



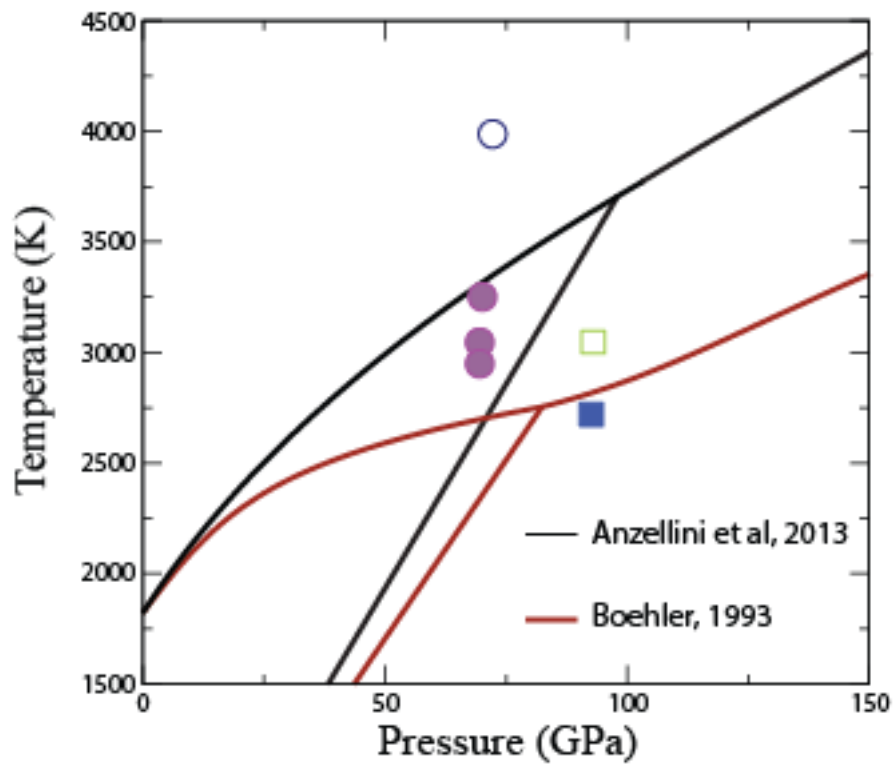
	<b>Experiment title:</b> Melts properties of Fe-O alloys under high pressure by Fe K-edge XANES and EXAFS	<b>Experiment number:</b> ES 358
<b>Beamline:</b> ID 24	<b>Date of experiment:</b> from: 30/09/2015 to: 06/10/2015	<b>Date of report:</b> 11/09/2017
<b>Shifts:</b> 18	<b>Local contact(s):</b> Angelika Rosa	<i>Received at ESRF:</i>
<b>Names and affiliations of applicants</b> (* indicates experimentalists):  Guillaume Morard (IMPMC, Paris) Marion Harmand (IMPMC, Paris) Silvia Boccatto (ESRF, Grenoble) François Guyot (IMPMC, Paris) Raffaella Torchio (ESRF, Grenoble) Eglantine Boulard (Institut Neel, Grenoble)		

## Report:

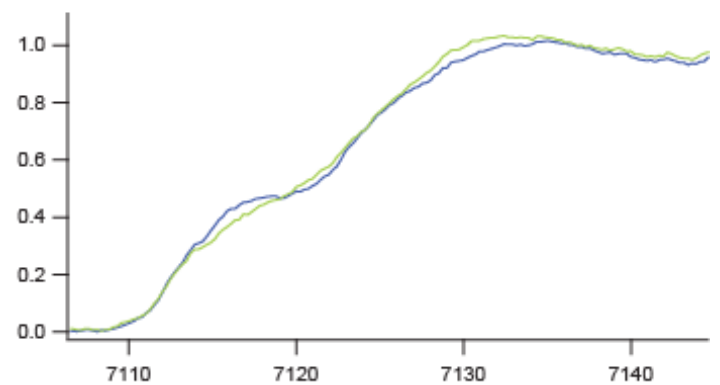
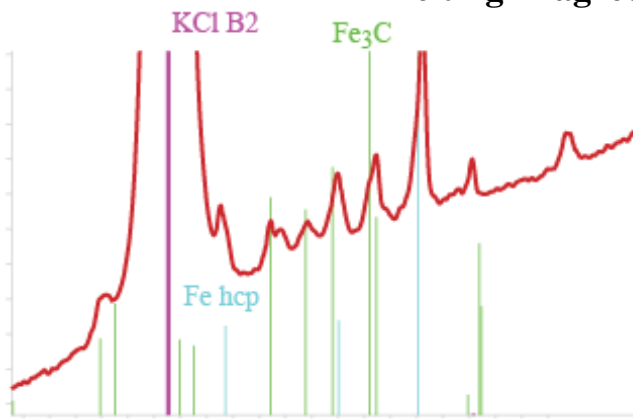
During this experiment, we investigated the melting of pure iron and of different iron alloys by in situ XANES using the Laser-Heating Diamond Anvil Cell setup available on ID24.

