiation

MECASENS 2021

Program & Abstracts

November 25 – 27, 2021
Prague, Czech Republic

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Contents

Welcome Message

Dear participants,

On behalf of the MECASENS 2021 Committee, we are honored to welcome you to the 10th International Conference on Mechanical Stress Evaluation by Neutron and Synchrotron Radiation (MECASENS) organized in cooperation of the Nuclear Physics Institute of the Czech Academy of Sciences and Faculty of Mathematics and Physics at Charles University, Prague. The conference is held in a hybrid form, i.e., with a choice of full real or full virtual participation on November 25-27, 2021.

The scientific program of the conference, which is focused on the field of neutron, synchrotron, and X-ray strain scanning studies of materials, will consist of numerous thematic sessions.

5 plenary lectures will be presented during the conference. The invited plenary speakers are: ***András Borbély*** *(Mines Saint-Étienne, France),* ***Stefanus Harjo*** *(J-Parc Centre, Japan Atomic Energy Agency, Japan),* ***Anna Paradowska*** *(Australian Centre for Neutron Scattering, Australia),* ***Xun-Li Wang*** *(City University of Hong Kong), and* ***Zhen Zhen Yu*** *(Colorado School of Mines, USA).*

The conference organizers express their gratitude to those who accepted invitations to present plenary and invited lectures and to those who will participate in the conference. They also thank all members of the International and Local Organizing Committees for their contributions to a successful meeting.

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Local Organizing Committee

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Conference information

Venue

The conference takes place on November 25-27, 2021.
The virtual part of the conference takes place on November 25-26, 2021.
The on-site part of the conference will be held on November 27, 2021 in the building of Physical Sciences of the Faculty of Mathematics and Physics (Ke Karlovu 5, Prague 2, room F2 - 1st floor).
The lectures from both, virtual and on-site, parts of the conference, can be followed on the portal: https://mecasens.mikeportal.online.

Registration Desk

Nov. 27 (Sat.) 07:30-12:00

The **secretariat office** will be located in the building of Physical Sciences of the Faculty of Mathematics and Physics (Ke Karlovu 5, Prague 2, room F2 - 1st floor).
The **secretariat office** will be open for participants during the entire day (Sat.).

Presentation guidelines

Oral presentation

The official language of the Symposium is English. Plenary lectures are planned for **45 min + 10 min**, invited talks - **25 min + 5 min** and other oral contributions - **15 min + 5 min**.

Poster presentation

Posters will be available at https://mecasens.mikeportal.online with an option to contact the authors.

Lunch & Coffee Break

Refreshments will be served during on Nov. 27. (Sat.) the coffee breaks (10:30 – 10:50 & 16:00 - 16:20) in the front of F2 lecture hall.

Lunches will be served during in-person conference on Nov.27 (Sat.) at conference site.

Wireless Internet Access

Wireless network ***eduroam*** is available on the place.

Social Program

Beer party

The beer party will be held in Legenda restaurant, Legerova 1820/39, 120 00 Prague, on Nov.27 (Sat.) from 19:00.

Technical program

The virtual part of the conference takes place on November 25-26, 2021.
It can be followed on the portal: https://mecasens.mikeportal.online

The on-site part is held on November 27, 2021, at Faculty of Mathematics and Physics, Charles University, Ke Karlovu 5, 121 16 Prague 2, lecture room F2 (1st floor). Live streaming of the lectures can be followed on the portal: https://mecasens.mikeportal.online

Plenary Speakers

VIRTUAL

Nov. 25 (Thu.) 08:00 — **A. Paradowska** — ANSTO, Australia
Industrial applications of neutron diffraction

Nov. 25 (Thu.) 16:00 — **Z. Yu** — Colorado School of Mines, USA
In-situ and ex-situ diffraction studies of material behavior during welding

Nov. 26 (Fri.) 8:00 — **S. Harjo** — J-Parc, Japan
In situ neutron diffraction for monitoring of internal stresses, deformation & transformation behavior of engineering materials

Nov. 26 (Fri.) 14:00 — **X.-L. Wang** — City University of Hong Kong
Low-temperature deformation in high-entropy alloys

IN-PERSON

Nov. 27 (Sat.) 08:30 — **A. Borbély** — Mines Saint-Étienne, France
Microstrain distribution in crystals

Conference at glance

Day	Time	Aula (1st floor)
Nov. 25 (Thursday) Virtual	7:50-8:00	Opening Ceremony
	8:00-8:55	Plenary lecture Paradowska, A.
	9:00-12:20	Oral Presentations
	12:20-14:00	*Lunch*
	14:00-16:00	Oral Presentations
	16:00-16:55	Plenary lecture Yu, Z.
	17:00-17:30	Invited lecture Balogh, L.
	17:30-18:00	Invited lecture Noyan, I.C.
	18:00-20:20	Oral Presentations
Nov. 26 (Friday) Virtual	8:00-8:55	Plenary lecture Harjo, S.
	9:00-9:30	Invited lecture Polatidis, E.
	9:30-12:10	Oral Presentations
	12:10-14:00	*Lunch*
	14:00-14:55	Plenary lecture Wang, X.-L.
	15:00-15:30	Invited lecture Samothrakitis, S.
	15:30-20:10	Oral Presentations
	20:10-20:40	Invited lecture Brügger, A.
Nov. 27 (Saturday) On-site & live streaming	8:30-9:25	Plenary lecture Borbely, A.
	9:30-10:00	Invited lecture Thiry, M.
	10:00-10:30	Invited lecture Marciszko-Wiąckowska, M.
	10:30-10:50	*Coffee break*
	10:50-13:00	Oral Presentations
	13:00-14:00	*Lunch*
	14:00-14:30	Invited lecture Beran, P.
	14:30-15:00	Invited lecture Farkas, G.
	15:00-16:00	Oral Presentations
	16:00-16:20	*Coffee break*
	16:20-18:00	Oral Presentations
	20:10-20:40	Invited lecture Brügger, A.

Posters will be available on-line during entire conference.

Virtual conference

Nov. 25 (Thursday)

07:50-08:00	**Lukáš, P.:** Opening Ceremony
08:00-08:55 Plenary	**Paradowska, A.:** Industrial applications of neutron diffraction
09:00-09:20	**Yescas, M.:** Neutron diffraction residual stress measurements of a nuclear power plant valve with hardfacing material
09:20-09:40	**Tomota, Y.:** Neutron Bragg-edge transmission imaging for FSSW-joined Mg alloy plates
09:40-10:00	**Marais, D.:** Residual stress in a thick Al 7050 T7451 plate
10:00-10:20	**Tsumura, Y.:** Investigation of residual stress and mechanical properties of steelwork after laser cleaning
10:20-10:40	**Glaser, D.:** The use of Bragg edge neutron transmission for evaluation of strains produced by laser shock peening for low pressure steam turbine blade applications
10:40-11:00	**Naeem, M.:** Deformation pathway of CrMnFeCoNi high-entropy alloy at low temperatures (15 – 295 K) – an in situ neutron diffraction study
11:00-11:20	**Gong, W.:** In-situ neutron diffraction study of deformation behavior of AZ31 alloy at 21K
11:20-11:40	**Mao, W.Q.:** Strain hardening behavior of metastable austenitic steel with TRIP effect: Insights from stress and strain partitioning
11:40-12:00	**Hayashi, Y.:** Type III stress measurement using scanning 3DXRD
12:00-12:20	**Kot, P.:** Direct diffraction measurement of critical resolved shear stresses and grain stresses in magnesium alloy
12:20-14:00	***Lunch Break***
14:00-14:20	**Yang, D.:** Annealing of focused ion beam damage in gold microcrystals: an in situ Bragg coherent X-ray diffraction imaging study
14:20-14:40	**Tapar, O.B.:** In-situ monitoring of microstructure evolution and stress generation during low pressure carburizing and quenching

14:40-15:00	**Wronski, S.:** The second order plastic incompatibility stresses in hexagonal polycrystalline materials
15:00-15:20	**Epp, J.:** Fast in-situ analysis of temperature and stress fields during grinding of steel by high-energy X-ray diffraction
15:20-15:40	**Charni, D.:** In-situ analysis of strain fields during rotary swaging of steel using synchrotron X-ray radiation
15:40-16:00	**Buxton, O.G.:** Investigating lattice strains and phase transformation in hydrogen charged Zirconium
17:00-17:30 Plenary	**Yu, Z.:** In-situ and ex-situ diffraction studies of material behavior during welding
17:00-17:30 Invited	**Balogh, L.:** Irradiation defects in Zr alloys: a comparison between transmission electron microscopy and diffraction line profile analysis
17:30-18:00 Invited	**Noyan, I.C.:** Investigation of precision, resolution, accuracy and trueness of time-of-flight neutron diffraction strain measurements
18:00-18:20	**Marais, D.:** Minimization of texture influences in diffraction assessments of solid samples
18:20-18:40	**Klaus, M.:** Reassessment of evaluation methods for the analysis of near-surface residual stress fields using energy-dispersive diffraction
18:40-19:00	**Simon, N.:** On the oscillating course of 2Θ-$\sin^2\psi$ plots for plastically deformed, cold rolled duplex stainless steel sheet
19:00-19:20	**Otte, A.L.:** Diffraction analysis of phase transformation behavior and stress development in short-term heat treatment of Ti-6246
19:20-19:40	**Pulvermacher, S.:** Load partitioning and micro residual stress development of two duplex steels with different phase contents
19:40-20:00	**Burca, G.:** Recent developments on the IMAT diffraction project
20:00-20:20	**Pirling, T.:** New approaches for in-situ measurements at the SALSA strain scanner

Virtual conference

Nov. 26 (Friday)

08:00-08:55 Plenary	**Harjo, S.:** In situ neutron diffraction for monitoring of internal stresses, deformation & transformation behavior of engineering materials
09:00-09:30 Invited	**Polatidis, E.:** Studying the TRIP effect under multiaxial loading using neutron diffraction
09:30-09:50	**Woo, W.:** Through-thickness variations of residual stresses in functionally graded steel-stainless steel structures additively manufactured by direct energy deposition
09:50-10:10	**Chae, H.:** Tailoring mechanical properties of metallic materials via additive manufacturing followed by heat treatment
10:10-10:30	**Kim, Y.S.:** Multiple deformation scheme in direct energy deposited CoCrNi medium entropy alloy at 210K
10:30-10:50	**Ostergaard, H.E. :** Microstructure and residual stress interactions in metal additive manufacturing: post-build assessment and new in-situ methods
10:50-11:10	**Evans, A.:** Residual stresses in additive manufacturing determined by diffraction techniques
11:10-11:30	**Serrano-Munoz, I.:** Influence of the scanning strategy on the RS state of a LPBF IN718 material
11:30-11:50	**Moztarzadeh, H.:** Residual stress in plasma transferred arc (PTA) cladding for hybrid additive manufacturing (AM)
11:50-12:10	**Yong, C.K.:** Synchrotron XRD Evaluation of Residual Stress Distribution for Additive Manufactured Inconel 718 for High Temperature Applications
12:10-12:30	**Abreu Faria, G.:** P61A, a new white beam beamline optimized for residual stress analysis
12:30-14:00	***Lunch Break***
14:00-14:55 Plenary	**Wang, X.-L.:** Low-temperature deformation in high-entropy alloys

15:00-15:30 Invited	**Samothrakitis, S.:** Microstructural characterization through grain orientation mapping with Laue three-dimensional neutron diffraction tomography
15:30-15:50	**Larsen, C.B.:** Grain-resolved strain analysis with Laue three-dimensional neutron diffraction tomography
15:50-16:10	**Rouquette, S.:** Validation of plane stress assumption on SS316L specimen with one layer
16:10-16:30	**Silveira, A.C. de F.:** Microstructure and stress development during laser metal deposition analyzed by synchrotron X-ray diffraction
16:30-16:50	**Degener, S.:** Material science with a new high energy white beam station – Prospects and challenges
16:50-17:10	**Landesberger, M.:** High accuracy neutron diffraction measurement and positioning with an industrial robot system at the STRESS-SPEC instrument
17:10-17:30	**Genzel, Ch.:** A concept for residual stress gradient analysis in cubic materials with mosaic structure
17:30-17:50	**Ramadhan, R.S.:** Quantitative analysis and benchmarking of positional accuracies of neutron strain scanners
17:50-18:10	**Apel, D.:** The potential of high-flux liquid anode X-ray sources for microstructure and stress analysis
18:10-18:30	**Cui W.:** Ferritic benchmark specimens for cross-comparison of diffraction and destructive residual stress measurement techniques
18:30-18:50	**Wimpory, R.C.:** Strain Scanning on E3 at BERII at the HZB, a retrospective
18:50-19:10	**Venter, A.M.:** Residual stress in sintered WC-VC-Co disks
19:10-19:30	**Lavanya, S.:** Effect of tensile strain on martensite formation and its influence on residual stress distribution in type 304 austenitic stainless steel
19:30-19:50	**Nielsen, M.-A.:** Residual stresses in additively manufactured aluminum alloys and 316l-steel
19:50-20:10	**Brown, D.:** In-situ heat treatment of additively manufactured Ti-6Al-4V

20:10-20:40 **Invited**	**Brügger, A.:** Protecting Suspension Bridges against Fire with Neutron Diffraction

On-site conference & live streaming

Nov. 27 (Saturday)

08:30-09:25 **Plenary**	**Borbély, A.:** Microstrain distribution in crystals
09:30-10:00 **Invited**	**Thiry, M.:** When industry meets large facilities **(virtual lecture)**
10:00-10:30 Invited	**Marciszko-Wiąckowska, M.:** In-depth evolution of residual stresses and effect of free surface on stress relaxation determined using X-ray diffraction Laplace methods
10:30-10:50	***Coffee Break***
10:50-11:10	**Sobotková, N.** - Delivery of neutron optics system for the BEER diffractometer in ESS
11:10-11:30	**Donath, T.:** EIGER2 CdTe detectors for hard X-ray research
11:30-11:50	**Šittner, P.:** Oriented internal stress in plastically deformed NiTi shape memory alloys
11:50-12:10	**Henningsson, A.:** A framework for equilibrium constrained strain estimation and tomography
12:10-12:30	**Wierzbanowski K.:** Modification of mechanical properties and microstructure of titanium grade 2 processed by hydrostatic extrusion
12:30-12:50	**Ozcan, B.:** In-situ neutron strain imaging during direct metal deposition of Ni-based Inconel 718 alloy
12:50-14:00	***Lunch Break***
14:00-14:30 **Invited**	**Beran, P.:** The material engineering diffractometer BEER at ESS
14:30-15:00 **Invited**	**Farkas, G.:** Line profile analysis and rocking curve evaluation in individual grains of β-Ti polycrystal

15:00-15:20	**Canelo-Yubero, D.:** Residual stresses in Al-Cu clad composites processed by rotary swaging
15:20-15:40	**Čapek, J.:** Optimization of post-built annealing of Ni Alloy718 processed by powder bed fusion
15:40-16:00	**Németh, G.:** Residual stresses in Titanium prepared by CONFORM ECAP
16:00-16:20	***Coffee Break***
16:20-16:40	**Heller, L.:** Prediction of martensite textures in NiTi wires
16:40-17:00	**Bian, X.:** In-situ synchrotron x-ray diffraction texture analysis of tensile deformation of nanocrystalline NiTi wire in martensite state
17:00-17:20	**Kehres, J.:** Utilization of laboratory energy dispersive X-ray diffraction for stress determination in polymers as a supplement to synchrotron experiments
17:20-17:40	**Abreu Faria, G.:** P61A, a new white beam beamline optimized for residual stress analysis
17:40-18:00	**James, A.:** Tomography driven diffraction capabilities of the new DIAD beamline

Poster presentations

P1 **Drozdenko, M.:**
Configuration of deformation rig developed for beamline at European Spallation Source

P2 **Olsen, U.L.:**
Depth resolving stress in amorphous polymers

P3 **Leemreize, H.:**
Multiscale residual stress analysis using x-ray and neutron dark-field microscopy

P4 **Leemreize, H.:**
Harmonisation and standardization of industrial residual stress measurement using neutrons and synchrotron x-rays

P5 **Nguyen T.D.:**
Microscopic stress-strain evaluation of age-hardened AA7075 during repeated stress relaxation at elevated temperature

P6 **Baczmański, A.:**
New analysis method of multireflection grazing incidence X-ray diffraction

P7 **Oponowicz, A.:**
Saccharine effect on the microstructure and stress state in nickel electrodeposited on copper substrate

P8 **Setoyama, D.:**
Diffraction measurement condition suitable for stress analysis of Polyphenylenesulfide component

P9 **Setoyama, D.:**
Non-destructive analysis of lead-free solder degradation in power electronic module by neutron diffraction

P10 **Kendall, O.:**
Influence of heat treatment on the residual stress in laser clad hypereutectoid rail components using neutron diffraction

P11 **Chae, H.:**
The effect of carbon content on deformation mechanisms of high Mn steels at elevated temperature

P12 **Su, Y.H.:**
Strain and microstructure distributions around a fatigue crack tip studied by neutron diffraction

Abstracts

Industrial applications of neutron diffraction

Paradowska, A.

