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Keynote - Precise quantitative mineral analysis by EDS-SEM

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The advance of modern EDS detector technology has unlocked significant potential for quantitative mineral analysis by EDS-SEM. Because count rates of large EDS detectors far surpass those of the WDS-based electron microprobe, quantitative mineral analysis by EDS-SEM has the potential to be very precise, while at the same time enabling rapid sample throughput. Here we present a case study of quantitative mineral analysis by EDS-SEM. Over the course of seven months, more than 100 samples of mantle peridotites were analysed at Cardiff University using a FEG-SEM equipped with dual Oxford Instruments 150mm² EDS detectors. These peridotites represent the first long section of mantle rocks recovered from the ocean basins, and provide a unique opportunity to reconstruct the processes of mantle melting as well as the mantle's oxidation state. However, because the constituent minerals of mantle peridotites vary little in composition, high precision is required to resolve variations and hence reconstruct these processes. To monitor precision, the same external peridotite standard was analysed over the course of the study, with over 500 analyses each of olivine, orthopyroxene, clinopyroxene. We found that the precision of critical compositional parameters ranged from 0.11% (2s; Fo content of olivine) to 0.13% (2 σ ; Mg# of orthopyroxene). Precision of minor elements such as Al in orthopyroxene (~4.4 wt%) and Ni in olivine (~0.5 wt%) was 2.7% and 12.4% (2 σ) respectively. Cr-spinel Fe³⁺/ Σ Fe, calculated from stoichiometry following EDS analysis, matched independent Mössbauer spectroscopy data for Fe³⁺/ Σ Fe exceptionally well, and better than previous electron microprobe analyses. The achieved precision, coupled with the high beam stability, has enabled us to clearly resolve compositional trends on a range of scales in this unique sample set. This demonstrates that quantitative mineral analysis by SEM-EDS is a very powerful approach for precise quantitative mineral analysis.

No WDS? No Problem! Why Do Geoscientists Continue to Overlook EDS?

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Element mapping of the lawsonite to epidote transition

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Lawsonite is a significant reservoir for H₂O (<11.5wt%) and contains geochemically-important trace elements and transition metals[1]. Its breakdown releases these components from the subducting slab into the overlying mantle wedge and ultimately contributes to arc magmatism. Epidote group minerals are common reaction products of lawsonite decomposition and, like lawsonite, incorporate Sr, Pb, rare earth elements (REEs), and H₂O (~2wt%) (e.g.,[2,3]). Despite the significance of this transition, the partitioning of elements during this transition remains poorly understood beyond water loss.

To investigate the element partitioning behavior across the lawsonite-to-epidote transition, we employed EPMA-WDS and LA-ICP-MS analysis on a suite of metabasalts from New Caledonia, including lawsonite, lawsonite+epidote, and epidote blueschists. EPMA-WDS analyses of lawsonite show that Ti-sector zoning is common[1]; LA-ICP-MS mapping reveals that Th, Pb, Sr, Y, and La also display sector zoning in lawsonite. Zoning can be used to track the lawsonite-to-epidote transition because epidote typically contains relict (or partially-reacted) lawsonite inclusions and/or "lawsonite ghosts" – tabular (i.e. lawsonite)-shaped epidote domains with lower-Fe and higher-Al than the surrounding epidote. An analyzed lawsonite-epidote blueschist displays Th, U, Pb ghost-sector zoning in low-Fe epidote and Sr, Y oscillatory zoning in the host-epidote. Higher-temperature epidote blueschists contain lawsonite inclusions with poorly defined ghost textures or lawsonite-absent with multiple generations of lawsonite ghosts with varying Fe content. Both show preferential elemental partitioning of Sr, La, and Y into the host epidote rims.

The relict compositional zoning and observed replacement textures have not been previously reported and would not have been possible without high-resolution element mapping. This work provides new insights into the chemical evolution of subduction-zone minerals and the processes controlling fluid-mediated mass-transfer in high-pressure environments, particularly the behavior of trace elements and transition metals.

[1] Ueno (1999)

[2] Spandler et al. (2003)

[3] Enami et al. (2004)

From Sparse Measurements to Full Maps: Machine Learning Prediction of Mineral Phase Data

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Scanning electron microscope (SEM) – energy dispersive spectroscopy (EDS) data is usually only collected for a few interesting areas of a larger sample. The challenge lies in bridging the gap between this data and the rest of the sample, particularly in regions of interest where no EDS data was collected. This study presents three distinct lithologies (granite, Muschelkalk and sandstone) analyzed using MaPro software (Jiang et al. 2022). This software uses a physics-informed decision tree to analyze EDS data in conjunction with high-resolution BSE data for each lithology. Once thresholds have been applied, the various phases are segmented based on the EDS maps, thus providing the ground truth for subsequent prediction. Compared to the original EDS data, the ground truth enables pixel-wise phase analysis, which is crucial for data processing. A machine learning (ML) algorithm based on random forest classification was trained using MaPro phase analysis to predict phases over a larger area. The results show a good match with the MaPro-based ground truth (Figure 1). For homogeneous samples such as sandstone and granite, the results correspond more closely to the ground truth than for a heterogeneous sample such as Muschelkalk. The fine-grained regions of the samples produce the largest errors in the MaPro software and, consequently, in the ML prediction results. In these areas, phases with similar mineralogy are more difficult for the ML tool to distinguish and typically require more ground-truth data than minerals with more distinct mineralogical differences. Overall, the results provide a reliable overview of the entire sandstone sample as well as large areas of the granite and Muschelkalk samples, enabling mineral phase analysis across extensive sample surfaces within short measurement times.

