

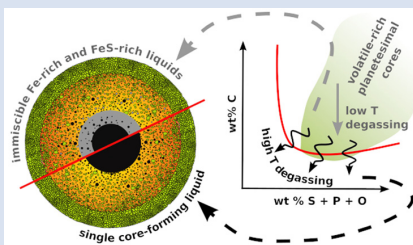
The extent of liquid immiscibility in planetesimal cores

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Abstract



We report results of experiments in the system $\text{Fe}_{0.9}\text{Ni}_{0.1}$, S, P, C, O, which constrain the extent of liquid immiscibility in planetesimal cores. Immiscibility results in segregation of Fe-rich (P-rich, C-rich) and FeS-rich (O-rich) liquids, with the extent of immiscibility dependent on volatile/light element content. Parental liquids to iron meteorites are volatile-poor, and based on our results, mostly represent miscible core-forming liquids. However, as these parental liquids were variably modified during/after planetesimal disruption, they are unlikely to fully represent original compositions of planetesimal cores. To better constrain planetesimal core compositions, we use data from chondrite meteorites to provide upper bounds on core volatile element

content. Modelled 'chondrite cores' are mostly immiscible liquids. The extent of immiscibility in planetesimal cores was sensitive to the degree of volatile loss during core formation, which was likely variable across planetary bodies, and dependent on thermal history and planetary differentiation processes. As such, evidence for immiscibility in core-forming liquids is useful in constraining the extent of degassing during differentiation.

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Introduction

Separation of Fe-rich, core-forming liquids from silicate was a defining process in planetary formation in the inner solar system, and left a geochemical signature on the cores and mantles of rocky planets (Wood, 2008). Differentiated asteroids, such as 4-Vesta, and the existence of iron meteorites, imply that core formation also occurred in a proportion of smaller bodies, possibly ranging down to a few 100 km in diameter (McCoy *et al.*, 2006; Goldstein *et al.*, 2009). Intriguingly, isotopic data from iron meteorites imply formation ages within a few Myr of the onset of accretion in the solar system (Kleine *et al.*, 2009). As such, building blocks of planets such as Earth likely consisted of a proportion of differentiated planetesimals, *i.e.* small rocky bodies with compositions modified by high temperature processes.

In addition to Fe and Ni, planetary cores contain variable concentrations of light elements: S, P, O, C, Si and H (Wood, 2008; Goldstein *et al.*, 2009). Towards lower pressures, mixing in Fe-rich liquids becomes increasingly non-ideal, resulting in 2-liquid fields in which immiscible O-poor/O-rich or S-poor/S-rich liquids coexist (Urakawa *et al.*, 1987; Chabot and Drake, 2000; Corgne *et al.*, 2008; Dasgupta *et al.*, 2009). Liquid immiscibility affects the composition of the cores of rocky bodies up to the size of Earth's Moon, would result in core stratification (Dasgupta *et al.*, 2009), and might influence core segregation processes (Bromiley, 2023). However, constraints on the extent of core immiscibility are limited, with most previous experimental work confined to simplified systems and/or conducted at pressures less relevant to planetesimal interiors (>3 GPa). In addition, although C has been shown to have a key role in reducing miscibility in Fe-rich liquids (Corgne *et al.*, 2008;

Dasgupta *et al.*, 2009), these studies were conducted in C-saturated or C-rich systems, *i.e.* in systems not directly comparable to volatile-depleted planetesimals (Hirschmann *et al.*, 2021).

Here, we describe superliquidus experiments which constrain liquid immiscibility in the system Fe, Ni, S, P, C, O from 0.5 to 1 GPa, at 1673 K, *i.e.* under conditions relevant to differentiation in planetesimals a few 100 km in diameter (Grewal and Asimow, 2023). Using constraints on the compositions of planetesimal cores from meteorite data, we then assess the importance of immiscibility in planetesimal cores.