Australian Centre for Neutron Scattering (ACNS, ANSTO)

The OPAL research reactor at ANSTO has several instruments available for materials science and engineering applications. The instruments have a unique non-destructive ability to determine critical imperfections assist performance of engineering apparatus via radiography and tomography, measure internal residual stresses and textures in crystalline materials, such as metals, alloys, ceramics, and composites. These measurements can be carried out on real engineering components, mock-ups, or test samples with minimal preparation. This information provides direct impact into optimization of modern manufacturing processes, improved product reliability, enhanced design performance, reduced production cost, and extended life prediction on significant engineering assets. The versatile team has established a strong record in assisting Australian and international researchers and engineers across a wide range of engineering projects.

Over time, we have built an exceptional body of skills, experience and technical expertise, which is now on offer to support industrial research and development.

The Industrial Liaison Office (ILO) at the Australian Centre for Neutron Scattering was set up in April 2014 to promote the use of the ANSTO neutron facilities for applied industrial research and to manage technology transfer.

This presentation will focus on the challenges and highlights of first few years of operation.

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Neutron Diffraction Residual Stress Measurements of a Nuclear Power Plant Valve with Hardfacing Material

Yescas M.[1]*, Paddea S.[2], Bouchard P.J.[2], Le Bot L.[1]

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Keywords: Neutron Diffraction; Residual stresses; Valve; Nuclear plant

In recent times Stellite hardfacing material, typically used in nuclear power plant valves, has been replaced by cobalt-free materials in order to reduce the radiation exposure to plant maintenance personnel. However, a new iron-base hardfacing alloy, known as NOREM02, can present high welding residual stress levels. Recent improvements in the manufacturing process have been made to reduce those high residual stress levels. This paper describes neutron diffraction measurements conducted in the NOREM02 deposit of a nuclear power plant valve plug for the purpose of quantifying the residual stresses present. The neutron strain measurements were undertaken at the ENGIN-X beamline of the ISIS Neutron & Muon Source, which is part of the Rutherford-Appleton Laboratory in the UK. Measurements were made at the weld deposit start/stop and diametrically opposite positions along three lines spanning the NOREM02 deposit. The trend and magnitude of residual stresses along the three lines analysed were very similar to each other. The stresses were highest in the hoop direction at the start/stop position.

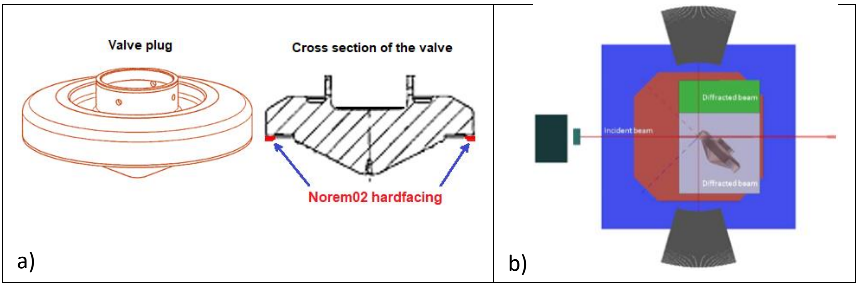

Figure 1: a) Nuclear plant Valve plug with Norem02 hardfacing, b) Simulation of Neutron diffraction residual stress measurements.

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Neutron Bragg-edge transmission imaging for FSSW-joined Mg alloy plates

Kino, K.[1,2], Itoh, T.[2,3], Tomota, Y.[1,2*], Watazu, A.[1,2], Tanaka, M.[1,2], Oshima, N.[1,2]
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Keywords: Neurton Bragg-edge imaging; Mg alloy; FSSW; Rietveld type profile analysis

Mg alloy (AZ31) plates with 3 mm thickness were welded by FSSW. The samples were characterized using pulse neutron Bragg-edge transmission imaging under a four-point bending condition at BL22 (RADEN) of J-PARC MLF. Bragg-edge spectra obtained with a nGEM detector, along the plane normal (ND) and transverse (TD) directions of a sample were analyzed using a Rietveld type profile analysis software, RITS, developed by Sato et al. [1]. Examples of the Bragg-edge specta obtained from the ND and TD measurements for the base plate and joint region are presented in Fig. 1. As seen, the obtained spectara are different from each other. These spectra were well fitted by RITS; the texture was evaluated by March-Dollase parameters while the crystallite size from the primary extinction effect, showing good agreements with the results obtained by SEM/EBSD observations. From the change in TD spectra along the plate thickness, elastic strain distribution under bending was roughly estimated.

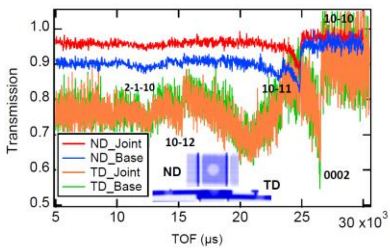

Fig.1 Bragg-edge spectra obtained from TD and ND for the base plate and FSSW joint. region.

[1] H. Sato, et al.: Mater. Trans., 52(2011), 1294-1302.

This work was based on the results obtained from a project commissioned by the New Energy and Industrial Technology Development Organization (NEDO), Japan. The neutron measurements were performed at J-PARC MLF through user program 2018A0157. We wish to thank experimental supports by Drs. Y.H. Su, J.D. Parker and T. Shinohara of J-PARC for neutron measurements and Drs. Y.X. Wang and T. Ohmura of NIMS for SEM/EBSD measurements.

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Residual stress in a thick Al 7050 T7451 plate

Marais, D.[1]*, Seger, M.B.[2], Malik, A.S.[2], Venter, A.M.[1]

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Keywords: residual stress; Al 7050 T7451

Neutron diffraction analyses of the residual stress field were performed on an aluminium series 7050 T7451 plate typically used in the aerospace industry for the manufacture of light weight high strength structures. These were done on a smaller sample with dimensions of ~ 335 × 400 × 90 mm^3 (X,Y,Z) and 31 kg in weight cut from a large plate which was hot rolled during the manufacturing process before it was heat treated and mechanically stress relieved.

Investigations were performed using the angular dispersive neutron diffraction stress measurement technique as employed at the MPISI instrument at the SAFARI-1 research reactor. Measurements of the lattice plane spacings respectively along three orthogonal directions were taken at discrete points along a 3-dimensional grid that covered the entire volume of the sample. The neutron gauge volume was fully embedded at all the measurement positions.

Determination of stress-free lattice plane spacings from a single representative cube proved inaccurate. This was remedied by having cubes smaller than the neutron gauge volume, taken through the plate depth (Z positions) with retention of the directional d_0 spacings. These results enabled calculation of the tri-axial residual stresses at each measurement position by incorporating the elastic moduli of the alloy.

Results show that the residual stresses are generally constant over the XY planes (constant Z) and have prominent variations through the plate depth which lie in a band between -30 to +30 MPa. With depth, the stress profile is symmetric about the mid plate plane with compressive stresses in the outer surface regions (up to ~20% depth from both surfaces) and tensile in the centre. These values are low compared to the certified material yield strength (427 to 472 MPa) and confirms that the plate had been subjected to stress relieve treatments. The compressive stresses in the surface regions would be beneficial to mitigate surface cracks. Preliminary depth resolved crystallographic texture measurements will also be presented.

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Investigation of Residual Stress and Mechanical Properties of Steelwork After Laser Cleaning

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Keywords: keyword; Laser ablation, residual stress, microstructure, metals and alloys.

Surface preparation of steelwork for structural repainting is often conducted by sandblasting method in which abrasives (sands) are blasted onto the painted surface at high speed/, removing the old paint and rust/dirt by the impact of the blast. This conventional method would cause irreversible damage to the underlying substrate, deteriorating the mechanical and fatigue performance. Laser cleaning has attracted attention as an alternative to conventional cleaning methods as an environmentally friendly and economical technology that removes paint and corrosion efficiently while inducing minimal damage towards the surface of the material.

This research investigates the mechanical properties and residual stresses of the laser cleaned steel samples from the Sydney Harbour Bridge. Laser cleaning using nanosecond laser was performed on the structural steel plates removed from the Sydney Harbour Bridge. The plates were then tested at the Australian Nuclear Science and Technology Organisation (ANSTO) for residual stress measurement and the University of Sydney for the microstructure characterization and microhardness testing.

A modelling work has also been carried out to get a better understanding of the experimentally observed changes in residual stress distribution and material properties. The model presented here incorporates a physical and mathematical model required to handle complex multi-physics phenomena, such as laser-substance interactions, thermal-mechanical interactions between melt pool and solid, and melting and re-solidification processes. Using this finite element model, simulations were run to determine the region in the material that is subjected to phase transformation and to determine how residual stresses change in the steel during laser ablation.

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The use of Bragg Edge Neutron Transmission for Evaluation of Strains Produced by Laser Shock Peening for Low Pressure Steam Turbine Blade Applications

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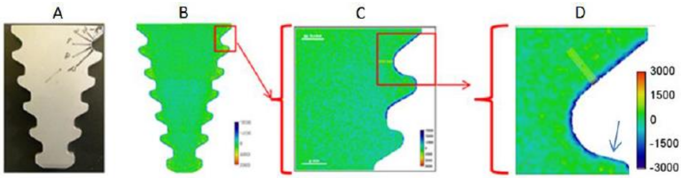
Keywords: Strain Mapping, Laser Shock Peening, Bragg Edge Neutron Transmission

Low Pressure steam turbine blades typically receive Mechanical Shot Peening (MSP) in the blade attachment regions to mitigate crack-based mechanisms such as fatigue and stress corrosion cracking [1]. Laser Shock Peening (LSP) is currently being developed due to deeper levels of compressive residual stress achieved with a lower surface roughness compared to MSP. This investigation focussed on the evaluation of strains produced by the LSP process on a scale 2D slice of the component geometry to ensure consistency of the surface treatment. Bragg Edge Neutron Transmission at the IMAT facility at ISIS, STFC (UK) is utilised, as the technique allows for strain mapping over a complex surface, through the depth of the sample, with a high degree of spatial resolution. An example of resulting strain maps for a 15 mm thick 2D slice of the blade attachment is shown in the images A-D below.



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Deformation pathways of multicomponent medium- and high-entropy alloys at cryogenic temperature

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Keywords: high-entropy alloys; cryogenic deformation; stacking faults; deformation twinning; martensitic transformation; dislocation density; neutron diffraction

High-entropy alloys are multicomponent materials that exhibit an increase in both strength and ductility at cryogenic temperatures. The unusual improvement in their ductility at low temperatures has recently sparked a lot of interest. The activation of multiple deformation mechanisms, which are responsible for outstanding mechanical properties at low temperatures, has been reported. However, a clear picture of deformation pathways at ultralow temperatures has been lacking. To address this issue, we used time-of-flight neutron diffraction to investigate the cryogenic deformation behavior of equiatomic CrMnFeCoNi, CrFeCoNi, and CrCoNi multicomponent alloys. The evolution of lattice strain and texture along different orientations provided the distinct deformation behavior at low temperatures. In contrast to deformation by dislocation slip at room temperature, the cryogenic deformation also involved stacking faults, twinning, and martensitic transformation (for CrCoNi alloy). The in situ data revealed the early activation of stacking faults and a higher stacking fault probability with decreasing the deformation temperature. The deformation at ultralow temperature also involved massive serrations. The samples at ultralow temperature deformed more uniformly and had a negligible necking region, thus imparting excellent ductility. This study sheds light on the activation and interaction of multiple deformation mechanisms at cryogenic temperatures, thus outlining the deformation pathway responsible for the extraordinary strength–ductility combination.

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In-situ neutron diffraction study of deformation behavior of AZ31 alloy at 21K

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Keywords: magnesium; neutron diffraction; cryogenic deformation; extension twinning; lattice strain

Magnesium (Mg) and its alloys have potential for application in various fields, and then have to face various environments. In which, the cryogenic temperature applications such as aerospace, storage, and transport of liquid cryogenics, are becoming increasingly important and widespread in modern materials. However, the deformation behavior at cryogenic temperature in Mg alloys is still far from being understood due to the limited studies and lacking method. Here, we conducted *in-situ* neutron diffraction experiments to investigate the temperature dependence of deformation mechanisms at cryogenic temperature in an AZ31 Mg alloy.

Cylinder specimens with a diameter of 8 mm and a length of 16 mm were cut from the a commercial extruded AZ31 Mg alloy bar for *in-situ* neutron diffraction experiments, while the loading direction was parallel to the extrusion direction. *In-situ* neutron diffraction experiments during compressive deformation at 21K and 298K tests were performed by the engineering diffractometer "TAKUMI" at Japan Proton Accelerator Research Complex (J-PARC).

The compressive stress-strain curves show that the strength and fracture strain at 21 K increased simultaneously compared to that at 298 K. The lattice strain results demonstrate that the basal slip exhibited a much higher sensitivity to temperature than the extension twinning. As a result, the extension twinning was enhanced at cryogenic temperature, which can provide additional capacity for strain accommodation and work hardening. Moveover, the contraction double twinning, as known to be the crack initiation source in Mg alloys, was suppressed at 21K. The different sensitivity to temperature of various deformation modes is considered to be the reason for the simultaneous increase in strength and ductility of the commercial AZ31 alloy at 21K.

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Strain hardening behavior of metastable austenitic steel with TRIP effect: Insights from stress and strain partitioning

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Keywords: metastable austenite; martensitic transformation; TRIP; strain hardening; neutron diffraction;

It is well known that the strain hardening of metastable austenite can be enhanced by the deformation induced formation of martensite. In this study, strain hardening behavior of Fe-24Ni-0.3C (wt.%) metastable austenitic steel having deformation induced martensitic transformation (DIMT) during deformation was investigated by tensile test with in-situ neutron diffraction, aiming to clarify the mechanism of the enhanced strain hardening caused by the DIMT. The results suggested that the evolution of phase stress, i.e., average internal stress, of martensite during the deformation plays an important role in the strain hardening. It was found that during deformation the phase stress of martensite firstly increased rapidly from a low value, and then the rate of increase decreased as it approached 1.8 GPa. A dramatic increase in the stress partitioning between austenite and martensite was generated due to the rapid increase of martensite phase stress, which contributed significantly to the increase in the overall strain hardening rate of the material. The analysis of plastic deformation of austenite and martensite reveals that the rapid increase in stress partitioning occurred during the elasto-plastic deformation stage and arose from the occurrence of the plastic strain misfits.

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Type III stress measurement using scanning 3DXRD

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Keywords: 3DXRD; type III stress; steel; plastic deformation

Three-dimensional X-ray diffraction (3DXRD) microscopy allowed us to measure grain-resolved (type II) stress tensors for grains deeply embedded in bulk alloys [1]. The type II stress measurement is useful for characterizing mechanical behavior of alloys [2]. However, 3DXRD cannot be used to measure intragranular local (type III) stresses, which can facilitate the understanding and prediction of plastic deformation and failure of alloys. Here, the measurement method of intragranular local (type III) stresses is shown using scanning 3DXRD [3]. In this method, multiple grains embedded in a bulk sample are illuminated by a synchrotron-based high-energy X-ray microbeam with a beam size smaller than a grain size. Non-overlapped multiple diffraction spots per grain are detected through a conical slit. Type III elastic strain tensors are determined from the differences between observed scattering angles and stress-free Bragg angles. Thus, a map of voxel-resolved stress tensors is obtained. The result of a scanning 3DXRD experiment for plastically deformed low-carbon steel is delivered in this presentation.

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Direct diffraction measurement of critical resolved shear stresses and grain stresses in magnesium alloy

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Keywords: neutron diffraction; slip systems, twinning process; self-consistent modelling, magnesium alloy

A new methodology based on neutron diffraction experiment was developed in order to measure stresses localised in polycrystalline grains during plastic deformation. In this method lattice strains were determined at different orientations of scattering vector and different hkl reflection during tensile and compression tests [1]. The method was used for textured Mg alloy and the components of stress tensor were determined directly from measured lattice strains corresponding to chosen orientations of crystallite lattice. Using the experimental data the evolution of von Mises stress was calculated for selected groups of grains. As the result, a large difference in the hardness of crystallites having different lattice orientations was found. Moreover, the critical resolved shear stress (CRSS) for different slip systems and for tensile twinning were determined directly from the experiment. Experiment allowed us to determine grain stress evolution during and after twinning process.

The neutron measurements were conducted on TKSN 400 (HK9) diffractometer in the Nuclear Physics Institute in Řež (Czech Republic), and on EPSILON-MSD and FSD diffractometers in Joint Institute for Nuclear Research in Dubna (Russia).

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This work was partially supported by grants from the National Science Centre, Poland (NCN), No. UMO-2017/25/B/ST8/00134. Przemyslaw Kot has been partly supported by the EU Project POWR.03.02.00-00-I004/16

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Annealing of focused ion beam damage in gold microcrystals: an *in situ* Bragg coherent X-ray diffraction imaging study

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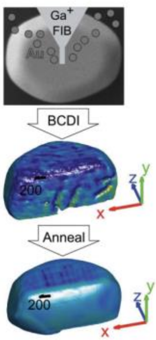
Keywords: Bragg coherent X-ray diffraction imaging; annealing; strain mapping; cross-correlation; facet growth

Gallium ion focused ion beam (FIB) techniques are commonly used to machine, analyse, and image materials at the micro and nanoscale. However, FIB milling can induce defects[1], which can cause undesired lattice distortions that modify the integrity of the sample. Methods have been developed to minimize FIB-induced sample damage, but these protocols need to be evaluated for their effectiveness. Here, we study the in situ annealing of FIB-milled gold microcrystals using non-destructive Bragg coherent X-ray diffraction imaging (BCDI)[2]. Two reflections, which provide strain information in two non-collinear directions, are simultaneously measured for two different crystals during a single annealing cycle. We evaluate the annealing process using various methods: facet area evolution, average strain plots, and cross-correlation maps of the displacement field and binarized morphology. Here, we attribute the evolution of the crystal's strain and morphology to be caused by the diffusion of gallium in gold below ~280°C and the self-diffusion of gold above ~280°C. The majority of FIB-induced strains are removed by 380–410°C, depending on the reflection being considered. These observations demonstrate the ability anneal out FIB-induced strain, at the expense of a slight change in morphology. This experiment further highlights the importance of measuring multiple BCDI reflections to unambiguously interpret material behaviour.



