



# Polarized Resonant Soft X-ray Scattering (P-RSoXS) as a New Technique for Characterizing Amorphous Astromaterials

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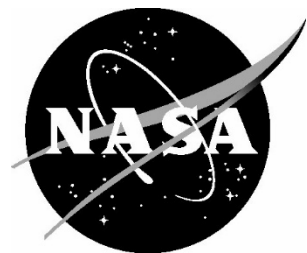
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## **Abstract**

In the coming years, samples will be returned from several asteroids, the lunar surface, and the first material returned directly from the Martian surface. Previous *in-situ* and remote studies indicate these samples will contain abundant amorphous or weakly crystalline materials. However, detailed characterization and quantification of these amorphous materials remains challenging. Conventional techniques, including electron microscopy and X-ray diffraction, provide important information on material structure, but are generally limited to crystalline materials. Polarized resonant soft X-ray scattering (P-RSoXS) is a synchrotron-based X-ray scattering technique that has been used to characterize and quantify weakly crystalline systems, including soft materials. This research aims to translate this technique to geologic and extraterrestrial materials, building off the knowledge of using P-RSoXS to interrogate soft materials. P-RSoXS is well-suited to interrogate geologic materials, which are often multiphase, heterogeneous systems with crystalline and amorphous components, and domain sizes on the order of tens of nanometers. It is anticipated that future work will help reveal new chemical and structural information in geologic materials. Maturation of this preliminary work to develop P-RSoXS to characterize astromaterial-relevant samples will provide new understanding of the secondary processes responsible for the development of amorphous astromaterials and further elucidate our knowledge of the geologic history and past alteration processes.

## **Introduction**

In the next ten years, samples will be returned from asteroids, the lunar surface, and potentially the first material returned directly from the Martian surface. Samples have recently been brought back from several bodies, including Ryugu on the Hayabusa2 mission and lunar regolith from Chang'e-5. The OSIRIS-REx mission just returned samples from the carbon-rich asteroid Bennu to earth, highlighting the urgent need to develop proper material

characterization tools so the scientific community can study these valuable materials and learn new insights into the history and development of astromaterials. Many of the common characterization techniques used to study planetary samples, including electron microscopy and X-ray diffraction (XRD), are limited to identification and quantification of crystalline materials. However, we know that extraterrestrial surfaces have a high concentration of amorphous materials, and this is evidenced by previous analysis, including the “original” return samples from Apollo 11, and from other planetary surfaces and non-Earth satellites (Bibring, et al., 1972; Holmes, et al., 1973; Horgan & Bell III, 2012; Morrison, et al., 2018; Nakamura, et al., 2022; Rampe, et al., 2012; Smith, et al., 2018; Zhao, et al., 2023). Amorphous materials are a major component of most rocky planetary surfaces it is imperative that we can accurately characterize them.

Glasses from volcanism, impact-generated melting, and impact-generated shock have been identified in samples returned from the Moon and lunar meteorites (Delano, 1986; Plescia & Cintala, 2012; Schaal, et al., 1979), and thin (<200 nm thick) rims of amorphous material have been identified on surfaces of lunar soil grains from space weathering (Cymes, et al., 2021). Spectra of carbonaceous chondrites and some asteroids are consistent with amorphous silicates, suggesting a primitive origin for the asteroids with amorphous silicates (McAdam & Gustafsson, 2021). Orbital infrared data and in-situ spectral and X-ray diffraction data from the Martian surface show a variety of X-ray amorphous materials. These amorphous materials have been interpreted as volcanic glasses, silicates from aqueous alteration, secondary nanophase Fe oxides/oxyhydroxides, and sulfates (Bandfield, et al., 2000; Morris, et al., 2000; Rogers & Christensen, 2007; Ruff, et al., 2011; Rampe, et al., 2011; Rampe, et al., 2012; Sun & Milliken, 2018; Chipera, et al., 2023). Amorphous materials are also recognized in Martian meteorites, including shocked plagioclase (maskelynite), volcanic glass, and secondary amorphous silicates in veinlets (Treiman, 2005; Treiman, et al., 1993; Mikouchi, et al., 1999; Changela & Bridges, 2010; Hallis, et al., 2014; Rubin, 2015). The composition and any short-range atomic order within these extraterrestrial amorphous materials can tell us more about how the amorphous materials formed and the geologic history of a particular planetary body. With samples returned from or soon to be returned from asteroids, comets, the lunar south pole, Mars, and Phobos, identifying the best techniques for characterizing amorphous materials is critical.

Mars-analog amorphous materials have been studied in terrestrial laboratories to characterize their short-range order, composition, and formation mechanisms. Allophane, Fe-substituted allophane, imogolite, ferrihydrite, and amorphous sulfates have been synthesized in the laboratory and studied with various techniques (e.g., XRD, VNIR and TIR spectroscopy, evolved gas analysis, TEM, X-ray absorption spectroscopy) (Rampe, et al., 2012; Bishop, et al., 2013; Baker, et al., 2014; Morris, et al., 2015; Rampe, et al., 2016; Jeute, et al., 2021). Natural analog materials, including basaltic soils and sediments from Hawaii, paleosols from the John Day Fossil Beds in Oregon, fluvial sediments from Iceland, and proglacial sediments from Three Sisters, Oregon, have also been studied using analog instruments and electron microscopy (Morris, et al., 1993; Rutledge, et al., 2018; Smith, et al., 2018; Thorpe, et al., 2019; Rampe, et al., 2022). These studies demonstrate that secondary amorphous materials are primarily Fe-, Si-, and/or Al-bearing and have heterogeneous compositions, often within the same sample.

