Exploring the potential of Xe$^+$ Plasma FIB for minimum mass-loss sectioning of meteoritic analogs of coarse-grained (0.2-1 mm) asteroidal samples from the JAXA Hayabusa 2 sample return mission to asteroid Ryugu.

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Introduction: The JAXA Hayabusa 2 sample return mission will collect samples of Ryugu, a spectral type Cg asteroid, in autumn 2018 and return them to Earth in December 2020. It is expected that a significant component of the material collected will be regolith particles that are ~0.2-1 mm in diameter. These comparatively large carbonaceous chondrite fragments will be the focus of research of the Coarse-grained Sample Analysis Team. Samples in this size range present significant challenges for sample preparation. It is highly desirable that the samples can be cut into two or more subsamples for analysis using different techniques, while leaving material that can be archived for future study. Typical cutting techniques such as wafering saws using wires or blades are highly wasteful in terms of mass. In preparation for these challenges, we are exploring the application of Xe$^+$ plasma FIB (P-FIB) techniques as a potential methodology for sectioning coarse-grained Hayabusa 2 particles, with minimum mass loss.

Background: Focused ion beam techniques have become the method of choice for the site-specific sample preparation of a wide range of synthetic and natural materials. Ga$^+$ FIB has become an established tool for the preparation of small volume samples for cross sectional imaging and TEM analysis [1]. However, the relatively low beam currents which can be obtained from Ga$^+$ ion sources limit their ability to section materials that are more than a few 10s of microns in size. The more recent development of P-FIB technology utilizing Xe$^+$ ions, delivers much higher beam currents and enhanced sputtering rates (10-30% higher and up to 300% for some materials [2]), has revolutionized the field of large area cross-sectional analysis, as well as enabling the extraction of large volumes of material for ex-situ analysis.

Methodology: A Thermo Fisher Dualbeam® Xe$^+$ P-FIB at the University of Manchester was used as a proof of concept instrument for our preliminary studies. A sample of the Murray CM2 chondrite was used as an analog material with properties that are likely to be a good match to samples that will be returned by the mission. Murray is a complex, heterogeneous meteorite with materials of diverse densities and grain sizes that sputter at different rates and represent a significant challenge. A chip of Murray, ~1x2 mm in size, was secured to an aluminium SEM stub using Superglue and was examined uncoated in the P-FIB.

Results: The first order sputtering behavior of Murray was determined by carrying out a cleaning cross section on a relatively flat surface of the sample. At instrumental conditions of 30 kV, 0.47 µA, a 50 x 50 µm^2 cleaning cross section depth of 30 µm (the calculated cutting depth for Si) sputtered to a measured depth of ~56 µm in 2 minutes. Sample sputtering was quite homogeneous across the whole area, except for an inclusion with much lower sputtering rates that was removed after a second cleaning cross section. A satisfactory surface was produced in approximately 3 minutes. A second experiment was performed to determine the viability of cutting larger regions of sample. A regular cross section was set up on the edge of the fragment with dimensions 316 µm (L), 66 µm (W) and 120 µm (D). Using a beam current of 1.3 µA, the pattern cut through to a depth of ~192 µm in 30 minutes (Fig. 1A). A sputter-resistant particle remained in the lower part of the surface that required three additional cleaning cross sections to reduce in size. These experiments show that sputtering is remarkably fast over large areas, even in regular cross section patterning mode, where the tip of the ion beam is removing material.

In order to section a single, coarse-grained particle, a complete cut across the entire diameter of the particle that maximizes the depth to width ratio of the cut is required. Due to increased re-deposition as the ion beam penetrates deeper into the sample, the sputtering rate will decrease with depth and the width of the cut will also become smaller. We explored two different approaches to cutting the sample to maximize the depth to width ratio of the trench. In the first case, a rectangular pattern 50 µm long, 5 µm wide and 30 µm deep was run at a beam current of 470 nA (40 seconds duration) on a relatively flat region of the sample surface. A regular cross section cut normal to the first rectangular pattern was then run to look at the geometry of the trench, which was carrot-shaped with a poor depth to width ratio of 2:1 (Fig. 1B, trench 1). A slight improvement in the depth to width ratio was obtained if the trench was open at one end at the start of the pattern (Fig. 1B, trench 2). This result demonstrates that sputtering using a standard rectangular pattern is inefficient and not a viable approach for sectioning this kind of chondritic material. An alternative approach is to take advantage of the higher sputtering rates when the ion beam interacts with the sample at glancing angle and sputtering occurs at the edge of the beam. For this experiment, we utilized a cleaning cross section pattern, but with dimensions that are essentially the reverse of those used typically for polishing a sample surface, i.e. the face that the beam is cutting is very narrow, but the pattern is highly elongate. A pattern with a width of 5 µm (on the cutting face), a depth of 50 µm and a length of 50 µm resulted in a trench in the sample with a depth to surface width ratio of ~9 (90 µm deep) (Fig. 1B, trench 3) in 40 seconds.

