



	Experiment title: Melting line of Iron under the Earth's Core conditions	Experiment number: HS-4741
Beamline: ID27	Date of experiment: from: 01/11/2012 to: 06/11/2012	Date of report: 31/03/2013
Shifts: 15	Local contact(s): M. Mezouar	<i>Received at ESRF:</i>
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Report:

This proposal was the direct continuation of HS-4477. The aim of this project was to measure the melting line of iron up to pressure-temperature conditions closest possible to the inner-core boundary conditions inside the Earth's Core (330 GPa, several thousands of K). Indeed, the temperature at the inner core boundary – which is impossible to measure directly - is expected to be close to the melting point of iron at 330 GPa, because the outer (inner) core is mainly composed of molten (solid) iron. Despite intensive experimental and theoretical efforts, there was little consensus on the melting behavior of iron at these extreme pressure and temperature. In particular, the melting points measured in laser-heated diamond anvil cell [1] were ~1000K lower than the melting points measured with shock compression. We wanted to confirm the melting line measured in diamond anvil cell using another melting diagnostic, based on X-ray diffraction (XRD).

During beamtimes HS-4477 and HS-4741, we have measured the phase (γ -Fe, ϵ -Fe or liquid) of iron at P-T conditions varying between 50 and 210 GPa, and 300 and 4800K. The sample was compressed to the target pressure and gradually heated with increasing lasers power. XRD spectra were taken every ~4 s during heating. The experimental conditions for each run of HS-4741 are summarized in **table 1**.

Run	Anvils culet size(μ m)	Pits	P range (GPa)	T range (K)	Pressure medium
CDMX6	300	N	45-70	300-3800	KCl
CDMX12	70x300	Y	60	300	KCl
CDMX11	50x300	Y	50-150	300-3000	KCl
CDMX131	150x300	N	45-140	300-4200	KCl
CDMX7	50x300	Y	5-100	300-2935	KCl
CDMX19	200x300	N	60-100	300-3800	KCl

CDMX11	50x300	Y	50-210	300-3500	KCl
CDMX7	70x300	Y	50-190	300-4000	KCl

Table 1: conditions of each experimental run for HS-4741.

The temperature was measured by pyrometry. The pressure measurement was based on the equation of state of KCl pressure medium [2], and cross-checked with Fe equation of state [3]. The temperature measurement was validated with the measurement of the thermal expansion of the sample during a heating series.

In the first runs with CDMX12, CDMX11, CDMX7, we have observed a chemical reaction between iron sample and the diamond anvil for the anvils with pits. It was due to a partial graphitization of the anvil during the focused ion beam machining; graphite reacts with iron more efficiently than diamond. After removal of the graphite layer by chemical etching, the reaction between iron and diamond anvil was inhibited. However we had difficulties to heat up the sample above 3500-4000K with the pitted anvils.

The P-T points, with phase information, are represented in **Fig. 1**. Above a certain temperature single crystal spots appeared and disappeared at each XRD exposure, which we will refer to as “fast recrystallization”. The melting line measured with the XRD diagnostic is substantially higher than the melting line measured in Ref. [1]. It can be noted that the fast recrystallization threshold, represented as dotted line in Fig. 1, corresponds approximately to the melting line measured in Ref. [1]. This suggests that fast recrystallization causes the surface motion observed in Ref. [1] which had been wrongly attributed to melting.

These results have been published in Science magazine [4].

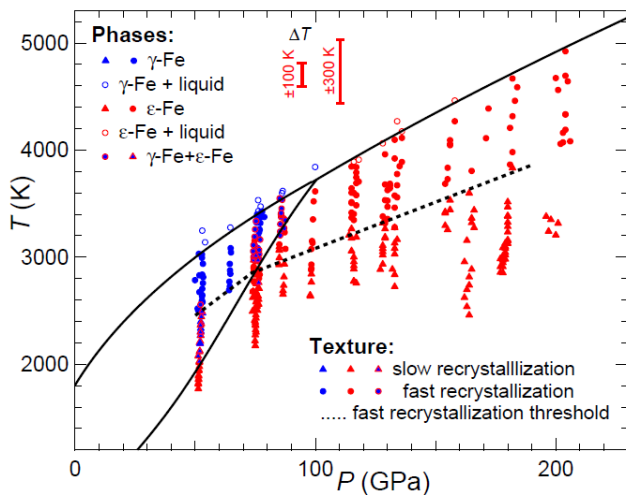


Fig. 1: Pressure - temperature conditions at which XRD patterns have been collected. Different symbols correspond to different Fe phases and textures. The continuous black lines represent the phase boundaries deduced from our measurements.

[1] R. Boehler, Temperature in the Earth’s core from melting-point measurements of iron at high static pressures. *Nature* 363, 534 (1993).

[2] A. Dewaele et al., High-pressure-high-temperature equation of state of KCl and KBr. *Phys. Rev. B* 85, 214105 (2012).

[3] A. Dewaele et al., Quasihydrostatic Equation of State of iron above 2 Mbar. *Phys. Rev. Lett.* 97, 215504 (2006).

[4] S. Anzellini et al., melting of iron at Earth’s inner core boundary based on fast X-ray diffraction, *Science* 340, 464 (2013).