ESRF

New Opportunities in Diffraction Microscopy

8 - 11 January 2024

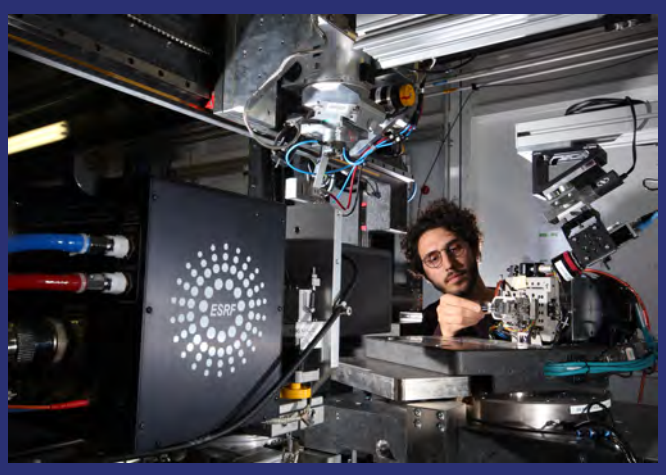
The primary objectives of this workshop are to foster the growth of the user community for the newly upgraded Dark Field X-ray Microscope (DFXM) instrument at ID03, and to define the scientific cases for a potential future upgrade of optics at ID11.

8 January
Metallurgy I
Phase Transformations I

9 January
Industrial Applications
Metallurgy II
Damage, Fatigue, Creep, Aging
News from ID03 and ID11

10 January
Modelling
Semiconductors
Ceramics
Software

11 January
Functional Materials
Phase Transformations II
Executive Session



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STREAMLINE

ESRF
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EPN Science Campus
71 avenue des Martyrs, 38000 Grenoble

https://www.esrf.fr/home/events/conferences/2024/DiffractionMicroscopy.html

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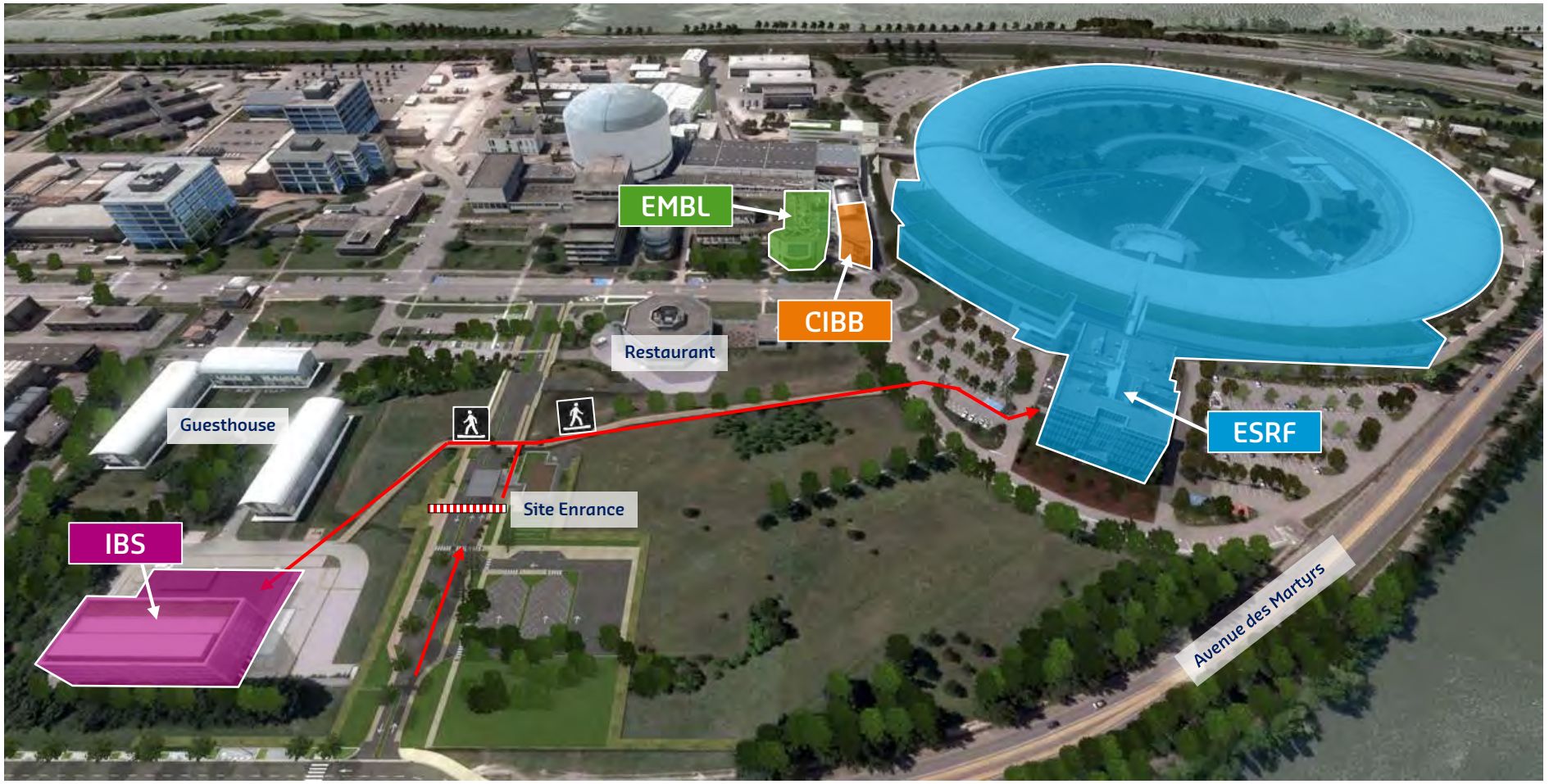

Grenoble downtown

Train station
Highway to Lyon
Shuttle from/to Lyon Airport
Shuttle from/to Geneva Airport
Tramway

Highway

To Chambery / Geneva

CIBB	Carl-Ivar Bränden Building – Partnership for Structural Biology
EMBL	European Molecular Biology Laboratory Grenoble
ESRF	European Synchrotron Radiation Facility
IBS	Institut de Biologie Structurale

Poster	Last Name	First Name	Affiliation	Title
P1	Atzen	Madlen Denise	Universitaet Ulm	Abnormal Grain Growth in the Aluminum Alloy 5252 Investigated in 3D by LabDCT
P2	Borbély	Andras	Ecole Nationale Superieure des Mines	Femto-second laser induced sub-surface hardening in metals
P3	Boronski	Claire Emile	University of Colorado Boulder	Transformative Approach to Controlling Lead in Drinking Water informed by Advanced Diagnostics
P4	Bose	Subham	Johns Hopkins University	CT and 3DXRD on Non-Ideal Crystals for Geomechanics Applications
P5	Chen	Yunhui	RMIT	How Deep Does It Go: Revealing 3D Strains in Additively Manufactured Alloys
P6	Dake	Jules	Ulm University, Germany	Tracking the emergence and persistence of abnormal grain growth in aluminum
P7	Detlefs	Carsten	ESRF	ID03: The new Hard X-ray Microscopy Beamline
P8	Foster	Daniel Jon	ESRF	The correlative potential of in-situ EBSD and in-situ synchrotron X-Ray diffraction
P9	Frewein	Moritz Paul Karl	Institut Fresnel	Texture tomography for polycrystalline materials: Principle and first results
P10	Gayoso	Antonella	Technican University of Denmark	Dark Field X-ray microscopy for the determination of chemical strain
P11	Hamilton	Dylan Charles	University of Colorado Boulder	Understanding chemo-mechanical failure mechanisms in solid-state batteries using X-ray diffraction computed tomography
P12	Heller	Ludek	Institute of Physics ASCR	Microstructure, Strains, and Stresses in NiTi Wire Under Tension Prior Stress-induced Martensitic Transformation Reconstructed Using Scanning 3DXRD
P13	Kalacska	Szilvia	Mines St. Etienne	TBC
P14	Ladbrook	Evangeline	University of Warwick	Going head-to-head with domains: 3DXRD for discovering domain structure in hybrid improper ferroelectrics
P15	Lauridsen	Erik	Xnovo Technology	Advanced acquisition with Lab-based X-ray Diffraction Contrast Tomography
P16	Li	Qianqian	Imperial College London	Advanced Microstructure Analysis of SiC nanowhiskers reinforced magnesium (Mg) composites
P17	Moya	Janice	University of Michigan	In-situ characterization of martensitic phase transforming CuAlNi shape memory alloys using X-ray Topotomography and diffraction contrast tomography
P18	Prikoszovich	Konrad	Karlsruhe Institute of Technology	Lattice strain distribution within BCC polycrystals – Bridging the statistical gap between experiment, simulation and theory
P19	Rack	Alexander	ESRF	Beamline ID19: a versatile station for synchrotron-based full-field hard X-ray microimaging for real-time in situ studies in materials research
P20	Resende	Pedro	ESRF	In-situ characterization of stress assisted grain boundary oxidation in Ni superalloys
P21	Rodriguez Lamas	Raquel	ESRF	4D mapping of epitaxial stress relaxation through dislocation arrays in complex oxide superlattices
P22	Tambe	Indrajeet	Malmo University	Investigating Precipitation Hardening and Grain Growth in Open Cell Aluminum Foams: In-Situ Analysis during Controlled Annealing.
P23	Thiercelin	Léo	Arts et Metiers Institut de Technologie	Development of digital twins for in-situ martensitic transformation of high entropy and shape memory alloys
P24	van Hulzen	Martijn	Delft University of Technology	Quantifying heterogeneity in Li-ion Battery Cathodes
P25	Wright	Jonathan	ESRF	Scanning 3DXRD reconstructions of a K-Type Thermocouple
P26	Yildirim	Can	ESRF	4D Microsctructural Evolution in a Heavily deformed Ferritic Alloy: A new perspective in Recrystallization Studies
P27	Zelenika	Albert	Technican University of Denmark	Studying Dislocation Patterning During Plastic Deformation of Pure Aluminium by Means of Dark-field X-ray Microscopy

New Opportunities in Diffraction Microscopy
ESRF - Grenoble - France
8 - 9 - 10 - 11 January 2024

STREAMLINE

	MONDAY 8 JANUARY	
13:00 - 13:45	**Registration and Welcome coffee**	**ESRF Central Building Entrance Hall**
	SESSION 1 - Metallurgy I (Chair: Carsten Detlefs)	
13:45-14:00	Welcome and scopes and objectives of the workshop	**Gema Martínez-Criado** Director of research, ESRF
14:00-14:40	**Invited -** Critical demand of full field microstructural characterization for recrystallization and grain growth	**Yubin Zhang** DTU
14:40-15:00	Diffraction imaging on creep-resistant self healing alloys	**Haixing Fang** ESRF
15:00-15:20	Non-destructive multimodal 3D quantification of recrystallization nucleation	**Elisabeth Knipschildt-Okkels** DTU
	COFFEE BREAK	
	SESSION 2 - Phase Transformations I (Chair: Jon Wright)	
15:40-16:20	**Invited -** Studying martensitic phase transformations and deformation twinning with in-situ DFXM and topotomography	**Ashley Bucsek** University of Michigan
16:20-16:40	Grain-level effects on in-situ deformation-induced phase transformations in a complex-phase steel using 3DXRD and EBSD	**James Ball** ESRF
16:40-17:00	3DXRD operando study of the nucleation and growth dynamics of multiple Fe intermetallic phases of Al alloys in the solidification processes	**Jiawei Mi** University of Hull
18:00	**POSTER SESSION**	**ESRF Central Building Entrance Hall**
	TUESDAY 9 JANUARY	
	SESSION 3 - Industrial Applications (Chair: Henning F. Poulsen)	
08:50-09:30	**Invited -** Unveiling the Impact of Intragranular Strain Localization on the Functional Response of Technological Components: Insights from a DFXM Perspective	**Jozef Keckes** University of Leoben
09:30-09:50	Revealing the Full Strain Tensor in microelectronic devices by Nanobeam Scanning X-ray Diffraction Microscopy	**Cedric Corley-Wiciak** IHP Microelectronics
09:50-10:10	High resolution reciprocal space mapping at the Swedish Materials Science beamline P21.2 at PETRA III	**Ulrich Lienert** DESY
10:10-10:30	High-resolution spatio-temporal strain imaging revealsloss mechanisms in surface acoustic wave device	**Tobias Schulli** ESRF
	COFFEE BREAK	
	SESSION 4 - Metallurgy II (Chair: Can Yildirim)	
10:50-11:30	**Invited** - New Insights Into Hierarchical Microstructure Formation During Green Ironmaking With Hydrogen	**Yan Ma** Max Planck MPIE
11:30-11:50	Three-dimensional stress localizations and grain yielding under elastoplastic axial-torsional loading	**Jerard Gordon** University of Michigan
11:50-12:10	Improvements in 6D Reconstruction for Diffraction Contrast Tomography and Topo-tomography	**Zheheng Liu** INSA Lyon
	LUNCH BREAK	
	SESSION 5 - Deformation, Fatigue, Creep, Aging (Chair: Wolfgang Ludwig)	
13:40-14:20	**Invited** - Correlative Diffraction Microscopy Imaging experiments to investigate crystal plasticity	**Henry Proudhon** MINES ParisTech
14:20-14:40	In-situ investigation of elastic strain and stress during brittle rock failure using scanning 3D X-Ray Diffraction	**Jean-Baptiste Jacob** University of Oslo
14:40-15:00	Resolving intragranular orientation and stress fields in plastically deformed titanium using scanning 3D X-ray diffraction	**Wenxi Li** University of Michigan
	COFFEE BREAK	
	SESSION 6 - Semiconductors (Chair: Tobias Schulli)	

15:20-16:00	**Invited** - On the opportunities offered by X-ray microscopies for the advanced characterization of microelectronic components	**Patrice Gergaud** CEA Grenoble
16:00-16:20	Evidence of Strain localization at high angle grain boundaries in CdTe solar cell using Scanning 3D X-ray diffraction microscopy	**Aditya Shukla** DTU
16:20-16:40	In situ observation of plastic relaxation dynamics in InxGa1-xN thin films by full-field X-ray diffraction microscopy	**Carsten Richter** Leibniz Institute for Crystal Growth (IKZ)
	WEDNESDAY 10 JANUARY	
	SESSION 7 - Modelling (Chair: Henry Proudhon)	
08:50-09:30	**Invited** - The Neper/FEPX Project and its Applications to 4D Experimental/Numerical Studies	**Romain Quey** CNRS St. Ettiene
09:30-09:50	Advances in DFXM Simulations for Comprehensive Dislocation Analysis	**Henning Friis Poulsen** Technical University of Denmark
09:50-10:10	Full-Field Crystal Plasticity and Phase Field Modelling of elasto plastic damage behaviour of ice	**Soroush Motahari** Max Planck MPIE
10:10-10:30	The evolution of grain scale stresses: 3D-XRD vs CPFE	**Hamidreza Abdolvand** Uni. of Western Ontario
	COFFEE BREAK	
	SESSION 8 - Deformation, Fatigue, Creep, Aging II (Chair: Christoph Kirchlechner)	
10:50-11:30	Pushing the Deformation Limit of the ID11 Scanning 3DXRD Microscope	**Axel Henningsson** Lund University
11:30-11:50	Femto-second laser induced sub-surface hardening in metals	**Andras Borbely** MINES St. Ettienne
11:50-12:10	DFXM Imaging of Dislocation Dynamics in in-situ Deformed Aluminum	**Felix Tristan Frankus** DTU
	LUNCH BREAK	
	SESSION 9 - Phase Transformations II (Chair: Jozef Keckes)	
13:40-14:20	**Invited -** Probing electronic phase separation in highly correlated systems through 3DXRD	**Mark Senn** University of Warwick
14:20-14:40	Diffraction and Imaging studies of Neuron-Mimicking Devices and Their Environments	**Elliot Kisiel** University of California SD
14:40-15:00	3D Grain Growth inelectrical steel sheet studied by laboratory DCT	**Masato Yasuda** Nippon Steel
	COFFEE BREAK	
	SESSION 10 - Emerging Methods, Software and Instrumentation I (Chair: Andras Borbely)	
15:20-16:00	**Invited** - Future of High Energy Diffraction Microscopy: Going towards more complex specimens and environments	**Peter Kenesei** Argonne National Laboratory
16:00-16:20	Real-time imaging of acoustic waves in bulk materials with X-ray Microscopy	**Theodor Holstad** DTU
16:20-16:40	Wide Angle X-ray Scattering Tensor Tomography	**Mads Carlsen** PSI
	SESSION 11 - Facility update ID03 - ID11	
16:40-17:00	ID03	**Carsten Detlefs** ESRF
17:00-17:20	ID11	**Jon Wright** ESRF
19:00	**WORKSHOP DINNER**	**ESRF Central Building Entrance Hall**
	THURSDAY 11 JANUARY	
	SESSION 12 - Ceramics (Chair: Raquel Rodriguez Lamas)	
08:50-:09:30	**Invited -** Texture tomography for polycrystalline materials: Principle and first results	**Moritz Frewein** Institute Fresnel
09:30-09:50	Study of CSTZ ceramics plasticity with synchrotron light and correlative characterizations	**Marcelo Demetrio de Magalhaes** INSA
09:50-10:10	Can residual stresses disrupt the energy storage functionality of antiferroelectric NaNbO3 ceramics?	**Leonardo Soares de Oliveira** DTU
	COFFEE BREAK	

SESSION 13 - Emerging Methods, Software and Instrumentation II (Chair: Alexander Rack)		
10:30-11:10	**Invited -** Fast and ultrafast: stroboscopic DF-XRM	**Trygve Magnus Ræder** DTU
11:10-11:30	Diffraction Microscopy Developments at the Advanced Photon Source	**Peter Kenesei** APS
11:30-11:50	Neural networks for rapid phase quantification of Cultural Heritage X-ray powder diffraction data	**Victor Poline** UGA
11:50-12:10	Laue Microscopy	**Jean-Sébastien Micha** ESRF
12:10-12:20	Closing Remarks	
LUNCH BREAK		
EXECUTIVE SESSION		

New Opportunities in Diffraction Microscopy 8 to 11 January, 2024

Critical demand of full field microstructural characterization for recrystallization and grain growth

Yubin Zhang[a]

Traditionally, recrystallized grains/nuclei developed during annealing of single-phase deformed metals have been considered to be stress-free. However, cutting-edge synchrotron studies[1,2], using for example dark field X-ray microscopy and three-dimensional X-ray Laue micro-diffraction, have recently challenged this view by revealing the presence of residual stresses within these grains. The levels of the residual stresses are substantial in both partially and fully recrystallized structures when compared to the relevant driving forces of the relevant process, i.e. from either stored energy for recrystallization or boundary curvature for grain growth.

