	Experiment title: Single crystal x-ray diffraction of solid oxygen up to above 250 GPa: equation of state of the metallic phase and search for the atomic phase.	**Experiment number**: HS4077
Beamline: ID27	**Date of experiment**: from: 18 June 2010 to: 22 June 2010	**Date of report**: 25/08/2010
Shifts: 12	**Local contact(s)**: Gaston Garbarino	*Received at ESRF:*

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Report:

Above 10 GPa, solid oxygen transforms into the dark-red ε-phase, stable up to 96 GPa, above which pressure it transforms into a molecular metal, the ζ-phase. The structures of these two phases have been determined only recently by single-crystal x-ray diffraction. In each case, the structural determination has been essential to reveal an intriguing physics. A molecular association of 4 O_2 molecules into O_8 entities has been observed in the ε-phase[1, 2]. The mechanism of the insulator-metal phase transition has been disclosed [3]: the metallization of solid O_2 by band gap closure at 96 GPa causes an instability of the lattice that evolves through a displacive phase transition into an isosymmetric structure with the dissociation of O_8 entities (connecting along the b direction). The aim of this proposal was to pursue the single-crystal x-ray measurements in the metallic phase up to 300 GPa in order to extend the determination of the equation of state and to observe, if any, the pressure induced dissociation of the O_2 molecule.

Experimental method

Two membrane diamond anvil cells were equipped with diamond anvils with culet size of respectively 50 microns and 70 microns for this experiment. The cells were loaded with an oxygen-helium mixture (1.6 mol% of oxygen) at ambient temperature and high pressure (1000 bar). The fraction of oxygen in the mixture was significantly reduce in comparison with experiment HS2757 that was limited to 1.4 Mbar. In both case, a single-crystal of pure oxygen was grown at 100° C in the middle of the compression volume. A total of 20 loading was necessary to prepare the two samples. The typical size of the sample and the single crystal was 15 μm and 8 μm in lateral dimensions. Pressure was measured using the ruby and gold pressure calibrants. We performed angular dispersive X-ray diffraction with a monochromatic beam of energy E = 33 keV at the ID27 beamline. The X-ray diffraction images were collected with an on-line image plate detector (MARCCD). The beam was focussed down to 4 μm in diameter.

Results

In the first cell equipped with the 50 microns culet anvils, the sample had rapidly diffused into the gasket. We suspect a chemical reaction between the gasket and the oxygen during the crystal growth at high temperature. This problem can be avoided by doing gold deposition on the gasket surface. In the second cell, equipped with the 70 microns anvils, the X-ray diffraction measurements were performed at ambient temperature for different pressures up to 190 GPa, pressure at which, one of the anvil failed. Despite the very weak initial oxygen concentration, the crystal diffraction peaks were observed up to the maximum pressure (see figure 1). Thanks to the microfocussed beam of ID27, we were able to avoid most of the X-ray diffraction arising from the gasket material containing the sample. The present experiment confirmed the displacive structural transformation observed at the metallization at 96 GPa. As already reported, the transition significantly damage the crystal. Future study will require annealing of the crystal by laser heating. In the current status of the analysis, no additional transformation is observed up to 190 GPa.

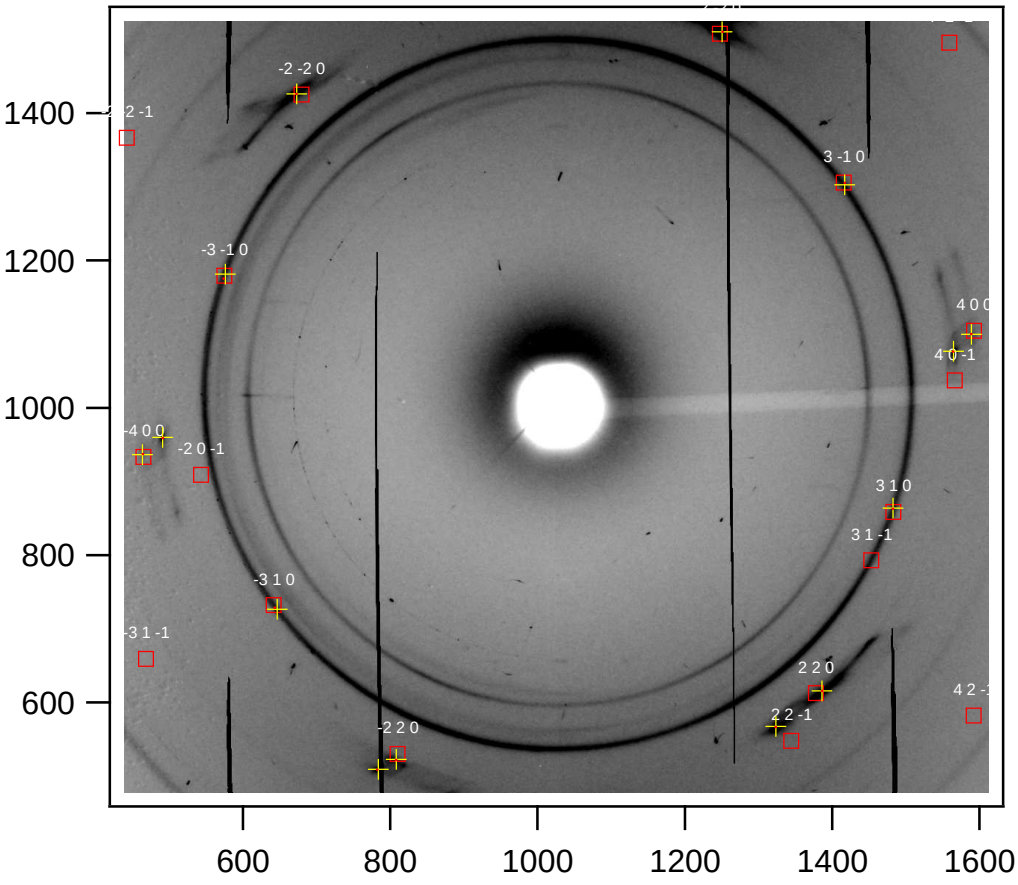

Figure 1: Panoramic X-ray diffraction pattern of oxygen at 187 GPa overlaid with the predicted (red square) and the observed (yellow cross) diffraction peaks.

References

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[3] G. Weck, P. Loubeyre, S. Desgreniers and M. Mezouar, Phys. Rev. Lett., **1002**, 255503, 2009 [3] L. F. Lundegaard, G. Weck, M. I. McMahon S. Desgreniers and P. Loubeyre *Nature* 443, 201 - 204 (2006)