



	<b>Experiment title:</b> Melting line of iron under the Earth's core conditions	<b>Experiment number:</b> HS-4477
<b>Beamline:</b> ID27	<b>Date of experiment:</b> from: 20/9/2011 to: 23/9/2011 27/11/2011 29/11/2011	<b>Date of report:</b> 6/2/2012
<b>Shifts:</b> 15 <a href="http://www.cecam.org/workshop-667.html">http://www.cecam.org/workshop-667.html</a>	<b>Local contact(s):</b> M.Mezouar	<i>Received at ESRF:</i>
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### Report:

The aims of this experiment were: (1) the determination of the melting points of iron under Earth's core conditions; (2) the reduction of the discrepancies between the melting points obtained by different high pressure techniques.

Fe sample were pressurized in Diamond Anvil Cells (DAC) with an automatic pressure driver. The sample was a thin disc obtained by compression of powder grains between two diamond anvils or a powder grain for the highest P runs. Different pressure media: KCl, Ne and He were tested to thermally/chemically insulate the sample from the anvils and to guarantee hydrostaticity. In some runs, diamond anvils with pits have been used. The pressure was first increased to the desired range. Then it was gradually heated by the two lasers; X-ray and pyrometry signal recorded every ~4s during each heating series. Several heating series were performed at each pressure step. Alignment of X-ray, laser beam and pyrometry were checked for each heating series. Various sample assemblies have been used in the conditions summarized in **Table 1**.

Run	Anvils culet size(μm)	Pits	P range (GPa)	T range (K)	Pressure medium	Melting	Ch. React.
1	200x300	N	65-85	300-3800	KCl	N	N
2	100x300	N	92-103	300-3073	Ne	N	Y
3	300	N	50-65	300-3200	KCl	N	Y
4	200x300	N	8-75	300-2730	KCl+He	N	Y
5	100x300	N	5-100	300-2935	KCl	N	N
6	75x300	Y	30-130	300-5300	Ne	N	Y

7	75x300	Y	3-140	300	KCl	N	N
8	300	N	47-90	300-4698	KCl	Y	N
9	75x300	N	85-200	300-5500	KCl	Y	N

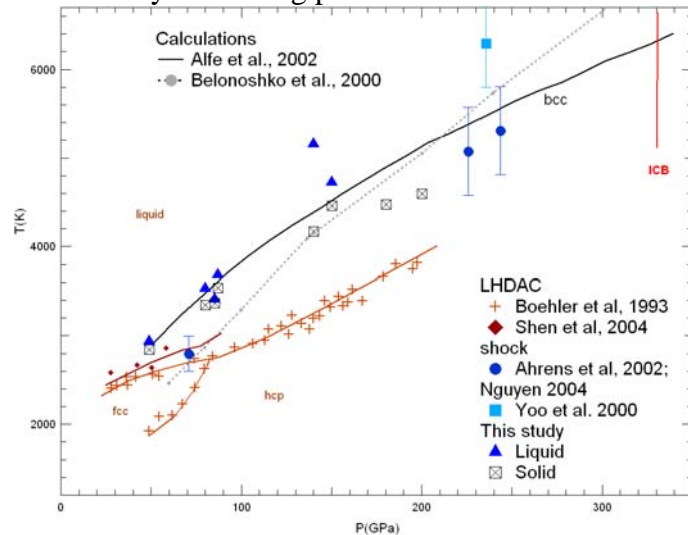
**Table 1:** conditions of each experimental run.

The experiments show that the Fe-C reaction cannot be minimized by a careful sample loading: sample with a very flat surface and the right thickness seem to be the most important parameters. We found KCl to be the best pressure medium to be used; it better kept sample insulation during heating probably because its melting point is higher than Ne and He, the other pressure media tested in this study.

The melting criteria were the observations of the diffuse x-ray scattered signal of the fluid and the concomitant observation of a jump followed by a plateau in the pyrometric signal. The pyrometry temperature was checked using Fe high P-high T EOS [1]. Temperature measurement was considered reliable when pyrometry and EOS temperatures, based on measured Fe's volume, were in agreement. During runs 1,2,3, we weren't able to reach very high temperatures. This is probably due to the sample shape and the laser focusing. In runs 2,3,4 and 6 we observed some chemical reactions, possibly Fe-C.

The P-T conditions of some of our runs, with the melting observation, are summarized in **Figure 1**. We have been able to measure several melting points in the 50-150 GPa range in Runs 8 and 9, without any chemical reactions. We have also evidenced that the sample remains solid at temperatures higher than the melting line previously reported. The presence of saturated single crystal spots of Fe on the image plate ("active recrystallization"), sometimes used as complementary melting criterium, were observed at each run but the diffused liquid signal was recorded at higher temperatures (~450 K). The current melting point agree with theoretical predictions [2] and shock experiments [3], but are higher than previous DAC measurements [4]. In these experiments melting was optically detected, movement on the sample surface being interpreted as melting.

Our observations prove that "active recrystallization" happens at ~450 K below melting, could result in surface movements. This could be the cause of a general underestimation of the melting temperature in DAC when optical detection of melting is used. The current work emphasizes the necessity of an *in situ* X-ray diagnostic to understand the changes undergone by laser-heated samples in diamond anvil cells, in particular for the study of melting phenomena.



**Figure 1:** Comparison between the current results (blue triangles: liquid phase; empty squares: solid phase) and previous studies [3][4][5].

## References

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