

HF VAPOR ETCHING FOR STARDUST PARTICLE EXTRACTION: A PROGRESS REPORT.

K. A. McCain¹, T. M. Hahn¹, R. M. G. Armytage¹, W. P. Buckley¹, C. J. Snead², and A.N. Nguyen³. ¹Jacobs JETSII contract, NASA JSC, Mail Code XI3, Houston, TX 77058 (kaitlyn.a.mccain@nasa.gov), ²ARES, NASA JSC, Mail Code XI2, Houston, TX 77058, ³ARES, NASA JSC, Mail Code XI3, Houston, TX 77058.

Introduction: The Stardust mission captured thousands of particles from Jupiter-family comet 81P/Wild 2 using aluminum foil and silica aerogel collection media [1]. Most studies of Wild 2 material examine refractory terminal particles >1 μm in diameter which are extracted individually from the ends of tracks produced by the particle's impact into the aerogel medium. A substantial (>65 %) mass fraction of the impactors is present as fine-grained (i.e. <1 μm) material in the 'bulb' and walls of the tracks, which may contain preserved organic material and presolar grains [2]. Few presolar grains have been identified in Stardust samples, which has been interpreted as owing to destruction upon impact into the foils and preferential sampling of terminal particles rather than a lack of presolar grains in Wild 2 samples [3-5]. By isolating the fine-grained material from these bulbs and performing microbeam and isotopic analyses (i.e., scanning electron microscopy and NanoSIMS), organic matter and presolar grains can be identified and constraints can be placed on the constituents of primitive outer solar system materials.

Prior efforts to extract and concentrate fine-grained material from the bulb of Stardust aerogel tracks have crushed or destroyed silica aerogel using HF vapor etching or plasma ashing techniques to free the fine-grained material, which is intimately mixed with the aerogel [6-8]. During our previous development of an HF vapor etching procedure, we constructed an apparatus from readily-available materials with the goal of slowly etching silica aerogel to free the fine-grained size fraction while minimizing alteration of the captured particles [6]. Secondary electron imaging of analog mineral grains exposed to various concentrations of HF vapor in this setup revealed Mg-fluoride 'bubbles' or coatings on Mg-silicates (e.g. olivine and enstatite) while other minerals remained intact. The extent of Mg-fluoride formation depended upon the etching rate and production of liquid water droplets around the minerals. Lower concentrations of HF resulted in lower etch rates and water droplet production and, therefore, less Mg-fluoride [6].

In this work, we present two further iterations upon this setup aimed to further decrease Mg-fluoride formation by discouraging water droplet production: (1) Carrying out the etching procedure at higher temperatures to keep water in the vapor phase and (2) physically separating HF vapor from the injected solution using a separate reservoir chamber.

Methods: Minerals ranging from <1 to 30 μm in size with known chemical and O isotopic compositions were pressed into HF-cleaned gold foils and mounted on an HF-compatible graphite stub alongside a fragment of silica aerogel to serve as analogues for the effect of the etching procedure on captured cometary minerals [6]. In the first test, the stub will be placed into the HF-compatible etching chamber held at 50 °C using a hot plate inside of a fully-exhausting fume hood. 1 mL of dilute (0.24 N or 0.5%) HF will be injected into the chamber using an HF-compatible syringe, and the etching reaction progress will be monitored using a Dino-Lite Edge digital microscope. Once the aerogel is fully consumed, the lid of the etching chamber will be removed inside of the fume hood to stop the reaction.

In the second test, a similarly prepared stub will be placed in an HF-compatible etching chamber connected to a reservoir chamber by HF-compatible tubing and feedthroughs fitted with a Parker PTFE needle valve. After injecting HF solution into the reservoir, the vapor will be allowed to equilibrate between the two chambers before closing the needle valve. This will limit the interaction of water vapor from the HF solution with the aerogel and minerals. As described above, the etching procedure will be monitored and the reaction stopped once the aerogel is fully consumed.

After etching is complete, the stubs containing the analogue minerals will be coated with 15 nm of Au and imaged with the JEOL 7900F SEM at NASA JSC. The extent of Mg-fluoride production produced by each setup will be evaluated and the procedure and setup adjusted as needed.

Discussion: The results of the revised procedures will be presented at the meeting. Once the etching procedure has proven capable of destroying silica aerogel while having minimal impact upon ~1 μm Mg-silicate analogues, we will apply the procedure to fragments of bulbs and a complete test keystone produced from Allende particles fired into aerogel. Any potential effects of the etching procedure on the O isotopic compositions will be evaluated by measuring the O isotopic compositions of the etched analogue minerals and Allende residues using the NanoSIMS at NASA JSC. After these tests are complete and results are satisfactory, we will proceed with etching Stardust bulb fragments.

Acknowledgements: This work was supported by the NASA LARS Program grant NNH20ZDA001N to A. N. Nguyen. We also thank Timmons Erickson (Jacobs JETSII contract, NASA JSC) for assistance with the SEM, and Anna Butterworth (Space Sciences Lab, University of California at Berkeley) for HF etching background information.

References: [1] Brownlee, D. et al. (2006) *Science*, 314, 1711-1716. [2] Flynn, G. J. et al. (2006) *Science*, 314, 1731-1735. [3] Messenger, S. et al. (2009) *LPSC XL*, Abstract #1790. [4] Leitner, J. et al. (2010) *LPSC XLI*, Abstract #1607 [5] Floss, C. et al. (2013) *Astrophysical Journal*, 763, 140. [6] McCain et al. (2023) *LPSC LIV*, Abstract #2515 [7] Ogliore, R. C. et al. (2015) *Geochimica et Cosmochimica Acta*, 166, 74-91. [8] Westphal, A. J. et al. (2004) *LPSC XXXV*, Abstract #1860. [9] Haas, B.A. et al. (2018) *LPSC XLIX*, Abstract #2245.