Experiment Report Form

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Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

ESRF	**Experiment title:** Identification of cometary particles by micro-analysing techniques (fluorescence and XANES); Preparation for the return of the Stardust mission.	**Experiment number**: CH 1934
Beamline: ID21	**Date of experiment**: from: 15 June 2005 to: 20 June 2005	**Date of report**: 2005-08-18
Shifts: 15	**Local contact(s)**: Dr Diane EICHERT	*Received at ESRF:*

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Introduction

The aim of the experiment was to prepare the return of the Stardust mission that will bring back on Earth in January 2006 aerogel collectors in which grains of comet Wild-2 as well as interstellar grains have been trapped [1]. In order to develop an analytical protocol, we analyse Stardust samples analogues that consist of grains trapped inside pieces of aerogel named keystones [2]. Using synchrotron X-ray techniques (micro-fluorescence and XANES) we can identify and analyse the trapped grains in a non destructive manner, still in-situ in the aerogel. This allows us to study the particles evolution during their slowing down in the expanded silicon gel.

This experiment is a continuation of CH1574 where we have shown, on sample LC01B2, the feasibility and the importance of synchrotron techniques for the study of cometary grains trapped in their collectors [4,5,6]. During this experiment we have analysed four samples:

- ***LC01B2, 2D0401** and **2D0403***: samples originating from the ODCE (Orbital Debris Collection Experiment) of the NASA, exposed outside the MIR station for 18 months in 1996-1997. Sample ***LC01B2*** was analysed again since we were in optimum experimental conditions for XANES measurements compare to the previous run (CH1574). Furthermore the study of different samples in the same experimental conditions allows a direct comparison between them and it is also a way of testing if our results can be reproduced or not.

- ***8JUN05B***: Allende meteorite grains shot with a dust gun into remaining Stardust's aerogel. The aerogel used for the Stardust mission is particular since it has a gradient of density to gradually slow down the incident hypervelocity particle.

Fe-XANES spectra have also been acquired on standard materials (siderite, diopside, enstatite, fayalite, goethite and hematite) to be used as references for the data obtained on our samples.

Main results

Micro-fluorescence results

The first sample studied, ***2D0401***, was a special one. Indeed it was a keystone which has been cut along the penetration track of the incident grain so that enventual grains deposited along the track are in the open air. The advantage of this technique is that the beam can be focused on the grains without having to go throught aerogel but we feared that the trapped material could have been lost. As we can see on Fig.1, showing the iron distribution in the sample, this is not the case and even if some grains could have been lost there is still a lot of material in the track (see the hotspots along the penetration track in (b)) and the final grain has been identified.

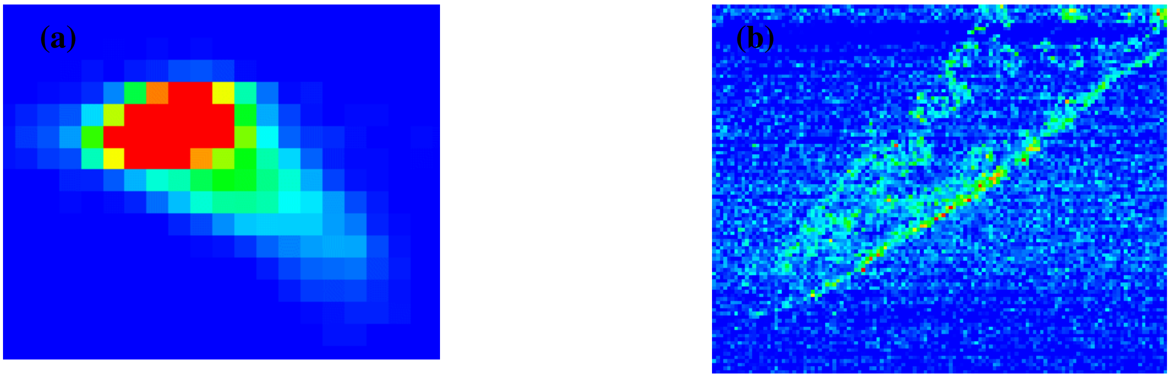

Fig. 1 – Fe repartition in 2D0401: (a) the final grain 4 µm in size. (b) the penetration track, about 250 µm long, with a spatial resolution of 2 µm.

Micro-fluorescence mappings have been performed on the other three samples. The final grain and the track have been clearly identified for LC01B2 and 2D0403. Nonetheless it was a bit more difficult for the latter one because the aerogel broke into several pieces when the chamber has been put under vacuum. As far as ***8JUN05B*** is concerned, the incident particle doesn't seem to have broken up into fragments during the slowing down in the aerogel. In the track entrance only iron beads, used to implant the Allende with the dust gun, have been found. They have been identified by their perfect sphericity and because they only contain Fe^0. This observation could indicate that Stardust's aerogel is very suitable for slowing down hypervelocity particles.

