SCANNING TRANSMISSION X-RAY MICROSCOPY AS A TOOL FOR ANALYSIS OF INTERSTELLAR DUST CAPTURED IN AEROGEL. Anna Butterworth[1], Tolek Tyliszczak[2] Zack Gainsforth[1], Ryan Ogliore[1], Christopher Snead[3] and Andrew Westphal[1], [1]Space Sciences Laboratory, UC Berkeley, 7 Gauss Way, Berkeley, CA 94720-7450, [2]Advanced Light Source, Lawrence Berkeley National Laboratory, 1 Cyclotron Road, Berkeley, CA 94720. [3]Department of Earth and Space Sciences, UCLA, Los Angeles, CA 90095-1567.

Introduction: The Stardust mission returned to earth in January 2006 with two aerogel collectors. Cometary dust samples from the Jupiter-family comet Wild2 were studied during the first months after recovery in the so-called Cometary Preliminary Examination [e.g. 1,2]. Like the cometary collector, the interstellar (IS) collector contains 132 aerogel tiles and 240 foils The IS collector was exposed to the interstellar dust stream for 196 days. In comparison with the hundreds of Wild2 comet dust impact tracks containing thousands of particle fragments, we expect the interstellar dust to be orders of magnitude smaller and rarer.

Volunteers in the Stardust@Home public project have scanned more than half a million microscope images searching for rare features in the aerogel which may be impact tracks from dust particles [3]. The candidates identified so far will be carefully extracted from the aerogel using computer controlled micromanipulators and analyzed [4]. Some candidates might turn out to be impact tracks of interstellar origin particles.

These are the first contemporary samples of interstellar dust ever collected. Because we lack laboratory analogs, we cannot truly predict what they will look like in the simplest terms like mechanical strength and track morphology, degree of crystallinity, and elemental composition.

Stardust Interstellar Preliminary Examination (ISPE): The ISPE [5] will investigate candidates extracted from the aerogel collector using only non-invasive and non-destructive techniques. This means that all fragments of particles will remain in a small volume of aerogel, thin enough for optical and X-ray microscopy. Coined "picokeystones" for the picogram mass of the impactor, these tiny aerogel wedges are about 50 μm thick and contain an entire track, maybe <30 μm in diameter.

We will use the Scanning Transmission X-ray Microscope (STXM) at the Advanced Light Source (ALS) Beamline 11.0.2, at Lawrence Berkeley National Laboratory (LBNL) to analyze Stardust interstellar dust candidates. Here we describe the capabilities of the beamline for analyzing samples in aerogel, and demonstrate a method for quantifying radiation damage to susceptible organics in a sample. Radiation damage to interstellar organics is possible due to the high absorption coefficient of C,N,O by soft X-ray energies.

STXM Beamline Specifications: The STXM at ALS Beamline 11.0.2.2 has an elliptically polarized undulator soft X-ray source, providing an energy range from 200 to 2000 eV. The resolving power (E/ΔE) is 2500-7000 (ΔE = 80 meV at C K-edge). The scanning microscope focuses the synchrotron beam to a <40 nm spot using a Fresnel zone plate. The very high spatial resolution is well suited to searching for sub-micron dust fragments in aerogel tracks. The microscope may also be slightly defocused in order to efficiently map relatively large areas.

A photon-counting detector behind the sample records the intensity of the transmitted radiation, generating an image pixel by pixel during rastering. Elemental maps are produced by acquiring an image of the sample just below and just above the edge for each element.

Detecting very low concentrations of elements, while limiting photon dose to the sample usually requires that we use a strong resonance peak for mapping, giving an increase in sensitivity of up to 5 times over the edge jump. The height of the resonance peak depends on the chemical nature and crystallography of the target material. For example, all simple silicates for which we have so far obtained Mg K-edge XANES, have a strong absorption at 1314 eV, which is consistent with Mg-XANES of olivines measured and modeled by Wu et al. [6]

Radiation Damage of Epoxy Resin: Epoxy resin (EMBED-812) is easily damaged by low energy X-ray photons. Figure 1 shows C and O K-edge XANES spectra.. We made several overlapping maps for different elements on the epoxy as shown in figure 2. We used a beamline set-up exactly like analyses we have made on Stardust cometary aerogel.

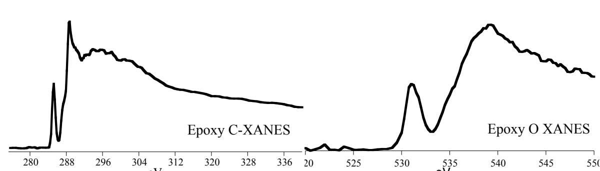


Figure 1: XANES spectra from the sample of epoxy used for testing. C XANES and corresponding O XANES.

