EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

Experiment Report Form

	Experiment title: Electronic and magnetic properties of unquenchable iron oxides	**Experiment number**: HC-5323
Beamline: ID15B	**Date of experiment**: from: 13.07.2023 to: 15.07.2023	**Date of report**: 30.08.2023
Shifts: 6	**Local contact(s)**: Michael Hanfland	*Received at ESRF:*

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Report:

The experiment focuses on exploring the electronic configurations and magnetic properties of unquenchable novel iron oxides (e.g. Fe_5O_7, $Fe_{25}O_{32}$ and so on), using the Single-Crystal X-ray diffraction (XRD) at ID15B. We have also applied for the Synchrotron Mössbauer Source spectroscopy (SMS) at ID18 in the proposal but the proposal fell just below the cut-off for allocation of beamtime in a highly competitive context on ID18.

During the experiment, we prepared 3 BX-90 type diamond anvil cells with diamonds with 250, 150 and 50 μm culet sizes. For the 250 μm culet cell, FeO, Fe_2O_3 and Fe_3O_4 were loaded as starting materials. We compressed the cell to ~ 54 GPa and laser-heated all three samples above their melting temperature (~ 3200 K). All three starting materials were converted to a mixture of coexisting phases with different relative abundances, namely high-pressure Fe_2O_3, high-pressure Fe_3O_4, Fe_4O_5 and Fe_5O_7. An investigation of the latter was one of the goals of this experiment. The structure details and pressure dependence of the unit cell volume of Fe_5O_7 were determined from the XRD data, measured upon decompression of the cell (Figure 1a-b). We found a discontinuity in the pressure dependence of the unit cell volume of Fe_5O_7 at 47 GPa without any changes in the structure. Thus, the volume drop is most likely related to the spin crossover.

We loaded very thin (~ 2 μm) Fe_2O_3 and Fe_3O_4 samples in the 150 μm culet cell and compressed it to ~ 80 GPa. After heating (~3500 K), both Fe_2O_3 and Fe_3O_4 decomposed to Fe_5O_7 and $Fe_{25}O_{32}$. We were able to solve the structures of both novel oxides at 80 GPa but were not able to trace their pressure change due to the diamond's break.

FeO (1-2 μm) was loaded in the 50 μm culet cell, compressed to 180 GPa and laser-heated at ~ 4000 K. Laser heating resulted in a transition from the B8 phase to a new phase with higher symmetry. The detailed data processing and solving of the new structure is currently in progress.

We successfully synthesized different unquenchable iron oxides. However, the investigation of their electronic and magnetic properties requires additional experiments, particularly utilizing the SMS.

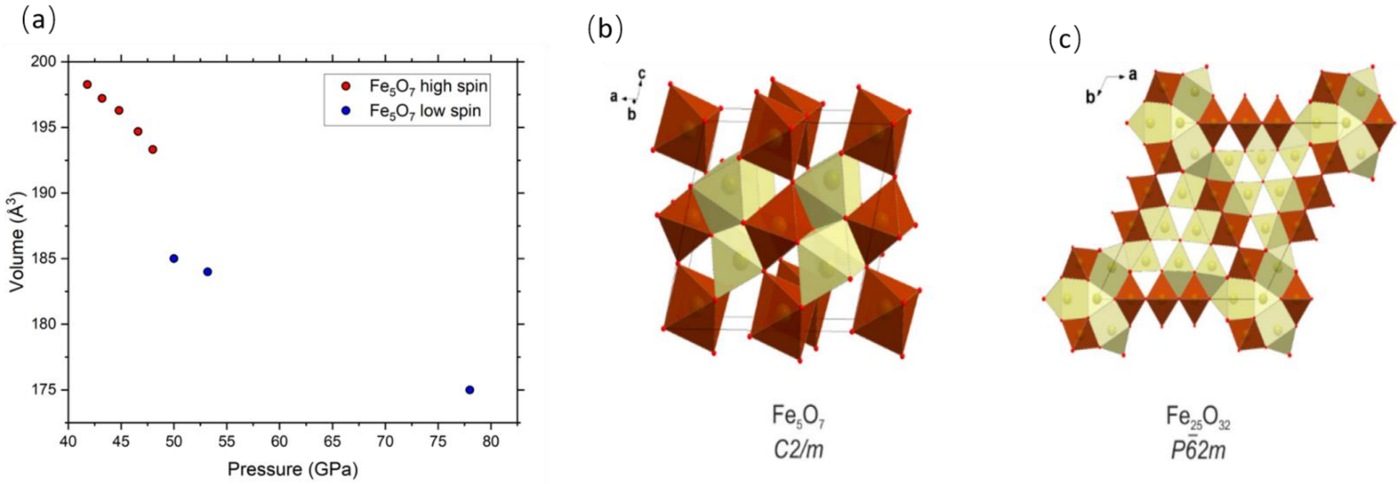

Figure 1. (a) Pressure dependence of the unit cell volume of Fe_5O_7. (b) The structure of Fe_5O_7. (c) The structure of $Fe_{25}O_{32}$. Different kinds of iron polyhedra are shown in different colors: FeO_6 octahedra are shown by brown color; FeO_6 trigonal are designated by citron color.