Fe-XANES results

During ***CH1574*** we have shown with the sample named ***LC01B2*** that the Fe redox state seems to vary from the entrance of the track (Fe^{3+}) to the end and the final grain (Fe^{2+}) [6]. In order to confirm this, Fe-XANES

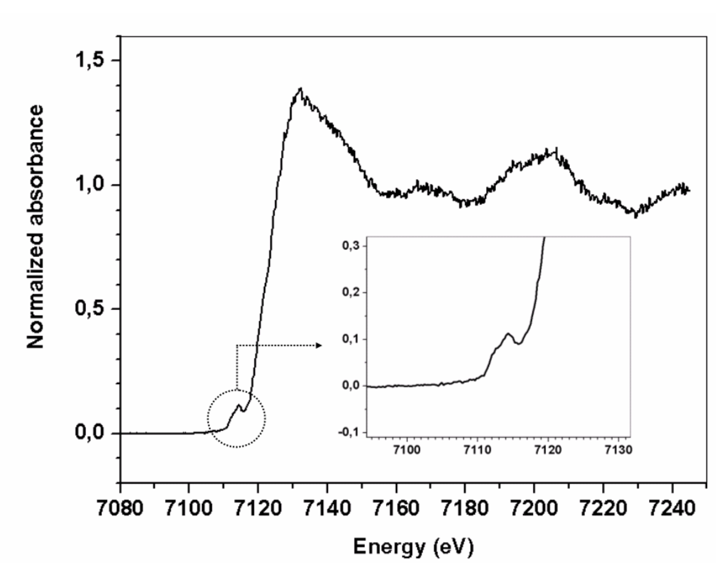

Fig. 2 – Fe K_α-XANES spectrum of the final grain of 2D0401.

Sample	Region	Edge energy (eV)	Pre-edge centroid (eV)
2D0401	(a)	7123.7	7114.2
	(c)	7119.9	7113.4
	final grain	7119.5	7113.4
2D0403	(b)	7119.2	7114.0
	(c)	7118.3	7113.4
	final grain	7119.7	7113.5
LC01B2	(a)	7123.2	7114.6
	(c)	7119.6	7113.6
	final grain	7119.7	7113.1
8JUN05B	final grain	7119.5	7113.5

(a) track entrance, (b) middle of the track, (c) end of the track
precision on the energy of 0.25 eV

Tab. 1 – Edge and pre-edge characteristics of Fe-XANES spectra.

spectra (Fig. 2) have been acquired in hotspot regions of each sample using a Si(220) double-crystal monochromator with an incident energy ranging between 7,1 and 7,28 keV. The beam size at the sample was 0,5x0,5 μm^2 with an energy resolution of 0,25 eV. Our spectra were calibrated in energy using an iron foil (edge at 7112 eV).

For each spectrum we determine the energy of the absorption edge of Fe which is shifted to higher energy when the oxidation state of Fe increases. We also studied the pre-edge feature deconvoluted into two Gaussian components using the software PeakFit 4, the centroid, area and amplitude of each component are derived. The centroid of the pre-edge is thus obtained by averaging the centroids weighted by their amplitudes. The principal results are summarized in Tab.1.

Spectra acquired on the standards indicate a shift of 1eV towards higher energy compare to what is found in the literature. Taking this into account, both the edge energy (7119.6 ± 0.1 eV) and pre-edge centroid (7113.3 ± 0.2 eV) indicate that the iron of the final grain and the end of the track is in Fe^{2+} form for all samples [7]. In contrast, in the track entrance we find the iron in its Fe^{3+} form. Based on the results obtained for the three first tabulated samples (no grain has been identified along the penetration track of the ***8JUN05B*** sample) we conclude that the iron constituting the incident grain undergoes a reduction during its slowing down into the aerogel.

Conclusions

The results presented here show the importance of Synchrotron X-Ray techniques for the in-situ identification of the cometary grains that will be brought back on Earth next January by the Stardust mission. We validate the ability to analyse the grains still trapped in a cut track. This new way of preparing the samples allows the coupling of other analytical techniques like Raman and IR microspectroscopies which are impossible in a bulk aerogel. Furthermore we have shown that hypervelocity particles do not seem to fragment when entering the Stardust's aerogel (characterised by a gradient of density), this could indicates that the cometary grains will be less dispersed than we are used to observe in aerogel with no gradient of density. Our Fe-XANES studies have shown that the iron of the incident grain is reduced during the slowing down in the aerogel. The data analysis is still in progress in order to quantify the amount of ferrous ions against ferric ones and to explain this reduction since heating is expect to rather have an opposite effect.

References

[1] Brownlee, D. E. Et al. (2004) The Stardust: A successful encounter with the remarkable comet Wild 2. *LPSC XXXV*, Abstract #1981.

[2] Westphal, A. J. et al. (2004) Aerogel keystones: Extraction of complete hypervelocity impact events from aerogel collectors. *Met. & Planet. Sc.*, **39**, 8, 1375-1386.

[4] Borg, J., Deboffle, D., Djouadi, Z., Eichert, D., Martinez-Criado, G., Snead, C.J., Somogyi, A., and Westphal, A.J. (July 2004) In-situ analyses of extraterrestrial grains trapped in aerogel. *35th COSPAR, Paris.*

[5] Borg, J., Djouadi, Z., Matrajt, G., Martinez-Criado, G., Snead, C.J., Somogyi, A., and Westphal, A.J. (2004) In-situ analyses of Earth Orbital grains trapped in aerogel, using Synchrotron X-ray microfluorescence techniques. *LPSC XXXV, Abstract #1580, Lunar and Planetary Institute, Houston (CD-ROM).*

[6] Borg, J., Djouadi, Z., Grossemy, F., Eichert, D., Martinez-Criado, G., Westphal, A.J., Deboffle, D., Snead, C.J., and Somogyi, A. (2005) Synchrotron X-rays in-situ analyses of extraterrestrial grains trapped in aerogel. *Accepted for publication in Advances in Space Research.*

[7] Wilke, M., Farges, F., Petit, P-E., Brown Jr, GE. And Martin, F. (2001) Oxidation state and coordination of Fe in minerals: An Fe K-XANES spectroscopic study. *Amer. Min.*, **86**, 714-730.