ESRF	Experiment title: P-V-T Equations of State in the Fe-Si and Fe-C Systems at Extreme Conditions	Experiment number: HC-3080
Beamline: ID27	Date of experiment: from: 20 Apr 2017 to: 25 Apr 2017	Date of report: 01/09/2017
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High-pressure and high-temperature x-ray diffraction (XRD) measurements were carried out at beamline ID27 on Fe and on a series of Fe-Si and Fe-Ni-Si alloys.

Methods

High-quality polycrystalline samples of Fe-Si and Fe-Ni-Si alloys were synthetized either by PVD or by an ultra-rapid quench method starting from Fe (99.9%, GoodFellow), Ni (99.9%,GoodFellow), and Si (99.9%, GoodFellow). Composition and homogeneity below 1 micron scale of the final products (Fe-5wt%Si, Fe-10wt%Si, Fe-5wt%Ni-5wt%Si, Fe-5wt%Ni-10wt%Si and Fe-5wt%Ni-15wt%Si) were confirmed by scanning electron microscopy and by electronic microprobe.

Angle dispersive 2D diffraction patterns were collected at each investigated pressure and temperature point with a monochromatic wavelength of 0.3738Å (iodine K edge). X-ray beam was focused at sample position to 3x3 μm^2. All data were calibrated against measurements made on a CeO_2 standard.

Membrane-drive diamond anvil cells (DACs) were equipped with diamonds with culet diameters ranging from 250µm to 100µm depending on the target pressures of each experimental run. For all experiments, Re was used as the gasketing material. For ambient temperature experiments, Ne was used as the pressure-transmitting medium (PTM) to ensure quasi-hydrostatic compression. Additionally, Pt and Mo were loaded together with samples and used as internal pressure calibrants. For high-temperature experiments, the samples were sandwiched between 5-15 µm thick, dry KCl plates (depending on sample chamber dimensions), which were used as both the PTM and pressure calibrant. This setup maximizes thermal isolation of the sample from the diamonds and inhibits chemical reaction between the Fe-alloys and the diamonds. Double-sided laser heating was performed with the YAG lasers (λ= 1.064 µm, hot spot diameter ~20 µm) available on ID27. The sample temperature was measured by spectroradiometry, by a Wein fit to the measured graybody spectra. Between 3 and 5 temperature measurements were taken during each diffraction pattern measurement. For each diffraction pattern, the collection time was 30-60 seconds.

Experiments

At 300K in Ne PTM, Fe-5wt%Si was measured to 1.1 Mbar (Fig. 1) and Fe-10wt%Si was measured to 1.3Mbar.

High-temperature isotherms were measured for: Fe at 1500K to 70 GPa, Fe-5wt%Si at 1500K and at 2150K to 100 GPa (Fig. 1), Fe-5wt%Ni-5wt%Si at 1500K and 1850K to 90 GPa. The temperature stability was remarkably good throughout all the experimental runs.

Data analysis is still ongoing for these experiments. Ambient temperature data will be combined with high temperature data to establish P-V-T equations of state for the investigated alloys.

Additionally, high temperature measurements were successfully performed at selected pressures on Fe-5wt%Ni-10wt%Si and Fe-5wt%Ni-15wt%Si up to 2700K and 90 GPa and up to 2400K and 50 GPa, respectively. These *in situ* data, coupled with in-house SEM measurements of recovered samples will allow for the assessment of the phase diagram of the Fe-Ni-Si system and for the estimation of the Si solubility in Fe-Ni alloys at high P-T conditions.

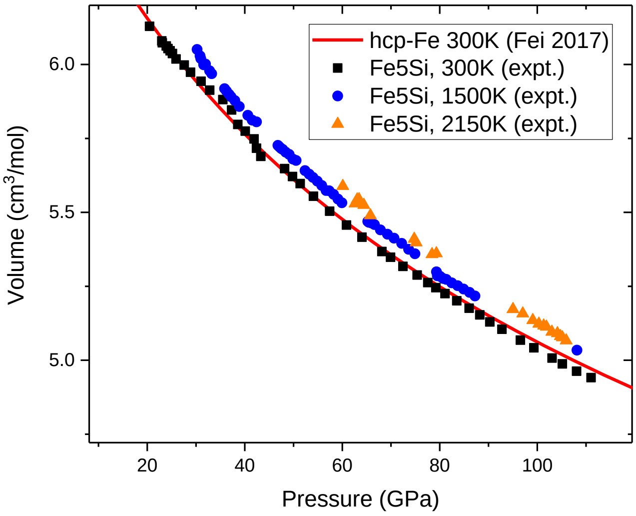

Fig. 1 – P-V isotherms of Fe-5wt%Si measured at 300K, 1500K and 2150K. Red line: literature equation of state for hcp-Fe at 300K.

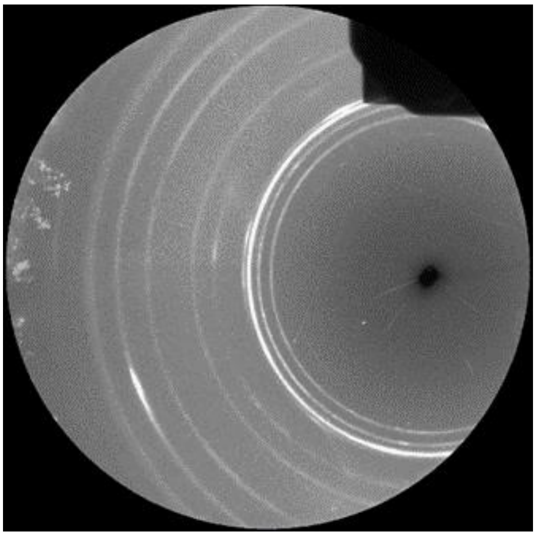

Fig. 2 – 2D diffraction pattern of Fe-5wt%Si in Ne at 108 GPa.