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In-situ monitoring of microstructure evolution and stress generation during low pressure carburizing and quenching

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Keywords: Low pressure carburizing; synchrotron diffraction; stress development; carbon diffusion; carbide formation; carbide dissolution

The low-pressure carburizing (LPC) is an experience-based process for surface layer hardening. To extend the process understanding, a systematic LPC parameter study was carried out based on in-situ X-ray diffraction experiments at the German Electron Synchrotron Facility (DESY) in Hamburg (Germany) using a specially developed process chamber. The experiments provide fundamental information about the evolutions of both microstructrual composition as well as residual stresses.

Regardless of the chosen process parameters, carbon saturation in austenite was reached whithin a few seconds during the carburizing step, leading to carbide formation at the surface.

During quenching, thermal stresses are induced as soon as the temperature started to drop. The carbon depth distrubution leads to an in-depth gradient of the martensite start temperatures. A combination of different cooling rates between the surface and the core, and the depth gradient of the martensite start temperatures caused inhomogeneous martensitic transformation throughout the material.

Additionally, martensite formed at the surface in the range of the martensite start temperature had a lower c/a ratio than the martensite formed at significantly lower temperatures. This difference is credited to the early transformation of austenite regions having lower carbon contents. Furthermore, it was noticed that the final carbon content determined in the martensite is lower compared to the carbon content of the austenite before quenching. This reduction is attributed to self-tempering effects, which affect the induced residual stress state as well.

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The second order plastic incompatibility stresses in hexagonal polycrystalline materials

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Keywords: X-ray diffraction; Second order stresses; Self-consistent modelling; Elastoplastic deformation

One of the important reasons for the formation of residual stresses in polycrystalline materials is the anisotropy of the plastic deformation process. Different slip systems activity leads to different plastic deformations of polycrystalline grains. The resulting misfit (incompatibility) between neighboring grains is the source of the second order incompatibility stresses. These stresses cannot be measured directly but can be predicted by elastoplastic deformation models. They are correlated with the nonlinearity of lattice strains $<\varepsilon(\psi,\phi)>_{\{hkl\}}$ vs. $sin^2\psi$ plots, determined experimentally [1].

In this work the stresses in deformed magnesium and titanium alloys are studied [3]. The grazing incidence X-ray diffraction measurements performed during "in situ" tensile test and a novel method of experimental data interpretation allowed to determine evolution of macroscopic and second order stresses during elastic-plastic deformation followed by sample unloading. Additionally the values of the critical resolved shear stresses were determined for slip systems activated during plastic deformation. Significant second order plastic incompatibility stresses were found in the magnesium sample, while these stresses were much smaller in the titanium alloy.

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Fast in-situ analysis of temperature and stress fields during grinding of steel by high-energy X-ray diffraction

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Keywords: in situ measurement; synchrotron radiation; grinding process; internal material load; residual stresses; process signature

In situ measurements during grinding process were performed using high-energy synchrotron X-ray diffraction in transmission with a specially designed grinding machine at the synchrotron radiation facility PETRA III at DESY in Hamburg, Germany at beamline P07. Measurements were performed at steel samples around the contact zone of the grinding wheel with time resolution up to 100 Hz by using fast Pilatus 2D detector. Local strain determined from several vertical z-scans below the processed surface at several positions around the process zone allow for a 2D reconstruction of the internal thermomechanical material load and the material modifications being introduced into the surface layer. Based on evaluation methods already developed from mechanical deep rolling process analysis [1], strain and stress components as well as peaks` full-width at half maximum could be analyzed to evaluate thermal and mechanical contributions through hydrostatic and deviatoric stress components. This first attempt makes it possible to separate the effects of these process influences and assess the effect of the grinding parameters for different settings and initial material states.

The results show the thermal gradients and contact load distribution that create the specific compressive longitudinal residual stress observed in the grinding process and make it possible to correlate the experimentally measured values, to be compared with previous studies with ex situ stress measurements and simulated temperature data [2].

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In-situ analysis of strain fields during rotary swaging of steel using synchrotron X-ray radiation

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Keywords: Rotary swaging, in-situ cold forming, synchrotron radiation, residual stress, steel

The automotive and aerospace industries are seeking for manufacturing lightweight components with a possibility of reducing the manufacturing cost and energy consumption. Therefore, the production shifts to innovative process chains using hollow steel components with improved mechanical properties and skipping heat treatment processes. In this context, rotary swaging was investigated to analyze the process conditions and the resulting material modifications in order to control and optimize the mechanical properties of the finished parts. Rotary swaging is an incremental cold forming process for axisymmetric components. It can be used to reduce the diameter and profile the workpiece with radial oscillating dies. In this work, analogous process to rotary swaging was performed allowing transmission measurements for in-situ synchrotron X-ray radiation investigation to characterize and map the material modifications generated in steel rings under load and after unloading at several steps of rotary swaging process. The determined stress maps showed inhomogeneous stress distribution in the ring cross section under load and strong stress gradients were observed. This inhomogeneity was found to be smaller with smaller stroke height. The residual stresses gradually build up with successive strokes but after several strokes, the residual stresses are reduced with further strokes, especially after the final calibration strokes.

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Investigating Lattice Strains and Phase Transformation in Hydrogen Charged Zirconium

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Keywords: SXRD, Zirconium, Hydrides, Transformation, Nuclear
Zirconium alloys continue to be the primary structural material in nuclear reactor cores but prolonged exposure to the reactor environment leads to the absorption of hydrogen and eventual precipitation of hydrides. The presence of hydrides deteriorates the mechanical properties of the material, which is of particular concern during transportation and storage of used nuclear fuel rods, yet the deformation behaviour of the hydrides remains unclear [1, 2]. Results from recent in-situ synchrotron X-ray diffraction experements are presented, where potential mechanisms associated with the extreme lattice strains observed in Zirconium hydrides under loading [1,2] are investigated. In-situ loading experiments were conducted using high energy synchrotron x-ray diffraction on fully recrystalised Zircaloy 4 samples charged to 400ppm hydrogen and heat treated to generate fine, medium and coarse hydride morphologies. In the case of the fine hydride morphology condition the δ-(111) develops an extraordinary large elastic strain while a γ-(111) ZrH peak appears after 2% strain suggesting a phase transformation exists under specific microstructural conditions, Figure 1. Repeat loads on a sample with the transformed γ-ZrH fail to reproduce the extreme lattice strains as seen in δ-ZrH peaks indicating this structure is more stable under loading, Figure 2. The transformed γ-(111) ZrH peak does not appear to be stabilised by stresses of up to 300MPa when heated to temperatures above the γ → δ phase boundary [3] suggesting the transformation happens more readily during plastic deformation,

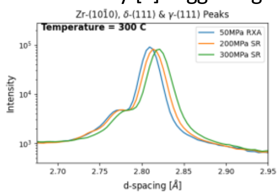

Figure 3.

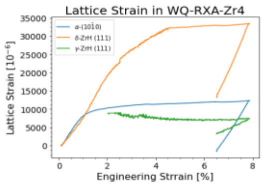

Figure 1: Extreme strain in d-(111) and appearance of g-(111)

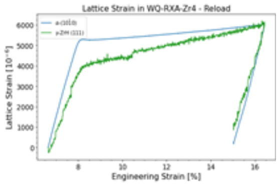

Figure 2: No extreme strain response in g-(111)

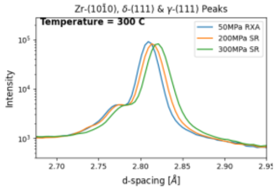

Figure 3: Prescence of the g-(111) peak under load and temperature

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In-situ and Ex-situ Diffraction Studies of Mateiral Behavior during Welding

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Keywords: Solidification; Non-equilibrium phase transformation; residual stress; neutron diffraction; synchrotron diffraction

A fundamental understanding of the transient and nonequilibrium material behavior and the residual stress evolution in welding processes is essential in the pursuit of process control and optimization to produce defect-free and reliable welds, and prolong their service life in harsh environments.

In-situ diffraction techniques using neutron and synchrotron x-ray enable monitoring of solidification and far-from equilibrium phase transformation behavior during welding of complex alloy systems (such as dual phase advanced high strength steels and Ni-base supperalloys) using newly developed fillers with sub-second temporal resolutions. Such a fundamental understanding provides critical inputs for filler metal development (e.g., multi-principal-component alloy fillers and low transformation temperature welding consumables) to generate welds matching the properties of advanced base metals. The deep penetration capability of neutrons enables ex-situ measurement of weld-induced residual stress distributions in various steel grades. Such information provides critical guidelines for optimization of weld procedure and post weld heat treatment for mitigation of failure mechanisms such as stress relaxation cracking and stress corrosion cracking in various industrial applications.

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Irradiation defects in Zr alloys: a comparison between transmission electron microscopy and diffraction line profile analysis

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Keywords: irradiation defects; diffraction line profile analysis; Zr alloys

Irradiation induced dislocations significantly affect the mechanical properties of Zr alloys, altering slip and influencing creep and growth. Thus, the quantitative characterisation of irradiation defects is important for models that attempt to predict their impact on mechanical properties. Whole pattern diffraction line profile analysis (DLPA) is a modern tool for microstructure characterization based on first-principle physical models, well-established for dislocation density measurements in plastically deformed materials. However, applying these DLPA methods directly to irradiated materials yields higher than expected dislocation density values in comparison to historical TEM measurements. To understand these differences, a new microstructural model was developed for DLPA to specifically address dislocation structures consisting of elliptical <a> and <c>-component loops. For comparison, unirradiated and irradiated Zr-2.5Nb samples were characterized with both the refined DLPA method and with TEM measurements. The capabilities of both methods will be compared, and the main sources of uncertainties will be discussed.

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Investigation of precision, resolution, accuracy and trueness of time-of-flight neutron diffraction strain measurements

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Keywords: lattice strain, precision, resolution, accuracy, trueness

The results of a simple statistical analysis which yields the precision, resolution, accuracy and trueness of diffraction-based lattice strain measurements will be presented [1]. The procedure consists of characterizing the thermal expansion induced in each component of an ideal, non-reacting, two-component, crystalline powder sample *in situ*. One component, with a high coefficient of thermal expansion (CTE), serves as an internal thermometer. The quantities of interest are obtained by determining the smallest, statistically significant, thermal lattice strain which can be detected through diffraction analysis in the second, low CTE, component in response to controlled temperature changes. This procedure also provides a robust check of the alignment of the diffraction system and is able to reveal the presence of systematic errors. The application of this technique to a time-of-flight engineering diffractometer/strain scanner will also presented, and implications of this analysis on strain/stress measurements with diffraction techniques will be discussed.

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Minimization of Texture Influences in Diffraction Assessments of Solid Samples

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Keywords: crystallographic texture; powder diffraction

The Rietveld refinement technique is used to extract phase specific information from crystalline materials by analyzing the Bragg diffraction peaks with respect to position, width and intensity. As peak intensities are highly dependent on the presence of crystallographic texture in a sample, eliminating this effect prior to refinement will improve results. The Rietveld technique does enable 1^{st} order corrections for texture contributions by means of spherical harmonics and March-Dollase function perturbations to the structural models, but these are inadequate in many cases.

Texture can be eliminated by grinding a solid sample into a fine powder, thereby reducing the grain sizes and randomizing the crystallite orientations, but this method is not always practical. Bunge has noted that texture effects in a solid sample can be eliminated by integration of the intensities over all sample directions or for all crystal directions [1]. Common methods towards randomizing crystal texture, to different extents, are to rotate the sample about a single axis (horizontal rotation) to the extreme of employing a 'sample randomizer' such as proposed by Gandolfi [2].

This study will show the limitations of these methods with respect to pole figure coverage and introduce practical measurement protocols in mitigation.

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[2] G. Gandolfi, Discussion upon methods to obtain X-ray powder patterns from a single crystal, *Miner. Petrogr. Acta* 13 (1967) 67-74.

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Reassessment of evaluation methods for the analysis of near-surface residual stress fields using energy-dispersive diffraction

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Keywords: energy-dispersive diffraction; multi-reflection analysis; elastic anisotropy; diffraction elastic constants

In this talk two evaluation methods for X-ray stress analysis by means of energy-dispersive diffraction are reassessed, which are based on the $\sin^2\psi$ - measuring technique [1]. Advantage is taken of the fact that the $d_{\psi}^{hkl} - \sin^2\psi$ data obtained for the individual diffraction lines E^{hkl} do not only contain information about the depth and orientation dependence of the residual stresses, but also reflect the single crystal elastic anisotropy of the material. It is demonstrated that even steep residual stress gradients can be determined from $\sin^2\psi$-measurements, that were performed up to maximum tilt angles of about 45°, since the $d_{\psi}^{hkl} - \sin^2\psi$ distributions remain almost linear within this ψ – range. This leads to a significant reduction of the measuring effort and at the same time also makes more complex component geometries accessible for X-ray stress analysis.

Applying the modified multi-wavelength plot method for data analysis it turns out that a plot of the stress data obtained for each reflection hkl by linear regression versus the maximum information depth $\tau_{\psi=0}^{hkl}$ results in a discrete depth distribution which coincides with the actual LAPLACE space stress depth profile $\sigma(\tau)$. The sensitivity of the residual stress depth profiles $\sigma(\tau_{\psi=0}^{hkl})$ to the diffraction elastic constants $\frac{1}{2}S_2^{hkl}$ used in the $\sin^2\psi$ – analysis can be exploited to refine the grain interaction model itself. With respect to the universal plot method the stress factors F_{ij} which reflect the materials anisotropy on both, the microscopic scale (single crystal elastic anisotropy) and the macroscopic scale (anisotropy of the residual stress state) are used as 'driving forces' to refine the strain-free lattice parameter a_0 during the evaluation procedure.

[1] M. Klaus & Ch. Genzel, *J. Appl. Cryst.* 52 (2019), 94 – 105.

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On the oscillating course of 2θ-$\sin^2\psi$ plots for plastically deformed, cold rolled duplex stainless steel sheet

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Keywords: Duplex stainless steel; phase-specific residual stresses; plastic anisotropy;

Duplex stainless steels are widely used in mechanical and chemical engineering due to their excellent combination of properties in terms of strength, ductility and corrosion resistance. Since the material consists of the two phases ferrite and austenite, which differ in their mechanical behaviour, significant phase-specific micro residual stresses can develop after plastic deformation that are superimposed on the macro residual stresses. The sign and magnitude of the phase-specific micro residual stresses are affected by the degree of plastic deformation and the phase-specific elasto-plastic behaviour, which depends i.a. on the specific material composition, crystallographic texture and previous heat treatments. Phase-specific residual stresses are often determined by the $\sin^2\psi$ method using X-ray diffraction. However, in cold deformed polycrystalline materials pronounced non-linearities, i.e. oscillating courses in the 2θ-$\sin^2\psi$ plots can appear which is due to the phase-specific crystallographic texture (elastic anisotropy) and the plastically induced microstresses (plastic anisotropy) [1].

In this work, the phase-specific lattice strain response of cold rolled duplex steel sheet X2CrNiN23-4 under continuously increasing tensile loading was analysed by an in situ experiment using 2D high-energy synchrotron X-ray diffraction. From the continuously detected Debye-Scherrer rings, 2θ-$\sin^2\psi$ plots were analysed for several lattice planes of both phases. For comparison, the similarexperiment was carried out for cold rolled single-phase stainless steel X6Cr17. Depending on the lattice plane family considered, more or less pronounced deviations from a linear dependency were observed for increasing load. From these results, the contribution of elastic and plastic anisotropy to the course of the 2θ-$\sin^2\psi$ curves was evaluated.

[1] A. Baczmański, C. Braham & W. Seiler, *Philos. Mag.*, 83:28 (2003) 3225-3246.

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Diffraction analysis of phase transformation behavior and stress development in short-term heat treatment of Ti-6246

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Keywords: Ti-6246; in-situ dilatometry; martensitic transformations; short-term heat treatment; near-surface induction heat treatment

The α+β Ti-alloy Ti-6Al-2Sn-4Zr-6Mo (Ti-6246) is mainly used in the field of light weight constructions for automobile and aircraft applications due to its advantageous features such as high specific strengths even at elevated temperatures up to approx. 500 °C and its high corrosion resistance. Furthermore, the alloy shows a good heat-treatablility by making use of the metastable orthorhombic α”-martensite which forms after quenching from the high temperature β-regime.