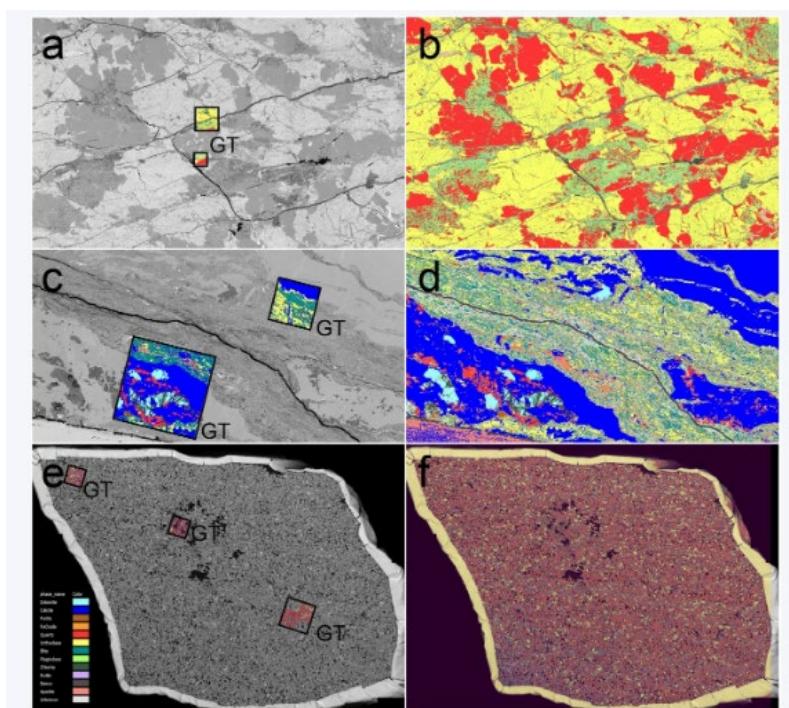


Figure 1: SEM-BSE images with pixel-wise labelled ground truth (GT) from MaPro software for granite, Muschelkalk and sandstone (a,c,e) and ML prediction (b,d,f).

Quantifying Facies-Controlled Pore Connectivity in Heterogeneous Porous Media with Alloy Intrusion Porosimetry (AIP)

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Porosity in rocks has traditionally been measured with volumetric techniques such as mercury intrusion porosimetry (MIP) or gas pycnometry. While these methods provide reliable estimates of bulk porosity, they cannot resolve pore space connectivity, which critically controls reservoir quality, fluid transport, and production rates. To address this gap, the Alloy Intrusion Porosimetry (AIP) method was developed. AIP is based on the injection of low-melting-point alloys into interconnected pore space, enabling direct characterization of pore connectivity down to the nanometer scale (Klaver et al., 2015).

Unlike MIP, AIP uses Field's metal (51% In, 32.5% Bi, 16.5% Sn) as the infiltrating medium. The alloy is heated above its melting point and injected under pressure into the connected pore system. Subsequent cooling under pressure solidifies the alloy, preserving pathways for imaging. Broad ion beam–scanning electron microscopy (BIB-SEM) of impregnated samples then provides high-resolution visualization of the three-dimensional pore structure. The combined AIP–BIB-SEM approach resolves pore throats as small as 3 nm, significantly extending the range of image-based pore network analysis.

Beyond qualitative insights, the method yields quantitative microstructural parameters such as tortuosity, essential for modeling flow and transport. Linking pore connectivity with mineralogy and lithofacies, derived from EDS- and AI-assisted phase analysis, further enables upscaling strategies. The MaPro software (Jiang et al., 2022) supports this integration by providing automated mineral phase characterization (Fig. 1).

Overall, AIP bridges the gap between traditional porosimetry and direct imaging, combining connectivity analysis, microstructural quantification, and mineralogical correlations to improve predictions of reservoir performance in hydrocarbon production and fluid storage applications.

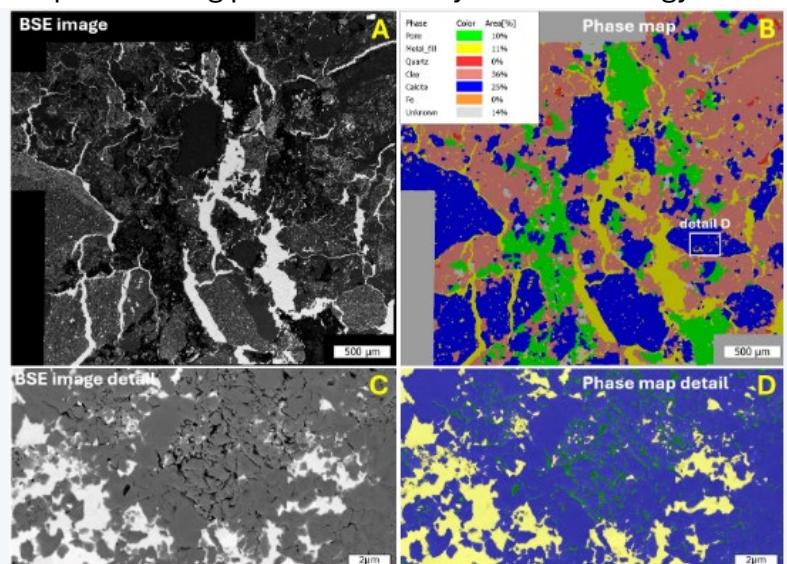


Fig. 1. BSE image of a marl sample partly intruded by an alloy (A), with corresponding phase map (B), BSE detail image (C), and detail phase map (D); see legend in B for phase labels.