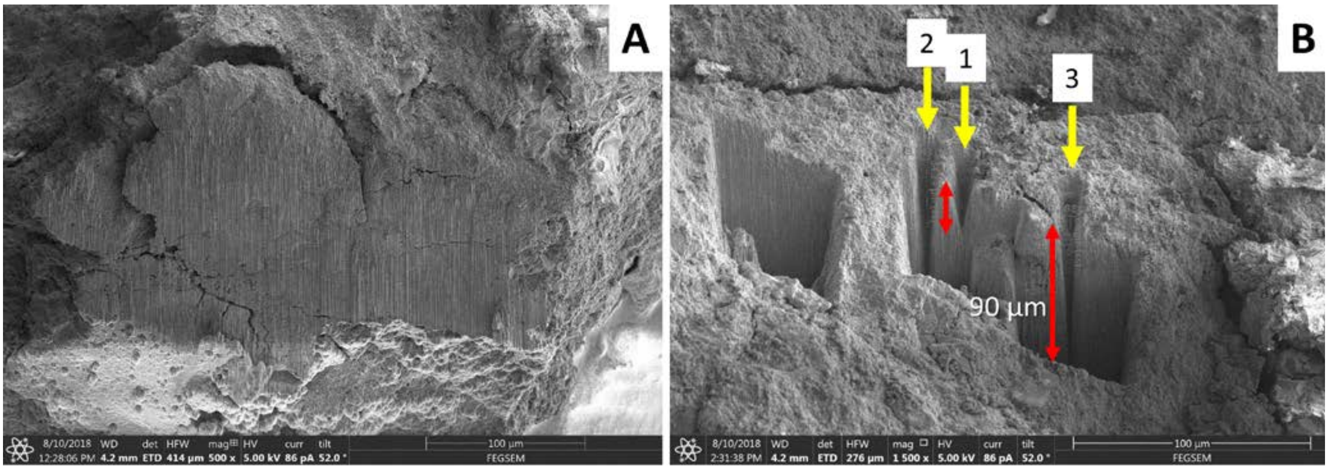

Figure 1 - A) SE image of Xe P-FIB cross section cut on the surface of a fragment of the Murray CM chondrite. The face is over 300 µm in width. B) SE image of trenches cut into Murray sample using different types of patterns. Trench 1 was cut using a rectangular pattern with closed ends that produced a carrot-shaped cross section. Trench 2 was cut using the same pattern, but the trench was open at one end resulting in a deeper trench. Trench 3 was cut using a narrow cleaning cross section, producing a deeper trench with improved depth to width ratio.

Conclusions: Although we have yet to cut an entire particle, these experiments demonstrate that P-FIB has great potential for sectioning Hayabusa 2 particles. Preliminary results show that it should be possible to section 500 µm diameter particles in time periods of ~1-2 hours. However, the heterogeneity of the sample in terms of sputtering rates does require that cutting through more resistant phases must be factored into the milling time. It may be advantageous sectioning the sample in two separate cuts run from opposite sides of the particle. Based on the depth to width ratio (9:1) obtained from the sample of Murray cut using a cleaning cross section, we estimate that the width of the top of the cut would be around 50 µm. Cutting particles larger than 500 µm in diameter in half would be more challenging and time consuming. A better strategy may be to remove serial slices of lower diameter. We are continuing to develop this method, including assessment of the extent of beam damage to the sample, adjacent to the cut.

References: [1] L.A. Giannuzzi, and F.A. Stevie (1999) Micron, 30, 197-204. [2] Burnett, T.L. et al. (2016) Ultramicroscopy 161, 119-129.