ESRF	**Experiment title:** Effect of shear deformation on the high pressure behavior of Si, GaSb, and Fe	**Experiment number**: HS-4199
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In this project, we planed to investigate influence of large shear deformations on phase transitions, stress, and development of texture in single-phase powder aggregates of Fe, Si, and GaSb submitted to high pressures. The deformations were generated using a unique diamond anvil cell with rotating anvils (SDAC) where one of the anvils can be rotated through 360º (Figure 1)

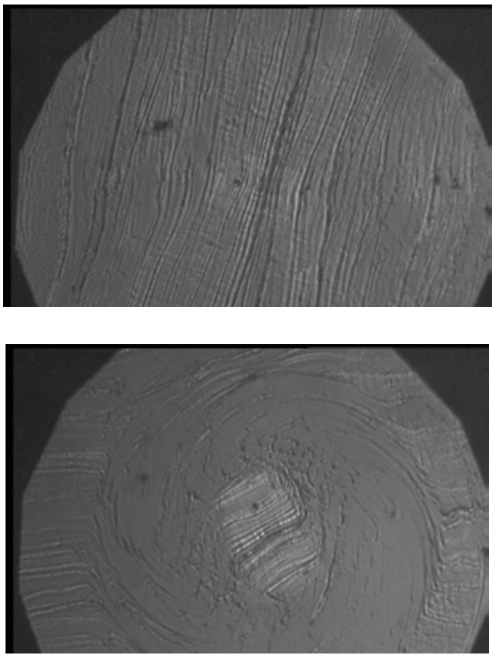

Figure 1. Photographs of the Fe-sample before and after shear deformation (by ~70º) in the SDAC.

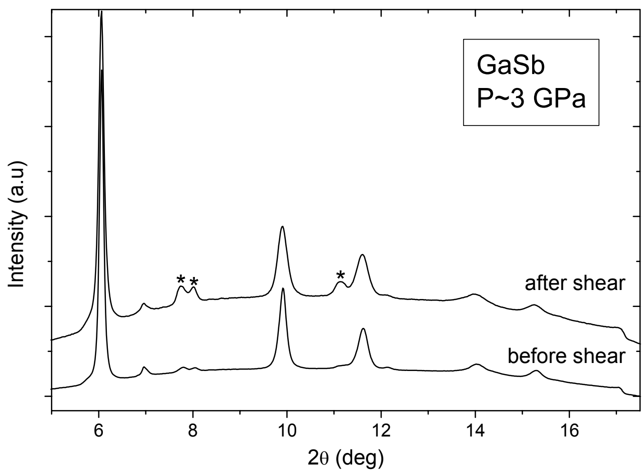

Figure 2. X-ray powder diffraction patterns of the GaSb sample measured at the distance of ~70 μm from the sample center. In the XRD pattern obtained before shear deformation the peaks of the HP metallic phase (indicated by asterisks) are much weaker than in the pattern measured after the deformation by ~20º.

We succeeded to examine the influence of large shear deformations on the semiconductor-to-metal phase transition in GaSb, both on compression and decompression. Initially, the results obtained for GaSb were planned to be used only as a reference in experiments on Si which shows, in addition to the semiconductor-to-metal phase transition, an intermediate phase (the so-called Kasper phase, Si-III). We have found, however, that the nature of the direct (upon pressure increase) and reverse transitions in GaSb are more complicated than initially expected because the type and amount of the transition products depend not only on the pressure or degree of deformation but also on time. This observation resulted in a significant extension of the experiments on GaSb at expense of the beam-time planned for the experiments on Si.

The powder of GaSb was compressed between the diamond anvils and XRD patterns were measured across the sample in 10 µm steps, before and after shear deformation. The step size was comparable with the lateral resolution of our XRD measurements. An example of the diffraction patterns collected before and after the deformation, at the distance of 70 µm from the sample center, is shown in Figure 2. From the Figure follows that the amount of the high pressure phase increased after the shear deformation even when the total load applied to the diamond anvils remained constant.

We also continued experiments on texture, stress, and a phase transition in iron subjected to large plastic deformations at high-pressures which we have started in 2009. Five samples of Fe were studied in the shear DAC. For all, and at each step in pressure or strain, data were collected every 20 µm across the diamond tip. This data will be used to study the variations of pressure, phase proportions, grain size, stress, and textures with distance from the rotation center as a function of pressure and applied strain.

Samples 1, 2, 3, and 5 were studied at pressure below 12 GPa in the sample center. In those cases, the sample was compressed to the target pressure, in multiple steps, and shear was applied to investigate the influence of plastic deformation on the bcc to hcp transitions in Fe. In those 4 cases, the bcc to hcp was not observed.

Sample 4 was compressed up to a pressure of about 16 GPa in the sample center. In this case, the hcp phase of Fe was observed at the center of the sample, where peak pressure is also located. This sample was then deformed by applying 3 subsequent steps of shear deformation at constant load. Figure 3, for instance, presents the pressure deduced from the measured unit cell parameters of α-Fe as a function of distance from the rotation center and applied shear. Figure 4 shows the coherent scattering domain size deduced from our XRD measurements as a function of applied shear. The full analysis of this dataset will be used for the study of the behavior of materials in the shear DAC.

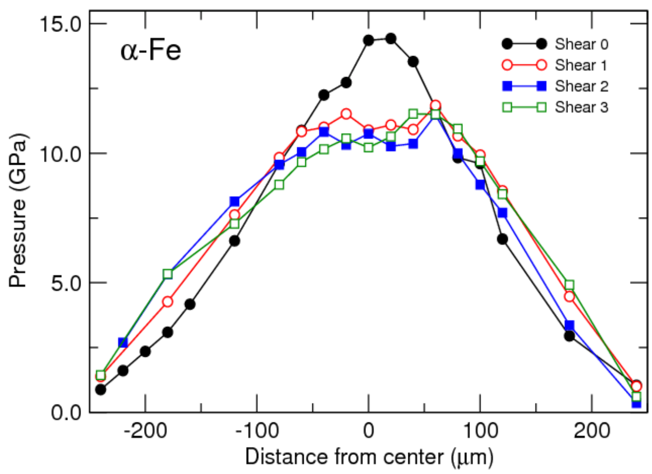

Figure 3. Evolution of the pressure distribution in the Fe sample as a function of the applied shear deformation.

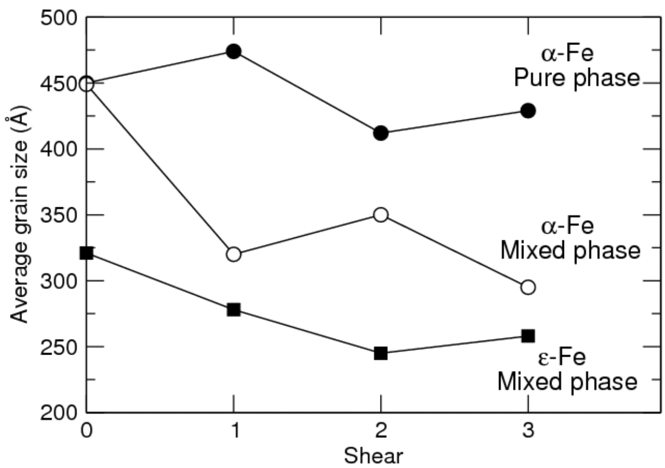

Figure 4. Evolution of the grain size of the α- and ε-phases of Fe in the pure α-phase- and in the two phase regions as a function of the applied shear deformation.