ESRF	**Experiment title:** Local structure of molten Fe Ni alloys at high pressures.	**Experiment number**: HC-2254
Beamline: ID24	**Date of experiment**: from: 3/02/2016 to: 9/02/2016	**Date of report**: 29/02/2016
Shifts: 18	**Local contact(s)**: Raffaella Torchio	*Received at ESRF:*

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Report:

Here we present first XAS measurements on melting of FeNi alloy at the Ni K-edge (8333eV). The data where recorded on the beamline ID24 using the recently installed laser heating system which allows to measure in simultaneous the absorption spectrum and the temperature of a laser heated sample in a diamond anvil cell.

A Goodfellow $Fe_{64}Ni_{36}$ foil 8 μm thick was loaded in a diamond anvil cell where the chemical and thermal insulation was provided from both sides by two KCl patalets 10 μm thick. A ruby loaded in the cell makes possible the measurement of the pressure thanks to its fluorescence spectrum which shifts with pressure. The sample is heated with two YAG (1064 nm) lasers and the temperature is measured through pirometry as a fit of the Planck function in the range 650-950 nm.

In this experiment we probed the melting temperature of $Fe_{64}Ni_{36}$ at different pressures between 30 GPa and 100 GPa reaching temperatures up to 3500 K, as shown in Fig. 1.

We adopted two ways to detect melting. In the XANES melting criterion we detect changes in the near edge region of the absorption spectrum as a function of temperature: when the edge at around 8340 eV flattens and the two bumps in the first post edge between 8345 eV and 8365 eV disappear the sample is considered molten (Fig. 2).

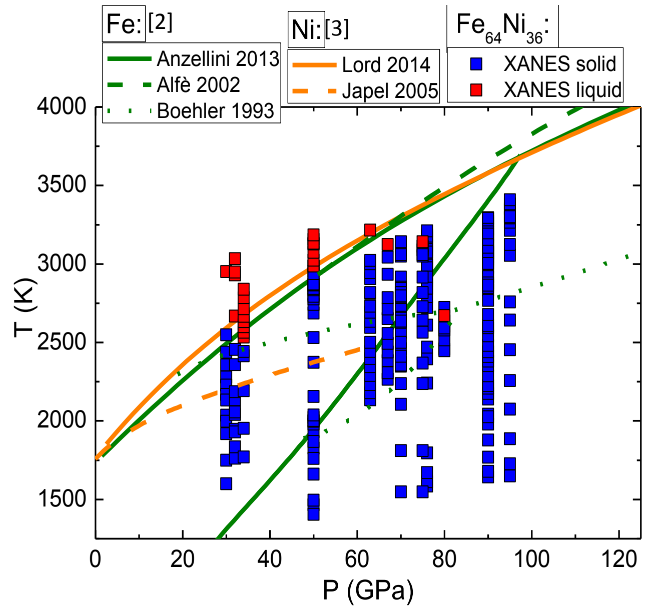

Figure 1. Comparison of the melting of pure Fe, pure Ni and $Fe_{64}Ni_{36}$ detected with different techniques and calculations.

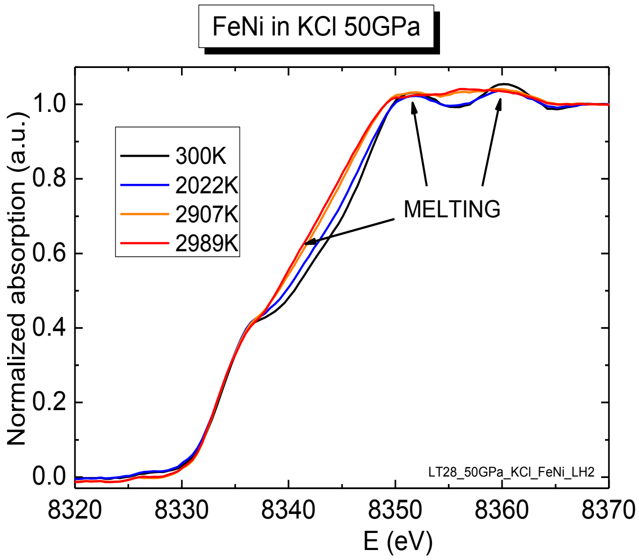

Figure 2. Detection of the melting on a XANES spectrum.

In the EXAFS melting criterion, instead, changes in the Fourier transform of the absorption spectra are detected as a function of temperature: when the second shell disappears the spectrum is considered molten (Fig. 3).

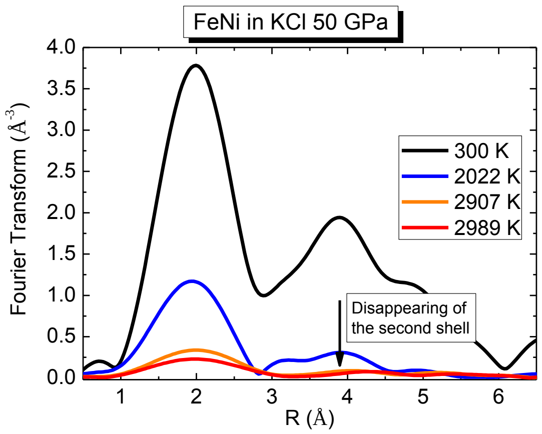

Figure 3. Detection of melting with EXAFS criterion

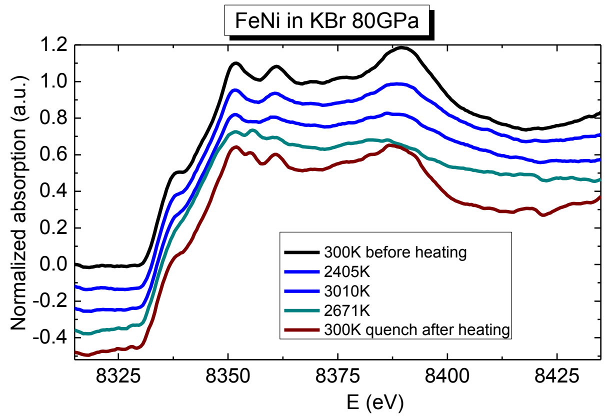

Figure 4.

In Fig. 1 is shown the melting curve obtained with the XANES melting criterion. Up to 60 GPa the agreement with XRD melting curves for Fe and Ni found in literature (Anzellini 2013 and Lord 2014) is quite good, while there is no agreement with the melting curves found with speckle (Boehler 1993 and Japel 2005). At higher pressures, starting from 60 GPa, melting was more difficult. The three points at 65 GPa, 75 GPa and 80 GPa need to be analysed in detail to check if there is any signature of reaction.
Moreover most of the times heating up the sample (even at temperatures lower than the melting temperature) the absorption spectrum was changing probably due to structural changes or chemical reactions maybe induced by the exposure to the laser of about 1 second. This aspect certainly deserves further analysis, in the framework of the understanding of FeNi alloys relevant for the study of Earth's interior.

A quantitative analysis of the local structure of the molten phase, as well as the study of the melting curve by means of the EXAFS melting criterion, is still ongoing.