ESRF	**Experiment title:** Mechanism of the pressure-induced body centered cubic to hexagonal close packed transition of iron	**Experiment number**: HS-4605
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Report:

The aim of this experiment was to further elucidate the mechanism of the α-Fe->ε-Fe phase transition which takes place around 13 GPa at ambient temperature. A few mechanisms have been proposed for this martensitic transformation [1]. The microstructure in recovered samples after shock compression are consistent with the Burger path [1], with no variants selection. In-situ X-ray diffraction (XRD) measurements on well controlled samples and experimental conditions (eg single crystals under hydrostatic conditions) are needed to confirm and better understand this mechanism.

Four diamond anvil cells have been loaded each with one α-Fe single crystal, embedded in a soft pressure transmitting medium (helium or neon). For each cell (see Table 1), the α-Fe/ε-Fe forward and reverse transition has been followed two or three times by cycling the pressure between 5 GPa and 20 GPa, at ambient or near ambient temperature.

The phase fraction, based on XRD relative intensities, is represented in Fig. 1 as a function of pressure. The coexistence domain of α-Fe and ε-Fe is much narrower than under non-hydrostatic compression [2]. The transition pressure is 12.5 GPa.

XRD spectra of the initial α-Fe sample (a), the child ε-Fe (b) and the recovered α-Fe (c) after one pressure cycle are presented in Fig. 2. These spectra have been interpreted in terms of sample texture and orientation. The initial single crystal (a) transforms into a powder (b) which exhibits strong preferred orientations. These orientations are consistent with the 6 variants predicted by the Burgers mechanims for bcc to hcp phase transformation. This powder is thus likely to be a mixture of single crystals, which have been formed from the initial single crystal, with the 6 possible orientations allowed by the Burgers path. Surprisingly, the α-Fe sample obtained after reversion (c) is a single crystal with the same orientation as the initial sample (although with a larger mosaicity that the initial sample). This was unexpected because the number of variants allowed by the Burgers path was 13.

Cell name	Pressure medium	P range (GPa)	T range (K)
CDMX17	Ne	5-20	300
CDMX8	He	5-20	380
CDMX131	He	5-20	300
CDMTHT3	He	5-20	300

Table 1: conditions of each experimental run.

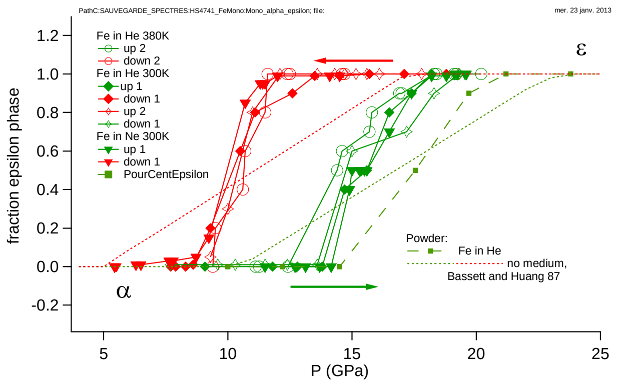

Fig. 1: fraction of ε-Fe as a function of pressure measured on pressure increase (in green) and pressure decrease (in red).

(a) (b) (c)

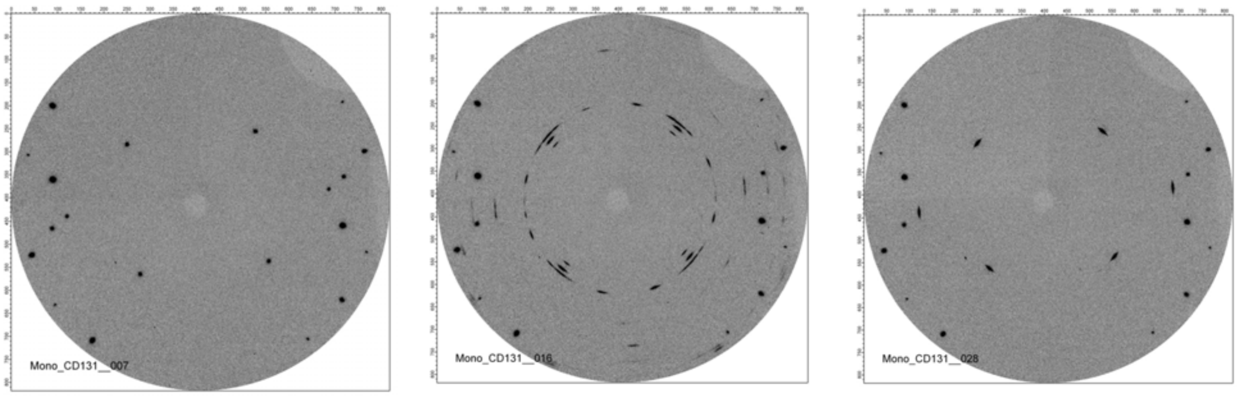

Fig. 2: XRD patterns taken (a) at 5 GPa, (b) at 20 GPa, (c) at 5 GPa after one pressure cycle. The round spots which are identical on Figs (a), (b) and (c) correspond to the XRD peaks of the diamond anvils. The remaining peaks are from the Fe sample.

The reason for the selection of variants on the reverse transformation may be due to geometrical constraints (grains shape) or stresses which prevent the system from exploring all energy minima offered by the transformation. Microscopic texture analysis on recovered samples as well as mesoscopic calculations are planned in order to better understand this phenomenon.

No important elastic strains have been measured in α-Fe and ε-Fe in the coexistence domain. This observation is different from what has been reported for a powder compressed with no medium [2]. It thus appears that the results reported in Ref. [2] were affected by non-hydrostatic pressurizing conditions.

[1] S. J. Wang *et al.*, Microstructural fingerprints of phase transitions in shock-loaded iron, Sci. Rep. 3, 1086 (2013)

[2] W. A. Bassett and E. Huang, Mechanism of the body-centered cubic-hexagonal close-packed phase transition in iron, Science 238, 780 (1987).