

Properties of liquid iron alloys in the 1.5-3.5 Mbar range

High pressure-high temperature experiments have been performed on the ID27 beamline using the available laser heating diamond anvil cell experimental set-up. We continue the investigation of Fe-O and Fe-C systems performed in previous proposal (HS4752, HC1104) by studying different sample composition and preparation. This experiment was dedicated to melting experiment on iron alloys above 1.5 Mbar pressure. Samples consisted in a homogeneous sample, made by sputtering iron under O₂ flow (DEPHIS Company), sandwiched between two disks of sapphire. High pressures were generated using membrane DACs (Le Toullec type) mounted with Boehler Almax anvils (Boehler & De Hantsetters, 2004), with culet diameters of 300/70 μm (bevelled anvils).

During this experiment, we succeeded to obtain melting data over 150 GPa for Fe-O and Fe-C systems. In addition to previous experiments HS4752 and HC1104, we obtain now complete range of melting temperatures for Fe-O and Fe-C systems up to 200 GPa (Figure A and B).

Two scientific papers are currently under progress. One will be re-submitted before the end of September 2015 (melting of Fe-O alloys) and the other one will be submitted before the end of this year (phase diagram in the Fe-C system up to 2Mbar).

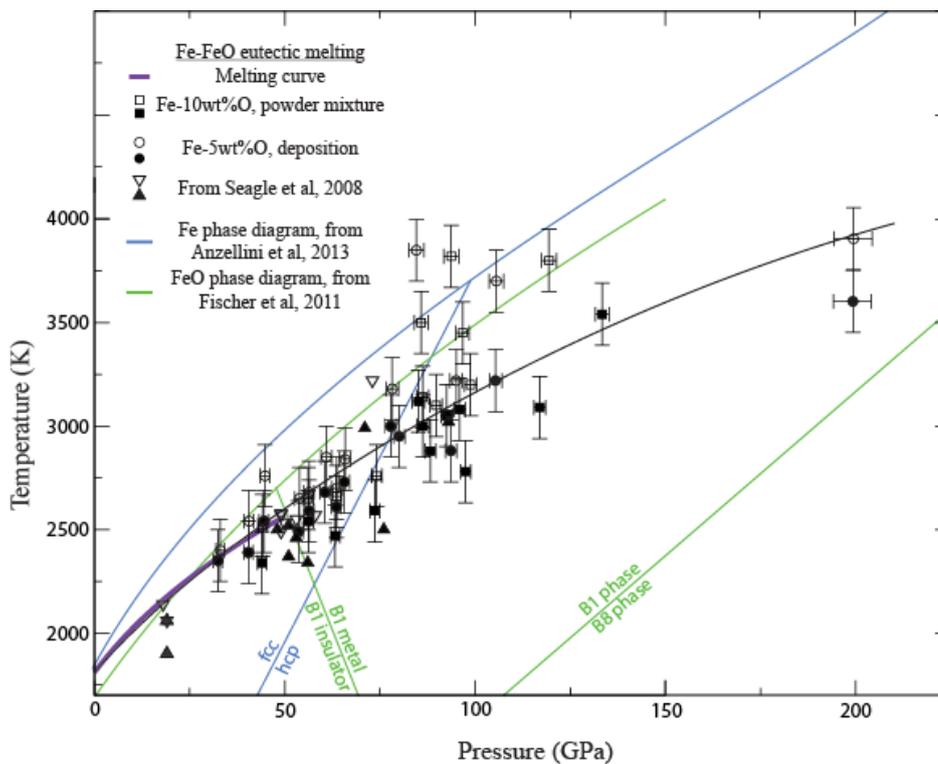


Figure A : The eutectic melting temperature in the Fe-FeO system as a function of pressure. Open symbols correspond to the temperature of the first diffraction pattern where the diffuse scattering signal was recorded, while solid symbols correspond to the temperature of the last diffraction pattern in which no such diffuse signal was seen. Two different starting materials

were used in this experiment: a mechanical mixture of Fe and FeO powders (square symbols) and a uniform alloy obtained by sputtering deposition (circle symbols). Good agreement is observed between the two starting materials. Triangles indicate eutectic melting temperatures reported in a previous experiment (Seagle, Heinz, Campbell, Prakapenka, & Wanless, 2008). Pure FeO and pure Fe phase diagrams are also shown in this figure (Anzellini, Dewaele, Mezouar, Loubeyre, & Morard, 2013; Fischer & Campbell, 2010).

