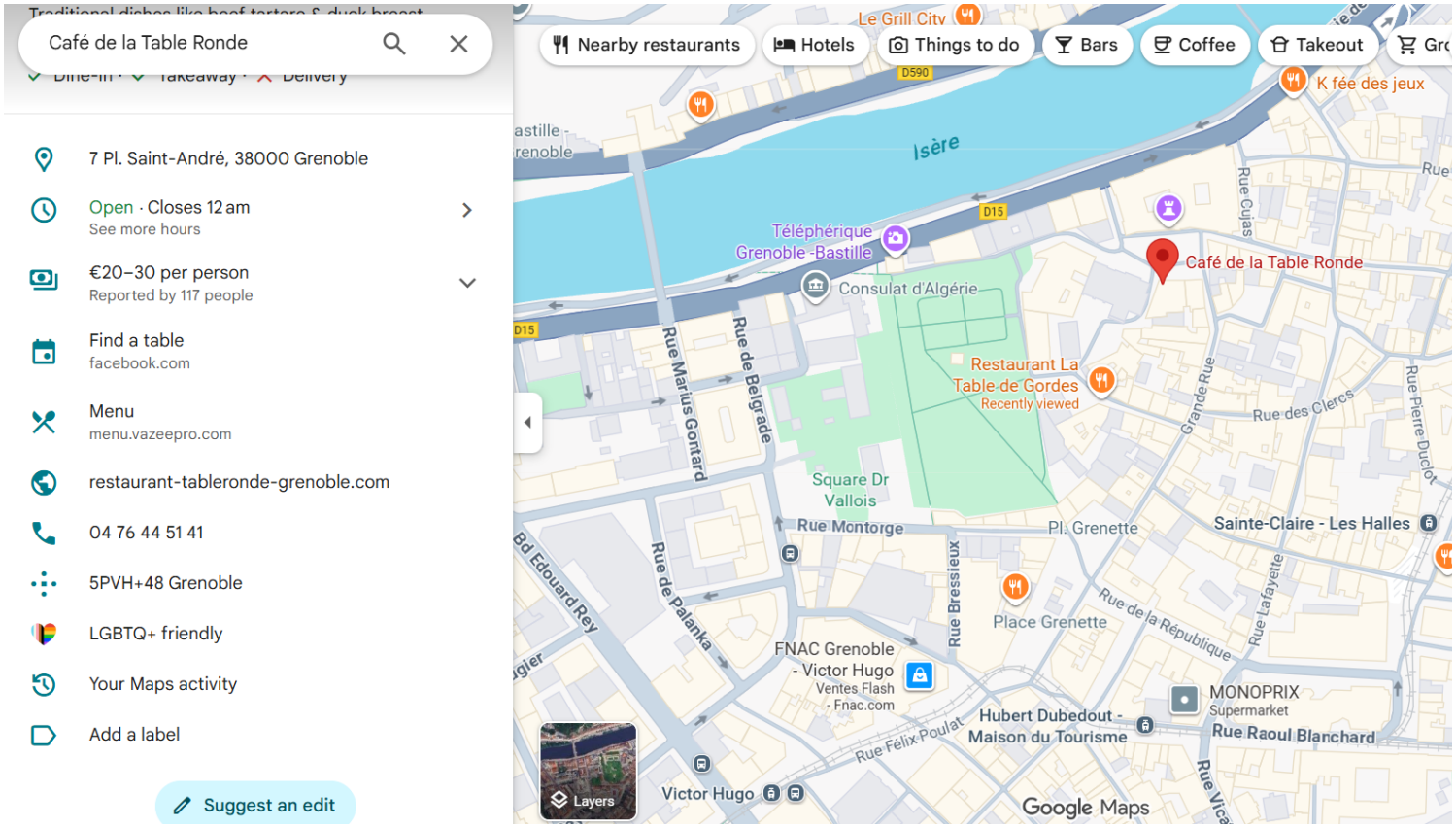
Practical information and abstracts

Workshop is at the auditorium



Tram B from ESRF goes to city center. Get down at Victor Hugo/Sainte-Claire and have a short walk to the cafe. In case of emergency call

Jaianth - 0032 473205390 (same for whatsapp)

22.09.2025	Name	Topic
Duration 09:00-20:00		
09:30-10:10	Organizers	Welcome + about ESRF/Remade
10:10-10:40	**Prof. Miguel Garcia Arnada**	X-ray imaging of current cementitious materials to image new sustainable cementitious materials
10:40-11:00	Dr. Robet Fischer	Time-resolved X-ray microtomography for studying dynamic processes in materials at beamline ID19 of the ESRF
11:00-11:10	Break	
11:00-11:30	**Dr. Minjian Wu**	SEM/TEM - TBD
11:30-11:50	Dr. Nicola Vigano	X-ray fluorescence ghost imaging: acquisition setup, reconstruction algorithms, and advantages for science applications
11:50-12:10	Dr. Masoud Dialameh	Three-dimensional elemental mapping at nanometer-scale resolution by atom probe tomography (APT)
12:10-13:30	Lunch at canteen	
13:30-14:05	**Prof. Susan Bernal**	The role of materials science in the development of sustainable and climate resilient infrasturcture
14:05 -14:25	Mr. Ilies Bentamene	Synchrotron X-ray nano-tomography as a tool for 3D strain partitioning in Al alloy designed for Additive Manufacturing
14:25-14:45	Dr. Nicolas Charpentier	Optical and spectroscopy imaging for advanced electronic waste sorting
14:45-15:05	Dr. Thibault Rosier	High throughput XRF study of electronic components to enable better understanding of the urban mine composition

15:05-15:25	Break
15:15-17:30	ESRF Tour
17:30-20:00	Poster + wine + cheese + food

23.09.2025	Name	Topic
Duration 09:00-17:00		
09:00-09:30	**Dr. Jiawei Mi**	Operando studies of the dynamic evolution of complex intermetallic phases of recycled Al alloys in the solidification process
09:30-09:50	Dr. Ken Aldren Sumaya	Probing Anisotropy-induced Toughening in Ti3C2Tx MXene Materials using Wide-Angle X-ray Scattering
09:50-10:10	Dr. Yan lu	Electrostatic potential of latex sphere using off-axis electron holography
10:10-10:30	Dr. Laurenz Schoroer	Contrast enhancing staining agents to image biofilms using lab-based micro-computed tomography
10:30-10:40	Break	
10:40-11:10	**Dr. Can Yilidrim**	Looking Inside: Multiscale Diffraction Imaging from Millimeters to Nanometers
11:10-11:30	Ms. Giesel Fernandez	Synchrotron diffraction and tomography analysis of damage evolution in AA2050 under high-temperature loading
11:30-11:50	Dr. Himanshu Dilip Khadse	Recycling of CIGS solar modules and electrochemical recovery of Cu and CRMs from waste
11:50-13:30	Lunch	
13:30-14:00	**Dr. Nikolay Kardjilov**	Recent advancements in neutron imaging
14:00-14:20	Prof. Guillaume Brotons	In-situ Phase-Contrast Tomography and Small-Angle X-ray Scattering at synchrotron sources to unveil multiscale-structured bone scaffolds made of bioglass nanoparticles for regenerative biomaterials
14:20-14:35	break	

14:35-15:05	**Dr. Sofiane Guessasma**	3D printing and microCT - TBD
15:05-15:25	Dr. Fabio De Marco	Probing CFRP material characteristics with speckle-based X-ray imaging – Workshop “Multi-probe imaging for materials
15:25-16:05	**Dr. Yunhui Chen**	New Insights in Advanced Engineering Materials from in-situ Synchrotron X-ray Imaging
16:05-16:30	Dr. Marta Mirolo	ID31 - TBD
16:30-16:45	Organizers	Thank you
19:00-23:00	Gala dinner	

23.09.2025	**Name**	**Topic**
Duration 09:00-16:30		
09:30-12:30	Dr. Heubert Taieb	Dragon Fly Hands-on
12:30-13:30	Lunch at canteen	
13:30-16:30	Dr. Olga Stamati	SPAM Python

X-ray imaging of current cementitious materials to image new sustainable cementitious materials

Miguel A.G. Aranda

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Portland cements are environmentally contentious, accounting for ≈8% of anthropogenic CO_2 emissions. If cement manufacture is considered a country, it would be the third emitter after China and USA. Thus, developing concretes with lower embodied carbon contents is central to maintaining our well-being. There are many strategies to decrease these emissions and X-ray tomography and powder diffraction are playing a key role on many of them.

Firstly, the most realistic proposals for sustainable low-carbon cements consist of replacing as much Portland clinker as possible with low-CO_2 footprint Supplementary Cementitious Materials. However, the main drawback of these cements is their slow hydration kinetics in the first days of hydration. Cement hydration must be understood to be enhanced. In this context we are working in two original contributions.

