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 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.

ESRF	**Experiment title:** Stability of Carbon and Sulfur-rich metals in the deep Earth's mantle	**Experiment number**: ES-632
Beamline:	**Date of experiment**: from: 29.11.2017 to: 04.12.2017	**Date of report**: 02.03.2018
Shifts: 18	**Local contact(s)**: Kristina Spektor	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Arno Rohrbach*, Vasily Potapkin*, Tobias Grützner*, Sebastian Hackler*: Institut für Mineralogie, Westfälische Wilhelms-Universität Münster, Correnstr. 24, 48149 Münster, Germany

Report:

The crystallization of superdeep big diamonds is believed to be closely related to the presence of reduced accessory phases in a molten from [1]. This hypothesis is strongly supported by recently performed analyses of metal, carbide and metal melt inclusions found in large diamonds like Cullinan, Constellation, and Koh-i-Noor [2]. As it is well known that alloying and incorporation of the light elements can significantly change melting temperatures, it is vital to know melting temperatures and the sub-melting phase diagram of the Fe-Ni-S-C system at relevant P-T conditions in order to unravel the diamond forming process. Up to now melting temperatures were solely based on quench experiments whose textures were generally difficult to interpret [e.g. 1]. Experiment ES-632 is an *in-situ* high-pressure high-temperature XRD study of the system over a wide range of pressures and temperatures where we derive subsolidus phase diagrams and the Clapeyron slope of solidus and liquidus to understand the nature of the reduced species at depth.

We performed four successful high P experiments at ID06 LVP, two runs at 10 and 19 GPa respectively. The fifth experiment failed during heating because of a high T blowout. We studied two different compositions with varying Fe/Ni and (Fe,Ni)/S ratios at each pressure and collected diffraction patterns continuously during heating from room T to max. 1893 K. Run pressures were calculated from EOS of Fe metal, Ni metal [3] and MgO [4]; temperatures were calculated from the thermal EOS of MgO [4].

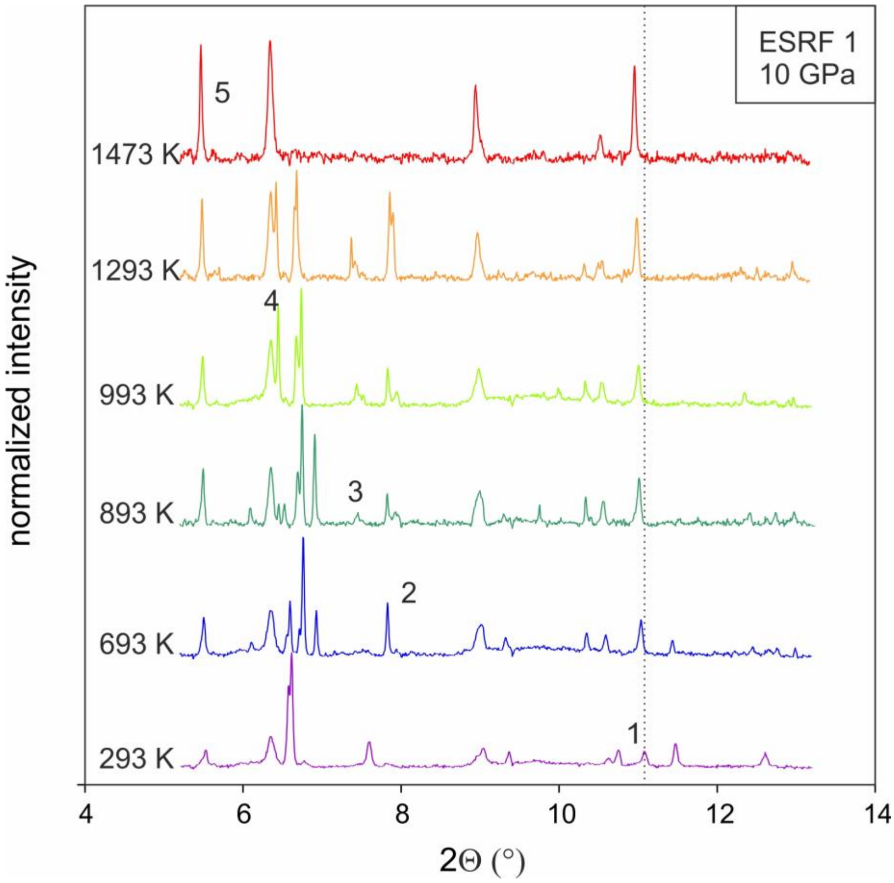

Figure 1: Diffractograms of experiment ESRF1 at 10 GPa between room T and 1493 K with a Fe-Ni-S-C composition, 10/4 assembly, Re heater, MgO single crystal capsule. Room T pressure calulation from MgO, Fe and Ni reflections. Note the shift of the MgO (222) reflection position to lower 2Θ as function of T (1). Key features of this experiment are (2) crystallization of (Fe,Ni)S, (3) crystallization of Fe_7C_3, (4) crystallization of diamond, (5) super-liquidus conditions with only MgO (capsule) and diamond refections.

We derived similar crystallization and melting sequences for the other experiments and analyzed quenched experiments at Münster University by SEM-EDX for major elements.

References:

[1] Rohrbach A, Ghosh S, Schmidt MW, Wijbrans CH, Klemme S (2014) The stability of Fe-Ni carbides in the Earth's mantle: Evidence for a low Fe-Ni-C melt fraction in the deep mantle. *Earth Planet. Sci. Lett.* **388**, 211–221.
[2] Smith EM, Shirey SB, Nestola F, Bullock ES, Wang J, Richardson SH, Wang W (2016) Large gem diamonds from metallic liquid in Earth’s deep mantle *Science* **354**, 1403–1405.
[3] Campbell AJ, Danielson L, Righter K, Seagle CT, Wang Y, Prakapenka VB (2009) High pressure effects on the iron–iron oxide and nickel–nickel oxide oxygen fugacity buffers. *Earth Planet. Sci. Lett.* **286**, 556–564.
[4] Speziale S, Zha C-S, Duffy TS, Hemley RJ, Mao H-k (2001) Quasi-hydrostatic compression of magnesium oxide to 52 GPa: Implications for the pressure-volume-temperature equation of state *J. Geophys. Res.* **106(B1)**, 515–528.