

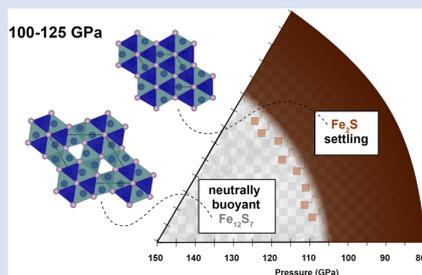
Stability of Fe₂S and Fe₁₂S₇ to 125 GPa; implications for S-rich planetary cores

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Abstract



The Fe-FeS phase relations were explored in the 22–25 wt. % S compositional range using single crystal X-ray diffraction in a laser heated diamond anvil cell. At pressures up to 125 GPa and at high temperatures, Fe₂S and Fe₁₂S₇ were determined to co-crystallise. The novel Fe₁₂S₇ compound adopts the Co₁₂P₇ structure and Fe₂S assumes the Fe₂P-type structure. Applying these results to an Fe-FeS binary phase diagram exposes a complex series of FeS phase assemblages in the 16–25 wt. % S range, whereby minor changes in S content significantly affect the crystallisation sequence of Fe-S rich planetary cores. For core compositions S-rich of the Fe₂S-Fe₁₂S₇ eutectic, the small density difference between solid Fe₁₂S₇ and Fe₂S is likely to result in the formation of a core slush rather than a gravitationally stable inner core.

Crystallisation of denser Fe₂S at eutectic conditions could then result in gravitational settling of an Fe₂S-rich inner core over time. As the Fe₂P-type Fe₂S has previously been identified forming at high temperatures to pressures as low as 22 GPa, the core crystallisation regimes determined here also elucidate that the Martian core sulfur composition must lie on the S-rich side of the Fe-Fe₃S eutectic or even the S-rich side of the Fe₃S-Fe₂S eutectic to maintain a fully molten core.

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Introduction

Earth and other terrestrial planets are composed of silicate mantles and Fe-alloy metallic cores (e.g., McDonough and Sun, 1995; Righter and O'Brien, 2011). Sulfur easily alloys with and lowers the melting temperature of iron (Fei et al., 2000; Campbell et al., 2007; Kamada et al., 2012; Mori et al., 2017), and plays a key role in early metal-melt formation and differentiation (Murthy and Hall, 1970; Kruijer et al., 2014; Terasaki et al., 2008). Therefore, the Fe-FeS phase equilibria and eutectic melting relations relevant to the *P-T-X* conditions of a given planetary core play a significant role in the core's evolving structure as it crystallises over time.

The terrestrial planets each have different interior structures, oxidation states, and proposed core sulfur contents. Cosmochemical models and metal-silicate partitioning experiments estimate that Earth's core contains ~2 wt. % sulfur (McDonough, 2003; Suer et al., 2017), and the sulfur content of the core is limited to <6 wt. % to explain the presence of an Fe-rich inner core (Mori et al., 2017). Mercury is small and dense, with a large core-mass fraction and a reduced core composition with sulfur contents <1.5 wt. % (Smith et al., 2012; Namur et al., 2016; Genova et al., 2019). Recent analysis of Marsquakes and geodetic data from the *InSight* mission reveal that Mars' core accounts for ~½ the planet's radius and is fully molten, such that it likely has a high sulfur content (Stähler et al., 2021). In a purely Fe-S Martian core, 25 wt. % sulfur is required

to explain the observations, while in a multicomponent core with cosmochemically plausible light element concentrations, a minimum of 10–15 wt. % sulfur is required (Stähler et al., 2021). Less is known about Venus: its core is likely fully molten, due to the lack of heat release through plate tectonics (Nimmo, 2002), which lends little constraint on how the core composition contributes to the core structure.

Reflecting on the 6–360 GPa pressure range and 1.5–25 wt. % potential sulfur concentrations in the terrestrial planetary cores in our solar system alone, it is inevitable that a much wider variety of core structures and compositions exists in planetary bodies outside of our solar system. Continuing to build the Fe sulfide phase diagram with pressure, temperature and composition is critical for interpreting core crystallisation in our solar system and beyond. Here we report new findings on the high *P-T* polymorphism of Fe₂S (22 wt. % S) and the discovery of Fe₁₂S₇ (25 wt. % S) to 125 GPa, and use these findings to model the structures of planetary cores with 16–25 wt. % S.

