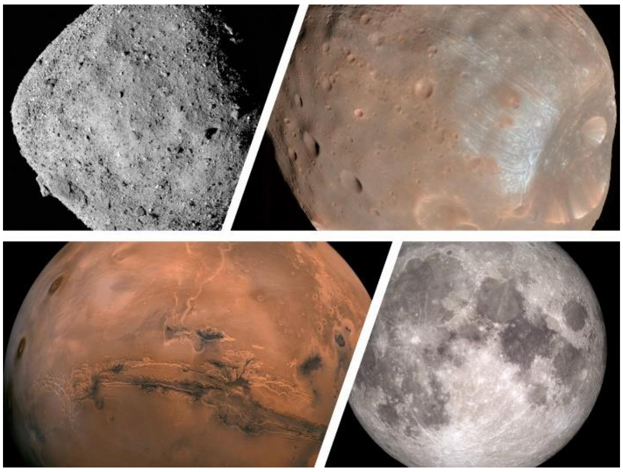
Analysis of Returned Extraterrestrial Materials: Current Capabilities & Future Opportunities

Virtual RAS Specialist Discussion Meeting

11th December 2020



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To date space missions have collected and returned to Earth samples of the Moon (Apollo/Luna), from low Earth orbit (e.g. LDEF), the solar wind (Genesis), a comet (Stardust), and a stony asteroid (Hayabusa). Returned extraterrestrial materials offer context for our meteorite collection, ground-truth astronomical and remote observations, and can be studied in unparalleled detail in the laboratory, leading to ground-breaking discoveries about planet formation that are not possible through in-situ spacecraft measurements alone. Furthermore, returned samples become available for the technology and scientific questions of the future.

Samples will soon be returned from the carbonaceous near-Earth asteroids Ryugu (Hayabusa2 in 2020) and Bennu (OSIRIS-REx in 2023), while there are ambitious plans to bring back materials from the Moon, Phobos and Mars within the next ~10–20 years. Successful characterisation of these complex and precious materials requires the planning and development of new analytical methods and protocols today.

This meeting will bring together the international sample analysis community to discuss the current and future analytical and curation capabilities that will maximise the scientific impact of extraterrestrial sample return missions. We aim to review recent advances in electron microscopy (e.g. SEM, FIB/TEM) and mass spectrometry (e.g. SIMS, ICP-MS), highlight the capabilities and upcoming opportunities at synchrotron facilities, and explore new pioneering techniques (e.g. nano-IR, atom probe, magnetic properties) and their application to extraterrestrial materials.

Organisers: Ashley King (NHM), Natasha Almeida (NHM), Luke Daly (University of Glasgow), Leon Hicks (University of Leicester), Natasha Stephen (University of Plymouth), Romain Tartese (University of Manchester) & Penny Wozniakiewicz (University of Kent).

ABSTRACTS

JAXA's MMX Mission and implications for sample analysis

Sara Russell, on behalf of the MMX Sample Analysis Working Team: Wataru Fujiya, Yoshihiro Furukawa, Haruna Sugahara, Ken-ichi Bajo, Nancy L. Chabot, Mizuho Koike, Yayoi N. Miura, Frederic Moynier, Sara Russell, Shogo Tachibana, Yoshinori Takano, Tomohiro Usui and Michael E. Zolensky

JAXA's MMX (Martian Moons eXploration) mission to Mars' moons will launch in 2024 and return in 2029. It will orbit both Deimos and Phobos and collect at least 10g of material from the surface of Phobos. There are two main goals of the mission. Firstly, the MMX mission will determine the origin of the moons of Mars, as either captured asteroids or the result of a giant impact. Secondly the mission will observe the processes that affect Mars and its environment (Usui et al 2020).

The Sample Analysis Working Team are making preliminary plans for sample analysis that will enable the science aims to be achieved. The plan is discussed in a recently submitted paper [2].

References: [1] Usui et al., (2020) Space Science Reviews 216, Article number 49 [2] Fujiya et al. (2021) Earth, Planets and Space, submitted.

Biogenic magnetic needles in the Martian sedimentary haystack: searching for magnetofossils in Jezero Crater lacustrine sedimentary rocks probed during the Mars Sample Return campaign.

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Magnetotactic bacteria (MTB) are microorganisms that produce intracellular chains of (typically) magnetite crystals with specific grain sizes that are used to assist navigation around the oxic-anoxic interface of aquatic habitats where they typically live. When MTB die, their magnetic remains (magnetofossils) are buried and can be preserved as intact chains within sediments provided that diagenetic conditions did not become sulphidic, which would cause magnetofossils to dissolve. Intact magnetofossil chains have distinctive magnetic properties, with strong magnetic interactions among individual magnetite crystals within a chain and negligible interactions among neighbouring chains. MTB live in aquatic environments worldwide, often under extreme environmental conditions analogous to those of early Mars. MTB arose on Earth in the mid-Archean (3.2-3.8 Ga), when oceans were stratified, hotter and mainly sulphidic and a magnetic field was strong enough to give MTB an evolutionary advantage. Evidence from Gale Crater indicates the existence of sulphurous and saline water masses in Mars, where clay-bearing fluvio-lacustrine deposits accumulated at a time (3.3-3.7 Ga) that overlapped with the presence of the final stages of an Earth-like Martian dynamo. The Mars2020 rover may discover similar, small-scale sedimentary deposits during its exploration traverse at Jezero Crater. Given the widespread occurrence of MTB in terrestrial lacustrine sediments, the potentially similar biochemical environment on Earth and Mars, and the great interest in identifying possible life on Mars, it would be important to evaluate the presence of magnetofossils in past Martian aquatic environments. If they were present, identifying magnetofossils in Mars2020 samples would be straightforward because they are detected readily with rock magnetic analyses. We propose to measure a combination of detailed isothermal remanent magnetization acquisition curves and first-order reversal curve diagrams, a tandem that enables distinction of the peculiar magnetic properties of magnetofossil chains from those of magnetite grains of inorganic origin. These measurements are non-destructive, only requiring subsampling of 1 cm^3 samples, and their reliability would not be compromised by initial sample sterilization using radiation. They might be, however, be sensitive to mid temperature pre-treatment conditions; magnetic analyses could, thus, be implemented as part of initial biological screening procedures. Although interest in searching for magnetofossils in returned samples from Mars is not novel, our proposal is, to our knowledge, the first to suggest a tailored, non-destructive and diagnostic rock magnetic study aimed at this goal.