This work provides an overview of the residual stress measurements conducted in several materials systems, revealing the universality of such stresses in single-phase polycrystalline materials. It is revealed that these stresses vary both within and across individual grains. The origins of these stresses are rationalized based on the medium- to long- range residual stresses developed in the deformed matrix, with correlations to both macroscopic processing parameters (e.g. deformation strain, annealing temperature and recrystallization extent) and microscopic microstructural characteristics (e.g. grain shape and neighbor environment). Additionally, evidence suggesting stress-driven boundary migration mechanisms possibly prompted by these residual stresses is presented. The potential impacts of these residual stresses on the recrystallization and grain growth process as well as the material thermal stability through such mechanisms are discussed. The critical demand for full-field microstructural characterization techniques to advance our understanding of recrystallization and grain growth is highlighted, concluding with the proposition of critical experiments and suggestions for technique development for future research endeavors.

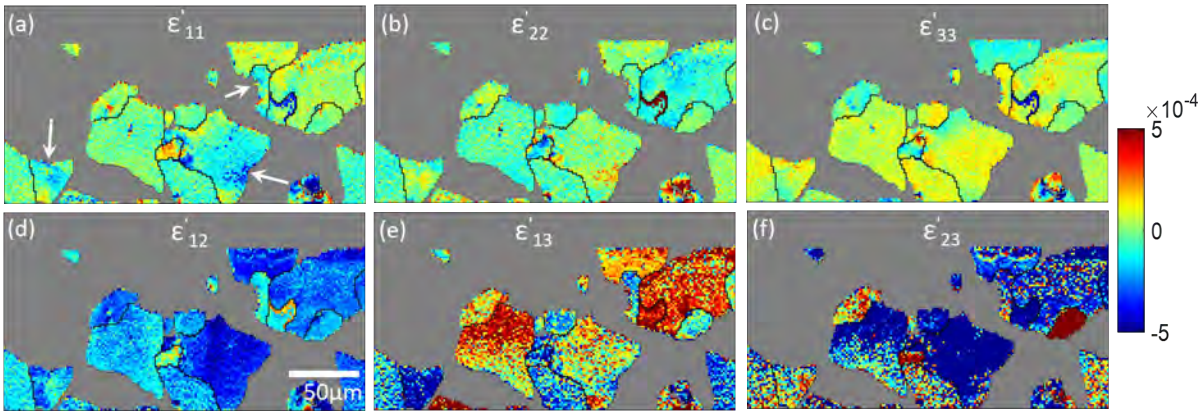

Figure: Deviatoric strain tensor maps showing the intragranular elastic strain distribution within recrystallized grains of a partially recrystallized pure iron sample. (a)-(c) are normal strain components, (d)-(f) are shear strain components. 1, 2 and 3 in the strain component subscripts represent the sample RD, ND, and TD, respectively. The white arrows in (a) mark local grain retrusions where large strain variations are seen.[1]

[a] Department of Civil and Mechanical Engineering, Technical University of Denmark, DK-2800, Kgs. Lyngby, Denmark

[1] Zhang et al. *Mater. Charact.* **191**, 112113 (2022).
[2] Lindkvist et al. *Mater. Res. Lett.* **11**, 942 (2023).

Diffraction imaging on creep-resistant self-healing alloys

Haixing Fang[1], Wolfgang Ludwig[1, 2], Jonathon Wright[1], Sybrand van der Zwaag[3] and Niels van Dijk[3]

Self healing is a promising new approach to control the delay or even remove the early stages of creep damage, hence prolong the lifetime of Fe-based alloys for high temperature structural applications. Recently we have demonstrated that efficient healing of creep damage can be achieved in binary Fe-Au and Fe-W alloys and Fe-Au-W ternary alloys due to intentional site-specific precipitation of Au enriched precipitates and Fe_2W Laves phase in grain-boundary creep cavities. Although the filling of cavities by precipitates is well captured by our previous synchrotron holotomography studies, there is no explanation why the cavities at some grain boundaries are filled nicely whereas other cavities are not filled and stay intact.

As a first attempt to answer this question, we have characterized the microstructures of two Fe-Au (3 wt.%) alloy samples with one creep interrupted and the other after creep ruptured using multi-modal imaging techniques available at beamline ID11, *i.e.* combining diffraction contrast tomography (DCT), phase contrast tomography (PCT) and 3DXRD (including the use of scanning focused beam) to resolve grain maps, cavities and gold precipitation. The results indicate a strong correlation between the changes in the lattice parameter (induced by possible residual strain and composition change) and the grain size and orientations. Variations of the compositions in the gold precipitates have also been found from the scanning 3DXRD indexing results. Analysis on individual grains linking the cavity filling by the gold precipitation as well as the grain orientations of neighbouring grains have also been performed. Typical examples of such analysis will be highlighted in this presentation. The next step to perform in-situ creep experiments at ID11 will also be sketched out.

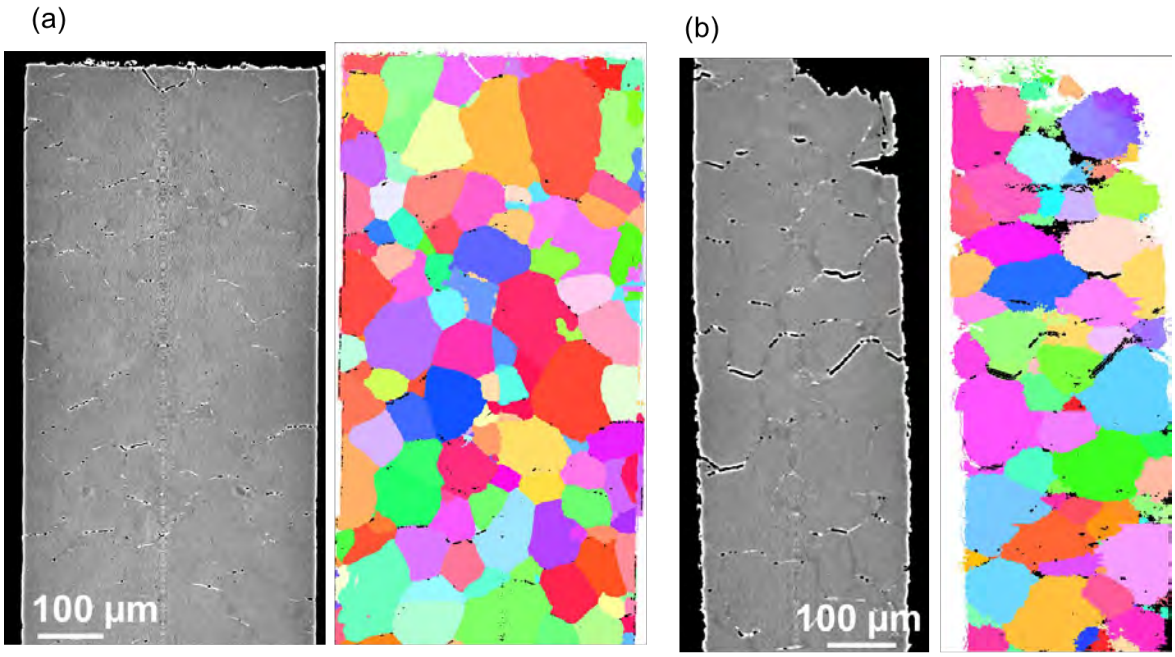

Figure: cross sections of Fe-Au alloy tomographic volumes and grain maps after (a) creep interrupted for a time of 188 h and creep rupture for a time of 376 h at 550 °C. In the tomographic slice, black corresponds to cavities and white corresponds to gold precipitates.

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[2] CNRS MatéIS, INSA-Lyon, Université de Lyon, 69621 Villeurbanne, France
[3] Delft University of Technology, 2629 JB Delft, The Netherlands

Non-destructive multimodal 3D quantification of recrystallization nucleation

E. F. F. Knipschildt-Okkels[1], Y. Zhang[1], X. Lei[2], T. Yu[1], W. Liu[3], S. Fæster[4], R. E. Sanders[5] and D. Juul Jensen[1]

Nucleation mechanisms are not well understood, partly due to the previous lack of suitable 3D investigation techniques, thereby most recrystallization characterizations have been carried out by 2D microscopy. However, characterizing nucleation in 3D of a bulk sample is now possible using some of the new X-ray techniques, e.g., Laboratory diffraction contrast tomography [1], 3D X-ray diffraction (3DXRD)[2], diffraction contrast tomography (DCT) [3], scanning 3DXRD [4] and Laue micro-diffraction [5].
The aim of this work is to study nucleation of recrystallization in metals using non-destructive multimodal 3D techniques. The focus is on quantifying particle stimulated nucleation in a large 3D volume of an AA5182 aluminum alloy cold rolled to 75 % reduction. Both second phase particles and aluminum nuclei are mapped in the same sample volume by laboratory X-ray absorption contrast tomography (CT) and synchrotron Laue micro-diffraction, respectively, see figure below. The two data sets are registered, and it is found that over 80 % of the nuclei are particle stimulated, though particles with different sizes and chemistry have different PSN efficiency. Strain analysis based on the Laue micro-diffraction data showed that residual elastic strain is present in the recrystallized nuclei, and these strains appear to stem from the deformed matrix. Further studies on effects of plastic strain in the deformed microstructure and residual elastic strain on recrystallization nucleation using DCT and scanning 3DXRD are discussed.

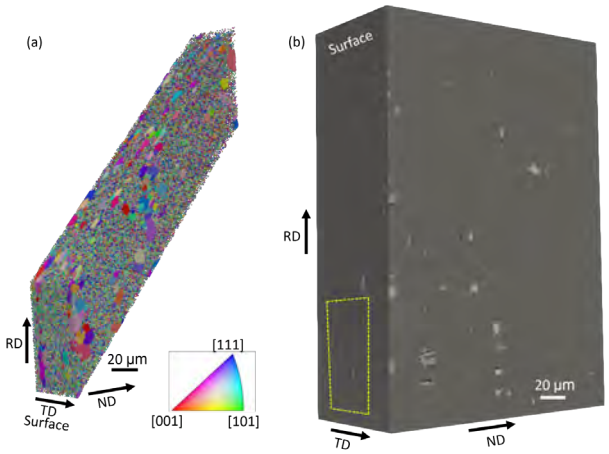

Figure: (a) Inverse pole figure map of DAXM data of the partly recrystallized AA5182 microstructure. (b) The raw CT data including the same volume in (a). The yellow dashed rectangle indicates surface area of volume in (a).

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[4] Department of Wind Energy and Energy Systems, Technical University of Denmark, Risø Campus, Roskilde, 4000, Denmark
[5] International Joint Laboratory for Light Alloys (Ministry of Education), Department of Materials Science and Engineering, Chongqing University, Chongqing, China
[1] Bachmann, Bale et al., *J. Appl. Crystallogr.* **52** 643 (2019)
[2] Oddershede, Schmidt et al. *Trans. Tech. Publ.* **652** 63 (2010)
[3] Reischig and Ludwig Curr. Opin. Solid State Mater. Sci. **24** (2020)
[4] Henningsson, Hall et al. J. Appl. Crystallogr. **53** 314 (2020)
[5] Hong, Zhang et al. *Scr. Mater.* **205** (2021).

Reconstructing the Structural and Electronic Heterogeneity in Functional Oxides with 3DXRD

Mark Senn1, Evie Ladbrook1 and Jon Wright2

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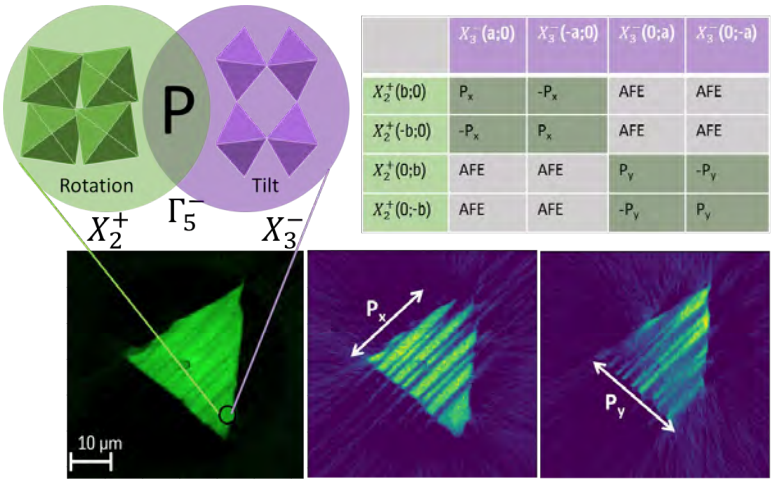
[2] *European Synchrotron Radiation Facility, 6 rue Jules Horowitz, 38043 Grenoble Cedex 9, France*

The conventional structure-property paradigm assumes that if we can understand atomic displacements within a material at the unit cell level, then we may start to rationalise its properties. However, in many functional materials, the technologically relevant properties arise from structural inhomogeneities that exist beyond the length scale of the unit cell. In this talk I will explore how *3DXRD* can be used to shed light on two such examples. Firstly, I will present how the rich domain structure that arises at this mesoscale in the hybrid improper ferroelectric $Ca_{2.15}Sr_{0.85}Ti_2O_7$ can be reconstructed from the diffraction contrast (Fig 1). The reconstructions shed insight into why experimentally these materials do not exhibit bulk switching of the polarisation. Secondly, in the colossal magnetoresistance manganite, I will show how very weak superstructure reflections associated with the electronic orderings can be used to produce a map of the percolative phase separation that underlines the materials functionality. Currently we are utilising only a very small fraction of the total information contained within the full 3DXRD dataset, and in these and other projects, we aspire in the future towards full spatially resolved crystallography at the sub 200 nm length scale.



	$X_3^-(a;0)$	$X_3^-(-a;0)$	$X_3^-(0;a)$	$X_3^-(0;-a)$
$X_2^+(b;0)$	P_x	$-P_x$	AFE	AFE
$X_2^+(-b;0)$	$-P_x$	P_x	AFE	AFE
$X_2^+(0;b)$	AFE	AFE	P_y	$-P_y$
$X_2^+(0;-b)$	AFE	AFE	$-P_y$	P_y

Fig 1: Reconstructions of the domain structure in hybrid improper ferroelectric Ca2.15Sr0.85Ti2O7. The improper ferroelectricity is driven by a distinct tilt and rotation of the TiO6 octahedra that couple to the ferroelectric polarisation (P). The diffraction contrast afforded by the associated superstructure reflections allows for a selective reconstruction of the associated domain structure and hence polarisation states.

Grain-level effects on in-situ deformation-induced phase transformations in a complex-phase steel using 3DXRD and EBSD

J. A. D. Ball[abc], C. Davis[d], C. Slater[d], H. Vashishtha[c], M. Said[c], L. Hébrard[e], F. Steinhilber[e],
J. P. Wright[a], T. Connolley[b], S. Michalik[b], D. M. Collins[c]

Many advanced steel alloys exploit a deformation-induced martensitic transformation (DIMT) to improve both tensile strength and ductility. In the DIMT, a metastable austenite (γ) phase transforms to a martensite (α') phase. The applied stress required to trigger this transformation is controlled by the austenite grain "stability". The direct γ→α' transformation is rare in steels — instead, an intermediate martensite phase (ε) is often observed as a precursor to α' nucleation, in a two-step γ→ε→α' transformation. Therefore, identifying the formation of ε within a γ grain provides a direct measurement of γ stability.

Studies of the stability of individual γ grains are rare, often measured two-dimensionally with surface-based techniques such as EBSD[1,2]. These studies can suffer from poor statistical significance, and cannot emulate the three-dimensional stresses experienced by γ grains inside the bulk of a sample. Three dimensional non-destructive techniques such as Three-Dimensional X-Ray Diffraction (3DXRD) are therefore required to measure a large number of γ grains in-situ. Some prior DIMT measurements have been done with these techniques, albeit on a limited number of grains[3,4]. The inherent strain limit of far-field 3DXRD (<1% applied strain) restricts the exploration of the DIMT in commonly-used alloy systems such as 301 or 304, where substantial phase transformations occur at much higher applied strains.

In this study, a model 304-based alloy system was developed to destabilise the austenite phase such that significant DIMT events occur at very low applied strains, just beyond the elastic limit. In-situ far-field 3DXRD measurements on an alloy sample were hence collected at the ID11 beamline of the ESRF.

A substantial number of ε formation events were captured, and linked to their parent γ grains via grain positions and orientations. Using the stress of the parent γ grains at the onset of ε formation as a measurement of γ stability, a number of γ stability relationships were confirmed with high statistical significance. Larger γ grains were found to be less stable than smaller grains (Fig. 1a). γ grains with {100} near-parallel to the loading axis were destabilised (Fig. 1b). Finally, γ grains with a more ferrite/martensite-dense neighbourhood were less stable against transformation (Fig. 1c). The accuracy of the "minimum strain work criterion" model was also validated — it correctly predicted ε martensite orientation variants 72% of the time, given the parent γ orientation. γ grains where the model failed were found to have lower Type II von Mises stresses, suggesting that local Type-III stresses, un-measurable with far-field 3DXRD, can modify the ε orientation that eventually forms.

