Supplementary Information

Heavy iron isotopic composition of iron meteorites explained by core crystallization

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Description of the run products

A total of 11 solid-liquid metal equilibrium experiments are reported in this study. Their experimental conditions and the chemical compositions of the run products are summarized in Supplementary Table 1. Back-scattered Electron (BSE) images of the polished sections for OCT2017 and S10-1 are shown in Supplementary Fig. 4 as examples for the run products. As verified by electron microprobe analyses, the solid metal phase of each experiment is homogeneous in composition and contains 10 to 13 wt% Ni (Supplementary Table 1). On the other hand, the liquid metal phase shows dendritic quench textures which caused slight variation in the chemical compositions between each analysis, resulting in a calculated bulk composition with higher uncertainty than the solid metal, especially for S (~10% relative uncertainty, Supplementary Table 1). The quench textures in the liquid metal are composed of Fe-Ni dendrites and S-rich interstitial phases, similar to previous experiments (e.g. ref. 1). Although small amounts of Os, W, and Ru were also doped in the starting powder, their concentrations are too low to be precisely determined by electron microprobe using our analytical conditions, especially for the liquid metal phase with high S content. Thus, we do not report values for these elements in Supplementary Table 1, but note that totals being low by ~1 wt%, especially in the solid metal phase as these three elements exhibit S avoidance behavior in the Fe-Ni-S system, is consistent with the presence of these elements.

Comparing partition coefficient data with previous studies

In this study, our experiments were conducted in the Fe-Ni-S system, and the partition coefficients for Ni are obtained as a by-product, which provide a way for us to compare our experiments with previous studies. Although small amounts of Os, W, and Ru were also doped in the starting powder, their concentrations are too low to be precisely determined by electron microprobe for calculating partition coefficients, especially for the liquid metal phase with high S content. Therefore, here we focus on comparing our Ni partition coefficients with literature data to confirm that our experiments are consistent with previous solid-liquid metal equilibrium experiments. As plotted in Supplementary Fig. 5, Ni partition coefficients between solid metal and liquid metal show good agreement with literature data, demonstrating that our experiments are consistent with previous studies.

Extrapolation of the Fe isotope fractionation factor to other temperatures

The experiments in this study were conducted at 1260 to 1470 ºC to study the potential effect of S on the Fe isotope fractionation factor. Ideally, the best approach to quantify the effect of S is to conduct experiments with variable S concentrations at the same temperature. Practically, however, S concentration in the liquid metal is coupled with the equilibrium temperature based on the Fe-Ni-S phase diagram. It is impossible to decouple the two parameters without dramatically adjusting the Ni content of the system.
As a result, the approach adopted in this study is to conduct experiments at a range of temperatures to achieve various S contents in the liquid metal, and correct all experimentally determined fractionation factors to 1300 °C before assessing the effect of S. It has been shown since the early work\(^3\)\(^4\) that at high temperatures (e.g. > 400 °C), equilibrium isotope fractionation decreases as a function of one over the square of temperature. Therefore, all $\Delta^{57}\text{Fe}_{\text{solid-liquid}}$ data are corrected to their equivalent fractionation factors at 1300 °C using the following equation:

$$\Delta^{57}\text{Fe}_{\text{solid-liquid, } 1300\, ^\circ\text{C}} = \Delta^{57}\text{Fe}_{\text{solid-liquid}} \times \frac{\tau^2}{(1300+273.15 \, \text{K})^2}$$  \hspace{1cm} (S1)

The temperature correction resulted in a 3% to 23% change on the experimentally determined $\Delta^{57}\text{Fe}_{\text{solid-liquid}}$, relatively small compared to the analytical errors (>30%).

The temperature-corrected Fe isotope fractionation between solid metal and liquid metal, as shown in Fig. 2, does not show significant dependence on the S content of the liquid metal, although sulfur content of the liquid metal has been found to be a parameter dominating trace element partitioning between the solid and liquid metal (e.g. ref. 2). Besides S, other compositional variables including Ni, Os, W, and Ru are unlikely to play a significant role in controlling Fe isotope fractionation. The total amount of Ni has been controlled to about 10 wt% in the starting powder, hence the end products show limited variations of Ni content in the liquid metal (8 to 12 wt%) and solid metal (10 to 13 wt%). Plotting Fe isotope fractionation versus Ni content of the liquid metal has confirmed the lack of correlation between them. For Os, W, and Ru, only trace amount of each (~ 0.3 wt%) has been doped in the starting powder and a measurable effect on Fe isotope fractionation is unlikely.

