



	Experiment title: Local structure of a new stable xenon oxide under pressure	Experiment number: HC-767
Beamline: BM23	Date of experiment: from: 27/02 to: 05/03/2013	Date of report: 20/02/2014
Shifts: 12	Local contact(s): O. Mathon	<i>Received at ESRF:</i>
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Report:

Proposal Summary : Using laser-heated diamond anvil cell, we have synthesized a new xenon oxide which is stable under high pressure. Its long-range order has been determined by X-ray diffraction. The aim of this proposal is to complete its experimental characterization by X-ray Absorption Spectroscopy (XAS), giving insights into the oxidation state and local structure of xenon atoms.

Experiments:

Two diamond anvil cells equipped with nano-polycrystalline diamond anvils (culets 150x300 microns) have been prepared and loaded with ~40% Xe-60% O₂ gaseous mixture. The use of this material for anvils allows the measurement of X-ray absorption spectroscopy spectra in a wide energy range without any parasitic signal created by diffraction on the anvils, which are usually single crystals [1].

The pressure has been increased to 80 GPa prior to the venue at ESRF. X-ray absorption spectroscopy (XAS) around the Xe K-edge (34.59 keV) has been performed on this starting sample, which indicated a local environment characteristic of a face-centered cubic material (the structure of pure solid xenon under this pressure). This confirmed that the large majority of xenon is phase separated from solid oxygen under high pressure [2].

After this measurement, the sample has been laser-heated off-line and a drastic change of the local environment of xenon atoms has been observed at the center of the heated zone (see figure 1), with an increased edge jump characteristic of an oxidized element.

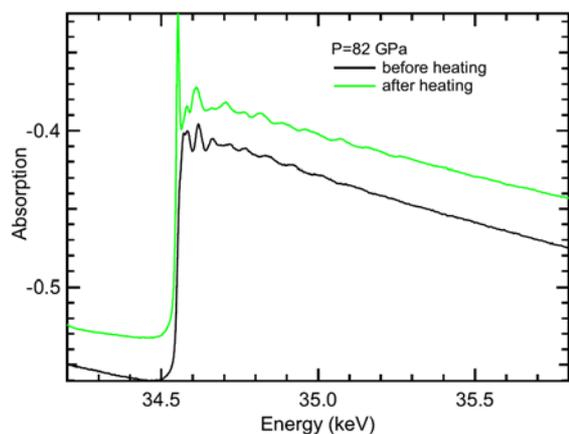


Figure 1: X-ray Absorption spectra of a 36 % Xe - 64 % O₂ mixture around 82 GPa, before and after laser-heating.

The place where the most important part of the sample had reacted has been located by performing a mapping of the edge jump in the high pressure chamber. This was made possible because of the 3x3 microns X-ray spot of the micro-XAS facility on BM23. The normalized edge jump map is plotted on figure 2. X-ray diffraction has been performed at the center of this reacted zone, which showed that the product was identical to the xenon oxide observed before (see proposal HC-767).

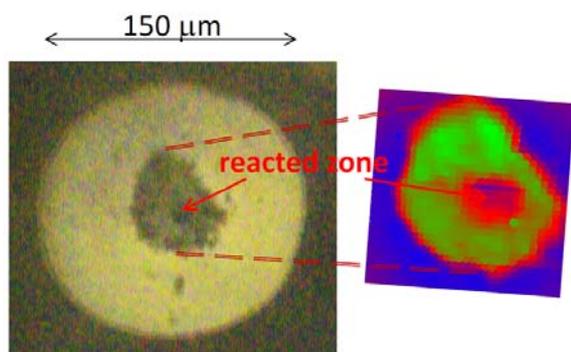


Figure 2: Left : microphotograph of the sample characterized with XAS after laser-heating at 90 GPa. The reacted zone appears in black. Right: mapping of the sample chamber based on XAS measurements. The different colors correspond to different measured values for the absorption just above the Xe K-edge ($E=34.57$ keV). The reacted zone appears in red.

Analysis:

The analysis of XAS spectrum plotted in Figure 1 suggests that each Xe atom has 4-6 oxygen neighbors at a distance of 1.93 angstrom and 2.35 angstrom and ~8 Xe neighbors at a distance of 3.15 angstrom at 82 GPa. Interestingly, this local structure is close, but not identical to the structure proposed for a new oxide predicted to be stable above 100 GPa, P21/C-XeO₂ [3]. The three nearest-neighbours shells coordination numbers suggest that the stoichiometry of the synthesized xenon oxide is close to XeO. Our study thus experimentally confirms the formation of a stable xenon oxide under high pressure, but not the oxide proposed in ref. [3].

References:

- [1] N. Ishimatsu et al., Glitch-free X-ray absorption spectrum under high pressure obtained using nanopolycrystalline diamond anvils, *J. Synchrotron Rad.* 19, 768, 2012
- [2] G. Weck, A. Dewaele and P. Loubeyre, Oxygen/noble gas binary phase diagrams at 296 K and high pressure, *Phys. Rev. B* 82, 0141120, 2010
- [3] Q. Zhu et al., Stable xenon oxides under high pressure, *Nat. Chem* 5, 61, 2013