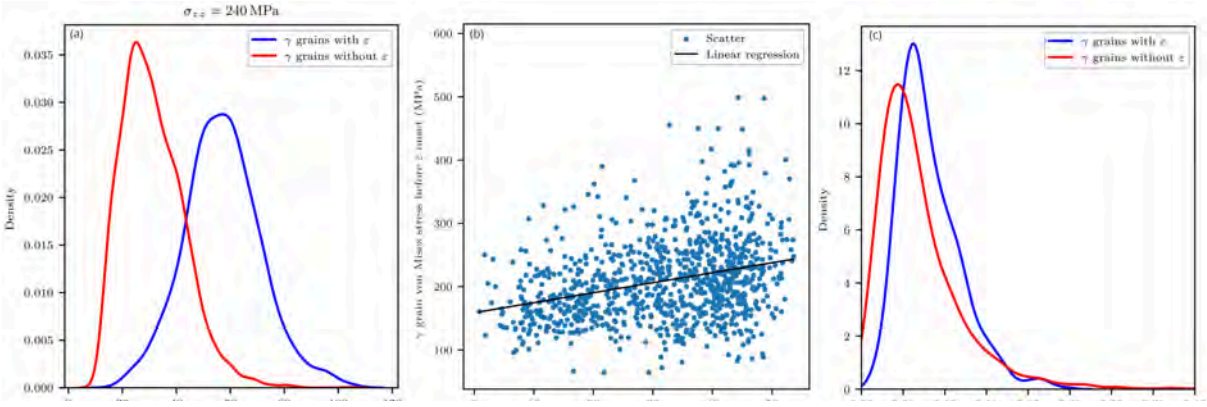


Figure 1: **(a)**: Grain sphere-equivalent diameter distributions for γ grains with and without detected ε formation, at an applied stress of 240 MPa. **(b)**: A scatter plot of (ε-forming) γ orientation vs grain-level γ von Mises stress at ε formation. **(c)**: Distributions of α/α' neighbourhood volume fraction for γ grains with and without detected ε formation.

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[b] Diamond Light Source Ltd., United Kingdom
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[d] WMG, University of Warwick, United Kingdom
[e] METAL group, INSA Lyon, France

[1] Kang et al., *Acta Mater.* **254**, 118985 (2023).
[2] Zhang et al., *Acta Mater.* **200**, 315 (2020).
[3] Hedström, Deformation induced martensitic transformation of metastable stainless steel AISI 301, Luleå, Sweden (2005).
[4] Hedström, Deformation and martensitic phase transformation in stainless steels, Luleå, Sweden (2007).

3DXRD operando study of the nucleation and growth dynamics of multiple Fe intermetallic phases of Al alloys in the solidification processes

K. Xiang[a], S. Huang[a], H. Song[a], K. Sungcad[a], H. Fang[b], J. Mi[a]

Maximizing the uses of millions tons of the recycled aluminum (Al) alloys currently circulating around the world is an essential step in achieving metal materials sustainability in a net-zero emission society in the near future. Recycled Al alloys contain multiple impurity elements and Fe is the most detrimental one, forming different brittle and irregularly-shaped Fe-rich intermetallics that damage mechanical properties. The nucleation and multiphase Fe-intermetallics evolution in recycled Al alloys (often having 4-8 elements) are very complex processes. Although many conventional ex-situ microstructural characterizations have been made to reveal the post-solidification structures, much less real-time and in-situ studies have been reported. Hence, the dynamic structure evolution from the onset of phase nucleation to a fully developed complex 3D phases have not been fully understood.

Very recently, we have conducted a systematic research at the ID11 of the ESRF and used the 3DXRD techniques to study in operando condition the nucleation and growth dynamics of the Fe-rich intermetallic phases in a number of multiple-component recycled Al alloys in the solidification processes. The 3DXRD technique is able to differentiate and quantify the different Fe intermetallics in 3D space, e.g., giving the unit cell and centre of mass of each crystal and their distribution in 3D space. The results provide very rich and systematic data that are complementary to the results we obtained previously using total scattering and tomography technique. It allows us to fully understand the complex and dynamic processes for multiple Fe phase nucleation and growth, providing essential theoretical guidance for future development of effective processing strategies for controlling and modifying the Fe-intermetallics for recycled Al alloys containing higher Fe content.

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Unveiling the Impact of Intragranular Strain Localization on the Functional Response of Technological Components: Insights from a DFXM Perspective

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Intragranular strain and stress concentrations significantly influence functional properties and lifetime of technological components made from polycrystalline materials. In this contribution, we present results from synchrotron dark field X-ray microscopy (DFXM) characterization of two sample systems.
In the first example[1], we used DFXM to characterize 20 µm thick Cu films, both in their as-deposited state and after 5×10^4 thermal cycles. The thermal cycling results in severe shear deformation of Cu grains and the emergence of microscopic voids, particularly at high-angle grain boundaries (HAGBs). DFXM provides experimental evidence that mosaicity of Cu grains, residual tensile and compressive elastic strain concentrations, and the full width at half maximum of Cu 111 reflections all increase simultaneously in the vicinity of the HAGBs (Fig. 1). This is interpreted as a result of vacancy condensation in front of the HAGBs after dislocations have moved across cycled grains and partly annihilated. Moreover, the observed HAGB decohesion and gradual void formation are correlated with the presumed hardening of regions near HAGBs, leading them to lose their ductility during cyclic elasto-plastic deformation. In general, DFXM identifies the inhomogeneous intragranular microstructural refinement and a gradual condensation of structural defects near HAGBs as driving forces for void formation in Cu metallizations during fast thermo-mechanical fatigue.
In the second example[2], we used DFXM to characterize strain and orientation gradients across ~20 µm large TiC and TiN particles embedded in a steel matrix. The results demonstrate gradual crystallographic orientation changes across the particles and the presence of sub-grains, in contrast to electron backscatter diffraction data. However, these gradients do not appear to be directly correlated with the observed strain gradients, which are mainly attributed to different strain states within the particles' interiors and envelopes.
In summary, the research conducted on industrially relevant materials has shown that DFXM provides valuable insights into the intragranular microstructure and stress state of polycrystalline materials, allowing for a further assessment of the structure-property relationship.

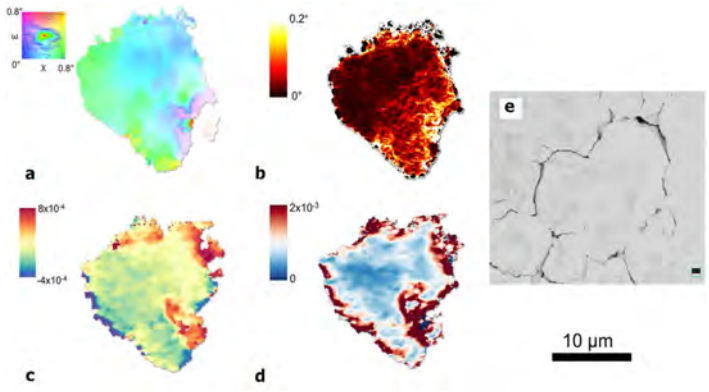

Figure 1: Maps reconstructed from DFXM data showing a Cu grain cycled 50,000 times. (a) depicts the mosaicity of the crystal lattice, in (b) the kernel-average misorientation indicates the diffuse variation of the diffraction vector, (c) shows the relative axial strain, corresponding to the distribution of 2nd order elastic strain and (d) presents the diffraction peak breadth, i.e. the concentration of lattice defects. A SEM micrograph of the same grain is shown in (e).[1]

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Revealing the Full Strain Tensor in microelectronic devices by Nanobeam Scanning X-ray Diffraction Microscopy

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Both historically and nowadays, there has been great interest in the study of lattice strains, e. g. for strain engineering in microelectronics. However, experimental access to the entire local strain tensor $\boldsymbol{\varepsilon}$ is challenging, especially in the study of small mechanical deformations acting over sub-micron length scales. This creates a need for experimental techniques probing all components of $\boldsymbol{\varepsilon}$ with microscopic resolution and high lattice sensitivity. In this work, we demonstrate that these requirements can be met by Scanning X-ray diffraction microscopy (SXDM) based on the latest methodical developments at beamline ID01/ESRF.

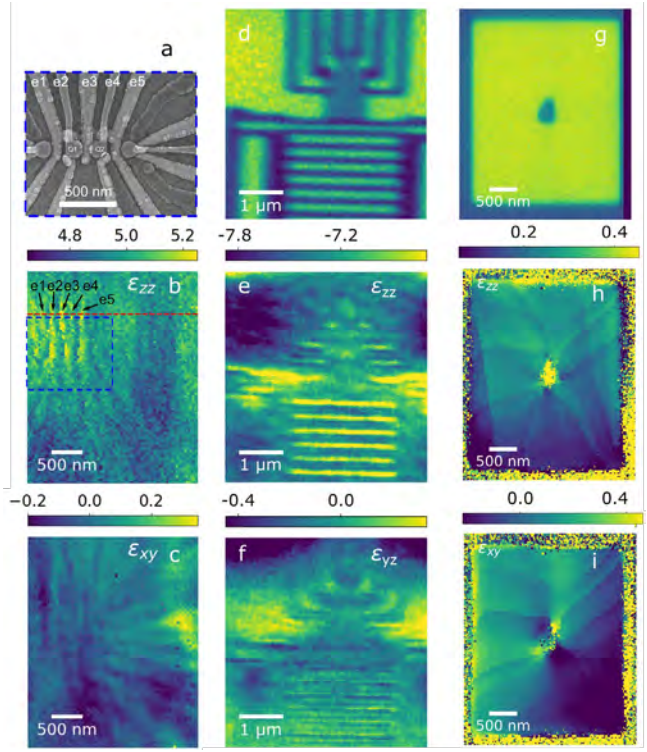

Figure Strain maps measured with SXDM: (a) SEM image of a functional hole spin qubit device[2], five Ti/Pd gate electrodes are labelled e1-e5, (b) map of symmetric strain ε_{zz} in the Ge QW, (c) shear strain ε_{xy}[3]. (d) X-ray Fluorescence (XRF) map of a QuBus test device with TiN electrodes[4], (e) map of symmetric strain ε_{zz} in the Si QW, (f) shear strain ε_{yz}[5]. (g) XRF map of a laterally grown $Si_{0.70}Ge_{0.30}$ VS[6], (j) map of symmetric strain ε_{zz}, (i) shear strain ε_{xy}. All strains are described by the colorbars in percent

SXDM employs a finely focused X-ray beam with spot size below 100 nm, created e. g. by a Fresnel Zone Plate. The sample is scanned continuously through the beam spot by a piezo stage, while the diffracted X-rays are recorded with an X-ray area detector. Combining datasets for several beam incidence angles yields a 3D reciprocal space map for every position. To probe all six lattice parameters and access the full strain tensor, such measurements are required for three non-coplanar Bragg reflections[1].

From the SXDM measurements, maps of all symmetric lattice strains ε_{ii} $(i \in \{x,y,z\})$, shear strains ε_{ij} and rotations w_{ij} are obtained. We present strain maps in different samples, including functional devices for quantum technology[2-5], and laterally grown $Ge/Si_{0.7}Ge_{0.3}$ virtual substrates[6]. For all examples, small modulations of lattice strain are observed in the 0.01 % – 0.1 % range, acting over length scales from a few 10 nm to a few µm. Depending on the sample, they are linked to the stressing action of metallic gate electrodes and structural defects in the epitaxial material. The experimentally determined strains are leveraged as input for Finite Element Method modelling and band structure calculations based on band perturbation potentials. Thus, due to the strains we predict spatial fluctuations of band edge levels of a few meV, similar to charging energies for single electron transistors.

The experimental results demonstrate small spatial resolution < 100 nm and fine lattice sensitivity < 0.01 % to all components of the strain tensor, including shear strain, in microelectronic heterostructures and devices Thus, we showcase the potential of SXDM for structural characterization in quantum- and microelectronics. Moreover, this method is model-free, non-destructive and universally applicable to single-crystal material systems, making it a promising new analysis tool for the field of strain engineering.

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High resolution reciprocal space mapping at the Swedish Materials Science beamline P21.2 at PETRA III

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The Swedish Materials Science beamline (SMS) on port P21.2 at the PETRA III synchrotron facility at DESY is a high energy beamline dedicated to in-situ materials characterization (bulk and surface) by a combination of WAXS/SAXS and imaging techniques. Sub-micrometer length scales are probed statistically by grain averaging WAXS and SAXS while real space imaging can cover micrometer to mm length scales. The available optics include an optional double channel cut high-resolution monochromator and long distance focussing enabling together with a large sample-to-detector distance efficient high resolution diffraction. The relevant beamline instrumentation will be presented and briefly discussed in view to the different modalities.

Case studies will concentrate on polycrystalline materials and include far- and near-field grain resolved diffraction. Emphasise will be put on the in-situ characterization of the deformation of metallic materials by high-resolution reciprocal space mapping (HRRSM). During deformation, typically characteristic dislocation structures form and evolve producing subgrains with very few dislocations separated by dislocation rich walls. HRRSM consists of recording 3D reciprocal space maps of individual grains. The technique is fast since only rotation around one axis is required and insensitive to minor sample displacements during deformation. The three-dimensional reciprocal space maps are typically analysed by azimuthal and radial projections. In suitable cases individual subgrains are observed and in general the radial profiles and azimuthal projections contain detailed information of the evolving dislocation structure. The technique will be outlined and a recent application to solid-state thermal cycling during additive manufacturing will be presented[1]. Individual grains in an austenitic stainless steel were followed during the deposition of additional layers. For the first time, direct evidence of the evolution of the dislocation structure during the layer deposition was obtained.

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High-resolution spatio-temporal strain imaging reveals loss mechanisms in surface acoustic wave device

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Surface acoustic wave (SAW) devices are key components for processing radio frequency signals in wireless communication, known for their high performance, compact size and low cost. A quantitative understanding of energy conversion and loss mechanisms is essential to improving device structures and materials. Using the 100 ps bunch length of The European Synchrotron ESRF, our stroboscopic full-field diffraction x-ray microscopy (s-FFDXM) studies of a prototypical one-port resonator device reveals multiple sources of acoustic loss. The exceptional strain sensitivity allows measurement of acoustic waves with picometer-scale amplitude. A difference in the apparent resonant frequencies observed in the x-ray and electrical measurements results from the spatial non-uniformity of the acoustic excitation and from substantial leakage out of the active area. The high-resolution spatiotemporal imaging demonstrated in this work is generally suited for studying nano-phononics, specifically when the feature size is smaller than the optical wavelength. Applying s-FFDXM and an associated wave decomposition analysis method in combination, we achieve high-resolution spatio-temporal imaging of the acoustically induced strain waves. The method has a strain sensitivity of ~ 10^{-7}, corresponding to a surface displacement on the order of 1 pm. Measurements on a prototypical one-port SAW resonator reveal multiple mechanisms of acoustic loss with distinct spatial and time dependencies. Specifically, the maximum acoustic energy in the cavity area was not observed at the intended resonance frequency despite a well-matched IDT period and a higher reflector efficiency. A substantial amount of acoustic energy was leaked to the side (22%) and through the reflector (13%), leading to a shift in the resonance frequency of –9 MHz.

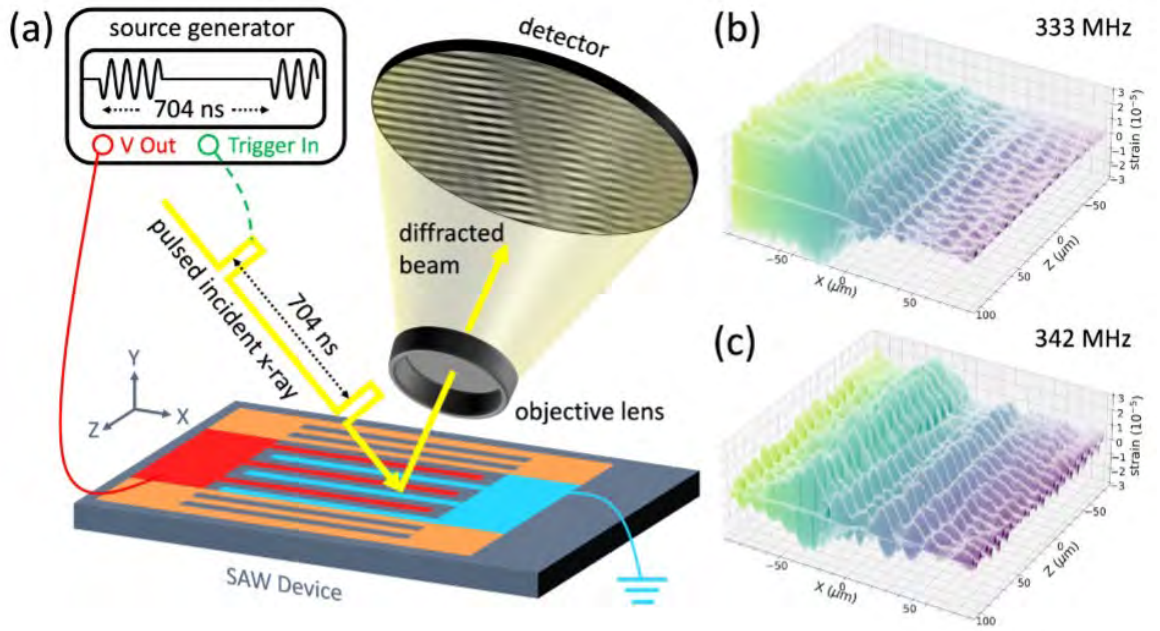

Figure 1: Stroboscopic full-field diffraction x-ray microscopy. (a) The SAW excitation was electronically synchronized to the x-ray pulses with a tunable delay. A dark-field image is formed on a two-dimensional x-ray detector by projecting a magnified image of the diffracted beam using an x-ray objective lens. Analysis of the dark field images acquired during a radial scan allows visualization of the strain wave, as shown in (b) and (c). The reflector gratings and the two IDT electrodes appear in orange, red, and blue, respectively.

