



## Experiment Report Form

**The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.**

Once completed, the report should be submitted electronically to the User Office using the **Electronic Report Submission Application:**

*<http://193.49.43.2:8080/smis/servlet/UserUtils?start>*

### ***Reports supporting requests for additional beam time***

Reports can now be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

### ***Reports on experiments relating to long term projects***

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

### ***Published papers***

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

### **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.



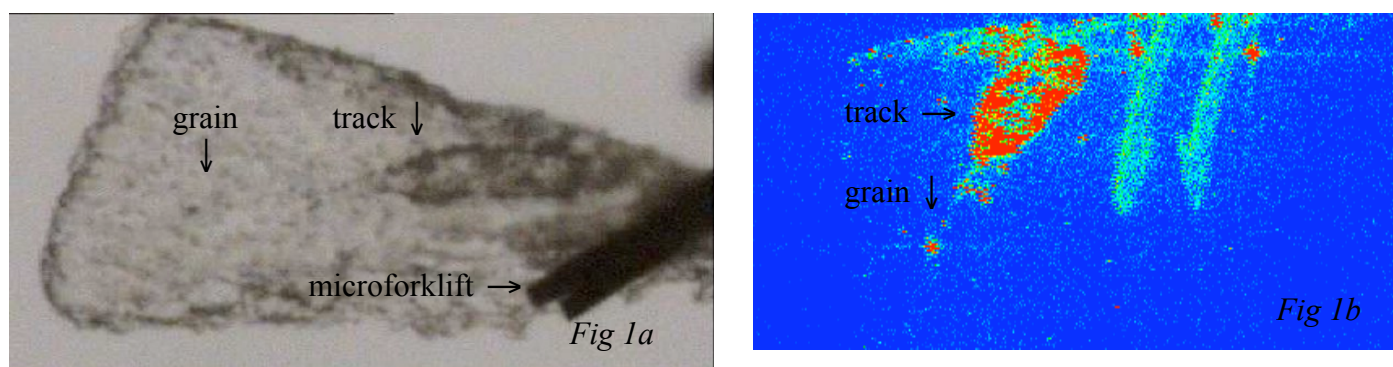
	<b>Experiment title:</b> Identification of interplanetary dust particles by micro fluorescence techniques	<b>Experiment number:</b> CH1574
<b>Beamline:</b> ID21 and ID22	<b>Date of experiment:</b> from: November 2003 to: January 2004 first run on ID22 : 12 – 17 nov 03 2nd run on ID21 : 29 – 31 janv 04	<b>Date of report:</b> 02/07/2004
<b>Shifts:</b> 30	<b>Local contact(s):</b> Andrea Somogyi and Jean Susini	<i>Received at ESRF:</i>
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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 aerogel collectors in which cometary grains of comet Wild-2 will be trapped. 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 200\*400  $\mu\text{m}$ , in which a grain is trapped (fig 1a).

## Main results

The grain, less than 3  $\mu\text{m}$  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  $\mu\text{m}$  down to steps of 1  $\mu\text{m}$ , 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 (< 3  $\mu\text{m}$ ), 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 aggregate in many sub-grains. In any case, our results show that one must be extremely cautious when analysing the Stardust samples, not to only concentrate on the study of the terminal grain which might not be an exact signature of the pristine grain.

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 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 more 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. The complete analyses of our data are not yet finished, as we have problems with Artemis on this beamline : for the moment, we only have Fe-images and no Xray – spectrum is available.

During the same run, we tested the possibility of XANES investigation on grains trapped in aerogel. This information is needed in order to better understand the behaviour of the incident grains inside the aerogel. We performed an absorption study of the main element present in the grains, that is Fe, and followed its oxydation state evolution along the track. Our data are compared with data for 2 standards, namely siderite ( $\text{Fe}^{2+}$ ) and hematite ( $\text{Fe}^{3+}$ ). Because of the very small size of the grains ( $\leq 3 \mu\text{m}$ ) and the lack of time, we only have very low signal/noise data; altogether, it seems that the Fe oxydation state varies from the entrance of the track ( $\text{Fe}^{3+}$ ) to the bottom ( $\text{Fe}^{2+}$ ) and that the terminal grain shows in equal proportions  $\text{Fe}^{2+}$  and  $^{3+}$ . This is not yet well understood, but, as a signature of the mineralogic state of the crystal to which the Fe belongs (most probably a silicate, if a meteoritical chondritic grain) is extremely important to confirm.

## Conclusions

Our results are the first complete attempt of a non destructive analysis of an extraterrestrial grain trapped in aerogel. Synchrotron Xray fluorecence and absorption techniques could be the only one available without extracting the grain from its collector, as IR and Raman spectroscopies give very poor and non exploitable results. It is important to pursue these analyses 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 will be presented at the COSPAR conference (Paris, July 2004). It will be submitted to Meteoritics and Planetary Science, or to another main review in Planetology.