Photogrammetry for Determining the Physical Properties of Meteorites

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The fundamental physical properties (such as density, porosity, magnetic susceptibility and electrical conductivity) of extra-terrestrial materials provide insight into the processes that controlled their formation and evolution, and the parent bodies from which they are derived [1]. For example, meteorite densities are used to make inferences about the composition and internal structure of asteroids [1,2], and to understand parent body thermal history [3]. Porosity can be used to understand the physical history of meteorite parent bodies, in terms of processes such as asteroid compaction, lithification, break-up and re-assembly [1]. It is a key parameter in understanding both meteorite survivability during atmospheric entry and asteroid survivability during impacts [4,5]. Magnetic susceptibility and electrical conductivity measurements, which are controlled by metal and metal oxide concentrations and distributions in the samples, can also be used to understand sample mineralogy [6,7] and aid the classification of recovered meteorites [6,8].

We are investigating physical properties of a suite of 20 meteorites returned by the UK-led 'Lost Meteorites of Antarctica' project [9,10], ranging in mass from ~1 g to ~2500 g, using a combined, non-destructive approach. We are also using a magnetic susceptibility-electrical conductivity field probe as part of the sample classification [7]. We used professional photogrammetry software [11] to create high-fidelity three-dimensional models of a suite of recovered meteorites [12]. Photogrammetry uses two-dimensional images to determine accurate information about the surface of the object [13]. These models are used to compute sample volume, which is used to determine sample density. The technique does not contaminate the sample and is scalable to samples of a range of sizes dependent on the resolution of the contributing images. We are calibrating volume measurements to understand the uncertainties and will use computed tomography studies of the same sample suite as a further point of comparison to understand sample volume, density and porosity.

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Instrumentation for Basic Characterisation of Mars Material under Containment

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Basic Characterisation (BC) is a set of non-contact measurements to identify the basic physical and geological characteristics of a returned Mars sample in order to inform its later detailed analysis [1]. It will include optical imaging and X-ray computed tomography. The BC imager will perform close-up sample inspection which will offer our first images of the collected extraterrestrial material in the Receiving Facility. Its role will be to inspect tube seal integrity, image dust on the outside of the tubes, examine sample shape and state, search for morphological biosignatures, check for visible contamination, measure optical characteristics, map geological features like grain size and shape, preliminary mineralogy, and early recognition of unusual or high priority features. We are developing a breadboard BC imager to test the range of sample measurements and general BC operation under containment. X-ray CT will be used to perform similar measurements on sample tubes before they are opened. We are investigating the feasibility and instrument requirements of CT for BC, and whether synchrotron-level scan quality is required for successful characterisation.

Strict contamination control measures must be taken for the purposes of planetary protection and to prevent contamination of the samples by Earth's environment. A likely containment environment for sample analysis is an isolation cabinet like the Double Walled Isolator (DWI). A DWI is a tightly controlled environment for BC and PE operations within containment. With ESA and Thales Alenia Space, Leicester has developed and demonstrated a DWI breadboard. It is an ultra-clean class III bio-safety cabinet supporting a large variety of analytical techniques and their requisite instrumentation [2]. The Mars Science Planning Group workshop 2 (pre-decisional) found that the breadboard demonstrates the feasibility of cabinet isolation [3]. This containment environment poses challenges such as space limitations and constraints on instrument materials.

Acknowledgements: The project was funded by ESA and the author's studentship by STFC.

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Non-contact millimetre-wave sensing of rock material properties

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Abstract: Non-contact characterisation of rocks will play a key role in the development of extra-terrestrial geo-mapping. This presentation outlines our research using millimetre-wave frequency modulated continuous wave radar (FMCW) to determine the properties of terrestrial rocks, with a view to both laboratory analysis of returned extra-terrestrial materials and potential deployment via a lab-on-a-bot for *in-situ* measurements.

Millimetre-wave radar has been shown to be an effective detector of geomaterial properties and sensitive to rock characteristics. We demonstrate that contrasts in radar returns correlate with static and dynamic changes in water content during injection to a sandstone sample, where signal returns were acquired during sample exposure in a neutron beam, a well understood means of benchmarking the sensor during water injection, and where the radar-detected flood-front position is corroborated by the synchronous neutron tomographic images. We also observe consistent signal variations corresponding to sample orientation during fluid injection, indicating that FMCW sensing offers insight into sample heterogeneity during rotation.

We advance the use of FMCW radar to assess response to uniaxially loaded sandstones in the laboratory, with contrasts in signal return interpreted to represent microfracture formation under compressive loading prior to sample failure. Our results indicate that FMCW is sensitive to the effects of axial-shortening and material composition. This sensing application yields repeatable and consistent results, which have not been observed in any other sensing modality and are currently verified for four laboratory-deformed sandstone samples, where characteristic radar returns occur approximately 20 seconds prior to macroscopic yield.

This continuing research demonstrates that FMCW radar sensing of rock properties offers new measurands for the study of dynamic properties of rocks and has potential to be a more accessible and deployable sensing modality than traditional methods, with the potential for integration with future extra-terrestrial geo-resource mapping missions. Millimetre-wave sensing represents a low-power, low-cost payload that could aid beyond visual line of sight (BVLOS) operations and form the basis of a lab-on-a-bot for materials analysis in the harsh environments of space, providing terrain and rock classification for live telemetry for human in the loop and parallel autonomous systems to utilise.

