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Experiment Report Form

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- 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.

	Experiment title: Identification of interplanetary dust particles by micro fluorescence techniques	Experiment number: CH1574
Beamline: ID21 and ID22	**Date of experiment**: from: November 2003 to: January 2004 first run on ID22 : 12 – 17 nov 03 2nd run on ID21 : 29 – 31 janv 04	**Date of report**: 24/01/2005
Shifts: 30	**Local contact(s)**: Andrea Somogyi and Jean Susini	*Received at ESRF:*

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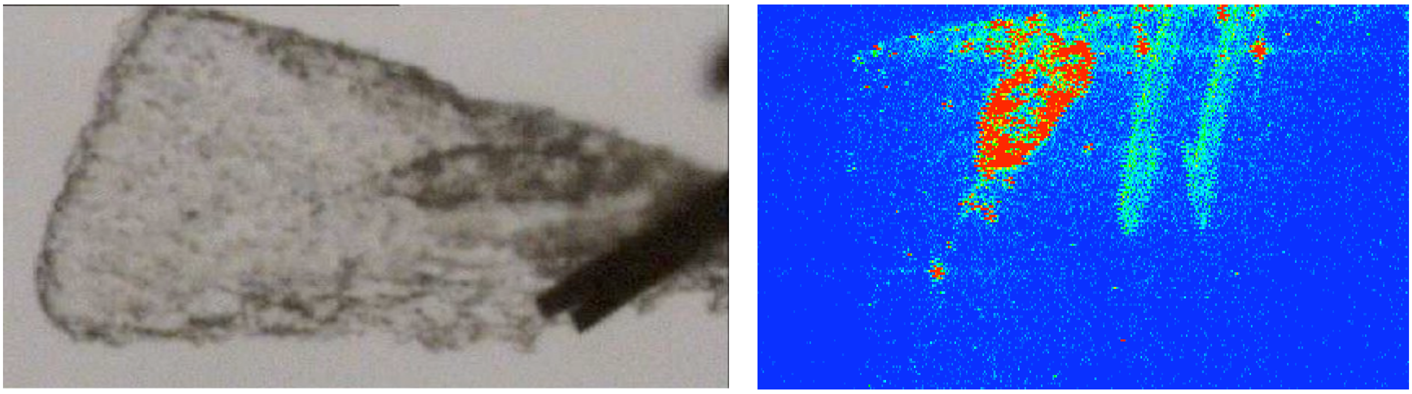
Introduction

The aim of the experiment was the preparation of the exploitation of the Stardust mission, that will return in January 2006 aerogel collectors in which cometary grains of comet Wild-2 will be trapped, as well as interstellar grains captured during the journey to the comet. As no modeling of the slowing down of the particles, incident at velocities of a few km/s, is available and only very seldom laboratory simulations have been performed, our results obtained on ID21 and ID22 on the slowing down of the grains in aerogel expanded silicon gels of very low density are extremely important. We analysed mainly one sample originating from a NASA experiment called ODCE (Orbital Debris Collection Experiment), exposed outside the MIR station for 18 months in 1996-97. This sample, called LC01B2, consists in a piece of aerogel 200x400 μm, in which a grain is trapped (Fig. 1a).

Main results

μ-fluorescence results on ID22

The grain, less than 3 μm in size, is visible at the end of the carrot-shaped penetration track. In order to analyse such samples, the use of synchrotron Xray microbeams is extremely valuable as the aerogel is transparent to incident beams with energies of a few keVs, which are only absorbed by the trapped grains, which are thus identified. We first worked with the beam delivered by ID22: a monochromatic beam, at energies of 10 and 7 keV, and fluxes of a few 10^{11} photons/s. After some preliminary investigation on aerogel samples that had been implanted with grains of the Murchison meteorite using a dust gun, we concentrated on the study of LCO1B2. The sample was self supported on a microforklift that was fixed on a goniometer head. Successive mappings at smaller and smaller steps were performed, from 10 μm down to steps of 1μm, in order to have the distribution of the main elements present in the grains at the end and along the track (Fig. 1b). The sample and the detector were placed in a He chamber to reduce the slowing down of



the light energies Xrays.

Fig. 1 – LCO1B2 in visible light (1a) and Fe-X image (1b). The terminal grain (<3μm), the penetration track and the self supporting microforklift are clearly visible.

By mapping several regions in the sample (terminal grain, other Fe-rich grains along the track), it has been shown that : i) the incident grain has a composition compatible with an extraterrestrial origin, based on the value of the Fe/Ni ratio, directly obtainable from the Xray spectrum, ii) the grains along the track show more or less the same composition, suggesting an erosion of the incident grain during its slowing down. Another possibility is a fractionation of an original agregate in many sub-grains. In any case, our results show that one must be extremely cautious when analysing the Stardust samples, and not only concentrate on the study of the terminal grain which might not be an exact signature of the pristine grain.