Without measurable dependence on S or other compositions, the 11 experiments conducted in this study can be simply described by an error-weighted average of $\Delta^{57}\text{Fe}_{\text{solid-liquid, } 1300\, ^\circ\text{C}} = 0.129 \pm 0.067$ ‰ (2 s.d.), indicating that the solid metal becomes heavier in $\delta^{57}\text{Fe}$ by $0.129 \pm 0.067$ ‰ when in equilibrium with the liquid metal at 1300 °C. For the purpose of the crystal fractionation modeling, the experimentally determined $\Delta^{57}\text{Fe}_{\text{solid-liquid, } 1300\, ^\circ\text{C}}$ at 1300 °C is extrapolated to the temperature range of 930 to 1350 °C via:

$$\Delta^{57}\text{Fe}_{\text{solid-liquid}} = (0.129 \pm 0.067) \times \frac{(1300+273.15 \, \text{K})^2}{\tau^2} = 3.19(\pm1.66) \times 10^5/T^2,$$  \hspace{0.5cm} (S2)

in which $T$ is the temperature in K and $\Delta^{57}\text{Fe}_{\text{solid-liquid}}$ is the Fe isotope fractionation between the solid and liquid metal phases in per mil. Note that even though the fractionation factors at temperatures used for the model are obtained through extrapolation, it is applied to a limited temperature (930 to 1350 °C) requiring < 80 % extrapolation.

**Lack of sulfur dependence of Fe isotope fractionation between solid and liquid metal**

Sulfur concentration in the liquid metal has been shown to have a dominant influence on the solid metal – liquid metal partitioning behavior of trace elements (e.g. ref 2), but not Fe isotopes according to our experiments. It is possible that the effect of S on the bond strength of Fe in the liquid metal is not strong enough to be reflected in its isotope fractionation. Interestingly, a recent ab initio study reached the same conclusion that the S content of the liquid metal does not affect Fe isotope fractionation between solid and liquid metal. Meanwhile, experimental studies in the metal/silicate system found that S content in the metal could potentially have a strong effect on Fe isotope fractionation between metal and silicate\(^5\)\(^7\). It is necessary to point out that, the experimental data referred to in these studies all come from Shahar et al.\(^5\). Compared to the solid/liquid metal equilibrium experiments conducted in this study, the metal/silicate equilibrium experiments in Shahar et al.\(^5\) were done at 1 GPa in a piston
cylinder. Due to the capsule material (boron nitride) and the experimental condition, the quenched liquid metal phase contained variable amounts of Ni (1 to 6 wt%), and significant amounts of O (up to 2 wt%) and N (up to 4 wt%), very different from the pure Fe-Ni-S metallic liquid in our experiments. It is unclear how the existence of N and O in the liquid metal would affect the properties of the liquid metal. In addition to the composition, experiments in Shahar et al.\textsuperscript{5} were done at a much higher temperature (1650 °C), while our experiments were all conducted at the solid/liquid metal phase boundaries. Electrical resistivity studies on liquid Fe\textsuperscript{8,9}, for example, indicated a higher abundance of long-range order in the liquid metal structure at the solid/liquid metal boundary, which could potentially affect the bonding environment in our experiments, but not those in Shahar et al.\textsuperscript{5} done at temperatures well above the solid/liquid metal phase boundary. Overall, the experiments conducted in our study is probably the best approach so far to study the core crystallization process of interest for asteroid-sized planetary bodies, but might not be directly comparable to the silicate/metal experiments in the literature. In the future, it will be interesting to directly evaluate the apparent difference in S dependence of Fe isotope fractionation between metal/silicate and solid/liquid metal through three phase equilibrium experiments between silicate melt, solid metal, and liquid metal.