In order to fundamentally characterise the phase transformation behaviour during short-term heat treatments, in situ diffraction analysis were carried out on Ti-6246 during short-term heat treatments using a quenching dilatometer and high-energy synchrotron X-rays at the High Energy Materials Research Beamline P07B at the German Electron Synchrotron (DESY). This profound knowledge is important to control targeted near surface induction heat treatments, which we aim at. The results are also important to assess the microstructure development after repeated short-time heating and cooling cycles, which appear in AM processing. Phase transformations and phase-specific mircostress evolution were studied by means of two target temperatures (945 °C = β-transus temperature; 970 °C) and holding times of 0.5 seconds followed by ultra fast quenching (100 K/s and 1000-1400 K/s). Multiple cycles were conducted in a supplementary manner to assess, whether the cyclic short-term heat treatment contribute to a stabilisation effect in phase composition. In this regard, the in-situ study indicates an increase in martensite content with increasing number of cycles. Further, an initial trend of decreasing α-contents for the first cycles was followed by a stabilisation of the phase content at approx. 10 wt.% after final cooling to ambient temperature.

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Load partitioning and micro residual stress development of two duplex steels with different phase contents

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Keywords: phase specific stress; micro residual stresses; load partitioning; duplex steel; neutron diffraction

Duplex steels are coarse multiphase materials, which combine the properties of austenitic and ferritic steels in a unique way. They are characterised by good mechanical properties, with high strengths and good ductility, and are also extremely corrosion resistant. For the design of technical components, the load partitioning behaviour on the phases austenite and ferrite is of particular interest. Knowledge of the development of phase-specific micro residual stresses is important for the analysis and evaluation of production-related residual stresses. Both characteristics are influenced by the phase contents and further by the phase-specific texture that is often present.

We have specifically addressed this issue by means of neutronographic in situ diffraction studies during uniaxial loading and subsequent unloading tests with two duplex steels (X2CrNiMoN22-5-3 and X3CrNiMoN27-5-2), which clearly differ by their phase contents and the existing degree of the specific phase-specific texture. These diffraction studied were carried out at the SALSA strain scanner at Institute Laue-Langevin, Grenoble. The load partitioning behaviour was investigated for various {hkl} reflections per phase during stepwise loading up to total strain of 9% and after each unloading step the development of the phase-specific micro residual stresses was investigated. The results indicate that under applied load, for both materials the phase-specific stress in ferrite was significantly higher than in austenite, which in turn provides information about the strength and the hardening behaviour of the phases. Plastic anisotropy effects occur when comparing the {hkl}-specific strain data of the single phases. Furthermore, significant phase-specific mico-residual stresses develop, which must be taken into consideration i.a. for diffraction stress analysis, when the strain-free interplanar lattice distance d0 is required for stress evaluation.

Recent developments on the IMAT diffraction project

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Keywords: neutron diffraction; materials science; tomography-driven diffraction

IMAT [1, 2], a recently built cold neutron beamline at the second target station at the ISIS Neutron and Muon Source, was designed for neutron imaging and diffraction applications to probe a broad range of novel materials for the advancement of engineering science. The current user programme exploits the IMAT neutron imaging oportunities using white beam neutron radiography and tomography, or energy-resolved neutron imaging techniques.

Taking advantage of new detector technology [3] and coupled with recent developments of the 90-degree diffracted beam collimation, the new implementation will substantially improve IMAT's scientific capabilities for rapid phase analysis, residual strain analysis, and texture analysis whilst complementing the engineering diffraction research at ISIS currently done only on the engineering diffractometer, Engin-X.

Neutron tomography and diffraction will complement each other through tomography driven diffraction technique (TDD) [4] to study geometrically complex samples making use of the recent developments of SScanSS software [5, 6].

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New approaches for in-situ measurtements on the SALSA strain scanner

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Keywords: neutron strain scanning, in-situ, data acquisition

The strain scanning method using neutron diffraction has become a mature technique and is applied to more and more complex problems in materials science and engineering, including industrial developments. Complex in this context does not only mean more complex shaped samples but as well in-situ studies in order to help understanding the evolution of stress development during fabrication and processing. Thanks to its beam optics and robotic sample stage, the stress diffractometer SALSA at the Institut Laue Langevin (ILL) is already well suited for hosting and manipulating complex samples and sample environment.

In-situ studies require high neutron flux, but as well storage of several measuring parameters, such as temperature, force etc., synchronized with neutron data. To meet these requirements, we have implemented an acquisition card, that provides 4 analogue channels, digital trigger and motor encoder inputs. All signals are recorded synchroniously with neutron data. Every neutron event gets a time stamp and an address, representing its position on the 2-dimensional detector. The time resolution is 10 ns which is practically real-time acquisition. Reconstruction of the detector image and correlation with recorded signals is done during data analysis. The great advantages here is that binning can be done in a most flexible way, depending on the findings during analysis. Correlation with the additionally recorded signals allows continuous measurements, i.e. continuous tensile testing, and even stroboscopic type of measurements.

We have already applied this acquisition mode on in-situ studies, including additive layer manufacturing, continuous tensile testing and fatigue cycling, using a stroboscopic mode.

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In situ neutron diffraction for monitoring of internal stresses, deformation & transformation behavior of engineering materials

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Keywords: neutron diffraction, *in situ*, stress, deformation, transformation

The applications of neutron diffraction (ND) in the field of engineering materials were mainly for *ex situ* measurements of residual stresses and texture, until 2-3 decades before, because of the low neutron flux and undeveloped measurement methods. *In situ* ND measurements during loading began at research reactors with high fluxes, and grew fastly by utilizing pulsed neutrons with the time-of-flight (TOF) method. *In situ* ND measurements have been then expanded to observe various phenomena after high intensity pulsed neutron sources[1,2] started their operations.

TAKUMI[3] is a pulsed neutron diffractometer dedicated for materials engineering installed in J-PARC[2], one of the high intensity pulsed neutron sources. Careful analysis of the Bragg peaks in a neutron diffraction pattern can reveal important structural details of a sample material such as internal stresses, phase conditions, dislocations, texture etc. Experiments in TAKUMI vary from internal strain mapping in engineering components, microstructural evolutions during deformations of structural or functional materials at various temperatures, microstructural evolutions during manufacturing (thermo-mechanical) processes, as well as texture analyses of engineering materials. In order to carry out these experiments, TAKUMI has developed various sample environmental (SE) devices either independently or together with the users. In the presentation, the SE devices and the scientific trends using *in situ* ND in TAKUMI with some research examples will be introduced.

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Studying the TRIP effect under multiaxial loading using neutron and synchrotron X-ray diffraction

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Keywords: Phase transformation; TRIP; martensite; biaxial; diffraction

A key element for the validation of continuum plasticity models is their ability to model the kinetics of the Transformation Induced Plasticity (TRIP) effect under multiaxial loading states, which occur during operation or metal forming processes. Most of the existing studies, implement kinetic laws where the triaxiality factor, defined as the ratio of the hydrostatic stress to the von Misses equivalent stress, is used as a fit parameter affecting the kinetics of the martensitic transformation. However, there is a discrepancy in the litereature whether the transformation is a monotonic function of stress triaxiality.

In this contribution it will be shown how in situ neutron and synchrotron X-ray diffraction experiments, under equibaxial or non-proportional biaxial loading states, have helped unveiling the effect of loading state on the TRIP behavior in different grades of technologically important steels. In specific, the effect of uniaxial/multiaxial loading on the TRIP effect was studied in situ with neutron diffraction, in two metastable austenitic stainless steels with different stacking fault energy (SFE) [1,2]. Moreover, the effect of different stress triaxiality factor on the TRIP effect was investigated experimentally and modelled on a low-alloyed Quenched and Partitioning (Q&P) TRIP-Bainitic Ferrite steel with dispersed metastable austenite particles [3].

These studies indicate that the stress triaxiality, which is associated with the growth mechanism of martensite, cannot solely account for the differences in the transformation kinetics between different loading states, but the loading state plays a key role on the nucleation mechanisms in these materials.

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Through-thickness variations of residual stresses in functionally graded steel-stainless steel structures additively manufactured by direct energy deposition

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Keywords: Residual stress, additive manufacture, functionally graded structure, direct energy deposition; neutron diffraction (up to 5 words)

Through-thickness distributions of residual stresses were measured in a steel-stainless steel structure using neutron diffraction. A total of five different types of specimens were additively manufactured by directed energy deposition (DED) process. The specimens have a functionally graded material (FGM) structure, which has been deposited with variation of chemical composition of ferritic and austenitic steel powders in each interlayer on a steel substrate. Neutron diffraction provided three orthogonal stress components of the specimen and the results were compared to the two-dimensional stress map obtained by the contour method. Significant variations from tension to compression (up to 950 MPa) in the sine-wave stress profile were alleviated to about 430 MPa when the specimen was deposited with orthogonal or island DED scanning strategies with interlayers. Gradual changes (16.3 to 12.1 x 10^{-6} /°C) of the thermal expansion coefficient were measured in the DED FGM parts and grain structure with defects along the interface was examined by neutron tomography.

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Tailoring mechanical properties of metallic materials via additive manufacturing followed by heat treatment

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Keywords: Additive manufacturing; Stainless steel; Microstructure; Anisotropy; Heat treatment

In this work, we deal with tailoring mechanical properties of metallic materials via additively manufacturing (AM) technology that has been frequently used in the areas of structural materials processing due to its advantages on fabricating a complicated shape. A 15-5PH martensitic stainless steel which possesses high strength and outstanding corrosion resistance was studied. The two dog-bone-shaped stainless steels with the different building direction by AM were manufactured to examine the effect of thermal history on microstructure and mechanical/fracture anisotropy by using in-situ neutron neutron diffraction and electron backscattered diffraction (EBSD). Then, we conducted various post heat-treatments (e.g., solid solutionizing, solid solutionizing followed by aging, and direct-aging) to tailor the microstructure and investigated the contributions of respective deformation mechanisms through the quantitative analyses based on small angle neutron scattering (SANS) and transmission electron microscopy (TEM). To retain superior mechanical properties, we contemplated the importance of phase stability of retained austenite as well as a texture, and these were possibly controlled by adjusting the manufacturing processing conditions and various heat-treatment strategies.

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Multiple deformation scheme in direct energy deposited CoCrNi medium entropy alloy at 210K

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Keywords: Additive manufacturing; Medium entropy alloy; Multiple deformation scheme; In-situ neutron diffraction; Peak profile analysis

Remarkable attention has been drawn to a new class of entropy alloys in materials science, which makes it possible to overcome the trade-off effect on the mechanical properties for strength and ductility due to lattice structure distortion, sluggish diffusion kinetics and the cocktail effect. The developments in manufacturing technology and designing new classes of alloys are great importance in materials science. One of the newly emerging process technologies is direct energy deposition of additive manufacturing (AM-DED). It is a powerful fabrication method which has a short lead time and design freedom. In addition, small melting powders undergo rapid heating and cooling rate, which create considerably different microstructures, such as non-equilibrium phase and directional grains/sub-grains, compared to conventional alloys. Such unique microstructures developed by the AM process have been shown to enhance mechanical properties. In this work, we manufactured CoCrNi medium entropy alloy and stainless steel 316L by the direct energy deposition process (DED CoCrNi MEA and DED SS316L). In-situ neutron diffraction coupled with diffraction peak profile analysis was used to compare tensile deformation behaviors and their deformation mechanisms at RT and 210K for cryogenic application. A combination of CoCrNi MEA and an AM-DED process achieves a synergistic effect of strength, ductility and toughness due to the intrinsic alloy design and processing control.

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Microstructure and residual stress interactions in metal additive manufacturing: post-build assessment and new in-situ methods

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Keywords: metal additive manufacturing, residual stress, in-situ neutron scattering

Layer-wise addition of metal to directly form components or add coatings via laser powder bed fusion (LPBF) or laser directed energy deposition (DED) can generate very high levels of residual stress which affect component durability if not adequately addressed. These techniques also result in novel, non-equilibrium microstructures, sometimes with desirable features, that interact with traditional residual stress relief and microstructure manipulation heat treatments.

In LPBF nickel superalloy 718, neutron diffraction was used to demonstrate that a complex residual stress state can persist through a non-recrystallising heat treatment at 960 °C plus subsequent aging. The same treatment has been previously shown to relieve residual stresses and promote grain growth in conventionally manufactured material. This discrepancy is attributed to the presence of nano-scale intercellular precipitates and a large concentration of existing dislocations, both consequences of the LPBF process, which act to impede recrystallisation and creep processes. The residual stress state is shown to influence the long-crack fatigue threshold at low stress ratios. Higher temperature annealing successfully relieved residual stresses but resulted in recrystallisation and grain growth which reduced the yield stress.

To further explore residual stress and phase evolution during additive manufacturing, an in-beamline laser DED capability is being developed at the Australian Nuclear Science and Technology Organisation for both neutron and synchrotron use.

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Residual stresses in additive manufacturing determined by diffraction techniques

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Keywords: diffraction, large scale facilities, additive manufacturing; residual stress

Additive manufacturing (AM) technologies are experiencing a rapid growth, driven by their potential through layer wise deposition for transformational advancements of engineering design, leading to efficiency and performance improvements. Laser Powder Bed Fusion (LPBF) is an AM method which permits the fabrication of complex structures that cannot otherwise be produced via conventional manufacturing methods. Nevertheless, the rapid cooling rates associated with this process results in the formation of significant and complex residual stress (RS) fields. Diffraction-based methods using penetrating neutrons and high energy X-rays at large scale facilities offer the possibility to non-destructively spatially resolve surface and bulk residual stresses in complex components. These techniques also offer the possibility to track the changes of RS following applied thermal or mechanical loads. This presentation will overview some of the success stories of using large scale facilities by the BAM (German senior scientific and technical Federal institute for Materials Research and Testing) for the characterization of residual stresses in additively manufactured metallic alloys. In particular, the study of the influence of process parameters including scanning strategies on the residual stress state and the relaxation of these stresses through heat treatment will be presented. Additionally, some of the challenges for diffraction-based RS analysis in AM materials will be discussed.

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Influence of the scanning strategy on the RS state of a LPBF IN718 material

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Keywords: as-built LPBF IN718 alloy; scan strategy influence; neutron and X-ray diffraction; stress balance condition; distortion upon baseplate removal

For metal-based additive manufacturing (AM) to achieve leaner designs and enable longer life predictions, it is imperative to gain a detailed knowledge of the residual stress (RS) built-up. Laser powder bed fusion (LPBF) is an AM technique particularly prone to RS because of the highly localized heat source, extremely high cooling rates (in the order of 10^3-10^7 K/s), and successive cooling and heating cycles of the solidified material. Furthermore, RS analysis of LPBF materials by diffraction methods is *peculiar* because of the complexity of the thermal history, the possibility of encountering high levels of surface roughness, spatial textural variations and/or changes in solute concentrations at the component scale.

Diverse aspects of the influence of scanning strategies on the as-built residual stress state of a LPBF IN718 alloy will be presented, with particular focus on the challenges that AM microstructures pose for a reliable RS determination [1-4].

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Residual Stress in Plasma Transferred Arc (PTA) Cladding for Hybrid Additive Manufacturing (AM)

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Keywords: Plasma Transferred Arc (PTA); Residual Stress, Titanium, Neutron Diffraction, Synchrtron, Additive Manufacturing (AM)

Plasma Transferred Arc (PTA) cladding has been used as a hybrid Additive Manufacturing (AM) technique, which enables using a powder or wire as the material supply. The material is fed to a molten pool, which moves according to the geometrical pattern of individual layer to manufacture parts on a layer-by-layer basis. The PTA AM technique is categorised as a Directed Energy Deposition (DED) process, according to the ASTM standard. The process enables a high deposition rate for AM, which makes it a unique solution for high value manufacturing sector. It also provides a unique opportunity for multi-material deposition and integrating surface conditioning and/or machining as part of the AM process.

As a typical issue with all metal-based AM technologies, PTA AM components suffer distortion and shape and size inaccuracy, mainly due to repetitive heating and cooling phenomena which affect the microstructural, mechanical and geometrical properties. The evolution of residual stress is one of the main challenges to successful industrial implementation of this technology. We have developed a fundamental understanding of the PTA AM process, by investigating mechanical and material considerations associated with PTA AM. By employing a range of non-destructive and destructive techniques, the residual stress was determined in PTA AM parts, manufactured with different combinations of process parameters, to provide a baseline for mapping residual stress in PTA AM parts. Neutron diffraction and X-ray synchrotron diffraction were implemented at The Institute Laue-Langevin (ILL) and European Synchrotron Radiation Facility (ESRF), Grenoble, France. Scanning through a number of scan-lines produced a map of residual stress in PTA AM parts. The destructive Contour Method was also employed to provide a map of residual stress on the plane-of-interest.

The knowledge obtained through the research work will enable the AM sector to identify operating protocols to minimise residual stress and increase shape and size accuracy for metal-AM technologies.

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Synchrotron XRD Evaluation of Residual Stress Distribution of Laser Shock Peened Additive Manufactured Inconel 718 for High Temperature Applications

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Keywords: Laser Shock Peening; Residual Stress; Additive Manufacturing; Inconel 718; High-temperature

Additive Manufacturing (AM) is rapidly developing applications in safety-critical components for aerospace and power generation gas turbines. A significant problem is the presence of residual stresses generated by the AM process due to the layer-by-layer printing process. Surface treatments such as laser shock peening (LSP) are commonly used to improve its fatigue performance, and the scientific community are looking towards translating these techniques to increase AM parts useability. LSP stands out as it has been shown to induce deeper and higher residual stresses. It is a much-preferred technique as it produced low cold work on the surface as it helps to stabilise a component's high temperature fatigue performance. The objective of this experiment was to determine the individual effect of the thermal and mechanical load has on LSPed AM IN718 specimens that has undergone elevated temperature high-cycle fatigue test. Preliminary findings showed that the beneficial compressive strains produced by the LSP process is retained after 15 hours of thermal exposure at 550°C. In a separate experiment, AM LSP IN718 samples could retain their initial stress state after going through a high-cycle fatigue environment at room temperature of up to 1,000,000 cycles at 30% yield strength.