Artemis 4 is planned to put humans back on the moon in 2028, and now is the time to develop proper analysis protocols of the lunar regolith. Simulants, although not strictly astromaterials, are an important class of materials that also contain a significant amorphous fraction that has remained difficult to analyze. Many lunar simulants contain upwards of 50% by weight amorphous or glassy material (Schrader, 2010). It has remained difficult to develop high-fidelity simulants due to challenges in identifying the amorphous phases (Kovtun, 2023). Accurate simulants are needed for testing dust mitigation techniques, extra-vehicular activities, suit development, and external structures and mechanisms. Traditional pXRD (powder X-ray diffraction) has fallen short of providing mineralogical and morphological information for rigorous simulant analysis and quality control, leading to a loss of proper testing and immediate shortcomings in insight and mitigation efforts (Kovtun, 2023). This provides a timely opportunity to study and demonstrate new capabilities that can obtain information about amorphous phases in simulants.

This technical memorandum presents resonant soft X-ray scattering (RSoXS) as a promising new tool to characterize astromaterials that contain significant amorphous character. This research is predicated on direct, remote, and *in-situ* data that indicate amorphous materials are abundant throughout our Solar System (Abreu & Brearley, 2010; De Gregorio, et al., 2016). Preliminary data suggest that geologic samples are well-suited for RSoXS characterization and show that RSoXS can provide unique information on length scales associated with chemically distinct phases in amorphous materials and inform alteration mechanisms. This will provide a more comprehensive

view of the effects that aqueous, volcanic, atmospheric, meteoritic, and potentially biologic alteration processes played on planetary and satellite surfaces.

## Technical Background

Resonant Soft X-ray Scattering (RSoXS) is an emerging technique that can characterize weakly crystalline or traditionally X-ray amorphous polymers and electronic and magnetic ordering in metallic materials (Ade, et al., 2009; Fink, et al., 2013; Comin & Damascelli, 2016; Carpenter, et al., 2015; Milne, et al., 2014; Collins, et al., 2012). RSoXS provides molecular and nanoscale morphological information of amorphous (i.e., lacking atomic order) and weakly crystalline (i.e., having short-range order over  $\sim 10$ s of nm), multiphase systems that is difficult to obtain with other methods like X-ray absorption spectroscopy (XAS) or XRD. This technique has recently emerged to study multiphase polymer systems (Su, et al., 2016; Collins, et al., 2011) with significant amorphous content and it is poised to uncover new insights into formation or development processes in amorphous or weakly crystalline geologic materials. The power of RSoXS to characterize amorphous phases and elucidate short-range structure in seemingly amorphous material will provide invaluable information about the geologic history of the solar system.

Although TEM with electron dispersive spectroscopy (EDS) has shed light on the short-range order and composition in many analog amorphous materials, this local technique can only be used to study individual grains and may not be representative of the whole sample. X-ray scattering methods provide statistically averaged information on atomic to mesoscale periodicities due to the relatively large sample area (up to hundreds of  $\mu\text{m}$ ) illuminated by the X-ray beam. Resonant soft X-ray scattering (RSoXS) has been used by the soft and biologic material community for over a decade. By tuning the energy of incident X-rays in resonance with core electron transitions, it is possible to overcome the limited electron density contrast between materials with similar elemental makeup and high concentrations of amorphous material and enhance contrast between materials, as described by the schematic in Figure 1. RSoXS combines X-ray absorption spectroscopy and scattering to provide structural and morphological information of amorphous or weakly crystalline systems (Swaraj, et al., 2009; Virgili, et al., 2007; Ade & Hitchcock, 2007; Ade, et al., 2009). While extensive experimental work has been performed at energies near the absorption edges of the elements in soft materials (C, N, and O K-edges), there have been some studies that leverage the L-edge of first-row transition metals and alkaline earth metals, such as calcium and iron, and biologic samples (Ingham, et al., 2018; Ingham, et al., 2018; Kuiper, et al., 1993; Ye, et al., 2018). RSoXS has successfully studied heterogeneous materials with phases of different elemental compositions on the order of tens of nanometers in size, like the Martian materials identified by the Alpha Particle X-ray Spectrometer APXS (Thompson, et al., 2019; Smith & Horgan, 2021; Virgili, et al., 2007; Ade & Hitchcock, 2007).

Polarized RSoXS (P-RSoXS) is uniquely able to probe molecular orientation by taking advantage of polarized synchrotron X-rays. Such local molecular orientation can lead to scattering anisotropy, a phenomenon seen in both organic and inorganic materials (Ingham, et al., 2018; Litofsky, et al., 2019; Mukherjee, et al., 2025; Mukherjee, et al., 2021; Nguyen, et al., 2024). By collecting scattering profiles with different incident polarizations, we can further quantify nanoscale domains in complex heterogeneous materials. In a sample with multiple domains, such as a heterogenous inorganic sample, differences between the alignment or orientation between the units can provide orientational and chemical contrast; this orientational contrast gives rise to anisotropic scattering. Rather than traditional radial integration as a function of scattering vector  $q$ , these anisotropy studies calculate the scattering parallel to and perpendicular to the electric field. To quantify the degree of scattering anisotropy of the sample, the anisotropy ratio,  $A(q)$ , can be used. The value is calculated as:

