

In situ X-ray observation of a new high-pressure phase of FeS

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Iron sulfide (FeS) has been supposed to be one of the major components of the core of terrestrial planets such as Mars. In the recent decade, in situ X-ray diffraction studies using multi-anvil high pressure apparatus provided some important data on the phase stability relationship of FeS at high pressure and high temperature. The results showed that FeS (troilite; hexagonal NiAs-type related phase) transforms to FeS II (orthorhombic MnP-type phase) above 3.4 GPa and then to FeS III (monoclinic phase) above 6.7 GPa by room temperature compression (King and Prewitt, 1982, Kusaba et al., 1997; Nelmes et al., 1999). Above room temperature, FeS undergoes two successive phase transitions, from FeS to FeS IV (hexagonal phase) and then to FeS V (NiAs-type phase) with increasing temperature in the pressure range from 0 to 20 GPa (Fei et al., 1995; Kusaba et al., 1998; Urakawa et al., 2004). However, no studies have yet been conducted to investigate the phase stability of FeS under higher pressure conditions.

High pressure in situ X-ray experiments were performed using a laser heated diamond anvil cell (LHDAC) at BL10XU of Spring-8. Powdered reagent grade FeS (troilite) was loaded into a 80 micron hole drilled in rhenium gasket together with a pressure medium of MgO polycrystalline pellets. The sample was compressed with 250 micron culet diamond anvils and heated with a focused Nd: YAG laser using a double-side heating technique (Watanuki et al. 2001). Pressures were estimated by the volume of MgO before and after heating at room temperature.

Several X-ray diffraction patterns were collected during room temperature compression of FeS up to 80 GPa. Above 20 GPa, all of the collected patterns are similar to those of FeS III phase reported. However, some of the major diffraction peaks (e.g. 001, 11-1, 221, 302 in Kusaba et al., 1997) are missing. This is possibly due to the non-hydrostatic compression of the sample in DAC, or may suggest that the patterns are brought from a different but similar structure. During heating at 1300 K and 80 GPa, two strong sharp peaks with d-spacings of 2.11 and 1.49 were newly appeared on the diffraction patterns. The strong peaks do not correspond with the diffraction peaks of any high pressure and high temperature phases of FeS previously reported, even considering the unit-cell compression under such high-pressure conditions. With increasing heating temperature up to 2000 K, the two peaks ($d = 2.11, 1.49$) became stronger and sharper. These facts suggest that a new phase transformation of FeS occurred at high pressure and high temperature. The two diffraction peaks remained just after quench to room temperature, but completely disappeared within 30 minutes, suggesting that the new FeS phase is only stable at high pressure and high temperature.

We are now looking for possible candidates for the crystallographic structure of the new FeS phase. The new two peaks can be indexed by cubic unit cell ($a = 4.22$). Estimated density is about 5 % lower than that of FeS III at 70 GPa.