Experimental Methods

High pressure-temperature conditions were achieved on Fe₆₆S₃₄ (by atom) samples using a laser heated diamond anvil cell. Powder and single crystal X-ray diffraction techniques were employed during heating and after temperature quenching, respectively, at beamline 13 ID-D of the Advanced Photon

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Source, Argonne National Lab. Analysis of the diffraction data is described in detail in the [Supplementary Information](#).

Synthesis and Structure Determination of Fe_2S and Fe_{12}S_7

Two high P - T experiments were conducted at 105.3 (9) GPa (values in parentheses are propagated uncertainties on the last reported digits) and quenched from 2400 (120) K and 125 (1) GPa and quenched from 2260 (140) K ([Table S-2](#)). After each heating, rotational diffraction scans were collected at various locations around the heated region to assess the phase relations. At each pressure step two distinct lattices were identified at the centre of the laser heated spot and are likely the relevant high temperature sulfides at these P - X conditions ([Fig. 1](#)).

One set of grains was indexed to a hexagonal cell with parameters $a = 5.326$ (7) Å and $c = 3.125$ (3) Å, compatible with

3 formula units of Fe_2S ([Table 1](#), [Fig. 1a](#)). The systematic absences identified for this hexagonal phase are compatible with a $P-62m$ space group, and a Fe_2P -type (Pearson symbol: $C22$, $Z = 3$) structure was solved and refined to the data ([Table 1](#), [Table S-2](#), [Fig. 1b](#)) ([Rundqvist and Jellinek, 1959](#)). This $C22$ structure, shown in [Figure 1b](#), is composed of 4 fold coordinated and 5 fold coordinated iron sites arranged into columns of edge sharing square pyramids (green) and columns of corner sharing tetrahedra (blue) linked along edges in the c direction ([Fig. 1a](#); [Appendix A-1](#)). Additional Co_2P -type and Cr_2P -type Fe_2S polymorphs were identified in the lower temperature regions of the laser heated spot after each run and discussion of their structures are provided in the [Supplementary Information](#).

The second set of grains was indexed to a hexagonal cell with $a = 7.794$ (2) Å and $c = 3.1234$ (8) Å, compatible with 1 formula unit of Fe_{12}S_7 ([Table 2](#), [Fig. 1c](#)). Crystal structure solution and refinement reveal that Fe_{12}S_7 adopts the Co_{12}P_7 structure ($P-6$, $Z = 1$) ([Zurkowski et al., 2020](#)) ([Fig. 1d](#); [Appendix A-2](#)).

