



## 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> Study of Soot Growth and Nucleation by a Time Resolved Synchrotron Radiation Based X-Ray Absorption Method	<b>Experiment number:</b> SC-809
<b>Beamline:</b> ID09	<b>Date of experiment:</b> from: 21 June 2001 to: 25 June 2001	<b>Date of report:</b> 26 July 2001
<b>Shifts:</b> 12	<b>Local contact(s):</b> Dr. Michael WULFF	<i>Received at ESRF:</i>
<b>Names and affiliations of applicants (* indicates experimentalists):</b> <b>J. B. A. Mitchell*</b> , Université de Rennes I <b>C. Rebrion-Rowe*</b> , Université de Rennes I <b>J-L. LeGarrec*</b> , Université de Rennes I <b>G. Taupier*</b> , Université de Rennes I <b>N. Huby*</b> , Université de Rennes I <b>M. Wulff*</b> , ESRF		

## Report:

In a preliminary experiment, the absorption of x-rays by soot particles in an ethylene diffusion flame was examined, using a white beam on the ID09 beamline. The absorption was seen by measuring positive and negative currents on a thin (1 mm diameter) rod, mounted just above the beam pipe. The flame was displaced horizontally and vertically with respect to the x-ray beam in order to map the location of the soot particles in the flame. It was found that while low down in the flame, just above the burner mouth, there was little or no x-ray absorption by soot particles, higher up, the current due to soot absorption was a factor of about thirty greater than that due to absorption by the background air. (The experiment was performed at atmospheric pressure). Fig 1 a shows the horizontal scan of measured negative current at 32 mm vertical height above the burner mouth (where the soot particle density is maximum) while fig. 1b shows the current measured at a height of 2 mm above the burner where the soot density is small. The probe used was 6 cm long and was positioned along the beam axis. The burner diameter was 1.2 cm and so the collection length for air ions was about five times that for flame produced species.

This measurement shows that it is possible to use x-rays to identify the position of flame particulates and this could be a useful method of mapping soot particles in fuel rich flames which would be opaque to visible radiation. The technique could also be applied to the study of flame produced nanoparticles such as TiO<sub>2</sub> or SiO<sub>2</sub> that are produced industrially in large quantities using combustion methods and to probe particulates formed during silicon based plasma-processing operations. The experiment has very interesting applications in astrophysics. Dust particles in the interstellar medium are exposed to high fluxes of x-rays from hot stars and other violent phenomena and their response to this radiation must be known in order to interpret observations. A number of models of this interaction exist [1-3].

The fact that such a high soot absorption signal is seen in this paper is at first sight very surprising. Typical soot volume fractions in the flame used were of the order of  $4 \times 10^{-6}$  at their maximum. This has been determined by other workers using a number of different methods and was verified by us by studying helium/neon laser light absorption. If the absorption due to soot particles is calculated using an independent

atom assumption, (typical practice for x-rays in the energy range of 5-30 keV used in this experiment), the absorption due to soot particles should be about 1000 times weaker than that due to background air ionization. The fact that such a large signal is seen indicates that the particulate form and structure must play a dominant role here.

The process of absorption of a high energy x-ray photon in a particle involves a primary ionization event that leaves one or more inner shell vacancies in the target atom. This vacancy is filled by an electron from a higher shell either of the target atom or of a neighboring atom. In this event a high energy photon can be emitted (fluorescence) or the energy release can be absorbed by the ejection of another electron from the atom (the Auger effect). If a lower lying electron is ejected then a second such Auger process can occur etc. This is known as an Auger cascade though for carbon only one, 262.4 eV Auger electron is released [1].

Since the target atom is located within a solid matrix, the departing primary photoelectron and the Auger electron must traverse this material in order to escape. During this traversal, these electrons can undergo inelastic collisions with other atoms in the solid, resulting in secondary electron emission. Thus the primary x-ray absorption can lead to the ejection of a number of electrons. If such a process occurs in an electrically unbiased bulk solid, the electrons will probably return to the surface thus re-neutralizing it. In the case of a particle, particularly in the flowing environment of a flame where convection dominates charged particle movement, it is quite likely that the electrons will escape and this will leave the particle with a net positive charge. (Natural soot particles usually are electrically charged, whether positively due to thermionic emission or negatively due to electron or negative ion attachment. Flames also contain ions formed via chemical processes. These phenomena are responsible for the current measured with the flame but without the x-ray beam). If the charge due to x-ray absorption is sufficient, a very high electric field can be produced and this will lead to the phenomenon of field emission of electrons. This will be a runaway process and can lead to the actual disruption of the particle that will break up into positively charged fragments [2,3] and free electrons but we believe that this is the first time that actual experimental evidence for such an x-ray induced process has been reported.

Future experiments will explore this effect for other fuels and under vacuum conditions using a pre-mixed flame. This will allow the post-absorption process to be studied in more detail and efforts will be made to identify the charge carriers.

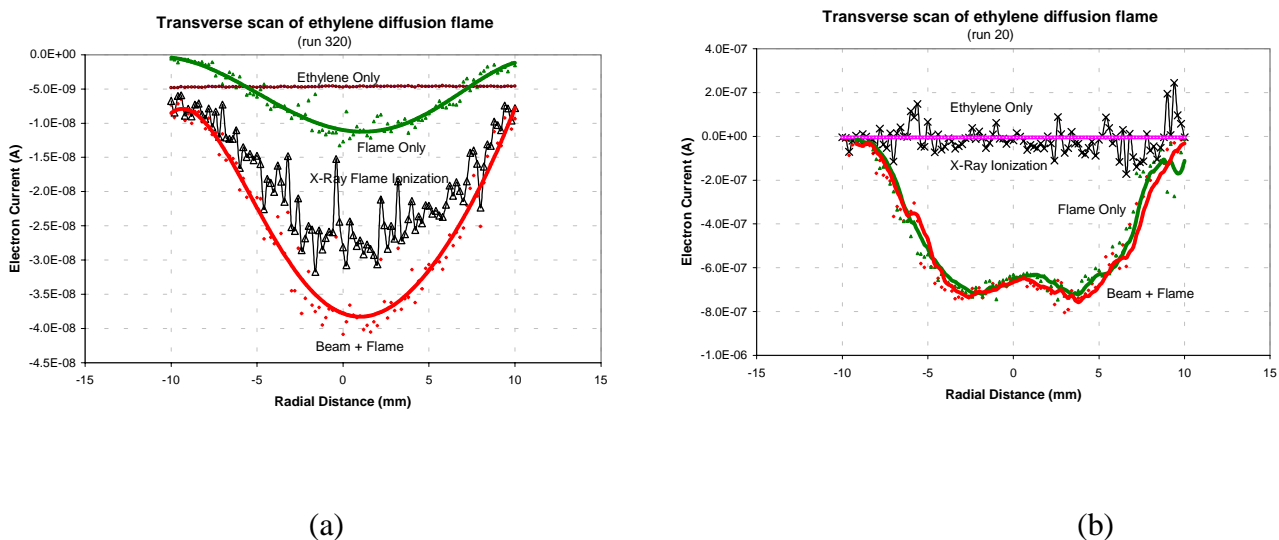


Fig. 1. Negative current measured with x-ray beam intersecting flame at heights of (a) 32 mm and (b) 2 mm above the burner mouth.

## References

1. B.T. Draine and E.E. Salpeter, *Astrophys. J.* 231, 77, 1979.
2. E. Dwek and R.K. Smith, *Astrophys. J.* 459, 686, 1996.
3. C.A. Chang, A.V.R. Schiano and A.M. Wolfe, *Astrophys. J.* 322, 180, 1987.



