STUDY OF FINE-GRAINED MATERIAL RECOVERED FROM A STARDUST AEROGEL TRACK USING PLASMA ASHER PREPARATION.
B. A. Haas[1], R. C. Ogliore[1], A. J. Westphal[2], T. K. Croat[1] and C. Floss[1]. [1]Laboratory for Space Sciences and Physics Department, Washington University, St. Louis, MO 63130, USA (bahaas@wustl.edu), [2]Space Sciences Laboratory, University of California, Berkeley, CA 94720.

Introduction: NASA's Stardust mission returned material from comet 81P/Wild 2 to Earth in 2006. Ultra-low density silica aerogel tiles captured material from the coma of Wild 2 at 6.1 km/s. However, the aerogel's insulating properties prevent the use of electron microscopy without extensive extraction efforts, impeding traditional material characterization techniques [1]. The fine (< 2 μm) component of Wild 2 is particularly difficult to study in the aerogels, as these materials are typically spread throughout large, bulbous impact cavities [1], further complicating the already challenging extraction procedures [2]. As a result, the fine component of the comet has not been thoroughly examined.

We attempt to simplify the study of the Stardust aerogels, particularly the fine component of the collected material, through the use of plasma ashing which heretofore has not been fully explored. We utilize the plasma asher's ability to react with Si-based materials to separate the collected cometary materials from the surrounding aerogel, allowing for further study with TEM.

Equipment: The SPI Plasma Prep II Etcher/Asher contains a cylindrical F-resistant quartz sample chamber sealable to 133.3 Pa. The asher draws a carrier gas (CF_4) over the sample and RF power, provided by a crystal-controlled oscillator at 13.56 MHz, ionizes the gas under vacuum, creating a plasma of CF_3^+ and F^- ions. F^- ions combine with materials in the sample, forming an ash (e.g. SiF_4) that is removed by the vacuum pump. Analysis of the samples post-ashing was performed on a JEOL JEM-2000 FX TEM. EDS spectra were collected and analyzed with traditional Cliff Lorimer techniques.

Experimental Methods: Aerogel keystones are extracted from the Stardust sample collector using an automated keystone system [2]. Our analysis was performed on a section of the bulb of track 35 (C2054,44,35,0,0) with dimensions of 190 x 100 μm. The sample was mounted in a custom-built copper stage at the Space Sciences Laboratory in Berkeley. The copper stage, resistant to F plasma, contained a standard pinpointer TEM grid, the enclosed aerogel keystone on top of the grid, and a Si_3N_4 window to protect the keystone during initial pump down of the plasma asher. The stage was then exposed to the F plasma for 10 minutes at 34.3 kPa in Washington University's plasma asher (Fig. 1). The Si_3N_4 window is fully ashed after approximately 3 minutes, exposing the aerogel to the F plasma. The aerogel's porosity allows it to ash faster than the collected cometary material [3].

Freed cometary material was deposited directly onto the TEM grid during the ashing process. Cu TEM grids with continuous carbon meshes are largely unaffected by the ashing process, maximizing retention of the cometary material and eliminating the need for further difficult material transfer processes.

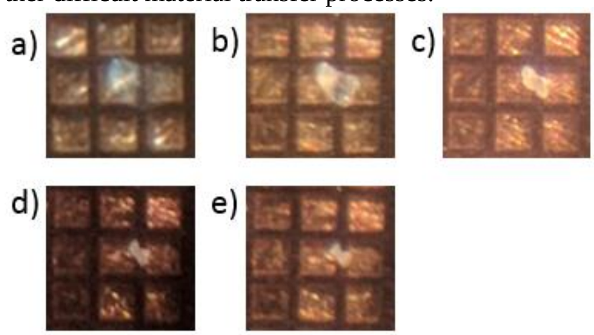

Figure 1: *Ashing process of the aerogel keystone as it rests upon the TEM grid. The size of the keystone a) pre-ashing and after b) 5 minutes c) 7 minutes d) 9 minutes and e) 10 minutes of exposure to the F plasma.*

Results: The aerogel was not fully ashed during the sample preparation process. Ashing rates decreased with continued exposure to the F plasma. The reduced ashing rates likely resulted from the aerogel's loss of porosity during ashing, as well as the gradual concentration of more F-resistant impurities known to be present within the Stardust aerogel [3, 4]. Ashing was halted once the aerogel's ashing rate was significantly slowed to prevent additional damage to the cometary material that had already been deposited.

Four large cometary particles were observed on the TEM grid in the same location that the aerogel was located during the ashing process (Fig. 2). Large grains ranged in size from 100 nm to 250 nm in diameter. Some of the large grains were also surrounded by tens of smaller grains only 1 to 5 nm in diameter.

The four large grains were largely composed of O/Si/Fe, with one grain containing significant Ca. Trace Ca was seen in the other 3 grains. Trace Mg/Cr/S/Al were also present in some of the EDS spectra. The lack of S alongside strong Fe signatures suggests that the Fe is a component of silicates rather than iron sulfides. Contamination in the form of F and Cl was also observed.

The larger grains appear to be aggregates composed of many smaller crystalline grains. Dark field images of the aggregates suggest the component grains range in size from 1 to 25 nm. Orientations of the component grains' crystal lattices appear to be random.