$$A(q) = \frac{I_{0^\circ}(q) - I_{90^\circ}(q)}{I_{0^\circ}(q) + I_{90^\circ}(q)}$$

where  $I(q)$  is the scattering intensity at a specific polar angle ( $\pm 5^\circ$ ). This ratio varies from +1 to -1, where +1 and -1 indicate perfect, universal alignment of the domains of interest. This analysis has also been shown to provide information about complex morphologies (Litofsky, et al., 2019; Tumbleston, et al., 2014; Mukherjee, et al., 2021; Wang, et al., 2011), crystallinity in X-ray amorphous materials (Litofsky & Gomez, 2019), novel morphologies in multiphase systems (Ye, et al., 2018; Litofsky, 2019), phase purity (Liu, et al., 2013; Mezger, et al., 2011), and protein

structures in living materials (Ye, et al., 2018). This added information enables more in-depth analysis of materials, including previously undetected phases from a variety of alteration processes.

In addition to polarization-dependent scattering contrast, one can generate contrast in P-RSoXS experiments by modulating the energy of the incident X-ray beam (Arges, et al., 2017; Ye, et al., 2018; Ye, et al., 2018; Ingham, et al., 2018). For multiphase systems, we will be able to analyze, in sequence, each of the constituent phases of a complex system to isolate specific features or effects and processes that have occurred (Litofsky, et al., 2019). Polarization and energy dependent studies of multiphase material systems have revealed 1) domains hypothesized but not previously seen; 2) resolution down to the sub-micron level; and 3) a method for quantifying the degree of crystalline ordering (Litofsky & Gomez, 2019; Ye, et al., 2018).

## Technical Approach and Methodology

Traditional XRD typically uses hard, or higher energy (~10 keV or greater) X-rays to probe the atomic structure of crystalline materials. This allows for thicker samples and atomic resolution (due to smaller wavelengths) relevant to unit cell parameters. However, XRD is often performed at a fixed high energy where contrast is based on electron density differences. This limits elemental and chemical specificity, especially among lighter elements. An innovative way to combat this chemical blindness is through tuning the incident X-ray energy to match the energy of transitions of core-level electrons to unoccupied states. Lower energy soft X-rays (~150-2000 eV) span core-level transitions of elements found in organic compounds (C, N, O), silicon, aluminum, and the L-edge of the first-row transition metals. Soft X-rays are uniquely able to probe carbon-based phases, biologic alterations, and detailed speciation among transition metals.

P-RSoXS leverages absorption differences between phases or materials. At energies near absorption edges, the scattering contrast is governed largely by changes in optical properties rather than by the electron density differences that govern hard X-ray contrast; thus, small differences in refractive indices can lead to large enhancements in scattering contrast, providing increased chemical specificity. In P-RSoXS experiments with linearly polarized X-rays, only bonds with transition dipole moments (TDMs) that are oriented parallel to the incident electric field interact with the X-rays. For example, in a benzene ring, the TDM associated with the excitation of a core level electron to an unoccupied  $\pi^*$  orbital points perpendicular to the ring plane. This phenomenon provides an orientation-based contrast mechanism in addition to the chemical contrast. Orientational contrast has been seen in inorganic materials in literature and in our work, including silicates, pyroxenes, and amphiboles (Dyar, et al., 2002; Dyar, et al., 2002; Munoz, et al., 2013).

Polymorphs with identical stoichiometry can produce different scattering signals with different anisotropy ratios, as indicated in Figure 1. The difference between well-known and easily distinguishable polymorphs, such as quartz and cristobalite, should be distinguishable via P-RSoXS. It would be informative to combine P-RSoXS with real space probes like TEM and IR nanospectroscopy imaging. P-RSoXS has the potential to elucidate new information, such as local ordering within amorphous phases, slight orientational changes, such as bending or twisting in the minerals.

We can use P-RSoXS to selectively interrogate specific phases by leveraging both chemical and orientational contrast contributions to scattering profiles. A sample of quartz and volcanic silica glass will have unique resonant energies to examine the contrast between the two phases, for example, the quartz phase vs. the glass phase. Energy tunability allows P-RSoXS to probe specific phases within a heterogeneous sample even if the phases are amorphous. Furthermore, P-RSoXS can identify formation mechanisms of materials, for example, it has been shown (Litofsky, 2019) and is expected to inform identification and quantification of contaminants, inclusions, biosignatures, or other alteration mechanisms as shown in our data (Figure 2). Changes that induce alignment or dehydration of lunar soil due to micrometeoroid impacts could be uniquely detected, characterized, and quantified by P-RSoXS (Benna, et al., 2019). Early work has already demonstrated the ability of P-RSoXS to detect novel structures within microbe-containing structures. To that end, the goals of this work are three-fold:

1. Evaluate the efficacy of P-RSoXS analysis of analog samples for detecting signatures of weathering and geologic history
2. Demonstrate the ability of P-RSoXS to detect alterations in carbon-containing material in analog samples

3. Compliment other techniques, such as electron microscopy, with information uniquely provided by P-RSoXS

## Materials and Methods

### Sample Selection and Preparation

Synthetic and analog samples were selected based on geologic similarities to those found in astromaterials or other return samples. Based on prior work, quartz, cristobalite, hematite, 2-line ferrihydrite, amorphous Mg-sulfate, amorphous Fe(III)-sulfate, and allophane were synthesized for the study (Rampe, et al., 2012; Rampe, et al., 2016; Morris, et al., 2015). Analog samples were selected from two proglacial mafic sediment samples from Three Sisters, Oregon. These samples include plagioclase, feldspar, clinopyroxene, orthopyroxene, olivine, magnetite, ilmenite, hematite, zeolite, and phyllosilicates that have been recently characterized by XRD and TEM-EDS (Rampe, et al., 2022). Many of these materials are geologically like those found on the Martian surface. Analysis of the analog samples will provide insight into characterizing amorphous materials in complex samples using RSoXS.