Nano-scale synchrotron and microscopy analysis of returned extraterrestrial materials

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X-ray synchrotron analyses have been performed using the I14 nanoprobe and I18 microfocus beamlines, at *Diamond Light Source*, to analyse sample return materials, measuring X-ray Absorption Spectroscopy (XAS), X-ray Fluorescence mapping, and transmission X-ray Diffraction (XRD). Both beamlines are capable of measuring a wide energy range (5-23 keV), and I14 is capable of a beam spatial resolution down to 50 nm.

Stardust Wild2 cometary tracks have been investigated using XRD and XAS at I18, identifying various terminal grain materials such as olivines and Fe-Ni metals. We have demonstrated the presence of Ni-bearing magnetite, suggesting that the parent body of comet Wild2 underwent hydrous alteration, similar to its occurrence within the matrices of many carbonaceous chondrites [1].

Recent work using the I14 nanoprobe, producing XANES maps of FIB lift-out sections from Hayabusa Itokawa asteroid surface samples, have revealed Fe-redox variations in the <100 nm space weathered surfaces of the grains, compared to the substrate ferromagnesian host mineralogy [2]. This analysis is achieved by comparing XAS spectra from selected regions across the sample and observing shifts in the Fe-K XANES pre-edge centroid, resulting from the $1s \rightarrow 3d$ transition in iron. The Fe-K XANES mapping results revealed an increase in oxidation within the partially amorphised space weathered surfaces. Additional analyses performed at *Diamond* also involved the use of aberration corrected electron microscopes at ePSIC, confirming the presence of the space weathered surfaces, along with npFe0 particles typically associated with space weathering [3]. Further investigations of space weathered samples also include Apollo returned lunar surface soil [4].

The techniques presented here allow us to constrain and identify the mineralogy of extraterrestrial materials, and the history of planetary parent-bodies. Current investigations at *Diamond* also include analysing carbonaceous chondrite meteorites in preparation for Hayabusa 2 sample return from asteroid Ryugu.

[1] Hicks et al. (2017), MAPS, 52, 2075-2096; [2] Hicks et al. (in press), MAPS; [3] Pieters and Noble (2016), JGR:Planets, 121, 1865-1884; [4] Hicks et al. (2020), LPSC51, abstract #2094.

Diamond is a Scientist's Best Friend: An overview of current, and future, analytical capabilities at the UK synchrotron facility

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Senior Beamline Scientist (Beamline I14 – Hard X-Ray Nanoprobe)

Diamond Light source is the UK's national synchrotron facility, the X-rays produced at Diamond are used across the beamlines by thousands of researchers to conduct research in a wide range of disciplines in life and physical sciences, including structural biology, health and medicine, solid-state physics, materials and magnetism, nanoscience, electronics, earth and environmental sciences, chemistry, cultural heritage, energy and engineering.

Synchrotron beamlines are essential, versatile, tools for advanced materials characterisation and research. Techniques such as X-ray fluorescence, spectroscopy, diffraction and imaging provide, physical, structural and chemical specific information on a sample across a range of length scales (nm-mm) from the atomic level (bond lengths, local chemical environments), to nano- and microstructural features (grain boundaries, precipitates, density variations) and macroscale arrangements (cracks, macropores).

This talk will highlight the capabilities across Diamond, presenting examples to enable discussion of how these techniques and capabilities can be applied to the study of extra-terrestrial materials, and how scientists from the community can access and fully exploit these instruments. Exciting future developments will also be presented, exploring how new beamlines such as DIAD (a Dual Imaging and Diffraction Beamline) which is planning to take first users early next year, and the Diamond-II source upgrade, will provide opportunities for analysis of material from future sample return missions.

Planetary Sample Analysis Laboratory (SAL) at DLR

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Laboratory measurements of extra-terrestrial materials like meteorites and ultimately materials from sample return missions can significantly enhance the scientific return of the global remote sensing data. The long-lasting heritage in spectral studies on planetary (analogue) materials and the available infrastructure at the DLR Berlin, motivated the extension of the Planetary Spectroscopy Laboratory (PSL) with a Sample Analysis Laboratory (SAL) with a joint support from the Astrobiology Laboratories.

SAL will add over the next 3 years capabilities in preparation to receive samples from sample return missions such as JAXA Hayabusa 2 and MMX missions, the Chinese Chang-E 5 and 6 missions as well as the NASA Osiris-REX mission.

SAL will focus on spectroscopical, geochemical, mineralogical and analyses at microscopic with the ultimate aim to derive information on the formation and evolution of planetary bodies and surfaces, search for traces of organic materials or even traces of extinct or extant life and inclusions of water.

Its setup has already started with the acquisition of a vis-IR-microscope and it will continue with the acquisition of Field Emission Gun - scanning electron microscope (FEG-SEM), Field Emission Gun – Electron microprobe analyser (FEG-EMPA), and X-ray diffraction (XRD) system with interchangeable optics for µXRD analysis and a petrologic microscope for high resolution imaging. The facilities will be hosted in a clean room facility (ISO 5) equipped with glove boxes to handle and prepare samples.

The DLR SAL will be running in close cooperation with the Museum für Naturkunde in Berlin and it will be operated as a community facility (e.g. Europlanet), supporting the larger German and European sample analysis community.

Ultimately, following the approach of a distributed European sample analysis and curation facility, SAL can be later extended to a full Sample Curation facility.