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P61A, a new white beam beamline optimized for Residual Stress Analysis

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Keywords: Residual Stress Analysis; Synchrotron EDXRD; $\sin^2\psi$

Energy-dispersive diffraction (EDD) is uniquely advantageous for residual stress measurements. EDD is well known for its use in near surface stress gradient measurements, but can also be applied on transmission conditions, even enabling gauge volume control by using incident and scattered beam slits. However, such measurement geometries require a high flux and high energy source, which can only be obtained through Synchrotron radiation.

In this work we present the new white beam beamline P61A, a new instrument from Helmholtz-Zentrum Hereon operating at the German synchrotron Desy. P61A was designed to be optimized for residual stress measurements with a uniquely bright white beam. The beamline has ten damping wigglers as its source, delivering up to 10^{12} photons/s.mm^2 at 40 keV and 10^{11} at 200 keV. Diffraction data is collected using two HPGe energy resolved detectors positioned at independently positioned diffraction planes, leading to advantageous measurement conditions [1]. P61A is open for external users and is capable of EDD near surface stress measurements at controlled depths, and transmission measurements through up to approximately 10 mm thick samples. The beamline is equipped to perform and analyze data with several different $\sin^2\psi$ based approaches such as multiwavelength method and universal plot [2]. Heavy load positioning devices are in place and are compatible with an extensive suite of sample environments.

In this talk, we will present the technical details of the instrument, typical experiment configurations, and setups and software available for users.

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Low-temperature deformation in high-entropy alloys

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Keywords: High-entropy alloys, dislocation, stacking fault, twinning, serration, low-temperature deformation.

Multi-component high-entropy alloys (HEAs) are a new class of structural materials with potentially excellent strength-ductility combination. Despite their complex chemistry, HEAs can form a single-phase solid-solution with an incredibly simple face-centered-cubic (FCC) structure. Due to the favorable crystal structure and the metastable FCC phase, several additional deformation mechanisms become available. In situ loading study with neutron diffraction has proven to be a powerful approach to characterize the actiation of different deformation modes and their interactions at low temperatures. Measurements at 15 K in CrCoNi-based HEAs have revealed a myriad of deformation mechanisms, including stacking fault, twinning, phase transformation, and serration, in addition to dislocation slip. The in situ neutron diffraction data are used to estimate the contributions of different deformation modes to strain hardening. It is shown that the cooperation of these different deformation mechanisms led to the extreme large ductility at low temperatures.

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Microstructural Characterization Through Grain Orientation Mapping with Laue Three-Dimensional Neutron Diffraction Tomography

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Keywords: Laue three-dimensional neutron diffraction tomography; neutron diffractive imaging; 3D grain indexing; 3D grain morphology reconstruction

For polycrystalline materials, key material properties including strength, deformation behavior, magnetic susceptibility, weldability and stress corrosion cracking resistance depend significantly on the texture of the crystalline microstructure. Conventional assessment of texture is either limited to thin surface regions or it is destructive while only probing small fractions of a bulk specimen. Only high energy X-ray diffraction at synchrotron sources and neutrons enable quantitative studies of bulk texture. Here, we report how transformative progress in advanced Laue three-dimensional neutron diffraction tomography enables to map several hundred grains and, thus, allows grain orientation assessment in the volume of centimeter-sized samples with statistical significance. The short exposure times and non-destructive nature of Laue 3DNDT will support in-situ studies, while future improvements in spatial resolution shall include more accurate grain morphology in corresponding microstructure studies.

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Grain-resolved strain analysis with Laue three-dimensional neutron diffraction tomography

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Keywords: Neutron diffraction tomography; strain analysis; ferromagnetic shape-memory alloy; Co-Ni-Ga

An approach for resolving grain-by-grain strain tensors from polychromatic neutron diffraction tomography data sets is presented. The analysis combines diffraction tomography methods with a forward modelling approach, as formulated in the recently developed Laue three-dimensional neutron diffraction tomography indexing method (Laue 3DNDT) [1], to obtain strain information from energy-integrated neutron data. We validate the method both through simulations and experimental measurements of a CoNiGa ferromagnetic shape-memory alloy, which has been characterized through several in-situ stress states.

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Validation of plane stress assumption on SS316L specimen with one layer.

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Keywords: Wire + Arc Additive Manufacturing, Gas Metal Arc Welding, Neutron Diffraction, Residual stresses.

Additive manufacturing (AM) techniques are based on layer-by-layer accumulation of molten material [1]. Compared with traditional manufacturing, Additive Manufacturing (AM) has the ability to produce complex components with less waste of materials and energy [1]. The processes used in this work is the Wire arc additive manufacturing (WAAM) technique. This technique has the potential to build up parts to tens of meters with relatively low forming costs and high manufacturing efficiencies [2]. In the WAAM process, the metallic wire is heated by the electric arc, then melted and transferred to the molten pool. In this work, the Gas Metal Arc welding – Cold Metal Transfer process (GMAW-CMT) was employed. The Cold Metal Transfer process uses low welding energy with a high control of deposition rate. Understanding the generation of residual stresses during the WAAM operation is of high interest especially in the aim to improve the mechanical properties of the part such as: fatigue life, …

In this study, the investigated specimens consisted of one layer only deposited on the edge and along the length of parent material hold vertically, see figure 1. Both the parent material and the filler wire are in stainless steel 316L. The experimental setup has been chosen in order to build AM parts under plane stress assumption so the analysis is reduced to two dimensions. The Neutron Strain Scanning technique has been used to characterise the strain and stress fields in the specimens (parent material and deposit). The strain-stress measurements were performed at the Institute Laue Langevin, SALSA diffractometer, Grenoble (France). The stress field is deduced from the strain measurements according to two approaches for determining the reference lattice parameter d_0 (or diffracted angle θ_0) [3]. The first approach is based on a reference specimen (comb-like) in order to cause "strain free" conditions in the area of neutron scanning whereas the second approach is based on plane stress assumption ($\sigma_{normal} = \sigma_{YY} = 0$). The reference $2\theta_0$ angle was only available along line V55. The validation of the two approaches was of high interest in order to compute the residual stresses along two other lines : V25 and V85 as shown in figure 1.

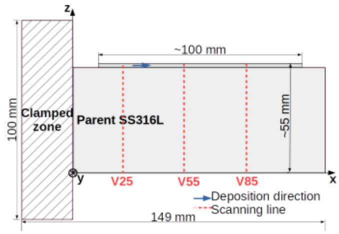

Figure 1 : location of the lines (red) where were performed the neutron strain measurements.

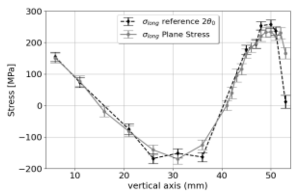

Figure 2 : comparison between the longitudinal stress calculated under plane stress and with reference $2\theta_0$ at the section V55.

The calculated residual longitudinal stress along line V55 are presented in figure 2. The two distribution are quite similar according the two considered approaches. In figure 2, one point was measured in the deposit at z ~ 54 mm. The first method gave a value close to zero while the second was around 180 MPa. The greatest discrepancy is observed in the deposit. The deposit (so melted zone) and the top zone of the parent are tensile while the core of the parent is compressive. The bottom par is also tensile due to the force balance in the parent. The parent was deformed under the thermal cycle with a bending towards the z direction. The results displayed in figure 2 validated the two approaches but some care has to be made in the melted zone (due to large grain size that are not shown here).

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Microstructure and Stress development during Laser Metal Deposition analyzed by Synchrotron X-ray diffraction

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Keywords: Laser Metal Deposition; Phase transformation; Residual stresses.

Laser Metal Deposition (LMD), also named Direct Energy Deposition (DED), is an additive manufacturing (AM) process in which a metallic powder is feed through nozzles by an inert gas flow and consolidated by a laser beam. Due to the layer-by-layer process, the microstructure undergoes several heating/cooling cycles throughout the process. These thermal gradients produce microstructure alteration and can generate high residual stresses leading to cracks and high distortions, hence, poor performance and low fatigue life. For hardenable steels, the residual stresses field is also affected by the strain due to martensitic transformation, adding another level of complexity into the manufacturing. Regions close to the deposition zone transform into virgin, brittle martensite and retained austenite (RA) during cooling, while at more distant regions, the previous virgin martensite is submitted to several tempering cycles and decomposition of the RA. These thermal characteristics are also known as Intrinsic Heat Treatment (IHT), and the understanding of these transformations is critical to achieving LMD components with superior properties.

To optimize the AM process, both in-process phase transformation and processing parameter effects on the residual stresses must be addressed. In this work, in-situ High-Energy X-ray Diffraction (HEXRD) was used during LMD manufacturing of thin X40CrMoV5-1 walls. Additionally, *ex situ* high energy and Low-Energy XRD scans were performed to evaluate in detail the microstructure variations over the sample height and the residual stress distribution. The obtained results indicate that defects are introduced in the austenite as the martensitic transformations occur. The *ex situ* results show that the IHT significantly modifies the residual stress state over the sample, generating two regions with different residual stresses states and microstructure over the height of the built samples. Additionally, process parameters, particularly laser power, was identified to have a pronounced effect on the resulting properties.

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Material Science with a new High Energy White Beam Station – Prospects and Challenges

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Keywords: energy-dispersive X-ray diffraction; residual stress analysis; additive manufacturing; preferred orientation analysis; shape memory alloys

X-ray diffraction techniques allow for non-destructive analysis in materials science, e.g., phase analysis, preferred orientation analysis and residual stress analysis in the near-surface region. In this regard, energy-dispersive X-ray diffraction enables the combined analysis of these characteristics [1]. Furthermore, various penetration depths as a direct consequence of the different beam energies allow for depth-resolved analysis of residual stresses [2].

High intensities at a wide range of energies up to 200 keV can be applied at the new white beam station P61A of Helmholtz-Zentrum Hereon at PETRA III, DESY. Here, transmission measurements and reflection measurements can be realized using the same setup. This allows for near surface and volume measurements with high spatial resolution eventually enabling analysis of complex microstructures. Such options are paramount for assessment of a specific mechanical behavior, e.g., in case of additively manufactured (AM) material [3] and shape memory alloys (SMA), respectively. For AM parts, the resulting microstructures and residual stress distributions imposed by the unique solidification conditions and thermal history stemming from the processing parameters are required to be assessed in-depth to establish paramount process-property relationships. Furthermore, recently developed iron-based SMAs Fe-Ni-Co-Al-X (X=Ta, Nb, Ti) [4] and Fe-Mn-Al-Ni [5] are promising candidates with good superelastic (SE) properties, but the presence of texture and a coarse grained morphology strongly affect the SE behavior. Here, the results of experiments at P61A will be presented tackling open issues highlighting prospects of this enhanced white beam station.

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High accuracy neutron diffraction measurement and positioning with an industrial robot system at the STRESS-SPEC instrument

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Keywords: neutron diffraction; residual stresses; high accuracy; additive manufacturing; sample position tracking

The Heinz Maier-Leibnitz Zentrum (MLZ) operates at Germany's sole neutron source FRM II the diffractometer STRESS-SPEC optimised for fast strain mapping and texture analyses. The STRESS-SPEC group was the first to pioneer the usage of industrial robots for sample handling at neutron diffractometers [1, 2]. However, the current robot is limited in its use due to insufficient absolute positioning accuracy of up to ± 0.5 mm in some cases. Usually an absolute positioning accuracy of 10% of the smallest gauge volume size – which in case of modern neutron diffractometers is in the order of 1×1×1 mm^3 – is necessary to allow accurate strain tensor determination and correct centering of local texture measurements. The original robot setup at the neutron diffractometer STRESS-SPEC has therefore been upgraded to a high accuracy positioning/metrology system. We will present the complete measurement process chain for the new robot environment. To achieve a spatial accuracy of 50 µm or better during measurement of the full strain tensor the sample position is tracked by an optical metrology system and actively corrected. Residual stress measurement within additive manufactured (AM) components with complex surfaces and a shell and support structure build-up principle represent an exemplary application [3]. Two new designed radial collimators create more space in the sample environment and enhance the residual stress analysis capabilities for large complex parts. In addition, a newly designed laser furnace can be mounted at the robot flange to conduct, for example, texture measurements at elevated temperatures of up to 1300 °C. A brief overview of the STRESS-SPEC instrument and its capabilities using the new setup will be given.

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A concept for residual stress gradient analysis in cubic materials with mosaic structure

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Keywords: mosaic crystal; crystallite group method; energy-dispersive diffraction; depth-resolved analysis; additive manufacturing

X-ray stress analysis (XSA) of materials with nearly single crystalline structure requires different methods than those developed for polycrystalline materials with random crystallographic texture, since the number of accessible measurement directions is restricted to a few distinct poles. An approach suitable for XSA of materials featuring pronounced texture is given by the crystallite group method, which treats crystallites of (nearly) the same orientation as single crystal [1,2].

In this talk we present a concept that enhances the crystallite group method by the feature of depth resolution [3]. The measurement strategy uses the scattering vector method which provides lattice strain depth profiles $\varepsilon^{hkl}(\tau)$ along the $\langle hkl \rangle$ poles by stepwise sample rotation around the diffraction vector [4]. The evaluation strategy is based on a segmentation of the $\varepsilon^{hkl}(\tau)$ – profiles parallel to the sample surface which yields the anisotropic strain state as function of depth. The in-plane stress components $\sigma_{ii}(\tau)$ $(i = 1,2)$ are obtained by solving the corresponding equation systems for any depth t. For samples with (001) orientation an alternative evaluation concept can be applied, which adapts the $\sin^2\psi$ – method to the case of single crystalline materials.

The concept is introduced by means of simulated examples inspired by real experimental conditions. The influence of the strain-free lattice parameter a_0 on XSA using both evaluation concepts will be discussed. Furthermore, experimental examples based on measurements performed on materials with mosaic structure produced by additive manufacturing will be presented.

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Quantitative analysis and benchmarking of positional accuracies of neutron strain scanners

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Keywords: neutron strain scanner, calibration protocol, positioning uncertainty

Positional accuracy is an important parameter in residual stress investigations with neutron diffraction, considering that precise measurements of strains at the same localised position along a number of sample orientations are required, including investigations of complete complex shaped engineering components. This study reports the development of a standardised approach for quantitative analysis of positional accuracy on neutron strain scanners that builds on previous campaigns and standards [1, 2]. The approach uses standardised sample sets with specific geometries that enable quantitative assessment of instrumental and sample alignment procedures and associated accuracies. This method has been implemented on four participating instruments: ENGIN-X (United Kingdom), MPISI (South Africa), SALSA (France) and STRESS-SPEC (Germany), to render results representative of monochromatic and time-of-flight strain scanners. The benchmarking results show comparable performance between the instruments with positional accuracies around 100 µm readily achieved. This standardised approach confirms the high positional precision attainable for non-destructive stress determination, to unequivocally benefit utilisation by academia and industry alike. It is envisaged that this common calibration protocol and reporting template that conforms to the newly developed Neutron Quality Label for Internal Stress Characterisation be adopted by other facilities to facilitate expansion of the supportive network.

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The potential of high-flux liquid anode X-ray sources for micro-structure and stress analysis

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Keywords: liquid metal jet; X-ray diffraction; imaging; microstructure; stress analysis

The main disadvantage of X-ray diffraction analysis with conventional laboratory sources lies in their low photon flux, which leads to long measurement times, especially in the investigation of polycrystalline materials. Fast in-situ analyses, the examination of large sample series or of components with complex geometry are therefore usually reserved for measuring stations at 3rd generation synchrotron sources, but their availability is limited.

Powerful laboratory high-flux sources such as rotating anode tubes or liquid metal anodes are an alternative in this respect. With the latter sources, primary spectra of particularly high flux densities and several characteristic Kα wavelengths can be realized when the liquid metal jet is an alloy of different elements such as gallium (9 keV) and indium (24 keV).

In this contribution the two recently commissioned MetalJet measuring stations are presented. The modular design of both stations, operated at 70 keV and 160 keV, are equipped with various detectors to enable in-situ and ex-situ measurements with different sample environments in angle- and energy-dispersive diffraction mode, as well as simultaneous imaging experiments. The potential of the measuring stations is demonstrated by first experimental examples. In addition, novel possibilities are discussed which arise, for example, for depth-resolved structural and stress analyses when the bichromatic radiation emitted by the sources is used for angle-dispersive diffraction experiments. Both measuring stations are available for external user groups as part of our Corelab concept.

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Ferritic benchmark specimens for cross-comparison of diffraction and destructive residual stress measurement techniques

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Keywords: diffraction; residual stress; standard; round robin; strain

Round-robin residual stress measurements with different underlying techniques have been previously carried out on a variety of material systems and with different sources of underlying residual stress, along with different underlying techniques. This includes VAMAS [1] which pursued neutron diffraction, and more recently NeT [2]. employed a similar approach to capture residual stresses in a series of welded coupons. To date, there is an absence of a specimen type with a well-characterised underlying stress state which has a configuration that is permissive of diffraction techinuqes (neutron, synchrotron X-ray and laboratory X-ray) as well as mechanical strain relief laboratory techniques. We present a series of replicable ferritic samples which are intended to provide a conclusive set of measurements across a host of techniques and practitioners. These samples will be employed as part of a round-robin measurement campaign as part of the EASI-STRESS programme, benchmarking residual stress measurements in a widely used ferritic alloy, and elucidate the potential variation due to the nature and source of these stresses. Further, the round-robin will inform techniques for the comparison/validation of computational predictive models of how residual stresses develop. The design and manufacturing protocols of these specimens will be presented, including how they pair with each residual stress measurement type, followed by preliminary neutron diffraction results and further intended measurements.