I) With the support of ERC-AdG "syn4cem", we are developing 4D (3D+time) cement hydration nanoimaging within a multiscale framework [1]. Full-field laboratory X-ray micro Computer Tomography (μCT) is used but the best spatial resolution is about 2 μm for a Field of View (FoV) of ≈1×2 mm (H×V) with measurements taking hours. Moreover, the contrast between the different components is poor. Full-field propagation-based phase-contrast synchrotron X-ray μCT can study similar FoVs ≈1×2 mm with better spatial resolution, ≈1.0 μm. The measurements are much faster, i.e. 5-10 minutes. Unfortunately, the component contrast is only slightly better. Cement hydration can be studied with much better contrast and spatial resolution by near-field ptychographic and/or holotographic nano-computed tomography. Examples will be shown.

II) With the support of the Spanish government, we are developing the "mix and measure" approach [2-3] for accurately studying the complex hydration reactions. Combined laboratory high energy X-ray powder diffraction (Mo$K\alpha_1$) and μCT decrease errors (i.e. multi-probe approach in exactly the same volume of the same sample). After water mixing, the cement pastes are syringed into 2.0 mm diameter glass capillaries, the ends are sealed with a polymer, and both type of data (XRPD and μCT) are sequentially taken. This removes the sample conditioning step which usually alters the microstructures. Selected examples will be discussed.

Secondly, the CO_2 footprint of cementitious materials can be much reduced by CO_2 curing. EIC-Pathfinder Challenges call-2024 'Towards cement and concrete as a carbon sink' has selected our proposal entitled -Real-time X-ray diffraction and microstructure imaging, accelerating the transformation of cementitious materials into CO_2 sinks-, acronym "X-SeeO2" for funding. The backbone of this proposal is *in situ* X-ray powder diffraction and microtomography during CO_2 curing. The scope of this 'Enabling Technology' project will be introduced.

Thirdly, and if there is time, our investigations on the upcycling by carbonation of Recycled Concrete Fines (a fraction of the Construction and Demolition Wastes) will be presented.

References

[1] - S. Shirani, et al. "4D nanoimaging of early age cement hydration" *Nat. Comm.* **14**, 2652 (2023)

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[3] - J. Fernandez-Sanchez, et al. "Mix and measure II – joint high-energy laboratory X-ray powder diffraction and microtomography for hydration studies of cement blends" *J. Appl. Crystallogr.* **57**, 1067-1084 (2024)

Time-resolved X-ray microtomography for studying dynamic processes in materials at beamline ID19 of the ESRF

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The high photon flux at a synchrotron beamline allows the acquisition of tomographic scans in rapid succession. We show how time-resolved X-ray tomographic microscopy (XTM) can be used to study otherwise inaccessible dynamic processes in opaque and dens materials.
We discuss on selected examples, with an emphasize on the capabilities at the ID19 beamline of ESRF, potentials and challenges of four-dimensional imaging (3D+time), experimental preparation and data processing strategies to extract scientific value of the typically very large imaging datasets.
XTM reveals heterogeneous pore network filling processes not captured by classical homogeneous uptake models for wicking in fibrous porous media like knits or wood. Heterogenous hygroscopic swelling introduces non-localized and inhomogeneous deformation of the wood structure.
By employing XTM, we can identify degradation processes in electrochemical cells including operando catalyst crumbling and membrane delamination in a CO2 electrolyzer and copper conglomerate formation during thermal runaway in Li batteries.
Linking in-situ dynamic microscale processes to the macroscale observable by complementary methods can help to better understand material performance in real applications.

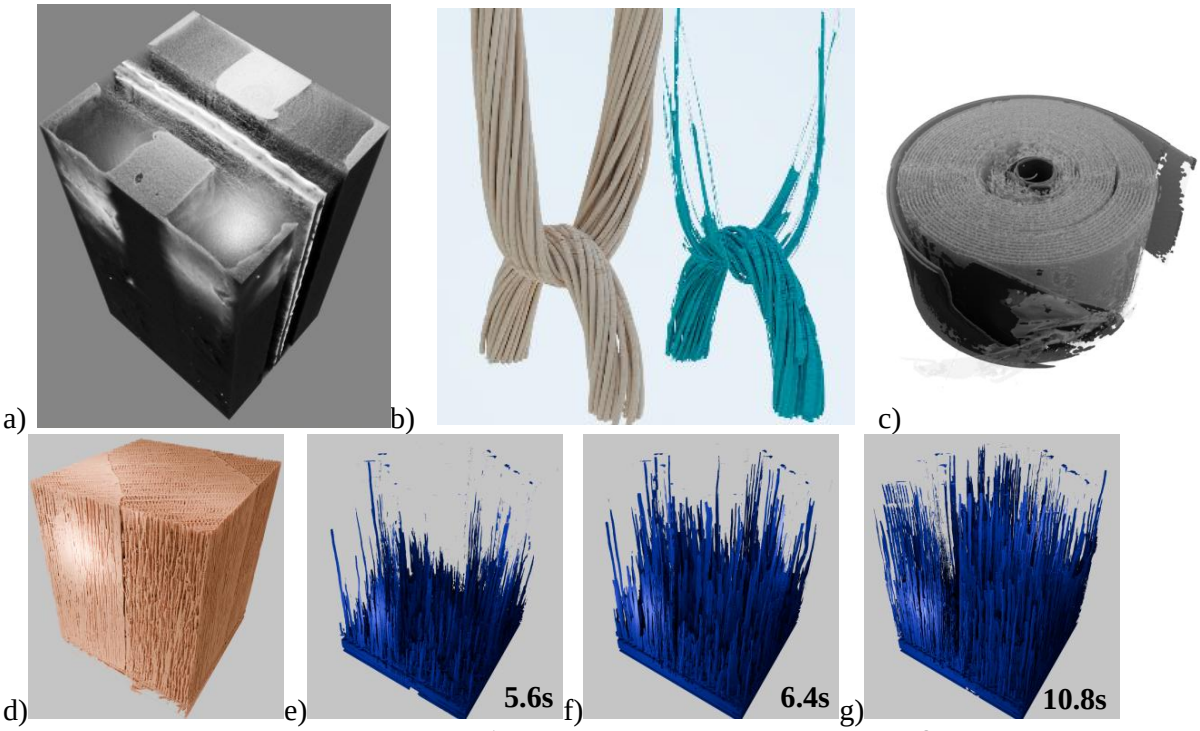

Figure 1: a) operando CO2-electrolyzer[1], b) capillary uptake in a knit loop[2], c) batterie side-wall rupture[3], d) solid matrix of spruce wood, e-g) capillary uptake in d)

[1] Fischer, R. et al. *ACS Applied Energy Materials* **2024**, *7* (9), 3590–3601.
[2] Fischer, R et al. *J Colloid Interface Sci* **2022**, *626*, 416–425.
[3] Venturelli, M. et al. *Journal of Energy Storage* **2025**, *131*, 117122.

Unravel the structure of molecular crystals in organic semiconductors using analytical TEM, 3D electron diffraction and 4D-STEM

Mingjian Wu

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Harnessing the complex nano-scale structure in organic semiconductors is essential to tailor their functionality and performance. For example, the orientation of π-stacking domains and grain boundaries are essential to their electric transport properties in organic field-effect transistors (OFET); in bulk hetero-junction (BHJ) organic solar cells (OSCs) it determines exciton dissociation and charge carrier pathways thus dictate performance of the cells. These structural properties at different length scales are highly sensitive to processing conditions and protocols. Unravel the structure of organic semiconductors from molecular to device scale is essential to establish the processing-structure-properties relationship. Fast electrons interact strongly with matter and can deliver a multitude of structural and analytical signals to study the structure, chemistry and composition of materials at high spatial resolution. Modern electron microscopes, as "a synchrotron in a microscope" [1], hold the potential for such studies. However, direct observation of structures of organic semiconductors using electron microscopy face certain challenges including radiation damage, limitations due to sample geometry and yet-to-established workflows etc.

In this talk, I will report our efforts toward systematically and correlatively characterize the structure of OSCs and organic semiconductor thin films using TEM: from nano-morphology of OSC using analytical methods (with energy-filtered TEM or spatially resolved electron energy-loss spectroscopy) [2], to evaluating their texture with EF-SAED (energy-filtered selection area electron diffraction) and 3D electron diffraction [3, 4]. The results are compared to that from identical sample using established X-ray based method using lab and synchrotron sources. [4] Finally I will showcase probing local structure information with our recently proposed, dose-efficient method 4D-scanning confocal electron diffraction (4D-SCED) technique [5]. Furthermore, I will discuss the merit when this technique is coupled to a state-of-the-art hybrid-pixel direct electron detector [6]. The unique combination of imaging, diffraction and spectroscopy methods in a single instrument of TEM enables straightforward, correlative study of organic semiconductors [4].