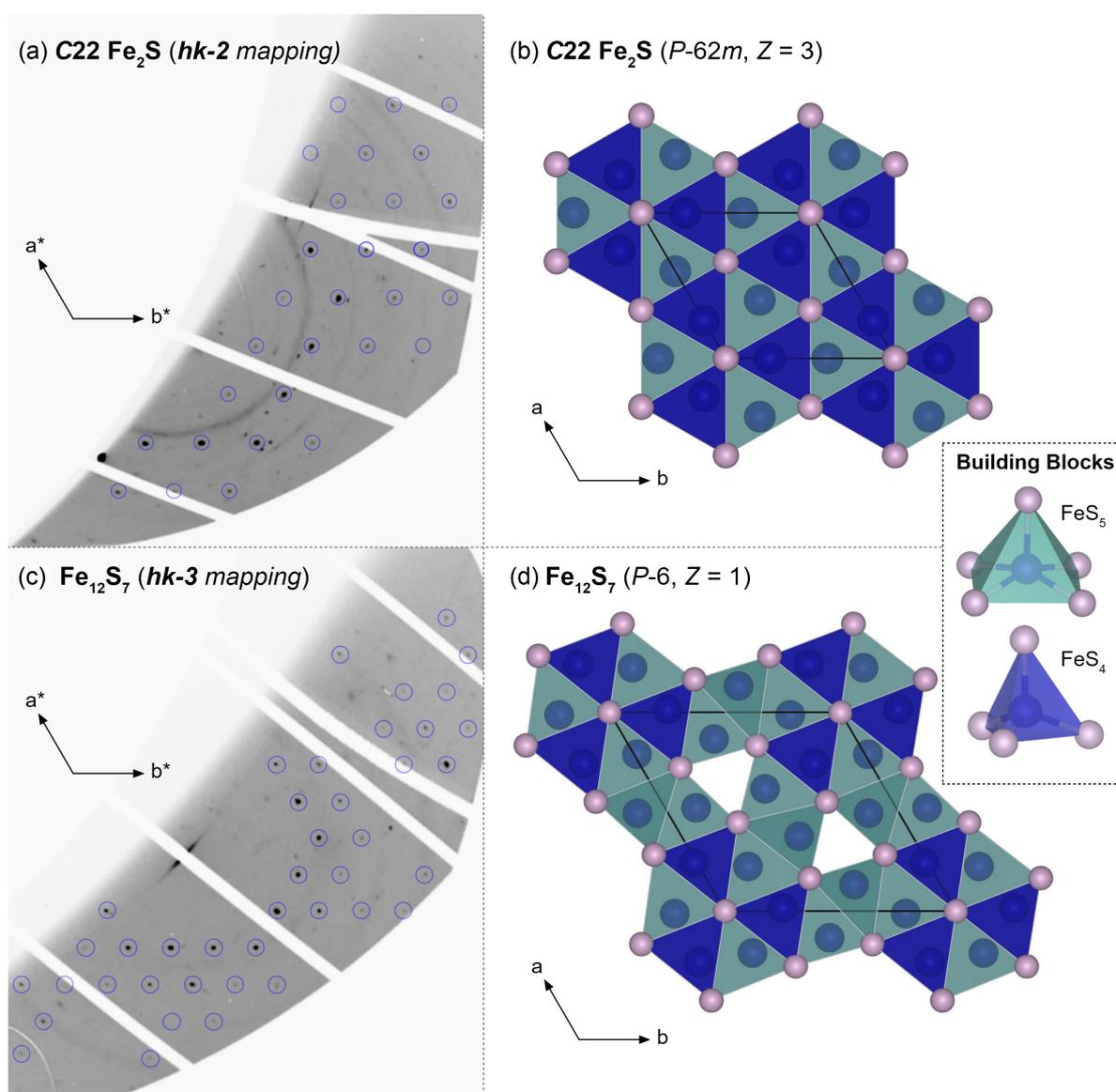


Figure 1 (a) Diffraction mapping of the hexagonal Fe_2S lattice collected at 105 GPa upon quenching from 2400 K. The reflections shown all satisfy the condition $hk-2$. (b) The crystal structure of Fe_2P -type Fe_2S (Pearson symbol $C22$). (c) Diffraction mapping of an Fe_{12}S_7 lattice identified at 105 GPa upon quenching from 2400 K. The reflections shown all satisfy the condition $hk-3$. (d) The crystal structure of Co_{12}P_7 -type Fe_{12}S_7 . The FeS_5 and FeS_4 building blocks observed in both the Fe_2S and Fe_{12}S_7 structures are shown in the centre-right inset.

Table 1 Unit cell parameters, volumes, and densities of hexagonal Fe₂S and Fe₁₂S₇ indexed at each pressure step upon temperature quenching. Values in parentheses are propagated uncertainties on the last reported digits.

Phase	<i>P</i> (GPa)	Synthesis <i>T</i> (K)	<i>a</i> (Å)	<i>c</i> (Å)	<i>V</i> (Å ³)	<i>Z</i>	ρ (g/cm ³)
Fe ₂ S	105.3 (9)	2400 (120)	5.340 (3)	3.133 (2)	77.38 (9)	3	9.27 (1)
	125 (1)	2260 (140)	5.282 (5)	3.062 (4)	74.0 (2)		9.69 (1)
Fe ₁₂ S ₇	105.3 (9)	2400 (120)	7.794 (2)	3.1234 (8)	164.33 (8)	1	9.05 (1)
	125 (1)	2260 (140)	7.700 (7)	3.104 (4)	159.4 (3)		9.32 (1)

Table 2 Atomic coordinates refined for the C22 Fe₂S and Fe₁₂S₇ structure models at 105 GPa.

