I075-005 Room: IC Time: May 27 14:30-14:45

Exploration of beta-Fe using Kawai-type apparatus equipped with sintered diamond

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Based on experiments using diamond anvil cell (DAC), several groups have claimed that a new polymorph of iron called beta phase is present under conditions higher than 35 GPa and 1500 K. Saxena et al. (1995) have proposed the double-hexagonal close-packed (dhcp) iron whereas Andrault et al. (1997) have maintained an orthorhombic (Pbcm) iron. In contrast, beta phase(s) was not found up to 84 GPa by Shen et al. (1998). Since the stability and structure of beta phase could be essential for understanding the constitution and state of Earth's inner core, we have explored the stability of beta iron using double-staged multianvil (Kawai-type apparatus) equipped with sintered diamond (SD).

MgO powder was mixed with the iron powder in a 1:1 weight ratio to suppress grain growth of the iron at high temperature. High pressure was achieved by compressing cubic anvils of SD in a press SPEED 1500 at SPring-8. The mixture of iron + MgO was put into an MgO sleeve which served as the pressure standard. The sleeve was set at the center of a cylindrical rhenium heater. Fine powder of diamond was packed into hollow spaces of the heater. A W97/Re3-W75/Re25 thermocouple was in contact with the central outer surface of the rhenium heater.

High-pressure in situ X-ray diffraction experiments were performed using an energy-dispersive detector and synchrotron radiation. The sample and the MgO standard were irradiated independently by incident white X-rays collimated to 0.05 mm horizontally and 0.1 mm vertically. The diffracted beam entered a Ge solid-state detector through a collimator of 0.05 mm width to ensure a diffraction angle 2Theta of ca. 6 degrees. The pressure was determined from the measured unit cell volume of the MgO standard using the equation of state for MgO by Jamieson et al. (1982).

We made four successive runs. Here we describe the results of run S614, in which highest pressure of 44 GPa was achieved. In the first heating cycle at 662 tons, iron initially assumed the hcp structure (epsilon iron) at 37(0.4) GPa and 300 K. Upon heating, diffraction peaks of the fcc structure (gamma iron) appeared at 1450(100) K and 38.4(0.8) GPa and intensified simultaneously with reduction of epsilon phase peaks as temperature increased to 1850(130) K and 39.2(1.1) GPa. A substantial amount of epsilon phase still survived at ca. 1850 K due to the temperature gradient through the sample. Upon cooling, enhancement of peaks for epsilon phase was noticed with a simultaneous reduction of peaks for gamma phase at ca.1350 K and 37.1 GPa, but a certain amount of epsilon phase remained even at 300 K and ca. 34 GPa, indicating a slow reaction rate for the gamma to epsilon transformation. Next the press load was increased to 803 tons, and the second heating was started at 300 K and ca. 36 GPa. No change in the diffraction pattern of iron was observed up to 1700 K. At 1750(120) K and 41.9(1.0) GPa, however, the growth of gamma phase was recognized together with reduction of epsilon phase, which was rapid with increasing temperature up to 2100(150) K and 44(1.8) GPa. Then the sample was quenched to 300 K by shutting off the power supply.

We carefully searched for reflections characteristic of dhcp-iron and orthorhombic iron in our in situ diffraction profiles, but all were absent. In conclusion, the results of the present study do not support the existence of beta phase with the dhcp structure or the orthorhombic structure (Pbcm) as a stable polymorph of iron. The beta phase(s) has mostly been observed in experiments where iron was compressed in a DAC without pressure medium or in solid media. In the present study using Kawai-type apparatus, the sample is kept in quasi-hydrostatic condition because an octahedral medium is squeezed in the eight equivalent directions. Therefore, it is possible that beta phase(s) might be present under the environment of strong deviatoric stress.