USING THE TECHNOLOGY DEVELOPED FOR MARS RETURN FOR OTHER EXTRATERRESTIAL MATERIALS. John Vrublevskis[1], Steve Duncan[1], Laura Hoyland[1], Lucy Berthoud[1], Michael Havouzaris-Waller[1], , John Holt[2], John Bridges[2], Caroline Smith[3], Allan Bennett[4], Francois Gaubert[5], Ludovic Duvet[6], Davide Nicolis[6], Dayl Martin[6], [1]Thales Alenia Space UK Limited, Building 660, Bristol Business Park, Bristol, BS16 1EJ UK john.vrublevskis@thalesaleniaspace.com, , [2]Space Research Centre, Department of Physics and Astronomy, University of Leicester, Leicester, LE1 7RH UK jmch1@leicester.ac.uk, [3]The Natural History Museum, Cromwell Road, London SW7 5BD UK caroline.smith@nhm.ac.uk, [4]Public Health England, Porton Down, Wiltshire SP4 0JG UK allan.bennett@phe.gov.uk, [5]ESA-ESTEC, Keplerlaan 1, Postbus 299, 2200 AG Noordwijk NL Francois.Gaubert@esa.int, [6]ESA-ECSAT, Fermi Avenue, Harwell Campus, Didcot, Oxfordshire, OX11 0FD UK Ludovic.Duvet@esa.int

Introduction: Thales Alenia Space UK Limited has led a Mars sample return technology development [1] that can be readily adapted for the analysis of other extra-terrestrial materials[2] under a programme of and funded by The European Space Agency* (ESA).

ESA have initiated two 'breadboard' developments to demonstrate the key technologies used in isolators using haptic robotic manipulation. Commercial Off The Shelf (COTS) hardware is being employed at room temperatu,re very clean dry nitrogen environment, to demonstrate current technology capabilities. Results from the isolators and robotic manipulation breadboards have shown the system to be feasible. The breadboards aim to give a first insight and assessment for the sample management in total isolation from human handling. A set of automated robotic systems will be housed inside the isolators and robotic manipulation used to provide both manipulation of the sample at macroscopic & microscopic level. The isolator will be fully integrated with instrumentation for preliminary analysis of sample material (e.g Environmental SEM, Raman Spectroscopy, X-Ray Tomography & Microscopy) as well as GC-MS to analyse isolator gas.

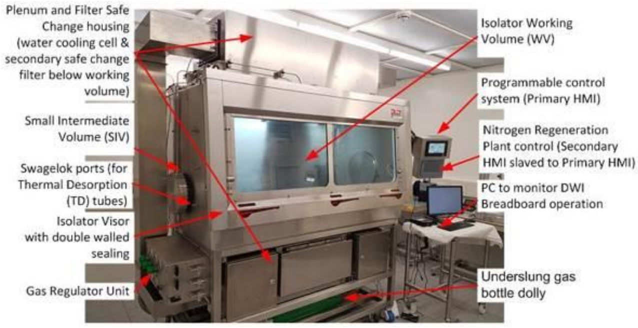

Fig. 1 Double Walled Isolator (DWI) Breadboard at the Space Research Centre, clean room University of Leicester.

This breadboard could be modified for use for other extraterrestrial materials. The robotic manipulation would enhance operator capability (e.g. autonomous actions in certain regions of the isolator) and remove mundane tasks (e.g. automatic isolator internal wipe down, sieving of fines and possibly thin section slices.

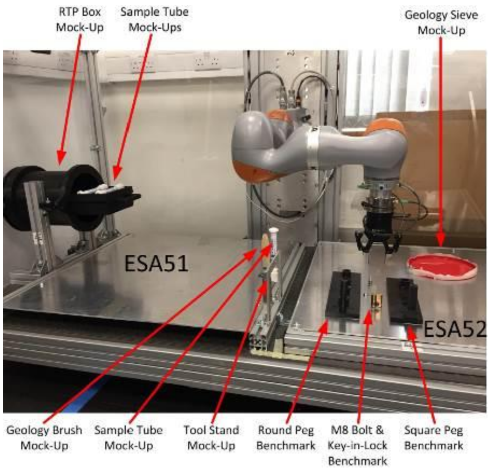

Fig. 2 Remote Manipulation (RM) Breadboard at Thales Alenia Space, Bristol

References: [1] J. C. Bridges and M. Guest, "Planetary Protection and Mars Sample Return," Journal of Aerospace Engineering, vol. 225, no. 2, pp. 239-246, 2011. [2] J. Vrublevskis et al., "Concept for a Lunar and Asteroid Receiving Facility (LaARF)", Science and Challenges of Lunar Sample Return Workshop, 19 February 2014. [3] J. Vrublevskis et al., "Using the European Space Agency (ESA) Technology developed for Mars Return for a Moon Cryo-Sample Return", European Lunar Symposium, Manchester, 21-23 May 2019.

*The view expressed herein can in no way be taken to reflect the official opinion of ESA.

Investigating returned samples with Mössbauer spectroscopy

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The mineralogy and speciation of the redox-active element Fe play an essential role in the co-evolution of planets and life. In meteorites representing undifferentiated parent bodies such as ordinary chondrites, iron partitions between its metallic state and ferrous state in increasingly oxidized minerals ranging from sulphides to silicates. In carbonaceous chondrites, aqueous alteration on the parent body resulted in the formation of ferric oxides. The comparison of these meteorites with samples returned from such parent bodies, the asteroids Ryugu and Bennu, by the Hayabusa 2 mission in 2020 and the Osiris-Rex mission in 2023, respectively, is highly anticipated. In lunar regolith, micrometeorite impacts reduce iron minerals leading to the formation of nanophase metallic Fe particles. The abundance of metallic Fe determines the regolith maturation index. For in situ resource utilisation purposes, lunar regolith can be artificially reduced which releases oxygen and produces metal. The process works the better the higher the FeO content of the regolith. Samples from areas where higher abundances of volatiles and water have recently been identified have yet to be returned and investigated in detail. How these volatiles in lunar regolith affect Fe oxidation states will be a matter of great interest. Oxidising conditions on the Martian surface have produced abundant Fe (ohyhydr)oxides. Redox gradient between ferrous igneous minerals and ferric alteration phases provide an energy source for microbial metabolisms. Iron (oxyhydr)oxides also play a significant role in the preservation of organic matter. Mössbauer spectroscopy is a powerful tool to determine Fe mineralogy and oxidation states and the technique has been applied to meteorites, to samples returned form the Moon during the Apollo era, and in the in situ investigation of the Martian surface with the Mars Exploration Rovers Spirit and Opportunity. Returned samples from any of these bodies are precious because of their small and finite quantities. We discuss non-destructive Mössbauer applications as well synchrotron-based Mössbauer applications that enable the analysis of microscopic sample volumes.

