	Experiment title:	Experiment number:
ESRF	**Experiment title:** Phase transitions in iron near the alpha-epsilon-gamma triple point	**Experiment number**: HC-2180
Beamline: ID27	**Date of experiment**: from: 03/12/2015 to: 06/12/2015	**Date of report**: 20/02/2016
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Report:

The aims of the proposal were: (i) to characterize the conditions and volume discontinuities for α–ε and α–γ transitions in iron near the α–ε–γ triple point; (ii) to better locate the position of the α–ε–γ triple point, which is poorly constrained in the literature; (iii) to determine the mechanism of these transformations from orientation relations between parent and child phases. One goal of this study is to better elucidate the interplay between magnetism and phase transformations between ferromagnetic α-Fe and paramagnetic ε-Fe and γ-Fe. For that purpose, we have used X-ray diffraction of a single crystal (or powder for one sample) compressed hydrostatically in a resistively-heated diamond-anvil cell.

We have carried out three experiments. The diamond anvil cells, loaded with iron samples embedded in neon pressure medium, were inserted in a vacuum resistive furnace which allows heating up to 850K on the samples. This device was provided by ID27. The temperature was measured by a thermocouple in contact with the back of one diamond anvil. The pressure was measured using a $SrB_4O_7:Sm^{2+}$ luminescence gauge and cross checked, when possible, with the neon pressure medium melting point. The phase and volume have been measured using angular-dispersive X-ray diffraction (see typical X-ray diffraction patterns in **Figure 1**). The conditions of the experiments are summarized in **Table 1**. The run FeBPHT_2 has been interrupted by a break of the heater.

Run name	Sample	Pressure medium	Pressure range (GPa)	Temperature range (K)
FeBPHT_1	Fe powder	Neon	5-19	300-481
FeBPHT_2	Fe single crystal	Neon	6-17	300-752
FeBPHT_3	Fe single crystal	Neon	6-12	300-813

Table 1: Conditions of the three experimental runs.

The P-T paths followed in the three runs are plotted in **Figure 1.** The measured conditions of direct and/or reverse α–ε–γ transformations (50% transformation) are plotted with large green or red symbols. The coexistence domains were relatively narrow (~2 GPa for α–ε).

We have made several interesting observations on the α–ε transformation:

- The dT/dP Clapeyron slope of the α–ε transformation decreases (in absolute value) by more than 50% above 600K, without any drastic change of the volume discontinuities. This suggests an increase in the entropy discontinuity between the phases with increasing temperature;

- Thermal expansions of α-Fe and ε-Fe progressively diverge from quasi-harmonic predictions when approaching the triple point. This could be explained by a magnetoelastic effect in α-Fe, although less dramatic than expected in Ref. [1], and needs to be further refined by more measurements;
- The α–ε mechanism determined from orientation relations, which was proven to be a Burgers path [2], remains unchanged up to 650 K (see **figure 1**, middle and right panels).

We have circled the triple point in run FeBPHT_3, which allowed us to constrain its location: 8<P<10 GPa, 723K<T<789 K. The uncertainties are due to both pressure drifts in this run and hysteresis in the transformations, which did not disappear under high temperature. We have not been able to determined orientation relations between α, ε and γ phases in this experiment. The volume and equation of state of γ-Fe needs to be constrained by more data points.

We have observed that as a result of high temperature recrystallization, ε-Fe progressively transforms into a highly mosaic single cristal around 700K. If we manage to get a better crystal, as well as in the γ phase, this should open new possibilities of characterization of iron under HP-HT, such as inelastic X-ray scattering studies.

To sum up, the conditions and mechanism of the α<–>ε transformation of iron have been accurately measured along the transition line up to conditions close to the α–ε–γ triple point. The position of this triple point has been constrained. The α–γ transformation (orientation relations, volume discontinuities) remains to be better characterized.

