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Report:

We report on the first high resolution X-ray diffraction (XRD) measurements aiming investigation of influence of large shear/plastic deformation on phase transitions, stress, and development of texture in single-phase aggregates submitted to high pressures. We used a prototype of a diamond anvil cell with rotating anvils, SDAC (Fig. 1), which allowed to plastically deform solids at pressures to 50 GPa [Blank and Zerr, 1992]. In the present experiments we studied the influence of shear on texture, stress, and a phase transition in iron and on the development of texture and stress in MgO plastically deformed in shear at pressures to about 30 GPa. For the XRD measurements on the beam-line ID09A we used a monochromatic radiation with the wavelength of $\lambda = 0.3654$ Å collimated to a spot of about 15 μm in diameter. XRD patterns were collected in the axial geometry where the X-ray beam propagated parallel to the rotational axis of the SDAC (Fig. 1). After each pressure increase and each shear deformation, the XRD patterns were measured from one sample edge to the opposite one, in two perpendicular directions in steps of 20-30 μm. This procedure allowed us to map the deformation state and, in the case of iron, phase distribution in the sample. For both substances we used powder samples.

In experiments on iron two surprising observations were done after shear deformation. The first unexpected result was an unusual pressure distribution in the sample with the maximal pressure on the sample periphery instead of in the center (Figure 2). To our knowledge, such form of pressure distribution was not reported in the literature. As a consequence of this pressure distribution, the high-pressure ε-phase of iron formed first on the sample periphery (Figure 2). Further shear deformation led to an expansion of the ε-phase region from the periphery to the sample center. The second unexpected result was a drastic increase of the stress state in the sample after shear deformation, even for the low pressure α-phase: For the chosen diffraction geometry, we did not observe any significant distortion of the Debye circles (e.g. variation of the *d*-values as a function the azimuthal angle, φ) (Fig. 3A). However, after a shear deformation via the anvil rotation to the total angle of about 37°, an unexpectedly large distortion of the Debye circles resulting in the *d*-values variation of

(d_{max}-d_{min})/d_{max} = 0.02, was observed for α-Fe. Such *d*-values variation corresponds to the pressure/stress contrast around 100%. We are not aware of reports on similar stress contrasts in α- or ε-phase of iron upon uniaxial compression, even if the XRD patterns were measured at comparable average pressures in the radial geometry. It should be clarified in the future work whether the observed effects are due to the intrinsic material behavior or is dependent on the nature of the starting material (powder vs. foil).

In the next set of experiments, we used a sample of pure MgO in order to characterize the stress and deformation applied to the sample. MgO is a simple material, with no phase transition in the P/T range of interrest and its elastic properties are known. Plastic properties of polycrystalline MgO have also been extensively studied in the past. It is therefore an ideal candidate for the characterization of the stress and strain state in the shear DAC.
Two pure polycrystalline MgO samples were studied, using 300 microns diameters flat diamond anvils, and a steel gasket.

- First sample was 140 µm in diameter and located at the center of the anvil. Pressure was increased to 5, 12, and 21 GPa. At each pressure, the sample was submitted to shear by rotating the top anvil to about 15° and back in multiple steps. After each deformation step, stress and strain distribution were mapped by performing XRD measurements at multiple locations in the sample.
- In order to increase the amount of plastic shear applied to MgO, second sample was 50 µm in diameter and placed off-center. Pressure was increased to about 14 GPa. At this pressure, shear was applied to sample multiple times and in multiple directions. After each shear event, stress and strain distribution were mapped by performing x-ray diffraction at multiple locations in the sample.

The XRD data we have collected are now under processing with the aim to characterize the state of stress and strain in the sample. Peak shifts and distortions of the Debye circles will be used to estimate stresses, and diffraction intensities to deduce lattice preferred orientation and characterize the plastic strain applied to the sample.

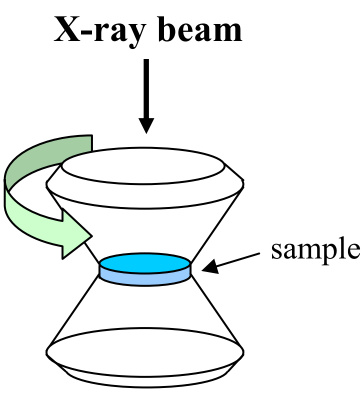

Figure 1. Schematics of an apparatus with rotating anvils allowing very large shear deformations at high pressures.

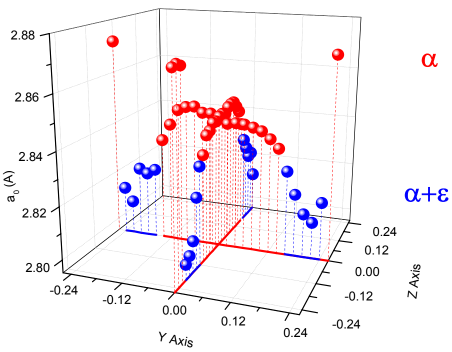

Figure 2 Averadged lattice parameter of α-Fe (corresponding to the average pressure in the particular part of the sample volume) and the phase distribution in the sheared Fe-sample.

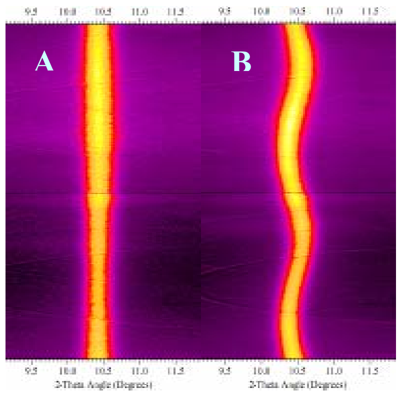

Figure 3 Examples of the diffraction angle, 2θ, variation and, accordingly, of the *d*-values as a function of φ for the reflex (110) of α-Fe: **A** – before and **B** – after shear deformation to the total angle of about 37 °.

References:

Blank V. D., Zerr A. Ju., *High Pressure Res.* **8**, 567-572 (1992)