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New insights into hierarchical microstructure formation during green ironmaking with hydrogen

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Abstract

The reduction of iron ores with carbon, such as coal and coke, generates 7-8% of the global CO2 emissions [1]. A paradigm shift from fossil-fuel-based to green-hydrogen-based ironmaking is urgently needed to address the decarbonization challenge in this sector and combat global warming. Hydrogen-based direct reduction of iron ores is one of the most promising solutions in this context when hydrogen is produced using renewable energy [2]. The byproduct of this reduction reaction is water. Hydrogen-based direct reduction is a multistep solid-gas reaction where the solid undergoes several non-volume conserving phase transformations and iron ores are gradually reduced into iron [2]. At the microscopic scale, the reduction process is associated with multistep solid-gas reactions and phase transformations, mass transport through heterogeneous media, and intense mechanical interaction among the phases [3-5]. This leads to the evolution of a complex defect cosmos, including vacancies, dislocations, interfaces, and free surfaces (cracks, pores), all with different transport features. These defects act as reaction, nucleation, and diffusion sites, shaping the overall reduction kinetics. This talk showcases the hierarchical microstructure formation during the hydrogen-based direct reduction and reveals its salient roles in the reduction kinetics. Particularly, the application of in-situ dark-field X-ray microscopy and nanotomography is highlighted to better characterize the dynamic evolution of the microstructural defects and understand the underlying reaction mechanisms.

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Three-dimensional stress localizations and grain yielding under elastoplastic axial-torsional loading

Jerard V. Gordon[1], Yaozhong Zhang[1]

Non-proportional (NP) multiaxial strain paths are well-known to contribute to fatigue life reduction in structural metals due to additional strain hardening mechanisms. However, our understanding of crystal-scale plastic deformation under NP loading is severely limited due to inherent difficulties of employing traditional specimen/load stage geometries within current SEM environments. We present two recent studies in leveraging *in situ* high energy diffraction microscopy (HEDM) and companion crystal plasticity finite element (CPFE) modelling that evaluate the development of stress localizations and grain yielding under NP axial-torsional loading. First, important stress metrics for ~ 300 grains are compared under two different loading conditions: (1) Torsion-dominated loading (low NP) and (2) Tension-torsion loading (high NP) in equiatomic NiCoCr, a representative multicomponent face-centered cubic (FCC) superalloy. Overall, significant stress localizations were discovered leading to a complex interplay between axial and shear stress components (e.g., stress coupling). This resulted in grain yielding near the sample surface largely driven by shear stress, whereas internal grain yielding was largely accommodated by axial stress. Grain-resolved stress localization trends were well-captured by the CPFE model, although some discrepancies in magnitude occurred, particularly for volumetric stress metrics due to initial type II residual stress distributions. Second, preliminary HEDM data and companion CPFE simulations are presented to better understand the role of microstructural features on plastic strain accumulation under combined axial-torsional cyclic loading. Interestingly, features such grain size and initial orientation play negligible roles in strain accumulation, while slip system-based metrics including maximum resolved shear stress strongly correlate with grain-resolved accumulated strain. This highlights that slip-system level behaviors play a major role in overall plastic deformation under NP loading; however, this information is largely unobtainable at relevant length and time scales. Thus, desired potential extensions to current synchrotron x-ray loading environments at user facilities are also presented that could provide further insight and construct a more detailed understanding on crystal-scale plastic deformation under multiaxial loading.

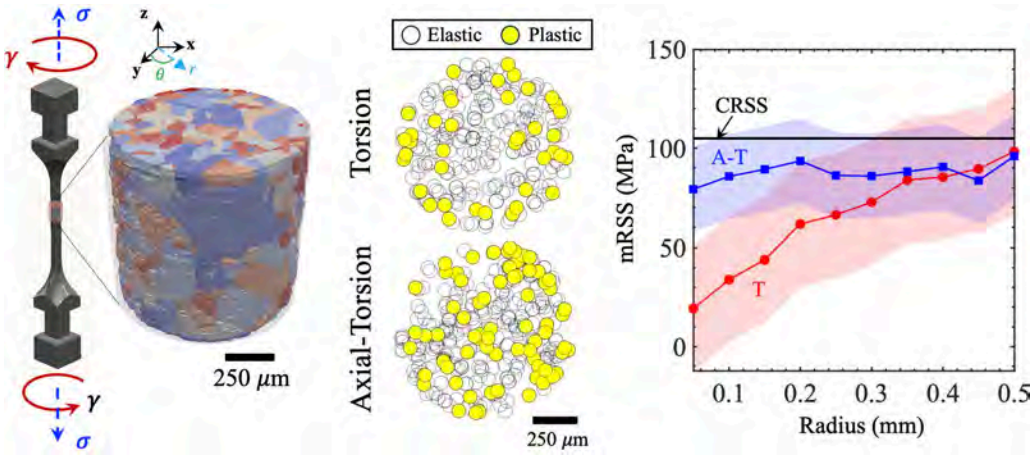

Figure: Investigating crystal-scale plastic deformation under NP loading via *in situ* HEDM and companion CPFE

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Improvements in 6D Reconstruction for Diffraction Contrast Tomography and Topo-tomography

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Diffraction contrast tomography (DCT)[1] is a synchrotron-based full-field X-ray imaging technique providing non-destructive observations of the 3D grain microstructure in polycrystalline materials. The lattice orientations and the shapes of the grains are reconstructed from the diffraction spots collected from near-field diffraction images. Topo-tomography (TT)[2] is a variant of 3D diffraction imaging, which can be used to scan individual grains of interest with improved spatial resolution. The dislocation structures give rise to lattice distortions and rotations, which can be visualized in the diffraction spots. The 6D grain reconstruction algorithm[3] is used to reconstruct intragranular lattice orientations and grain shape from the diffraction spots. Some crystal microstructures like slip bands can be observed in intragranular orientation reconstructions of the polycrystalline materials like Ti7Al. Therefore, we propose some methods to improve the 6D reconstruction, in order to obtain better observations on intragranular orientations.

Poisson noise in detector pixels and the errors in the model cause artifacts in the reconstruction. The weighted L2 cost function is introduced into 6D reconstruction to suppress the artifacts. The experimental results and the simulation results show that the weighted L2 cost function noticeably improves the reconstruction quality.

The joint reconstruction of DCT and TT scans with different parameters is investigated by simulation. The simulation results show that joint use of TT scans with different scattering vectors significantly improves the reconstructions of intragranular orientations. The application of identical crystal plane for TT scans facilitates the identical sample tilts and shifts, making subsequent scans easy to perform after the initial one. For joint use of 2 TT scans with the identical plane, optimal performance is attained by employing opposite scattering vectors. As utilizing a single crystal plane is limited to reconstructing plane normal directions (2D orientation space), joint reconstruction of TT scans involving different crystal planes enables the reconstruction of 3D lattice rotations. The 5D reconstruction from a single TT scan is underdetermined. Using a range of detector distances can improve it. Using line beam enables a complete reconstruction of 5D space.

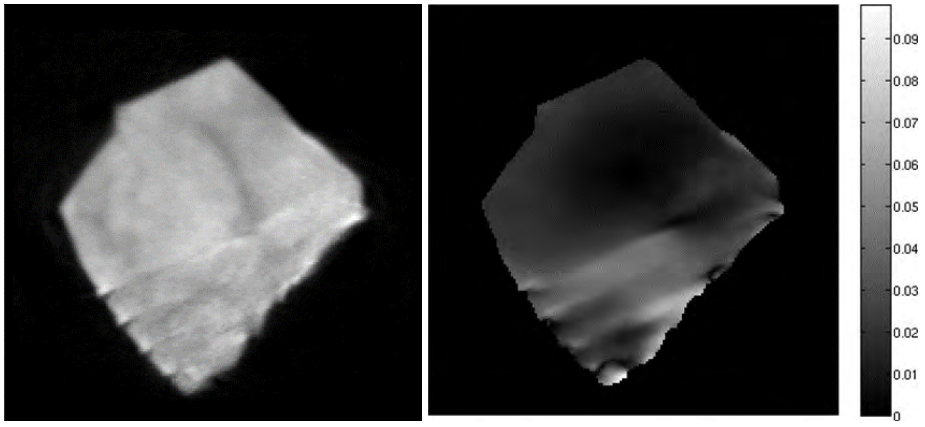

Figure: Volume intensity and intragranular misorientation (in degree) of Ti7Al grain reconstructed from TT scan.

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Correlative Diffraction Microscopy Imaging experiments to investigate crystal plasticity

H. Proudhon

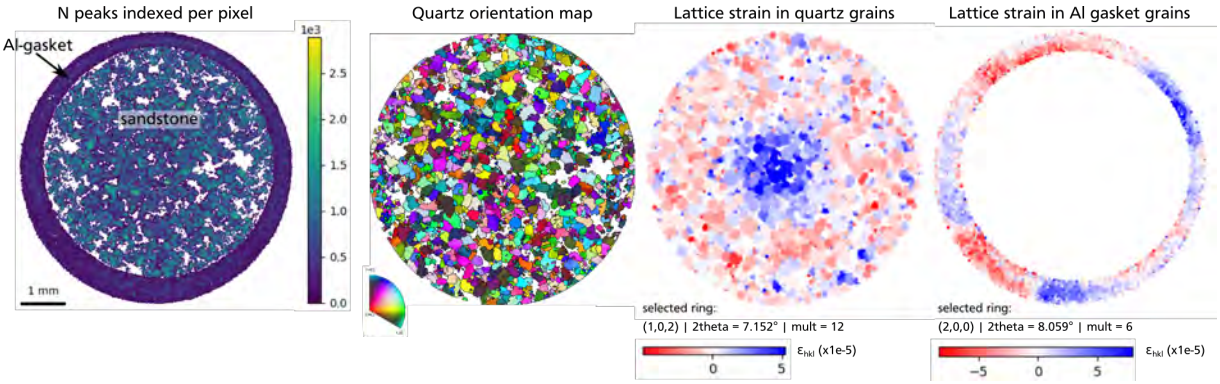
Key mechanical properties are governed by the three dimensional arrangement of the material microstructure. Synchrotron (and to some extent laboratory) X-ray based characterization methods now allows for non destructive analysis and in situ multi-modal, correlative measurements in microstructures containing thousands of grains. This has the potential to revolutionize material characterization, deformation and fracture mechanisms investigations, and constitutive behaviour identification at the grain scale.

This presentation will showcase results from several studies which bring new insight on crystal plasticity of structural metallic materials. Different characterization modalities have been combined on the same sample such as EBSD, HR-DIC, DCT and Topotomography at different stage of tensile deformation, sometimes in situ, sometimes ex situ. Slip band formations and build up of lattice rotation fields will be looked in detail. In all cases, measuring the same grain with different modalities is of great help to bring new insight on bulk crystal plasticity mechanisms. Finally, an outlook on the potential of correlative experiments with mesoscale mechanical simulation methods such as FFT, FEM and DDD will be given.

In-situ investigation of elastic strain and stress during brittle rock failure using scanning 3D X-Ray Diffraction

J-B. Jacob[1], F. Renard[1], B. Cordonnier[2], J. Wright[2]

Understanding the mecanisms that control brittle rock failure is a fundamental challenge in Geosciences, with implications for a large range of topics such as earthquake mechanics, structural integrity of tunnels and boreholes, or the evolution of fracture networks in geological reservoirs. Recent advances in triaxial compression experiments, dynamic shock experiments, and dynamic X-ray microtomography imaging have provided unparalleled insights into the i*n-situ* evolution of strain within deforming rocks subjected to quasi-static or dynamic loading. However, these experiments do not provide direct information on the heterogeneous internal stress state of the rock, which is crucial to predict how and where microfractures will nucleate and propagate, eventually resulting in macroscopic failure. In addition, rocks submitted to shocks, *e.g.* damaged during an earthquake, may have experienced transient stresses up to GPa-level, which could be partially preserved as residual strains in the damaged minerals. We used synchrotron scanning 3D X-ray diffraction (s3DXRD), in order to investigate the internal elastic strain field (and then deduce the stress field as well) of deforming rocks and track residual strains resulting from shock-wave propagation during dynamic rupture. We investigated the evolution of internal stress distribution in 5mm-diameter cores of sandstone during triaxial compression. We also investigated rock samples recovered after shock experiments that led to partial pulverization. Processing these data is challenging due to the large size and complex mineralogy of these natural samples. We developed a method based on identification of Friedel pairs in symmetrical s3DXRD scans, which allows to index mineral phases and crystallographic orientations on a pixel-by-pixel basis. Local stress is inferred from elastic strain measured on each grain. A first approach of strain analysis based on comparison between the fitted unit cell and a reference cell for each indexed grain yields a large spread across the sample up to 10^{-2} strain units, which seems incompatible with pure elastic strain and probably arise from pre-existing plastic defects in the grains. However, strains $\Delta d/d_0$ measured on individual diffraction rings show a much lower spread (ca. $\pm 10^{-4}$) and reveal spatial heterogeneities which we relate to internal stress variations. These results are among the first *in-situ* mapping of elastic strain in natural rock samples, and demonstrate the great potential of s3DXRD to investigate strain and stress in geological materials.



Processed s3DXRD data for a sample of sandstone recovered from a shock experiment. From left to right: Number of peaks indexed per pixel, mean grain orientation (quartz), residual lattice strain on selected Debye-Sherrer rings for quartz grains and for Al grains from the gasket surrounding the sample.

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Resolving intragranular orientation and stress fields in plastically deformed titanium using scanning 3D X-ray diffraction

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The response of a polycrystalline material to a mechanical load depends not only on the response of each individual grain, but also on the interaction between each grain and its neighboring grains. These interactions lead to local, intragranular stress concentrations that often dictate the initiation of plastic deformation such as slip and deformation twinning. Yet, few experimental studies have quantified intragranular crystallographic orientation and stress evolution coupling with plastic events across bulk, 3D volumes. In this presentation, we use in-situ scanning 3D X-ray diffraction at the ESRF (ID11) and the APS (1-ID-E)[1] to investigate intragranular plastic deformation in polycrystalline titanium. The results reveal the heterogenous stress distributions inside individual grains and across grain boundaries during deformation. Intragranular measurements of crystallographic orientation and the elastic strain tensor provide insight into the local stress evolution, which can then be related to local plastic deformation events. This work demonstrates the potential for understanding the local elastic and plastic deformation associated with networks of grains in polycrystalline for informing mesoscale phase field and crystal plasticity models. Finally, we will also briefly discuss current limitations and future advancements to scanning 3D X-ray diffraction experiments and data analysis approaches.

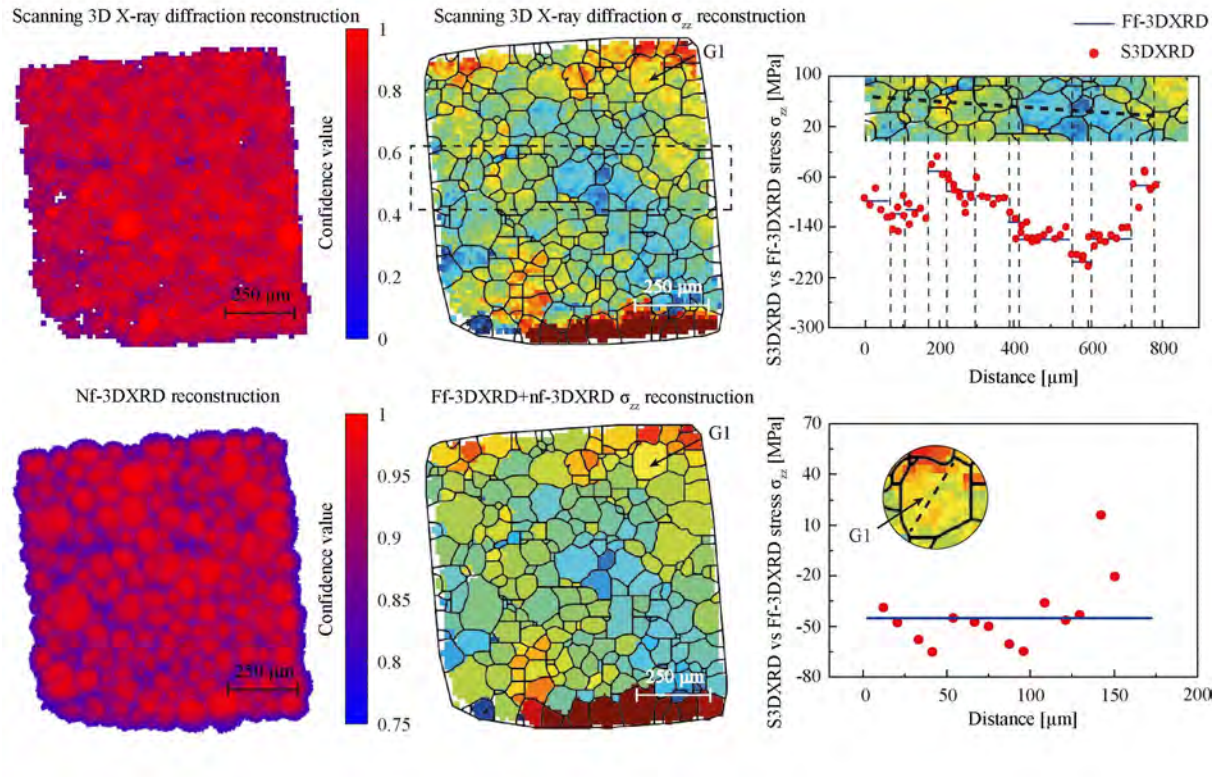

Figure: Comparison of scanning 3D X-ray diffraction versus far-field and near-field 3D X-ray diffraction reconstruction results.

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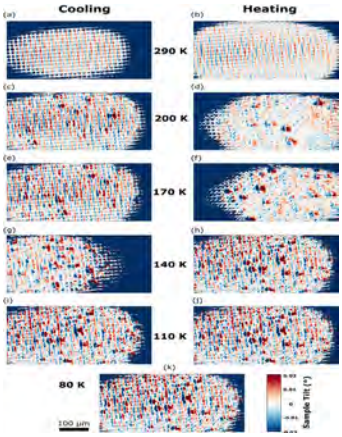
[1] Li et al. *Journal of Materials Research* **38**, 165 (2023).