Refs:
[1] Ramasse, Q. *Ultramicroscopy.* **180** (2017) 41
[2] Rechberger, S., et. al., *Solar RRL.* **4** (2019) 1
[3] Hawly, T., et. al., *ACS Appl. Mater Interf.* **14** (2022) 16830
[4] Kraus, I., et. al., arXiv:2502.11254 (2025)
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[6] Wu, M., et. al., *J Phys. Mater.* **6** (2023) 045008

X-ray fluorescence ghost imaging: acquisition setup, reconstruction algorithms, and advantages for science applications

N. R. Viganò[1], Dmitry Karpov[1], K. Joost Batenburg[2], Sharon Shwartz[3]

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X-ray Fluorescence Ghost Imaging (XRF-GI) was recently demonstrated for x-ray lab sources. It has the potential to reduce acquisition time and deposited dose by choosing their trade-off with spatial resolution, while alleviating the focusing constraints of the probing beam. In this talk, we present the realization of synchrotron-based XRF-GI [1]: We present both an adapted experimental setup and its corresponding required computational technique to process the data.

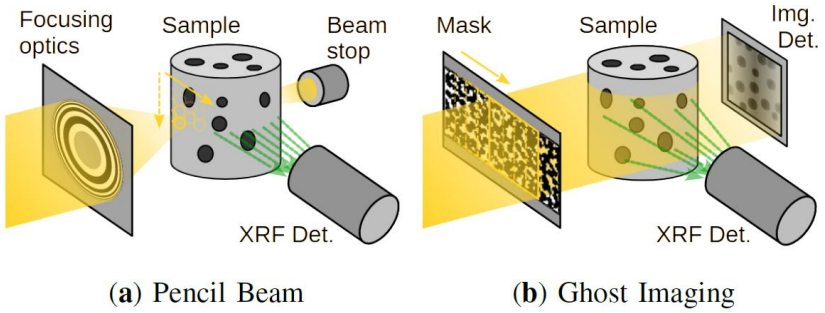

Figure 1: Comparison of the traditional XRF imaging setup (pencil beam) against an XRF-GI setup.

In particular, we present a new self-supervised deep-learning-based GI reconstruction method (called Noise2Ghost), which provides unparalleled reconstruction performance for noisy acquisitions among unsupervised methods [2]. Self-supervision removes the need for clean reference data while offering strong noise reduction. This provides the necessary tools for addressing signal-to-noise ratio concerns for GI acquisitions in emerging and cutting-edge low-light GI scenarios.

In conclusion, the highlights of our work are:

- Extension of the above-mentioned potential advantages of GI to synchrotron XRF imaging.
- A new strategy to improve resilience against drifts at all scales, and the study of previously inaccessible samples, such as liquids.
- A potential new avenue for the development of micro- and nano-scale x-ray emission imaging with dose-sensitive samples.

Notable applications that could benefit from our work include in-vivo and in-operando case studies for biological samples and batteries.

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Three-dimensional elemental mapping at nanometer-scale resolution by atom probe tomography (APT)

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[2] KU Leuven, Department of Physics and Astronomy, Quantum Solid-State Physics, 3001 Leuven, Belgium
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Advances in atom probe tomography (APT) have expanded its capabilities beyond conventional applications in metal alloys, enabling diverse types of analyses across a broader range of material systems, including complex semiconductors, advanced catalysts, battery and sustainable energy materials, each highly relevant to the circular economy. The developments are driven by the unique capability of APT to combine three-dimensional nanometer-scale spatial resolution with isotopically precise elemental identification of both heavy and light elements, including hydrogen [1]. These strengths enable diverse types of materials analysis, including voxel-based 3D compositional mapping with a sensitivity approaching parts-per-million level depending on the voxel volume [1]; detection and characterization of solute clusters, precipitates, and segregated regions as small as ~1 nm [2]; nearest-neighbour distribution analysis; and proximity histograms for quantitative interface characterization.
APT operates on the principle of controlled field evaporation from a needle-shaped specimen. During analysis, surface atoms are sequentially ionized, accelerated in a strong electric field, and detected by a position-sensitive detector coupled with time-of-flight mass spectrometry. Subsequent data analysis and three-dimensional reconstruction of the detected ions allow the original atomic arrangement within the specimen to be resolved, under ideal conditions. This provides atomic-scale insights from local, nanometer-sized regions of a material, in contrast to most X-ray based techniques that yield ensemble-averaged measurements. In practice, however, accurate APT analysis of complex material systems faces several challenges, including reliable specimen preparation, preferential ion evaporation in heterogeneous systems, and data reconstruction artifacts.
This presentation will provide a brief introduction to the fundamentals of APT, covering field evaporation, specimen preparation, and three-dimensional data reconstruction. A selected case studies of APT analysis on semiconductors and metal alloys will be presented, with the emphasis on potentials and the challenges of applying APT to such material systems.

References

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[2] De Geuser, Frédéric, and Baptiste Gault. "Metrology of small particles and solute clusters by atom probe tomography." *Acta Materialia* 188 (2020): 406-415.

The role of materials science in the development of sustainable and climate resilient infrastructure

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Abstract

Concrete is the most consumed material on Earth after water, and since its creation, it has shaped the way our built environment and society develops. From a materials science perspective, concrete is a porous composite material, whose performance is strongly dependent on the intrinsic properties of the binding (or 'glue') component, the type and quantity of aggregates added to it, and the developed physico-chemical properties of the multi-scale interfaces forming between the reacted binder fraction and unreacted particles (anhydrous cement and aggregates). Historically, direct (e.g. mercury intrusion porosimetry) and indirect methods (e.g. water uptake) have been applied to infer concrete's porosity; however, those approaches often required sample preconditioning (e.g. high temperature drying), which changes its pore structure. The use of non-destructive techniques such as X-ray computerised tomography (XCT) has enabled evaluation of undisturbed specimens, and 2D and 3D visualisation of the pore size distribution as a function of mix design or different physico-chemical stresses, providing novel and unique knowledge about factors influencing the performance of such materials.

This presentation provides a brief overview on the application of laboratory and synchrotron X-ray computerised tomography (XCT) techniques to cementitious materials research, centring on studies performed in novel cement formulations to elucidate their pore structure evolution, as well as structural alternations when exposed to aggressive environments (e.g. CO_2) as a function of time. Opportunities of using XCT results for simulating other key properties (e.g. diffusivity) influencing the longevity and CO_2 uptake capacity of concrete will be discussed.

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4. Yue Z., Su Z., Paul P.P., Marsh A.T.M., Macente A., DiMichiel M., Provis J.L., Withers P.J., Bernal S.A. 3D crystalline phase and pore structure evolution upon CO_2 exposure in sodium sulfate-activated cements pastes. Cement and Concrete Research. 2025. 187: 107716.

Synchrotron X-ray nano-tomography as a tool for 3D strain partitioning in Al alloy designed for Additive Manufacturing

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The new generation of synchrotron sources offers the ultimate state-of-the-art instrumentation in terms of resolution, brilliance and coherence. Coupled with a nano-probe, it allows new mechanisms and processes to be studied in materials sciences and engineering. Beamline ID16B [1] at the ESRF - The European Synchrotron - proposes a multi-modal approach allowing the combination of several nano-scale techniques such as X-ray fluorescence (XRF), X-ray absorption spectroscopy (XANES), X-ray diffraction (XRD), X-ray Excited Optical Luminescence (XEOL), X-ray Beam Induced Current (XBIC) and 3D phase contrast nano-imaging. The presentation will detail the use of synchrotron X-ray nano-tomography to understand the mechanical response of an additively manufactured Al alloy with heterogenous microstructures.

Indeed, the microstructures of aluminum alloys designed for laser beam powder bed fusion (LB-PBF) can be heterogeneous across all scales. A bimodal grain structure is often observed with fine-grained regions (FG) consisting of submicron grains near the melt pool boundaries, and coarser columnar grains (CG) in the melt pool interiors. In the FG and CG regions, the morphology and size of intermetallic particles differ as does the composition of the solid solution due to changes in local solidification conditions at the melt pool scale. This specific grain arrangement leads to a 3D architecture at the mesoscale. The mechanical response of such microstructures was found to be influenced by the microstructure heterogeneity using digital image correlation based on SEM images collected during in situ tensile tests [2]. The response of such heterogeneous microstructures is affected by the relative mechanical behaviour of the FG and CG regions.

