ESRF	**Experiment title:Single crystal x-ray diffraction of solid oxygen: EOS, structure of the metallic phase and search for dissociation**	**Experiment number:** HS493
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Report:

The insulator-metal phase transition in solid oxygen was first reported at 96 GPa from reflectivity studies [l]. More recently, this metallic state was reported to be superconductor at 0.6 K [2]. The two important properties of this metal that remain unknown are its structure and whether it is a molecular or an atomic metal. A recent angle-dispersive x-ray diffraction study at the ESRF has proposed a structural phase transition within the same monoclinic lattice (space group A2/m) high pressure phase [3]. Nevertheless, this result was speculative due to the small number of diffraction lines observed. On the other hand, a recent ab-initio calculation has proposed a displacive phase transition from a monoclinic to an orthorombic structure **[4]**.

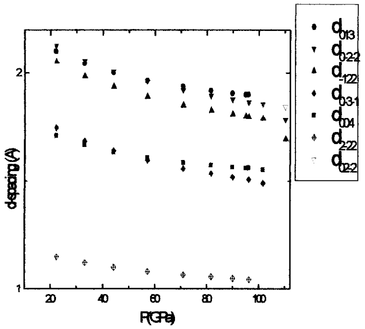
We have performed very accurate structural measurements of solid 02 with the single crystal x-ray technique in the energy dispersive mode up to 115 GPa. The single-crystal of ε-O_2 was embbeded in helium so as to reduce its fragmentation and to assure a quasi-hydrostatic compression (complete immiscibility of He and 02 was exploited in a mixture containing 2.6% of oxygen in helium). Pressure was generated in a membrane diamond anvil cell with a 76” x-ray aperture on both sides. Beveled diamonds with culets of 100

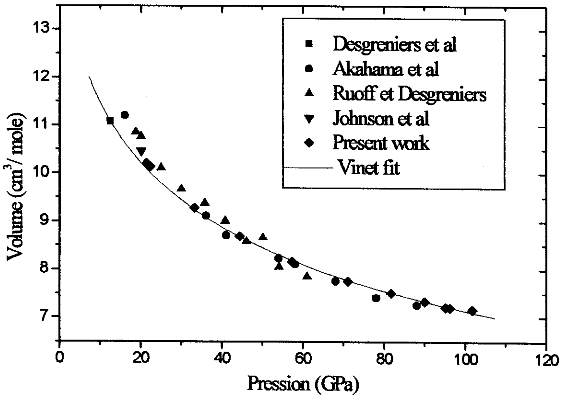
μm and 150 μm were used to go in the 100 GPa range and the single crystal of oxygen was typically of 15 μm diameter. The pressure was determined both with the $SrB_4O_7:Sm^{2+}$ luminescence gauge and with the x-ray determination of the volume of Au.

Six classes of reflexions were followed to 96 GPa (see figure 1). Up to 96 GPa, these reflexions were well-related with the A2/m orientation matrix. However, a smaller compressibility than previously reported was measured. A reason for this discrepancy could be the crossing of d-spacings, as seen in figure 1, that was not observed in the ADX study. At 96 GPa, the lattice parameters show a trend to a structural change with an associated change of the intensity of the reflexions. At 110 GPa, most of the diffraction lines have disappeared, and a new one is observed with strong intensity at angle expected for the (02-2). The three remaining lines do not fit with the monoclinic structure, both for the d-spacings and the angles. This is due to the fall in d-spacing values of (O-2-2) and (-122) at 110 GPa, and to the change of their diffraction angles (illustrated in figure 3 by the (02-2) rocking curve at different pressures). In a second experiment, the (100) peak has been followed through the phase transition without any discontinuity in the angles or the energy of the peak. These observations clearly indicate a displacive phase transition with sliding of the [**100]** planes that seems in good agreement with recent ab-initio simulations. Yet, only four peaks were observed that is not enough to refine the structure.

In conclusion, we obtained: - Very accurate V(P) data in the a-phase. -Clear evidence of a displacive phase transition at metallization from a monoclinic to a possible orthorombic structure.

Unfortunately, the diamond anvils broke at 116 GPa. Further experiments are needed.





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