	Experiment title: Melting of iron at high pressure using X-ray absorption spectroscopy	**Experiment number**: HD313
Beamline: ID24	**Date of experiment**: from: 12/11/2008 to: 22/11/2008	**Date of report**: 27/02/2009
Shifts: 33	**Local contact(s)**: Giuliana Aquilanti	*Received at ESRF:*

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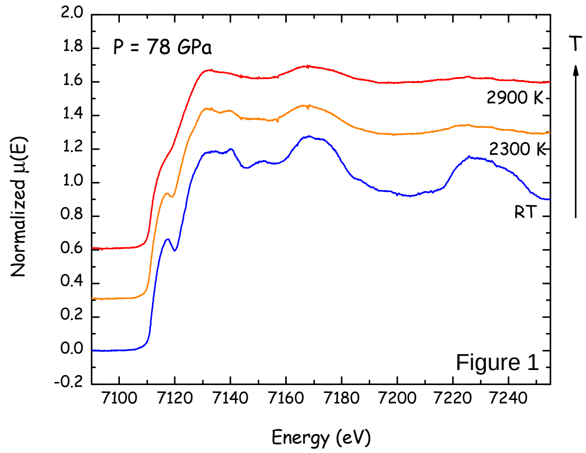
Report:

The structure of molten elements, as well as the thermodynamic conditions of melting under high pressure are of great interest in different fields: from fundamental physics to planetary interior's studies. More specifically, melting of iron has geophysical implications because it is the major element of our planet. Its melting temperature at high pressure constrains the temperature in the Earth's core and, thus the thermal gradient and heat flows in the core and in the mantle. The melting temperature of Fe at the inner outer core boundary has been determined with many different techniques with temperature differences of over 2000 K [1]. These discrepancies are too large for modelling of the thermal history of the Earth. Besides the geophysical implications, melting of iron is very interesting from a fundamental point of view. Iron is a classic d-electron metal and – together with other transition metals – is worth to be investigated in the liquid phase at high pressure to verify experimentally the hypothesis according to which liquid frustration, i.e. the presence of local structures in the liquid phase would lower the freezing point in those metals with partially filled d-bands [2].

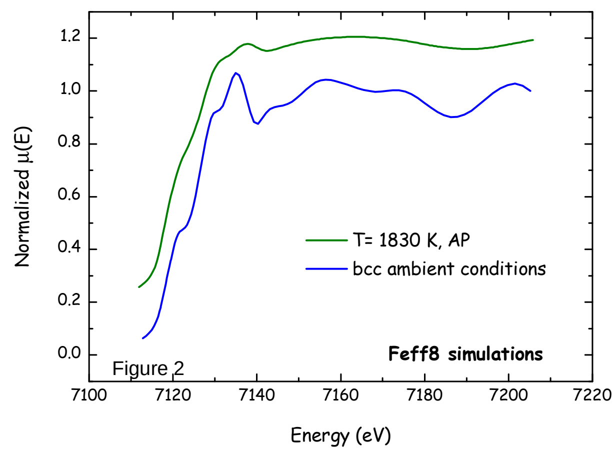
The aim of this proposal was twofold:

- To ascertain the suitability of EDXAS as a new alternative method to detect melting in iron
- To provide evidence of preferred local ordering in liquid iron at high pressure using a local probe such as XAS.

We have performend XANES measurements at Fe K-edge at ID24. The source consisted in 3 undulators whose gap was adjusted to have the mazimum of the first harmonic at Fe K-edge. The beam was focused horizontally using a Si(311) polychromator and vertically by a silicon mirror at 4 mrad. The beam size was 5x5 mm^2 FWHM. The absorption of the diamonds was minimized reducing to 1.3 mm the thickness of the anvils. Several samples were tested at several pressures. The sample resulted to be best suited to be laser heated and measured with a good S/N ratio consisted in an iron grain squeezed and incapsulated between two polished ruby platetd and embedded in Ar. The sample was laser heated from both sides using a portable laser stand built at the Max Planck Institute of Mainz (Germany) and tested at both ID 24 and ID 27 [3]. The iron sample was pressuized to 78 GPa. The temperature was progressively increased from 2300 K to 2900 K and after each spectrum at high temperature a spectrum at room temeprature was recorded. The normalized data are shown in Figure



1. As the temperature is raised from ambient to 2300 K all the spectral features of the hcp iron are conserved. The oscillations are damped because of the increasing thermal disorder. At 2900 K the changes in the spectrum are dramatic: the pre-peak disappears and so does the double oscillations in the 7130-7140 eV energy range suggesting the occurrence of a solid-liquid phase transition. An important feature of these X-ray absorption measurements is the remarkable reproducibility in the spectra when cycling through the entire temperature range, including melting and freezing events. To verify whether the disappearance of the prepeak can be a reliable signature of melting we performed XANES simulations using Feff8 on clusters obtained using the EPRS [4] method applied on existing structural data of liquid Fe. The simulations, shown in figure 2, evidence the disappearance of the prepeak when going from solid to liquid iron in agreement with the experimental data. This result shows that EDXAS can be an alternative, reliable method to detect melting in iron. The present result on the melting temperature of iron is in very good agreement with previously measured melting curves obtained by a number of groups. The average melting temperature in these studies at 78 GPa is slightly below 2800 K [5]. The analysis to provide evidence of preferred local ordering in liquid iron at high pressure using a local probe such as XAS is underway and, since a structural model at HP is not available it requires additional XAFS data, possibly at lower pressure in order to be able to compare the simulations with the room pressure liquid iron and to see the evolution of the data as a function of pressure. In conclusion, we have recorded a XANES spectrum of liquid iron at 78 GPa. More data at other pressures are needed both to map the melting curve of iron and to determine with increasing density the occurrence of preferred local structure in the liquid phase.



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