**Inward crystallization for the IIIAB parent body**

The crystal fractionation model employed in this study is purely a chemical model and does not depend on whether the parent core of IIIAB irons crystallized inward or outward. Regardless the crystallization direction, according to the Fe-Ni-S phase diagram (Fig. S6), it should precipitate Fe-Ni alloys with decreasing concentrations of siderophile elements, while the remaining melt gradually becomes S-enriched. Cooling rate measurements for IIIAB irons by ref. \textsuperscript{10}, however, found higher cooling rates for the early-crystallized, low-Ni meteorites compared to the late-crystallized, high-Ni meteorites. Hence, the authors concluded that the parent core of IIIAB irons must have crystallized inward.

Probably first proposed by Haack and Scott\textsuperscript{11}, inward asteroid core crystallization is supported by numerous cooling rate studies on IIIAB irons\textsuperscript{10} and IVA irons\textsuperscript{12,13}. These careful studies on the cooling rates of individual iron meteorites found a negative correlation between Ni content and cooling rate within each group, indicating inward crystallization for their parental cores with little or no mantle\textsuperscript{12}. The inward crystallization model for IVA parent body is further supported by paleomagnetic evidence for a dynamo driven by inward crystallization\textsuperscript{14}. In the literature, effort was also made to theoretical model the dynamo generation during inward core crystallization for a mantle-stripped metallic asteroid\textsuperscript{15,16}. In addition, the inward crystallization model is fundamental for the occurrence of ferrovolcanism, which refers to the eruption of less dense, S-rich Fe-Ni melt to the surface of metallic asteroids that can potentially explain the formation of pallasites\textsuperscript{17,18}.

Whether a planetary core crystallizes inward or outward depends on its internal pressure and the thermal profile. In the case of a planetary body similar to the size of Earth, its internal pressure is high enough to significantly lower the liquidus/solidus at the center of the core relative to the core-mantle boundary, causing the core to crystallize outward. For smaller planetary bodies, the pressure effect on the liquidus/solidus could be negligible across their cores. Therefore, core crystallization could occur from the core-mantle boundary inward because of the lower temperature at the core-mantle boundary during cooling. Complications arise when inward crystallization occurs in the form of small Fe-Ni alloy particles. With higher density relative to the parental liquid core, they are expected to descend to the center of the core,
generating an “Fe snow” that effectively accumulates from the inner core outward\textsuperscript{15,16}. Crystallization in the form of “Fe snow”, however, is inconsistent with the cooling rate studies\textsuperscript{10,12,13} because the early-crystallized Ni-poor alloys will settle at the center of the core and have lower cooling rates, opposite to the observations. For the parent bodies of IIIAB and IVA irons, the proposed mechanism is that the newly crystallized metal attaches to the overlying silicate shell and is prevented from sinking. The solidification front can then proceed downward, either concentrically or dendritically\textsuperscript{15}. The parent bodies of IIIAB and IVA irons had their mantles stripped away in hit-and-run impacts in the early Solar System\textsuperscript{12}. Cooling down with little or no mantle, these metallic asteroids had the opportunity to quickly grow an inactive, stagnant crust that allows further inward crystallization\textsuperscript{16}. The large asteroid Psyche, made almost entirely of Fe-Ni metal, might be a live example of the parent bodies of IIIAB and IVA irons. Understanding whether it crystallized inward or outward is, in fact, a major scientific objective for the NASA Psyche mission\textsuperscript{19}.

**Length scale of equilibration for metal/troilite fractionation**

At the temperature of \(\sim 930 ^\circ C\) when troilite forms in the parent core of IIIAB irons, a significant portion of the core would have crystallized. Due to the high yield strength of Fe-Ni alloy, further isotopic equilibration between metal and troilite below this temperature would likely be controlled by a diffusive process instead of a mechanical mixing process. Based on the cooling rate of 56 to 338 \(^\circ C/\text{My}\) estimated for IIIAB irons\textsuperscript{10}, and Fe diffusivities reported in ref. \textsuperscript{20}, the diffusion length scale can be estimated for Fe after troilite formation.