The sample consisted of a flake of pure Fe or Fe alloys (made either by deposition (see (Morard et al. 2017) or rapid quench (Morard et al. 2011)) sandwiched between two KCl disks, loaded in a DAC. The whole DACs were dried under vacuum for several hour in order to remove the potential water absorbed by the KCl. Pressure was measured before and after the laser-heating cycle using either Raman shift from the diamond tip (Ocelli, Loubeyre, and LeToullec 2003) or the fluorescence of a ruby sphere placed in the sample chamber (Syassen 2008). Recovered samples were cut using FIB and the texture was also used as aternative melting criteria. Furthermore, XRD mapping has been also performed on this recovered samples.

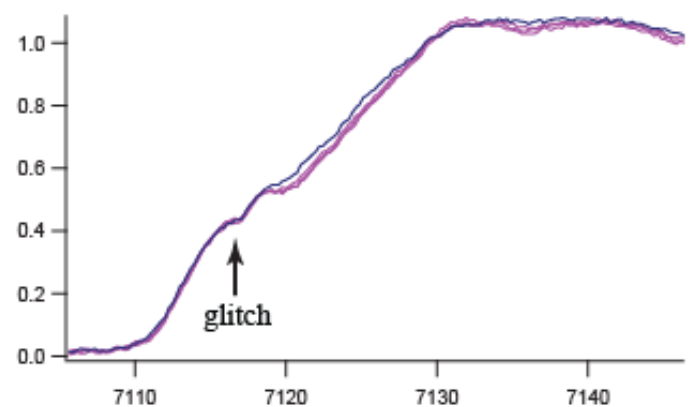
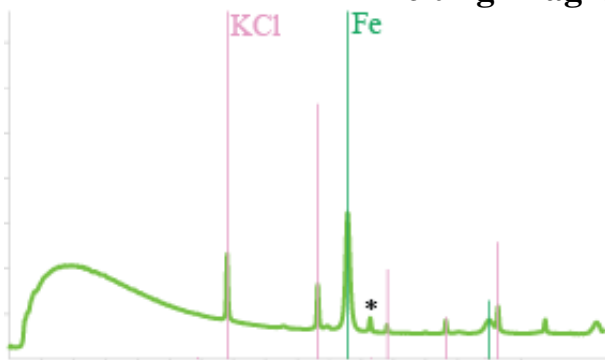
Regarding melting of pure Fe, we perform similar experiment as (Aquilanti et al. 2015), but our overall results are significantly different. In fact, the controversy between (Boehler 1993) and (Anzellini et al. 2013) emphasize a difference over 1000 K in the position of the iron melting curve under a pressure of 100 GPa. We used the same melting criteria, but we succeed to find similar results as in (Anzellini et al. 2013). The explanation of this difference came from the fact that carbon contamination drastically reduce the melting point of iron. Therefore, data from (Aquilanti et al. 2015) is a signature of melting in the Fe-C system and not the melting of pure Fe.



**Melting in agreement with Boehler curve**



**Melting in agreement with Anzellini curve**



*Figure 1 : Melting of iron measured in the present experiment. Melting point in agreement with Boehler curve shows evidence of Fe<sub>3</sub>C peaks on the recovered sample, whereas melting point in agreement with Anzellini curve shows pure Fe in the quench sample.*

At the same time, change upon melting for the XANES edge has been collected for different alloys, and the melting signature was quite different depending on the type of alloy studied. But overall, the melting temperature measured were in excellent agreement with the published melting curves measured by in situ XRD (Morard et al. 2011; Morard et al. 2017).

