ESRF	**Experiment title:** Twins and dislocations in hcp-Fe at high pressure	**Experiment number**: HS-4089
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Report:

In this run, we wanted to study the plastic properties of the α and the ε phases of iron under high pressure and identify twins and dislocations within the sample. To this aim, individual grains had to be extracted from the polycrystalline diffraction patterns using 3D-XRD methods. High resolution diffraction spots are then used for obtaining the substructure of those grains.

Two samples were studied. In both cases, the sample was a pure Fe powder, along with platinum and ruby as pressure markers. For sample 1, we used a fairly hydrostatic pressure medium (methanol-ethanol). For sample 2, we chose to enhance deviatoric stresses by using SiO_2 powder as pressure medium.

For all measurements, an area detector was used in a close (105 mm) and a far (500 and 550 mm) distances from the specimen. In the close detector position, the diffraction patterns were collected in steps of $\Delta\omega = 0.2°$, 0.3° or 0.4° with an accumulation time of 15 to 30 seconds. In far position, diffraction patterns were recorded in 4x4 grids with different $\Delta\omega$ steps and accumulation time depending on the quality of the data and time constraints.

Sample 1 was studied at 16 GPa, at the α-ε transition, and above the transition at 22 GPa, 30 GPa, and 34 GPa. In this data, many spots originating from the α or ε phase can be identified. In addition, unexpected spots have been found at 16 GPa that seem to originate from an intermediary fcc structure. This will be investigated in details during data processing.

Sample 2 was first studied at 5.5 GPa. At this pressure, we realized that the grains were very small and could not be indexed. We therefore performed in-situ laser heating in order to promote grain growth and repeated the close detector data collection. Sample was then studied at about 6 GPa, before the α-ε transition, 14 GPa,

at the transition, and 25 GPa, above the transition. At this pressure, we performed another run of in-situ laser heating to promote grain growth and collected a final set of data at 40 GPa.

Figure 1 shows diffraction spots extracted for sample 1 with the detector in close position for one ω angle at 22 GPa. Spots originating from the ε phase can be identified. In addition, unexpected spots have been found that seem to originate from an intermediary fcc structure. Figure 2 shows a typical high resolution image for a 002 diffraction spot of ε-Fe at 25 GPa. If the grain indexation is successful, the peak broadening in the patterns of the far detector position data, will be used to study dislocations in this phase. This could give many important information about the ε phase of Fe since nothing is known about dislocations in this material.

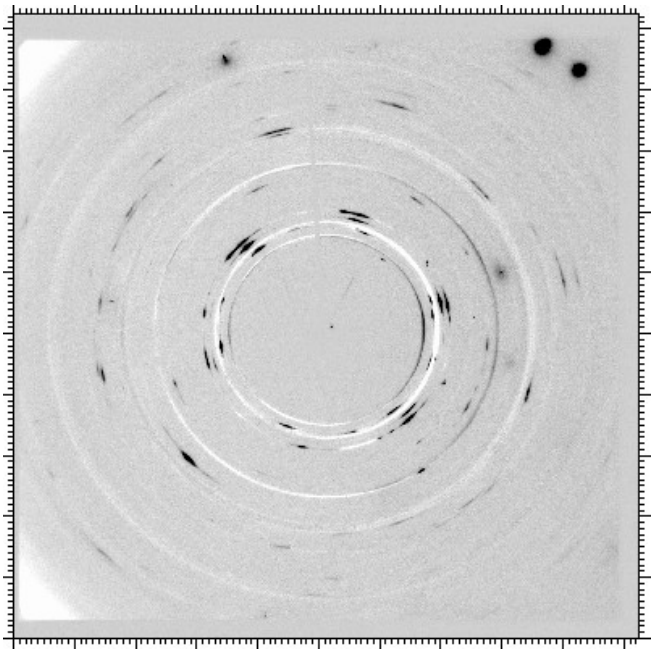

Figure 1: Diffraction spots of ε-Fe and the unknown phase extracted from an image at one ω angle and 22 GPa.

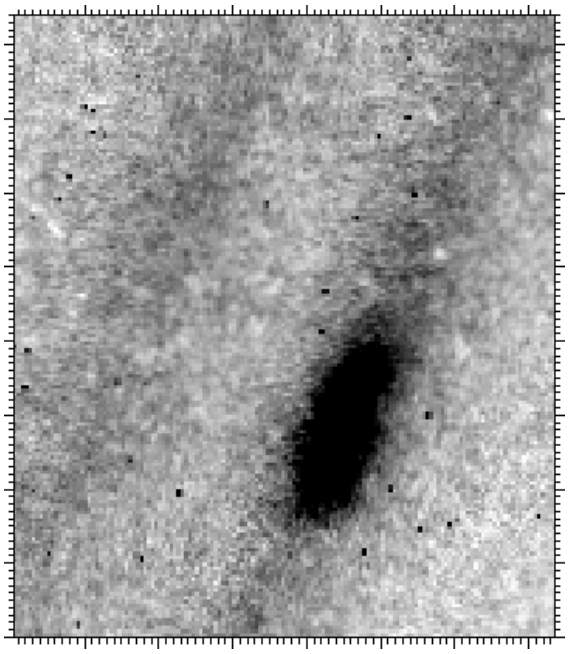

Figure 2: High resolution image of a 002 diffraction spot of ε-Fe at 25 GPa.