NanoFTIR: The Technique, the Results and One Researcher's Experience of it

Wren Montgomery

The Natural History Museum, London

The nanoFTIR technique will be briefly introduced; I will summarize my experience with the technique 5 years ago and discuss the technical and scientific advances which have occurred since.

Nano-IR Analysis of Returned Carbonaceous Asteroid Material

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ABSTRACT

Mid-infrared (MIR) spectroscopy has been used to great success to quantitatively determine the mineralogy of geologic samples. It has been employed in a variety of contexts from determining bulk composition of powdered samples to spectroscopic imaging of rock thin sections via micro-FTIR. Recent advances allow for IR measurements at even finer resolutions, specifically at the nanoscale, which we have previously used to understand nano-scale compositional variation in ordinary and carbonaceous chondrites [1].

Near field infrared (nano-IR) imaging and spectroscopy enable understanding of the spatial relationships between compositionally distinct materials within a sample. This will be of particular use when analyzing returned samples from Bennu and Ryugu. Both asteroids are thought to be compositionally similar to CI or CM2 carbonaceous chondrites, which have undergone considerable aqueous alteration. Returned samples will likely contain olivine/pyroxene chondrules that have been altered into hydrous phyllosilicates, sulfides, carbonates, and other alteration phases. Use of near-field infrared techniques to probe the boundaries between once pristine chondrules and alteration phases at the nanoscale is a novel approach to furthering our understanding of the compositional evolution of carbonaceous asteroids and the processes that drive that evolution.

Near-field infrared images and spectra of returned materials from Bennu and Ryugu can be acquired at the Synchrotron Infrared Nano Spectroscopy (SINS) beamline at the Advance Light Source at Lawrence Berkeley National Laboratory. Spectra will be collected using a neaspec neaSNOM near-field system coupled with an AFM tip for focusing the synchrotron infrared beam source. Imaging will be conducted using the same neaspec instrument and a series of tuneable lasers (6 µm and 10 µm) with spatial resolution of 20 nm/pixel.

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Applications of the Neutron-Irradiation Noble Gas Mass Spectrometry (NI-NGMS) technique to determining the halogen composition of returned extraterrestrial materials

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John Pernet-Fisher & Lorraine Ruzié-Hamilton

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The halogens (Cl, Br and I) are volatile elements that are present in most geological samples, typically in trace amounts (ppb-ppm). Despite their generally low abundance, halogens are important volatile species that can provide information on both the primary (e.g., magmatic volatiles) and secondary (e.g., aqueous alteration) history of samples. The low halogen abundance in many samples (especially Br and I) means that their distribution in a wide range of planetary materials is not yet well-constrained. The Neutron-Irradiation Noble Gas Mass Spectrometry (NI-NGMS) method for halogen analysis is particularly well-suited to bulk planetary materials because it is capable of determining low abundance (e.g., ppb Br and I) halogens in the small sample masses (~1 mg) that are typically available for study. Based on the same principle as ^{40}Ar-^{39}Ar dating, NI-NGMS is already well-established for the analysis of chondrite and martian meteorites [e.g., 2-5] and lunar materials [e.g., 6,7]. Neutron-irradiation of samples converts halogens into noble gas isotopes (^{38}Ar, ^{80,82}Kr and ^{128}Xe) through the (n, γ, β) reactions which can then be easily measured by traditional mass spectrometry [1]. Additional information on the chemistry of samples, including K, Ca, Ba and U can be obtained in the same sample extraction from (n, p), (n, α) and (n, γ, β) reactions. Proxy noble gas isotopes can be extracted from mineral phases by laser heating or ablation. Conversion of noble gas to halogen parent concentration requires standards of known halogen concentrations in the neutron irradiation package, placed in close proximity to the samples. Ongoing developments in extractions techniques, such as step-crushing, step-heating, and in situ UV laser-probe at micron-scale spatial resolution, can provide a wealth of information on halogen siting and distribution. The potential applications (and limitations) of the NI-NGMS method to planned (e.g., Ryugu, Bennu, Chang'e 5) and future (e.g., Mars) sample return missions will be discussed.

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3D ANALYSIS OF THE APOLLO 17 73002 CORE SAMPLE TO IDENTIFY STRUCTURES LINKED TO THE LIGHT MANTLE LANDSLIDE EMPLACEMENT MECHANISM.

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The Apollo Next Generation Sample Analysis (ANGSA) program is part of the NASA efforts to maximize the science derived from samples returned by the Apollo Program in preparation for future lunar missions anticipated in the 2020s and beyond. In order to achieve this, NASA recently solicited research on specially curated materials from the Apollo 15, 16, 17 sample collections. The samples were deliberately saved so to take advantage of future's more advanced and sophisticated technology. After being carefully stored and untouched for nearly 50 years at NASA's Johnson Space Center, these precious lunar samples will be opened and studied for the very first time, continuing the science legacy of the Apollo missions.

The NASA ANGSA Science Team is going to examine the Core Sample Vacuum Containers (CSVC) from the Apollo 17 mission. These samples comprise about 800 g of material that penetrates the landslide deposit in the Taurus-Littrow Valley, down to about 60 cm depth. The availability of these lunar samples collected from the deposit of the Light Mantle avalanche represents an unique opportunity to study the stratigraphy of an extraterrestrial landslide deposit.

We will use the XCT-scanning imagery of the 73002 core that has been obtained at the NASA JSC Acquisition and Curation Facility in order to describe 3D characteristics of the landslide deposit. We will conduct a 3D analysis of the grain-size distribution, sorting, and preferred orientation. We will be also looking at microstructures of grains (e.g., fractures) and of the deposit (e.g., shear zone), for these details may reveal information regarding the landslide emplacement mechanism. The results will be integrated with the mineralogy analysis of other ANGSA research groups so to better constraining the modelling of the stratigraphy, temperature, and volatiles of the sample material.