After taking 6 element maps, we took a single image at 540 eV (σ* resonance) from which we could measure change in any damaged regions.

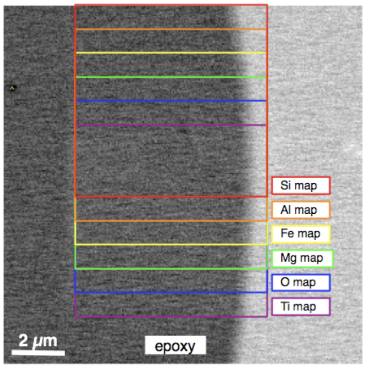


Figure 2: Testing radiation damage. Absorption image (540eV) of ultra-microtomed epoxy. The positions of 6 overlapping element maps (2 energies per map) are shown. Almost no change is visible.

Results: The change in oxygen bonding in the epoxy was derived from the 540 eV absorption image. The optical density ($OD = -ln(I/I_{zero})$) scales with density:

$$OD = \mu \rho t$$

where μ is mass absorption coefficient cm^2/g, ρ is density g/cm^3 and t is thickness, cm. The photon fluences of the maps are shown in Table 1.

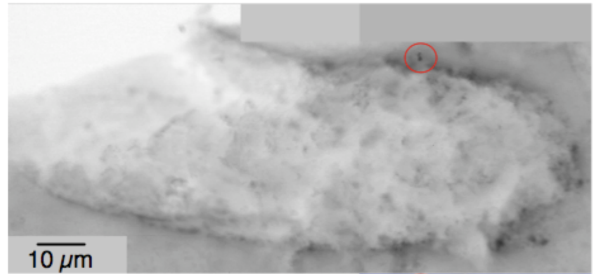
For cumulative Ti, Fe, Mg, Al and Si maps, OD_{540eV} fell less than 2%. The oxygen map resulted in a drop in OD_{540eV} of 5%, since epoxy is more susceptible to radiation at the O K-edge. The first pixel of each row has an extra 3 ms dwell time, the extra photon dose resulted in a 10% fall in OD_{540eV} for a vertical strip by the O map and 2% for the other 5 element maps.

Also shown in Table 1 are attenuation lengths of some elements with edges in the energy range for STXM. Major elements pose little problem for picokeystones, as demonstrated in figure 3.

Table 1: Photon fluence for mapping several elements at ALS 11.0.2.2 STXM

	Map Energies (eV)	Photon Fluence (x10^{14} cm^{-2})	Attenuation length aerogel (μm)	Attenuation length olivine (μm)
Ti K	450, 453	5.6	73	0.4
O K	525, 540	8.1	33	0.3
Fe-L	702, 708	9.4	64	0.4
Mg K	1304, 1314	13.1	324	1.1
Al K	1560, 1570	29.5	528	1.8
Si K	1825, 1845	19.1	245	2.0
C K	280, 288		26	0.2
N K	398, 406		54	0.4

Also shown are attenuation lengths into 20 mg/cm^3 SiO_2 aerogel and a ~3 g/cm^3 silicate. Picokeystones (~50 μm thick) are optically too thick for CNO.



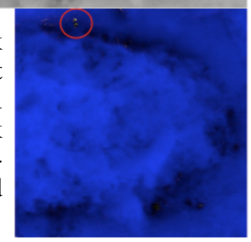


Figure 3: A small ODCE track in a pico-keystone, imaged at 1304 eV. Variation in aerogel density occurs at the track edges. Mg map (1304, 1314 eV), right, with one highlighted particle fragment

Mixing of the impactor with surrounding aerogel during capture may reduce the sensitivity of element maps. In this case, a XANES spectrum, say Mg K-edge, would discriminate between amorphous and crystalline material. The fluence would be higher in acquiring a XANES ‘stack’ depending on how many images are required (the energy resolution).

Several standards show that mineral identification is possible using Mg, or even Si XANES in aerogel. We have begun producing a library of standard minerals for Mg K, Al K, Si K, and Fe L edges. At present we can distinguish amorphous from crystalline material, and narrow down a sample to a mineral group. These abilities are consistent with the broad approach of ISPE, leaving detailed min/pet to post-ISPE research and full particle extraction.

Conclusions: We have presented a powerful technique for non-destructive analysis of picogram impactors in aerogel providing chemical and structural information. With the exception of Oxygen mapping, common elements may be mapped at very high spatial resolution, high sensitivity and with less than 2% measurable damage to epoxy resin, a readily available and easily damaged material.

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