Analog samples were prepared similar to TEM preparation (Rampe, et al., 2022). Samples were suspended in ethanol and deposited, or “picked up,” on a 5mm by 5 mm silicon frame with a 1 mm x 1 mm 100 nm thick silicon nitride X-ray windows (Norcada) (Litofsky, et al., 2019). Synthetic samples were microtomed to appropriate size and thickness and then suspended in ethanol for deposition onto the X-ray windows.

### Beamline X-ray Scattering

X-ray scattering was performed at both Beamline 11.0.1.2 at the Advanced Light Source (ALS) at Lawrence Berkeley National Lab and Beamline SST-1 at the National Synchrotron Light Source II (NSLS-II) at Brookhaven National Lab. A transmission geometry was used under vacuum near the carbon (280 eV), nitrogen (400 eV), and oxygen (530 eV) K-edges and the iron L-edge (700 eV). An off-resonance measurement at 900 eV was also performed. The X-rays were linearly polarized in the parallel (0°) and perpendicular (90°) directions. 2D scattering patterns were integrated azimuthally as a function of scattering vector ( $q = 4\pi \sin(\theta/2) / \lambda$ , where  $\theta$  is the scattering angle and  $\lambda$  is the wavelength). Data were corrected for the dark current and changes in beam flux, and the beam stop was masked. More information can be found in supporting literature (Virgili, et al., 2007).

## Results

### Computational Contrast Analysis

The ability to distinguish between polymorphs, while already achievable through other methods, provides an appropriate proof-of-concept demonstration for P-RSoXS. This demonstration can show how low energy polarized X-ray scattering can reveal orientational or structural contrast within chemically identical materials, a necessity when attempting to elucidate structures in amorphous materials. Figure 1 shows simulated near edge X-ray absorption fine spectra (NEXAFS) spectra for two polymorphs of silicon dioxide (quartz and cristobalite) and hematite, demonstrating that P-RSoXS can distinguish between these phases.

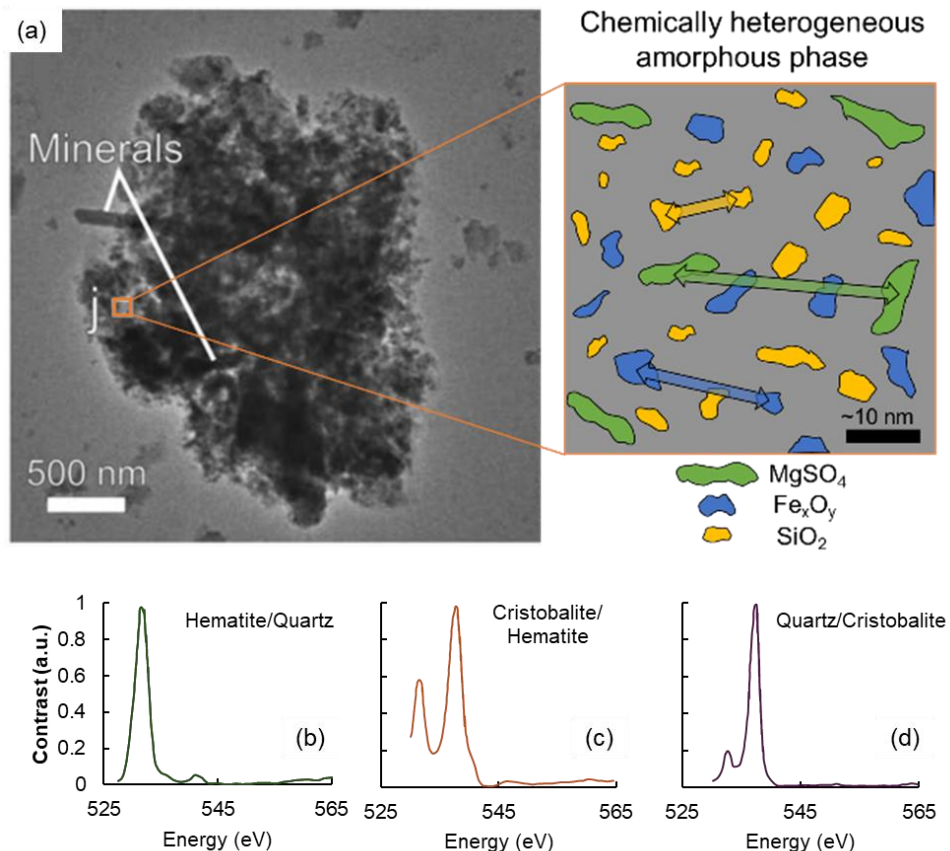


Figure 1. Schematic depicting the ability of RSoXS to identify, distinguish, and quantify domain size, spacing, and chemical identity in an amorphous Martian analog material (a). Micrograph adapted from (Smith & Horgan, 2021). (b-d) Preliminary scattering contrast between different material pairs calculated based on simulated NEXAFS spectra generated by RSoXS in potential phases within an inorganic sample.