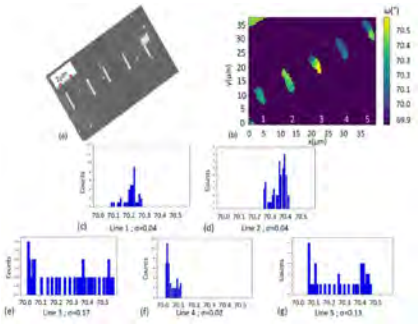
On the opportunities offered by X-ray microscopies for the advanced characterization of microelectronic components

P. Gergaud[1]

Among the X-ray microscopies techniques offered by the most advanced synchrotron facilities such as 3D X-ray Diffraction, Scanning X-ray Diffraction Microscopy or Diffraction Contrast Tomography, the Dark Field X-ray Microscopy (DFXM) is non-destructive, very well suited to simultaneous multiscale characterization or in-situ measurements and can achieve a direct spatial resolutions of 30-100 nm and a time resolution of 100ms. If the application of this technique have been largely demonstrated for metallurgical studies, fuel cells as well as bio minerals, it is only scarcely performed on microelectronics devices. It is, however, a particularly interesting technique for microelectronic objects because it makes it possible to observe the individual behaviour of a large number of objects, which can range from several hundred to several thousand of them. By this way, it is therefore possible to detect phenomena of defectiveness or random growth. Among the other particularities of this technique, there is its ability to provide three-dimensional (3D) mapping of strain and orientations in a crystalline materials at a nano scale and to generate a real space image of the illuminated volume. Within this talk, I will illustrate through 2 examples the advantage of DFXM for the development of microelectronic devices:



1/ The first example concern hybridized focal plane array HgCdTe (MCT) sensors which are the workhorse of high performance infrared detectors covering a broad range of applications from space investigation to gas monitoring. Despite the improvements in the performance of these sensors in the last decades, device failure due to the lattice and thermal expansion mismatches between MCT and the Si readout circuit still affects the overall MCT detector performance. In this work, we use in-situ DFXM to map the structural variations of a fully operable MCT sensor at temperatures down to 80 K. We report, for the first time, on the nanoscale structural evolution over a large population of photodiodes at operating temperatures with high spatial and angular resolution.



2/ the second example is focused on growth and coalescence of GaN nanodots. GaN is an extremely attractive semiconductor for optoelectronics applications. At CEA-LETI we develop a new growth methodology based on epitaxial structures of gallium nitride (GaN) on top of GaN/AlN/Si/SiO2 nano-pillars. The nano-pillars are intended to allow independent GaN nanostructures to coalesce into a highly-oriented film due to the SiO2 layer becoming soft at GaN growth temperature. By this approach, lower dislocations densities are expected. Thanks to DFXM, we show that extremely well oriented lines of GaN (standard deviation of 0.04°) as well as highly oriented material for zones up to 10 x 10 µm2 in area are achieved with this growth approach.

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Evidence of Strain localization near high angle grain boundaries in CdTe solar cell using Scanning 3D X-ray diffraction microscopy

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Cadmium Telluride (CdTe) solar cells have emerged as a promising candidate to help boost green energy production. Impurity and structural defects are the major barriers to further improving solar power conversion efficiency. Grain boundaries often act as aggregation sites for these impurities and hence show strain localization in these areas of high diffusion[1]. In this study, we demonstrate the use of Scanning 3D X-ray diffraction microscopy[2] to non-destructively make 3D maps of the grains – their phase, orientation, and local strain[3] within a CdTe solar cell absorber layer with a resolution of 100 nm. We quantify twin boundaries and suggest how they affect grain size distribution and orientation distribution. Local strain analysis reveals that strain is primarily associated with high misorientation grain boundaries, whereas twin boundaries do not show high values of strain. We also observe that high-strain grain boundaries form a connected region in space from the CdS substrate. Hence, this high strain is believed to be associated with the diffusion of sulfur along grain boundaries, corroborating previous work [1]. The method and analysis demonstrated in this work can be applied to different polycrystalline materials where the characterization of grain boundary properties is essential to understanding the microstructural phenomena.

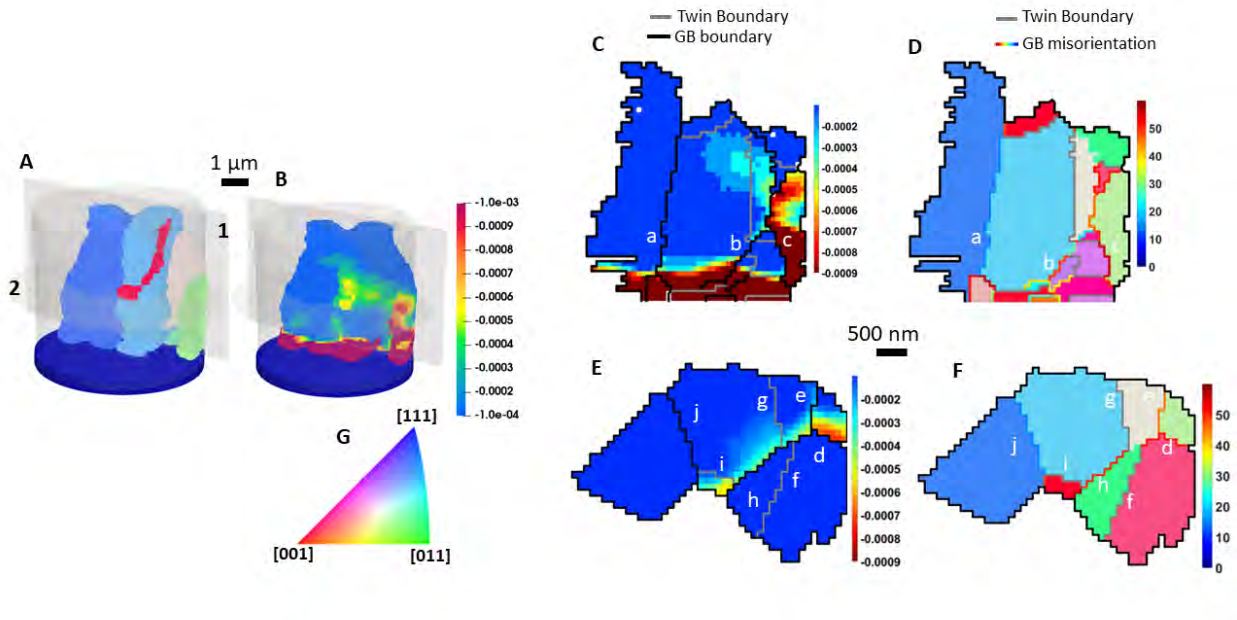

Fig1. A) and B) are 3D visualizations of grain orientation and strain, respectively, within the CdTe p-type layer in a selected region where high strain is observed. The transparent grey cylinder is the sample volume studied; the dark blue disk is the CdS layer. In A, the colour scale is the IPF colour code for cubic symmetry. In B, the colour scale is saturated near the CdS layer to emphasize the weak strain in the bulk C) Vertical cross-section of B) along plane 1 showing strain with annotation of boundary types. D) Grain orientations in the same plane as C) with annotation of grain boundary character. E) and F) Similar cross-sections now along the horizontal plane 2 in A. Grain boundaries are marked with lowercase letters. We observe that high misorientation angle grain boundaries c, d and h allow localization of strain whereas twin boundaries coloured in grey do not show localization of strain.

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[1] I. Calvo-Almazán et al. *IEEE Journal of Photovoltaics*, vol. **9**, no. 6, pp. 1790-1799 (2019)
[2] H.F Poulsen et al. *J. Appl. Cryst.* **45**, 1084-1097 (2012)
[3] A. Henningsson et al. *J. Appl. Cryst.* **54**, 1057-1070 (2021)

In situ observation of plastic relaxation dynamics in $In_xGa_{1-x}N$ thin films by full-field X-ray diffraction microscopy

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The prospect of implementing white light sources based on the monolithic integration of three-color light emitting diodes within one material family has motivated an enormous amount of research. The $In_xGa_{1-x}N$ system appears as the most promising candidate for this purpose, since its optical emission can, theoretically, be tuned to cover the whole visible range by adjusting the indium content. However, increasing the indium-content above a level of 30 % is a prevailing challenge. One particular obstacle here is the relatively large lattice mismatch of 10% between InN and GaN, which leads to elastic stress and the formation of defects in the active region when the indium content is increased. The growth of relaxed InGaN buffer layers as virtual substrate for the subsequent growth of higher indium-content quantum wells (QW) thus appears as a promising approach to realize a better QW material quality[1,2].

The dominant mechanism of plastic strain relaxation in extended 2D layers of (0001) oriented wurtzite films is the nucleation and glide of (a+c) dislocations[3]. Due to their large Burgers vector such dislocations are expected to exhibit high formation energies and are typically dissociated into partials leading to a complex glide behavior slowing down the relaxation process. In this work, we examine the glide dynamics of such (a+c)-type dislocations in coherently strained (In,Ga)N layers on GaN in real-time by in-situ full-field X-ray diffraction microscopy (FFXDM). The plastic relaxation was activated by annealing in a temperature range between 420 °C and 480 °C. FFXDM measurements have been performed at beamline ID01/ESRF in a grazing-incidence geometry enabled by fine-tuning the energy of the X-rays (see Fig. 1). Using a condenser lens for focusing the incident beam, the footprint on the sample surface has been matched with the field of view of the FFXDM measurement. This way, a time resolution of ≈ 2 s could be achieved for the in-situ experiment.

Fig. 2 shows a zoomed in snapshot of an image sequence, recorded at a temperature of 420 °C. Misfit dislocations (MDs) are visible in bright contrast. Next to an irregular stop-and-go behavior, we notice an apparent change of the glide direction for this MD segment. As each line direction belongs to a different set of Burgers vectors, which was confirmed via TEM, we can exclude cross-slip as origin, suggesting a different mechanism based on a dislocation reaction. Tracking over 100 MDs in the same way as in Fig. 2 within the entire temperature range, allows a statistical analysis of glide velocities and the rate of glide events, allowing to quantify a minimal glide activation energy of around 1.0 ± 0.2eV. Furthermore, we observed a decreasing rate of glide events with time for each annealing temperature, which suggests a saturation of the relaxation process. These dynamics can be explained by the formation of jogs occurring during the glide process. As each time a MD blocks itself, the energy required for reactivation can vary, thus the reactivation energy increases statistically over time.

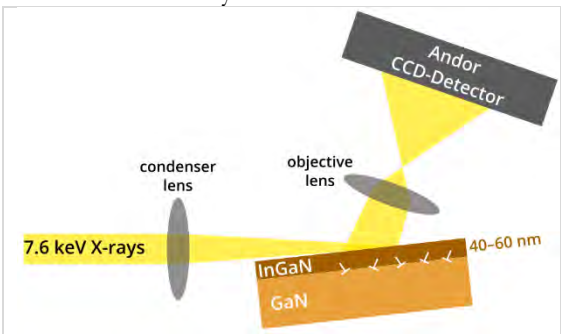

Fig. 1: FFXDM measurement in grazing-incidence geometry as seen from top.

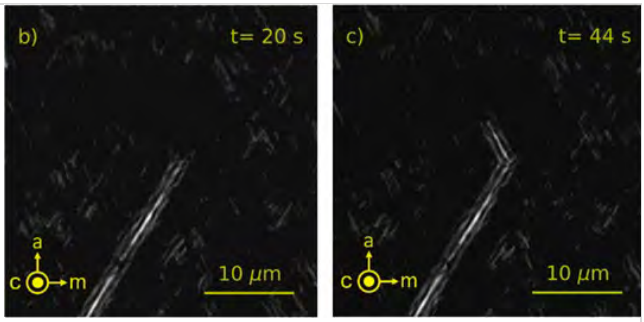

Fig. 2: Snapshots of an individual (a+c) dislocation at the interface of an InGaN layer on GaN during thermal annealing at 420°C.

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The Neper/FEPX Project and its Application to 4D Experiment/Simulation Studies

R. Quey[1], M. Kasemer[2]

Neper[1,3] and FEPX[2,3] are two complementary free/open-source programs for the study of polycrystal deformation. The programs can be used to solve a wide range of scientific problems in metallurgy (and other fields, such as geology), while offering methods specifically dedicated to the (large) plastic deformation of metals, particularly in relation to experiments based on synchrotron X-ray diffraction. Both programs cover all stages of a typical polycrystal plasticity study.

Neper first generates "digital samples" of a material from experimental data, which can be either statistical (grain size distribution, ODF, etc.) or grain-scale (as provided by 3DXRD or DCT experiments), and meshes them into finite elements. FEPX is a massively parallel crystal-plasticity finite-element program, which solves the local deformation of the polycrystal under prescribed loading. Typical results of a simulation include the local stress, strain and related variables (slip rate, etc.), as well as the microstructure evolution (lattice rotation, hardening, etc.). Finally, Neper offers post-processing and visualization capabilities for polycrystal deformation results, whether coming from the original experiment or the simulation, which greatly facilitates comparative studies.

Using both programs, the (large) deformation of 1000+-grain polycrystal can be simulated (and analysed) routinely. Several examples of applications, including a recently-published work carried out at ID11[4] (see Figure 1), will be presented.

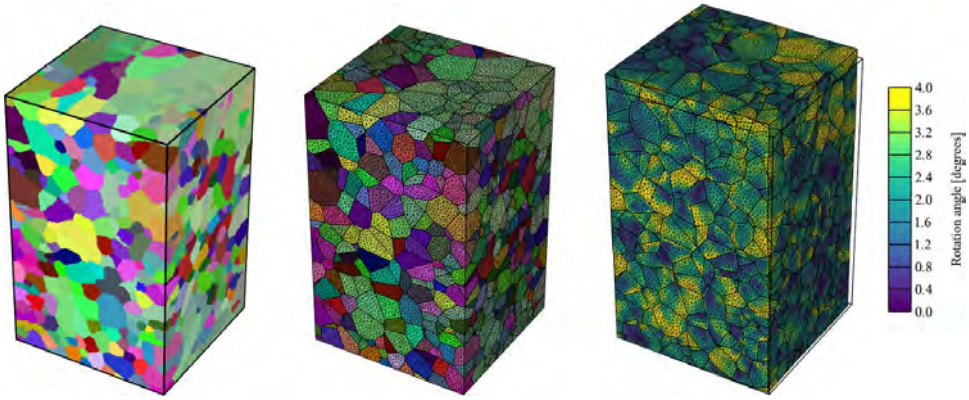

Figure 1: Deformation of an Al-Mn polycrystal[4]. (a) DCT sample, (b) finite-element mesh obtained with Neper, and (c) rotation angle at 4.5% strain simulated with FEPX.

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[2] Department of Mechanical Engineering, University of Alabama, Tuscaloosa, AL 35487 United States

[1] https://neper.info

[2] https://fepx.info

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Advances in Dark-Field X-ray Microscopy Simulations for Comprehensive Dislocation Analysis

S. Borgi[1], G. Winther[2] and H. F. Poulsen[1]

This presentation explores the current state of forward modelling Dark-Field X-ray Microscopy (DFXM) images using geometrical optics.[1] The focus will be on modelling individual dislocations in an Aluminum single crystal, offering a visual exploration of dislocation patterning and demonstrating the tool's strength for potential beam time users. The simulations provide insights into sample expectations, and the possibility of guiding experimental design when utilizing DFXM. Results on contrast mechanisms will be discussed, covering rocking/rolling curves, beam height configuration effects, pink beam contrast for dislocations (Figure 1), and contrasts with varying dislocation densities.[2] Additionally, I'll discuss transitioning from images to the micro-mechanical model of dislocations using center-of-mass (COM) maps. Hereby also the restrictions of this transition with experimental data, with the introduced artifacts/effects by the strong beam condition.

The work aims to enhance the understanding of dislocation patterning and dynamics by integrating discrete dislocation dynamics (DDD) modelling with geometrical optics.[3] As the DDD modelling will be able to give realistic behavior of dislocations during deformation, the vision of this combination will be to directly compare a 3D volume of a simulated sample, directly to a 3D volume of an experimental sample. Depending on the progress, I will report on the direct comparison of individual dislocations in an experimental sample and from the forward modelled images.

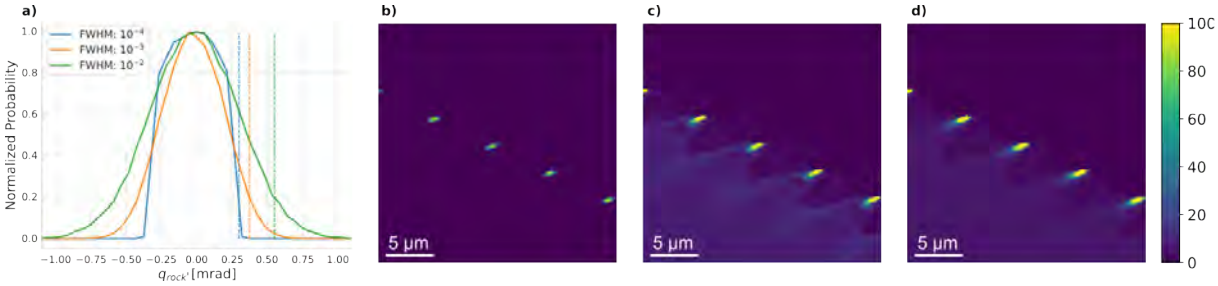

Figure 1: a) Normalised distributions of the reciprocal space resolution function in direction qrock' at different energy band widths (FWHM). b)-d) Forward modelled DFXM images in the weak beam condition with dislocations in a straight edge wall. b) energy bandwidth of $1.41 * 10^{-4}$, corresponding to the usual DFXM setup, at 300 µrad in the rocking curve, blue dashed line in a). c) energy bandwidth of $1.41 * 10^{-3}$, corresponding to the DFXM setup at an XFEL, at 375 µrad in the rocking curve, orange dashed line in a). d) bandwidth of magnitude $1.41 * 10^{-2}$, corresponding to the bandwidth from the pink beam setup, at 550 µrad in the rocking curve, green dashed line in a). The DFXM images have the same exposure time and same color scale for direct comparison, with max intensities of 100, 815 and 1806 in b-d), respectively.