Assuming an asymptotic cooling history of:

\[
T = T_0/(1 + t/\tau_c),
\]

where \(T\) is the temperature in K, \(T_0\) is the eutectic temperature (930 \(^\circ C\)), \(t\) is time, and \(\tau_c\) is the cooling time scale, the cooling rate \((q)\) can be related to the cooling time scale through:

\[
q = -\frac{dT}{dt} = \frac{\tau_c^2}{\tau_c \tau_0}.
\]

Note that the asymptotic cooling model is chosen here instead of the linear cooling model because it is more realistic. The linear cooling model is usually avoided in diffusion calculations because temperature would falsely become negative if it decreases linearly over time. With the assumed thermal history, the diffusion distance \((L)\) of Fe can then be estimated using the following equation (Section 3.2.8.1, ref. \textsuperscript{21}):

\[
L = \sqrt{D_0 \tau_c (RT_0/E)},
\]

where \(D_0\) is Fe diffusivity at \(T_0\), \(E\) is the activation energy for Fe diffusivity (269 kJ/mol for Fe\textsubscript{90}Ni\textsubscript{10}, ref. \textsuperscript{20}).

As a result, the diffusion distance for Fe can be estimated to be 8 to 19 mm for IIIAB irons after troilite formation. Such an Fe diffusion distance is quite significant compared to the typical radius of troilites measured for Fe isotopes in literature (3 to 5 mm, ref. \textsuperscript{22}), supporting diffusive exchange as the mechanism for Fe isotope exchange between metal and troilite below 930 \(^\circ C\). Depending on the spatial distribution of troilites, however, such a diffusion distance might not be sufficient to homogenize Fe isotopes on a broader scale in iron meteorites. Potential Fe isotope gradient could have been recorded in iron meteorites as a function of distance to troilite, with metals adjacent to troilite nodules being heavier in Fe isotopic composition compared with those at distance to troilite. Unfortunately, existing Fe isotope data on iron meteorites did not record the context of the samples, which makes it impossible to evaluate this possibility in detail. But in general, Fe isotope fraction between metal and troilite reported in ref.
$^{22}$ is consistent with an equilibrium temperature significantly lower than 930 °C (up to 0.79 ‰, $T_{ae} = 389 \degree C$). Future studies on Fe isotopes of iron meteorites are recommended to sample at different distances to troilite nodules to examine whether such a mixing profile exits.
Supplementary Figure 1 | Iron isotope compositions for iron meteorites by group. Magmatic and nonmagmatic iron meteorite groups are plotted in solid and open symbols, respectively. The chondritic $\delta^{57}$Fe value of 0.006 ± 0.018 ‰ is calculated based on data reported in ref. 23. Iron meteorite data are from refs. 24,25,22,26. Error bars plotted in the figure represent 2 standard errors reported in the original literature.
Supplementary Figure 2 | Iron isotope fractionation between metal and troilite by NRIXS studies\textsuperscript{27} and measurements in iron meteorite samples\textsuperscript{22}. The possible temperature range for metal-troilite fractionation is defined by the troilite stability temperature of ~930 °C (estimated from ref. \textsuperscript{28} based on a bulk composition of Fe\textsubscript{0.48}Ni\textsubscript{0.08}S\textsubscript{0.44}) and the highest metal-troilite fractionation observed in iron meteorites\textsuperscript{22}.

Supplementary Figure 3 | BSE images for experiments a. OCT2017 and b. S10-1 as examples of the run products. The liquid metal phases of the experiments show dendritic quench textures that vary with the sulfur content (~22 wt% for OCT2017 and 16 wt% for S10-1). Tungsten carbide drill bits used for sampling the two phases have diameters from 300 to 700 μm.
Supplementary Figure 4 | Comparing non-mass dependent Fe isotope fractionation ($\Delta^{56}$Fe) between the solid and the liquid metal phases to check for isotopic equilibrium. The starting powder mixtures of the experiments were doped with $^{54}$Fe metal to disturb the natural $\Delta^{56}$Fe in the solid metal. During the experiment, Fe isotopes exchanged between the solid and liquid metal to achieve the same mass-independent isotope fractionation in both phases. Our data all fall on the 1:1 line in the figure, indicating that Fe isotope equilibrium was achieved between the two phases.