However, the complexity of the microstructure topology requires a 3D approach to further improve our understanding of the mechanical response of such Al alloys. Thanks to 3D images collected using synchrotron X-ray nano-tomography (voxel size of 25nm3) at the beamline ID16B after different strain increments, we determined the 3D strain maps, at the scale of few melt pools, in a high strength aluminum alloy designed for LB-PBF. The high resolution of the 3D images allowed capturing the fine intermetallic network decorating the microstructure. This enabled to perform high resolution digital volume correlation (DVC) since these intermetallics act as a natural speckle and allow subsets as small as 2 µm3 to be used. The evolution of the 3D strain fields as a function of the macroscopic stress is considered and the heterogenous distribution of strain is explored.

References

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Optical and spectroscopy imaging for advanced electronic waste sorting

Nicolas Charpentier[1], Ange Maurice[2], Dong Xia[3], Andrea Brambilla[1], Jean-Christoph Gabriel[1]

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We present an integrated approach for high-precision sorting and characterisation of electronic components from E-waste by combining machine vision, X-ray absorption spectroscopy imaging and machine-learning classification. This study demonstrates the effectiveness of optical sorting based on machine vision coupled with classification algorithms such as convolutional neural networks (CNN). This combination allows similar electronic components to be efficiently grouped together, making them easier to recycle. In addition to optical sorting, X-ray absorption spectroscopy is being introduced to overcome the limitations of optical sorting by providing crucial information on the elemental composition of electronic components. The integration of these sorting methods into a single process, supported by the construction of a prototype, demonstrates the relevance of this approach, demonstrating up to 96.9% accuracy. The overall process offers the opportunity not only to group similar electronic components efficiently, but also to significantly enrich the final streams with targeted elements, enabling the recovery of previously lost elements due to their low concentration in electronic waste with elemental enrichments by up to 10,000 for targeted elements. This study opens the door to large-scale industrial application of the process, making it economically viable to recycle many elements of interest.

High throughput XRF study of electronic components to enable better understanding of the urban mine composition

T. Rosier[1,2], L. Admans[1,3], J.C.P. Gabriel[1,4], A. Stolidi[3]

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Our dependence to electronic tools has made the supply of critical raw metals (CRMs) crucial. Waste printed circuit boards (PCBs) is a promising source of such CRMs, but most of the latter are not recoverable with current industrial recycling practices [1]. One major issue to be solved: the overall composition, and therefore the value, of this urban mine comprising of PCBs. Indeed, this is necessary for stakeholders to build accurate and viable business models to mitigate criticality issues and enable their recycling. To solve this, one need to build a database of the chemical composition of the millions of electronic components (ECs) to be found in such e-wastes. However, full-element analysis of ECs lacks standardization in literature and is often proprietary piece of information from electronic manufacturers [2].

Hence, our team propose to demonstrate the feasibility of high throughput X-ray fluorescence imaging of waste ECs using synchrotron radiation to populate a reference database of ECs (@BM23, ESRF). This would enable online estimation of the e-waste flux composition, and educated sorted strategies toward sorting bin that are simpler, less variable and more concentrated in targeted CRMs [3].

Our team also investigate hard x-ray hyperspectral scans as signatures to access a reference components entries in the database. Such scan is acquired online using a transmission setup with an x-ray tube and a pixelated photon counting detectors, and provide a more unique, composition-related signature than an RGB image.

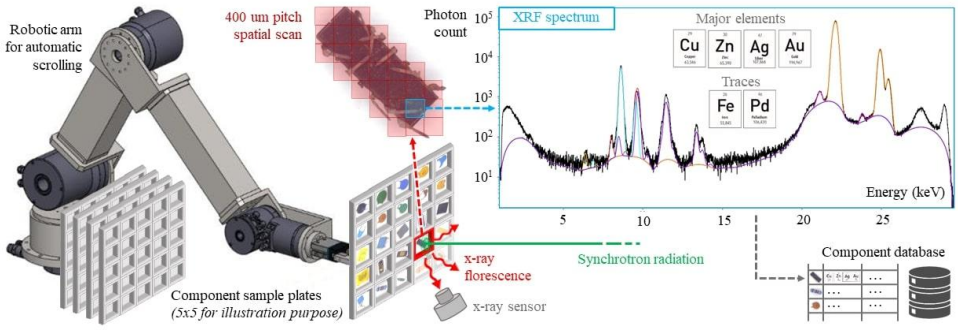

Figure: Experimental setup suggested for high-throughput XRF at BM23 of electronic components

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Operando studies of the dynamic evolution of complex intermetallic phases of recycled Al alloys in the solidification process

Jiawei Mi[1°]
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Globally, the annual demand for primary aluminium (Al) is estimated at ~70 million tons and the uses of secondary Al or recycled Al consume only ~7% of the energy compared to the primary Al. Hence, it is vital to maximise the reuses and recycling rate of secondary Al to reduce the greenhouse gas emissions in Al industry. In almost all commercial Al alloys, Fe is the most common detrimental element, which is often accumulated in the sorting and remelting processes and form different type of brittle Fe-rich intermetallic phases, damaging greatly castability and mechanical properties of the alloys. It is critical to develop efficient and effective methodologies to restrict the detrimental Fe phases or to modify/change the damaging phase morphology into beneficial ones in order to improve the mechanical properties.

Here, I present the recent operando research work of using the fast synchrotron X-ray diffraction and tomography techniques at the ESRF plus machine-learning assisted phase segmentation methods to study the 3D nucleation dynamics of the Fe phases in a typical multiple-component recycled Al alloy; and how co-growth of the multiple Fe phases lead to the formation of the complex and convoluted 3D Chinese-script phases. In addition, the beneficial effects of applying ultrasound to control the primary Al dendrites and to alter the Fe phase growth dynamics as well as the final 3D morphology were also discussed and elucidated in this work.

Probing Anisotropy-induced Toughening in $Ti_3C_2T_x$ MXene Materials using Wide-Angle X-ray Scattering

K.A.S. Usman[1], K.M. Sisican[1], R.P. Ibabao[1], R.M. Ramos[1], J.M. Razal[1]

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Two-dimensional (2D) transition metal carbides and/or nitrides, referred to as MXenes, have recently gained considerable attention due to their unique combination of properties, such as excellent electrical conductivity and high electrochemical capacitance. However, translating these nanoscale properties into bulk or macroscopic structures remains a challenge, as both pure and composite MXene assemblies often suffer from performance trade-offs. For instance, while pure $Ti_3C_2T_x$ MXene can yield films and fibers with outstanding conductivity (>10,000 S cm^{-1}), their tensile strength remains relatively low (*i.e.*, around 34 MPa for films and 40 MPa for fibers). To overcome this drawback, we explored the use of bridging additives (*e.g.*, polydopamine [1], nanocellulose [2], and silk nanofibrils [3]) between MXene layers to replicate the hierarchical architecture of natural materials like nacre. This strategy referred to as "sequential bridging" (SB), enhances the mechanical robustness of free-standing MXene films and fibers while preserving electrical and electrochemical performance. For example, fibers fabricated using this approach demonstrate tensile strengths >200 MPa, conductivities >8000 S cm^{-1}, and volumetric capacitances >900 F cm^{-3} [4]. In addition, we emphasized the role of preserving sheet alignment, tracked using Wide-Angle X-ray Scattering (WAXS), in ensuring uniform electrical contact and enabling rapid ion transport. Methods for controlling alignment, such as shear-induced orientation during processing or the application of tensile forces after fabrication, were also discussed. These findings offer valuable insights for developing durable and multifunctional MXene-based materials tailored to specific applications. This technique may be extended to other 2D nanomaterials, fostering their broader implementation in next-generation technologies.

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Electrostatic potential of latex sphere using off-axis electron holography

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Electrostatic potential, including both that contributed by electron-beam-induced specimen charging and intrinsic material-related mean inner potential (MIP), is crucial because it is influence the reaction between charged particles, chemical reactivity, and dielectric properties. Off-axis electron holography is a powerful TEM technique that can be used to map local variations in electron optical phase shift, which are in turn sensitive to electrostatic potentials and magnetic fields. In the absence of magnetic contributions to the phase shift, the recorded phase is proportional to the projected electrostatic potential within and outside the specimen. Insulating nanoparticles with simple geometries are ideal objects for the study of specimen charging in the TEM.

Polystyrene latex beads were examined temperature-dependent behavior of the MIP and electron-beam-induced charge from room temperature down to 5.3 K in a FEI Titan G2 TEM at 300 kV. The diameter of latex spheres is in the range of 230 nm to 600 nm. By using a model-independent approach for the quantification of the spatially-dependent projected charge density from a recorded phase image [1,2], the amount of positive charge on the sphere at each temperature was determined. Isolating the electrostatic potential contributed by the electron-beam-induced charge, the MIP was obtained at high precision, revealing a significant increase of 16.8% as temperature decreases from RT to 5 K.