Phase	Site	<i>x</i>	<i>y</i>	<i>z</i>	sof	U11	U22	U33	U23	U13	U12	Ueq
C22 Fe ₂ S	Fe1	0.5990	0	-1/2	1/4	0.010	0.010	0.009	0	0	0.0052	0.0095
	error	0.0008				0.001	0.001	0.002			0.0007	0.0008
	Fe2	0.2594	0	0	1/4	0.009	0.008	0.011	0	0	0.0041	0.0092
	error	0.0006				0.001	0.002	0.001			0.0008	0.0007
	S1	2/3	1/3	0	1/6	0.007						
	error					0.001						
Fe ₁₂ S ₇	S2	0	0	1/2	1/12	0.011						
	error					0.002						
	Fe1	0.0151	0.2549	0	1/2	0.010	0.020	0.014	0	0	0.005	0.0159
	error	0.0006	0.0006			0.001	0.002	0.001			0.001	0.0008
	Fe2	0.1335	0.6233	0	1/2	0.009	0.010	0.014	0	0	0.004	0.0112
	error	0.0006	0.0005			0.001	0.001	0.002			0.001	0.0009
	Fe3	0.2324	0.2178	1/2	1/2	0.032	0.024	0.012	0	0	0.024	0.0185
	error	0.0006	0.0007			0.002	0.002	0.001			0.002	0.0008
	Fe4	0.5170	0.1334	1/2	1/2	0.009	0.009	0.014	0	0	0.005	0.0106
	error	0.0005	0.0005			0.001	0.001	0.002			0.001	0.0009
	S5	0.1638	0.4476	1/2	1/2	0.011						
	error	0.0008	0.0010			0.001						
S6	0.446	0.281	0	1/2	0.013							
error	0.001	0.001			0.001							
S7	0	0	0	1/6	0.020							
error					0.002							

The Co₁₂P₇ structure is closely related to the C22 Fe₂S structure, in that it is composed of the same building blocks arranged into similar edge-sharing columns, but an increased ratio of square pyramid to tetrahedral building blocks results in the formation of trigonal channels along the *c* direction in Fe₁₂S₇ (Fig. 1d). Previous studies have asserted that this structure is not likely stable in Fe-rich systems as it is composed of a majority 5 fold coordinated sites (Dhahri, 1996), but this work reveals that this structure stabilises in the Fe-S systems with sufficient pressure and temperature.

Crystallisation Sequences in S-rich Planetary Cores

The novel Fe₂P-type Fe₂S (22 wt. % S) and Co₁₂P₇-type Fe₁₂S₇ (25 wt. % S) phases were then used to construct an updated binary *X-T* phase diagram between 100 and 125 GPa (Fig. 2). The Fe-rich portion (<16 wt. % S) of this phase diagram has been characterised at these conditions previously (e.g., Kamada *et al.*, 2012; Mori *et al.*, 2017), but the addition of this study exposes the complexity of Fe-S phase assemblages in the limited 16–25 wt. % S range: Fe₃S + Fe₂S (16–22 wt. %) and Fe₂S + Fe₁₂S₇ (22–25 wt. %) (Fig. 2a).

As the structures of planetary cores are dictated by the density difference between the crystallising phases and remaining liquid, these Fe-S phase relations determine the possible S-rich core crystallisation regimes for an approximately Venus-sized planet (Aitta, 2012), such as the rocky exoplanet TRAPPIST-1e ($R_{T1e} = 0.96 \times R_{Venus}$) (Grimm *et al.*, 2018). The visualisations in Figure 2b depict the intricacies of these S-rich crystallising cores. With core compositions ranging from 16–22 wt. % sulfur, two crystallisation models are possible: Fe₃S may crystallise into the inner core leaving a lower density, more S-rich liquid outer core or, for more S-rich compositions, Fe₂S would be the crystallising phase from a more Fe-rich liquid, producing gravitationally buoyant 'snow' that would remix and not produce a solid inner core (Fig. 2b; Scenarios 1, 2).

Then for 22–25 wt. % sulfur concentrations, further variations are possible. A gravitationally stable Fe₂S-rich inner core could form for core compositions on the Fe-rich side of the Fe₂S-Fe₁₂S₇ eutectic (Fig. 2b; Scenario 3). Alternatively, for more S-rich compositions shown in Scenario 4 in Figure 2b, the crystallisation of Fe₁₂S₇ from a more Fe-rich liquid could result in a neutrally buoyant slush due to the near equivalent densities of an Fe₂S-rich liquid and Fe₁₂S₇ solid. At 105 GPa, a ~2.5 % volume increase of Fe₂S upon melting results in equivalent solid Fe₁₂S₇ and liquid Fe₂S densities. As changes in volume from

