INTEGRATING ANALYTICAL TECHNIQUES FOR ANALYSIS OF COMET 81P/WILD2 IN STARDUST TRACK C2052,2,74. Z. Gainsforth[1], A. L. Butterworth[1], L. Bonal[2], D. E. Brownlee[3], S. C. Fakra[4], G.R. Huss[2], D. Joswiak[3], M. Kunz[4], M.A. Marcus[4], K. Nagashima[2], R. C. Ogliore[1], N. Tamura[4], M. Telus[2], T. Tyliszczak[4], A. J. Westphal[1]. [1]Space Sciences Laboratory, UC Berkeley, USA, [2]Hawaii Institute of Geophysics and Planetology, University of Hawaii at Manoa, USA. [3]Astronomy Dept., University of Washington, USA, [4]Advanced Light Source, Lawrence Berkeley Laboratory, USA.

Introduction: NASA's Stardust mission brought back samples of Comet 81P/Wild2 in 2006 [1] necessitating the development of an array of new experimental and sample preparation techniques. This case study focuses on sample preparation and instrumental issues for a terminal particle in track C2052,2,74, named Iris. Our intention is to aid future researchers in designing intelligent experimental pathways for future Stardust samples while avoiding non-trivial pitfalls.

Sample description: The sample was captured in aerogel during a a 6.1 km/s relative velocity flyby of

Figure 1: Stardust track C2052,2,74 in optical light shown with a 1 mm scale bar.

comet Wild2 [1]. During this process, the cometary particle fragmented and experienced rapid deceleration and heating in the aerogel to produce a type B track (bulbous with terminal particles) [2].

We report scientific results regarding Iris in Butterworth, et al. [3] and focus on experimental aspects in this article.

Maximizing scientific output for a single sample: Our objective was to carry out coordinated analyses of a single terminal grain using complimentary instruments while minimizing instrumental interferences. We were able to successfully examine Iris using synchrotron Fourier Transform Infrared spectroscopy (FTIR), X-Ray Fluorescence (XRF), X-ray Absorption Near-Edge Structure spectroscopy (XANES), X-Ray Diffraction (XRD), Scanning Transmission X-ray Microscopy (STXM), STEM/EDS/EELS, HRTEM, and EFTEM in roughly that order. Future work involves analysis with Ionization Mass Spectrometry (IMS) for isotopic work and additional TEM and STXM work.

Instrumental interferences: Techniques utilizing photonic probes are least damaging. By contrast, depending on the sample's robustness, e-beam techniques tend to damage the sample via amorphization, carbon deposition, and loss of volatile elements. Additionally, sample preparation tends to restrict further work (e.g. ultramicrotomy). Ion beam isotopic measurements must come last as they are the most destructive to the sample.

Synchrotron investigation: Without a priori knowledge of the track, the first task is to obtain a non-destructive survey using synchrotron XRF, XRD, and XANES. We used beamlines 10.3.2 and 12.3.2 at the Advanced Light Source (ALS), Lawrence Berkeley National Laboratory [4,7]. With XRF and Fe K-edge XANES, we were able to identify the bulk chemical and crystalline state of the Fe in the track [5]. As a result, we identified the presence of olivine, sulfide, iron metal, and additional Fe^{2+} minerals. The primary advantage of XRF/XANES/XRD is that it can provide high resolution (<1 μm) maps of a track and limited phase identification in situ with no measurable sample damage.

Separation of a terminal particle: After identifying a

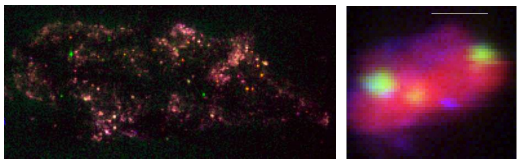

Figure 2: On the left is an FeCrNi RGB map of a portion of the bulb of track 74. Cr hotspots show up in green providing insight into the science to come. Image scale is 2.5 mm. On the right is an FeCrNi RGB map of the terminal particle named Iris. Cr hotspots show up in green and Ni hotspots in blue. Scale bar is 9 μm.

Cr-rich terminal particle we prepared it for detailed STXM/TEM work using a variation of the keystoning technique [6]. The aerogel was placed on a compliant surface (silicone covered with a 4 μm ultralene film). A second ultralene film in which a narrow slot had been made using a hot Nb wire was positioned over the aerogel with ~10 μm accuracy. This "surgical tent" was then pulled taut using monofilament tied to the corners of the ultralene. Previous work used glass spatulas to hold the aerogel in place, but we have learned that ultralene is much more gentle and leads to higher quality cuts with less damage to the aerogel. Ultralene is also cleaner than kapton and polypropylene films. The rest of the keystoning procedure is the same as in [6] using glass needles and micromanipulators for the cuts.