If time allows it, the perspective of this tool will be explored in terms of utilizing forward modelled images in machine learning (ML) models to create training datasets for supervised learning. The ML models aim will be to detect the location of the dislocation core, identify the Burgers vector of these dislocations, and connect them in 3D from the forward modelled images that act like 2D slices of this 3D volume.

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[1] Poulsen et al., *J. Appl. Cryst.* **50**, 1441 (2021).
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Full-field crystal plasticity and phase-field modelling of elasto-plastic damage behavior of ice

Soroush Motahari[1], Chuanlai Liu[1], Mohammad Khorrami[1] and Dierk Raabe[1]

The overarching objective of the present work is to examine the brittle characteristics of a polycrystalline ice aggregate in order to improve our understanding of the macroscopic large-scale formation of crevasses in Greenland and Antarctica. We predominantly utilize the full-field crystal plasticity approach to determine the plastic behavior of polycrystalline ice. In addition, the plastic constants of the phenomenological crystal plasticity model are found using the Bayesian parameter estimation technique. To achieve this, the results obtained from the creep deformation of polycrystalline ice are subjected to different uniaxial compression and different strain rates are compared with the laboratory data. To monitor the initiation and crack promotion within the representative volume element, the obstacle damage phase-field method is integrated with the implemented crystal plasticity model. Considering that hexagonal open-packed ice is the predominant form of ice on Earth, we regard this phase as a suitable medium to study ice. The findings suggest that, at low strain rates, incorporating the viscoplastic behavior of ice into the analysis reveals a discernible manifestation of the brittle to ductile transition within the simulations. Moreover, the integration of viscoplastic behavior into the model substantially advances the initiation of crack nucleation. This can be attributed to stress concentration between grains, arising from the contrast in plastic deformation among the grains. Additionally, the results indicate that the development of texture under shear in ice contributes to an increased brittleness of polycrystalline ice.

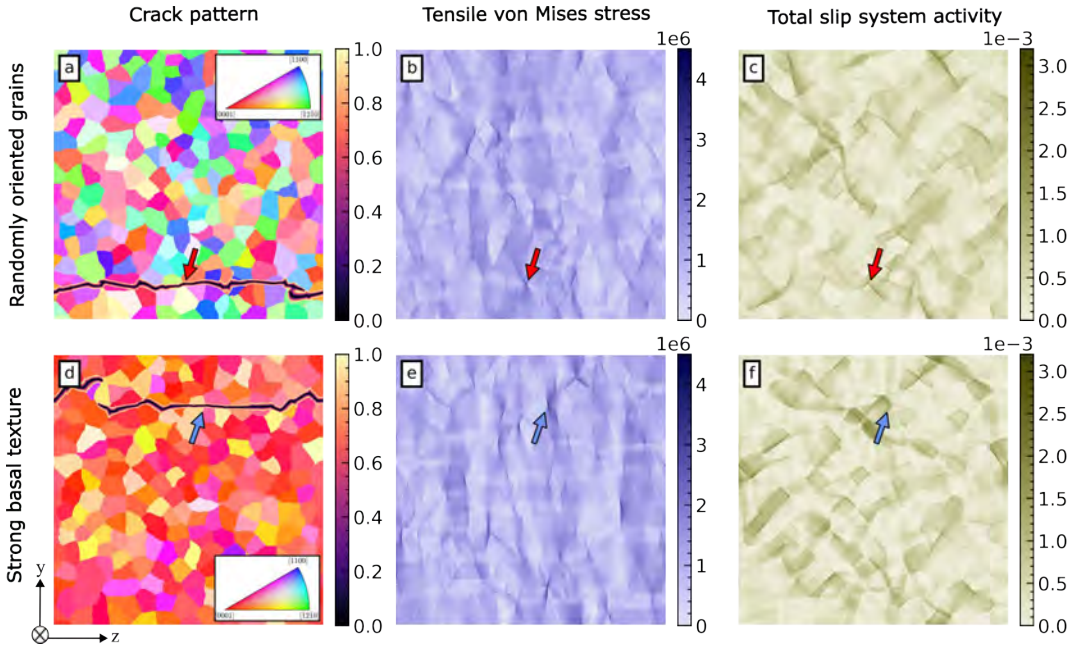

Figure: The crack propagation pattern for RVEs with random and strong basal texture is shown in (a) and (d) respectively. The color of grains corresponds to the crystallographic orientation that is aligned with the z-axis [001]. The tensile von Mises distribution before damage promotion reveals lower stress concentration with no texture (b) compared to the textured model (e). The total activity of different slips denotes the amount of plastic deformation for each grain that can be seen in (c) and (f). The small arrows pinpoint the crack nucleation spot.

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The evolution of grain scale stresses: 3D-XRD vs CPFE

Hamidreza Abdolvand[1]

Zirconium alloys are widely used to manufacture critical components of nuclear reactors. These alloys comprise of α-zirconium with hexagonal close packed (HCP) crystals. Similar to zirconium, the primary phase of magnesium and titanium alloys generally have HCP crystals. Under mechanical loads, plastic deformation in such alloys takes place by slip and twinning. Understanding the micromechanics of slip and twinning is important for developing predictive models focusing on the nucleation of mesoscale cracks. In this presentation, the use of three-dimensional synchrotron X-ray diffraction (3D-XRD) technique for characterizing grain-scale stresses in Zr, Ti and Mg alloys will be discussed. Attention will be given to the linking of the results with mesoscale modelling techniques such as crystals plasticity finite element (CPFE). Examples will be provided for how to use the measured data for improving materials constitutive equations.

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Pushing the Deformation Limit of the ID11 Scanning Three Dimensional X-ray Diffraction Microscope

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J. P. Wright[c], W. Ludwig[c], H. F. Poulsen[b], G. Winther[d]

Mapping the 3D evolution of texture and strains/stresses in the bulk of metals at elevated degrees of plastic deformation has provided a long-standing challenge for the X-ray diffraction microscopy community. Using Scanning Three Dimensional X-ray Diffraction Microscopy (S3DXRD) we demonstrate the reconstruction of grain orientation and strain maps for a 0.6mm thick Aluminium sample subject to a macroscopic tensile strain of >50%. These results were obtained with a standard pencil-beam S3DXRD acquisition, without conical slits[1,2], and a series of algorithmic upgrades to established diffraction data analysis methods[3,4]. We leverage the dependence of the diffraction model on the spatial positions of the voxels in the sample to reconstruct a primary, multichannel, orientation map, in which each voxel is associated to a set of possible crystal orientations. We then refine all orientation channels in the grain map using the full diffraction data set. Finally, to reduce the multi-channel grain map to a mono-channel grain map, we introduce an orientation filter that capitalise on spatial correlations within the sample. The reconstructed grain map, shown in Figure 1, revealed revealed intra-grain orientation gradients in the order of 5-10°. Our results mark a milestone for the ID11 S3DXRD probe in terms of enabling the study of intra-grain deformation mechanics at industrially-relevant degrees of plastic deformation.

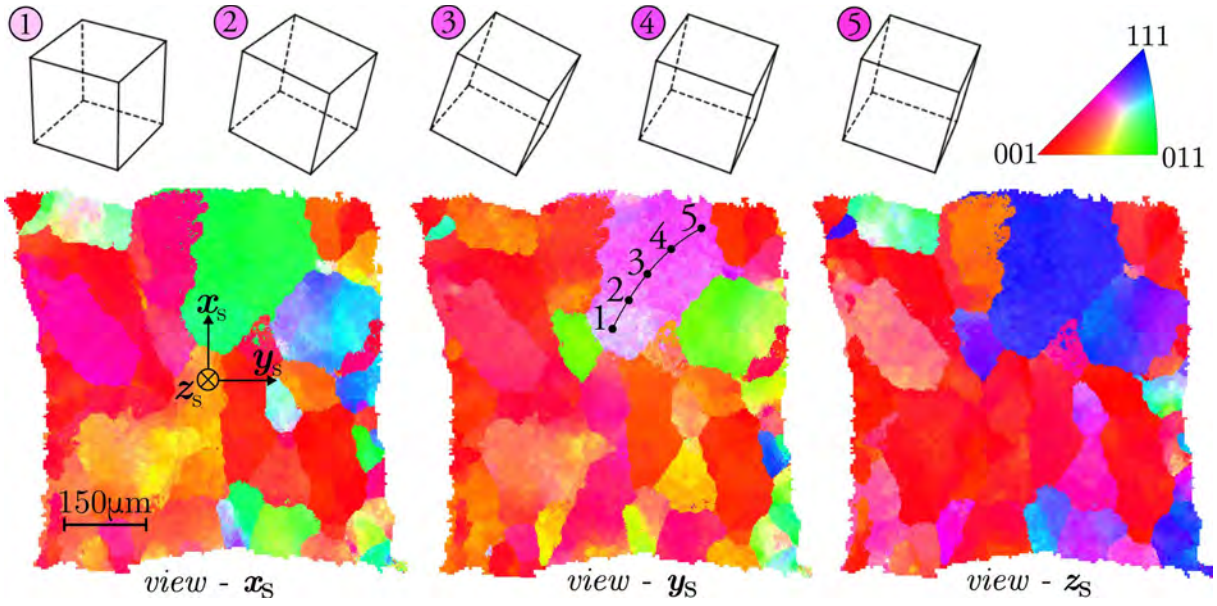

Figure: Super-sampled grain orientation map (bottom row) depicting a plastically deformed Aluminium sample subject to a macroscopic tensile strain of >50% (along $\boldsymbol{z}_l$). Each voxel in the reconstructed grain map is coloured by the inverse pole figure colour key (top right). The real-space sample coordinate view axes, to which the colours refer, are (*x, y, z*) as indicated from left to right in the figure. In the top row a ~7° intra-grain orientation gradient is illustrated as a sequence of magnified difference rotations with respect to the orientation present at point (1). Diffraction data were acquired at the ESRF ID11 beamline.

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Femto-second laser induced sub-surface hardening in metals

L. Rousseau[a], Sz. Kalácska[a], N. Peillon[a], X. Sedao[b] , J-P. Colombier[b], E. S. Ryan[c], J. Wright[c] and A. Borbély[a]

Laser peening is a surface treatment technique that improves the mechanical performance of metals. It can be used to increase the material resistance to crack initiation, extend fatigue life as well as to enhance the fatigue strength. Most of the works are using nano-second (ns) pulsed laser[1], which require a sacrificial coating that absorbs the laser energy and transforms it into plasma, protecting by that the work piece surface from melting. Recent studies[2-3] reported on femtosecond (fs) laser peening without protective coating, which makes this new process technologically attractive. However, due to the different energy absorption mechanisms active during fs and ns-laser peening (energy absorbed by the electrons and by the plasma, respectively) it is expected that the interaction region between the metal and the emerging shock wave will be different in the two processes. Experimental results indeed, indicate that ns-laser peening creates residual stresses at mm depths in metals, while fs-laser peening affects the metal up to depths of about 100 µm[2-3] only. The aim of this work is to evaluate this interaction depth between the shock wave created by fs-laser peening and different metals by measuring the dislocation density and the associated strain energy as function of the distance from the irradiated surface (Fig. 1a). XRD peak profile measurements were done at ID11 of the ESRF using a planar beam with a lateral length of 75 µm and thickness of about 1 µm. The results for Al, Ti, Fe and Ni show that the interaction depth during fs-laser peening is the highest for Al, where dislocations can be created until depths of about 40 µm (Fig. 1b).

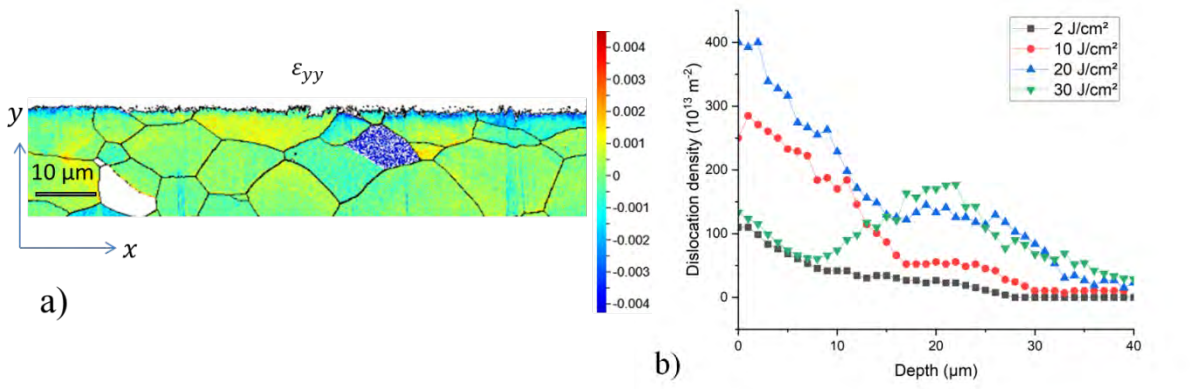

Figure 1: a) Normal strain component ε_{yy} measured with HR-EBSD on the cross section of an irradiated Fe specimen. Large compressive strains are formed close to the penned upper surface.
b) Variation of the dislocation density in Al as a function of depth for different laser fluences.

The interaction depth and the dislocation density for Al scale with the fluence (from 2 to 20 J/cm²) until a threshold value (~30 J/cm²) when ablation and surface melting takes place. In the same range of fluence the affected depths in Ti, Ni and Fe are limited to about 15-20 µm.

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[b] Univ Lyon, UJM-St-Etienne, CNRS, Institute of Optics Graduate School, Laboratoire Hubert Curien UMR 5516, F-42023 Saint-Etienne, France

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DFXM Imaging of Dislocation Dynamics in in-situ Deformed Aluminium

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Dislocations are line defects within the atomic arrangement of crystalline materials, notably metals. Irreversible deformation, or plasticity, of these materials is closely linked to the presence of dislocations, acting as local mediator of shearing motion within the crystal. Upon deformation, dislocations migrate through the crystal and agglomerate into micrometre-sized structures. These structures are understood to govern the nature of deformation processes as their evolution and morphology is tightly correlated with the applied deformation.

Traditionally, dislocation structures have been mainly studied using Transmission Electron Microscopy (TEM) necessitating thin foil specimens with a thickness of approx. 200 nm which limit access to volumetric information and dynamics. Consequently, the physics behind the formation and refinement processes of dislocation structures have remained elusive.

Dark-field X-ray Microscopy enables imaging individual dislocations deeply embedded in millimetre-sized specimens. In this talk, we will present a deformation-resolved movie of a three-dimensional dislocation configuration rearranging under a gradually applied external load. Through this, we identified individual dislocation lines and reconstructed the evolution of their three-dimensional network. These new insights are expected to enhance our understanding of the physics of plasticity and bridging the gap between atomic-scale rearrangements and macroscopic deformation.

Studying martensitic phase transformations and deformation twinning with in-situ DFXM and topotomography

A. Bucsek

This talk will discuss the use of high-resolution 3D in-situ characterization to study local deformation in metallic materials under mechanical loading. We present work on stress-induced martensitic phase transformations in CuAlNi shape memory alloys and stress-induced deformation twinning in lightweight structural Mg-4Al alloys. In both cases, the local stress concentrations that develop are critical for understanding both the local micromechanics (i.e., the activation of inelastic deformation mechanisms) and the macroscopic material behavior (i.e., ductility and fracture in the case of twinning, and hysteresis and functional fatigue in the case of martensitic phase transformations). Using dark-field X-ray microscopy (DFXM) on ID06-HXM and topotomography on ID11, we resolve the emergence and evolution of twin/phase interfaces and their 3D morphologies, interfacial strain fields, and interactions with defects under subsequent loading. Finally, we discuss our outlook for in-situ mechanical loading experiments on ID03 and ID11 with multimodal, multiscale diffraction microstructure imaging techniques.

Diffraction and Imaging studies of Neuron-Mimicking Devices and Their Environments

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Neuromorphic functionalities in memristive devices are commonly associated with the ability to electrically induce local conductive pathways by resistive switching. Using a full-field diffraction-contrast x-ray microscope, we explore the structural effects in VO_2 thin film devices under applied voltages. We observed that the filament formation follows a preferential path determined by the nucleation sites within the device. These sites become predisposed to certain structural phases which persist even after saturation back into the insulating state. In addition to these preferential paths, the structure of the filament is a combination of monoclinic and rutile phases further indicating local preference towards one of the phases. Moreover, an increase to the power in the system shows an overall increase in the size of the filament indicating a constant temperature of the filament near the transition temperature. Repeated voltage cycling of these devices indicate that threshold voltage lowering in the system is due to these structural phase preferences. Studying the sapphire substrate environment beneath the VO_2 film, we observe structural changes arising from imprinting of the filament on the substrate. These local changes cannot be explained by simple heating of the system and thus additional modifications from the film onto the substrate must be considered. We touch on the implications of these changes to the substrate on future network construction and thin film systems.