Supplementary Figure 5 | The solid-liquid metal partitioning coefficients for Ni are plotted versus S content of the liquid metal to compare with previous studies. Errors bars in the figure are 2 standard errors. Our data show good agreement with previous work. Literature data in the figure are from ref. 29 and references therein.
Supplementary Figure 6 | Fe-Ni-S phase diagram from ref. demonstrating the correlation between S content of the liquid metal and the equilibrium temperature. Solid blue curves in the phase diagram represent the liquid/solid metal phase boundary at different temperatures. With 10 at% Ni in the liquid metal, equilibrium S content in the liquid metal is only a function of equilibrium temperature. It is impossible to decouple S content in the liquid metal and the temperature unless Ni concentration is allowed to vary dramatically from experiment to experiment.
Supplementary Table 1. Run conditions and chemical compositions for all 11 experiments. Concentrations are in wt% and the errors are given in 2 standard deviation (except for S in liquid metal, which are in 2 standard error).

| Exp#  | Phase | T (ºC) | t (days) | Fe     | S        | Ni       | Total   |
|-------|-------|--------|----------|--------|----------|----------|---------|
| S2-1  | Solid | 1470   | 1        | 89.58±0.48 | 0.04±0.02 | 10.15±0.06 | 99.8±0.5 |
|       | Liquid|        |          | 84.29±0.27 | 4.33±0.28 | 10.90±0.10 | 99.5±0.4 |
| S5-1  | Solid | 1425   | 1.7      | 88.98±0.50 | 0.04±0.01 | 9.90±0.12 | 98.9±0.5 |
|       | Liquid|        |          | 83.82±0.71 | 4.75±0.55 | 10.59±0.09 | 99.2±0.9 |
| S10-1 | Solid | 1360   | 1        | 87.09±0.36 | 0.04±0.01 | 11.68±0.16 | 98.8±0.4 |
|       | Liquid|        |          | 73.59±0.47 | 15.93±0.61 | 10.58±0.15 | 100.1±0.8 |
| S15-1 | Solid | 1380   | 1        | 87.45±0.62 | 0.05±0.02 | 10.29±0.10 | 97.8±0.6 |
|       | Liquid|        |          | 77.64±0.93 | 11.65±1.11 | 10.26±0.23 | 99.5±1.5 |
| S15-2 | Solid | 1350   | 1        | 87.83±0.50 | 0.05±0.02 | 10.74±0.18 | 98.6±0.5 |
|       | Liquid|        |          | 72.74±0.34 | 17.60±0.44 | 9.55±0.18 | 99.9±0.6 |
| OCT17 | Solid | 1325   | 1        | 87.59±0.42 | 0.05±0.02 | 10.54±0.12 | 98.2±0.4 |
|       | Liquid|        |          | 67.67±0.43 | 23.48±0.50 | 8.17±0.16 | 99.3±0.7 |
| OCT17 | Solid | 1325   | 2        | 86.81±0.56 | 0.05±0.02 | 11.19±0.10 | 98.0±0.6 |
|       | Liquid|        |          | 67.95±0.44 | 22.63±0.45 | 9.08±0.12 | 99.7±0.6 |
| OCT20 | Solid | 1325   | 3        | 85.44±0.50 | 0.05±0.02 | 12.29±0.10 | 97.8±0.5 |
|       | Liquid|        |          | 67.73±0.33 | 21.78±0.37 | 9.78±0.16 | 99.3±0.5 |
| S25-1 | Solid | 1260   | 3        | 86.07±0.72 | 0.03±0.01 | 12.74±0.10 | 98.8±0.7 |
|       | Liquid|        |          | 63.57±1.21 | 26.77±1.34 | 8.07±0.40 | 98.4±1.8 |
| S25-2 | Solid | 1260   | 2        | 86.89±0.34 | 0.04±0.01 | 12.41±0.12 | 99.3±0.4 |
|       | Liquid|        |          | 64.79±1.13 | 26.07±1.40 | 7.95±0.40 | 98.8±1.8 |
| S25-3 | Solid | 1260   | 7        | 86.02±0.32 | 0.03±0.02 | 12.80±0.12 | 98.8±0.3 |
|       | Liquid|        |          | 64.03±1.17 | 26.48±1.40 | 8.13±0.42 | 98.6±1.9 |
**Supplementary Table 2.** Iron isotope compositions for the experiment products and geological standards. Errors are given in 2 standard error.