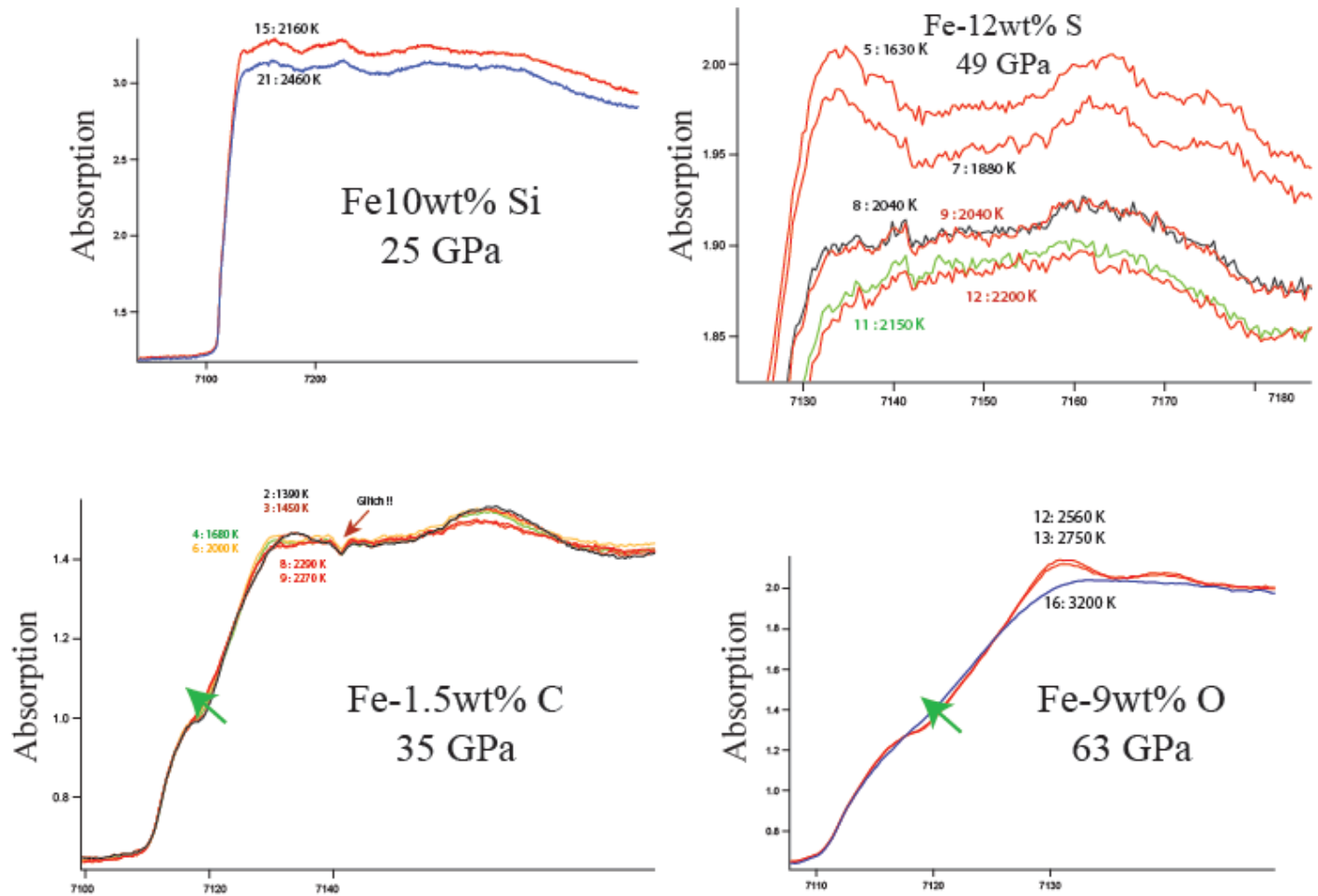


Figure 2: Change in the absorption edge of iron for different iron alloys upon melting.

Two scientific papers are actually in preparation to discuss the melting of pure Fe and the melting criteria in iron alloys. One paper related to this work has been already published (Torchio et al. 2016).

#### References:

- Anzellini, S, A Dewaele, M Mezouar, P Loubeyre, and G Morard. 2013. "Melting of Iron at Earth's Inner Core Boundary Based on Fast X-Ray Diffraction." *Science* 340 (1998): 464–66. doi:10.1126/science.1233514.
- Aquilanti, Giuliana, Angela Trapananti, Amol Karandikar, Innokenty Kantor, Carlo Marini, Olivier Mathon, Sakura Pascarelli, and Reinhard Boehler. 2015. "Melting of Iron Determined by X-Ray Absorption Spectroscopy to 100 GPa." *Proceedings of the National Academy of Sciences* 112 (39): 12042–45. doi:10.1073/pnas.1502363112.
- Boehler, R. 1993. "Temperatures in the Earth's Core from Melting-Point Measurements of Iron at High Static Pressures." *Nature* 363 (6429): 534–36. doi:10.1038/363534a0.
- Morard, G, D Andrault, D Antonangeli, Y Nakajima, A L Auzende, E Boulard, S Cervera, et al. 2017. "Fe – FeO and Fe – Fe<sub>3</sub>C Melting Relations at Earth's Core – Mantle Boundary Conditions : Implications for a Volatile-Rich or Oxygen-Rich Core." *Earth*

- and Planetary Science Letters* 473. Elsevier B.V.: 94–103.  
doi:10.1016/j.epsl.2017.05.024.
- Morard, G, D Andrault, N Guignot, J Siebert, G Garbarino, and D Antonangeli. 2011. “Melting of Fe-Ni-Si and Fe-Ni-S Alloys at Megabar Pressures: Implications for the Core-Mantle Boundary Temperature.” *Phys. Chem. Minerals* 38: 767–76.
- Occelli, Florent, Paul Loubeyre, and René LeToullec. 2003. “Properties of Diamond under Hydrostatic Pressures up to 140 GPa.” *Nature Materials* 2 (3): 151–54.  
doi:10.1038/nmat831.
- Syassen, K. 2008. “Ruby under Pressure.” *High Pressure Research* 28 (2): 75–126.  
doi:10.1080/08957950802235640.
- Torchio, R., S. Boccato, V. Cerantola, G. Morard, T. Irifune, and I. Kantor. 2016. “Probing the Local, Electronic and Magnetic Structure of Matter under Extreme Conditions of Temperature and Pressure.” *High Pressure Research* 36 (3).  
doi:10.1080/08957959.2016.1198904.