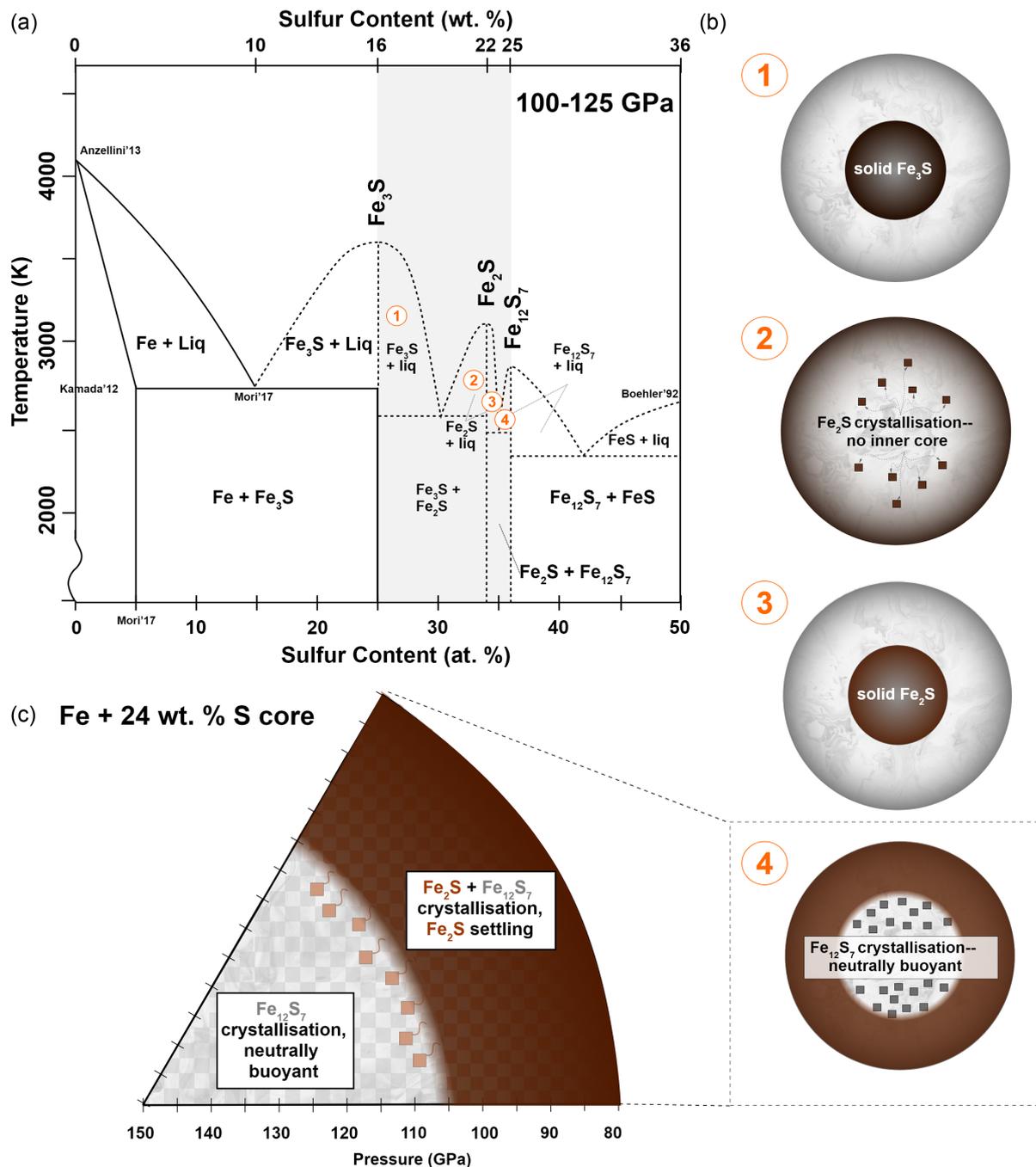


Figure 2 (a) Phase diagram for the Fe-FeS system between 100–125 GPa inferred from this study in the 16–26 wt. % S (25–36 at. %) range along with previous studies: Fe and FeS melting temperatures were taken from Anzellini *et al.* (2013) and Boehler (1992), and eutectic melting temperature and composition in the Fe-Fe₃S system are based on Kamada *et al.* (2012) and Mori *et al.* (2017). (b) In the 16–25 wt. % S range, four core crystallisation scenarios are possible depending on the crystallising phase and the density difference between it and the remaining core liquid. In case 4 in (b), the minimal density difference between solid Fe₂S and an Fe₂S-rich liquid, may result in a neutrally buoyant solid and liquid and the formation of a core slush. (c) A crystallisation model of an Fe + 24 wt. % S core spanning 80–150 GPa indicates that Fe₂S may begin to crystallise with Fe₁₂S₇ around 115 GPa, causing the denser Fe₂S to precipitate out and settle downwards.

