

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 via the User Portal:

<https://wwws.esrf.fr/misapps/SMISWebClient/protected/welcome.do>

Reports supporting requests for additional beam time

Reports can 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.



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| | Experiment title: Melting relations of Fe ₃ S at high pressures: implications for the mineralogy of the core | Experiment number: ES-574 |
| Beamline: ID27 | Date of experiment: from: 14 July 2017 to: 18 July 2017 | Date of report: 17 May 2018 |
| Shifts: 12 | Local contact(s): Volodymyr SVITLYK | <i>Received at ESRF:</i> |
| Names and affiliations of applicants (* indicates experimentalists): *Tetsuya Komabayashi (main proposer), *Helene Breton, School of GeoSciences, University of Edinburgh *Takaaki Kawazoe, Bayerisches Geoinstitut, University of Bayreuth *Yuta Shimoyama, Department of Earth and Space Science, Osaka University | | |

Report:

Within the allocated beamtime, we conducted high-pressure (P) and –temperature (T) in-situ X-ray diffraction (XRD) experiments on Fe₃S samples in a laser-heated diamond anvil cell at ID27. The principal research goal is to place constraints on melting phase relations of Fe₃S under high pressure.

In-situ XRD experiments were performed at ID27 with a monochromatic wavelength of 0.3738Å. The sample temperature was measured by the spectroradiometric system available on the beamline and the sample pressure was obtained from the Raman shift of the diamond anvil before and after heating.

We operated ten separate runs: 1 at 32 GPa, 2 at 48 GPa, 1 at 49 GPa, 3 at 56 GPa, 2 at 57GPa, and 1 at 101 GPa. First we increased the pressure at room temperature. At a constant load, the temperature was then increased by laser. XRD patterns were repeatedly collected during laser heating. Laser alignment was made on each sample and the amorphous starting material was turned into crystalline Fe₃S phase during the alignment. With further increasing temperature, the XRD peaks from Fe₃S disappeared. Also the temperature was getting hard to increase when we lost the peaks. The disappearance of XRD peaks and the observed so-called plateau in temperature may be associated with melting of the sample. Another important observation was that Fe₃S congruently melted as we did not observe any other solid phases in the system Fe-S.

Textural observation in a SEM will be made at University of Edinburgh to provide independent evidence of melting of Fe₃S.