APPLICATION OF PLASMA ASHING TO THE STUDY OF STARDUST MISSION AEROGEL SAMPLES. B. A. Haas[1], R. C. Ogliore[1], and C. Floss[1], [1]Laboratory for Space Sciences and Physics Department, Washington University, St. Louis, MO 63130, USA (bahaas@wustl.edu).

Introduction: NASA's Stardust mission returned the first unambiguous cometary material to Earth in 2006, but study of the returned material from comet 81P/Wild 2 remains challenging. Silica-based low-density aerogel tiles successfully collected cometary material despite collection velocities of 6.1 km/s. However, the aerogel's insulating properties prevent the use of traditional electron microscopy techniques without extensive extraction efforts, making study of the cometary material difficult [1].

Coarse (> 2 µm) cometary fragments have been found to survive at the ends of aerogel tracks, allowing for extraction and further study, but finer grained impactors are spread throughout large, bulbous impact cavities [1]. Separating the collected cometary material from the aerogel would greatly simplify the study of the fine-grained component of comet Wild 2.

While plasma ashers are well-known for their use in the manufacture of semiconductors, their application towards the study of Stardust samples has not heretofore been fully explored. By utilizing the plasma asher's ability to react with Si-based and organic materials, separation of the collected cometary material from the ubiquitous aerogel may prove possible, aiding in the study of these challenging samples. We outline here a process by which the Stardust aerogel samples may be prepared with plasma ashing for ultimate examination with electron microscopy.

Equipment: The SPI Plasma Prep II Etcher/Asher contains a cylindrical Pyrex sample chamber with a diameter of 10.5 cm and a length of 15.0 cm sealable to 133.3 Pa. The asher draws a carrier gas (e.g. O_2 or 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. The ions combine with materials in the sample, forming an ash that is removed by the vacuum pump.

Carrier gases are chosen based upon the materials to be ashed. CF_4 ionizes into CF_3 and F, resulting in a F plasma that interacts with silica-based materials such as the Stardust aerogel. O_2 results in O plasma that interacts with organic materials.

Planned Methods: Full impactor tracks are extracted from Stardust's aerogel tiles using an automated keystone system, resulting in individual keystones ranging from tens of microns to millimeters in length [2]. These keystones are placed on Au-plated mounts that are resistant to the ashing effect of both F and O plasma and provide a flat, conductive surface.

The low density of the aerogel makes these keystones difficult to work with, as even slight changes in pressure can cause free keystones to be lost. To protect the individual keystones during transportation to the asher as well as during the initial phases of the ashing process, Norcada Si_3N_4 windows are used. These windows cap a Si base and range from 10 nm to 1.0 µm in thickness. The lengths and widths of the windows defined by the Si base range from .01 by .01 mm to 2.7 by 2.7 mm, allowing for the study of a wide range of keystone sizes.

Typical Si_3N_4 ashing rates by F plasma at the standard operating pressure of 34.4 kPa are 1.7-2.0 µm/hr, allowing the F plasma to penetrate the window shortly after stable pressures are reached within the asher [3]. This prevents the loss of the delicate aerogel keystones while still allowing F ions to access the sample.

Ashing rates for SiO_2, the composition of the Stardust aerogel, are 2.0-2.4 µm/hr at standard operating pressure. However, the Stardust aerogels are extremely porous, with over 98% of the volume composed of ambient gases [4]. The porous nature of the aerogels greatly increases their effective surface area and significantly speeds up the ashing process.

C contamination, though limited in the Stardust aerogels, is known to compose 0.1-0.5% of the aerogel by weight [4]. O plasma can be utilized following the F plasma to remove organic contamination from the surviving cometary debris.

Following the plasma process the deposited material will be pressed into the Au plating, presenting an ideal surface for further study with electron microscopy.

Discussion: Traditional plasma ashers use sample chambers made from borosilicate glass such as Pyrex. These chambers are mildly reactive with F plasma due to their high Si contents, resulting in the creation of the gaseous molecule SiF_4. However, SiF_4 is also a product of F reactions with aerogel, making the full elimination of this contaminant difficult. We ran samples of Brenham and San Carlos olivines through hour-long runs in the Pyrex sample chamber with F plasma and found that F composes <1% of the atomic compositions of these samples after the run, suggesting contamination from the sample chamber is minimal and that the vacuum pump is effective at removing gaseous contaminants. Potential SiF_4 contamination can be mitigated through use of a quartz sample chamber resistant to fluorination if continued degredation of the sample chamber appears problematic. Potential contaminants from the aerogel also appear unlikey to bias results. Outside of C, few contaminants are present in concentrations larger than a few parts per million [4].