Non-destructive, quantitative measurement of olivine compositions in chondritic material, from synchrotron X-ray microcomputed tomography

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We are developing an X-ray microcomputed tomography (µCT) method for obtaining quantitative non-destructive analyses of olivine in chondritic meteorites and sample return materials. The method is based on density calibration of a set of well characterised olivine grains with a range of compositions across the solid solution series. Tomography was performed on the i13 beamline at the Diamond synchrotron. Since the i13 beam is close to monochromatic, there is a linear relationship between X-ray attenuation and (electron) density of the material, and the brightness values in reconstructed 3D images record a close-to-linear relationship with density. Since density variation across the olivine solid solution series is also linear, image brightness values can be correlated with olivine composition. This method is similar to that described by [1, 2].

We scanned three chondrules (C14, C15, C16), diameters 0.8-1.1 mm, that were extracted from the L3.00 ordinary chondrite, NWA 8276, by freeze-thaw disaggregation followed by gentle crushing. The chondrules are part of a study of I-Xe ages of chondrules (e.g. [3]), and the scans were made so that the petrography of the samples was known prior to destructive analysis for Xe isotopes. Olivine standards were measured in a single continuous helical scan along with the chondrules. Voxel size (xyz) was 1.125 µm. Calibration of the olivine standards, and measurement of unknowns, were carried out using *Fiji* [4]. We found a linear relationship ($R^2 = 1.00$) between olivine composition and brightness values over five olivine standards, with compositions from Fo0 (fayalite) to Fo81. The three chondrules all have different textures, olivine compositions and olivine zonation. As examples, olivine in C14 has a composition Fo99, and line profiles 20 µm in length across zoned olivine grains in chondrule C16 give olivine compositions Fo80 to Fo70. These compositions are consistent with type I and type II chondrules in ordinary chondrites, respectively [5]. The method is a robust non-destructive approach to determining quantitative olivine compositions in chondritic as well as other geological materials.

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The importance of considering martian material, including biomarkers, in MMX analyses of Phobos samples

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JAXA's Martian Moons eXploration (MMX) mission and associated CNES/DLR rover intends to investigate Phobos remotely and *in situ*, and subsequently return samples to Earth in 2029. It is hoped that the data received will supply compelling information towards an origin mechanism for Phobos [1,2].

However, as an airless body, Phobos has been exposed to harsh space weathering and ejected material from Mars that could occupy ~250 ppm of Phobos' regolith [3]. The addition of, and modification to, martian material in Phobos' regolith may result in the samples analysed by MMX being unrepresentative of Phobos' pristine bedrock. This may impede MMX from achieving its mission goals.

Furthermore, ongoing impact modelling using iSALE-2D [4], presented here indicates the possibility that, biomarkers present in martian ejecta could survive an impact into the Phobos regolith. This modelling has investigated the role of variable impactor/target materials and impact parameters to determine the most/least favourable conditions for biomarker survival within Mars-like projectiles impacting Phobos-like surfaces. Light gas gun impact experiments will validate the modelling outputs by firing Mars-like projectiles, including biomarkers, into a Phobos regolith simulant. Analysis of the post-impact simulant will constrain the survival and modification of biomarkers impacting Phobos and reveal the efficiency of impacts at delivering exogenous martian material to Phobos-like regolith.

If biomarkers survived impact, they could become incorporated within samples analysed and returned by MMX. Such material is significant for astrobiology and unravelling Mars' geological past but could invalidate results from MMX analyses. Therefore, a deeper understanding of material transport within the Mars-Phobos system is required. This investigation also illustrates the need for analytical techniques to be made available for returned samples that allow the detection of potential biomarkers and identification of Mars-derived exogenous material among indigenous Phobos materials.

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Evaluating the Mineralogy of Returned Samples from Bennu and Ryugu using Machine Learning Multivariate Infrared Spectral Analysis

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Estimating modal mineralogy for meteorites and asteroids is important for understanding compositional diversity and small body evolution in the early solar system. Meteorite composition can constrain the degree of aqueous alteration and/or thermal metamorphism experienced by the asteroid parent body. Mid-infrared (MIR) spectroscopy is a common telescopic, spacecraft, and laboratory tool used to determine mineral composition. Although mineral identification is possible through MIR spectroscopy, fine-particulates in the MIR do not mix linearly [1], making quantification difficult for some materials. Multivariate analysis removes the assumption of linear mixing across wavelengths and is therefore useful for spectroscopic techniques in which non-linear mixing occurs. It has been utilized as an effective tool for evaluating compositional abundances using several types of spectroscopy [2–4]. This technique requires preparation of a training set covering the relevant compositions and particle sizes. We have constructed an integrated fine (<50 μm) and coarse-particulate (>125 μm) albedo-constrained training set and MIR spectral library to build a machine learning multivariate analysis model for CI and CM meteorite compositions. This machine learning MIR spectral model can be used for quantitative unmixing for fine or coarse particulate materials returned from Bennu and Ryugu.

The Planetary and Asteroid Regolith Spectroscopy Environmental Chamber (PARSEC) at Stony Brook University, is a custom-built planetary environmental spectroscopy chamber for emissivity measurements. PARSEC can be pumped to ~10–4 mbar and cooled to <125 °C allowing for simulated asteroid environment measurements. The capability of PARSEC to simulate measurement conditions similar to Bennu and Ryugu and the development of the machine learning MIR model for quantitative mineralogy unmixing, could aid in characterizing returned sample. With ~500 mg of returned sample, we could estimate modal mineralogy through non-destructive MIR spectral measurements and the application of our model. MIR spectra of the sample from Bennu could be directly compared to OTES measurements for validation and ground truth of remote sensing measurements of primitive bodies.