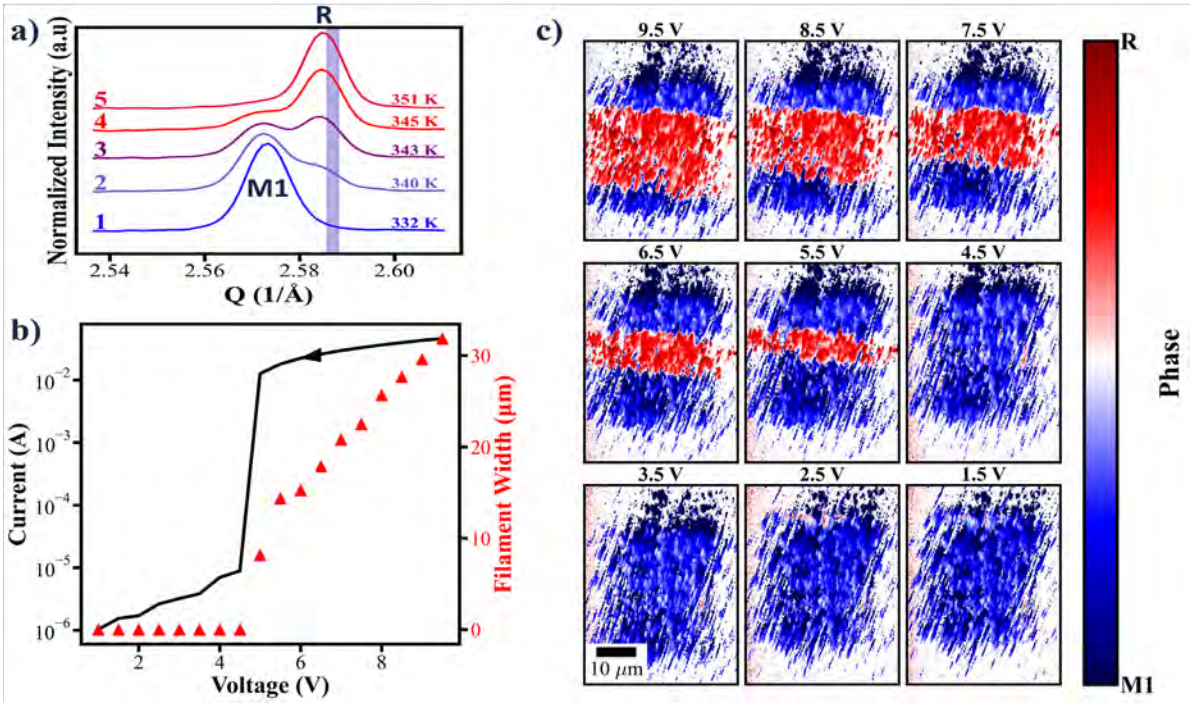

Figure: (a) Structural transition from the insulating monoclinic (M1) to the metallic rutile (R) in VO_2 as temperature increases. (b) The voltage-current curve for this device (black) and size of the filament as the power dissipated in the system increases (red). (c) Dark-field maps of the phase coexistence and phase differences of the filament in VO_2. Red regions indicate metallic rutile phase and blue regions indicate insulating monoclinic phase.

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3D grain growth in electrical steel sheet studied by laboratory X-ray diffraction contrast tomography

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Electrical steel sheets are widely used for transformers and motors. To increase the efficiency, it is critical to reduce iron loss of the electrical steel sheets. Iron loss is roughly given by the eddy current loss and the hysteresis loss. Si up to about 3 wt% is added to the steel to reduce the eddy current loss, while the microstructure, texture and grain diameter, should be controlled during grain growth to reduce the hysteresis loss. Conventional 2D techniques, such EBSD and XRD, have provided much information about the microstructure evolution during annealing. However, uncertainties related to the bulk kinetics remain. Therefore, 3D characterization methods have been developed to overcome the limitations in 2D characterization. Recently, laboratory X-ray diffraction (LabDCT)[1)] has become useful method due to its easy accessibility and non-destructive nature. In this study, grain growth within electrical steel sheets was investigated by statistical and local analysis using the LabDCT technique. The aim is to understand how the local grain structure as well as the texture affect the growth behaviour.

The material is Fe-3wt% Si, which was cold-rolled to 0.3 mm thickness with 80 % reduction and annealed to the fully-recrystallized state. This sample was annealed at 830 °C for total 3 steps. LabDCT measurements were performed using a Xradia 520 Versa X-ray microscope (Zeiss). The sample was scanned in the Laue focusing geometry. A total of 181 diffraction projections were obtained by a sample rotation of 360°. The grain reconstruction was performed with *GrainMapper3D* (Xnovo Technology ApS).

The results show that on average the grain growth follows Hillert's law[2)]: namely larger grains grow and smaller grains shrink (Figure 1). However, there are many grains that do not follow the law. To understand the reasons of this deviation, the local grain structure around a small grain (marked by the red circle in Figure 1) was analysed in detail. It is shown that this grain (labelled as grain G in Figure 2) is not consumed by the neighbouring large grains d and e because of the low angle grain boundaries between them. Furthermore, this small grain G was able to consume other even smaller neighbouring grains (a, b and c in Figure 2(a)) by the migration of high angle grain boundaries. This result underscores the importance of the local grain neighbourhood in determining the microstructural evolution process. This work further highlights the critical need for 3D non-destructive characterization to advance our understanding of the grain growth behaviour.

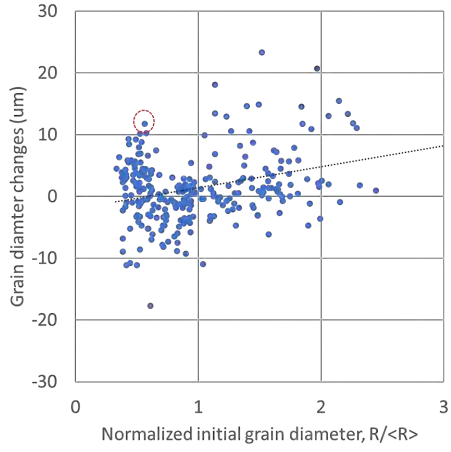

Figure 1 The relationship of the normalized grain diameter (individual grain diameter divided by the average) and changes in individual grain diameter during grain

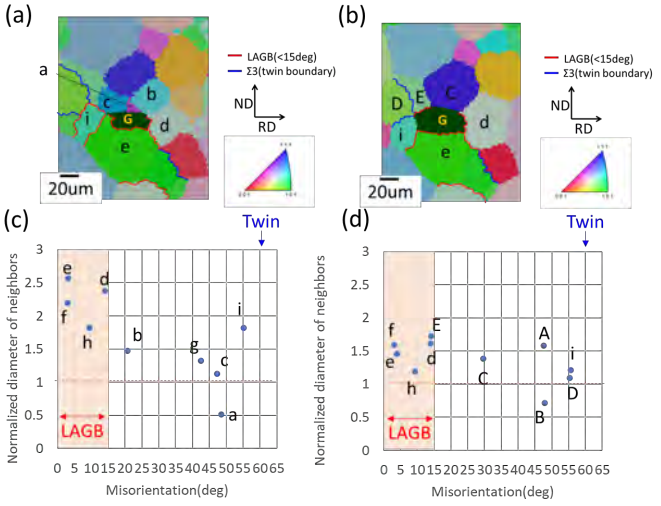

Figure 2 IPF map in a cross section of the 3D volume, and misorientation to as well as size of neighbouring grains to grain G.
(a,c) starting grain structure (b,d) after grain growth.

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Future of High Energy Diffraction Microscopy: Going towards more complex specimens and environments

H. Sharma

High Energy Diffraction Microscopy (HEDM) or 3DXRD is a powerful technique for in-situ studies of polycrystalline materials. In this talk, I will cover improvements, both software and hardware, at the Advanced Photon Source to extend the HEDM techniques to more challenging materials and environments. I will also cover recent developments to improve reconstruction accuracy using AI/ML techniques. New experiment modalities for studying various details in the samples will be highlighted.

Real-time imaging of acoustic waves in bulk materials with X-ray Microscopy

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Tim van Driel,[3] Matthew Seaberg,[3] Eric Folsom,[4]
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During the last decade, Dark-Field X-ray Microscopy (DF-XRM) has emerged as a powerful tool for mapping the microstructure within deeply embedded volumes in bulk crystalline materials [1,2,3,4,5]. So far, the time resolution has been limited to milliseconds, which is sufficient to image some dynamic processes in situ. For example, dislocation motion close to the melting point in aluminium [6] and structural transformations taking place in ferroelectrics during phase transitions [7].

Phonon dynamics, on the other hand, occur on the pico- to microsecond timescale. Until now, imaging of phonon waves has been limited to thin specimens in dark-field transmission electron microscopy [8] or to nanocrystals using Bragg coherent diffraction imaging [9]. In this work [10, 11], we implemented DF-XRM at an X-ray Free Electron Laser, the Linac Coherent Light Source at SLAC National Accelerator Laboratory, using a stroboscopic optical-pump / X-ray-probe scheme. We directly visualized the generation, propagation, branching and energy dissipation of longitudinal and transverse acoustic phonon waves in a mm-sized diamond single-crystal, demonstrating how mechanical energy thermalizes from picosecond to microsecond timescales.

This novel approach opens the door to direct visualization of a plethora of phenomena. For example, the interaction between phonon waves and microstructural features such as twin walls or dislocations, or diffusionless transformations where the phase fronts propagate close to or at the speed of sound (e.g. the martensitic transformation). We therefore anticipate that our findings will have a wide impact in the fields of materials physics and condensed matter science, and that it will inspire similar stroboscopic fast or ultrafast DF-XRM experiments at ID03.

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Wide Angle X-ray Scattering Tensor Tomography

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3D x-ray scattering microscopy techniques such as 3D-x-ray diffraction (3D-XRD)[3], diffraction contrast tomography (DCT) a scanning 3D XRD all share the same aim of mapping the grain structure of a polycrystalline sample from measurements of scattering patterns. All these methods rely on identification of individual Bragg-peaks stemming from single grains and are therefore fundamentally limited to samples with large and rather ideal grains.

Small-angle x-ray tensor tomography (SAXS-TT)[2] is another x-ray scattering technique, which aims to reconstruct a voxel-map of the sample, where each voxel contains a 3D-reciprocal space map of the anisotropic small-angle scattering cross section. The extension of SAXS-TT to wide-angle scattering (WAXS-TT) has so far been demonstrated for bone-samples with very smooth texture[3] but is yet to be tested on a wider range of systems.

Because WAXS-TT does not rely on identification and indexing of individual Bragg-peaks, it has the potential to be a compliment to other 3D scattering microscopy techniques, for samples where the grains are too small or too deformed to yield isolated spots.

We have performed initial experiments at the cSAXS beamline at the Paul Scherer Institute and have achieved promising results showing that with only small modifications of existing SAXS-TT algorithms, we can reconstruct spatially resolved pole-figures of samples with complicated texture at an angular resolution better than 10°.

The WAXS-TT experiment is essentially identical to a scanning 3D-XRD experiment except for the inclusion of a second rotation stage needed to access a wider range of directions in both real- and reciprocal space. It can therefore potentially be implemented as the same beamlines as a complimentary technique.

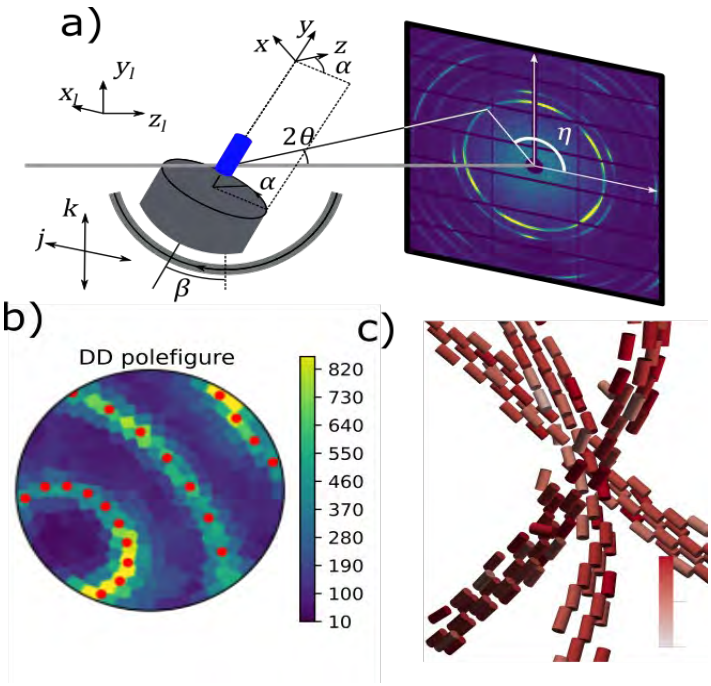

Figure: a) Sketch of the experimental geometry in WAXS-TT. The sample is raster-scanned along two orthogonal rotations *j*, *k* and rotated around two angles α, β. b) Pole figure of a single voxel of the reconstruction. c) 3D rendering of the reconstructed fibre-symmetry axis.

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[1] Liebi *et al.* 2015 *Nature* **527**, DOI: 10.1038/nature16056
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ID11

J. P. Wright[1], W. Ludwig[1,2], H. Fang[1], P-O. Autran[1], J. Ball[1], E. Lawrence Bright[1], C. Giacobbe[1], H. Gleyzolle[1], E. Papillon[1], J.M. Clement[1], E. Gagliardini[1]

The Materials Science beamline (ID11) at the ESRF offers a suite of instrumentation for diffraction and imaging of materials and their microstructures. The ESRF source upgraded to a "fourth generation" lattice that offers even higher X-ray brilliance than before. There is sufficient X-ray flux in the monochromatic beam at ID11 to be able to recrystallize some copper wire using beam heating, as shown in figure 1.

A photon counting CdTe Eiger2 4M pixel detector has been installed on the nano focus station. This allows fast (500 fps) scanning XRDCT measurements with X-ray beam sizes down to ~0.1 μm and energy from 40-70keV. These methods bridge the gap between traditional powder and single crystal diffraction approaches with a range of applications, and can be applied to amorphous samples as well as polycrystalline microstructures.

The 3DXRD station has been upgraded with two new sCMOS detectors for phase- and diffraction- contrast tomography (PCT, DCT) and a new CCD detector for topo-tomography (TT). These new detectors enable much faster scans, with no gaps for readout. The overall throughput has drastically increased thanks to the combination of higher flux and faster detectors. New scans have been developed within the new (Bliss) control system to exploit all these detectors for complementary diffraction and imaging experiments. Grains can be indexed and located via 3DXRD or DCT scans ready to be aligned for high resolution TT imaging measurements.

As well as a brief overview of the existing beamline hardware, this presentation should report on some of the scientific highlights from recent user experiments. Potential future upgrades will also be discussed, for example: a new monochromator could improve the resolution in strain for diffraction, as well as giving a larger field of view for tomography together with smaller imaging pixel sizes.

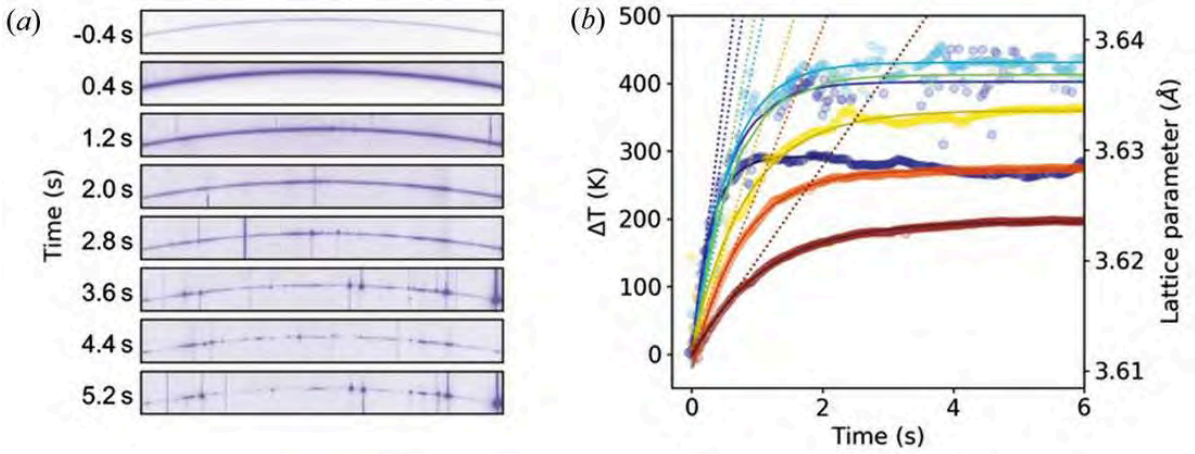

Figure 1: Beam heating in a 0.19 mm-diameter Cu wire sample. (a) images of the (222) reflection as a function of time after insertion of focusing lenses with a 0.86 mm-length sample, (b) the change in temperature as a function of time for different wire length (from [1]).

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Texture tomography for polycrystalline materials: Principle and first results

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The crystallographic texture is a key organization feature of many technical (steel, concrete, ceramics or energy materials) and biological materials (bone, teeth, wood, tendon). In these materials, and especially in those with hierarchical structure, the preferential alignment of its nano constituents is heavily influencing the macroscopic behaviour of the material.
In order to study local crystallographic texture with both high spatial and angular resolution, we are developing Texture tomography (TexTOM). This approach allows to model the diffraction data of polycrystalline materials by using the full reciprocal space of the ensemble of crystals and describe their texture via orientation distribution functions. Thus, we acquire quantitative texture information and address limitations associated with existing models: The TexTOM approach reduces the number of needed projections per sample, which at the same time increases the sample throughput and reduces the dose of X-rays on the material. In addition, it yields probability distributions of all unambiguous crystal orientations in each voxel.
In this contribution, we present the mathematical model, the inversion strategy and its current experimental implementation. Furthermore, we present 3D images obtained from a synthetic, inorganic model sample, the silica biomorph. We will use this example to show numerical and experimental benchmarking of the method.

Study of CSTZ ceramics plasticity with synchrotron light and correlative characterizations

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One of the obstacles preventing the extensive application of ceramics is their constrained ductility and toughness, which stems from the conventional trade-off between strength and ductility observed in numerous difficult-to-deform materials. However, the discovery in 1975 that by using doping agents on zirconia, a stress-induced martensitic transformation from a tetragonal to a monoclinic phase could be controlled to become the source of transformation induced toughening[1].

In this context, ceria-stabilized tetragonal zirconia (CSTZ) is advantageous when compared to the better-known yttrium oxide (Y_2O_3)-stabilized zirconia, stabilization with ceria reduces the critical stress required for phase transformation, enabling it to be set before cracks propagate. In addition, CSTZ ceramics exhibit a plasticity effect induced by phase transformation (TRIP for "Transformation-Induced Plasticity"), with a plastic deformation capacity of the order of one percent before fracture[2]. However, even though the TRIP effect has already been studied on a macroscopic scale[3], small-scale characterizations are needed for a thorough understanding.