A weakly crystalline Martian analog material including semicrystalline nanophases of iron oxide and silicon dioxide was used as a model system (Figure 1a) for this analysis. The absorption spectra, as shown in Figure 1b-d, highlights the distinct contrast between each of the examined phases in the model structure. This analysis was done at the oxygen K-edge, roughly between 525 and 550 eV, to provide appreciable scattering from each of the examined phases.

Contrast between quartz and cristobalite (Figure 1d) is significant between 530 and 540 eV. Because of the lack of chemical contrast between the two chemically identical species, the contrast seen in the figure is uniquely driven by orientational contrast; that is, the difference in the TDM interfacial alignment between the quartz and cristobalite phases. While the contrast between cristobalite and hematite (orange) and hematite and quartz (green) is driven by both chemical and orientational contrast, the substantial orientational contrast between quartz and cristobalite demonstrates P-RSoXS's utility and efficacy to examine heterogeneous materials. The spectra shown in Figure 1b-d demonstrate that there is more than substantial contrast to perform soft X-ray scattering experiments on these materials provided proper sample preparation. Even though there exists overlap between peaks on the spectra, the slight deviations between the individual spectra furthermore allow for tuning of the phase interest. For example, slight adjustments to the incident beam energy around 530 eV can preferentially select for the examination for any of the three phase pairs.

## Scattering Analysis of Synthetic Standards

Analysis of RSoXS data was performed through heritage methods (Litofsky, et al., 2019). The synthetic and analog samples were selected to verify the efficacy of RSoXS to reveal structures and signatures of formation mechanisms in relevant inorganic materials and the ability of the technique to provide quantitative characteristics, such as correlation lengths, orientation, and anisotropy, of planetary and geologic materials. Radially integrated data are shown in Figure 2. Initial RSoXS measurements reveal interesting and novel information, which is discussed in more detail below.

RSoXS data at energies that reveal the clearest scattering features are shown in Figure 2, as well as a comparative scan at an off-resonant energy of 900 eV to demonstrate changes due to resonance. Arrows in Figure 2 point to peaks or shoulders in the RSoXS curves. Our preliminary data show the ability of RSoXS to differentiate nanoscale features among polymorphs; for example, the tridymite-quartz-amorphous silica trio (d, e, and f, respectively) all have very distinct scattering patterns. Additionally, RSoXS clearly resolves the grain structure in tridymite (d), as seen by the shoulder at  $q = 0.1 \text{ \AA}^{-1}$  (6 nm) (Fröhlich, 2020; Hatipoğlu, et al., 2015) as well as the potential quartz (e) grain structure of 4 nm at  $q = 0.16 \text{ \AA}^{-1}$  (Buckley, et al., 2018) ( $q = 2\pi / d$ ). While this result can also be seen in pXRD, RSoXS will differentiate between other polymorphs, such as opal and cristobalite, where pXRD may need large sample sizes in powder form (Elzea, et al., 1994). Furthermore, RSoXS reveals a phase with a 3 nm correlation length in amorphous silica (f), as denoted by the peak at  $q = 0.21 \text{ \AA}^{-1}$ . This suggests that while the silica (f) may appear amorphous using pXRD, there are some larger length-scale structures or grains present in the material. Our preliminary data shows that RSoXS can differentiate between these silica polymorphs and be used to identify discrete formation mechanisms, geologic history, and potential alteration.

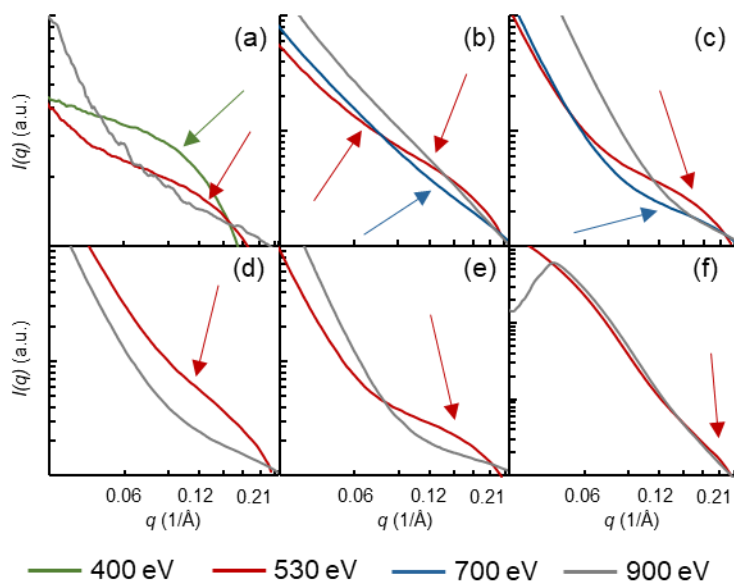


Figure 2. Radially integrated RSoXS scans at multiple energies for cryogenic magnesium sulfate ( $\text{MgSO}_4$ ) (a), 2-line ferrihydrite ( $\text{Fe}_5\text{O}_8\text{H}$ ) (b), hematite ( $\text{Fe}_2\text{O}_3$ ) (c), tridymite ( $\text{SiO}_2$ ) (d), quartz ( $\text{SiO}_2$ ) (e), and amorphous silica ( $\text{SiO}_2$ ) (f). Scattering profiles are shown at 400 eV (nitrogen K-edge; green), 530 eV (oxygen K-edge; red), 700 eV (iron L-edge; blue), and 900 eV (off-resonance; gray). Arrows highlight features in the curves.

