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.

	Experiment title: In-situ X-ray diffraction study of Fe-Si alloys in an internally-heated diamond anvil cell	Experiment number: ES-268
ESRF		
Beamline: ID27	**Date of experiment:** from: 18 April 2015 to: 23 April 2015	**Date of report:** 7 Sep 2015
Shifts: 12	**Local contact(s):** PARISIADIS Paraskevas	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

Within the allocated beamtime, we conducted high-presssure (P) and –temperature (T) in-situ X-ray diffraction (XRD) experiments on Fe-Si alloys in an internally-heated diamond anvil cell (DAC) at ID27. The principal research goal is to place constraints on the P-T location of the boundary between the face-centred cubic (FCC) structure and hexagonal close-packed (HCP) structure in Fe-4wt%Si.

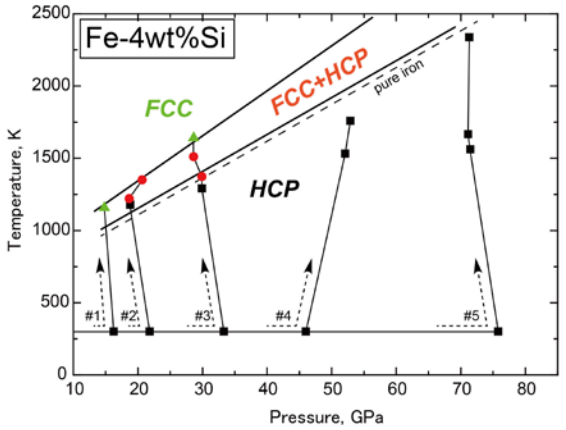
In-situ XRD experiments were performed at ID27 with a monochromatic wavelength of 0.3738Å. The sample temperature was measured by the spectroradiometric system availiable on the beamline and the pressure was obtained from the unit-cell volume of the HCP phase and its thermal equation of state (Tateno et al., 2015). Figure 1 shows a summary of the experimental P-T conditions and observed Fe-Si phases in Fe-4wt%Si in the internally heated DAC. Note that only selected data points are shown here. We conducted five separate sets of internal resistive heating experiments at different pressures. This has been the first time we ever used our internal heating techqnique at ID27. In all the runs we succesfully heated the sample at temperatures much higher than those currently achievable by external heating (T max ~1200 K), which demonstrates the feasibility and compatibility of this technique with ID27.

First we increased the pressure at room temperature. At a constant load, the temperature was then increased by directly appling a DC current across the metallic sample (thermally insulated from the diamods). The first cell was compressed to 16 GPa and then heated to 1160 K. We obseved disappearance of all the peaks of the HCP phase and appearance of FCC peaks at that temperature, the minumum value possible to measure by spectroradiometry in that run. Experiments on the second cell, showed coexistence of the FCC and HCP phases at 1220 and 1350 K. From experiments carried out on the third cell, we observed single FCC phase at the highest temperature and placed constraints on the HCP-out reaction. In the fourth and fifth runs, the HCP phase was observed stable till the highest reached temperatures.

For comparison, in Figure 1, we also plotted the HCP-FCC transition boudanry in pure iron (Komabayashi et al., 2009). The present experiments demonstrate that higher temperatures are needed to enter the FCC stability field for Fe-4wt%Si samples than pure iron, therefore. The FCC-HCP transition boundaries in Fe-4wt%Si is then placed at higher temprerature than in pure iron (Figure 1).

Figure 2 shows a comparison of the present data with existing literature data in Fe-3.4 wt%Si (Asanuma et al., 2008; Fischer et al., 2013). Note that Fischer et al. (2013) estimated the phase relations in Fe-3.4 wt%Si based on their experimental data in Fe-9 wt%Si. The results of the previous laser-heated DAC studies are clearly not in agreement. Moreover from comparisons with the pure iron boundary (Fig. 2), the two studies show opposite Si effect to the relative stability of FCC and HCP phases. Our new data are qualitatively consistent with those of Fischer et al. (2013), namely, addition of Si increases the transition temperature.

We also conducted two test runs in the laser-heated DAC on a Fe-6.5 wt%Si sample. We observed appearance of the FCC phase (i.e., FCC-in reaction) at 1860 K and 31 GPa and at 2370 K and 55 GPa. Such temperature conditions are higher than those for pure iron and therefore, confirmed the positive effect of Si on the transition temperature.



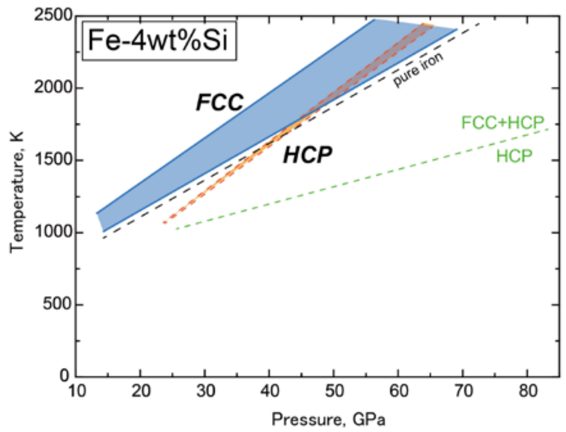


Figure 1 (left). Sumary of experimental conditions and results. Symbols are selected experimental data points: black, HCP; Red, HCP+FCC; Green, FCC. The FCC-HCP transition boundary in pure iron is also plotted (Komabayashi et al., 2009).

Figure 2 (right). Comparison of our FCC-HCP transition boundary in Fe-4wt%Si with the existing literature data in Fe-3.4wt%Si . Blue, this study; Red, Fischer et al. (2013); Green, Asanuma et al. (2008). The boundary in pure iron is alos plotted (Komabayashi et al., 2009).

References

Asanuma et al., 2008, GRL.
Fischer et al., 2013, EPSL.
Komabayashi et al., 2009, EPSL.
Tateno et al., 2015, EPSL.