ARGON ION SLICING (_ArIS_) OF MINERAL AND ROCK SAMPLES: A NOVEL TOOL TO PREPARE LARGE ELECTRON TRANSPARENT THIN FILMS FOR TEM USE.

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Introduction: *Argon Ion milling* is the basic conventional means used since decades when speaking of sample preparation for *TEM* analysis. Applied to earth and planetary samples (usually on hand as thin sections) the shortcomings are (a) nearly impossible site specific thinning and (b) only small electron transparent areas obtained. A new technique named *FIB* (*Focused Ion Beam*) developed over the past years overcame several of these draw backs [1]. However, *FIB TEM* sectioning remains time consuming and expensive. Furthermore the electron transparent area obtained is in most cases restricted to about 20µm x 5µm with a typical thickness of less than 100 nm and a major part of the area sampled is not seen in advance since it is located perpendicular to the sample surface. A novel alternative approach to site specific preparation for earth and planetary samples is presented here using an JEOL EM-09100IS Ion Slicer. *Argon Ion slicing* (*ArIS*) is based on a shadowing technique [2] and leads to high quality, homogenous, damage free, large electron transparent areas of up to 100 x 200 μm^2 (20,000 μm^2) even for complex polyphase samples.

Sample pre-treatment: Although any conventional thin section is suitable the use of removable glues (e.g. crystal bond or comparable) is recommended. The area of interest can be identified by light or electron microscope. The best results will be obtained using an ultrasonic drill to cut out a rectangular area (2mm x 3mm). Applying this technique the margins produced are straight and intact, which is a necessary precondition for later successful *ArIS*.

After cutting the rectangle out of the specimen a half circular copper grid is glued onto its surface. In most cases subsequent acetone treatment is sufficient to separate the sample from the glass part of the microscope slide. Samples initially not intended for *TEM* analysis may require longer acetone in some cases even chloroforme treatment to remove the glue.

Optimised thinning: The sample rectangle with its attached copper grid is mounted into the Ion Slicer sample holder (JEOL Ion Slicer) which had been slightly modified to avoid further use of any glue.

The sharp (typically 30 to 100 µm thick) edge of the sample is partially shaded by the sharp edge of a copper mask belt (10 µm) mounted above the sample. The Ion beam lies in the same plane as the sharp edges of sample and belt do. In this respect the tilt angle determines the final size of the thinned area obtained. The beam can be tilted in various angles from zero (parallel) to 6°. The slicing process alternates between front and backside whereas side change intervals are variable as are accelerating voltage and slicing time. Streaks, artefacts produced during slicing on the sample surface are minimized by a “swing-mechanism” rocking the sample stage slightly in the plane of the ion beam.

With a tilt angle of 1.5° the largest electron transparent areas were obtained. According to conventional *Argon Ion milling* an acceleration voltage 4.5 kV with an Argon flow rate of 7.8 was chosen to balance a fast slicing rate and minimum sample damage (amorphisation, phase changes, etc.). Applying theses conditions a large “lagoon shaped” area is obtained (Fig. 1). The thinned region in total covers an area more than 500 x 500 μm^2 (250,000 μm^2) with a thickness of less than 1 µm. Like in conventionally thinned samples a small hole is generated in the thinnest region of the sample. *ArIS* produces very large electron transparent areas surrounding the small hole.

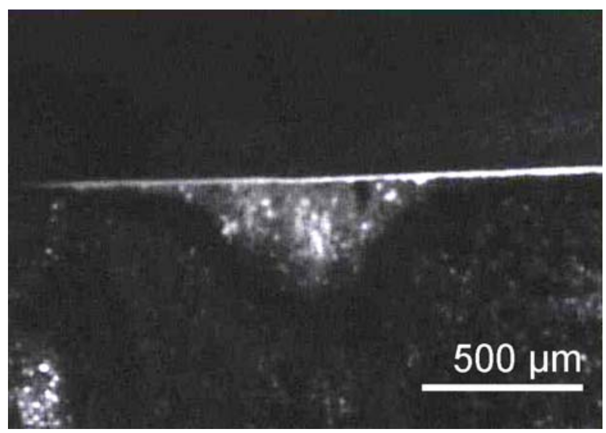

Figure 1. Typical thin (less than 1µm) “lagoon shaped” area (Carboneceous Microbiolith) produced by ArIS-technique.

Application to Earth and planetary materials: *ArIS* was applied on different geological samples, namely carbonaceous chondrites, a martian nakhlite, carbonaceous microbioliths and eclogites. TEM samples were produced according to the description in sample pre-treatment and optimised thinning.

In most cases non coherent electron transparent areas of approximately 1,000 to 5,000 µm^2 were achieved (Fig. 2) in routine runs. Compared to 100 µm^2 gained by a typical FIB section the advantage becomes apparent.

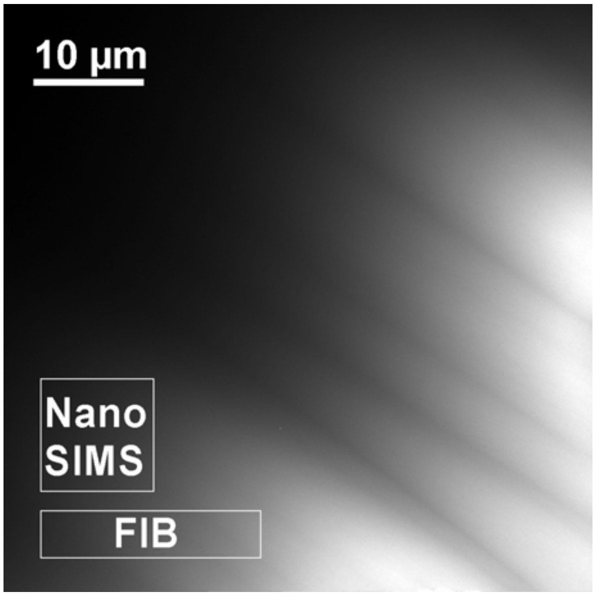

Figure 2. TEM bright field micrograph of an homogeneous electron transparent area of a pure SiO-glass test sample. For comparison the sizes of a typical FIB-section and a standard 10x10µm^2 NanoSIMS mapping area are shown.

Future perspectives of the *ArIS* technique: *ArIS* represents an alternative preparation technique to site specific approaches where large electron transparent areas are required. The electron transparent area obtained by conventional methods can be exceeded by orders of magnitude applying the *ArIS* preparation method. This will allow the detailed and statistically significant study of features in polyphase mineral aggregates like fine grained matrix or fine grained rims of pristine meteorites, a comprehensive study of alteration products in meteorites or planetary samples, etc.

The thin electron transparent areas are still surrounded by thick sample parts which enhance the stability of the sample mount and therefore enable the study of the same sample material by different analytical techniques like NanoSIMS, STXM, HR-SXRF [3, 4] and others.

References: [1] Wirth, R. et al. (2004) *EJM*, *16*, 863–876. [2] Yasuhara, A. (2005) *JEOL News, Electron Opt. Instrum. 40*, 46-48. [3] Wirth, R. et al. (2004) *EJM*, *16*, 863–876. [4] Vincze, L. et al. (2004) *Analytical Chemistry 76*, 6786-6791. [5] Vekemans, B. et al. (2004) *JAAS 19*, 1302-1308.