Liquid Immiscibility in the System Fe, Ni, S, P, C, O

Starting mixes were prepared from high purity reagents, with variable S, P and C, based on a fixed $\text{Fe}_{0.9}\text{Ni}_{0.1}$ ratio, broadly representative of a range of planetesimal cores (Goldstein *et al.*, 2009). Incorporation of minor O into run products is expected depending on oxygen fugacity, f_{O_2} , within experiments (Corgne *et al.*, 2008; Dasgupta *et al.*, 2009). Starting mixes were loaded into graphite (C-saturated), MgO or BN capsules (C-undersaturated), and inserted into piston cylinder assemblies. Experiments at 0.5 GPa exhibited significant P and to a lesser degree S loss. A second series of experiments conducted at 1 GPa exhibited only minor volatile loss, but with liquid compositions consistent with 0.5 GPa experiments (Fig. 1a, Fig. S-2a, Tables S-1 and S-2). For 1.0 GPa experiments, rapid capsule recrystallisation during annealing and heating inhibited volatile loss. For 0.5 GPa experiments, it is likely that greater volatility resulted in volatile loss during initial stages of experiments.

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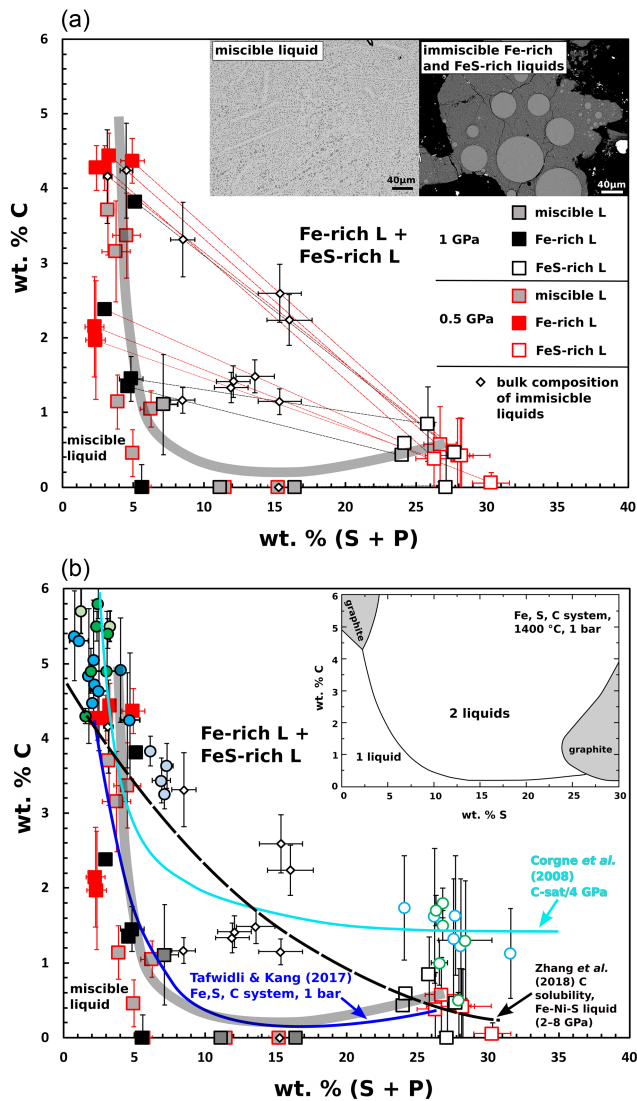


Figure 1 (a) Liquid compositions in the system $\text{Fe}_{0.9}\text{Ni}_{0.1}$, S, P, C, O at 0.5 (red)/1.0 GPa (black), and calculated bulk compositions for immiscible liquids. Tie-lines connect compositions of coexisting immiscible liquids. Insert: examples of run products (see Fig. S-1 for details). (b) Comparison with C-saturated/C-rich experiments, Green: Dasgupta et al. (2009) Fe, S, C system at 2 GPa, Light blue: Corgne et al. (2008) model chondritic system, and their miscibility curve at 4 GPa. Blue line: miscibility curve at 1 bar/1400 °C in the system Fe-S-C calculated using thermodynamic model of Tafwidli and Kang (2017); insert additionally shows fields of C-saturation. Dashed black line: pressure-independent C solubility in Fe/Ni-S liquid at 2–7 GPa (Zhang et al., 2018).

Capsule recrystallisation at 1673 K likely inhibited further volatile loss, followed by re-equilibration across sample volumes (see Supplementary Information for discussion). Rapid equilibration in Fe-rich liquids suggests that chemical equilibration would have been attained in experiments with several minutes, even with early volatile loss.