2-line ferrihydrite also shows interesting features (Figure 2b). It shows RSoXS features at around 10 nm ( $q = 0.06 \text{ \AA}^{-1}$ ) and 5 nm ( $q = 0.12 \text{ \AA}^{-1}$ ), potentially associated with variable grain sizes due to its composite structure (Sassi, et al., 2021; Funnell, et al., 2020; Michel, et al., 2007). Interestingly, the peaks corresponding to these different length scales appear at different energies; the 10 nm feature, and its second order reflection at  $q = 0.12 \text{ \AA}^{-1}$ , occurs at

the oxygen edge (530 eV) while the 5 nm feature occurs at the iron edge (700 eV). This indicates multiple alteration mechanisms or multiple phases within the sample with varying oxygen and iron compositions, speciation, or bonding environments. This also highlights the unique energy-based elemental sensitivity of RSoXS. RSoXS measurements at smaller energy increments around these elemental absorption edges and polarization-dependent scattering will further elucidate this complex structure.

The most interesting sample is the amorphous cryogenic magnesium sulfate (a). Strong resonant peaks are expected at 530 eV (O), 1.25 keV (Mg), and 2.31 keV (S). However, a peak is also seen at 400 eV (near the nitrogen K-edge). Because this peak is not present in any other sample or the bare substrate, this feature is unique to amorphous cryogenic magnesium sulfate. These data suggest the cryogenic magnesium sulfate sample may contain nitrogen inclusions with a 7 nm characteristic length scale ( $q = 0.09 \text{ \AA}^{-1}$ ), possibly from the sample preparation. As the sample is submerged in liquid nitrogen, some of the liquid may penetrate the sample. Once the sample returns to room temperature during testing, the liquid nitrogen evaporates to become gaseous nitrogen, and some of this vapor may be trapped in the sample. While this feature has not been seen before, the mechanism has been previously studied (Tedder, et al., 1985). This inclusion is clear evidence of the ability of RSoXS to identify previously hidden alteration processes in inorganic materials. Further analysis using the same scattering data can examine these inclusions further.

## Polarization-Dependent Analysis of Analog Samples

Polarization-dependent analysis can reveal insights on molecular orientation (Collins, et al., 2012; Litofsky & Gomez, 2019; Mukherjee, et al., 2025). This analysis was performed on a Martian analog material from Collier Glacier (Figure 3) (Rampe, et al., 2022). Even though these analog materials have been characterized by XRD and electron microscopy (Figure 3a), P-RSoXS elucidates novel morphological information with the potential to inform alteration mechanisms.

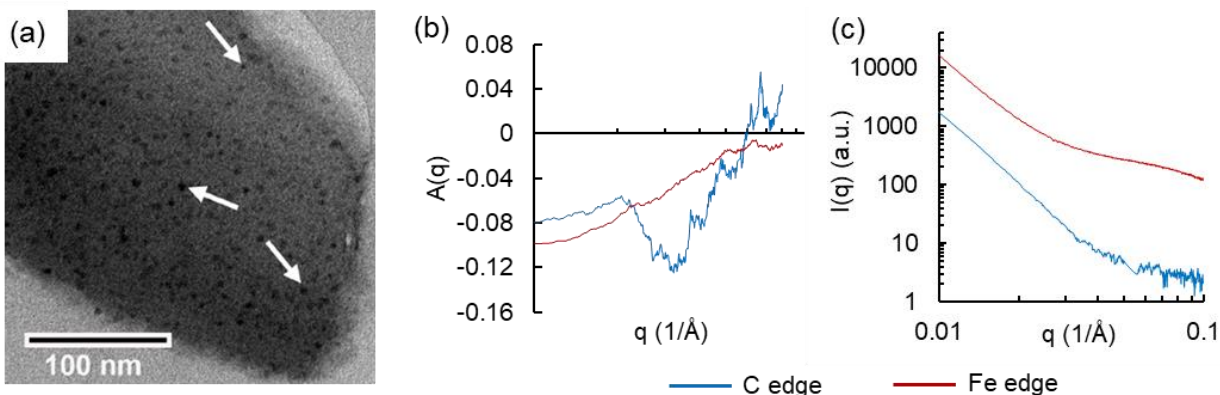


Figure 3. Analysis of Collier glacial flour. (a) TEM image of glassy grains with 5-10 nm nanoscale crystallites (reprinted from Rampe et al., 2022); (b) anisotropic and (c) traditional RSoXS analysis.

We observe at least two different mechanisms or morphologies at play within this sample of glacial flour. The first of these morphologies come from amorphous carbon-containing material in the glacial flour. The blue curve in Figure 3c shows intensity vs  $q$  data and Figure 3b shows anisotropic ratio analysis at the carbon edge. There is no indication of crystalline carbonate in the XRD, implying all carbon-containing materials are amorphous. The lack of a peak in the traditional RSoXS analysis verifies this morphology and indicates that there are no preferred sizes for amorphous carbon-containing grains.

However, as seen by the distinct peak at  $0.033 \text{ \AA}^{-1}$  ( $19.0 \text{ nm}$ ) within the P-RSoXS analysis (Figure 3b), there is a clear preferred orientation and spacing of the carbon-containing materials. This preferred orientation, coupled with additional knowledge about the sample, can inform alteration mechanisms (Litofsky, et al., 2019). While the

source of this carbon-containing material is unknown, it may be derived from dissolved organic matter (Hood, et al., 2009; Stubbins, et al., 2012; Stibal, et al., 2012). Certainly, more examination is needed into this material and this specific marker considering the presence of siderite, an iron carbonate, and other carbonates on the Martian surface (Archer, et al., 2020). The ability of P-RSoXS to resolve orientation within carbon-containing Martian analog samples, even when other techniques like XRD and electron microscopy could not, underscores the strength of P-RSoXS. While it is unclear the origins of this signal, indication of microbe activity or other biosignature cannot be ruled out until further investigation is performed.