A New Approach for X-ray Mapping Unprepared and Uncoated Samples: How SEM-BEX Helped Locate Some of the Solar Systems Oldest Zircons

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Angrites are among the oldest igneous rocks in the solar system, providing unique constraints on early planetary differentiation. However, the scarcity of such extraterrestrial materials often precludes destructive preparation, limiting opportunities for detailed study by conventional microscopy techniques. Here, we report a new approach for high-resolution elemental mapping of an unprepared angrite meteorite (~4.35 Ga) that led to the first documented occurrence of zircon within this class of meteorites.

SEM-BEX (backscattered electron and X-ray) mapping was performed at beam conditions of 15 kV and 4 nA in variable pressure mode (35 Pa) to mitigate charging on the uncoated angrite surface. To overcome X-ray shadowing from uneven topography and low count rates typical of such conditions, BEX uses a beneath the pole piece detector geometry that minimizes shadowing and maximizes X-ray collection efficiency. This approach enabled large-area mapping ($>4.5 \text{ cm}^2$) at high resolution (2.5 μm pixel size) to be achieved in four hours,

The dataset revealed ten zircon crystals ($<50 \mu\text{m}$) dispersed across the sample, representing an exceptionally rare phase at trace abundance. Several crystals were confirmed as zircon through quantitative elemental analysis using EDS. Large area maps now provide a means with which to target individual crystals for localised FIB ‘lift out’ and further analysis with mass spectrometry techniques. As zircon is a key chronometer for U–Pb dating, its presence in angrites opens new avenues for constraining early solar system chronology.

This discovery demonstrates the feasibility of identifying ultra-rare minerals in pristine samples without cutting or coating, preserving their integrity for subsequent analysis. Beyond the implications for angrite petrogenesis and solar system evolution, this approach demonstrates a powerful pathway for guiding targeted extraction of minerals for geochronology in rare earth and planetary materials.

The importance of being oriented: a review of thirty years of Electron Backscatter Diffraction research in Earth and Planetary Sciences

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Electron Backscatter Diffraction (EBSD) allows mapping of crystal orientation on submicron scale. Earth scientists realised the potential of the method in the 1990s. One of the first PhD theses applying it to rocks includes about 200 indexed quartz orientations; now we routinely collect orientation maps with more than a million points. In this talk I will focus on what we have done and might do with such large datasets.

Ideally, we have hypotheses in mind before collecting such data, although EBSD maps often stimulate new ideas. EBSD is excellent for deducing Crystallographic Preferred Orientations (CPOs) to help understand deformation. Here, one orientation per grain is likely to provide an adequate picture, so micron scale maps are not, strictly, necessary. In contrast such maps illustrate distortions *within* specific grains which carry a wealth of additional information. That information can be extracted by looking at *relative* orientations (“misorientations”) between points. Misorientations comprise an axis and an angle; the axis gives insights into, for example, dislocation slip systems. A newer related approach, using orientation gradients on maps, gives more direct insight into the slip vectors of dislocations. Not all distortions are due to deformation; they may be due to growth and thus give insights into that, but EBSD remains a key source of information.

EBSD also illustrates microstructural relationships between *different* minerals. A modified version of misorientation can be calculated and gives information on for example topotaxy and epitaxy, key aspects of solid-state mineral growth.

Today we have many analysis tools available from EBSD system vendors and academics (e.g. MTEX). These don’t tell you what the analysis outputs *mean* so we still need microstructural theory to underpin our work and need to be prepared to develop new algorithms that could exploit our data more fully.

The Space Nanomaterials atom probe (SNAP) providing an atoms eye view of the Solar System

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The SNAP laboratory is coming to the University of Glasgow in 2026. SNAP comprises a LEAP6000-XR instrument from CAMECA. SNAP will be the first APT (atom probe tomography) facility in Scotland and the first worldwide to specialise in space science challenges. The SNAP facility will comprise a deep-UV laser system and cryo-vacuum transfer solution enabling first-of-their-kind measurements of challenging materials, including extraterrestrial materials.

Atom probe tomography (APT) is an advanced technique for analysing materials at the atomic level, offering unparalleled insights into their structure and composition. Over the past decade, APT has significantly contributed to our understanding of both technological and geological materials. In planetary science, its applications include characterising pre-solar nano-diamonds, examining nanoscale damage from space weathering, tracking trace element movement during asteroid impacts, detecting nanophase magnetic minerals in iron meteorites, and studying aqueous alteration in asteroids. APT is an ideal tool to maximize scientific output from minimal sample volumes which is particularly important for sample return missions. Yet, the technique's full potential remains largely untapped, with many promising applications still to be explored.

Here we will outline the opportunities the SNAP laboratory can offer the UK Planetary Science community and detail our early access scheme.