In addition, this talk will present the quantification of electron-beam-induced charge on MgO nanocubes reflecting the amount of oxygen vacancies on the surface, which is essential for catalytical reaction [3].

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CONTRAST ENHANCING STAINING AGENTS TO IMAGE BIOFILMS USING LAB-BASED MICRO-COMPUTED TOMOGRAPHY

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Bacteria and other microorganisms form biofilms upon and within materials, affecting their physical and chemical properties, such as pore-scale clogging, mineral precipitation, or gas production. Upon the surface, biofilms can be studied by electron and optical microscopy. However, within opaque materials, other techniques such as micro-computed tomography (µCT) are necessary. Unfortunately, biofilms consist of water and other light materials, making them nearly indistinguishable from water. Contrast-Enhancing Staining Agents (CESAs) can improve visualization, but existing CESAs mainly stain the water phase, can precipitate, are toxic, or are time-consuming. In our research, five CESAs: Mono-WD POM, Hf-WD 1:2 POM, isotonic Lugol, Hexabrix® and CA4+ were evaluated for their ability to enhance the contrast of biofilms with conventional lab-based µCT. Two CESAs, Hf-WD 1:2 POM and isotonic Lugol, effectively increased the attenuation and contrast of the biofilm. It enabled the visualization of biofilms that could otherwise not be (fully) visualized. It included cyanobacterial biofilms colonizing sandstone [1] and biofilms of heterotrophic bacteria colonizing the pores of sand filters [2] (Fig. 1). Isotonic Lugol produced stronger attenuation but may induce shrinkage, whereas Hf-WD 1:2 POM could potentially induce swelling. Furthermore, spectral CT at TESCAN XRE was applied to further confirm the uptake of iodine (for biofilms stained with isotonic Lugol) and tungsten and hafnium (for biofilms stained with Hf-WD 1:2 POM) and to enhance further discrimination between the phases. While iodine signals correlated clearly with biofilm presence, tungsten and hafnium detection were less consistent. Future research should focus on the CESA-biofilm interaction and apply these CESAs within the industrial and research fields. Initial experiments have already been conducted in the scope of the BugControl project at Utrecht University and within the EXCITE Transnational Access at Ghent University.

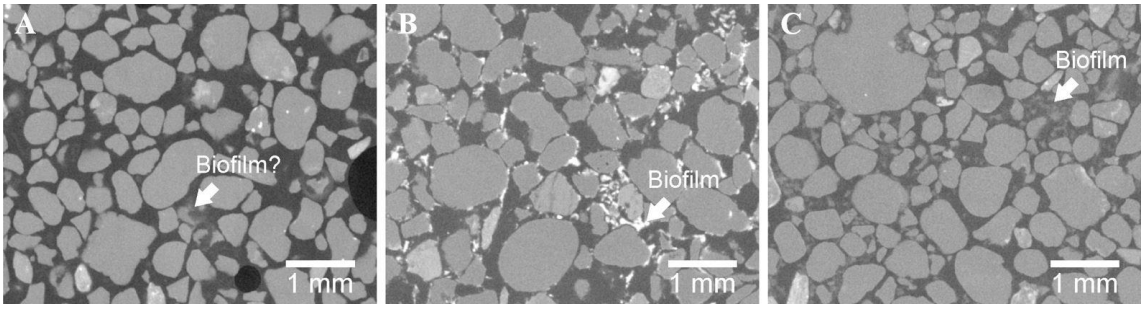

Figure 1: Biofilms colonizing sand filters, visualized without CESA (A), with isotonic Lugol (B) and Hf-WD 1:2 POM (C) (adapted from [2])

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Looking Inside: Multiscale Diffraction Imaging from Millimeters to Nanometers

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Macroscopic properties of advanced materials are governed by hierarchically-organized structures such as grains, subgrains, dislocations, and phase domains, spanning nanometers to millimeters. Understanding their dynamic interactions is key to property control and multiscale model validation. Dark Field X-ray Microscopy (DFXM) is a synchrotron-based diffraction imaging technique that enables non-destructive, three-dimensional mapping of orientation and strain in bulk materials, with submicron spatial and microstrain-level angular resolution. Using an objective lens to magnify diffracted X-rays, DFXM delivers full-field imaging of individual grains with high temporal resolution, enabling in situ studies of grain growth, dislocation dynamics, recovery, and recrystallization. Recent methodological advances have integrated DFXM with upstream grain mapping techniques such as 3DXRD, DCT, and labDCT, enabling seamless, multiscale targeting without dismounting the sample. At the ESRF, the upgraded ID03 beamline, part of the EBSL2 program, now supports both monochromatic and pink-beam modes, enhancing time-resolved and multimodal capabilities. We demonstrate the potential of this approach through recent studies on metal alloys, ceramics and semiconductors, highlighting how DFXM, combined with grain mapping, reveals new insights into structure–property relationships across length and time scales.

Synchrotron diffraction and tomography analysis of damage evolution in AA2050 under high-temperature loading

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The formability of Al2050 alloy is critical for manufacturing large and thick components while maintaining its outstanding performance [1]. Previous studies showed that pores and cracks occur during the hot deformation of this alloy and the pore growth follows three different paths (exponential, decelerated, mixed) [2]. To link the damage development during high-temperature loading with the alloy microstructure evolution, an in-situ tensile loading at 480 ºC was performed on this alloy using synchrotron diffraction and tomography, i.e. diffraction contrast tomography (DCT) to provide 3D grain maps at ~1.1 µm resolution and phase contrast tomography (PCT) to characterize pores and intermetallic phases with 0.55 µm voxel size and scanning 3DXRD (s3DXRD) to obtain 2D grain and elastic strain maps at 0.5 µm resolution. The pore density and volume fraction were quantified as a function of macroscopic strain up to 20% and three pore formation mechanisms were identified: growth from pre-existing pores, fracture of the intermetallic particles, and nucleation of new pores. The characteristics of the pore evolution are then linked with the grain structure (grain boundaries, orientations and strains) characterized by DCT and s3DXRD. Additionally, the grain maps show newly recrystallized grains, suggesting the presence of dynamic recrystallization. To exclude the possible explanation by annealing recrystallization, an in-situ annealing experiment at 480 ºC without external loading was performed and the results confirmed no recrystallized grains. This study demonstrates that correlating synchrotron grain mapping techniques with tomography offers comprehensive insight in linking the damage development with the microstructure evolution under high-temperature deformation.