P-RSoXS can also uniquely characterize iron in this glacial flour. While there are an abundance of iron-containing minerals in this sample, including augite, pigeonite, and orthopyroxene, the only feature in the iron L-edge  $I(q)$  plot (Figure 3c) exists as a shoulder at approximately  $0.08 \text{ \AA}^{-1}$ , corresponding to the 5-10 nm size aggregates as mentioned in the original analysis and shown in Figure 3a and seen in other literature (Rampe, et al., 2022; Sun, et al., 2017). The additional presence of a low  $q$  negative region at  $0.02 \text{ \AA}^{-1}$  and below suggests a preferential orientation of amorphous iron compounds at larger (100 nm and above) features. More experimentation, including a change of geometry to better probe the low  $q$  region, will verify this hypothesis; however, this preferential orientation has been verified computationally and previously seen in synthetic samples (Cölfen & Mann, 2003; Sun & Milliken, 2018; Dabat, et al., 2018). It is hypothesized that this anisotropy is derived from a pH gradient of approximately 4 to 7 during glass formation or the presence of organic matter (Parfitt, 1990; Perrott, 1978). This preliminary work provides the first experimental verification of this phenomenon on geologic samples and showcases the ability of RSoXS to decipher new alteration processes. Other structures containing collections of similar sized aggregates with organic material have been found (Janney, et al., 2000). Furthermore, using pH as a method for determining geologic history has a prevalent basis in planetary science, such as the identification of an alkaline environment in early Martian history that led to the development of phyllosilicates (Chevrier, et al., 2007; Baldrige, et al., 2009). The ability of P-RSoXS to identify structures that result from pH gradients may provide even deeper insight into planetary histories.

## Forward Work

### Anticipated Alternation Mechanisms

Because the features and signs that are seen through analysis of geologic materials by P-RSoXS are mostly unknown and novel, a better understanding of the mechanisms that cause these alterations will be needed. Identification of specific alteration mechanisms is crucial to using P-RSoXS as a precision tool for astromaterial characterization. A useful example can be demonstrated with hydraulic-induced alteration of hard materials. As seen in previous literature, dehydration induces an increased P-RSoXS anisotropy peak (Rongpipi, et al., 2021). This signature can be easily confirmed using analog samples. For lunar, asteroid, and Martian samples, this signature can be uniquely identified by RSoXS and support the historical presence of water. We aim to investigate other unique features like pH environments. While RSoXS has not been used to identify historical pH environments, RSoXS has clearly demonstrated its ability to identify active alteration mechanisms (Ye, et al., 2018; Ye, et al., 2018; Litofsky, et al., 2019; Rongpipi, et al., 2021). Due to the innate ability of high pH gradients to institute a preferential orientation within materials, materials exposed to these environments are expected to show higher magnitude anisotropy ratios than their counterparts; this is readily verifiable by the analysis of analog samples, and our preliminary data provides a basis for this. Lastly, the microscale erosion on the lunar surface due to meteoroid impacts, while extensively studied previously, will be better quantified through RSoXS (McDonnell, et al., 1972). High impact or force events, such as meteoroid impacts or shocks, have been shown to increase the degree of alignment of weakly crystalline materials, which can be revealed by P-RSoXS studies (Litofsky & Gomez, 2019).

### Multimodal Analysis

Multiple characterization probes are often needed to reveal a more complete picture of a material. Complementary real-space imaging with other synchrotron techniques, including infrared nanospectroscopy and X-

ray microscopy/tomography will complement statistically significant, reciprocal space RSoXS characterization to provide additional insight into the spatial distribution of phases and/or polymorphs in heterogeneous extraterrestrial samples and provide a unique multimodal characterization pipeline. Light sources have unique capabilities in synchrotron infrared nanospectroscopy (SINS) (Bechtel, et al., 2020). SINS leverages an atomic force microscope (AFM) tip for near-field IR spectroscopy with nanometer resolution, effectively combining the surface topography and phase information from AFM with the chemical sensitivity of IR spectroscopy at the resolution of the AFM tip (~25 nm). Since SINS can provide nanoscale spatially resolved chemical information on both crystalline and amorphous phases, this will provide a new way to map out the distribution of amorphous phases with distinct elemental composition, e.g., Fe-, Si-, or S-rich phases, or polymorphs, e.g., quartz vs. cristobalite. A recent study characterized samples from Alan Hills (ALH) 83100, a CM1/2 carbonaceous chondrite (Young, et al., 2022), with SINS demonstrating the utility of nanoscale FTIR. SINS revealed small, fine-grained phases in the meteorite that contribute to its bulk IR spectra, including resolving the distribution of Fe-Mg variations in the phyllosilicates surrounding chondrule rims. These findings suggest aqueous alterations in ALH83100. SINS can be performed on any sample that is AFM-compatible and should work well with the same samples used for RSoXS. Additionally, the Advanced Light Source (ALS) has recently developed a tender X-ray full-field transmission microscopy and nanotomography beamline (beamline 11.3.1) that can image in 2D down to 75 nm. The energy range (5-17 keV) complements RSoXS providing sensitivity to elements, for example, the Fe K-edge. This combination of complementary probes will help develop new multimodal characterization workflows for planetary samples.