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Strain Scanning on E3 at BERII at the HZB, a retrospective.

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Keywords: Strain Scanning; Neutron scattering; Residual Stress; Neuton diffraction.

In December 2019, BERII, Berlin's second research reactor, shut down for the final time, marking the end of almost 62 years of neutron research in Berlin.

E3, the strain scanner at BERII had seen a lot of development and innovation in the last 20 years. In this presentation we would like to mention the highlights and the milestones that shaped E3's success. A major advancement was a vertically focusing and horizontally bent perfect Si single crystal monochromator in 2007 [1], developed with the Institute of Nuclear Physics in Řež near Prague. This enhanced significantly the experimental turnover as well as the quality of the measurement results for the final 12 years of the instrument's use. Since then the continuation of upgrade activities, including a radial oscillating collimator, a new slit system and further advantageous tools to improve the performance on E3, were implemented [2].

E3's novel robust design has been used at FRMII (TUM, Munich Germany), SAFARI-1 (NECSA, Pretoria, South Africa) and CARR (CIAE, Beijing, China). In this case we can be rest assured that E3's legacy will continue well into the future.

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Residual Stress in Sintered WC-VC-Co Disks

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Keywords: residual stress; WC-VC-Co sinters

WC-VC-Co cermets produced using two different sintering routes, i.e. conventional sintering (CS; hot isostatic pressing (HIP)) and spark plasma sintering (SPS) have been investigated for the compositions, WC-12wt%Co and WC-10wt%VC-12wt% Co where the latter reduces weight whilst retaining hardness and toughness. To achieve high densification with CS methods a high sintering temperature and long holding times are required. This leads to significant increases in the WC grain size. The recently developed SPS technique enables equivalent densification at lower temperatures with shorter holding times leading to decreased grain growth.

The influence of the sintering method (CS/HIP vs SPS), the effect of cermet compositions (WC vs WC+VC) and effect of the erosion testing (at several impact angles) on the residual stress state were investigated. This was achieved by determining the depth resolved in-plane residual stresses in solid disk samples of 6 mm thickness using the neutron strain scanners MPISI@NECSA (with resolution 0.4 mm) and KOWARI@ANSTO (resolution 0.2 mm).

Although no stress depth dependences (within experimental uncertainty of ~50 MPa) were found in the investigated samples, the results of the measurements allowed determination of the bulk micro-stress and its connection to the production route and composition.

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Effect of tensile strain on martensite formation and its influence on residual stress distribution in type 304 austenitic stainless steel

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Keywords: SS 304, Tensile strain, Residual stress, Strain-induced martensite

Enhancing the service life of fabricated components by reducing the tensile residual stresses is most desirable [1-3]. One of the possible methods is mechanical stress relieving by plastic straining [4]. Researchers have tried to establish a relation between tensile strain and residual stress in high-strength dual-phase steel [5] and GH4169 alloy [6]. As expected, results indicate that plastic straining reduces the resultant residual stress. However, resulting residual stress is the synergistic contribution from plastic straining and phase transformation during deformation in transformation-induced plasticity steels. Present study investigates the influence of martensite on residual stress development during tensile deformation of type 304 austenitic stainless steel. The uni-axial tensile tests were carried out at room temperature to different tensile strains varying from material yield strength (YS) to ultimate tensile strength (UTS), i.e., 7.3 (≈ at YS), 15, 30, 45, and 60% (≈ at UTS) at a strain rate of 1.3×10-2 s-1. The residual stress (RS) and strain-induced martensite (SIM) were measured on the flat tensile samples before and after deformation at various locations along the sample length using the x-ray diffraction method. The residual stress in austenite phase is measured in three sample orientations (0°, along tensile direction, 45° and 90°, perpendicular to tensile direction) using Manganese Kα radiation (λ= 2.10314 Å) by Proto-iXRD diffractometer. The observed variation in the distribution of RS is explained based on the SIM fraction, and full width at half maxima (FWHM) of XRD peak.

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Residual stresses in additively manufactured aluminum alloys and 316l-steel

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Keywords: additive manufacturing; SLM; residual stress; high energy materials science

Additive manufacturing opens up new ways to produce parts with high geometric complexity, e.g., involving internal structures, which led to an increased interest of science and industry in the recent years. The mechanical behavior and load-bearing capacity of additively manufactured components, however, is still not really understood and subject of intensive research efforts. In particular, residual stresses (RS) play an important role, e.g., for the strength and fatigue properties. Therefore, RS distributions were investigated in various parts, fabricated from aluminium alloy powder (AlSi10Mg) and 316L-steel using the Selective Laser Melting (SLM) technique. The samples consist of simple walls with a wall thickness of about 3 mm and are made in different geometries involving, e.g., different edge curvatures. Residual stress fields were determined using high-energy X-ray diffraction. The diffraction study was carried out in transmission geometry using a photon energy of 87.1 keV that allows to penetrate thicker samples. The influence of specimen geometry and production parameters on the RS state will be discussed.

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In-Situ Heat Treatment of Additively Manufactured Ti-6Al-4V

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Additive Manufacture is currently being used for the production of metallic components because it can offer reductions in cost and waste as well as the ability to create optimized topologies/geometries that are not achievable through conventional manufacturing. To date, few AM components have been qualified for high-consequence structural applications because of insufficient knowledge of the process/structure/property relationship. AM Ti64 has received attention because of the high material cost and the innate ability of AM to minimize material waste. Originally, the Ti64 alloy was developed for casting applications which have very slow cooling rates. Its property combinations are still desired for AM built parts, but the much higher cooling rates, as high as 106K/s, associated with AM result in a distinct and metastable microstructure and, subsequently, distinct material properties. Post-build heat treatments are usually used on Ti64 components produced by AM to drive the as-built microstructure closer a wrought equivalent in microstructure and properties. In this work, we have collected diffraction data during in-situ heat-treating of AM Ti64, made with various processes, in order to monitor the microstructural evolution of the material. The evolution of the phase fraction, texture, internal stress and dislocation density during heat treatment will be presented.

Protecting Suspension Bridges against Fire with Neutron Diffraction

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Keywords: suspension bridge, fire, contact, friction, mechanics

Suspension Bridges carry both economic and strategic significance for the people whom they serve. In part due to their critical nature, they are subjected to various hazards, both natural (corrosion, adverse weather, and snow) and anthropomorphic (fire, blast, and impact) that must be resisted over 100+ years of expected service life. The main cables of a suspension bridge are failure-critical structural members; the failure of one cable invariably leads to the collapse of the bridge deck. The goal of this investigation is quantifying of the resilience of the main cables to high temperature loading while under tensile service load. We present results from experiments performed at multiple scales, ranging from the microscale of the steel making up the main cables to multi-ton full-scale laboratory cable models. In-situ nDif experiments are performed at the Los Alamos and Oak Ridge National Laboratories under various environmental conditions to answer various questions: First, the friction and contact forces between wires in a bundle (strand) are quantified in-situ to understand the internal packing mechanisms in the parallel wire bundles [1]. Second, we simulate multiple fire scenarios of single wires, constituent strands, and a full-scale 9200-wire bridge cable to observe the mechanical behavior including creep, plastic flow, and annealing/recrystallization of the steel both during and after a fire event [2]. Surprisingly, no data exists for the ASTM A586 steel that has been used to construct suspension bridge cables for the last century. Third, we quantify the thermal conductance of the bridge cable, which turns out to be highly orthotropic due to its packing order and contact mechanics. This broad research effort supports the unified goal of truly understanding the mechanics of suspension bridge main cables. These results in turn inform and optimize numerical models that quantify stochastically the internal mechanics of parallel wire bridge cables and predict their collapse risk under a broad array of hazards.

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Microstrain distribution in crystals

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Keywords: X-ray diffraction; neutron diffraction; electron diffraction; line profile analysis; dislocation density

An overview of the diffraction methods in use today for the evaluation of the dislocation density and crystallite size is presented. Following a chronological approach the main concepts and models of line profile analysis [1-4] are introduced and illustrated with real or numerical examples. Special emphasis is made on popular breadth methods and it is shown that the width at half maximum or the integral width of diffraction peaks depend on dislocation arrangement. Therefore, in spite of the possibility of automatic fitting of thousands of peaks, the evaluation of dislocation density and the elastic stored energy based on breadth methods should be avoided. It is shown that the stored elastic energy of two dislocation ensembles with the same density mainly depends on the correlations among Burgers vectors.

Novel techniques developed at synchrotron source and their implications for local microstrain analysis are equally examined. For example the 3DRXD method using hard X-rays opened new perspectives for a more complete and accurate characterization of deformation heterogeneity in polycrystalline aggregates. Scanning methods using sub-micron beams for directly assessing intragranular strains are equally discussed and their connection with conventional peak profile analysis as well as special laboratory techniques (such as high-resolution EBSD) is emphasized. An attempt will be made to highlight the significance of a few selected examples for materials science (unfortunately, not an exhaustive list).

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When industry meets large facilities

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Keywords: Diffraction; Synchrotron; Neutrons; Industry

GEMS is the central user platform of Helmholtz-Zentrum Hereon, which operates beamlines at PETRA III/DESY in Hamburg, Germany, and neutron instruments at the FRM II reactor in Garching, Germany. In addition to proposing its beamlines, instruments and expertise to external users from both academia and industry, GEMS is supporting the research of the HZG departments. GEMS provides instruments for imaging, scattering and diffraction, designed mainly for engineering science purposes. At PETRA III, the High Energy Materials Science beamline HEMS/P07 enables diffraction experiments using a monochromatic beam with energies ranging from 40 to 150 keV. Moreover, Energy-dispersive X-ray diffraction experiments with photon energies from 30 to 200 keV are now available at the beamline WINE/P61A which started its user operation in December 2020. In addition to the X-ray instruments, diffraction experiments are also possible at the neutron diffractometer STRESS-SPEC, installed at FRM II. Most of our users are coming from the academic world. However, thanks to several national, regional and European projects, the number of industrial users interested in our techniques has increased during the last few years. In this talk, a short description of selected projects will be presented as well as some successful examples of scientific collaboration between industry and large facilities.

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In-depth evolution of residual stresses and effect of free surface on stress relaxation determined using X-ray diffraction Laplace methods

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Keywords: near surface residual stress, angle- and energy-dispersive diffraction, elastic anisotropy, intergranular interaction, austenitic stainless steel

This work shows that the choice of the grain interaction model is one of the critical components of a correct residual stress analysis. In addition, a new model of grain interaction, called "tunable free surface" has been proposed, allowing the most accurate determination of the depth-dependent profile of the residual stresses measured experimentally. Moreover, the proposed new model has a strong physical justification. For the first time, the in-depth evolution of grain elastic interactions was determined using three complementary model verifications.

Residual stress analysis was performed on mechanically polished elastically anisotropic austenitic stainless steel and was performed by various evaluation methods of X-ray diffraction. To determine the evolution of stresses in a shallow region below the sample surface (up to 5 µm), the multiple-reflection grazing incidence X-ray diffraction (MGIXD) with classic monochromatic X-rays was used. For comparison, deeper volumes (up to a depth of 45 µm) were investigated by energy dispersion X-ray diffraction (EDDI) with high-energy synchrotron radiation. These complementary methods allowed the determination of the depth dependent stress profile and the evolution of the strain-free lattice parameter. Excellent follow-up and overlapping results have been achieved using different data analysis methodologies. Moreover, it has been found that very close to the polished surface, a significant compressive stress of about -500 MPa is generated, which gradually transforms into a tensile stress and then drops to zero at a depth of about 60 µm. On the other hand, the strain-free lattice parameter does not change significantly at this depth.

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Delivery of neutron optics system for the BEER diffractometer in ESS

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Keywords: Neutron optics system; BEER; ESS

In the talk the engineering, manufacturing and delivery process of the neutron optics system for the time-of-flight materials engineering diffractometer (BEER) at European Spallation Source (ESS) will be presented.

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EIGER2 CdTe detectors for hard X-ray research

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Keywords: Hybrid photon-counting X-ray detector; cadmium telluride; powder diffraction; time-resolved

In the last decade, Hybrid Photon Counting (HPC) X-ray detectors [1] like the PILATUS have transformed research at synchrotrons. They provide noise-free detection and enable new data acquisition modes. The most current HPC detector family EIGER2 enables even more ambitious X-ray research, for example in material science [2]. These detectors combine all advantages of previous generations while offering new acquisition features and improved performance: maximum count rates of 10^7 photons/sec per pixel, small pixels of 75 µm × 75 µm, two energy-discriminating thresholds, and frame rates up to 2 kHz with zero dead time (<100 ns) between exposures.

EIGER2 detectors were designed and optimized for the demands of synchrotron applications, and they are available for the laboratory as well. Equipped with CdTe sensors they provide high quantum efficiency at energies up to 100 keV, making them ideal for hard x-ray diffraction applications. Two energy thresholds allow for reduction of high-energy background such as from cosmic radiation, higher harmonics, or unwanted sample fluorescence. These benefits advance established X-ray diffraction methods like crystallography including powder diffraction as well as scattering techniques. Fast and gated measurements become possible and empower new fields of research, by enabling e.g., time-resolved or pump-probe techniques such as in laser-heating or scanning X-ray diffraction tomography.

We will demonstrate the advantages of the HPC CdTe technology for hard X-ray research with examples from synchrotron beamlines at ESRF, BSRF, and APS using loan detectors and recently installed EIGER2 CdTe systems.

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Oriented internal stress in plastically deformed NiTi shape memory alloys

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Keywords: martensitic transformation; shape memory alloy, NiTi; plastic deformation, internal stress

When deformed plastically, NiTi shape memory alloy undergo martensitic transformation followed by deformation twinning in martensite, which gives rise to deformation microbands with large localized deformation. Since this localized deformation is unrecoverable upon unloading and heating but the neighboring crystal lattice exhibits recoverable strains, oriented internal stresses are introduced into the austenitic microstructure by controlled plastic deformation. The origin of such oriented internal stress will be introduced and discussed based on the TEM observation of austenitic [1] and martensitic [2] microstructures in deformed NiTi wires. Practical implications of the oriented internal stress, particularly to the two way shape memory effect will be discussed.

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A framework for equilibrium constrained strain estimation and tomography

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Keywords: strain tomography; strain estimation; scanning-3DXRD; neutron transmission; neutron diffraction

We present a framework for strain estimation and tomography that constrains the resulting strain field to satisfy self-equilibrium. This framework models the strain field as a linear mapping of an underlying tensor potential field where these are both chosen such that the equilibrium constraints are always satisfied. The strain field can be estimated using measurements that are a linear operator transformation of the field; this includes line and volume integrals. Hence, it is applicable to a variety of neutron and x-ray measurements. Within this framework, several methods for strain estimation and tomography have recently been developed and successfully applied to different sources of experimental data. This includes triaxial strain tomography from neutron transmission data [1]; intragranular strain tomography from scanning-3DXRD [2], and strain estimation from neutron diffraction data [3]; (a)-(c) in Figure 1.

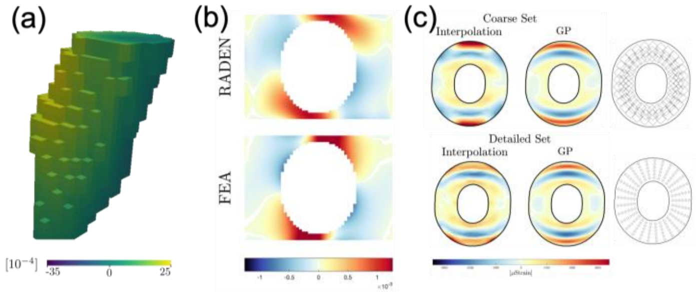

Figure 4: (a) Reconstructed e_{xx} intragranular strain for a columnar tin grain from scanning-3DXRD [2]. (b) Triaxial strain reconstruction from neutron transmission measurements [1]. (c) Strain estimation of a plane stress crushed ring from neutron diffraction measurements [3].

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Modification of mechanical properties and microstructure of titanium grade 2 processed by hydrostatic extrusion

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Keywords: titanium, hydrostatic extrusion, texture, residual stress, microstructure

The properties of metals after severe plastic deformation (SPD) are strongly modified in general. Their mechanical strength can be increased by a factor of two, the hardness and fatigue resistance are considerably increased and also other physical properties can be considerably improved [1[. In the present work the properties of hydrostatically extruded pure titanium (grade 2) were studied and analyzed in aspect of its application for dental implants instead of titanium alloys (e.g., grade 5). The advantage of use of pure titanium (grade 2) is that it does not contain doping elements, which can be harmful for organism.

The X-ray, EBSD and TEM techniques were used to study the properties of hydrostatically extruded titanium grade 2. Basing on these measurements the microstructure parameters, crystallographic texture and residual stress were determined. Also, the coherent domain size and the average elastic deformation were determined using the Williamson-Hall method. In the next step the strain-stress tensile tests and micro-hardness measurements were done and a set of mechanical characteristics was determined.