Resolving Mixed Spinel Signatures with EPMA Element Mapping in Metasomatized Peridotites

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Spinel chemistry (Cr#, Mg#) is widely used to infer mantle tectonic setting, but grain-scale metasomatism can introduce heterogeneity that is only visible with microscopy. In peridotites of the Santa Elena ophiolite (Costa Rica), electron probe microanalysis (EPMA) element maps identified Ti-pargasite that rims spinel grains. These rims drive sharp chemical heterogeneity within single spinel grains by redistributing Al at the rim. Analyses adjacent to Ti-pargasite exhibit elevated Cr# and lower Al and Mg, plotting in forearc fields, whereas analyses at spinel-plagioclase contacts mimic abyssal-field values. Prior core-rim spot analyses produced scattered tectonic signatures with no clear explanation; only high-resolution mapping revealed the cryptic amphibole phase and its control on spinel chemistry. These results show that in metasomatized peridotites, element mapping is essential to separate primary tectonic signals from localized reaction overprints.

Linking olivine crystal-preferred orientations to deformation: EBSD evidence from the Songshugou Peridotite Massif (North Qinling, Central China)

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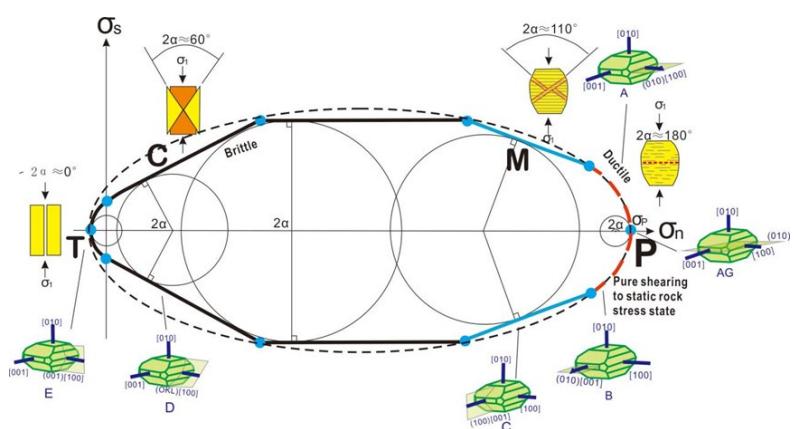
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Peridotite slabs in the North Qinling orogeny underwent a long and complex tectonic history over 500 Ma before the Devonian era. Unravelling complex olivine crystal fabrics from Songshugou peridotites from this region is one way we can better understand the tectonic history. During deformation, dynamic recrystallisation can result in preferred crystal orientations linked to different stress histories. By measuring the crystal-preferred orientations (CPOs) of olivine in peridotites from Songshugou, we demonstrate that there are six types of inner-crystal slipping systems recorded, namely AG (010), A (010)[100], B (010)[001], C (100)[001], D (0kl)[100], and E(001)[100].

We then study the relations among the inner-crystal slipping systems, considering the unified deformation criteria, which is a valuable tool for determining structural deformation types based on the plane angle between the deformation zone and the maximum principal stress. The unified deformation criteria form an ellipse, but are usually approximated by several special criteria, including the Tensile Criteria (Point T; tensile fractures), the Coulomb criterion (C; shear fractures), the maximum effective moment criterion (M; ductile shear bands), and the pure shearing point (P; lithostatic stress) (Figure 1).

By checking micro-structures in the small experimental areas of EBSD, it can be concluded that different deformation regimes results in different types of CPOs, for example, high temperature compression results show plastic deformation such as alignment of the unrotated AG type (D in Figure 1), the rotated B type, and the fluid accelerated and rotated C type, while low temperature compression results in alignment of the conjugate shearing fractured D type, and the jointed E type typical of more brittle deformation. By further investigating these crystal fabrics and deformation signatures, we hope to compile a more detailed tectonic history of the North Qinling orogeny.

Figure 1. The shear stress (σ_s)-normal stress (σ_n) diagram shows the unified deformation criteria ellipse, three different fracture criteria in Mohr space, and six inner-olivine crystal slip systems identified in the Sonshugou Peridotite Massif. Three styles of fracturing are depicted as tensile fractures (T), Coulomb criterion-related shear fractures (C), and maximum effective moment criterion-related ductile shear bands (M). The transition criteria among these three types are not displayed. Point P corresponds to lithostatic stress with zero differential stress, and the Mohr circles tangent to P represent unrotational compression caused solely by normal stress. From left to right, the plane angle (2α) is changing from zero (e.g. joints), an acute angle (e.g. brittle shear fractures), an obtuse angle (e.g. ductile shear zones), to the angle of 180 degrees (e.g. pure shearing without rotation).



High-resolution Scanning Electron Microscopy Imaging in Mudstone Thin Sections with Different Metallization Alternatives

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Mudstones constitute about 40% of all sedimentary rocks and play a fundamental role in petroleum systems because they can act as both seal and source. A full characterization is, therefore, essential for deep hydrocarbon exploration. The analysis of these nanometer-scale regions provides direct data on the interaction of formation fluids with the rock constituents (diagenesis), allowing the determination of pore abundance and type in order to identify the factors that control permeability—information that is fundamental for studies on the sealing capacity of these rocks. Sedimentary rock samples are highly insulating, requiring metallization for their analysis with scanning electron microscopy. However, conventional coatings using carbon or gold can obscure pore-related information. Imaging at low accelerating voltages can provide high-resolution images without metallization, but is not ideal for energy-dispersive X-ray spectroscopy (EDS) data collection. In the present work, different metallization alternatives were tested to enable high resolution imaging and EDS analysis on these porous sites without a conducting layer. They involved the use of different metals, design of the conducting framework on the surface, and aluminum castings for grounding while preserving pore integrity. The samples were analyzed using a Tescan Amber microscope equipped with an Oxford EDS Max-Pro detector, operated at 10 and 20 kV. The results showed that four alternatives were effective, allowing high-resolution imaging and EDS analysis in the porous regions.

