



	Experiment title: Structural transition in liquid iron around the triple point	Experiment number: HC-4874
Beamline: ID24	Date of experiment: from: 09/02/2023 to: 13/02/2023	Date of report: 12/09/2023
Shifts: 12	Local contact(s): Angelika Rosa	<i>Received at ESRF:</i>
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Report:

X-ray absorption spectroscopy maps were performed on iron samples at high pressure recovered after laser heating experiments coupled to X-ray diffraction (experiment HC-5073).

The pressure-temperature covered during this experiment was 50-170 GPa and 2000-5500 K, with the purpose of determining eventual changes in the local structure and compressibility of liquid iron at the pressure of the triple point (~100 GPa).

After laser heating and in situ XRD on ID27 beamline, samples were kept under pressure in the diamond anvil cell and mapped on ID24 to check for chemical reactions with carbon from the diamond, following the protocol established in [Boccato2020] and in [Morard 2018].

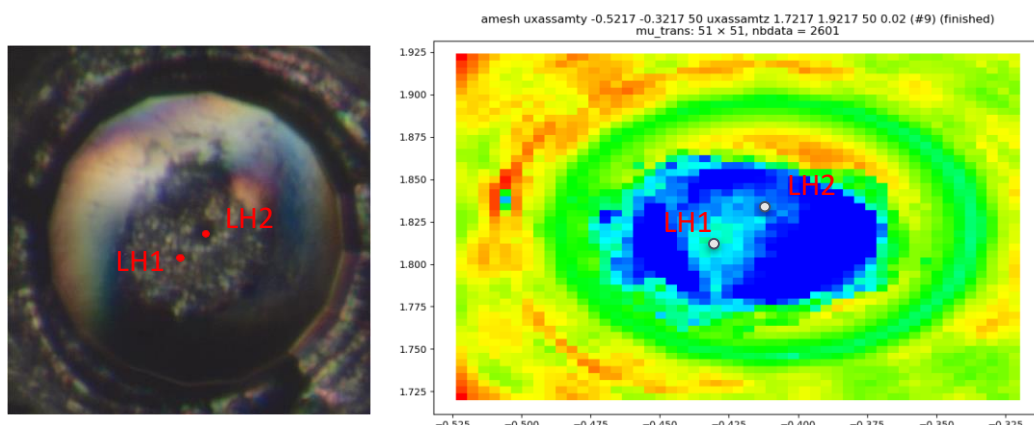


Figure 1. Comparison between the optical image (left) and the XAS mapping (right).

The Fe sample was a 10 μm thick, high purity (>99.99%) Goodfellow Fe foil loaded in a diamond anvil cell, between two previously dried KCl $\sim 10\ \mu\text{m}$ thick disks, which acted as pressure transmitting medium, as well as chemical and thermal insulators. The pressure was determined according to the thermal equation of state of KCl [Dewaele2006].

A first quick map allowed to identify the regions heated by the laser, also thanks to previously recorded optical images, as represented in Figure 1. Regions of about $20 \times 20\ \mu\text{m}^2$ were then selected around the hot spot for a fine mapping.

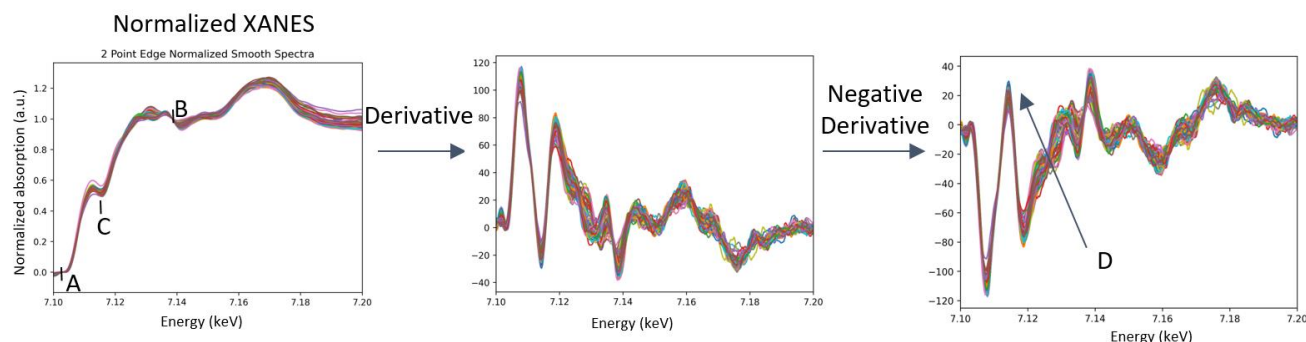


Figure 2. Left: Normalized XANES. Centre: XANES derivative. Right: Negative derivative.