Of greater concern is the loss of cometary silicates from overexposure to the F plasma. HF etching has previously been used to remove silicates from meteoritic material and IDPs to focus on Fe-Ni sulfides and organic materials [5,6]. The porosity and high Si content of aerogel both increase the rate at which it ashes due to the high surface area and total Si exposure to the F plasma, resulting in aerogel samples ashing quicker than denser silicates. Tests on similar silica-based aerogels ~1 mm^3 in volume resulted in complete ashing within one hour of exposure to F plasma at 17.2 kPa. Similar F plasma exposures on San Carlos and Brenham olivine granules resulted in damage to the materials on the scale of tens of microns, with larger grains surviving the ashing process (Fig. 1). The rapid ashing rate of the Stardust aerogel relative to other silicates should allow for its complete removal with minimal damage to collected cometary silicates. However, given the small sizes of the aerogel keystones and collected cometary materials, limited F plasma exposure is required. F plasma pressures and exposure times will need to be carefully adjusted to ensure minimal damage to collected silicates while still maintaining penetration of the protective Si_3N_4 window and complete destruction of the surrounding aerogel. Further testing to determine optimal F plasma pressures and exposure times is underway.

The use of O plasma for the destruction of organic materials presents its own problems. Our tests of O plasma at 34.4 kPa on 98.0% pure graphite grains found most graphite grains <5.0 µm in diameter were fully ashed within one hour of exposure time. These results suggest that the use of O plasma will destroy any surviving organic materials collected from comet Wild 2. However, prior investigations of organic compounds found many to be extensively thermally altered during the collection process, making investigation of these materials inherently difficult [7]. Successful investigation of organic materials in the fine-grained component captured by the aerogel therefore appears unlikely unless the O plasma processing step is eliminated. In contrast, O plasma processing appears to be beneficial for the study of silicates in the fine-grained component of Wild 2. We exposed San Carlos and Brenham olivine granules to O plasma at 34.3 kPa for one hour and found no observable damage to grains as small as 5 µm. Thus, studies focused upon characterizing silicates from the Stardust aerogel would benefit from O plasma processing by removing other contaminants without affecting these materials.

Successful sample preparation with the ashing process hinges upon precise determination of the minimum F plasma exposure times. Previous investigations of the aerogel have made use of analog samples made from Stardust-type aerogel cells [8]. Use of these analog aerogels would allow for test runs to be conducted and minimum exposure times, as well as silicate-material survival rates, to be calculated.

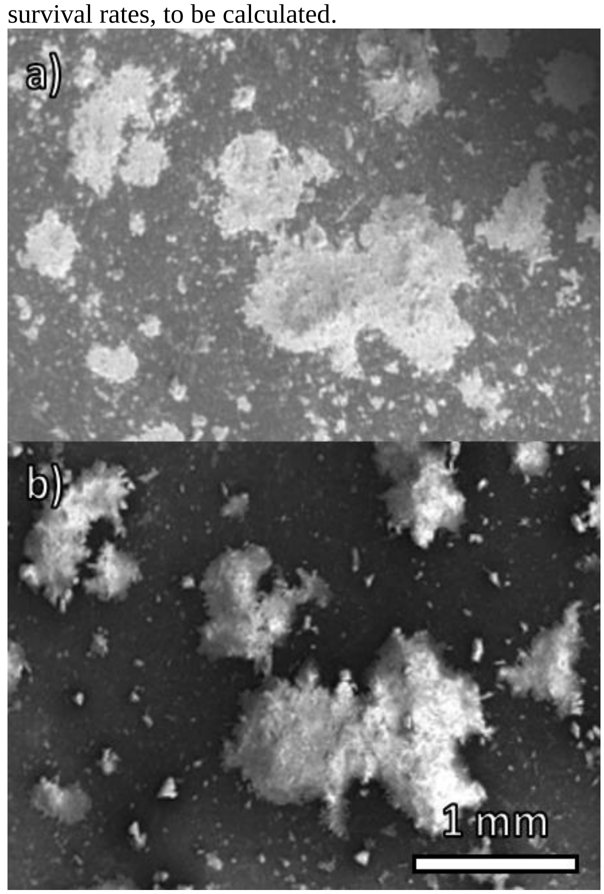

Figure 1: *SEM image of San Carlos olivine granules a) before and b) after exposure to F plasma at 17.2 kPa for one hour. Damage to the olivine samples suggests lower plasma pressures and run times will be required with aerogel samples.*

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] SPI Supplies (1996) Plasma Prep II Maintenance & Instruction Manual. [4] Burchell M. J. et al. (2006) *Annu. Rev. Earth Planet. Sci., 34*, 385-418. [5] Brownlee et al. (2000) *LPSC XXXI*, Abstract #1921. [6] Matrajt G. et al. (2004) *Astro. & Astrophys. 433*, 979-995. [7] Cody G. D. et al. (2008) *Meteoritics & Planet. Sci., 43*, 353-365. [8] Hoppe P. et al. (2006) *LPSC XXXVII*, Abstract #1546.