Telling the time with SEM-EDS: Application of quantitative EDS to diffusion chronometry

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Diffusion chronometry is a powerful tool for constraining the timescales of both magmatic and metamorphic processes, based on the relaxation of chemical boundaries. In volcanic environments, diffusion chronometry is often used to extract human-relevant timescales of pre-eruptive processes such as magma mixing and magma ascent. In mafic to intermediate rocks, Fe-Mg interdiffusion chronometry is particularly useful for constraining processes that occurred over timescales of days to years from the products of eruptions, independent of when the eruption occurred. The method relies on accurate and precise chemical profiles of the elements of interest. In olivine, these are typically extracted from EPMA-calibrated BSE images. However, access to EMPA instruments is sparse geographically and is often extremely expensive. Here we test whether recent developments in EDS detector technology have advanced the technique of quantitative EDS to the point where well calibrated EDS measurements of Fe-Mg interdiffusion could be used instead of EMPA for diffusion chronometry. Such an advance could revolutionise diffusion chronometry research in countries where access to electron microprobes is limited or has traditionally been prohibitively expensive.

Quantifying Porosity and Mineralogy in Rock Samples through Integrated SEM/EDS and Machine Learning Approaches

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Integrating Scanning Electron Microscopy (SEM), Energy Dispersive X-ray Spectroscopy (EDX), and advanced machine learning techniques such as the U-Net model provides powerful tools for microstructure analysis and mineralogical research. This interdisciplinary approach enhances the precision of data interpretation while broadening research methodologies.

This study presents a systematic framework for analyzing rock microstructures, with emphasis on quantifying porosity and mineral composition. Rock samples were first prepared using Broad Ion Beam (BIB) polishing to achieve high-quality surfaces for imaging. Secondary electron (SE2) images obtained through SEM revealed fine-scale microstructural features.

A key innovation is the application of a pre-trained U-Net model for pore segmentation (Klaver et al., 2022), enabling efficient and accurate delineation of pore networks within the rock matrix. Mineralogical analysis was then conducted using high-resolution backscattered electron (BSE) imaging in combination with EDX data. Semi-automatic phase segmentation, incorporating pixel-wise algorithms and decision-tree classification, facilitated precise identification and quantification of mineral phases (Jiang et al. 2022).

Finally, SE2 and BSE images were aligned to correlate porosity with specific mineral phases. This integration offers a comprehensive perspective on the spatial distribution of porosity within distinct minerals, advancing understanding of rock microstructural properties.

The methodology outlined here provides a robust and versatile framework for rock microanalysis. By combining high-resolution imaging, machine learning, and automated mineral classification, this approach delivers novel insights into porosity/mineral relationships with broad implications for geological research and industrial applications.

References:

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Klaver, J., et al., 2021. <https://doi.org/10.3997/2214-4609.202011981>

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Microscopic Meteorites: Art, Science and Stories from space

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As earth scientists, planetary scientists, and electron microscopy technicians, it has become routine for meteorites and rocks to be analysed in the microscope. This is in the hope that they show something meaningful and exciting about the solar system, or the ground beneath our feet. To the rest of the world what can be done is often a mystery that is hidden behind closed laboratory doors.

Microscopic Meteorites is a travelling exhibition, combining art and science to immerse the public in the mysteries of the universe that can be uncovered using analytical microscopy techniques of meteorites, and rock samples from Earth.

Five samples originating from different places (Mars, Moon and Vesta meteorites & Kpg boundary layers from Italy and Canada) were analysed under scanning electron microscope (SEM). Complementary in-situ techniques such as energy dispersive spectroscopy (EDS), and automated mineralogy (mineral liberation analyser by FEI), revealed structural and compositional properties of each sample which could then be used a main talking point for the exhibition.

Alongside bright colourful images taken during analysis, a teaching point for each sample was chosen to help introduce the public to new scientific concepts such as: what minerals can be found in meteorites, how do we know a rock is a meteorite, what environments do these rocks experience, and how that can change their texture, chemistry and morphology?

After a three-month residency at the Sherwood Observatory in Sutton-in-Ashfield, the exhibit received overwhelmingly positive reviews, showing that art and science can work together to help make these concepts accessible to the public that are commonplace for planetary and earth scientists, but mysteries for everyone else.

Building a pilot open-access correlative rock microscope

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*With the proliferation of *in situ* analysis on polished thin sections and grain mounts, we are producing vast multi-dimensional chemical and isotopic datasets within the spatial context of mineral grains. Despite great progress with data acquisition on many platforms (SEM, LA-ICP-MS, SIMS, Synchrotron XFM, etc.) there is a gap in archiving data in a spatially registered context and performing quantitative data analysis across all data modalities.*

To address this gap, we have developed a pilot open-source microscope viewer that can be cloud- or local server-hosted or deployed on a PC, which allows simultaneous inspection of optical, chemical, and crystallographic data. For education and public outreach purposes, the microscope viewer is similar to the Open University's 'Virtual Microscope' but offers easy virtual stage rotation across the entire scanned thin section.