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Characterizing Organic and Mineral Components in Asteroidal Particles to Constrain Planet Formation Models

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Pebble-accretion is one of the most successful models of planet formation (Johansen and Lambrechts 2017 Annual Review of Earth and Planetary Sciences). Efficient movement of pebbles from the outer to inner disk can lead to high abundances of carbon in the inner disk but that is in opposition to carbon-poor Earth (Andrews et al. 2020). Terrestrial planets formed by pebble accretion can be extremely dry (Coleman et al. 2019 Astronomy and Astrophysics), depending on the recycling capabilities in early formed protoplanets. However, we know that formation of Jupiter greatly hampered the inward drift of carbon- and water-rich pebbles. Addition of both water and carbon in planets via asteroidal delivery, therefore, is an extremely important step in planet formation process.

To understand the planetary 'fossils', the asteroids and their input to the inner solar system, the planetary science community needs to well-characterize samples from different classes of asteroids, including particles acquired by ongoing missions *Hayabusa2* and *OSIRIS-REx*. We have been preparing for analyses of these samples through work on *Hayabusa* samples and analog meteorites. For example, recent analyses of Itokawa particles acquired by the Hayabusa mission provided proof that early formed inner-solar system planetesimals likely formed wet (Jin and Bose 2019 Science Advances; Jin and Bose 2020 In review). Organic and inorganic forms of carbon in pristine unmelted micrometeorites are preserved, inspite of the high abundance of water on their parent bodies (Bose et al. 2020 LPSC). Finally, the identification of thiols, a novel sulfur-bearing molecule in pristine Antarctic carbonaceous meteorites have only been possible because of the non-destructive nature of XANES mapping (Bose and Root 2018 LPSC). Thus, armed with a variety of instrumentation aimed at determining the composition of the organic matter coatings on silicate particles as well as the volatile contents of silicate minerals, our laboratory at Arizona State University is developing a series of novel ways to separate the labile and refractory materials in samples returned through missions. We will process and mount them while maintaining the same level of cleanliness during the entire analytical sequence. I will briefly review our past and future work.

High precision bulk oxygen 3-isotope analysis of returned planetary / asteroidal samples

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High precision bulk O-isotopic measurements of returned extra-terrestrial material offers unparalleled insight into the thermal, aqueous and geological history of the mission targets as well as well as the inter-relationships with other samples [1]. Current and future return missions will likely only return small amounts of sample (grams to 100s of grams). Reliance on grab samples of mixed regolith also means that the returned material may be a mix of multiple lithologies. An example of the potential application of this technique to returned samples will be in determining the origin of the high albedo materials that have been identified by OSIRIS-REx on the surface of the otherwise dark, C-type asteroid Bennu. The spectral properties of these boulders indicate a strong similarity to 4Vesta and eucrite meteorites [2]. The O-isotopic composition of 4Vesta is well established through the study of HED meteorites, and therefore O-isotope measurements will unambiguously confirm/refute the notion that this material on Bennu was derived from 4Vesta, even if only small amounts are present within the returned sample.

The laser fluorination line at The Open University (OU) offers excellent oxygen isotope reproducibility on small samples (~2mg) – providing reproducibility for d^{17}O (±0.05‰ (2 SD)), d^{18}O (±0.09‰) and D^{17}O (±0.02‰) (1). For particularly precious materials, sample size can be pushed down to <100 mg silicate material, while retaining good accuracy and precision (Fig. 1). This capability has recently been demonstrated in the study of micrometeorites [3, 4]. Further studies are on-going to further reduce minimum sample size requirements, and to provide spatially resolved samples (50 µg -2 mg) extracted from CM chondrites using a New Wave Instruments MicroMill^{TM}. This permits careful and conservative extraction of powder from precious samples to be run for oxygen isotopes. Such protocol would prove invaluable when attempting to understand the geological context of returned samples from an oxygen isotopic perspective.

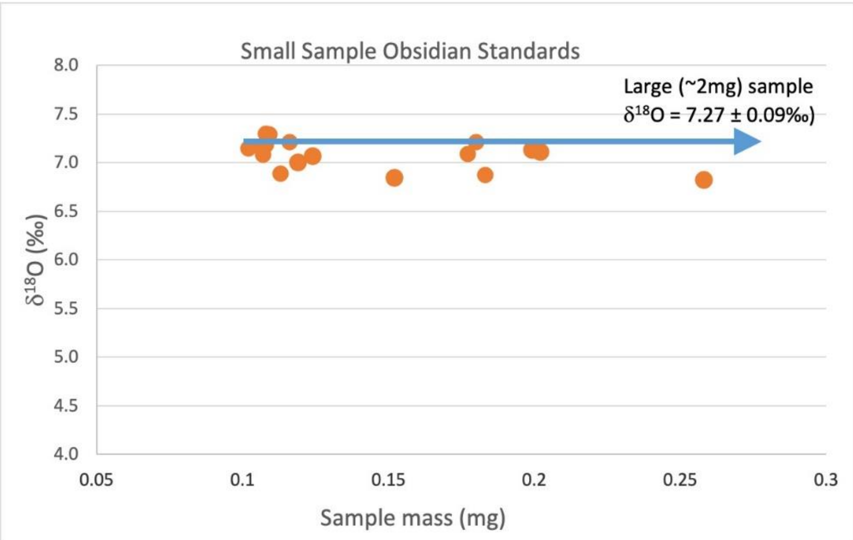

Figure 1. Replicate analyses of an internal obsidian standard (blue arrow) results in an avg δ^{18}O of 7.27 ± 0.095 ‰ (2σ) for 2 mg of powder. For smaller aliquots (orange circles) of 0.1 - 0.26 mg: δ^{18}O of 7.08 ± 0.30 ‰ (2σ) modified after [4].