## Impacts of Sample Sterilization

Return samples will be subjected to biological sterilization treatments to kill any potentially hazardous microbes. One possible sterilization method is heating the samples to 250 °C for 30 minutes or 180 °C for three hours (Neal, 2000). This type of sterilization may affect the short-range atomic order of sensitive hydrated amorphous materials and cause them to crystallize (partially or fully) into minerals. It is anticipated that by combining the minerals' orientation changes with correlation lengths, P-RSoXS can correlate these variations to their thermal environment during formation. To examine the effects of heat sterilization, materials can be heated and analyzed using P-RSoXS with *in-situ* heating. Complimentary temperature-dependent powder XRD can also be performed (Jin, et al., 2020).

Gamma radiation treatment is another sterilization technique used for returned samples. The radiation dose currently used is 100 Mrad, which can be achieved in less than 30 days using a variety of academic sources in the United States, such as the Co-60 source at the Breazeale Nuclear Reactor at The Pennsylvania State University. Previous work has suggested that intense gamma radiation exposure produced no discernable changes in the morphology or crystallinity of materials, as demonstrated on soil analogs (Allen, et al., 1999). No measurable changes were recorded in these samples across the surface and bulk isotopic and chemical properties. However, visible to near infrared (VNIR) spectroscopy indicated some changes in quartz and plagioclase, and these changes must be examined in more depth, along with analysis of amorphous content. P-RSoXS has the potential to identify mesoscale ordering changes, including changes in orientation, domain size, domain spacing, and elemental contrast. SINS could also provide additional insight into the local heterogeneities that may influence changes seen by bulk NIR spectroscopy after gamma radiation treatment.

## Astromaterial Sampling and Analysis

All testing described in this work was performed on synthetic or analog samples. However, characterizing true astromaterials with RSoXS is a longer-term goal. Lunar soils and glasses, and primary and secondary amorphous Martian astromaterials from meteorites are good candidates. Apollo 15 soil samples have a basaltic protolith, whereas Apollo 14 and 16 samples have feldspathic impact ejecta protoliths. The lunar samples, and their maturity indices, include: 14163 (57, submature), 14259 (85, mature), 15101 (70, mature), 15401 (5.6, immature), 61220 (9.2, immature), and 62231 (91, mature). It would be of interest to analyze the <25 µm split analyzed via micro-FTIR (Morlok, et al., 2022), which identified spectral bands from a glassy/amorphous component in all samples related to

the presence of glasses and space weathering. While the abilities of FTIR should not be forgotten, advantages of using RSoXS include enhanced contrast, and therefore easier structural elucidation, at low-Z elements, high sensitivity to weak molecular orientation, and the ability to examine buried interfaces (Ferron, et al., 2023).

Three Martian meteorites (MIL 090032, MIL 03346, and EETA 79001) can be sectioned via focused ion beam (FIB), ensuring maximum functionality and sample-use efficiency. Nakhilite MIL 090032 contains poorly ordered Fe-smectite/amorphous silicate gel in veins (Hallis, et al., 2014). Nakhilite MIL 00346 contains X-ray amorphous materials in the mesostasis, consistent with igneous glass, and ferrihydrite in veins (Ling & Wang, 2015). Shergottite EETA 79001 is well-known for having impact-shock melt (Mikouchi, et al., 1999).

## Conclusion

This preliminary work has demonstrated that polarized resonant soft X-ray scattering (P-RSoXS) can provide morphological information and correlation lengths in amorphous astromaterials. By leveraging existing analog and well-characterized synthetic materials, these data outline how to utilize this technique for gaining unique insight to return samples and other astromaterials, including its ability to detect previously unidentified inclusions and hypothesized pH gradients during mineral formation and dissolved organic matter within the amorphous matrix of samples. Further work is needed to verify these findings and develop this technique more broadly for planetary materials. It is anticipated that P-RSoXS could allow for the ability to better understand the thermal and radiation environment during formation, elucidate secondary alteration mechanisms, and identify past traces of water and potentially life.

As a community, it is imperative that we maximize the productive science that can be performed on the limited amount of astromaterials that can be tested in a lab. There is great interest in understanding planetary history, alteration processes, and potential signs of life in our solar system. While the field has, to great effect, used existing characterization techniques, there are opportunities to expand on the current methods to uncover new insights. P-RSoXS helps fill this gap by providing information that is difficult to obtain from traditional analytical and experimental methods. The chemical and orientational contrast to which P-RSoXS is sensitive can resolve ordering in materials that were previously thought to be fully amorphous. This ability can identify and quantify alterations in astromaterials, including effects from hydration, pH, temperature, and potentially life. Combining this technique with existing and newly maturing methods can improve our understanding of astromaterials and return samples. These samples need to be characterized with other tools, for example, electron microscopy, infrared nanospectroscopy, and X-ray microscopy/tomography, to validate and compare P-RSoXS results and form a comprehensive multimodal characterization framework for astromaterials. This research could lead to a new field of research by encouraging NASA's stated emphasis of multimodal synchrotron characterization for model, analog, and return samples, and could encourage NASA's eventual investment to support beamlines and/or endstations that can be dedicated for NASA research. As NASA prepares for and executes missions to the Moon, Mars, and beyond, utilizing the full workbench of characterization tools to address scientific questions will be imperative; P-RSoXS will be a great tool in this arsenal.

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