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ESRF	**Experiment title**: Single crystal x-ray diffraction of solid oxygen up to 200 GPa: structure of the metallic phase and search for dissociation.	**Experiment number**: HS2135
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Report:

The aim of the experiment was to study the structure of the molecular metallic phase of oxygen (ζ-O_2, above 96 GPa) and to search for a possible transition towards an atomic solid. Several experiments have already been done in order to determine the structure of the metallic phase of oxygen. First, Akahama et al. [1] have performed angular dispersive X-ray diffraction on powder of pure oxygen and concluded to an isostructural phase transition at the metallization; the technique used, however, provided limited data at very high pressure due to the low scattering power of oxygen. Moreover, because of the non-hydrostatic condition, the X-ray diffraction peaks recorded were very broad at high pressure and the diffraction patterns were thus difficult to refine. We have carried out energy dispersive X-ray diffraction on oxygen single crystals in helium [2]. The experiments gave access to the orientation matrix of the crystal avoiding any erroneous indexation. The X-ray diffraction peaks from single crystals were intense and sharp even at high pressure. In those experiments, we have highlighted a displacive transition at the metallization; however, only 4 peaks were still present after the transition and we could not refine the structure of the metallic phase. Under the current project, we have performed angular dispersive X-ray diffraction on oxygen single crystals using with helium as a pressure transmitting medium. We used an image plate detector.

Experimental method

Three membrane diamond anvil cells with high X-ray aperture ($2\theta_{max}$ = +/- 37 °, φ = 360°) were loaded with oxygen-helium mixtures at ambient temperature and high pressure (1000 bar). For each sample, a single-crystal of pure oxygen was grown in the middle of the compression volume. The typical size of the sample and the single crystal was 20 μm and 7 μm in lateral dimensions. The optical properties of the oxygen epsilon phase allowed us to choose different orientations for each single crystal. Pressure was measured using the ruby pressure calibrant. We performed angular dispersive X-ray diffraction with a monochromatic beam of energy E = 33 keV at the ID30 beamline. The X-ray diffraction images were collected with an on-line image plate detector (MAR3450). The beam was focussed down to 15 μm in diameter.

Results

X-ray diffraction experiments were performed at ambient temperature for different pressures up to 143 GPa. Despite of the low X-ray diffraction power of oxygen, we could obtain well defined X-ray peaks. Nevertheless, we were not able to avoid X-ray diffraction arising from the gasket material containing the sample, due to the actual beam spot size. At very high pressure this problem became critical since diffraction rings from the gasket broadened strongly and overcame the weaker X-ray peaks of the sample.

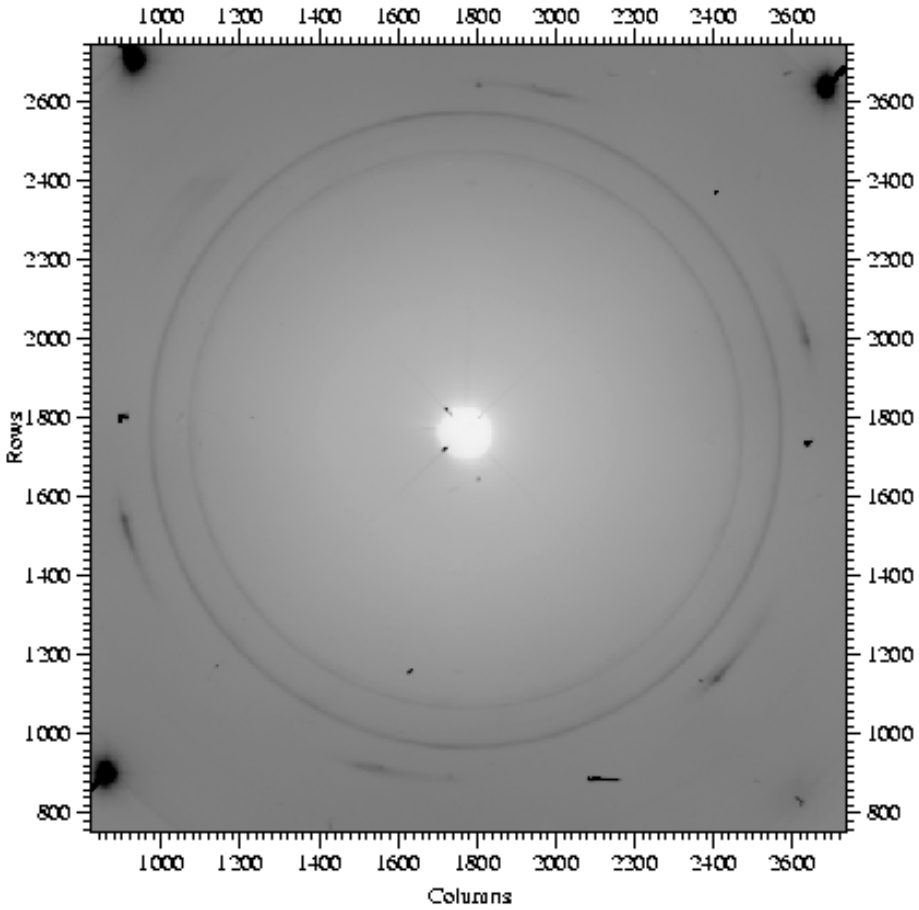

Figure 1: Panoramic X-ray diffraction patten of solid oxygen at 143 GPa

We have confirmed the displacive nature of the transition to the metallic phase already observed in the energy dispersive X-ray diffraction experiments. In Figure 1, we present the X-ray diffraction image of solid oxygen at 143 GPa. This X-ray diffraction images was obtained after the realignment of the X-ray beam. Due to the non-hydrostatic conditions and the displacive phase transition, the single crystal is strongly damaged (strained). The X-ray diffraction image seems to indicate a hexagonal symmetry for solid oxygen in the metallic phase. The different d-spacings could be measured but the peak positions in the reciprocal space were difficult to refine. Hence we could not determine an orientation matrix. We believe that the oxygen single crystal must be annealed in the metallic phase in order to remove internal strains and help to

eventually solve the structure. In our next experiments, we plan to grow single crystals of oxygen in helium and anneal them by *in-situ* laser heating prior to recording X-ray diffraction images.

References

[2] Y. Akahama *et al.*, Phys. Rev. Lett. **74**, 4690 (1995)
[3] G. Weck , P. Loubeyre, R. LeToullec, Phys. Rev. Lett. **88**, 035504 (2002)