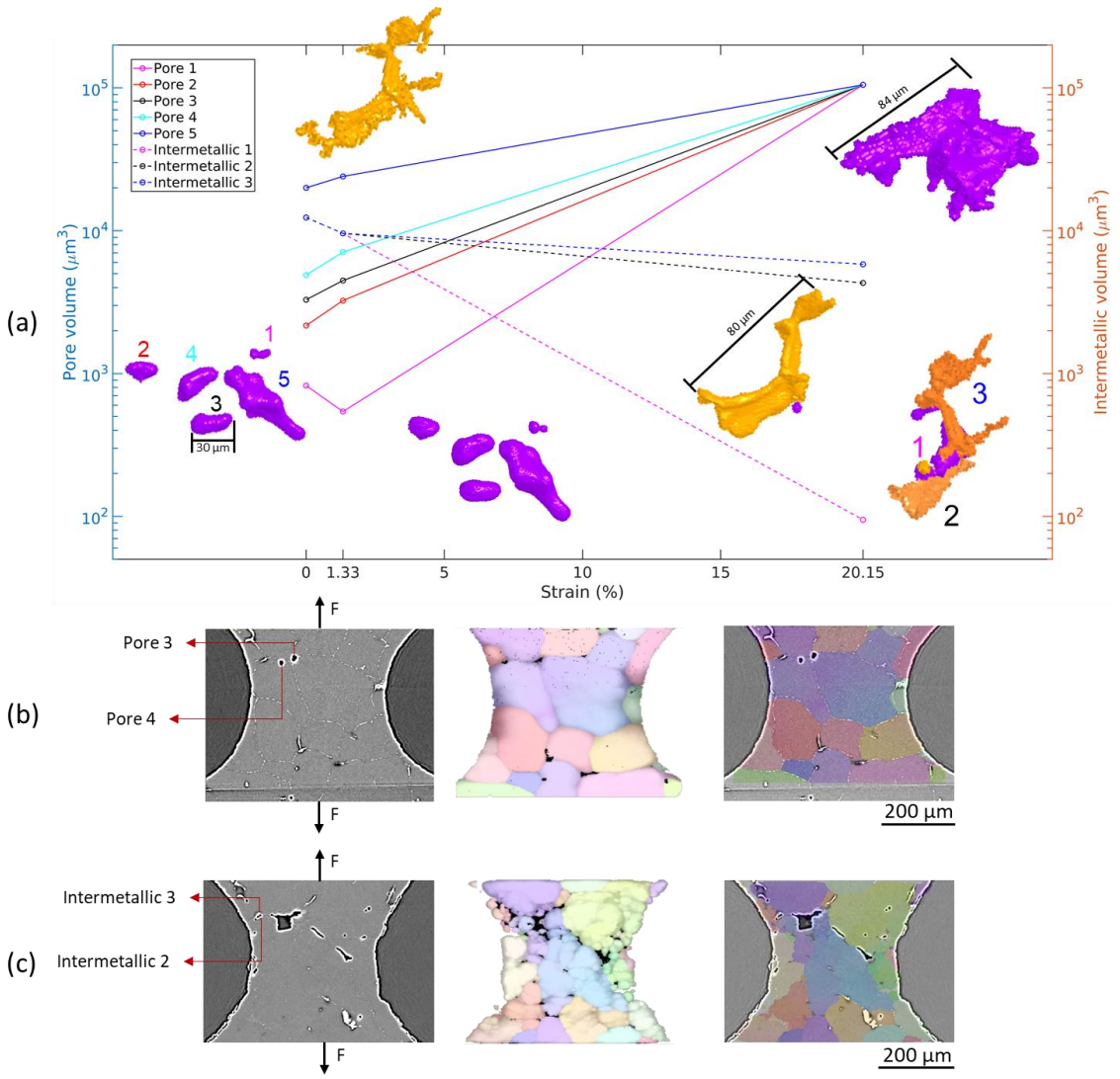

Figure 1: Damage development analysis through the three states measured (initial state – 0% strain, 1.33% strain, and final state – 20.15% strain). (a) Volume evolution of pores and intermetallic particles, showing the pores (no. 1-5) grow and connect with each other before finally coalescence into one large single pore while the intermetallic particle fractures into small ones with new pores forming in between. For (b) initial state and (c) final state, 2D slices showing PCT (left), overlays of grain map and completeness (middle) and PCT and grain map reconstructed by DCT colored by IPF-Z (right). Many sub-grains and newly recrystallized grains are observed in the final strained state.

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Recycling of CIGS solar modules and electrochemical recovery of Cu and CRMs from waste

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Copper-Indium-Gallium-di-Selenide or CIGS panels are one of the promising alternatives in the market for thin-film solar technologies. CIGS panels are lightweight, durable and use less semiconductor materials as compared to Si-based PV modules [1, 2]. According to EU CRM materials report 2023, indium gallium and copper which are raw materials for CIGS panels are rated as critical or strategic materials subject to their high supply risk and moderate economic importance [3]. The development of robust recycling methodologies for copper indium gallium selenide (CIGS) photovoltaic panels and associated production waste presents a viable pathway towards securing the supply of critical raw materials (CRMs). While a range of recycling processes have been explored, many current approaches demonstrate limitations. Specifically, challenges remain in achieving both efficient material separation and high material purity within a single process. Furthermore, certain methodologies necessitate operation under highly toxic conditions, resulting in the generation of secondary waste streams which require further management. Within this research electrochemical leaching for separation and recovery of materials from CIGS solar panels is studied. The scope of study is to determine efficiency of semicomductor coating separation from glass in $H_3C\text{-}SO_2\text{-}OH$ (Methanesulphonic Acid). Using ICP-OES/MS and XPS techniques the dissolution and de-coating efficiencies are calculated. The investigation includes the observing effective separation of the glass and coating material at different acid concentrations in novel drum electrode[4].Furthremore, studies have been conducted to recover Cu from mixed CRMs solution in high purity. Purity upto 98% for Cu was achieved. Further optimisation of parametes is needed to improve purity and yeild. Within the study, sustainability analysis of the processes from ecological, safety and social aspects are briefly discussed.

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Recent advancements in neutron imaging

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The material characterization by neutron imaging reached a new level after developing innovative techniques using different contrast mechanisms than the common beam attenuation. In this way properties of materials and complex systems can be resolved by position sensitive mapping of diffraction, small-angle scattering and refraction signals. In addition, the improved spatial and time resolution of the detector systems allows for micro tomography studies and 3D dynamic investigations.
Applications related to 2D and 3D visualization of material phase heterogeneities, texture, fluid dynamics, magnetic structures and phase transitions in applied materials will be presented [1].

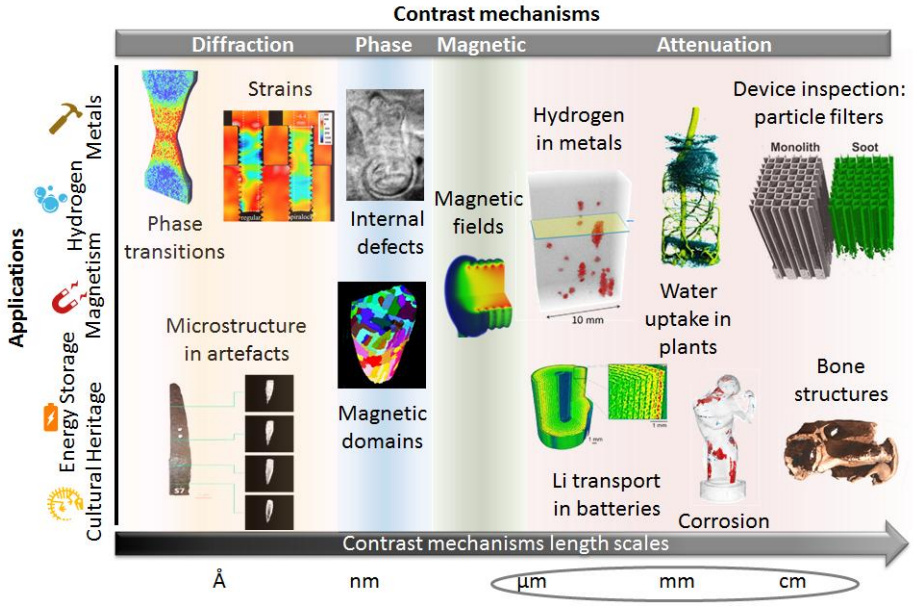

Figure 1: Different contrast mechanisms can be used to explore various length scales in materials and to study their properties and related processes. The relation between contrast mechanisms and different application fields is presented. The length scale presented on the lower axis relates to the corresponding contrast mechanism specified on the upper axis. For the attenuation-based image techniques the large length scale was emphasized by grouping the scales from µm to cm.

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In-situ Phase-Contrast Tomography and Small-Angle X-ray Scattering at synchrotron sources to unveil multiscale-structured bone scaffolds made of bioglass nanoparticles for regenerative biomaterials.

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A new-generation of synthetic bone scaffold is tailored using a bricks-and-mortar approach from bioactive glass nanoparticles BGNps (SiO_2-CaO-P_2O_5 doped with metal ions, the bricks), and customized polymers (PLA, poly (lactic acid), the mortar). Used as synthetic implants for substitutive and regenerative therapies targeting mandibular osteoradionecrosis (ORM), they must promote bone formation and cell adhesion, while exhibiting high porosity, adequate mechanical strength, and pro-angiogenic coupled with antibacterial properties. Freeze-casting solutions of BGNps and PLA derivatives can result in hybrid nanocomposite scaffolds[1], with a multi-scale porosity, offering improved mechanical properties and proper auto-catalytic degradation.

To shed light on the mechanisms behind the formation of the hierarchical structure of these scaffolds, the synthesis of BGNps derived from Stöber silica was studied using *in-situ* SAXS at synchrotron facilities (ID02@ESRF[2] and SWING@SOLEIL). The SAXS data revealed the preorganization of the particles in solution prior to freeze-casting.

Subsequently, thanks to a custom-built sample chamber, fast X-ray phase-contrast tomography *operando* experiments were performed using synchrotron beams (ID19@ESRF). They allowed to follow the controlled growth via a freeze-casting process of centimeter-cubed scaffolds made from nanoparticles (bricks) and polymers (mortar), at high resolution. We pictured the fabrication process from the initial suspension of nanoparticles to the finalized porous material, obtained via lyophilization that was also studied operando with X-ray tomography.

Keywords: Small-Angle X-ray Scattering, X-ray Tomography, Freeze-casting, Synthetic bone scaffolds, Bioglass nanoparticles.

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Probing carbon composite microstructure with speckle-based X-ray imaging – Workshop "Multi-probe imaging for materials"

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For speckle-based X-ray imaging, structures such as sandpaper are placed in a partially coherent X-ray beam to generate speckle patterns on a downstream detector. The sample is then added and the resulting local distortions to these patterns, due to the sample's interactions with the beam, are analysed by a dedicated algorithm. Due to their different effects on these patterns, maps of refraction, attenuation, and dark-field can be calculated [1]. This presents an advantage over propagation-based X-ray imaging methods, where attenuation and refraction cannot be treated independently, and the dark-field modality is either inaccessible or much more difficult to retrieve.

