A PREPARATION SEQUENCE FOR MULTI-ANALYSIS OF MICROMETER-SIZED EXTRATERRESTRIAL SAMPLES. A. Aléon-Toppani[1], R. Brunetto[1], J. Aléon[2], Z. Dionnet[3, 4], S. Rubino[1], D. Levy[4], D. Troadec[5], F. Borondics[6], and F. Brisset[7], [1]Université Paris-Saclay, CNRS, Institut d'Astrophysique Spatiale, 91405, Orsay, France (alice.aleon@ias.u-psud.fr), [2]IMPMC, MNHN, Sorbonne Univ., CNRS, Paris, France, [3]INAF-IAPS, Roma, Italy, [4]DIST-Università Parthenope, Napoli, Italy, [5]Institut d'Electronique, de Microélectronique et de Nanotechnologie, Lille, France, [6]SOLEIL synchrotron, Gif-sur-Yvette, France, [7]ICMMO, CNRS UMR 8182, Univ. Paris-Saclay, Orsay, France

Introduction: Since the past few decades, analyses of microscopic samples have become increasingly important to extract a wealth of scientific information from rare and precious samples. Samples, such as those that will be returned by Hayabusa 2 mission, are expected to be very heterogeneous (close to CM mineralogy) and require analyses with nanometer resolution techniques. In such conditions, it is necessary to perform multiple complementary analyses on micrometer-sized samples, beginning from the least destructive to the most destructive methods to maximize scientific outcome and minimize sample loss and contamination.

Here we present a new sample preparation sequence. Infrared tomography (IR-CT) is performed as a first step for a non-destructive characterization of the 3D structure and chemistry of the grain [1]. This notably can allow identification of sub regions of interest which can be targeted for further analyses. Subsequently, we have followed two different approaches 1) preparation of sequential focused ion beam (FIB) sections of single 20 µm grains, for different 2D analytical methods (e.g. IR spectroscopy, transmission electron microscopy,TEM or NanoSIMS) and 2) development of a new FIB section mounting protocol that allows a combination of different analytical methods on the same FIB section starting with NanoSIMS, followed by IR and/or TEM. Both approaches can be combined to maximize scientific output from a single grain.

Material and methods: Sample preparation was performed using a FEI Strata DB 235 scanning electron microscope (SEM) at IEMN (Lille). An Agilent Cary 670 IR microscope equipped with a Focal Plane Array Detector (FPA) and a globar source was used for the IR characterization of the sample (SOLEIL synchrotron, Gif-sur-Yvette). Isotopic imaging was performed using the NanoSIMS 50 at IMPMC (Paris). Mineralogical characterization of samples was performed using a 200 kV 2100+ JEOL TEM (Univ. Paris-Saclay).

IR-CT of single microscopic grain: Grains deposited on a conductive substrate are welded on a tungsten needle using either platinum (Pt) or electronic glue. Both glue and Pt are spatially restricted to the welding zone (fig. 1). Signature of contamination is very weak and different from that of meteoritical carbon. Fig. 1 shows an example of IR-CT of a grain from the NWA 5515 CK chondrite. Once a grain is mounted for one 3D method, other types of 3D characterization can be performed on the same grain (e.g. X-CT [2]).

Sequential sectioning of individual grains for TEM, 2D-IR mapping and NanoSIMS analyses: After analyses of a grain by IR-CT, the grain is recovered and sliced into several sections for further 2D mineralogical, chemical and isotopic analyses (figure 1). Using the Ga beam of the FIB focalized at the contact between the grain and the tungsten needle, the grain is removed from the needle and retrieved on a conductive substrate. Small and very heterogeneous grains are completely covered by platinum in order to be protected from irradiation damages and to improve cohesion. The Ga beam is subsequently used to cut the grain into three slices. Then, one after each other, the slices are connected to the W needle using Pt and separated from the substrate using the Ga beam. Dedicated sections for IR and TEM analyses are welded to a TEM grid using Pt-deposition. Sections for TEM analysis are finally thinned in situ down to ~100 nm for electron beam transparency and sections for IR mapping are thinned down to 1 micron.

In this process, we are able to obtain 3 slices of about 3 microns thickness from a ~20 µm grain.