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UNDERSTANDING THE SPACE WEATHERING OF RETURNED SAMPLES THROUGH COORDINATED ANALYSIS

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The surfaces of airless bodies experience solar wind irradiation and micrometeoroid impacts, a phenomenon collectively known as space weathering. These processes alter the chemistry, microstructure, and spectral properties of grains on airless surfaces. Understanding these effects is critical for interpreting remote sensing data for airless bodies, linking meteorites to their parent asteroids, and for understanding the characteristics of returned samples. Considerable work has been done to understand the space weathering of lunar and ordinary chondritic materials, but work focused on hydrated, organic-rich materials is still ongoing. In advance of sample return, we can simulate space weathering processes in the laboratory and characterize the resulting spectral, chemical, and microstructural effects of constituent space weathering processes. Here we present results from the simulated micrometeoroid impacts and solar wind irradiation of a carbonaceous chondrite. This work indicates that the effects of space weathering on these materials is complex and that the constituent space weathering processes may result in conflicting microchemical and optical characteristics. We will discuss these results in the context of OSIRIS-REx, Hayabusa2, and look forward to sample return missions from the moons of Mars. While space weathering effects the spectral properties of entire airless surfaces, in returned samples the effects are best studied at the nanoscale. We will discuss the importance of coordinated techniques (e.g., SEM, FIB, TEM, reflectance spectroscopy) in the understanding of space weathering features and describe the cutting-edge techniques to be used in the analysis of returned samples from ongoing and future missions.

Siderophile Elements as Tracers of Core-formation: Linking the Lab to Missions

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Siderophile ("iron-loving") elements are powerful tracers of early core-formation and metal-silicate differentiation in planetesimals, and lend insight into planetary formation and evolution. This study focuses on the highly siderophile elements and examines their abundances in mesosiderites, which consist of approximately equal amounts of silicate and metal phases. These meteorites are thought to be breccias that may have formed through impact, break-up and reassembly of a planetesimal. The objective of this work is to investigate the potential relationship between the silicate and metal phases in these meteorites, on the basis of siderophile element abundances. Measurements of mesosiderite metal phases yield chondritic abundances of siderophile elements, suggesting that this metal was molten at the time of impact and did not undergo fractional crystallization, such as that experienced by metal in iron meteorites.

In the context of asteroid missions, analytical advances in laboratory techniques may be linked to geochemical data obtained from mission instruments. While measuring trace elements, such as the "highly siderophiles" will remain difficult to do through remote techniques, it is important to continue these high-precision analyses in laboratory settings. This approach is relevant to future sample return missions because it will advance our knowledge to more robustly link the behavior of elements that are measurable on space probes and those that are currently restricted to the lab. In the case of core formation, the Psyche mission analytical approach emphasizes the importance of measuring Ni (a siderophile) on the surface of the asteroid. If possible, linking these remote measurements to laboratory measurements of highly siderophile elements may provide greater insight into early processes of core-formation on the Psyche asteroid.

The Lost Meteorites of Antarctica: field season results

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The Lost Meteorites of Antarctica project is the first UK led meteorite recovery expedition to Antarctica, with the goal of searching for surface and sub-surface buried meteorite samples [1]. In Austral Summer 2019, a two-person team searched for surface meteorites upon two Antarctic blue icefields: the Outer Recovery icefields, and Hutchison icefields, south of the Shackleton Mountain Range / Recovery Glacier region in the far Transantarctic mountain region in Coats Land [2]. In total 36 meteorites were recovered, ranging from 0.1 g to 2.9 kg in size. In Austral Summer 2020, a five-person team searched for surface and subsurface meteorites, returning a postulated 86 meteorites from the surface of the Outer Recovery area. The meteorites were shipped back to the UK frozen. To date, nearly all have been thawed under vacuum, to minimise any contact or reaction with liquid water.

A preliminary examination plan [2] is being carried out in order to classify all of the meteorites for submission to the Meteoritical Society Nomenclature committee. Data on the sample magnetic susceptibility and electrical conductivity has been collected to facilitate classification [4,5], showing that a wide range of meteorite types have been collected. Computed-tomography (CT) scans have been obtained for 19 of the first season meteorites, and photogrammetry models have been made of the surfaces for the same 19 meteorites to measure and compare their physical properties [6,7]. Aliquots from most of the first season specimens have been used to make thin sections and epoxy blocks, from which we have recently obtained petrographic and mineral chemistry data leading to our first provisional classifications.

We have demonstrated new UK capability to collect samples in the field with procedures designed to minimise contamination and to carry out a preliminary lab examination in a clean room environment. Now that we have established meteorite stranding zones on blue icefields that are accessible by the British Antarctic Survey, we hope to develop a long-term meteorite recovery project to add to the diversity of meteorites in our collection available for scientific study.

Acknowledgements: The Lost Meteorites of Antarctica Project is funded by The Leverhulme Trust, The University of Manchester, and field logistics supported by the British Antarctic Survey.

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Fifty Years of Curating NASA's Astromaterials Collections – Planning for the future

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ARES, Johnson Space Center

For the past 50 years, the Astromaterials Acquisition and Curation Office at NASA Johnson Space Center has curated all of NASA's astromaterial sample collections, with the dual goals of maximizing the scientific return on the samples, while preserving a significant portion of each collection for work by future investigators. The collections at Johnson Space Center include everything from large rock samples (Apollo and meteorites) to small "dust" particles (Cosmic Dust, Stardust, Hayabusa), extending even to solar wind atoms (Genesis). A common infrastructure supports requirements needed in all labs, while the unique requirements of each collection are met on a case-by-case basis. The "lessons learned" from curating each collection are applied retroactively to other collections (where possible) and feed forward into the curation of subsequent collections (e.g., OSIRIS REx), allowing for continual improvement in the overall state of curation of NASA's astromaterials collections.

On the Predicted Nature of the Sample from Asteroid 101955 Bennu

Harold Connolly, Jr.

Rowan University

The OSIRIS-REx mission is NASA's New Frontiers 3 asteroid sample return mission. The spacecraft arrived at asteroid Bennu in December of 2018 and after almost two years of unprecedented exploration of an asteroid with, the goal of winded a site suitable for sample collection, OSIRIS-REx touched the Bennu's surface on 20th October, capturing a significant mass of sample. My talk will summarize key achievements of the mission that inform on the nature of the sample and make predictions as to that nature to be revealed through analysis on Earth in 2023.