Underlying the new correlative platform are open-source software solutions that allow users to more easily manage and organise the data, simplify the image dimensions, register images from different instruments, annotate features across all data modalities, and perform classification. The ability to register chemical, crystallographic, optical and other data, as pioneered by ImageMatrix, permits users to produce their own phase maps from their understanding of the thin section in a more intuitive fashion than automated mineralogy purely on SEM-BSE and -EDX intensity/concentration maps.

*We will demonstrate how thin sections are rapidly explored with a stack of simplified optical and chemical images, while mineral classes are annotated on simultaneously displayed thin section surface images (SEM BSE and optical reflected light), while machine-learning classifiers are deployed across all original input channels (individual underlying maps). In our experience, the use of neural networks for chemical dimensionality reduction, holds significant potential to make many discoveries from *in situ* chemical data.*

Lithium to Uranium: A New Analytical SEM for Environmental Research hosted by UCL Earth Sciences

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The Department of Earth Sciences at UCL has recently acquired a state-of-the-art analytical Scanning Electron Microscope (SEM) dedicated to quantitative research across the breadth of Earth and Environmental science. The system is based around a TESCAN Clara Field Emission SEM that is equipped with a suite of cutting-edge detectors for studying the major, minor and light element chemistry of samples as well as their crystallographic texture. We anticipate this will offer a transformative platform for non-destructive microanalytical investigations.

A large area Energy Dispersive Spectroscopy (EDS) detector facilitates routine studies of samples' major element chemistry at 1-2 μm spatial resolution. An additional windowless EDS detector extends capability to allow higher spatial resolution (sub-micron) and increased detection of lighter element X-rays (Li-F). Coupled with standardised analytical routines we anticipate the new platform providing robust major element chemical mapping of a wide range of environmental sample types. Furthermore, chemical mapping is fully integrated with an electron backscatter diffraction (EBSD) detector. This allows efficient and simultaneous EDS and EBSD analyses across large sample areas, significantly enhancing studies of complex natural and synthetic samples.

Finally, the microscope is equipped with iXRF's latest SEM-based micro-X-ray fluorescence (XRF) system. This provides non-destructive trace element quantification and mapping capabilities. Dual operation of electron and photon beams should provide simultaneous excitation of light (via electrons) and heavy elements (via photons). Since XRF has increased sensitivity, by up to 3 orders of magnitude, compared with electron beam excitation we expect detection limits of ~ 20 ppm (or better) for all elements across the periodic table up to Uranium.

The system is currently being commissioned, such that this presentation will demonstrate initial capabilities using early results.

Micro to nanoscale characterisation of olivine, wadsleyite and ringwoodite in the Catherwood L6 ordinary chondrite and implications for the evolution of high-pressure mineral assemblages in planetary materials

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High-pressure minerals such as ringwoodite, wadsleyite and majorite are abundant in highly shocked chondritic materials and are commonly preserved in, or adjacent to, impact melt veins. However, the timing and mechanisms governing their formation are poorly constrained. Here, we conduct correlated electron backscatter diffraction (EBSD) and SEM-WDS analysis of impact melt veins in the highly shocked Catherwood L6 chondrite to place empirical new constraints on the formation mechanisms of these phases. Ringwoodite occurs as polycrystalline conglomerates intergrown with wadsleyite and displays hints of phase heritage from a precursor (olivine) structure, suggesting a solid-state (or martensitic) formation rather than melt-driven recrystallization. This is supported by identical chemical compositions for olivine and ringwoodite within the sample (within analytical uncertainty). Majorite, which occurs as the primary mineral within the impact melt vein, displays crystallographic preferred orientations (CPO) throughout the vein with correlated {001} axis orientations, suggesting the presence of constraining pressure during formation. There is no shape-preferred orientation (SPO) for majorite crystals across the impact melt vein, further supporting the rapid formation of the assemblage through frictional melt processes as formation from a viscous melt would generate flow textures with a clear SPO. Chemically, majorite does not match with pyroxene grains adjacent to the impact melt, suggesting interaction of the grains with a chemically distinct melt prior to crystallisation. These findings suggest that impact melt veins in the Catherwood L6 ordinary chondrite formed through frictional stress and heating, and that ringwoodite formed through solid state transformation rather than melting and recrystallization of an olivine precursor. These new insights into the origin and evolution of high-pressure mineral assemblages begin to place constraints on the P-T conditions experienced by chondritic materials and place new constraints on the formation of meta-stable high PT polymorphs in planetary materials.

Hydrothermal Alteration of Isle of Rum Peridotite – A Jezero Crater Analogue

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The Isle of Rum Layered Suite (RLS), Scotland, forms part of the British Tertiary Volcanic Province (BTPV), recognised as a site of interest to study magmatic processes that occur within layered intrusions [1]. In the Western Layered Series of the RLS, olivine-cumulate/wherlitic units of the Ard Mheall member have been identified as potential analogue samples for the Séítah formation - an olivine-rich igneous crater floor unit of Jezero Crater, Mars [2]. This is owing to their olivine-rich nature and evidence for hydrothermal alteration that, whilst observed, has not been studied in detail. The Séítah formation was sampled by the Perseverance rover in the early stages of the Mars2020 mission in preparation for Mars Sample Return (MSR).

