



	Experiment title: Study of the local structure of molten Ni and Co at high pressures.	Experiment number: HC-2882
Beamline: ID24	Date of experiment: from: 26/10/2016 to: 31/10/2016	Date of report: 01/03/2017
Shifts: 18	Local contact(s): Silvia Boccato	<i>Received at ESRF:</i>
Names and affiliations of applicants (* indicates experimentalists): Silvia Boccato (ESRF) Simone Anzellini (Diamond) Raffaella Torchio (ESRF)		

Report:

Here we present first XAS measurements on melting of Co at the K-edge (7709eV) and Ni at the K-edge (8333eV). The data were 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 Co deposition 4 μm thick and a Ni Goodfellow foil 4 μm thick were 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 Co and Ni at different pressures between 10 GPa and 110 GPa reaching temperatures up to 4000 K, as shown in Fig. 1 and Fig.2. This experiment allowed to fill the gaps at very low pressure and very high pressure of the phase diagram that we started to fill in previous experiments.

The criterion we adopted to detect melting is the following: the sample is molten when in the XANES region the edge at around 7704 eV in Co flattens and the two bumps in the first post edge between 7725 eV and 7735 eV disappear the sample is considered molten (Fig. 3). The same criterion was used for nickel, with the energies appropriately translated due to the different K-edge energy.

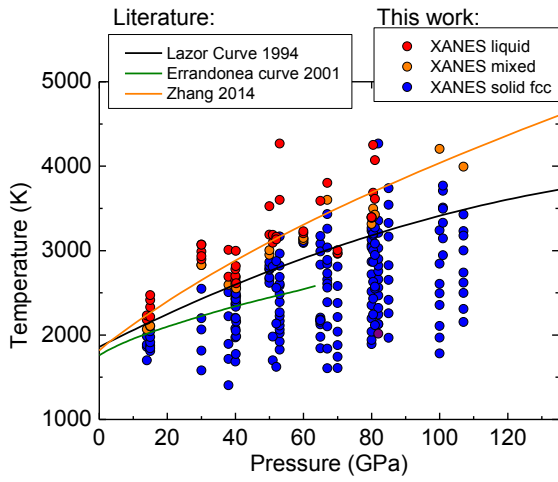


Figure 1. Melting curve of cobalt measured with X-ray absorption

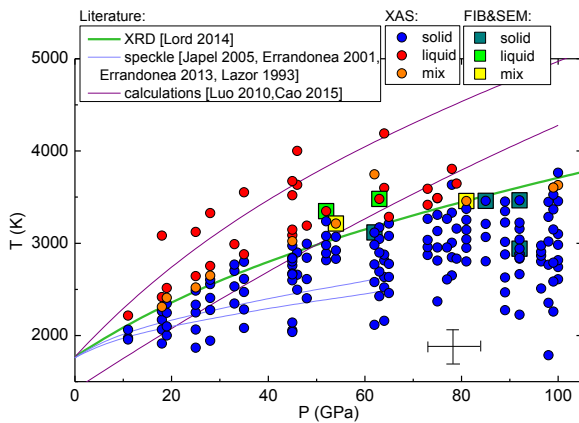


Figure 2. Melting curve of Nickel measured with X-ray absorption

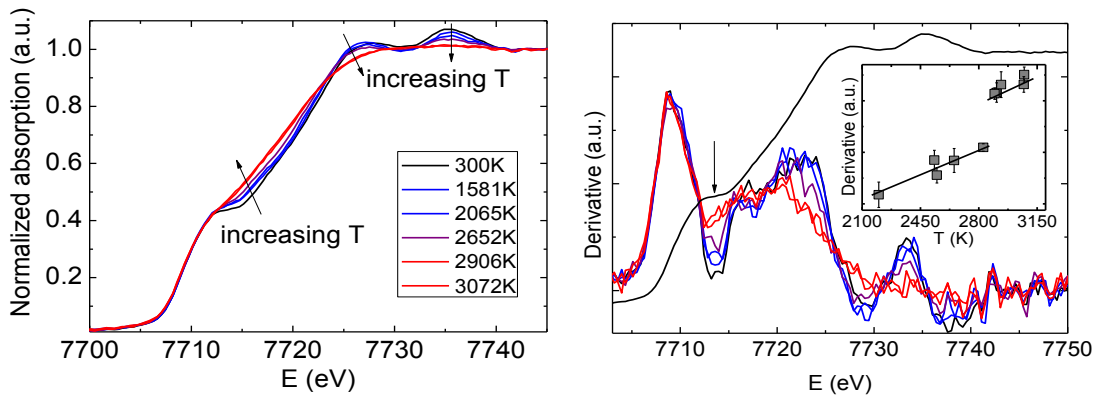


Figure 3. Detection of melting measured with X-ray absorption in the XANES region. Changes in the XAS spectrum are even more evident in the derivative. The red spectra are the molten ones.

In Fig. 1 is shown the melting curve obtained with the XANES melting criterion showed in Fig. 3. The best agreement is found with calculations performed by Zhang (Zhang 2014). While the agreement with previous melting curves measured with speckle (Errandonea 2001 and Lazor 1994) is very poor. Some of the points, such as the one at 70GPa need to be analysed in detail to check if there is any signature of reaction.

In Fig. 2 is shown the melting curve obtained for Nickel, where we found a very good agreement with recent X-ray diffraction measurements of melting (Lord 2014).

A quantitative analysis of the local structure of the molten phase, still to be refined, already gave promising results showing a compression of the first neighbours distances of the liquid as a function of pressure. A check of the temperature measurements with the two colors fit is still ongoing.