Fe8wt%Si Alloy at High Pressure and High Temperature

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Introduction

Iron is believed to be the most abundant element in the Earth's core. The phase diagram of iron has been extensively studied[1]. Body-centered cubic iron (bcc) transforms to the hexagonal close-packed phase (hcp) under high pressures, and the bcc phase transforms into the face-centered cubic phase (fcc) under high temperatures.[1] A high-pressure and high-temperature polymorph (β-phase) of iron has also been reported by various groups .[2,3] Studying the structure and properties of the phase diagram of iron plays an important role in understanding the properties of the Earth's core.

Silicon has been traditionally proposed to be an important alloying element in the Earth's outer core.[4] Iron-rich Fe-Si alloys are in the bcc structure under ambient conditions. The thermal equation of state (EOS) of Fe9wt%Si alloy in the bcc phase was studied in a cubic anvil apparatus,[5] and the bcc-fcc phase transformation was observed to occur at increasingly high temperatures between 6-9 GPa6. A bcc-hcp phase transformation in Fe8wt%Si began at 16 GPa and was completed at 36 GPa.[7] These studies indicate that iron-rich Fe-Si alloys have similar phase transformation behaviors in the pressure and temperature range studied.[7] However, the pressure and temperature range of previous studies is not comparable to the conditions at the Earth's core. Therefore, we studied Fe8wt%Si alloy in a laser-heated diamond anvil cell (DAC) in order to understand the effect of Si on the phase diagram of iron-rich Fe-Si alloys.

Materials and Methods

A powdered sample of Fe8wt%Si alloy from Goodfellow Corporation was used as the starting material. The sample was in the bcc structure with lattice parameter a = 2.8520 (+0.0040) Å (number in the brackets represents standard deviation), and an electron microprobe analysis showed that the sample contained 7.9 (+0.3) wt% Si (averaged from 5 analyses). The sample was ground into a fine powder (~1 μm grain size) and precompressed in a diamond anvil cell (DAC) to make a thin, flat disk. Stainless steel or rhenium gaskets were preindented to about 25 μm thickness, and a hole of 100 μm was drilled through them. The size of the samples in the DAC was about 50 μm and the thickness was about 10 μm. A sandwiched sample configuration, using NaCl, MgO, or Ar as the thermal insulator and pressure medium, was used in this study.[8] The thickness of each insulating layer was about 7 μm. When NaCl or MgO was used as the insulator, the diamond cell was heated for an hour at 100°C in a vacuum oven after sample loading, to eliminate water contamination in the insulator.

A double-sided laser heating system at 13IDD, GSECARS (APS, ANL) was used in this study.[8] A Nd:YLF laser operating in "donut" (TEM_{01}) mode or a multimode (TEM_{00}+TEM_{01}) was used to heat the sample from both sides of the DAC. The laser beam diameter was about 20 μm. Since the fluorescence of the x-ray beam on the sample and insulating materials were visible to a sensitive CCD camera, we were able to align the x-ray beam and the laser heating spot precisely together. The optical system was designed for *in situ* temperature measurement, and temperatures were determined by fitting the thermal radiation spectrum between 670 and 830 nm to the Planck radiation function.[8] In most of the experiments, the laser power and thermal radiation from the laser-heated sample were monitored for stabilizing temperatures by negative feedback to the laser power supply. The sample was laser-heated for about 1-5 min at each temperature, and thermal radiation spectra and x-ray diffraction patterns were collected during this time. The average standard deviation of temperature measurements was about 50 K.

A white beam or a monochromatic beam (wavelength = 0.4246 Å) was used as the x-ray source for energy-dispersive or angle-dispersive experiments, respectively. The synchrotron x-ray beam size was about 8 μm in diameter. The diffracted X-rays were collected by a germanium detector at a fixed angle (2θ) of about 8° in the energy-dispersive experiments, or by a CCD in the angle-dispersive experiments. Pressures were calculated from the room-temperature equation of state of NaCl[9] or MgO[10], or by the ruby fluorescence pressure scale in the Ar medium.[11] No thermal pressure corrections were made.