It was found that the average grain size at the transverse sample section was in the range of 300-400 nm. Moreover, a high density of structure defects was cumulated (increased value of kernel average misorientation – KAM), also the intragranular fragmentation was important, which was expressed by increased values of grain orientation spread (GOS). The circumferential and radial components of residual stress had the compressive character.

The mechanical properties of nano-crystalline titanium were strongly improved (higher yield and ultimate yield stress, higher micro-hardness, increased fatigue resistance).

Our study showed that pure titanium (grade 2) processed by hydrostatic extrusion has as good mechanical parameters as titanium alloy (grade 5), conventionally used for medical implants.

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In-situ neutron strain imaging during Direct Metal Deposition of Ni-based Inconel 718 Alloy

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Keywords: *additive manufacturing, DMD, Inconel 718, strain monitoring, neutron diffraction*

The quality and performance of the parts produced by additive manufacturing (AM) are highly sensitive to initial conditions and processing parameters[1]. The rapid solidification, complex thermal gradients, anisotropic microstructure, and challenging geometries substantially affect the strain formation, which in turn, play a significant role in residual stress (RS) distribution in the final part. This becomes more crucial especially for superalloys such as Inconel 718 (IN718) that are commonly used in critical engineering applications with high demanding in-operando conditions such as high temperature and dynamic loadings[2]. Although traditional approaches are mainly employed to determine RS in AM parts, there is still controversy on the formation, distribution, and ultimately the evolution of RS. In order to disclose strain contributions during AM and to adjust the controlling process parameters for optimization, a novel in-situ approach is hereby used.

In this study, we propose a state-of-the-art in-situ strain imaging during Direct Metal Deposition (DMD) via neutron diffraction. This "time and spatially resolved" in-situ experiment was performed on a monochromated instrument, SALSA (Strain Analyzer for Engineering Applications), at Institute Laue-Langevin, Grenoble, FR. IN718 wire feedstock was used to produce cylinder walls by the laser equipment with a coax laser cladding head (COAXwire).

The present study reports the evolution of γ-matrix Ni-311 in the tangential component at a constant wavelength of λ = 1.62 Å and 2θ = 95°. Imaging of spatially resolved strain distribution in height and time including the cooling period was achieved. A continuous data acquisition approach, *event mode*, was used during the experiment. Results were later investigated in time and processing regions starting from melt pool towards baseplate named as melt pool, near-melt pool, and far-field. A hypothesis was put forward to explain overall strain contributions by considering temperature, microstructure, and stress-based events as total strain ($\varepsilon_{(hkl),T}$) is contributed by thermal ($\varepsilon_{(hkl),TH}$), microstructural ($\varepsilon_{(hkl),M}$), and RS-based ($\varepsilon_{(hkl),S}$) strain. Complementary post-mortem microscopy and neutron powder diffraction analysis proved the utmost relevance of in-situ studies towards understanding the fundamental controlling parameters of RS formation in AM.

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The Material Engineering Diffractometer BEER at ESS

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Keywords: material science; instrumentation; modulation technique

The time-of-flight engineering diffractometer BEER [1] (Beamline for European Materials Engineering Research), which is under construction at the European Spallation Source (ESS), will offer new opportunities for investigations of microstructures, residual stress evolutions and in-situ phase transformations under near-processing conditions.

BEER combines the high brilliance of the ESS source with large instrument flexibility. The diffractometer includes a novel beam-shaping technique, the so-called modulation technique [2]. By a time-encoded extraction of several short pulses from the long ESS pulse, a substantial intensity gain of up to an order of magnitude compared to a pulse shaping method (one pluse extraction) for high-crystal-symmetry materials can be achieved without compromising the resolution. More complex crystal symmetries or multi-phase materials can be investigated by the standard pulse shaping method. The variable chopper set-ups and advanced extracting techniques [3] offer broad intensity/resolution ranges that can be adjusted for the experiment's needs. This flexibility opens up new possibilities for in-situ experiments studying materials processing and performance under operating conditions. Advanced sample environments dedicated to thermo-mechanical processing are foreseen to fulfil this task, e.g. a quenching and deformation dilatometer.

Here we present the main components and features of BEER and the sample environment to be available on day one of operation. We give an outlook on future upgrades that will further increase the performance and uniqueness of BEER at the ESS.

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Line profile analysis and rocking curve evaluation in individual grains of β-Ti polycrystal

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Keywords: Slip systems; Dislocation densities; β-Ti; HEXRD; in-situ deformation

In this work X-ray diffraction was measured during in-situ tensile deformation test on β-Ti polycrystalline material. During the experiment 105 grains diffraction patterns were detected. The used X-ray diffraction-based technique was able to evaluate dislocation densities in each grain individualy. Furthermore, the technique discriminates dislocation densities of different slip modes, slip systems and dislocation character. For each grain was measured 55 reflections in average and these were fitted with CMWP program to obtain contrast factor for each reflection. In furher analysis Monte-Carlo-type algorithm was involved in order to determine the dislocation structure best representing the single crystal diffraction peak profiles.

In the model two different slip modes were taken into account: <111>{110} and <111>{112}, altogether with 28 slip systems. The evaluation of profiles was made for different deformation steps with the aim to monitor the evolution of dislocation structure in individual grains. According to the results at the beginning of plastic deformation the nucleation of screw dislocations is much pronounced than the nucleation of edge dislocations. On the other hand, in the final stages of plastic deformation the screw dislocation density growth stops and the edge dislocation density with <111>{110} mode starts to increase dominantly. The crystallographic orientation of grains also shows influence on the dislocation density evaluation.

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Residual stresses in Al-Cu clad composites processed by rotary swaging

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Keywords: severe plastic deformation; composite material; neutron scattering; residual stress

Novel materials, together with modern production technologies, are needed for a sustainable development in the transport industry as well as in energy production, distribution, and storage. There is an ongoing transition towards multiphase and composite materials with microstructures and properties tailored for special functionalities. For example, the automotive industry and electrical engineering search for novel alloys and composite materials for replacement of relatively expensive and heavy copper conductors.

Intensive plastic deformation is a very advantageous way for modification of conventionally fabricated metallic materials in order to achieve ultrafine-grained or nanocrystalline structures. Technologies imposing intensive shear strain includes rotary swaging (RS) which improves not only the mechanical properties of the processed work-pieces, but also the quality of their structures without deteriorating their physical or utility properties. Among the well-known composites are the clad composite materials prepared by plastic deformation [1].

Residual stress within clad composites can decrease their lifetime and could have fatal consequences for special applications. Neutron diffraction technique is valuable experimental tool for non-destructive investigation of residual stress within materials.

The primary motivation of this work is to investigate the effect of parameters of the intensive plastic deformation technologies on the *residual stress*. This goal requires to prepare *Al-Cu clad composites* and subject them to *RS under different processing routes*. The Al filaments will be specially arranged to observe their effect within the samples.

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Optimisation of post-built annealing of Ni Alloy718 processed by powder bed fusion

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Keywords: Nickel superalloy, additive manufacturing, residual stresses, precipitation

Laser powder bed fusion provides valuable prospects for nickel-based superalloys that are used in many applications e.g. aerospace, automotive, chemical, and nuclear industries. However, the microstructure and mechanical properties of these materials are especially sensitive to the manufacturing conditions and post-treatments that are applied to relieve the internal stresses, as they are susceptible to the formation of different types of precipitates, depending on the alloy chemistry, temperature and time [1]. Generally, a two-step annealing is performed to obtain the desired microstructure, meanwhile resulting in stress relief in the as-built condition[2]. Such treatments are however non-economic and change significantly the as-built microstructure, which is often deliberetly manipulated by tailoring the process parameters.

A combination of in situ high-temperature neutron diffraction and synchrotron X-ray diffraction was used to study the evolution of the residual stresses and precipitation in nickel based Alloy 718 for a temperature range from 600°C to 1000°C. Samples with cylindrical symmetry were prepared and by using the 2-bank detector system at the Engin-X beamline, ISIS UK, it was possible to follow the evolution of residual stresses. Rietveld analysis on the X-Ray diffraction data was used to evaluate the initial phase composition and its evolution during the annealing [3]. These results allow for the optimisation of one-step annealing treatments to relieve residual stresses and, in the meantime, control the microstructure and improve the mechanical properties of the final material.

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Residual Stresses in Titanium Prepared by CONFORM ECAP

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Keywords: stress, ECAP, dislocation, texture, diffraction

The CONFORM technique combined with the equal angular pressing (ECAP) enables the industrial-scale production of the ultrafine-grained (UFG) materials, which are well known due to their enhanced mechanical properties. Since these processes belong to the severe plastic deformation methods, residual stresses can remain in the processed material which increases the probability of the reduced lifetime of the workpiece. The present study is focusing on the residual stress (RS) field in a UFG titanium grade 2 after multiple passes of the CONFORM ECAP (C-ECAP) process.

Commercially pure titanium grade 2 rods, prepared by one to three passes of the C-ECAP process, were studied by neutron (ND) and X-ray (XRD) diffraction methods. Three mutually perpendicular - axial, radial and hoop components of the RS were calculated based on the ND strain scanning results in the circular cross-section of the cylindrical samples. At the ND scanning points, local macro-textures and dislocation densities were evaluated with the help of XRD. Moreover, samples cut from studied regions of the rods were mechanically tested in compression.

As the results show, a highly heterogeneous distribution of the RS was found in the samples and the largest RS gradient appeared in the sample after a single pass of C-ECAP process. However, after subsequent passes, the RS gradients were gradually reduced. The measured average dislocation density distributions show similar evolutions, which are in agreement with the RS distributions. Also, the results of texture measurement and compression tests correlate with results provided by previous ND and XRD measurements. These findings helped to enlighten the micromechanical processes responsible for the formation of the RS field in the samples.

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Prediction of Martensite Textures in NiTi wires

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Keywords: shape memory alloys; nitinol; martensitic transformation; twinning; lattice correspondence;

Superelastic or actuator cold drawn NiTi wires are used in biomedical and industrial applications [1]. Their excellent functional and structural properties are ascribed to reversible martensitic transformation and strong preferential orientation of austenitic <111> directions along the wire axis. Due to low symmetry and multiple twinning modes of the monoclinic B19’ martensite structure, the cubic B2 austenite single fibre texture transforms into martensite multiple fibre textures [2-4]. Actual number of fibers depend on deformation history governing the activation of twinning deformation processes.

In this work, we calculated martensite multiple fibre textures in NiTi wires by using an ideally austenite single fibre textured model. The model accounts for lattice correspondence between austenite and martensite, transformation strain and all martensite twinning systems, including irreversible deformation twins. The calculations enabled to construct theoretical inverse pole figures, including the twinning strains, that facilitated interpretation of martensite texture evolution experimentally observed in a martensitic NiTi wire during tension up to large plastic deformations.

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In-situ synchrotron x-ray diffraction texture analysis of tensile deformation of nanocrystalline NiTi wire in martensite state

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Keywords: synchrotron X-ray diffraction; in-situ texture; NiTi shape memory alloys; tension; Rietveld refinement;

Tensile deformation of nanocrystalline superelastic NiTi wire at -90°C in martensite state until fracture was investigated by in-situ synchrotron x-ray diffraction texture analysis. In-situ texture evolution was studied by Rietveld refinement of sequential 2D diffraction patterns recorded continuously using fast 2D detector, and was presented using pole figures and inverse pole figures [1,2]. The sequential 1D diffraction spectra along the loading/wire axis direction were fitted to obtain information on the integrated intensity, lattice strain and peak width of individual grain families diffracting along that direction.

The obtained results were interpreted with (i) austenite-martensite lattice correspondence upon transformation, (ii) deformation mechanisms during martensite reorientation and plastic deformation [3], (iii) TEM analysis of lattice defects left in the austenitic microstructure of the deformed, unloaded and heated wire [4], (iv) an abstraction of "*ideally fibre textured <hkl> polycrystal*" that accounts for austenite-martensite lattice correspondence, and all martensite twinning modes, including irreversible deformation twins. It shows that synchrotron x-ray diffraction is an effective tool for in-situ texture evolution during thermomechanical loading of in thin NiTi wire.

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Utilization of laboratory energy dispersive X-ray diffraction for stress determination in polymers as a supplement to synchrotron experiments

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Keywords: Laboratry EDXRD; Spectral 2D detectors; stress-strain polymers

Granted beam time for experimental work at synchrotron facility is usually very limited and therefore investigation of the conditions yielding stress in the polymer structure prior to the beam time are advantageous.

Characterization of the samples using a conventional laboratory X-ray diffractometer with monochromatic X-rays generated from the element specific fluorescence is hampered by the low flux density and material penetration due to the low energy of the X-ray photons. Energy dispersive X-ray diffraction (EDXRD), utilizing the full Bremspectrum emitted by the X-ray source, provides both a higher flux density and material penetration due to the use of the high energy part of the spectrum.

Feasibility studies of tensile stress-strain experiments on polymer samples were performed in a laboratory EDXRD utilizing an industrial X-ray source providing photons with energies up to 160 keV. For the data acquisition two different types of detectors were utilized, (1) a spectral point detector with very good energy resolution and (2) a fully spectral pixelated area detector. (2) owes a high spatial resolution which together with a larger area and the option of placing the detector closer to the sample provide a faster data collection, Furthermore since (2) resolves and collects the the azimuthal distribution of the signal investigations of the anisotropy of the stresses are possible, but has a worse spectral resolution.

The results from EDXRD data are evaluated with the outcome of in situ high energy XRD stress-strain experiments which were conducted at a synchrotron facility.

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Tomography driven diffraction capabilities of the new DIAD beamline

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Keywords: in-situ; strain; diffraction; tomography

The Dual Imaging and Diffraction (DIAD) beamline is a new instrument constructed at Diamond Light Source, UK. The beamline has been designed to conduct correlated in-situ/in-operando experiments combining synchrotron X-ray diffraction for phase identification and strain analysis, and tomography for three-dimensional microstructure analysis.

Operational photon energies of 8-38 keV, use of a micro-focused diffraction beam (fixed spot size 50 μm x 50 μm) and a 1.2 mm x 1.0 mm imaging beam with a 1 μm imaging resolution makes the beamline suited to studies of in-situ processes from the same specimen. Using two independent incident beams the DIAD instrument permits switching between diffraction and imaging operational modes at up to 10 Hz with independent energy selection for X-ray imaging (radiography or tomography) and diffraction.

To permit working directly in a calibrated imaging reference frame the dual-beam instrument design is based on a moving diffraction source. This represents a fundamentally different approach to diffraction data collection approaches applied on other beamlines. In this presentation results from work on the geometric calibration of DIAD's moving beam diffraction geometry, alignment methodology for the diffraction and imaging coordinate systems and key instrument resolution parameters (e.g. strain accuracy) will be presented.

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Configuration of Deformation Rig Developed for Beamline at European Spallation Source

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Keywords: Deformation rig; BEER; ESS

Medium-size deformation rig was designed and constructed in the Nuclear Physics Institute of the Czech Academy of Sciences. It will be used as a part of the sample-environment equipment of Beamline for European materials Engineering Research (BEER) at European Spallation Source (ESS) [1]. The developed rig will allow performing in-situ materials testing and physical experiments in combination with various neutron analytical techniques.

The deformation rig is equipped with 100kN actuator (nominal load is 60kN) and a vacuum chamber for high temperature deformation experiments. Fast Joule heating of the sample is foreseen with two 6kW power supplies. The control system is based on the CompactRIO (real-time embedded industrial controller), which includes FPGA module for fast data processing. The firmware and software have been developed in LabVIEW. Communication between the rig and beamline at ESS will be implemented with EPICS through MQTT. The rig will allow performing deformation tests with a strain rate in the range of 10^{-3}-100 mm/min. Various types of holding grips will be developed with respect to the shape and the size of testing samples.

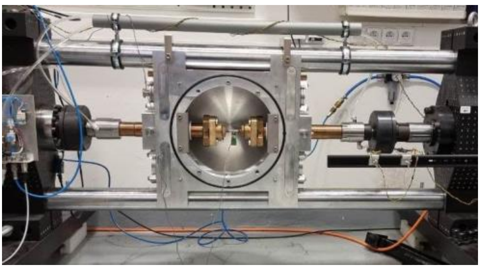

Fig.1 – Deformation rig appearance

[1] https://europeanspallationsource.se/instruments/beer#sample-environment

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Depth resolving stress in amorphous polymers

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__Keywords__: EDXRD; Spectral 2D detectors; strain; polymers; reconstruction

In production, the strain distribution in a component can give vital information about mechanical properties and product lifetime. In Poulsen *et al* [1], characterization of strain was shown for amorphous material in the form of metallic glass. Polymers similarly have an amorphous phase and the same methodology can be used to access the strain tensor components [2]. Scanning a focused x-ray beam will provide information of strain in the two scan-directions but mapping the third depth dimension is equally necessary for an accurate analysis. We use 2-dimensional detectors for the measurements to resolve the strain tensor for the axial strain ε_{11}, the transverse strain ε_{22} and the in-plane shear component ε_{12} through the expression:

$$\varepsilon_i = \varepsilon_{11} \sin^2(\eta_i) + \varepsilon_{12} \sin(\eta_i) \cos(\eta_i) + \varepsilon_{22} \cos^2(\eta_i)$$

where η_i corresponds to the azimuthal angle where the local relative shifts ε_i of the diffraction peaks are found.