In particular, the dark-field modality visualises small-angle X-ray scatter by microstructures below the resolution limit of the imaging system. It may thus be of particular interest for the analysis of fibrous materials such as carbon-fibre-reinforced polymers (CFRPs), as it can provide microstructural information without a need to directly resolve individual fibres.

Besides an introduction to the imaging technique and the image retrieval approach used by the "Unified Modulated Pattern Analysis" (UMPA) model [2], we will present results from two speckle-based imaging experiments on CFRP samples performed at the ID19 beamline of the ESRF. The first of these explored directional dark-field imaging, showing that fibre directions in CFRP samples can be reliably extracted by analysing the local anisotropy of small-angle scattering characteristics (Fig. 1a,b). Furthermore, the used image retrieval method allows striking a balance between dark-field SNR levels and spatial resolution [3].

The second experiment demonstrates that the dark-field modality provides increased sensitivity for detecting barely visible impact damage (BVID) in two CFRP samples (Fig. 1c,d). Subsequent analysis via computed laminography shows that damage in the two samples is primarily due to matrix-cracking and delamination, respectively [4].

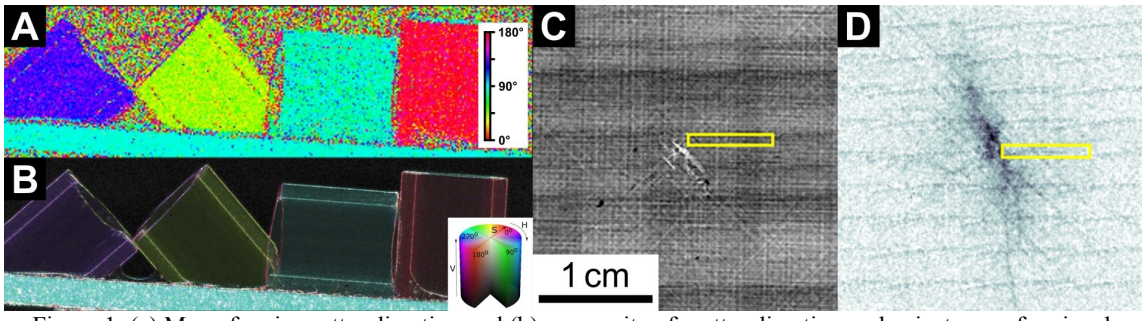

Figure 1: (a) Map of main scatter direction and (b) composite of scatter direction and anisotropy of variously oriented CFRP tubes. (c) Transmittance and (d) dark-field of damaged area of CFRP panel (matrix cracking).

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Poster: Microstructure and Mechanical Performance of Molongó Wood: Unveiling an Unexplored Amazonian Timber

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In this work, we provide the first assessment of the microstructure and mechanical properties of Malouetia Tamaquarina (Aubl.) (Apocynaceae), commonly known as Molongó wood. The timber grows in the Amazonian region in blackwater areas and is employed in arts and crafts by indigenous populations.

The microstructure was characterised via scanning electron microscopy (SEM), X-ray computed tomography (XCT), and mercury intrusion porosimetry (MIP). In particular, XCT scans were performed at two different resolutions (3.1 µm and 0.594 µm) to evaluate the cell wall thickness and pore size at different length scales. The wood exhibits a highly porous cellular structure, with porosity values reaching up to 87.8%, as measured through XCT and MIP. Rectangular cells are observed with walls ranging from 2.6 to 10 µm in thickness and pores diameter between 20 and 80 µm, exhibiting a similar morphology to that of softwood trees (shown in Figure 1). Additionally, pits are observed along the cell walls, which regulate water transport across the cells during the plant's cyclic flooding.

Mechanical testing was employed to assess the response in tensile, compressive, flexural, and Charpy impact loading. A strong anisotropy is observed, where the longitudinal direction exhibits the highest strength and stiffness due to the wood's cellular structure. Tensile and flexural moduli reach 0.85 GPa and 0.82 GPa, respectively. In contrast, the impact resistance is highest in the transverse direction due to the deformation of the pits. The lignin content of the wood is 27.1%, which is in line with that of lightweight angiosperm woods.

The results suggest that Molongó wood combines low density and favourable specific properties, indicating potential for lightweight, sustainable structures.

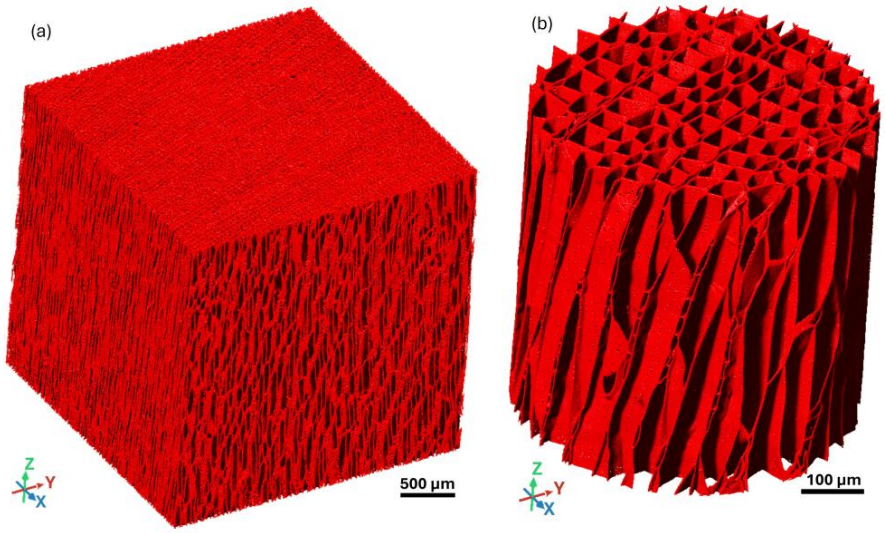

Figure 1: Wood morphology obtained via XCT imaging at different scales: (a) micro-CT and (b) nano-CT.

Poster: Synchrotron diffraction contrast tomography for reconstructing local orientations in deformed microstructure

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To extend the applicability of synchrotron diffraction contrast tomography (DCT) towards more plastically deformed materials, we have developed a forward-model based reconstruction method to reconstruct grain shapes and local orientations in materials exhibiting levels of intragranular orientation spread that can no longer be handled with the conventional Friedel pair based indexing and tomographic reconstruction approach. This method consists of seed and regional indexing, in which an exhaustive searching and fitting of orientations is first performed to index the seed orientation and then a regional indexing by testing a list of local orientations around the seed orientation is carried out to maximize the completeness. The capability of this novel method was benchmarked and compared with the reconstructions based on the conventional Friedel pair matching and tomographic reconstruction method using samples made from fully recrystallized Al-Cu alloy, moderately deformed α-Ti alloy and 10% creep ruptured Fe-Au alloy. The results show that this method has a great prospect in overcoming the deformation constraint and can reconstruct reasonably well the intragranular orientations. It is also suitable for multi-phase reconstruction and both box-beam and line-beam acquisition geometries. The implementation has been made flexible supporting the use of single or multiple GPU machines. The strengths and weaknesses of the current forward-model based reconstruction have been discussed in detail with respect to the conventional Friedel pair matching method. To fully exploit and complement the strengths of the two methods, the code to implement the current forward-model based reconstruction has been fully integrated with the existing DCT code and is open sourced for beamline data processing.

Keywords: Diffraction contrast tomography; Grain mapping; Forward model reconstruction; Synchrotron X-ray diffraction; Intragranular orientation

Poster: Tailoring Pore Structure in Porous Carbon toward Enhanced Lithium-Ion Storage Performance

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Conventional graphite anodes face limitations in capacity and rate capability, motivating sustainable carbons with hierarchical porosity to shorten lithium-ion transport paths and stabilize interfaces[1,2]. Herein, biomass-derived porous carbons were prepared by hydrothermal pretreatment followed by high-temperature activation to regulate pore architecture for lithium-ion battery anodes. The samples ZPC, NZPC, and NZHPC exhibit specific surface areas of 1213/2242/2360 m^2/g, total pore volumes of 0.48/0.93/1.08 cm^3/g, and micropore fractions of 95.8/94.6/88.9%. Adjustment of the solution chemistry during hydrothermal pretreatment promotes the subsequent activation, thereby increasing the surface area and average pore size and introducing mesoporosity, while retaining a high density of micropores. The optimized NZHPC delivers a reversible capacity of 1624.5 mAh/g at 50 mA/g, and maintains 656.1 mAh/g after 800 cycles at 1 A/g as well as 154.3 mAh/g after 1000 cycles at 5 A/g. Rate and cycling data indicate lower polarization and more stable kinetics compared with ZPC and NZPC. The approach used in this study led to outstanding materials that largely surpass the capacity and cyclability observed in standard graphite anodes, which is attributed to the particular pore structure obtained through our methodology. However, to fully understand the behavior of these materials, in situ synchrotron SAXS/WAXS measurements will be conducted in the future to probe pore evolution and validate the transport mechanism. These planned experiments aim to provide direct evidence for a clear correlation between pore architecture and electrochemical performance and to support a practical route to high-performance biomass-derived carbon anodes through rational control of pore size distribution.

