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.

<table>
<tr><td>ESRF</td><td>Experiment title:
Single-crystal X-ray diffraction studies of Fe-C-N system at deep Earth's interior conditions</td><td>Experiment number:
ES-392</td></tr>
<tr><td>Beamline:
ID27</td><td>Date of experiment:
from: 13.04.2016 to: 17.04.2017</td><td rowspan="2">Date of report:
11.09.2017

Received at ESRF:</td></tr>
<tr><td>Shifts:
12</td><td>Local contact(s):
Mohammed Mezouar</td></tr>
<tr><td colspan="3">Names and affiliations of applicants (* indicates experimentalists):
*Maxim Bykov (Bayerisches Geoinstitut, University of Bayreuth, Bayreuth, Germany)
*Leonid Dubrovinsky (Bayerisches Geoinstitut, University of Bayreuth, Bayreuth, Germany)
*Elena Bykova (Bayerisches Geoinstitut, University of Bayreuth, Bayreuth, Germany)</td></tr>
<tr><td colspan="3"></td></tr>
</table>

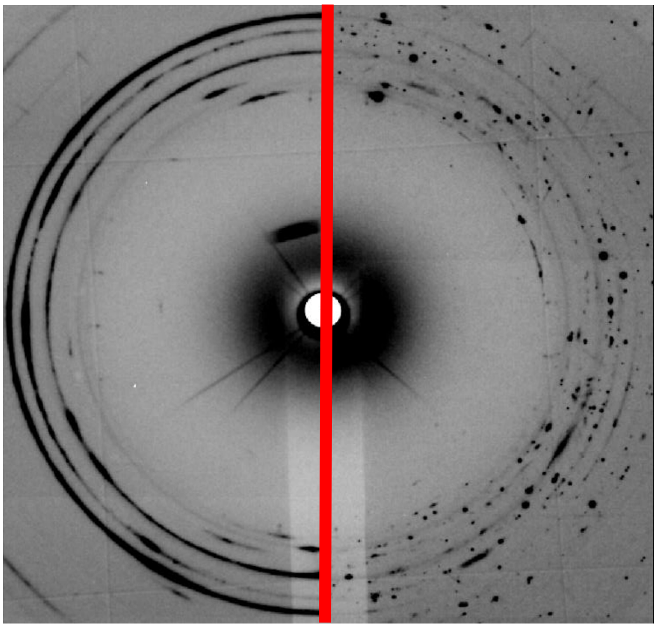
Report:

A piece of ^{57}Fe was placed inside a 60 µm hole in a Re gasket, preindented to the thickness of 22 µm. The sample chamber was loaded with nitrogen, which served as a pressure-transmitting medium. We used BX90 diamond anvil cells equipped with Boehler-Almax type diamonds (culet diameter of 120 µm). The double-sided laser-heating systems of the beamlines ID18 and ID27 of ESRF were used to heat the sample at the three pressure points: 60, 106 and 130 GPa.

Whereas the starting material, a polycrystalline iron foil, gives characteristic Debye-Scherer rings in the diffraction pattern, after the laser-heating in solidified nitrogen, we clearly observed well defined, sharp diffraction spots from multiple grains of new high-pressure high-temperature phases (Figure 1). We were able to identify the diffraction spots belonging to certain domains, find their orientation matrices, refine the unit cell parameters, and solve structures of the new phases against single-crystal diffraction data.

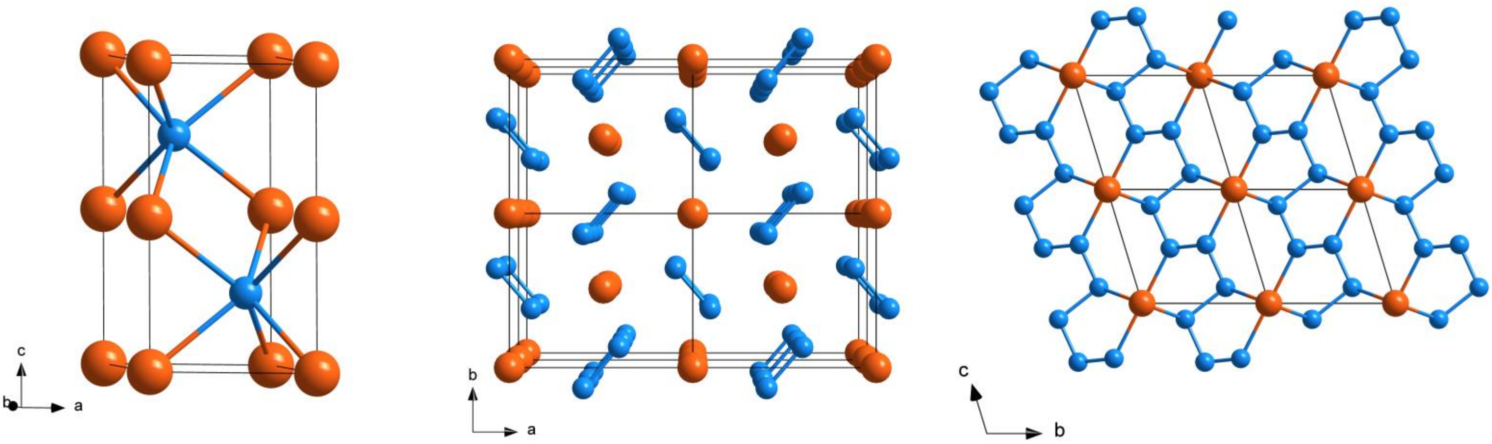
Heating at 60 GPa resulted in the formation of novel iron nitrides FeN and FeN_2 (Figure 2 a,b). FeN has B8-structure type, while FeN_2 has the marcasite structure type (space group *Pnnm*).

Heating at higher pressures (106 and 130 GPa) resulted in the appearance of nitrogen-rich nitride FeN_4. Accurately determined atomic coordinates and interatomic distances allowed us to unambiguously identify the bonding type within the nitrogen chains and show that FeN_4 contains poly-[tetraz-1-ene-1,4-diyl] anions in its structure (Figure 2c). FeN_4 is the first experimentally obtained and unambigously characterized nitride containing such polymeric nitrogen chains.



before heating **after heating**

Figure 1. Example of the observation of phase transformation in Fe-N system during the laser-heating. Left side of the image – non-heated sample. Right-side of the image taken after the laser-heating.



(a) **(b)** **(c)**

Figure 2. Crystal structures of FeN (a), FeN_2 (b) and FeN_4 (c). Blue balls show the position of nitrogen atoms, orange balls – iron atoms.