ESRF	**Experiment title:** High Resolution X-ray Diffraction of Dense Solid Oxygen	**Experiment number:** HS-741
Beamline: ID30	**Date of experiment:** from: 19-nov-98 to: 24-nov-1999	**Date of report:** August 20, 1999
Shifts: 12	**Local contact(s):** T. Le Bihan	*Received at ESRF:* 30 AOUT 1999

Names and affiliations of applicants (* indicates experimentalists):

* Dr. Serge Desgreniers, Université d'Ottawa, Département de physique, Ottawa, Canada

* G. Weck, CEA, DIF/DPTA/PTIC, Brugère le Chatel, France 91680

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, in order to do structure refinements;
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.
4. Record powder X-ray diffraction patterns of polycrystalline oxygen sample grown in He and Ne.

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 samples contained in a large angular X-ray aperture diamond anvil cell which was fully rotated about the X-ray beam axis (χ-rotation) while rocked by a total of 20° about the vertical axis (ω-rotation) during the imaging plate exposures. 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].

X-ray diffraction patterns of the ε-O_2 phase were also recorded up to 118 GPa, past the insulator-metal transition which occurs at 96 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. We were not successful in obtaining more crystallographic information of the metallic phase. We will have to increase the diffracting volume. This will be the aim of our future attempt.

We have also recorded X-ray diffraction from polycrystalline oxygen grown in He and Ne. (samples prepared by P. Loubeyre at the CEA, France). This constituted our very first attempt to record X-ray diffraction patterns for oxygen under good hydrostatic conditions. Unfortunately, the oxygen/noble gas ratio turned out to be unfavorable. Yet beam time at ID30 has be crucial in identifying all the problems with our first sample preparations and in deciding what to try next to succeed.

It was not possible to use the ESRF laser for sample annealing. Annealing is important for obtaining the best quality X-ray diffraction data for structure refinement. We hope to do it next time.

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