COORDINATED STUDIES OF PRISTINE CONCORDIA MICROMETEORITES. M. Gounelle[1], P. Bleuet[2], L. Bonal[3], J. Borg[4], M. Chaussidon[5], L. d'Hendecourt[4], Z. Djouadi[4], J. Duprat[6], C. Engrand[6], T. Ferroir[7], P. Gillet[7], F. Grossemy[4], C. Le Guillou[8], L. Lemelle[7], H. Leroux[9], B. Marty[5], A. Meibom[1], G. Montagnac[7], S. Mostefaoui[1], E. Quirico[3], B. Reynard[7], F. Robert[1], J.-N. Rouzaud[8], A. Simionovici[7] and B. van de Moortèle[7]. [1]LEME, MNHN, 75005 Paris, France (gounelle@mnhn.fr). [2]ESRF, 38043 Grenoble, France. [3]LPG, 38041 Grenoble, France. [4]IAS, 91405 Orsay, France. [5]CRPG-CNRS, 54501 Vandoeuvre-lès-Nancy, France. [6]CSNSM, 91405 Orsay. [7]LST, ENS, 69007 Lyon, France. [8]Lab. Géol. ENS, 75231-Paris. [9]LSPES, 59655 Villeneuve d'Ascq, France.

Introduction: The NASA Stardust space mission will bring back to Earth dust collected in the coma of the Jupiter Family Comet, Wild 2 [1]. Cometary dust might be similar to Interplanetary Dust Particles (IDPs) collected in the stratosphere [2], to polar micrometeorites collected in the Antarctic ice cap [3], to carbonaceous chondrites [4] or be totally different from any known extraterrestrial material. The size of Stardust samples is expected to be on the 1-10s µm size range so the use of state-of-the-art microscale analysis is imperative [5]. We have set up a consortium of French scientists specialized in the microanalysis of extraterrestrial matter. We have tested our ability to generate reliable scientific data, using a large diversity of instrumental techniques on submillimeter-sized samples (Concordia micrometeorite's fragments) within a limited amount of time. Two sets of samples were circulated between the laboratories, and analyses were completed within one month.

Sample collection and preparation: Analyses were performed on two Concordia micrometeorites (MMs), 03-31-Y and 03-32-S, that were collected at Dome C in 2002 [6]. The initial sizes of the 2 MMs were 100 x 100 µm and 90 x 60 µm, respectively. They were split in several fragments, with one of them (~ 30µm) embedded in epoxy and polished. SEM examination revealed a fine-grained, iron-sulfide rich matrix for both MMs (Figure 1).

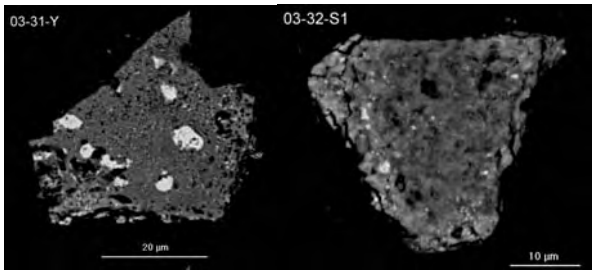

Figure 1 : SEM micrographs (backscattered electrons) of 03-31-Y and 03-32-S.

Another fragment of 03-31-Y (~ 20 µm) was embedded in epoxy and ultramicrotomed, producing 50 nm, 80 nm and 400 nm thick sections, while a fragment of MM 03-32-S (~20 µm before crushing) was pressed in a gold foil.

TEM Mineralogical Examination: Several ultramicrotomed sections of 03-31-Y were examined using a Tecnai G2-20-twin transmission electron microscope (TEM) at 200 kV, equipped with an EDAX X-ray detector for Energy Dispersive Spectroscopy (EDS). 03-31-Y is made of olivine, pyroxene and iron oxides cemented by an amorphous, SiO_2-rich phase (Figure 2), in agreement with previous TEM studies of micrometeorites [7]. Elemental mapping revealed the fine-grained and heterogeneous nature of 03-31-Y. Complementary high resolution TEM studies potentially allow to image directly the variety of insoluble carbons (nanodiamonds, graphitic and disordered carbons). Sections performed by ultramicrotomy (50 nm thick) appear convenient to obtain reliable images. In situ EDS elemental analyses are however required to discriminate strongly disordered carbons and amorphous silicates.

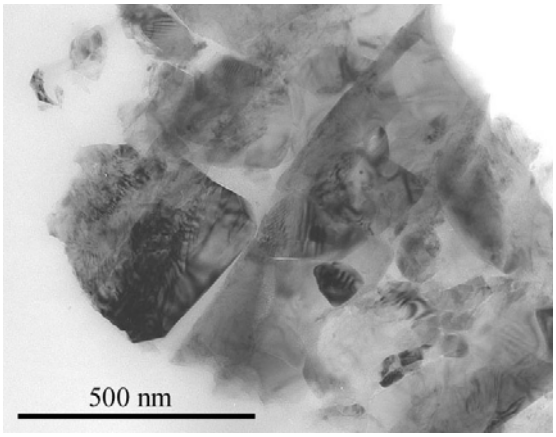

Figure 2 : TEM micrograph of 03-31-Y showing an olivine-pyroxene-magnetite assemblage, cemented by a SiO-rich glass.