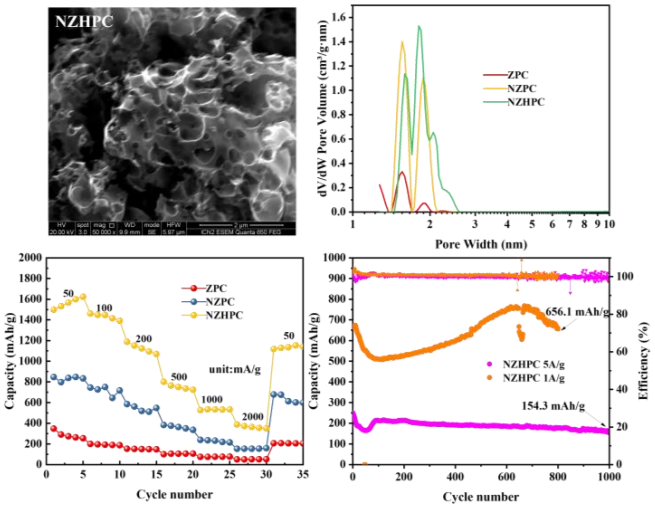

Fig. 1 Morphology and electrochemical performance of porous carbons.

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Poster: Cellulose nanocrystals /Co-doped ZIF-8 Derived Mesoporous Single-Atom Sites for High-Performance ORR

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Most ZIF-derived carbons are rich in micropores, which slows oxygen transport to single-atom sites [1, 2]. We report a template-free, CNC-guided route that builds mesopores at the precursor stage and fixes atomically dispersed Co-N_4 centers in carbon. During in situ assembly, Co/ZIF-8 nucleates densely on cellulose nanocrystals and forms thin, open shells. After pyrolysis and acid washing, these shells turn into interconnected 4-6 nm channels within a conductive framework. XAS shows Co bonded to nitrogen and no Co-Co contribution, confirming isolated Co atoms [3]. Nitrogen sorption reveals that mesopore volume increases with the Co/Zn ratio while the micropore fraction stays nearly constant, so the extra porosity mainly comes from mesopores set by the assembly step rather than created by leaching. This architecture improves access to Co-N_4 sites and wets the electrode more effectively [4]. In alkaline media the catalyst gives a high half-wave potential (~0.85 V), a 55.9 mV dec^{-1} Tafel slope, and stable activity under repeated LSV cycling (100000 cycles). Using cellulose nanocrystals-a green, water-processable feedstock-avoids hard templates and harsh activation, lowers cost and waste, and is well suited to scale-up. This work shows a simple path to durable Co-N_4 catalysts with programmed mesoporosity.

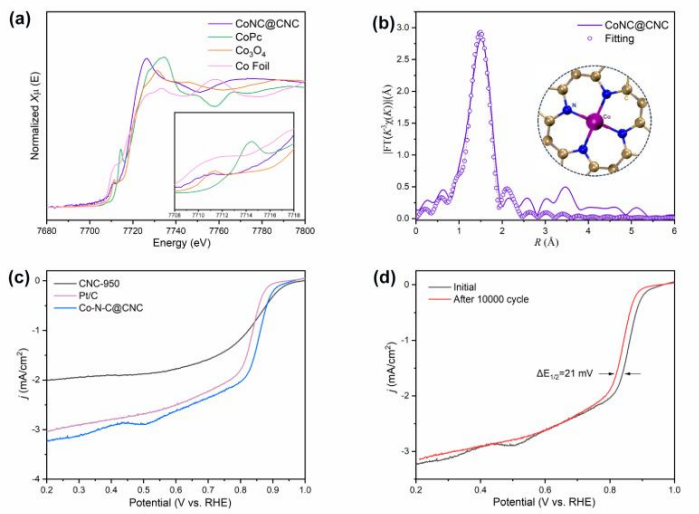

Figure 1: (a) XANES of the Co K-edge and (b) FT-EXAFS fitting curves of Co K-edge. (c) LSV curves for ORR in 0.1 M KOH and (d) Cyclic stability curve of Co-N-C@CNC

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Poster: Microstructural Analysis and Modeling of Alkaline Electrolyzer Electrodes from Advanced Imaging Techniques

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Hydrogen, as a high-potential energy carrier, stands out as a key solution for storing and regulating electricity generated from renewable sources [1]. In this context, ADELE HYDROGEN™ develops cutting-edge electrodes for alkaline electrolyzers, avoiding the use of noble metals or critical raw materials. However, further innovation is needed, particularly in optimizing electrode topology to address performance limitations such as ohmic and activation losses.

A deeper understanding of the multiphysics phenomena governing water electrolysis—especially gas bubble dynamics—is critical to improve electrolyzer efficiency. Ohmic losses, for instance, are exacerbated by gas bubble accumulation, which disrupts electron and ion transport within the porous electrode structure. To mitigate these effects, high-resolution imaging of the electrode's complex, often heterogeneous microstructure is essential. This enables precise segmentation of phases (solid, void) and quantification of structural parameters like porosity and tortuosity, which directly influence bubble nucleation, growth, and detachment.

In this study, we combine advanced imaging techniques—X-ray tomography, FIB-SEM, and electron microscopy—with numerical simulations to extract key structural parameters. Our objective is to use these data to reconstruct the 3D electrode microstructure and develop 3D simulations of multiphysics phenomena at the microscopic scale. These microscopic insights will then be upscaled to inform a macroscopic model, enabling the study of overall electrolyzer performance and stability under real-world operating conditions.

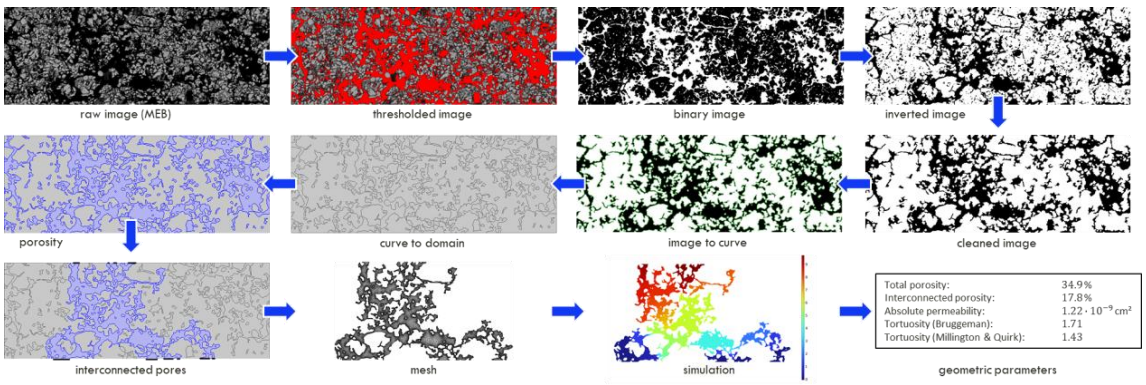

Figure 1: From raw imaging to the determination of geometric parameters.

References

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Poster: Capacitors at low temperatures revealed by cryo-synchrotron X-ray phase contrast microtomography

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Electronic devices are used in extreme conditions such as in outerspace or polar regions, exposing them to extreme low temperatures. [1,2] Understanding their behaviour is important for optimizing their functionality and to better design and manufacture individual electronic components. Capacitors are such electronic components sensitive to temperature. Conventional low temperature electrical characterization of capacitors typically has no spatial information. Here we combine synchrotron cryomicrotomography down to 77 K (carried out at beamline BM18), and electrical characterization down to 20 K (carried out at beamline BM28), to correlate the electrical and structural properties of commercially available capacitors. We characterized multilayer ceramic, Ta2O5 and Al2O3 electrolytic capacitors. Our experiment reveals that a change in capacitance at low temperature is dominated by structural changes than from possible intrinsic changes such as in resistivity, crystal structure or dielectric permittivity. Our result shows a new insight towards the functionality of commercial capacitors at low temperatures and provide important benchmark for developing and designing future capacitors for extreme environmental conditions.

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