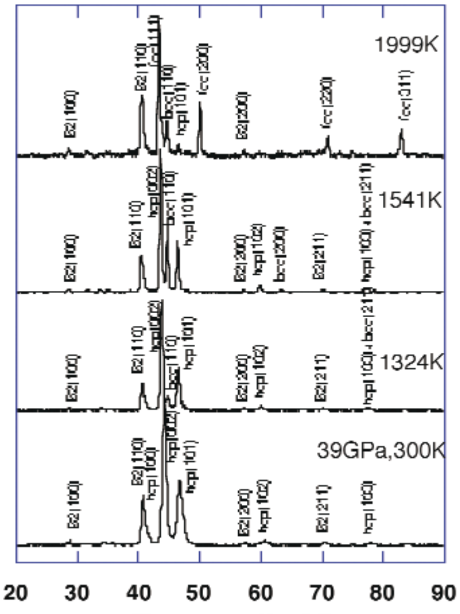

FIG. 1. A series of x-ray diffraction patterns of Fe8Wt%Si at 39 GPa during increasing temperature. The hcp-bcc phase transformation occurred at 1324 K, and the bcc-fcc phase change occurred above 1742 K. B2 indicates the high-pressure structure of NaCl, which was used as the insulator and pressure calibrant.

Results and Discussion

The Fe8wt%Si alloy was laser-heated in a DAC between 14 and 84 GPa and up to 2400 K, and the x-ray diffraction patterns were collected simultaneously. Figure 1 is a representative series

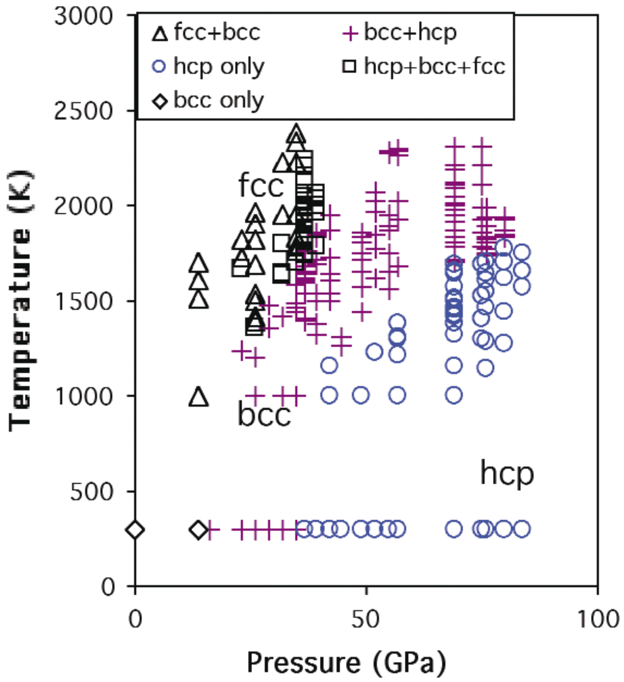

FIG. 2. The pressure-temperature phase diagram of Fe8wt%Si alloy. Open circles=hcp phase only; open diamonds=bcc phase only; crosses=bcc+hcp; open triangles=fcc and bcc; open squares=hcp+bcc+fcc. Multiple phases were observed in most of the experiments, indicating a phase loop in between hcp+bcc and fcc+bcc. Electron microprobe analyses of the quenched samples also show that Si partitioning occurred in between phases. Both hcp-bcc and bcc-fcc phase boundaries have a positive slope; but the bcc-fcc phase boundary is steeper than the hcp-bcc phase boundary.

of x-ray diffraction patterns under high temperatures at 39 GPa. A hcp-bcc phase transformation was found at 1324 K, and, upon further heating, the fcc phase was detected at 1963 K, where the bcc and fcc phases coexist. The P-T phase diagram of Fe8wt%Si alloy is plotted in Fig. 2. The cell parameters of the bcc phase and hcp phases at 39 GPa and 1541 K are listed in Table 1. The volume difference between the two phases is about –1.0%; the bcc phase has a slightly smaller unit cell volume than the hcp phase.

Our study indicates that a few weight percent of silicon in iron may significantly change the phase diagram of iron. The phase diagram of pure Fe has a triple point at 8.3 GPa and 713 K, beyond which the bcc phase does not exist. However, this study has shown that alloying Fe with a few weight percent of Si stabilizes the bcc phase to much higher pressure and temperature conditions (Fig. 2). This observation has profound implications for the structure of the proposed Si-bearing inner core of the Earth.

Table 1. Cell parameters of hcp and bcc phase at 39 GPa and 1541 K. Volumes are listed on a per atom basis.

hcp	**d-spacing, 1541 K**	**bcc**	**d-spacing, 1541 K**
(100)	2.1235	(110)	1.9269
(002)	1.9752	(200)	1.3610
(101)	1.8572	(211)	1.1148
(102)	1.4397		
(110)	1.2276		
(103)	1.1148		
a (Å)	2.452 (0.0080)	a (Å)	2.7259 (0.0044)
c (Å)	3.930 (0.0110)		
V(Å3)	10.231 (0.073)	V (Å3)	10.127 (0.049)

Acknowledgments

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