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ESRF

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

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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.

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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.

ESRF	**Experiment title:** Commissioning of a Mobile Laser Heating System at ID20 for Resonant X-ray Emission Spectroscopy and X-ray Raman Scattering at Extreme Conditions	**Experiment number:** ES-356
Beamline:	**Date of experiment:** from: 22 Jul 2015 to: 28 Jul 2015	**Date of report:** 7 March 2017
Shifts: 18	**Local contact(s):** Ch.J. Sahle	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

The aim of this experiment was to comissioon a mobile laser heating system, developed by the Bayerisches Geoinstitut Bayreuth (BGI) [1], and currently in use at ID09A and ID18, for usage at the inelastic x-ray scattering beamline ID20. The goal was to enable resonant- and non-resonant X-ray emission spectroscopy ((R)XES) and non-resonant X-ray Raman scattering (XRS) spectroscopy of materials at pressure and temperature conditions prevailing in the Earth's mantle and core. We were especially interested in determining the system's capabilities and limitations for the investigation of the electronic structure of materials under extreme conditions using the inelastic X-ray scattering techniques available at ID20.

At current, X-ray spectroscopic measurements at combined high pressure and high temperature in a Laser Heated Diamond Anvil Cell (LHDAC) are challenging and can only be carried out at very few synchrotron beam-lines across the world. Therefore, only few electronic structure studies in extreme-pressure mineralogy exist that follow the expected mantle geotherm. However, we need to elucidate the effect of simultaneous temperature and pressure when investigating questions related to the Earth's mantle and core. As one explorative question, we turned to the temperature dependence of the iron transition from a high to a low electron spin configuration in lower mantle minerals under compression, which has received considerable attention in the last few years [2-4]. This spin-transition of iron causes a drastic change in the elastic properties of minerals [5], making this process a question of primary importance for geophysics [6].

In a first experimental session, we used the high resolution spectrometer situated in experimental hutch EH2 of ID20 to study the Fe K-beta emission line of $FeCO_3$ under

simultaneous high-temperature and high-pressure conditions. A specially developed setup enabled us to laser heat along the compression axis in the horizontal direction while measuring the emitted X-ray photons in a „through-the-gasket“ geometry perpendicular to the compression axis, i.e. in the vertical direction. Fig. 1 a) shows the experimental setup as used on the high-resolution spectrometer in EH2 of ID20. Selected results of measurements at ambient temperatures and at 2000K are shown in Fig. 1 b). The presented spectra demonstrate the feasibility of our setup, but also demonstrate the weaknesses: the utilized high pressure Be gasket contained traces of Fe metal which resulted in spurious metallic Fe signal that could not be separated completely from the Fe K-beta line of the FeCO3 sample.

In a consecutive experimental session, we attempted to use the portable laser heating system at the large solid angle spectrometer for non-resonant inelastic X-ray scattering in EH3 of ID20. The utilized setup was the same as used for the XES experiments in EH2: laser heating parallel to the compression axis, whereas the inelastic X-ray scattering took place in the vertical scattering plane. Thus we could exploit all vertical detector modules or 36 analyzer crystals.

These XRS measurements were intended to allow studying the spin-transition in FeCO3 at the Fe M23 edge, i.e. as a complementary probe to the performed XES measurements. Although successfully reaching high-pressure and high-temperature conditions, the obtained Fe M23-edge spectra defy definite conclusions and several experimental difficulties have to be circumvented for future attempts. To avoid temperature gradients, the sample was chosen to be of smaller extend than both, the laser and X-ray beam size, i.e. of the order of 10 microns. This leads to considerable background noise in the overall scattering signal.

In conclusions, we have tested and used the portable laser heating system of ID18 to be used at ID20. Resonant and non-resonant emission spectroscopy experiments are well within reach of the current experimental setup. However, care has to be taken for the choice of pressure medium and gasket material. Therefore, we suggest to next explore the emission lines of Co, Ni, Mn. Non-resonant inelastic X-ray scattering remains challanging at present, but we see considerable potential in conjunction with the expected smaller X-ray beam size after the ESRF EBS upgrade program.

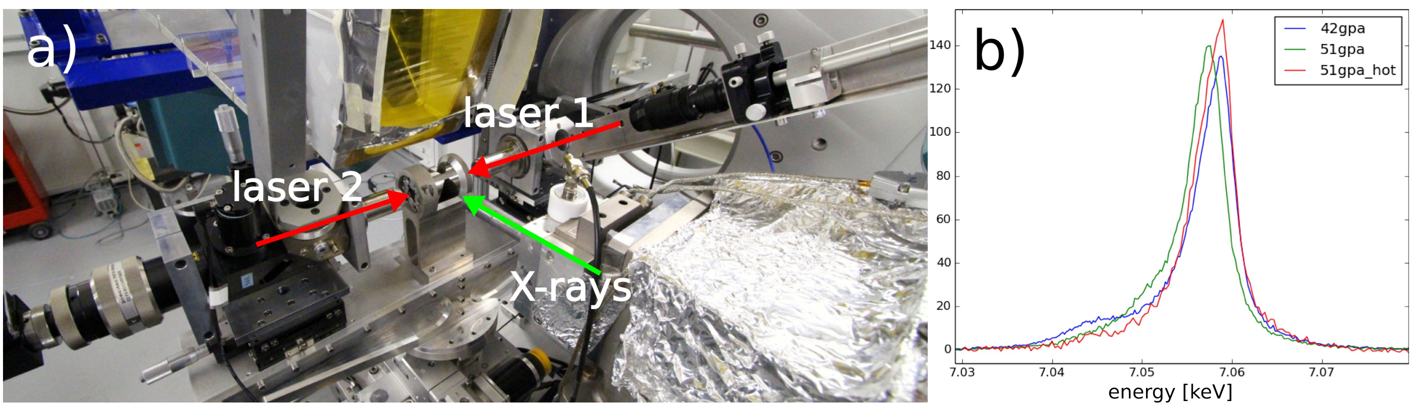

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Figure 1: a) Fotograph of the experimental setup in EH2. b) example data taken at the Fe K-beta emission line.

[1] Kupenko et al. (2012) Rev. Sci. Instrum., 83, 124501. [2] Badro (2014) Annu. Rev. Earth Planet. Sci., 42, 231-248. [3] McCammon et al. (2013) High Pressure Res., 33(3), 663-672. [4] Kupenko et al. (2014) Lithos, 189, 167-172. [5] Speziale et al. (2007) J. Geophys. Res., 112, B10212. [6] Antonangeli et al. (2011) Science, 331, 64-67.