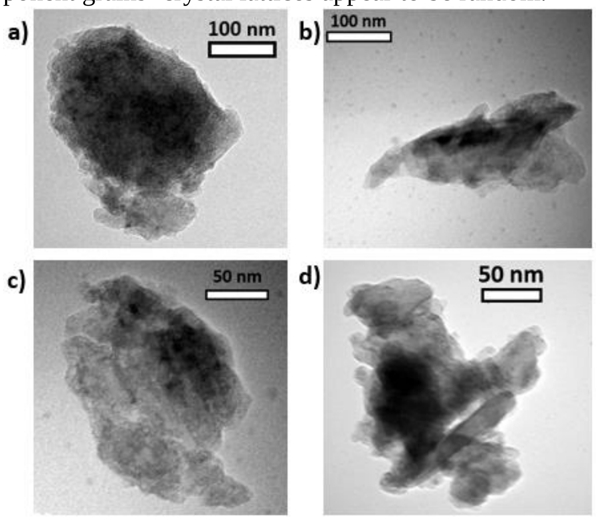

Figure 2: *Bright field TEM images taken at 200 KeV of grains deposited during the ashing process. Smaller grains only a few nm in size can be seen surrounding the grains in b) and c).*

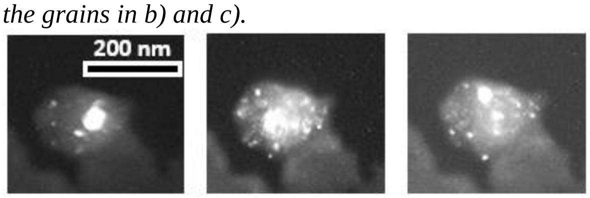

Figure 3: *Dark field TEM images taken at 200 keV at different sample tilt values for grain c) in Fig. 2. Variations in the image contrast with tilt angle suggests the grain is an aggregate composed of crystalline subgrains with different lattice orientations.*

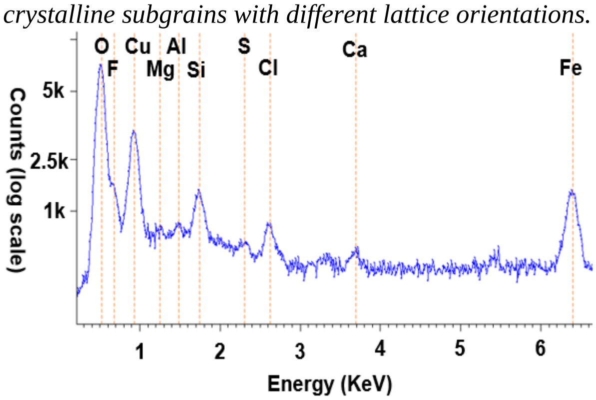

Figure 4: *Logarithmic EDS spectra of grain a) from Fig. 2. The Cu peak at 0.93 keV is a background effect.*

Discussion: The plasma ashing sample preparation appears to have been successful. We were able to extract fine grained cometary material from the aerogels and prepare them for further study with electron microscopy. Though the grains show some signs of contamination from the ashing process, they appear to have largely survived exposure to the F plasma as expected from our previous analog studies [5]. The behavior of the aerogel during the ashing process mirrored results previously seen using HF vapor etching [4]. Further investigations are required to determine to what extent extraterrestrial materials may be damaged during the ashing process. Probing the aerogels for grains prior to ashing is difficult, making estimates of how many grains may be lost during the ashing and transfer to the TEM grid unreliable.

Previous investigations of the coarse materials from Wild 2 have shown that they mostly formed far from the accretion location of the parent body [6], but the origins of the fine materials from the comet have not been well constrained. O isotopic compositions of the fines cover the full range of known Solar System materials, suggesting that the fines either sampled a variety of reservoirs in the inner Solar System or represent the Solar System's parent molecular cloud [7]. The grains we observed differed from both CI chondrites [8] and interplanetary dust particles [9] by lacking significant amounts of Mg. However, a much larger sample size is required to determine the nature and origins of the Wild 2 fines. As this sample preparation technique is easily applicable to the NanoSIMS, we hope to use this method to determine the isotopic compositions of these fine grains in addition to their mineralogies. Presolar grain abundances would help in determining the amount of processing experienced by the comet's fine component [7].

Further ashing studies will allow for sample preparation of fine grained particles from the aerogels, allowing for many more particles of these sizes to be isolated and studied by TEM and NanoSIMS. Complementing these studies with analyses of fine impactors in the Stardust foils [10] will allow for the characterization of the Wild 2 fine component.

References: [1] Burchell M. J. et al. (2008) *Meteoritics & Planet. Sci.*, 43, 23-40. [2] Zolensky M. et al. (2008) *Meteoritics & Planet. Sci.*, 43, 5-21. [3] Burchell M. J. et al. (2006) *Annu. Rev. Earth Planet. Sci.*, 34, 385-418. [4] Westphal A. J. et al. (2004) *LPSC XXXV*, Abstract #1860. [5] Haas B. A. et al. (2017) *LPSC XLVIII*, Abstract #2058. [6] Brownlee D. et al. (2012) *Meteoritics & Planet. Sci.*, 47, 453-470. [7] Ogliore R. C. et al. (2015) *Geochimica et Cosmochimica Acta*, 166, 74-91. [8] Lodders K. et al. (2010) *Principles and Perspectives in Cosmochemistry*, 379-417. [9] Zolensky M. et al. (2008) *Meteoritics & Planet. Sci.* 43, 261-272. [10] Kearsley A. T. et al. (2008) *Meteoritics & Planet. Sci.*, 43, 41-73.