ESRF

ESRF	**Experiment title:** Thermal equation of state of epsilon-iron	**Experiment number**: HS2860
Beamline: ID27	**Date of experiment**: from: 06/04/2006 to: 11/04/2006	**Date of report**: 28/02/2007
Shifts: 15	**Local contact(s)**: Nicolas Guignot	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Anne Line Auzende*, James Badro*, Guillaume Fiquet*

Département de Minéralogie, IMPMC et IPGP, Université Paris 6, Université Paris 7, CNRS, Campus Boucicaut, 140 rue de Lourmel
75015 Paris

Evgueni Gregoryanz*

School of Physics, CSEC, University of Edinburgh, Erskine Williamson Building, Mayfield Road, Edinburgh EH9 3JZ, UK

Report:

The high-pressure, high-temperature behaviour of iron has been investigated to 140 GPa and 3500K by in situ synchrotron X-ray diffraction with double-side laser-heated diamond anvil cells at. We found that only α-bcc, γ-fcc, and ε-hcp Fe can be clearly verified as the stable solid phases in the explored *P–T* range.

A lot of care was taken for sample preparation, made of a mixture of pure polycrystalline MgO and fine grained iron. This mixture was first hot pressed for 24 hours in very reducing conditions, so as to obtain a dense starting material free from any iron oxides. This sample was then subsequently thinned down to 15 μm and shaped into discs 30 μm in diameter, and finally loaded in diamond-anvil cell devices under a dry neon atmosphere in a 2000 bar gas vessel.

Diamond-anvil cells were then aligned on the newly installed double-sided laser heating set-up available at ID27 beamline. At pressure exceeding 85 GPa, ε-Fe is observed to P-T conditions approaching those existing in outer core. No evidence could be found for any phase transition toward d-hcp structure as previously reported by Saxena et al. (1995) or to a Pbcm orthorhombic phase as proposed by Andrault et al (1997). The diffraction pattern shown in Figure 1 is a perfect illustration of the quality of the pattern we were able to collect over the whole pressure range. Such a diffraction pattern can unambiguously be interpreted as a mixture of MgO and hcp-iron. In addition, all samples were recovered and prepared for some analytical TEM study, that have shown no significant reaction between iron and the MgO matrix.

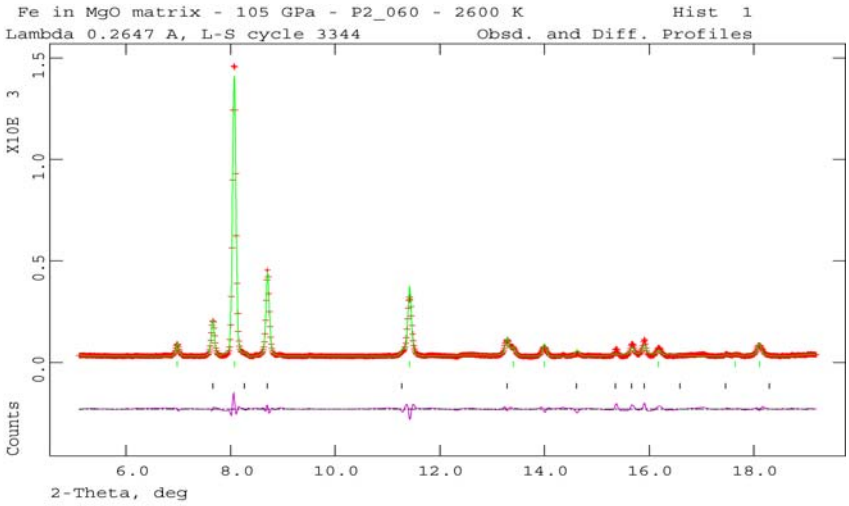

Figure 1: X-ray diffraction pattern collected at 105 GPa and 2600 K, at a wavelength of 0.26472 Å. Upper ticks denote MgO reflections whereas lower ticks correspond to hcp iron. A very small peak around a 2-theta angle of 9.3 corresponds to the most intense reflection for neon, quite weak at these extreme temperatures.

Within the *P–T* range examined, we did not observe a significant change with pressure or temperature on the *c*/*a* ratio of ε-Fe (see Figure 2). This observation is quite in disagreement with theoretical calculations of Steinle Neumann (2001), that reported a large variation of this ratio with increasing temperature at high pressure. Our observation casts a new light on the change of anisotropy proposed by this theoretical approach, since the large change in c/a ration was a key feature in these theoretical calculations.

In Figure 3, we report a phase diagram where the triple point γ-ε-liquid is accurately determined with the use of periclase as an internal pressure standard. Our results slightly differ from a recent study by Ma et al. (2004), where no internal pressure standard was employed. Our measurements yield the triple point at around 90 ± 3 GPa.

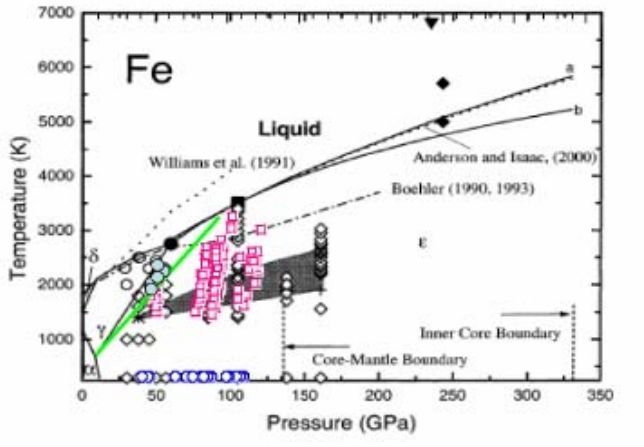

Figure 2: P-T data set and phase diagram, along results reported by Ma et al. (2004). At high-temperature, blue dots stand for fcc-iron, pink squares for hcp-iron. The main feature is that the triple point γ-ε-liquid is shown to be at higher pressures than previously reported.

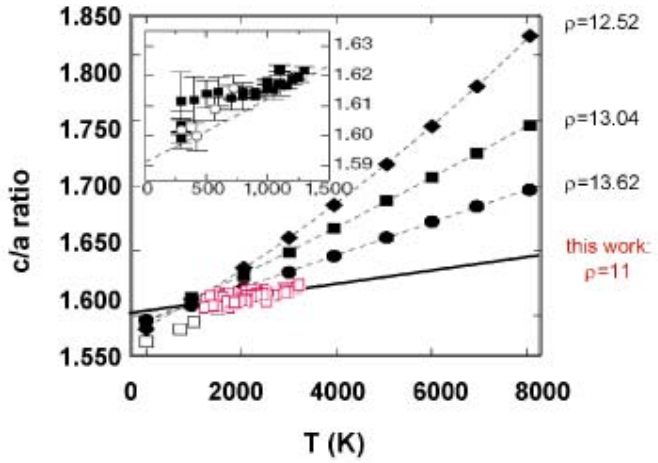

Figure 3: evolution of the c/a ratio at high-temperature at different densities. Solid symbols are from Steinle-Neumann (2001). Our measurements show a very different evolution at high-temperature, which suggests the need for improved theoretical treatment of the elastic anisotropy of iron at high pressures and temperatures

Data analysis is almost completed now, and should provide a reliable P-V-T equation of state, as described in the original proposal.

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

Ma Y. et al., **Phys. Earth Planet. Int.**, 143-144, 455, 2004.

Saxena et al., **Science**, 269, 1703, 1995.

Steinle Neumann et al., **Nature**, 413, 57, 2001.