Here, we use a correlative multi-technique approach including X-ray diffraction, scanning electron microscopy and transmission electron microscopy was used to characterise a peridotite sampled from the Ard Mheall member to determine its suitability as an analogue for MSR and investigate any evidence of hydrothermal alteration. If suitable, this sample could inform analytical workflows for analysis of the Séítah formation sample once retrieved and returned to Earth. Initial results demonstrate the peridotite as an effective analogue for the Séítah formation, hosting a poikilitic texture much-like that observed in the Séítah formation by the Perseverance rover [3]. Our analyses not only reveal serpentine veining like some aspects of nakhelite Martian meteorites [4], but a complex history of water-rock interactions indicating multiple episodes of hydrothermal alteration in the RLS, providing an insight into the potential hydrothermal alteration that occurred within the nakhelite cumulate pile and Jezero Crater on Mars.

[1] Hepworth et al., (2020), *Contrib. Mineral. Petrol.*, DOI: 10.1007/s00410-019-1652-9. [2] Thiessen et al., (2024), 55th LPSC, Abstract #1208. [3] Farley et al., (2022) *Science*, DOI: 10.1126/science.abo2196. [4] Hicks et al., (2014) *Geochim. Cosmochim. Acta*. DOI: 10.1016/j.gca.2014.04.010.

Constraints on Basaltic Volcanism on Vesta: Insights from Pb-Pb Isotope Systematics in Eucrites

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Eucrites provide unique insights into the early magmatic evolution of the asteroid Vesta. This study comprises 11 samples: five basaltic, two cumulate, and four brecciated eucrites. We used Secondary Ion Mass Spectrometry to collect Pb-Pb isotope systematics, using methodology resembling previous studies [1-2]. Radiogenic Pb isotope compositions are indicative of sample age, whilst initial Pb compositions provide an isotopic record of a rock's source at crystallisation. The age and initial Pb combined enable estimates of the $^{238}\text{U}/^{204}\text{Pb}$ source ratio (μ -value), which can provide an indication of volatile loss from a rock's mantle source or parent body.

Seven samples date between 4.54 and 4.51 Ga, consistent with reported eucrite formation ages [e.g., 2]. The remaining samples range from 4.4 to 3.9 Ga; some dates may be explained by the brecciated nature of specific samples, potentially related to an impact event at \sim 4.1 Ga [3]. The range of Pb-Pb dates suggests ongoing processes, likely impact-related, across this more extended period.

The initial $^{204}\text{Pb}/^{206}\text{Pb}$ values indicate source μ -values in the range 91 – \sim 3000, significantly higher than terrestrial values (\sim 8-10) and those previously reported for eucrites (\sim 14–150; [4-5]). However, they are somewhat comparable to reported lunar mare basalt source μ -values (\sim 350–700; [e.g. 1]), thus supporting a volatile-depleted model of Vesta. We are currently conducting further work to analyse the trace element chemistry of individual minerals, using LA-ICP-MS. These will be used to reconstruct parent melt compositions and identify samples originating from chemically distinct source regions.

References: [1] Snape J. F. et al. (2016) EPSL 451:149–158. [2] Kouvatsis I. et al. (2023) GCA 348:369–380. [3] Miyahara M. et al. (2014) PNAS 111(30):10939–10942. [4] Tera F. et al. (1997) GCA 61:1713–1731. [5] Lowe H. et al (2024) 86th Annual Meeting of the Meteoritical Society, Abstract 6303.

Analysis of the CM chondrite meteorite Winchcombe by FIB-TOFSIMS and SEM-EDS as advanced complimentary techniques

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Introduction: Time Of Flight Secondary Ion Mass Spectrometry (TOFSIMS) is a highly sensitive surface analysis technique, generating large quantities of reproducible data. When used as a fully integrated detector within a dual beam Focused Ion Beam Scanning Electron Microscope (FIBSEM) it allows identification of a site of interest and characterisation by SEM-EDS prior to TOFSIMS analysis. TOFSIMS providing major, minor, trace and isotope chemistry, including detection of light elements, often with higher resolution than EDS. We applied these techniques to a sample of the Winchcombe meteorite, a CM chondrite breccia [1]. It has eight distinct lithologies ranging from CM2.0-CM 2.6 [2] with many interesting microscopic features. This study will present a range of results from the varied features observed within Winchcombe by SEM-EDS and FIB-TOFSIMS.

Methods: A Winchcombe rock chip in a polished block, P31494, loaned from the Natural History Museum, UK, was analysed by SEM imaging, EDS and FIBSEM based TOFSIMS on a Tescan Solaris X with a TOFWERK TOFSIMS.

Results and conclusions: The target features, revealed structures in the TOFSIMS data that the SEM-EDS alone could not readily detect. Such as a chondrule rim with well-defined inner rim incorporating approximately 1 micron particulates rich in Al^{27+} Ti^{48+} , Cr^{52+} and an outer rim enriched in Na^{23+} , Mg^{24+} , Si^{28+} , K^{39+} , Ca^{40+} . The action of the FIB could also be used as a localised surface preparation technique, revealing additional detailed structure, figure 1. FIBSEM TOFSIMS systems are incredibly powerful technology in application to planetary science samples, a high-resolution method complimentary to EDS.