Nanosims analyses of FIB section and subsequent TEM and IR analyses. NanoSIMS analyses are usually performed after all other analyses (e.g. TEM) because they are destructive at the 100 nm scale [3]. Here, we developed a new sample mounting procedure to perform NanoSIMS imaging of FIB sections before synchrotron-based microspectroscopies and/or TEM of the same sample (fig. 2). This approach has several requirements: (1) the section of interest must be thicker than the depth of NanoSIMS analysis, (2) the section must be mounted on a holder suitable for subsequent analyses or must be recovered after SIMS analysis, (3) instrumental effects due to sample preparation must be minimal for NanoSIMS analysis and (4) the atomic layers damaged by the ion beam must be removed before subsequent mineralogical analyzes. To solve these

issues, 2 to 3 µm thick FIB sections are deposited on flat conductive NanoSIMS sample mounts, here polished Al-disks. In order to recover the FIB sections from Al-disks, they are mounted on 1-2 µm bridges made of Pt deposited onto the Al-disk in the FIB-SEM (Fig. 2).

In order to evaluate the instrumental effects associated with FIB section mounting and preparation, two FIB sections of the Bamble standard amphibole were deposited directly on top of the polished, Au-coated, crystal from which they were extracted. The first FIB section was directly deposited on the surface of the amphibole and the second FIB section was deposited on Pt bridges. H isotopes were measured in both FIB sections and in the polished section and yielded D/H ratios identical within error, indicating that the preparation of the FIB section and mounting on Pt bridges did not introduce measurable isotopic effects.

Using Pt bridges, FIB sections can be recovered after NanoSIMS analysis and undergo minimal thinning for FTIR imaging (fig. 2), in order to remove the layer damaged by ion sputtering during NanoSIMS imaging. After FTIR analysis, the section can be thinned a second time down to a thickness of ~100 nm for TEM imaging.

Conclusion:, The analytical sequences we developed in this work enable the extensive characterization of individual very small grains by a wide variety of 2D and 3D complementary techniques including microspectroscopy, microscopy and microscale mass spectrometry. Such analytical sequences could be very useful for the forthcoming Hayabusa 2 and OSIRIS-Rex sample return missions.

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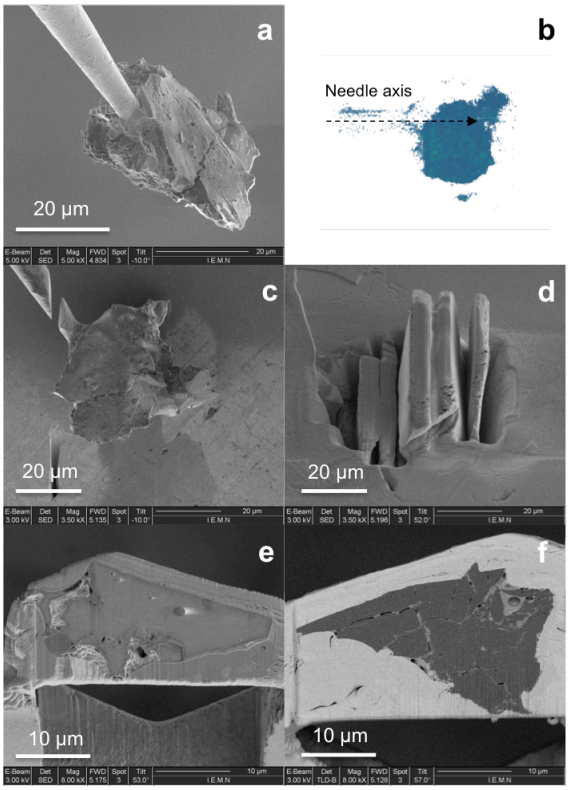

Figure 1: Preparation sequence of a ~20 µm grain from NWA 5515. a) grain welded at the end of a tungsten needle, b) SiO (1048 cm^{-1}) reconstruction by IR-CT, c and d) recovery and slicing of the grain respectively. FIB sections for TEM (e) and IR analyses (f).

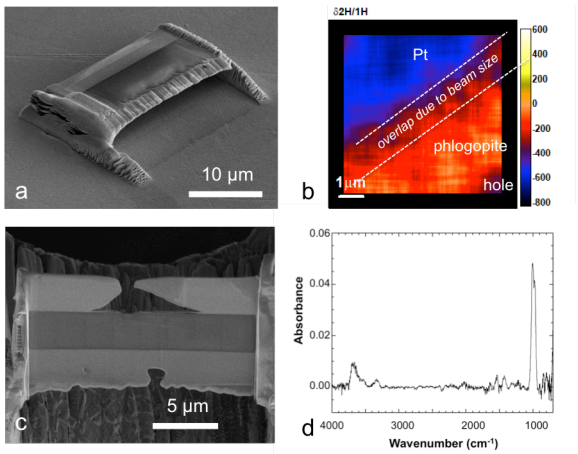

Figure 2: Combined NanoSIMS-FTIR imaging of the CRPG phlogopite standard. a) thick FIB section mounted on Pt bridges. b) NanoSIMS image of D/H ratio. c) FIB section recovered after NanoSIMS and thinned for FTIR. d) FTIR spectrum of the FIB section.