Bulk composition: Synchrotron X-ray fluorescence (SXRF) mapping at 13 keV incident energy was performed on two 400 nm thick sections of 03-31-Y on TEM grids using the micron-sized high intensity beam of ID22, ESRF [8]. Quantitative analysis performed using SRM fluorescence standards allowed quantification of elements between Si and Zn (Figure 3). The Fe/Ni ratio varies from subchondritic to chondritic (Fe/Ni = 15 ± 0.8 and 17.5± 0.6). For the actual

Stardust grains we will also perform diffraction and Xanes directly on aerogel keystones to assay the mineralogy and oxidation states along the track.

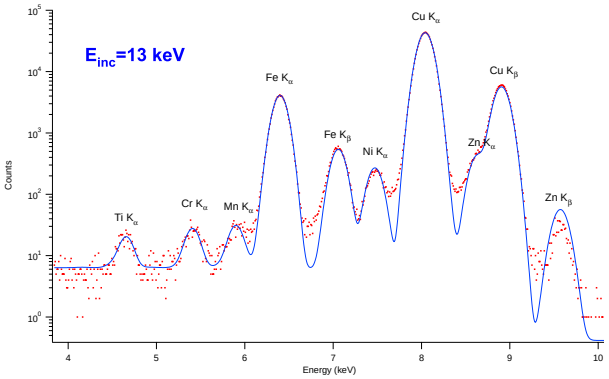

Figure 3 : SXRF spectrum for elements from Ti to Zn for 03-31-Y.

Raman Spectroscopy: Raman and LIF measurements on 400 nm thick sections of 03-31-Y on a TEM grid were performed under argon with a Jobin-Yvon Labram spectrometer, equipped with a x50 long working distance microscope objective, and an Ar^+ laser (514 nm excitation). The power onto the sample was < 100 µW, and the diameter of the spot was ~2-3 µm. UV Raman measurements were acquired with a similar instrument with a 244 nm excitation. The high carbon content prevents from the detection of any mineral features but gives clues on the structure of organic material. High fluorescence of the embedding epoxy masked signatures from the sample. The fluorescence intensity of this latter is however much lower than that measured for CI and CM matrix grains. The intensity and width of the D and G carbon bands obtained with a 514 nm excitation indicate that this particle has endured little thermal metamorphism [9].

Infrared Spectroscopy: IR-microscopy analyses were performed in the transmission mode using Fourier Transform InfraRed (FTIR) spectroscopy, with a Nicolet Magna-IR 560 ESP spectrometer attached to a Nicolet Nicplan Infrared microscope. For each sample, we did an average of ten 512 scans spectra in the 4000 to 650 cm^{-1} spectral range, at a resolution of 4 cm^{-1}. Spectra obtained on 03-31-Y for the two thin sections (80 and 400 nm) we analysed show typical bands of silicates around 1000 cm^{-1} and weak features around 2980-3000 cm^{-1} that can be attributed to C-H stretch signatures in CH_2 and CH_3.

NanoSIMS analyses: Following TEM and SXRF investigation, oxygen and hydrogen isotopic imaging were done on the same 400 nm thick sections of 03-31-Y using the CAMECA nanoSIMS 50 at the MNHN Paris. A 25 x 25 μm^2 region was scanned with a beam size of ~100 nm, producing a 256 x 256 pixels image. Internal precisions (1σ) are on the order of a few %, 2% and 1% for the ratios D/H, $^{17}O/^{16}O$ and $^{18}O/^{16}O$, respectively. No isotopic anomaly was detected for oxygen. A deuterium enriched value $\delta D = 352 \pm 54$ ‰ was identified (zone 1 in Figure 4). Additional hydrogen and oxygen isotopic images were obtained on 03-32-S (pressed in gold foil) using similar instrument setting, and showed solar compositions.

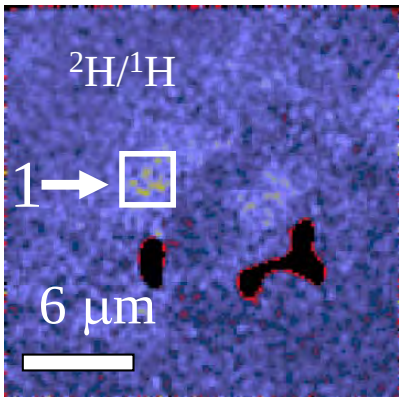

Figure 4 : D/H mapping of Concordia Micrometeorite 03-31-Y by NanoSIMS.

Conclusions: We have demonstrated our ability to obtain data on MM fragments with sizes comparable to that of the forthcoming Stardust samples within a limited time range (1 month). Microscale mineralogy seems to indicate that 03-31-Y has endured some moderate thermal metamorphism, which is not evidenced by the degree of structural order of the polyaromatic organic matter derived by Raman spectroscopy. Bulk composition is in line with a roughly chondritic sample. Infrared spectroscopy has identified CH_2 and CH_3 bonds. NanoSIMS analyses have identified a marginal deuterium excess.

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References:

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