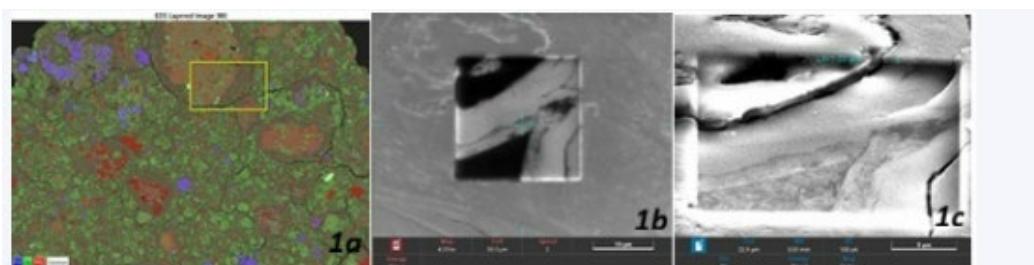


Figure 1. (a) SEM-EDS false colour map of Winchcombe, P31494, NHM (UK), highlighting an area studied with FIB-TOFSIMS, (b) FIB image after TOFSIMS analysis, (c) SEM SE image after TOFSIMS analysis.

References: (1) King A.J., et al., *Sci. Adv.*, 8(46) 2022. (2) Suttle M.D. et al., 75th Metsoc.

4D STEM and Correlative EDS Analyses of Planetary Materials

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In order to fully characterise complex phases such as phyllosilicates, both structural and compositional information is required. In conventional (S)TEM techniques, as the electron beam is rastered over a sample, the electrons that are scattered by the sample that fall into a specific range of detection angles are recorded. In 4D STEM, at every 2D probe position (x, y) a 2D diffraction pattern is acquired (k_x , k_y), resulting in a 4D dataset. The technique is enhanced further by simultaneously acquiring EDS data at every probe position. The final result is a rich dataset comprised of spatially correlated crystallographic and compositional information.

Secondary mineral assemblages in carbonaceous chondrites show that they experienced extensive aqueous alteration whilst on their parent bodies (Suttle, 2021) resulting in the formation of serpentine-like phyllosilicates (King, 2015, 2022; Garvie, 2021; Ito, 2022). Analyses of these phases can tell us more about the delivery of water and volatiles to the terrestrial planets. In turn, this characterisation informs about key geochemical cycles such as the S-cycle in the early Solar System (Topping et al, in press). In more recent geological history, the hydrothermally altered nakhlite Martian meteorites contain saponite, resulting from the alteration of anhydrous silicates. The abundance, composition and structure of this phase can be used to infer the relative emplacement of the meteorites on the Martian surface, with respect to the fluid source, and the nature of the secondary assemblage's origin on Mars.

Here, we present analyses of phyllosilicates in the CM carbonaceous chondrite Winchcombe (Topping et al, in press), and in Martian nakhlite NWA 15364. Our results thus far show that the novel 4D STEM and correlative EDS technique is capable of identifying complex mineral phases with differing compositions and structures in a variety of planetary materials.

Novel Combinations of Quantum Diamond Microscopy and Electron Probe Microanalysis Reveal Intricate Intergrowths in an Unusual Meteorite

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Ungrouped achondrite Northwest Africa (NWA) 15915 is an unusual meteorite composed of FeO-free augite and enstatite, complexly intergrown sulfide spherules of alabandite, daubréelite, troilite, caswellsilverite, oldhamite, and djerfisherite, and Ni-free Fe-metal. Silicate compositions, observed sulfide species, large grain sizes, and reported oxygen isotopes [1] suggest potential formation on a Mercury-like body. Here, we present combined electron microscopical (EM) and remanent magnetism (RM) analysis, and how observations made during RM analysis can be a valuable guide for targeted EM.

Sulfide species were identified by energy dispersive and wavelength dispersive spectroscopy (EDS/WDS) at the Characterization Facility, University of Minnesota using a Thermo Fisher Scientific Apreo 2S Lo-Vac SEM with Oxford Instruments Ultim Max 100mm2 EDS detector and AZtec v6.1 software, and a JEOL 8530F Plus EPMA and Probe for EPMA software respectively. The presence of Fe-metal, daubréelite, and troilite were independently identified through room temperature saturation isothermal remanent magnetism using a Quantum Design MPMS3 at the Institute for Rock Magnetism, University of Minnesota. The behaviour of the magnetic response during thermal cycling suggested that the sulfides were highly intergrown, and was subsequently verified by low voltage EM.

Further, quantum diamond microscopy (QDM) mapping of the sample show extensive small dipoles throughout in addition to large dipoles associated with Fe-metal spherules – signaling a distinct magnetic carrier. Low voltage EM analysis finds euhedral cubic and hexagonal submicron Ni-free Fe-metal grains hosted within the pyroxenes and aligned to the direction of exsolution. QDM mapping shows that although these grains are observable throughout the sample, they are heterogeneously distributed across individual pyroxene grains. At typical EDS/WDS conditions, these submicron Fe-metal grains are largely lost in the interaction volume of the electron beam. Thus, the combination of EM and RM techniques can reveal unique features that would otherwise be overlooked.

[1] Rider-Stokes et al. (2025), Icarus 441:116713.

*Laser-induced breakdown spectroscopy (LIBS) as a tool for planetary exploration:
From the macroscale to the microscale on Mars and beyond*

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Laser-Induced Breakdown Spectroscopy (LIBS) uses a rapidly pulsed laser to ablate a small volume of material and generate a plasma whose emitted light contains the characteristic spectral signatures of the elements present. Over recent years, LIBS instruments have become a popular technology for planetary exploration given the remote capabilities, rapid analytical time, ability for the LIBS shockwave to clear dust from a target, and relatively low power requirements. In this talk, I will discuss the combined use of LIBS with textural analysis on the SuperCam instrument to explore Jezero crater's geological history and the current development to miniaturize this technology further for fine-scale element mapping on Mars and other planetary bodies.