15 % have been estimated upon melting in previous studies of iron-rich systems (Anderson 2003; Kuwayama *et al.*, 2020), these values credit the possibility of neutrally buoyant rather than density settling crystallisation in such an S-rich core. Once eutectic temperature and composition is reached in this system, the crystallisation of both Fe₁₂S₇ and Fe₂S will result in solidification of an inner core over time.

A core crystallisation model of an S-rich core was then constructed to further assess this scenario (Fig. 2c). The model

begins with a fully molten exoplanetary core with Fe + 24 wt. % S composition that spans an arbitrary pressure range of 80–50 GPa with radius of 950 km. These pressure-depth values were assumed using a similar relationship as constrained for Earth’s core. Equations of state of Fe and Fe₂S in this pressure range were then used convert from weight percent to volume percent of sulfur in the bulk starting liquid (Dewaele *et al.*, 2006; Zurkowski *et al.*, 2022). The model proceeds by crystallising along the S-rich side of the Fe₁₂S₇ eutectic in radial shells,

assuming temperatures are at the liquidus in each step and following the approximate Fe_{12}S_7 liquidus in Figure 2a. The volume fractions of Fe and S were then deducted from the remaining core liquid and the S content of the core liquid was recalculated. For this model core, eutectic conditions would be met around 115 GPa, resulting in the co-crystallisation of a denser Fe_2S with Fe_{12}S_7 (Fig. 2c). Depending on the dynamics of a specific iron sulfide core, further texturing of this multiphase solid inner core would be possible.

Applications for the Martian Core

Marsquake and geodetic data obtained from the recent *InSight* mission on Mars indicates a large, fully molten core requiring more sulfur-rich core compositions than previously thought (Stähler *et al.*, 2021). As grains of C22 Fe_2S have also been synthesised at 22 GPa and 1300 K (Koch-Müller *et al.*, 2002), it is likely that the C22 Fe_2S high temperature stability field extends from at least 19–125 GPa; encompassing pressures relevant to the Martian core (18–40 GPa) (Stähler *et al.*, 2021). Fe_3S is also known to be stable at the pressures spanning the Martian core; therefore, models of an iron sulfide Martian core may involve crystallisation of Fe_3S and/or Fe_2S , like the higher pressure phase diagram in Figure 2a. The regions of the phase diagram in Figure 2a that are Fe-rich of Fe_2S may therefore be generally applied to assess possible core S contents to sustain a molten core. Based on these core-structure models (Fig. 2), the Martian core sulfur composition must lie either on the S-rich side of the Fe- Fe_3S eutectic, such that Fe_3S is the crystallising phase from a more Fe-rich liquid, or even S-rich of the Fe_3S - Fe_2S eutectic, such that Fe_2S is crystallising from a denser Fe-S liquid. Core compositions more sulfur-rich than Fe_2S are geochemically unlikely (Steenstra and van Westrenen, 2018).

Conclusions

Here we present the novel characterisation of Fe_2S and Fe_{12}S_7 from *in situ* X-ray diffraction experiments conducted up to 125 GPa and apply these findings to exoplanetary core crystallisation regimes in the 16–25 wt. % sulfur range. The Fe_3S - Fe_2S - Fe_{12}S_7 phase stabilities based on core sulfur contents result in intricate core solidification structures including: gravitationally stable inner core crystallisation, low density sulfide snow and remixing in a sustained molten core, and neutrally buoyant sulfide crystallisation forming a core slush. As the stability fields of Fe_3S and Fe_2S also extend as low as 21 GPa, the phase relations Fe-rich of Fe_2S were generally applied to the molten Martian core to constrain that its sulfur contents must be S-rich of the Fe- Fe_3S eutectic or S-rich of the Fe_3S - Fe_2S eutectic. The presented experimental results and core crystallisation models highlight the sensitivity of core structure to sulfur content and the utility of the Fe-FeS phase relationships for interpreting seismically detected core structures.

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Additional Information

Supplementary Information accompanies this letter at <https://www.geochemicalperspectivesletters.org/article2217>.



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