μ-fluorescence results on ID21

In order to investigate the light elements distribution and their behaviour during the slowing down in the collector, we also performed mappings at ID21. As we needed at the same time Fe-XANES investigation (see below) and because of time considerations, we only worked at an incident energy of 7 keV, which is not the most favorable for light elements identification. Furthermore, in order to use the ID21 sample holder, it is needed to place the sample under vacuum and we had to install our samples between 2 thin ultralen films. This operation resulted in the loss of 2 samples originating from ODCE (LCO1B1 and B3), that were extremely precious for confirming our results obtained on LCO1B2. Fortunately, we did not lose LCO1B2 and after many vain tentatives, we could finally place it correctly in the ultralen sandwich. We used the same rules as on ID22 for the fluorescence emission characterisation and obtained images at higher resolutions of the grain and its track. Unfortunately spectra obtained are very noisy and have given no further information on elements present in grains at the end and along the track. Longer counting times are required to possibly see light elements like Al or Mg.

Fe-XANES results on ID21

During the same run we tested the possibility of XANES investigation on grains trapped in aerogel. Indeed the analysis of XANES spectra can complete the information we have on the matter constituting the track in order to better understand the slowing down of the incident grain inside the aerogel. Fe-XANES spectroscopy provides direct information for the Fe redox state, which can reveal eventual heating during entrance of the grain and can help us determining which mineralogical state of crystal Fe belongs to.

We used an energy range between 7 and 7,5 keV to completely cover the K-edge of Fe at 7125 eV. The beam size at the sample was 0,5x0,5 _m^2 with an energy resolution of 0,3 eV. Our spectra were calibrated in energy using 2 standards: siderite ($FeCO_3$, pre-peak position at 7112,6 eV) and hematite (Fe_2O_3, pre-peak position at 7113,5 eV) containing Fe^{2+} and Fe^{3+} respectively. Because of the very small size of the grains (≤ 3_m) and the lack of time we only have very low signal/noise ratio data. Nevertheless in figure 2 which shows the normalized Fe K -XANES spectrum for the terminal grain in LC01B2, the pre-edge feature can be observed and its centroid position is found at 7112,3 eV. A pre-edge feature is clearly observed for other regions studied (Fig. 3) showing that the Fe redox state varies from the entrance of the track where Fe is in the form of Fe^{3+} (pre-edge centroid at 7113,4 eV) to the end where Fe is in the form of Fe^{2+} (pre-edge centroid at 7111,8 eV).

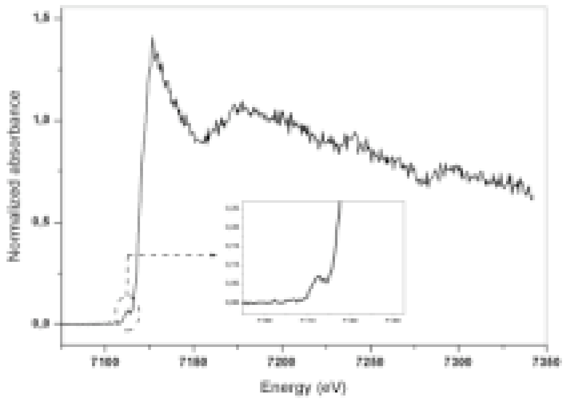


Fig. 2 – Fe K_-XANES spectrum of the terminal grain. The spectrum has been normalized by taking a mean absorption coefficient between 7200 and 7300 eV, so that the absorption is 1 in the EXAFS region.

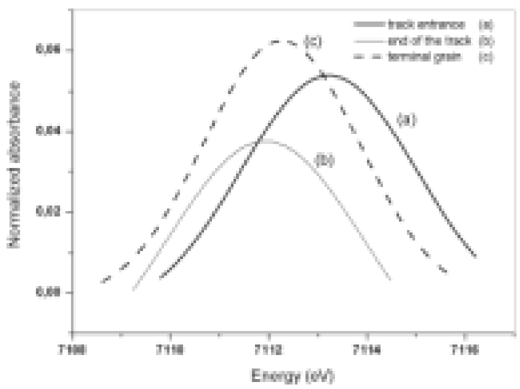


Fig. 3 – Centroid positions for three mainly studied regions of LC01B2: the entrance of the track (a), its end (b) and the terminal grain (c).

Conclusions

The results we have obtained are preliminary and we have tentatively interpreted them in terms of understanding how to better handle the Stardust samples. They already indicate the essential role that will be played by Synchrotron X-ray fluorescence and absorption techniques in the identification of the composition of the incident grain, without extracting the grain from its collector. These non-destructive techniques might be among the only ones to be used for analysing the interstellar grains trapped in the Stardust aerogel and for those cometary grains that might have lost most of their material along the penetration track. It seems reasonable to think that the material that will be brought back by Stardust from comet Wild-2 will qualitatively resemble the material analysed here: instead of dealing with a primary coarse grain, it will most probably consist in an aggregate that might disperse even more than what is observed here. So this first step of conserving the total of the incident particle in an individual keystone is crucial if one does not want to introduce a bias in the analysis of the primary grain. Until the return of the Stardust aerogels, big efforts will have to be made in the extraction of the grains and their complete characterisation.Our results are the first complete attempt of a non destructive analysis of an extraterrestrial grain trapped in aerogel. It is important to pursue these analysis on new samples, at higher resolutions in order to confirm these first results.

This work was presented at the 35th Lunar and Planetary Conference (Houston, March 2004) and at the COSPAR conference (Paris, July 2004). It has been accepted for publication in Advanced Space Resaearch review.

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