ESRF	**Experiment title:** High Resolution X-ray Diffraction of Dense Solid Oxygen	**Experiment number:** HS-334
Beamline: ID30	**Date of experiment:** from: Nov. 26 to: Nov. 28,1997	**Date of report:** Feb. 15,1998
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Report:

Solid oxygen has been the object of numerous studies as it constitutes an excellent "laboratory" for the study of molecular magnetic interactions in the solid state. In fact, magnetic interactions give rise to a rich phase diagram comprising several crystalline structures. In using the intense X-ray beam at experimental line ID30 of ESRF, we aimed our effort at assessing several crystalline structures of solid oxygen for which complete structural parameters have not been determined. The goals of the experiment were as follows:

1. Record, using angle-dispersive X-ray diffraction with imaging detectors, the best powder patterns of ε-O_2 at relatively low pressure. The new ESRF data were to be combined with previously recorded synchrotron data for a full Rietveld refinement of the atomic positions.
2. Record patterns of ε-O_2 in its pressure stability field, i.e., from 12 GPa to 96 GPa at 300 K;
3. Record powder diffraction patterns across the ε-O_2 / ξ-O2 transition to gain more information regarding the structural transformation at the insulator-metallic transition.

All angle-dispersive X-ray diffraction patterns were recorded using imaging plates from powdered samples of solid oxygen grown under pressure in diamond anvil high pressure cells. Samples were kept close to room temperature and were not annealed following

pressure increases. All pressures were measured by $Al_2O_3:Cr^{3+}$ luminescence. Excellent angle-dispersive X-ray diffraction patterns were recorded from a sample contained in a large angular X-ray aperture diamond anvil cell which was fully rotated about the X-ray beam axis (x-rotation) while rocked by a total of 20" about the vertical axis (o-rotation) during the imaging plate exposures. A resulting diffraction intensity profile is shown in Figure 1. Refined lattice parameters of the ε-O_2 phase (A/2m, Z=8 molecules), at 13.5 GPa and 300K, are in agreement with previous results [1, 2, 3]. Rietveld refinements are still being carried out to take into account possible texture effects. Refinement of the atomic positions, a first for the ε -phase of solid oxygen, will most likely be possible with the new ESRF data sets [4]. Analysis of diffraction patterns recorded at pressure between 8 -10 GPa are also underway to assess the possible existence of the postulated but not well-documented ω- and χ- phases.

X-ray diffraction patterns of the ε-O_2 phase were also recorded up to 60 GPa. We did not succeed in recording diffraction from the pressure-grown samples at higher pressures due to weak diffraction . Weak diffraction intensities were most likely the result of very small diffracting volumes, necessary to generate pressures in excess of 100 GPa as planned, and the use of less sensitive imaging plates. Unfortunately during our beam time, the more sensitive planar detector FASTSCAN II was not functioning. If this had not been the case, we would have had a better chance to reach higher pressures on the prepared samples and to make an optimal use of the beam time scheduled.

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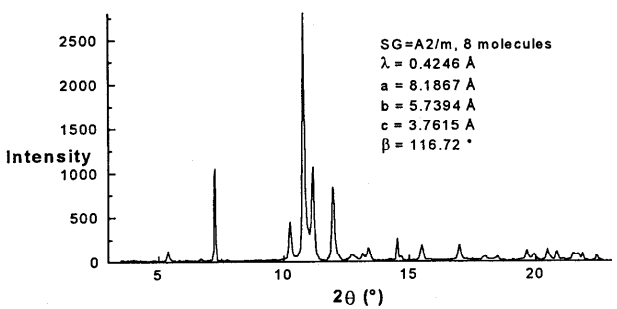

Figure 1. X-ray diffraction pattern of ε-O_2 recorded at 13.5 GPa and 300K at line ID 30. Insert indicates the refined lattice parameters.