The beam size of about $0.7 \times 0.7\ \mu\text{m}^2$ and a step of $2\ \mu\text{m}$ allow to finely probe chemical reactions around the heated spot. All the spectra in the fine map are normalized setting the intensity of the XANES before the edge (point A in Figure 2) at 0 and the XANES after the edge (point B) at 1 for all spectra. The derivative of XANES spectra is then calculated and inverted. The prominence of the peak indicated as C in Figure 2 will be referred to as “Edge bump prominence”, the edge jump previous to the normalization will be referred to as “Edge prominence”, and the prominence of the peak indicated as D will be indicated as “Negative Edge Bump Slope”. These three parameters can be plotted to identify and localize chemical reactions, as shown in Figure 3.

Once the reaction has been localized, using the protocol developed in [Boccatto2020] it is possible to quantify the amount of carbon in the hot spot. This analysis is still ongoing.

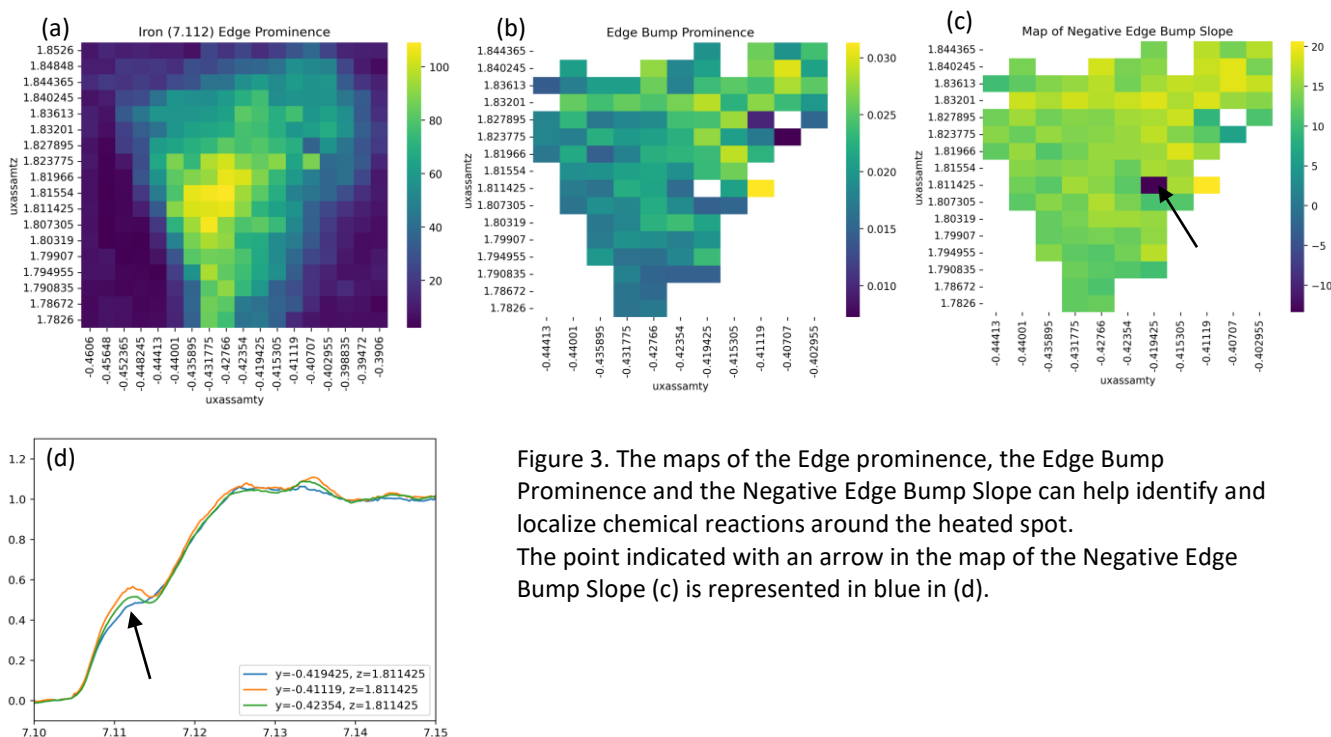


Figure 3. The maps of the Edge prominence, the Edge Bump Prominence and the Negative Edge Bump Slope can help identify and localize chemical reactions around the heated spot. The point indicated with an arrow in the map of the Negative Edge Bump Slope (c) is represented in blue in (d).

