EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

Experiment Report Form

	Experiment title: Phase relations in the Fe-Si-C system at extreme conditions	**Experiment number**: ES-819
Beamline: ID 27	**Date of experiment**: from: 21.11.2018 to: 25.11.2018	**Date of report**: 06.09.2021
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Report:

In this experiment, we have performed compression up to 143 GPa and laser heating up to 3700K of Fe-Si-C alloys. These alloys were Fe – 3.6 Si – 3.2 C, Fe – 5.2 Si – 3.6 C, Fe – 6.0 Si – 4.1 C, Fe – 3.8 Si – 3.2 C (numbers are weight percents). They were synthesized using the arc-melting method and characterized beforehand with electron micro-probe analysis in the University of Münster. The melt drops were polished down to ~ 20 μm, sliced to ~20x20 μm^2 slabs, and analyzed with EMPA. On ESRF our measurements were conducted on the facilities of the ID27 beamline.

We did XRD of the samples in the diamond anvil cells with rhenium gaskets using KCl as a pressure-transmitting medium and as a pressure standard. Initial samples were the mixtures of body-centered cubic iron (bcc-Fe alloyed with silicon and carbon) and cementite (Fe_3C). Upon compression bcc-Fe in these mixtures start to transform into hexagonal closest packed at 17 GPa. Upon further compression, up to 130 GPa at room temperature, no other phases were detected except hcp-Fe and cementite. However, heating changes the situation dramatically. In the composition Fe – 3.6 Si – 3.2 C during heating at 1300 K at 20 GPa we have observed the appearance of face-centered cubic iron (fcc-Fe), and iron silicide with B2 structure (B2-FeSi). In another mixture with a higher percentage of silicon (Fe – 5.2 Si – 3.6 C), at 58 GPa and 1400K, we noticed the Fe_7C_3 phase along with fcc-Fe and B2-FeSi. It is worth mentioning that amount of hcp-Fe is diminishing and its decomposition reaction takes place:

$$Fe(Si,C) \rightarrow Fe + FeSi + Fe_7C_3 \quad (1)$$

This reaction also happens with Fe – 3.8 Si – 3.2 C at 130 GPa but at around 2000 K and without hcp to fcc transformation. From these results, we infer that silicon substitution into iron upon heating leads to increased precipitation of carbon-bearing phases such as Fe_7C_3. At the same time presence of carbon shifts the B2-FeSi phase stability field to a more iron-rich region compared to the pure Fe-Si phase diagram[1]. Another interesting feature of mutual silicon and carbon impact is the existence of the fcc phase at more elevated pressures than it was previously established for Fe – Si phase digram[1]. A relatively high level of silicon substitution leads to FeSi precipitation upon heating which was not the case in the work of Miozzi et al.[2] where silicon weight percent was only 1.6. Comparison between room temperature diffraction plot and at 2000K at 59 GPa is shown in Fig.1.

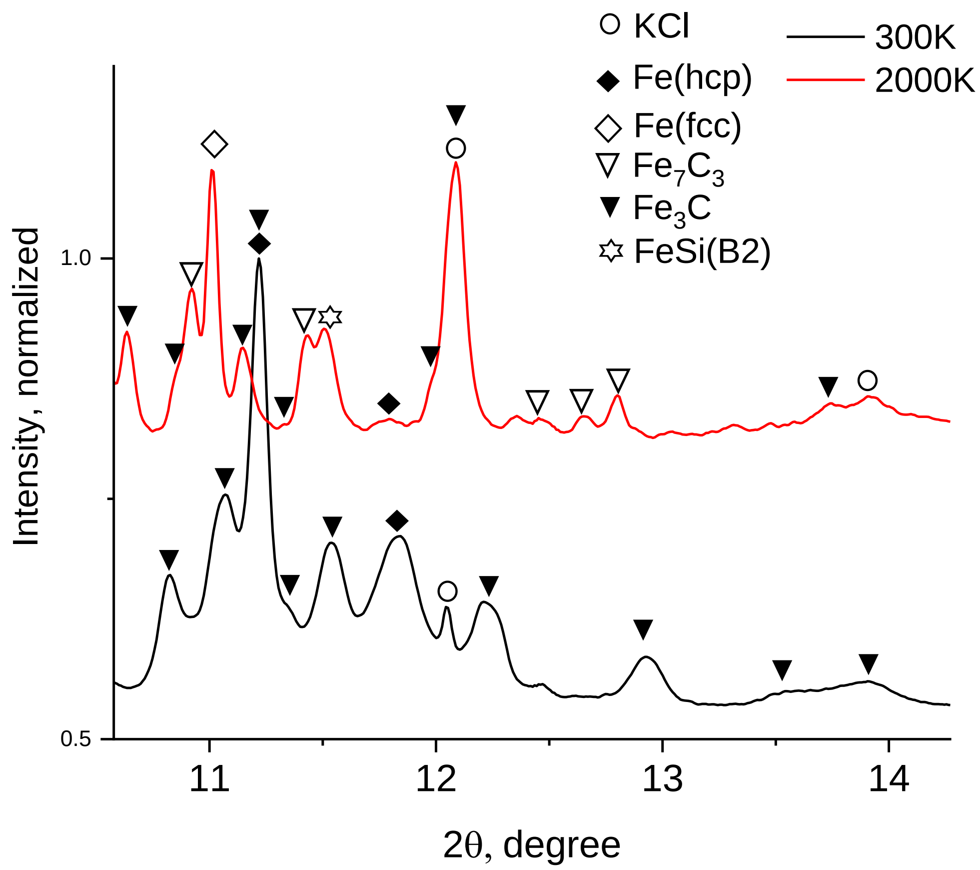

Figure 1. Diffraction plot of Fe – 5.2 Si – 3.6 C at room temperature and 2000 K (59 GPa).

We have performed the analysis of the measured data and identified the phase relations in the Fe-Si-C system up to about 70 GPa. Yet, in order to properly constrain it at higher pressures, additional experiments are required. Moreover, we have identified the formation of a previously unknown phase upon laser heating of the sample at 130 GPa. The single-crystal refinement indicates the cubic lattice with approximate composition $C_{6.7}Fe_1Si_{4.7}$.Yet the quality of the data does not allow excluding a chemical reaction with KCl pressure medium and incorporation of K and/or Cl into the crystal structure. Additional experiments with a different pressure transmitting medium are required to exclude such a chemical reaction and to confirm the new phase.

Supporting literature

1. Fischer, R. A. *et al.* Phase relations in the Fe–FeSi system at high pressures and temperatures. *Earth Planet. Sci. Lett.* **373**, 54–64 (2013).
2. Miozzi, F. *et al.* Eutectic melting of Fe-3 at% Si-4 at% C up to 200 GPa and implications for the Earth's core. *Earth Planet. Sci. Lett.* **544**, 116382 (2020).