We present data from synchrotron, as well as laboratory measurements using a detector with high spatial and spectral resolution. Finally, we investigate the influence of experimental parameters on the separation between diffraction spectra along the axial direction of depth using simulations. For distributions in a two-dimensional sample we an experimental strain resolution of the order 10^{-4}. This is achieved through analysis of the diffraction signal acquired with a 2D detector which allow us both a fine radial and an azimuthal resolution. For 3-dimensional samples we report how the third dimension can be resolved from multiple EDXRD measurements by reconstruction.

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Multiscale residual stress analysis using x-ray and neutron dark-field microscopy

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Keywords: multiscale analysis; dark field microscopy; diffraction; synchrotron x-ray; neutrons (up to 5 words)

Residual stresses exsist on several length scales from macroscopic (type I) to individual grains (type II) down to sub-grain level (type III). All of these stresses affect the performance and lifetime of a material and hence component. To fully understand a material's properties, the stresses on all lengths scales hence have to be analysed.

The type III stresses can be analysed using synchrotron x-ray dark-field microsopy revealing the variations in the stresses within individual grains [1], such as the stress fields around individual dislocations [2]. This technique has here been shown to also enable insights into the type II stresses by probing the individual grains within a powder type sample.

To probe the type I stresses, neutrons are preferred because of their penetration power into metallic structures. With the right optics [3] an equivalent setup as used with x-rays can be used to analyse stresses in a neutron dark-field microscope [4]. Challenges in the manufacturing of this optics is discussed while initial tests of compound refractive lenses for neutron microscopy are shown.

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Harmonisation and standardization of industrial residual stress measurement using neutrons and synchrotron x-rays

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Keywords: standards; industry; stress measurement; data format

Non-destructive measurement of bulk residual stresses using neutron and synchrotron x-ray diffraction has been employed for studies on industrial components over the past 40 years. Despite this, the measurement techniques have not attained widespread industrial acceptance and design standards, such as EUROCODES 3, do not refer to measured bulk residual stresses. Instead, large design safety factors are imposed resulting in over-dimensioned components and added material use and cost.

The EASI-STRESS project (EC no. 953219) [1] has assembled key stakeholders from industrial sectors and neutron and synchrotron facilities. The consortium partners aim to lower the barrier for wider industrial use of non-destructive bulk residual stress measurement techniques by:

- benchmarking the non-destructive techniques with more accepted destructive measurement techniques used in industry such as contour mapping and hole drilling,
- ensuring interoperability and reproducibility by harmonizing protocols and data formats and ensuring they are compatible with industrial modelling software,
- developing a new standard for synchrotron x-ray residual stress measurement and posing recommendations for improvements to the existing neutron residual stress measurement standard [2],
- demonstrating the industrial applicability of the measurement techniques across several industrial sectors.

The consortium includes eight industrial partners from the following sectors: energy, metal manufacturing, additive manufacturing, aerospace, and automobile. The consortium also includes two neutron and two synchrotron facilities in order to ease the uptake of the new measurement procedures and protocols across all interested facilities.

[1] EASI-STRESS - European Activity for Standardization of Industrial residual STRESS characterization: https://cordis.europa.eu/project/id/953219
[2] ISO 21432:2019: Non-destructive testing — Standard test method for determining residual stresses by neutron diffraction.

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Microscopic stress-strain evaluation of age-hardened AA7075 during repeated stress relaxation at elevated temperature

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Keywords: Aluminum 7075 alloy, in-situ neutron diffraction, stress relaxation, mechanical properties

Aged-hardened AA7075 aluminum alloys have increased widespread application due to high strength-weight ratio and good recyclability. Especially, AA7075 is often used at high temperatures for components and structures adjacent to the aircraft engines or automotive heat exchangers. In these cases, fatigue, stress relaxation, and creep are major concerns. Therefore, structural parts of AA7075 must be tested to ensure their safe and predictable lifetime, and thus the evaluation of mechanical properties and stress relaxation is crucial to elucidate the changes in microscopic stress-strain behavior during tensile and repeated stress relaxation. In this study, the repetitive stress relaxation test with in-situ neutron diffraction was performed to investigate the different macroscopic and microscopic stress relaxation behaviors of AA7075 at 150 ^oC under the different heat treatment conditions. The entire diffraction patterns monitored during in-situ stress relaxation enabled us to examine (1) lattice strains, (2) shear stress between different orientation, and (3) microstructure development. Regardless of heat treatment conditions and relaxation cycles at high temperature, a stress drop was almost identical due to the activities of dislocation climb by diffusion, and the creep mechanism became more predominant. Moreover, the responses of the {hkls} lattice strains at 150 ^oC were different with those at 25 ^oC [1]. The current study provides insights on stress relaxation mechanisms at elevated temperatures.

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New analysis method of multireflection grazing incidence X-ray diffraction

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Keywords: residual stress; grazing incidence diffraction; coating layer; elastic constants

In this work, a new analyses of experimental data obtained using MGIXD measurements are proposed and successfully tested on Ni samples exhibiting significant elastic anisotropy of crystals [1]. Three different ways of biaxial stresses and lattice parameter determination were compared. In the first approach, the calculations were performed using liner least square method and next two simplified procedures based on the simple linear regression (weighted and non-weighted) were applied. It was found that all the tested methods give similar results, i.e. almost equal values of the determined stresses, lattice parameter as well as the uncertainties of their determination. The advantage of the analyses based on simple linear regression is their simplicity and straightforward interpretation enabling easy verification of the influence of texture, presence of shear stresses, as well as graphical determination of stress free lattice parameter.

It was found, that the anisotropy of XSFs (or XECs) is well predicted by the Reuss, Eshelby-Kröner and Free-Surface methods but it is not possible to decide which model is the best one. The worst result was always obtained with Voigt method.

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Saccharine effect on the microstructure and stress state in nickel electrodeposited on copper substrate

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Keywords: Nickel electrodeposition, Diffraction analysis, SEM, AFM, microstructure

Saccharin effect on the nickel electrodeposition on copper substrate was a goal of the present paper. It was found that a tensile stress of about 500 MPa is produced during the deposition from an elementary Watts solution at a current density of 5 A/dm^2. However, if a small amount of saccharin was added to the solution (the brightening compound), no stress was found in the coating. Microscopic study (SEM, TEM and AFM measurements) and X-ray diffraction show that the saccharin addition effect on the residual stress is related to the deposited layer microstructure and crystallographic texture. The volumetric analysis indicated also the effect on the hydrogen content in the deposited layer. Electrochemical studies clearly indicate that the electrolysis process is controlled by adsorption. The performed experimental study leads to conclusion that saccharin builds a layer on the surface of depositing nickel, which changes the layer growing characteristics and blocs escape of hydrogen.

Density Functional Theory based calculations (Gaussian software) show that two types of the saccharin-Ni complex can be formed and their energy difference is less than the energy of thermal vibration. Therefore, these forms of the saccharin-Ni complex can be treated as the resonance ones.

In the stress analysis performed for the Ni-coatings the Multireflection Grazing Incidence X-ray Diffraction Method (MGIXD) was used [1]. This technique allowed us to determine stress gradient in the deposited layer.

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This work was partially supported by grants from the National Science Centre, Poland (NCN), No. UMO-2017/25/B/ST8/00134. Oponowicz A. has been partly supported by the EU Project POWR.03.02.00-00-I004/16

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Diffraction measurement condition suitable for stress analysis of Polyphenylenesulfide component

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Keywords: synchrotron diffaraction, PPS, strain anisotropy

Polyphenylenesulfide (PPS), which is one of the engineering thermoplastic resins, is widely adopted as a light-weight structural material. Since PPS is partially crystalline, x-ray diffraction technique is applicable for measuring the stress and strain condition. When PPS is used as composite materials or is embedded in arbitrary components, however, it is necessary to avoid overlapping of the diffraction peaks with those of the other materials. In this study, authors conducted synchrotron radiation x-ray diffraction measurements to select suitable diffraction peaks of PPS in the composite materials or components.

The synchrotron radiation x-ray energy of 29 keV was applied for smooth tensile specimen of PPS under uniaxial loading. It was found that the stress sensitivities of the peaks were strongly related to the crystal structure. This result would give us the second or third best diffraction peaks for the stress measurement when the first best peak unfortunately overlaps with the peaks from the other component materials.

The synchrotron radiation experiments were performed at the BL33XU of SPring-8 with the approval of the Japan Synchrotron Radiation Research Institute (JASRI) (Proposal No. 2017A7012, 2017B7012 and 2018A7012).

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Non-destructive analysis of lead-free solder degradation in power electronic module by neutron diffraction

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Keywords: power electronic module, neutron diffraction, orientation

Reliability of the power electronic module for automobiles [1] is one of the most important factors for both the energy efficiency and the safety. The module mainly consists of Si power semiconductors and copper heat sinks, and then a lead-free solder layer is used to joint two parts, and finally the module is surrounded by an epoxy resin composite. This condition gives a difficulty to measure their elastic/plastic strains by X-ray, because of the poor x-ray attenuation in the thick copper plates. On the other hand, neutron diffraction has potentials to visualize the internal state non-destructively.

In this study, the authors have analyzed degradation of lead-free solder layer of power electronic module surrendered under thermal fatigue. The degradation was analyzed by the texture change measured at BL19 (TAKUMI), J-PARC and the collective crystal rotation were studied. The neutron experiment at the Materials and Life Science Experimental Facility of the J-PARC was performed under a user program (Proposal No. 2020B0061).

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Influence of heat treatment on the residual stress in laser clad hypereutectoid rail components using neutron diffraction

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Keywords: laser cladding, hypereutectoid, repair, residual stress, neutron diffraction

Railway infrastructure is relied upon globally for freight and passenger transit for a growing population. Increasing speeds, frequency, and tonnage of rolling stock places extreme demands on critical railway networks leading to premature failures. This necessitates the continuous development of maintenance methods to meet the maintenance demand. Laser cladding can be used to apply coatings to recondition worn rail profiles. This process uses a high energy laser to melt a metallic powder at the rail surface to form a metallurgically bonded deposition [1]. As laser cladding is a thermal process, a heat affected zone (HAZ) is created below the fusion boundary containing a complex residual stress state due to changes in volume, solidification stresses and phase changes [2]. The internal stress in conjunction with cyclic wheel-rail contact stress governs the fatigue performance, therefore high or tensile residual stresses are undesirable, and they may reduce the service lifetime [3]. Understanding the residual stress is critical in predicting fatigue behaviour and the residual stress can be accurately measured using non-destructive neutron diffraction techniques. Laser cladding of a martensitic stainless steel has been carried out on sections of premium hypereutectoid heavy-haul rail. Triaxial residual stress was measured on the Kowari strain scanner at ANSTO using through thickness line scans, traversing the cladding, HAZ and substrate. Two tempering processes were applied to determine the influence of post-cladding heat treatments on the microstructure and stress generation. The outcomes from this investigation will assist in understanding the effect of post-cladding procedures on internal stress and extending the fatigue performance. This will aid in determining the optimal parameters for cladding repairs.

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The effect of carbon content on deformation mechanisms of high Mn steels at elevated temperature

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Keywords: Mn steel; In-situ neutron diffraction; Carbon; TRIP; TWIP

Mn steel reveals outstanding strength and elongation due to its activation of various deformation mechanisms, which leads the alloy to be widely used in automobile and shipbuilding industries. To understand the underlying deformation mechanisms under mechanical loading in real-time is a topic of crucial importance in controlling the mechanical properties of the alloy. In this study, in-situ neutron diffraction was employed to evaluate the effects of carbon content (0.1, 0.3, 0.5 wt.%) on the mechanical behavior and relevant deformation mechanisms (transformation/twinning-induced plasticity, TRIP/TWIP) of 17 Mn steels during tensile loading at 150˚C. The change in phase fraction and lattice strain between the phase constituents during deformation will be revealed, and stress contributions of each phase on the mechanical strength will be unfolded. Moreover, the phase stability of austenite during deformation will be discussed in terms of stacking fault energy.

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Strain and Microstructure Distributions around a Fatigue Crack Tip Studied by Neutron Diffraction

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Keywords: Fatigue crack; compact-tension specimen; residual strain; neutron diffraction

Fatigue damage is known as one of the major concerns in maintaining the integrity of large-scale engineering components. The nonlinear strain/stress distribution around a fatigue crack tip has a great impact to understand the fatigue and crack growth behavior of engineering component. Two kinds of steels (SUS304 and SUS329J4L) were used in the study. The fatigue tests on the compact-tension (CT) specimens were conducted with a servo-hydraulic fatigue testing machine. We performed the time-of-flight neutron diffraction strain mapping on the fatigued CT specimens at BL19 TAKUMI, an engineering diffractometer at MLF/J-PARC. Neutron diffraction data were collected with the gauge volume of 2×2×2 mm^3 and analysed by the Z-Rietveld software to obtain the lattice constant. Fig 1. shows the the obtained residual strain distributions in three directions at the middle thickness of a 6 mm thick SUS304 specimen, which was subjected to constant-amplitude cyclic loading mode (P_{max} = 7400 N, P_{min} = 740 N and frequency of 10 Hz) until a crack of 25 mm was developed after 2.9×10^5 cycles. It reveals strong compressive residual strains at the crack tip and rising to near-zero with increase of distance from the crack tip in the TD and RD directions, while those in the ND direction show tension near the tip and decreased to near zero at about 7 mm from the crack tip. Diffraction results for specimens tested under different loading conditions and comparisons with surface strains by digital image correlation method will be presented.

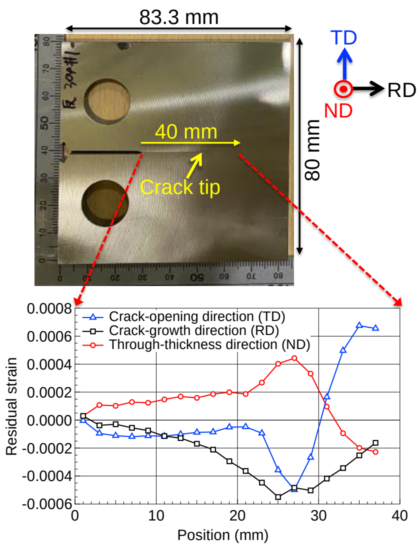

Fig.1 Residual strain determined via neutron diffraction.

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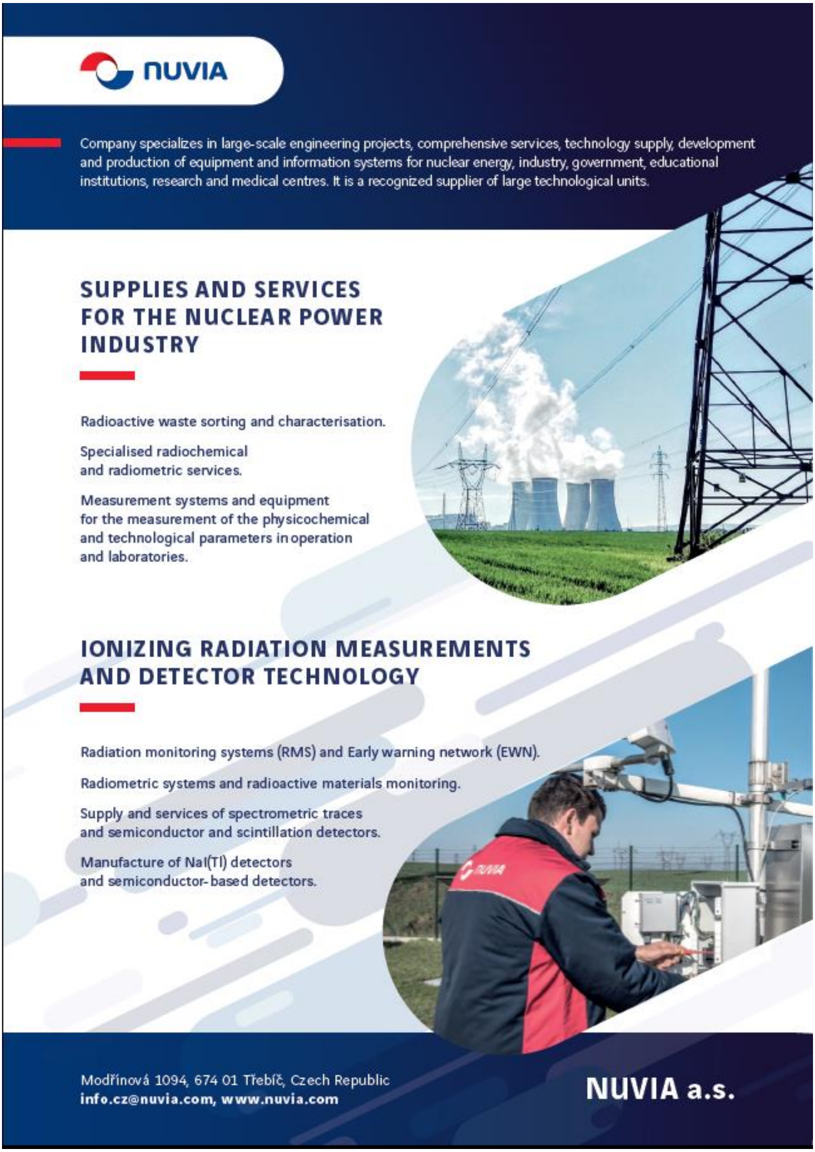


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