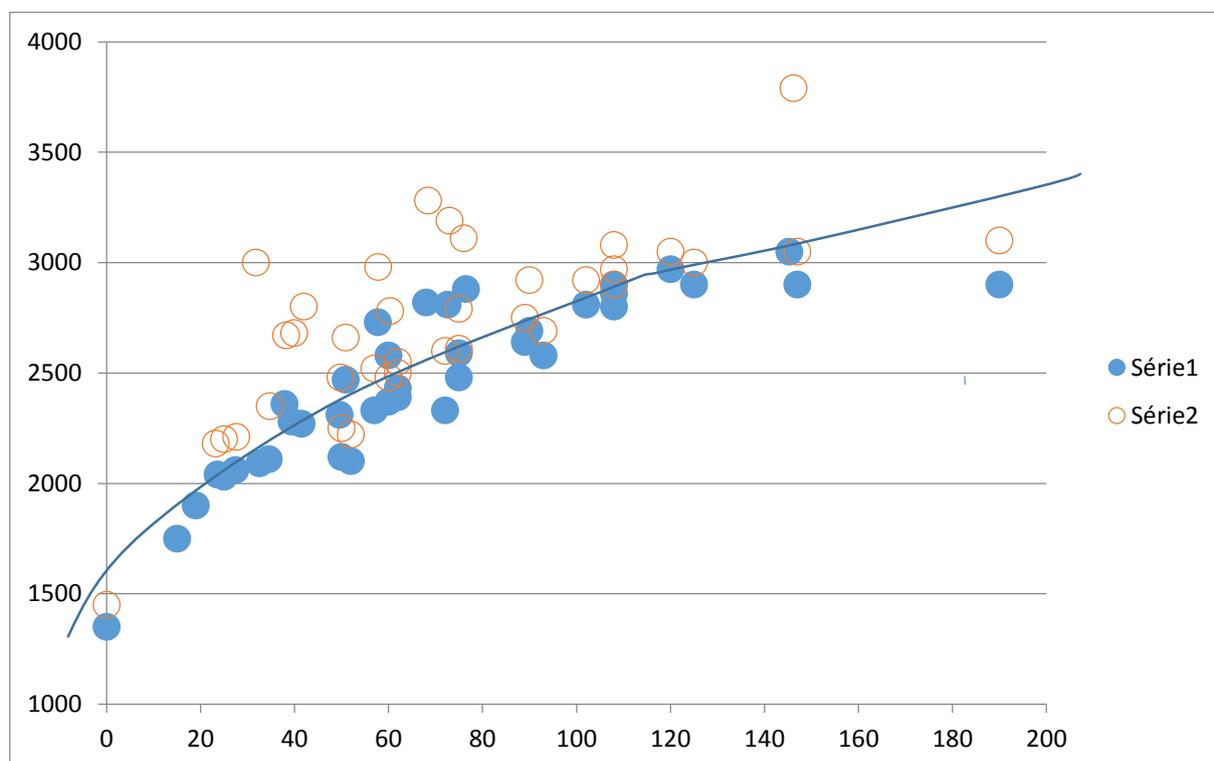


Figure B : The eutectic melting in the Fe-Fe₃C system. Legend is similar to Figure A.

In addition to the eutectic melting temperature Fe-C system, we were able to determine the eutectic composition by Rietveld refinement of the quench sample after melting (the sample were reheated to ~1500 K and fine recrystallization occurred, allowing Rietveld refinement of the diffraction pattern). Also, stability of Fe₃C up to 200 GPa before melting has been noticed in this experiment, in strong disagreement with previous lower pressure experiments (Lord, Walter, Dasgupta, Walker, & Clark, 2009) or thermodynamic studies (Fei & Brosh, 2014), that predicted the dissociation of Fe₃C under core conditions to form Fe₇C₃.

Test for sample encapsulation into single crystal sapphire were also performed on Fe-S samples (Figure C). This new technique could help to maintain the liquid iron alloys under high pressure and high temperature into the X-ray beam. Unfortunately, we did not succeed to heat the cell with the YAG lasers, due to insulation problem under extreme pressure, but this tricky loading, performed by Y. Kuwayama, is really promising for future study of liquid iron alloys under extreme conditions.

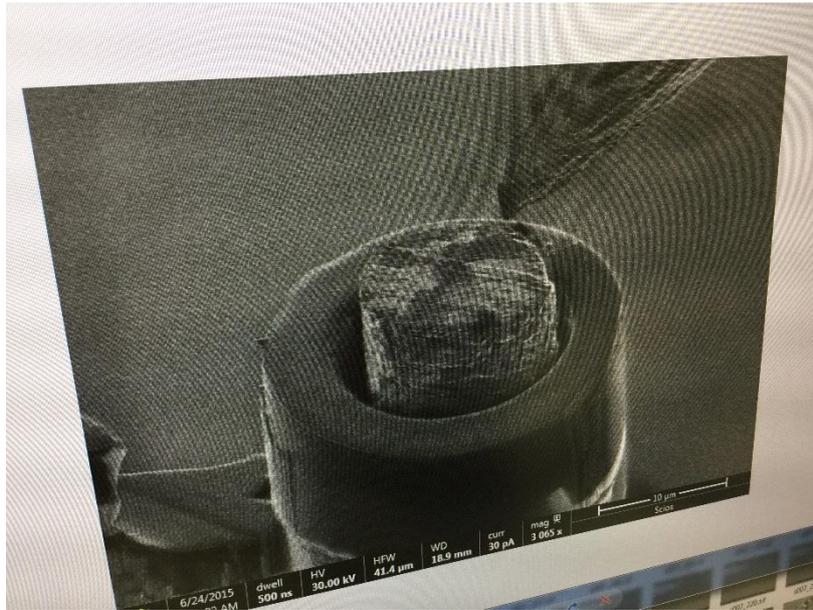


Figure C: SEM image of Fe-S sample loaded into a single crystal sapphire. All the elements were cut at the nanometer scale using FIB technique. These pieces were then loaded into a diamond anvil cell.

References

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