In the framework of this project, mapping of previously laser heated Fe-O samples was performed, Figure 4. The fine mapping allows to easily localize and identify the hot spots and perform linear combination analysis to determine the composition of the hot spot after heating. This analysis, performed on heated Fe-O at different pressures will allow to better constrain the compositional phase diagram of Fe-O.

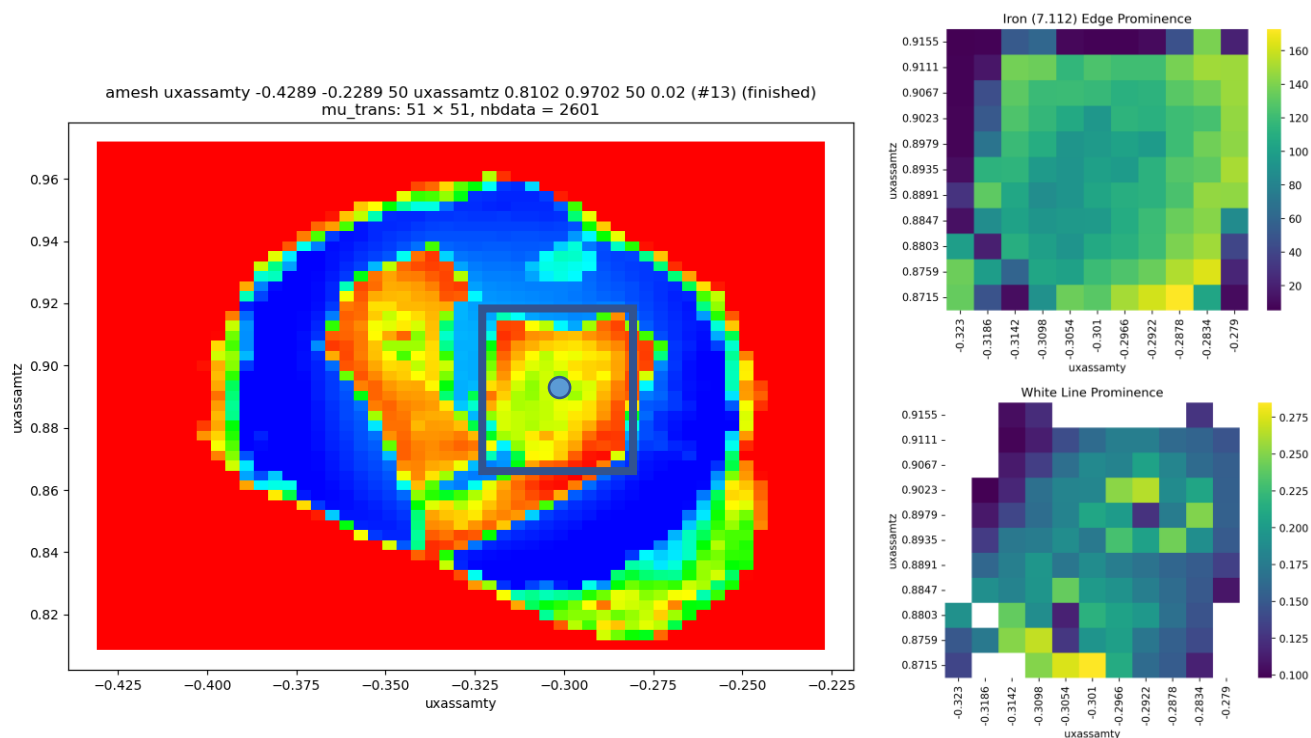


Figure 4. Test on Fe-O sample. Changes of composition can be easily identified with the mapping, and the composition of the recovered sample can be determined by linear combination analysis of reference spectra under pressure of Fe and stoichiometric FeO.

References

Boccatto, S. et al. Sci. Rep. 1–13 (2020)

Dewaele, A. et al. Phys. Rev. Lett. 97, 215504 (2006)

Morard, G. et al. Geophys. Res. Lett. 45, 11, 074–11, 082 (2018)