The result was a 100 μm thick slab of aerogel cut perpendicular to the track, containing only the terminal

particle and about 50 μm of track just above it. We oriented the grain so that microtomy would approach the particle from our direction of choice.

Back to the synchrotron: We returned to the synchrotron to obtain more detailed analyses of Iris using XRD/XRF at beamlines 10.3.2 and 12.3.2 at the ALS. XRD requires very strict control over the sample-to-camera distance which we obtained by sizing the aerogel slab only a few microns thicker than a 100 μm thick Si_3N_4 window frame. Thus, the sample could be pressed securely between two Si_3N_4 windows, and a diffraction standard glued to the window structure. This afforded us a sample to camera distance with an uncertainty on the order of the size of the sample itself.

XRD mapping confirmed the presence of olivine but also revealed that at least 10 other phases were present, whereas the XRF map showed only 3 distinct phases. XRD also allowed us to investigate the nanocrystalline and strain properties of the grain.

Embedding: We chose to use Embed-812 epoxy for ultramicrotomy. Brownlee et al., have pioneered the use of acrylic with the very significant advantage that it can be removed using chloroform [8]. However, acrylic is also more easily damaged by an analytical synchrotron x-ray beam. Because the particle was primarily olivine (index of refraction: n ~1.7), we were uncertain whether it would show sharp optical contrast after embedding in epoxy (n~1.5). Therefore, we chose an embedding medium that would allow us to measure the depth of the particle from the surface of the epoxy using the synchrotron. We then ultramicrotomed sections ~100 nm thick onto grids with an amorphous carbon (α-C) substrate.

STXM: STXM and TEM are in many ways synergistic techniques. TEM provides a powerful tool for analysis, but tends to be destructive and quantification is typically done with EDS which involves complex matrix effects that must be accounted for. TEM is also limited in the quality of its near edge spectra via EELS. STXM on the other hand uses x-ray transmission which rests on very simple single photon physics. One obtains very high count rates and low elemental detection limits on the nanoscale with less potential for sample damage. One also obtains very high quality near edge spectra ($\Delta E < 0.1$ eV) for elemental valence state and bonding analysis. Hence, a protocol involving TEM work benefits tremendously from STXM pre-analysis. For example, STEM/EDS analysis volatilized some Na in a Na-rich plagioclase before we had fully analyzed the mineral, and so it might have been difficult to quantify successfully. However, we were able to use our previous STXM data to obtain an excellent quantification.

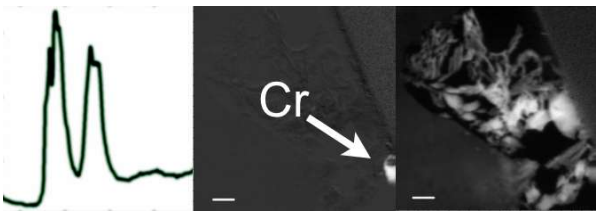

Figure 3: On the left is a Cr spectrum resolving both the L2 and L3 lines, as well as clearly resolved substructure related to the chemical environment of the Cr. Middle image is a STXM Cr map of Iris Grid B9. On the right is a STXM O-K. Intensities are quantitative and related to photoionization cross-section. 1 μm scale bar.

TEM: Three grids were examined using TEM at UW and UC Berkeley which provided complimentary information. TEM has spatial resolution far below STXM's 15 nm limit, higher image contrast, and micro/nano diffraction. These techniques proved very useful for nanophase analysis. TEM also allowed us to use phase contrast techniques for atomic imaging and sample tilt which can expose obscured features.

Conclusion: As a consequence of the tight integration between the above techniques, made possible through the development of sample preparation methods and an instrumental sequence unique to Iris, we were able to characterize striking similarities between Iris and Type IIA chondrules, measure fugacity, constrain heating profiles, and identify shock features all within the same particle.

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Acknowledgements: The operations of the Advanced Light Source and National Center for Electron Microscopy at Lawrence Berkeley National Laboratory are supported by the Director, Office of Science, Office of Basic Energy Sciences, U.S. Department of Energy under contract number DE-AC02- 05CH11231. We are indebted to Laura Westphal for naming Iris.