| Sample | Phase  | \( \delta^{56}\text{Fe} \) (‰) | \( \delta^{57}\text{Fe} \) (‰) | \( \Delta^{56}\text{Fe} \) (‰) | \( \Delta^{57}\text{Fe}_{\text{solid-liquid metal}} \) |
|--------|--------|-------------------------------|-------------------------------|-------------------------------|--------------------------------|
| S2-1   | Solid  | -5.91±0.06                    | -6.04±0.05                    | -1.81±0.05                    | 0.14±0.08                      |
|        | Liquid | -6.02±0.03                    | -6.18±0.07                    | -1.83±0.04                    |                                |
| S5-1   | Solid  | -10.77±0.06                   | -10.88±0.04                   | -3.42±0.04                    | 0.10±0.05                      |
|        | Liquid | -10.86±0.05                   | -10.98±0.04                   | -3.42±0.06                    |                                |
| S10-1  | Solid  | -8.04±0.03                    | -8.12±0.03                    | -2.53±0.02                    | 0.13±0.05                      |
|        | Liquid | -8.11±0.02                    | -8.25±0.04                    | -2.52±0.04                    |                                |
| S15-1  | Solid  | -1.09±0.03                    | -1.15±0.06                    | -0.31±0.04                    | 0.08±0.09                      |
|        | Liquid | -1.09±0.03                    | -1.23±0.06                    | -0.28±0.03                    |                                |
| S15-2  | Solid  | -1.02±0.02                    | -1.03±0.05                    | -0.32±0.03                    | 0.07±0.06                      |
|        | Liquid | -1.07±0.03                    | -1.10±0.03                    | -0.32±0.04                    |                                |
| OCT1717| Solid  | -5.98±0.05                    | -6.04±0.08                    | -1.90±0.07                    | 0.23±0.13                      |
|        | Liquid | -6.11±0.07                    | -6.27±0.10                    | -1.87±0.03                    |                                |
| OCT1817| Solid  | -5.87±0.13                    | -5.90±0.10                    | -1.86±0.04                    | 0.24±0.12                      |
|        | Liquid | -6.08±0.08                    | -6.14±0.08                    | -1.87±0.06                    |                                |
| OCT2017| Solid  | -6.01±0.02                    | -6.03±0.03                    | -1.92±0.03                    | 0.15±0.05                      |
|        | Liquid | -6.11±0.02                    | -6.18±0.05                    | -1.92±0.02                    |                                |
| S25-1  | Solid  | -3.38±0.04                    | -3.42±0.05                    | -1.06±0.02                    | 0.15±0.08                      |
|        | Liquid | -3.43±0.02                    | -3.56±0.05                    | -1.01±0.04                    |                                |
| S25-2  | Solid  | -3.12±0.02                    | -3.14±0.04                    | -0.99±0.02                    | 0.13±0.05                      |
|        | Liquid | -3.22±0.02                    | -3.27±0.04                    | -1.01±0.02                    |                                |
| S25-3  | Solid  | -2.83±0.03                    | -2.92±0.05                    | -0.85±0.03                    | 0.12±0.08                      |
|        | Liquid | -2.97±0.05                    | -3.04±0.06                    | -0.91±0.02                    |                                |
| BHVO-2 | Bulk   | 0.14±0.03                     | 0.20±0.05                     | 0.00±0.04                     | n/a                            |
| BIR-1  | Bulk   | 0.05±0.04                     | 0.13±0.05                     | -0.04±0.03                    | n/a                            |
| AGV-2  | Bulk   | 0.09±0.05                     | 0.18±0.05                     | -0.03±0.03                    | n/a                            |
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