In that regard, the objective of this work (ANR-21-CE08-0019 NANOTRIP) is to unveil the mechanisms that control the TRIP effect, focusing on the crystallographic and mechanical aspects, as well as how the transformation propagates between grains. For that, we have used synchrotron light for *in situ* compression experiments in CSTZ micropillars. Correlative analyses such as *in situ* SEM compression tests, molecular dynamics simulations, and the theory of martensitic transformation were performed in parallel. By combining both experimental, simulation, and theory, a more detailed understanding of the crystal orientation dependence of plastic deformation can be obtained, the determination of critical stresses for transformation, and also a selection criterion for the transformation variant (A, B, or C).

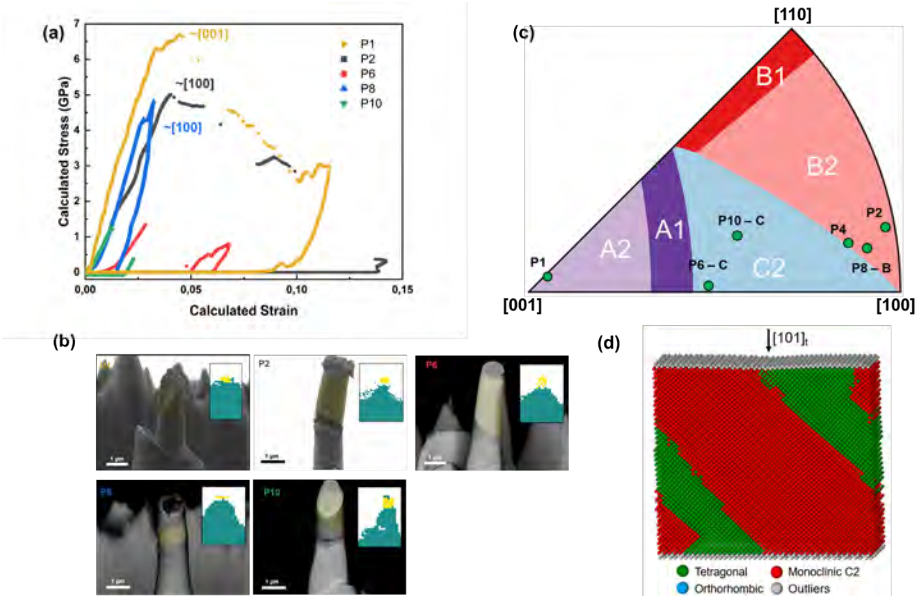

Figure: (a) Stress-strain curves and (b) respective SEM images of compressed micropillars highlighting the transformed region (yellow). (c) Variant selection criterion based on the crystallographic theory. Laue microdiffraction variants are shown in green points in agreement with the theory. (d) Molecular dynamics simulation with a $[101]_t$ compression axis, exhibiting the transformation for a C2 variant.

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Can residual stresses disrupt the energy storage functionality of antiferroelectric $NaNbO_3$ ceramics?

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Stabilizing lead-free antiferroelectrics at room temperature is key for creating greener and more efficient energy storage devices.[1] While $NaNbO_3$ solid solutions hold great promise for high energy density applications, its pure form displays structural instabilities arising from irreversible electric-field induced phase transitions and/or an unwanted coexistence with its ferroelectric polymorph.[2] To unravel how mechanical constraints imposed by residual stresses, structural defects, and microstructure disrupts the stability of the $NaNbO_3$ antiferroelectric state, we used in situ Dark-Field X-ray Microscopy (DFXM) to map local microstructural deformations in a single embedded $\{100\}_{pc}$ grain. By replicating typical heat treatment conditions, we show that the ferroelectric phase nucleates as a result of the coupled interplay between residual shear and compressive strain distributions that manifest during cooling towards ambient temperature. In addition, the microstrain relaxation behavior indicates that long-range defects carrying local ferroelectricity preferentially nucleate at the expense of the antiferroelectric phase in regions at sub-micrometer distances from the grain center. We believe our findings introduce a significant perspective on the structure-property relationship of $NaNbO_3$, showing that adequate temperature control during low temperature sintering, heat treatments, or in operando conditions may be vital for preserving the antiferroelectric properties. Therefore, our work impacts the shear-strain engineering of the current ceramic processing methods, paving the way to the development of more reliable $NaNbO_3$-based antiferroelectrics with enhanced energy storage efficiency.

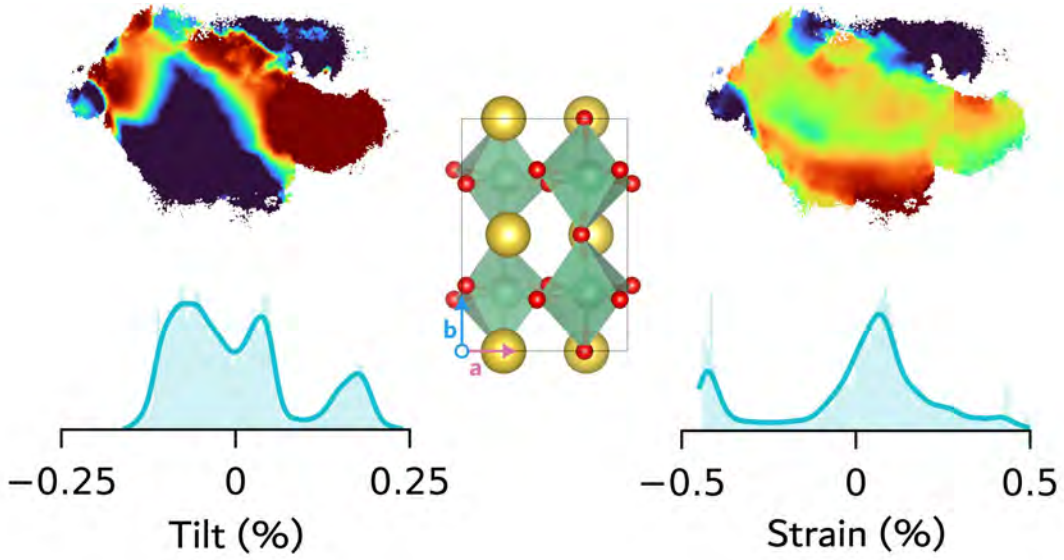

Figure: DFXM maps of lattice tilt and elastic strain in a $\{100\}_{pc}$ grain at ambient conditions, post-cooling from the high-temperature phase. Our results suggest that the unexpected transformation to the ferroelectric structure (in the figure), displacing the antiferroelectric structure, is driven by a shear-strain coupling mechanism. Specifically, a positive peak in the tilt distribution is induced by a compressive strain peak, shedding light on the underlying dynamics of this transformation.

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Fast and ultrafast: stroboscopic DF-XRM and functional materials

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Functional materials, such as dielectrics, ferroelectrics and piezoelectrics are ubiquitous in electronics. The functional properties of these materials are intimately tied to their local structure, a structure that varies in space and changes during operation. This brings a central challenge to characterization: If the structure changes during operation, what local structure do we wish to explore? For a long time this question has had a simple answer, as ex-situ characterization was the only option to characterize local structure. However, with the increasing brilliance of X-ray sources, the time has come to revisit this central question, and provide a different answer: Characterize all the in-situ structures!

X-ray microscopy provides ample space around the sample to build complicated experimental setups, and in this talk I will show how the group at DTU have used stroboscopic DF-XRM to characterize samples from kHz to GHz[2]. By combining stroboscopic experiments and DF-XRM we can study local processes in the bulk with bespoke time resolution for the first time. In doing so, we can provide new answers to old questions, such as: How do ferroelectrics switch in real materials? Is ferroelectric switching a stochastic process? Why does the performance decrease over time?

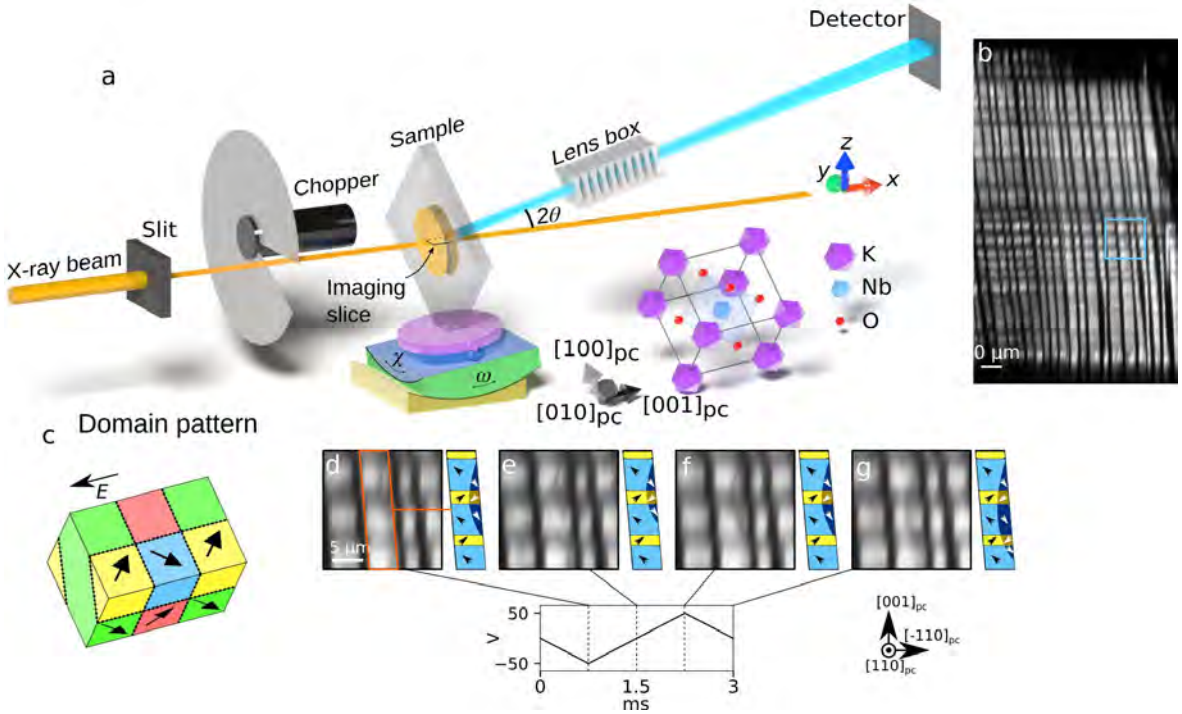

Figure: Subfigure (a) illustrates a stroboscopic DF-XRM experiment on ferroelectric $KNbO_3$ at 300 Hz. The intensity map (b) reveals the prevailing domain pattern (c). The motion of domain walls during cycling is shown in the cut-outs (d-e). Note that both thermodynamically stable (head-to-tail domain walls) and thermodynamically unstable (head-to-head domain walls) are present.

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Diffraction Microscopy Developments at the Advanced Photon Source

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At the Advanced Photon Source (APS) at beamline 1-ID we have almost two decades experience with high-energy diffraction microscopy techniques. Currently the APS is undergoing an upgrade which opened the opportunity to rethink the user programs and the provided experimental setup for researchers and industry according their current needs and interests.
In this presentation I will overview the past decades developments which served as the base for the future expansion of possible measurement modalities to provide users with a more detailed and information rich picture about the microstructure of their specimen in situ or operando. As the advanced material systems have more complexity the experiments need to adjust to the challenges and be ready for investigations on several time and length scales. Our team of engineers and scientists unified forces to address this challenge and we hope the result will satisfy the next generation scientific interests for the coming decades.
The new developments require modern approaches on both the hardware and software side and planned to be improved continuously even after the new beamline will be taken over by user measurements.

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Neural networks for rapid phase quantification of Cultural Heritage X-ray powder diffraction data

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Recent developments in synchrotron radiation facilities have considerably increased the amount of data generated during acquisitions, requiring fast and efficient data processing techniques. Here, we present the application of Artificial Neural Networks (ANNs) to data treatment of X-ray diffraction computed tomography (XRD-CT) experiments. Processing involves mapping the phases in a tomographic slice by predicting the phase fraction in each individual pixel. ANNs were trained on sets of calculated XRD patterns generated using a python algorithm developed in-house. In addition, an initial Rietveld refinement of the tomographic slice sum pattern provides additional information (peak widths and integrated intensities for each phase) to improve the generation of simulated patterns and make them closer to real data. A grid search was used to optimize the network architecture and demonstrated that a single fully connected dense layer was sufficient to accurately determine phase proportions. This perceptron was used on XRD-CT acquisition of a homemade mock-up and an historical sample of highly heterogeneous multi-layered decorations of late medieval statues, so-called "applied brocade". The phase maps predicted by the ANN were in good agreement with other machine learning methods, such as non-negative matrix factorization[1], and sequential Rietveld refinements performed with TOPAS software. The method was evaluated by regenerating experimental patterns from predictions and using weighted R profile as agreement factor. This assessment allowed to confirm the accuracy of the results.

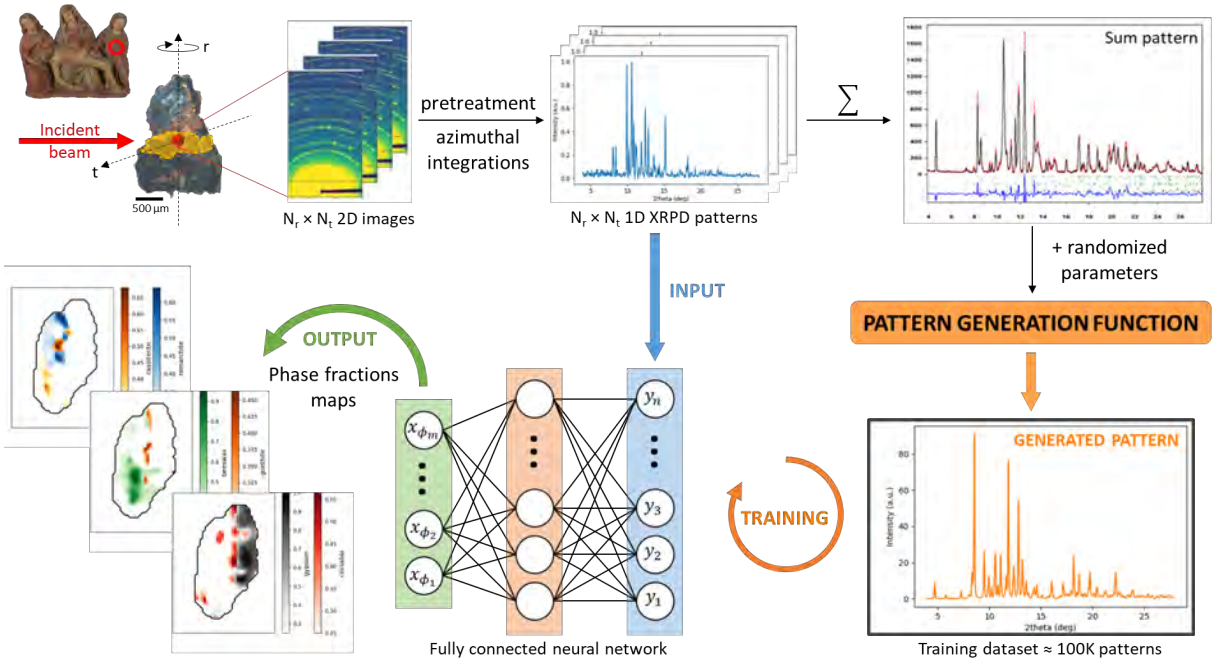

Figure: Summary of the method different steps from acquisition to phase fraction mapping

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Laue Diffraction X-Ray Microscopy

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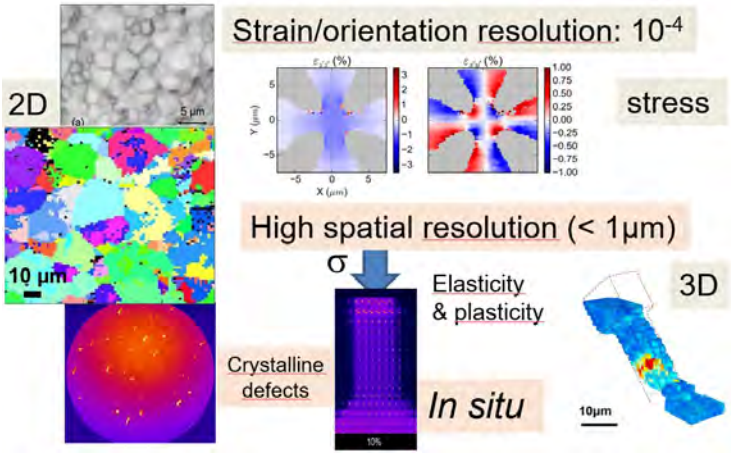
X-ray Laue microdiffraction (µLaue) is a well-established technique to characterize microstructural and mechanical fields in polycrystalline specimens at the sub-micron scale with an absolute strain resolution as fine as 0.01%. Without any particular sample preparation, this non-destructive technique collect scattering signals from individual crystals and crystalline objects. Performing raster scans with a few hundreds nanometer beam is particularly suited for materials science studies to obtain orientation and strain maps [1] even in case of large strain heterogeneity (e.g. plastic regime). Moreover in situ or operando experiments can be carried out. This standard technique [2] on CRG-IF BM32 beamline at ESRF can be complemented by additional measurements with resp. energy and depth resolution for resp. full stress/strain tensor determination (without any mechanical assumption)[3] and 3D microstructure imaging [4]. The Laue patterns analysis comprises schematically several steps from Laue pattern images to structural parameters. Nowadays materials (nanosized and numerous crystals, large deformation, presence of extended crystal defects) require fast automatic and flexible software tools to obtain reliable information. Complex collected Laue patterns composed of more than a thousand Laue spots originating from dozen of crystals can be indexed rapidly thanks to the recent open source LaueNN software based on neural network recognition of Laue spot corresponding hkl Miller indices [5]. Perspectives offer by the ongoing upgrade will be discussed.

Figure: Laue Diffraction X-ray Microscopy capabilities on CRG-IF BM32 at ESRF



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