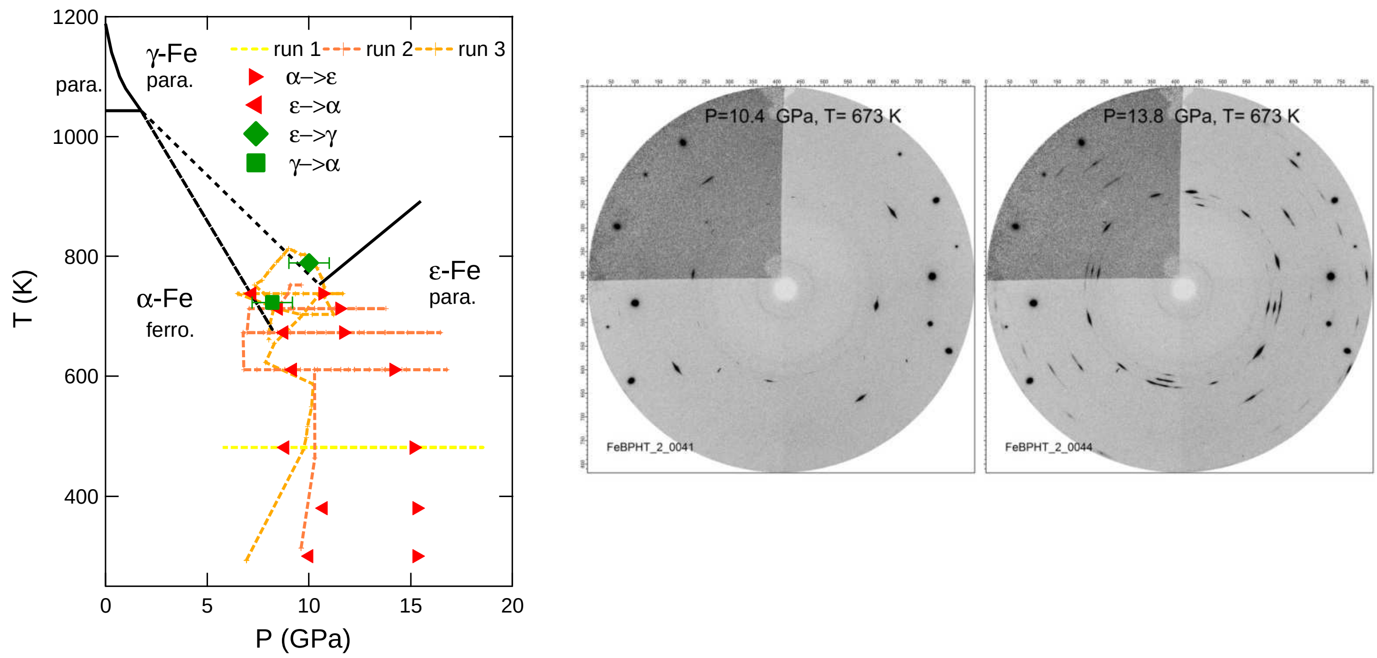

Figure 1: (left) P-T paths followed for the three runs (dashed coloured lines) and location of phase changes (symbols, see the legend). The transition lines found in the literature are plotted as black lines. **(middle)** One X-ray diffraction pattern recorded in the stability domain of α-Fe (rotation of the diamond anvil cell by ±13°). **(right)** Pattern taken in the stability domain of ε-Fe. The round (elliptical) spots correspond to x-ray diffraction peaks of diamond anvils (Fe sample). The orientation relations between α-Fe and ε-Fe are characteristic of a Burgers path.

References:

[1] J.M. Besson and M. Nicol, An EOS of γ-Fe and magnetoelastic effects on measurements on the α–ε–γ triple point, J. Geophys. Res. 95, B13, 21717, 1990; S. Klotz et al., Elastic properties of α-Fe at HT by HP neutron scattering, J. Phys. D: Appl. Phys. 44, 055406, 2011

[2] A. Dewaele, C. Denoual, S. Anzellini, F. Occelli, M. Mezouar, P. Cordier, S. Merkel, M. Véron and E. Rausch, Mechanism of the alpha-epsilon transformation in iron, Phys. Rev. B 91, 174105, 2015