ESRF	**Experiment title:** **Diffraction studied under extreme conditions of pressure and temperature**	**Experiment number:** HS330
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Shifts: 18	**Local contact(s):** Tristan LeBihan	*Received at ESRF:* 05 MAR. 1998

Names and affiliations of applicants (* indicates experimentalists):

D. Andrault *

G. Fiquet*

A. Dewaele*

T. Charpin*

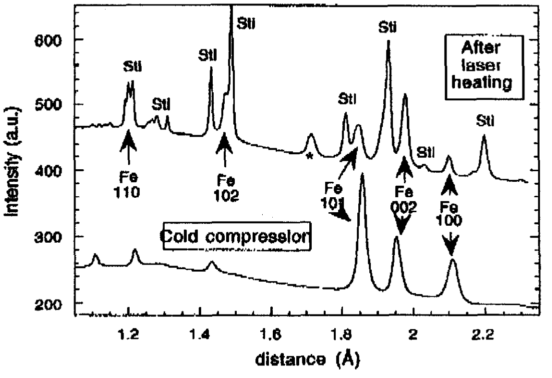
Report:

During this beam-time we have performed 3 different experiments; (1) We extended the PVT equation of state (EOS) of $MgSiO_3$ perovskite up to 86 GPa and to 2700 K (2) We checked the occurrence of the orthorhombic polymorph of iron at high P and T, using either SiO_2 or Al_2O_3 as pressure transmitting medium, and Re or W as gasket material (3) We performed preliminary experiments on PVT-EOS of MgCI periclasc, which can be considered as the second major constituent. of the Earth's lower mantle.

<u>(PVT) equation of state of $MgSiO_3$ perovskite</u>:
The experiments were carried out using a high power YAG multimode laser combined with a diamond-anvil cell. The use of this laser-type allows heating al higher pressure than the previously used CO_2 laser, but the experiments claims for a smaller X-ray beam, compatible in size with that of the hot spot (about 15 µm). We thus reached the extreme conditions of 86 GPa and 2700 K, that is about 2/3 of the maximum pressure in the Earth mantle. The thermoelastic parameters were determined after inversion of this PVT data set, and it represents to date the best experimental constraint to the mineralogical model of the Earth's lower mantle (Fiquet et al., 1998). Our goal was also to check the $MgSiO_3$ perovskite stability that was recently put into question. We found the perovskite stable, and it adopts the same Pbnm orthorhombic lattice at all P and T investigated in this study. The quality of the diffraction pattern even allowed us to carry out Rietveld full structure refinements (see experimental report HS325), which gave for the first time a very detailed structural behavior of this compound.

<u>The orthorhombic structure of iron:</u>
After our publication proposing an orthorhombic lattice for iron at high P and T [Andrault et al., 1997], we encountered large discussions about this structure solution. In particular, we received official comments that somehow asked for more proofs that our results were not related to experimental artifacts. These artifacts could have been due to (i) chemical reaction of our starting material, (ii) occurrence of Re gasket diffraction lines, (iii) deviatoric stress in the pressure chamber, and (iv) large temperature gradient.
We thus took time to reproduced the same orthorhombic lattice using different experimental conditions. We showed that the occurence of the new characteristic orthorhombic diffraction lines is not due to any reaction with pressure transmitting medium or to gasket material, but to iron itself. We also showed that the temperature onto the sample is quite homogeneous with very reduce stress. This was done tracing the diffraction peaks FWHM as a function of pressure and temperature.

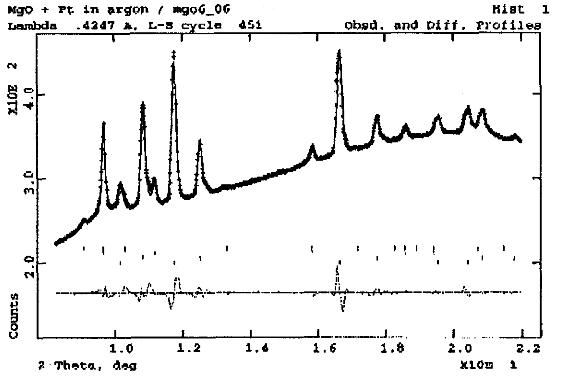
Figure 1: Integrated diffraction patterns of Fe in SiO_2 **pressure** medium, and using a W-gasket at 35 **GPa. Upon** cold compression (bottom spectrum), all e-hcp Iron diffraction lines are visible, and there is no SiO_2-feature because quartz has become amorphous. The top spectrum was recorded after several laser-heating sequences. No oxidation of the iron-sample was encountered, since all diffraction peaks can be indexed as a mixture of iron and stishovite (* stands for metastable g-fcc iron, quenched on cooling).



<u>**Preliminary results**</u> on PVT-EOS of **MgO:**

The are several interests in the study of **periclase** at high P and T, It is the second major constituent of the Earth's lower **mantle,** and thus its PVT-EOS is needed to produce **accurate** mineralogical model for the deep Earth. MgO is **often** used as a reference in various high-pressure **experiments, and thus one has to extend the P and T range in which its physical properties are well known. Finally, the mixing with FeO provides a** useful solid solution to analyse the effect of iron on the elastic behavior of the MgO-rich minerals. X-ray diffraction pattern have thus been recorded in situ at high-pressure and high-temperature, on a mixture of MgO and platinum, loaded cryogenically in argon. Figure 2 shows an example of diffraction pattern, after collection and integration of an imaging plate exposed 120 s at a wavelength of 0.4247 **Å.**

Figure 2: In this experiment, the sample was compressed at 16.7 GPa while heated with a CO, infrared laser at around 1900 K, as determined by the analysis of the sample thermal emission. Such results, however, need to be extended to higher pressures and more investigations arc **needed** to a slightly enriched iron composition, **in** order to identify correctly a **reliable** set of thermoclastic parameters



References:

Andrault et al., Science, 278, 831-834, October 1997

Fiquet et al. , Phys. Earth Planet. Int., in press, 1998