As expected, light element content controls liquid stability in our experiments, driving immiscibility and separation of what we term Fe-rich (low S, high P, high C, lower O) and FeS-rich (high S, low P, low C, higher O) liquids. Figure 1b shows comparison of our results with higher pressure C-saturated experiments (Corgne et al., 2008; Dasgupta et al., 2009) and the calculated 1 bar Fe-S-C phase diagram (Tafwidli and Kang, 2017). Under conditions of planetesimal interiors (*i.e.* <1 GPa), miscibility in

core-forming liquids is restricted to volatile-poor compositions. C is particularly important in defining the extent of immiscibility in core-forming liquids; *e.g.*, <1 wt. % C results in immiscibility in otherwise miscible liquids at S contents >5 wt. % S. This mirrors the compositional dependence of C solubility in Fe/Ni/S liquids, which is markedly lower at higher S contents (Zhang et al., 2018; Fig. 1b). Conversely, C solubility increases with increasing Ni/Fe ratio, implying that the miscibility field in Figure 1 expands with increasing Ni content. P also reduces miscibility in Fe-S liquids (Chabot and Drake, 2000), although to a lesser extent than C (Fig. S-2b). Our data cannot differentiate the influence of P and O on miscibility. However, coincidence of our $\text{Fe}_{0.9}\text{Ni}_{0.1}$ miscibility curve at 0.5/1 GPa with the 1 bar Fe-S-C miscibility curve from Tafwidli and Kang (2017), suggests that wt. % concentrations of P and O significantly reduce miscibility. By extension, in planetesimals with interior pressures <0.5 GPa, the miscibility field in core-forming liquids is likely further reduced compared to that shown in Figure 1.

Discussion

At pressures up to 1 GPa, miscibility in core-forming liquids is limited to Fe-rich liquids with low light element contents and/or high Ni contents. With increasing planetesimal size and internal pressure, immiscibility is restricted to increasingly light element-rich core compositions. The most significant uncertainty in assessing the importance of immiscibility in planetesimals is the light element content of their metallic cores.

Volatile-poor planetesimal cores: iron meteorite parent liquids. Magmatic iron meteorites are believed to have originated as the cores of differentiated, rocky planetesimals (Goldstein et al., 2009), and as they date from a few Myr after the onset of solar system accretion (Kleine et al., 2009; Spitzer et al., 2021), provide insight into the early stages of planetary formation (*e.g.*, Chabot and Drake, 2000; Hirschmann et al., 2021; Grewal and Asimow, 2023; Grewal et al., 2024). However, as magmatic iron meteorites formed by near-fractional crystallisation, the composition of liquids from which they crystallised must be inferred. There are various models for reconstructing compositions of magmatic iron meteorite parental liquids (IMPLs), although results from these are broadly consistent, at least for elements such as S, which have a strong influence on element partitioning (*e.g.*, Goldstein et al., 2009; Hilton et al., 2022; Zhang et al., 2024). As shown in Figures 2a and S-2b, IMPL compositions are light element-poor and, as a consequence, except for IIC (Fig. S-2b), single liquids regardless of planetesimal size. However, the possibility that other IMPLs were low C/moderate S immiscible liquids cannot be entirely dismissed.

IMPLs are parental liquids which solidified to form magmatic iron meteorites. Although they originated as core-forming liquids in planetesimals, the extent to which they evidence the original light element content of planetesimal cores is unclear, as they could have been modified by degassing, *i.e.* loss of volatile elements, during and after planetesimal disruption. Hirschmann et al. (2021) argued that compositions of IMPLs largely reflect the primary volatile content of planetesimal cores. Specifically, they noted that low C/S in IMPLs mirrors trends in $\log C/S$ vs. $\log C$ in chondrites, which they identify as a signature of metamorphic degassing in rocky planetesimals. They argued that degassing in planetesimals during subsequent melting and differentiation would further reduce C/S to values recorded in IMPL compositions. By contrast, volatile loss from unshielded planetesimal cores, *i.e.* following planetesimal disruption, would result in preferential S loss and an increase in C/S (Hirschmann et al., 2021).

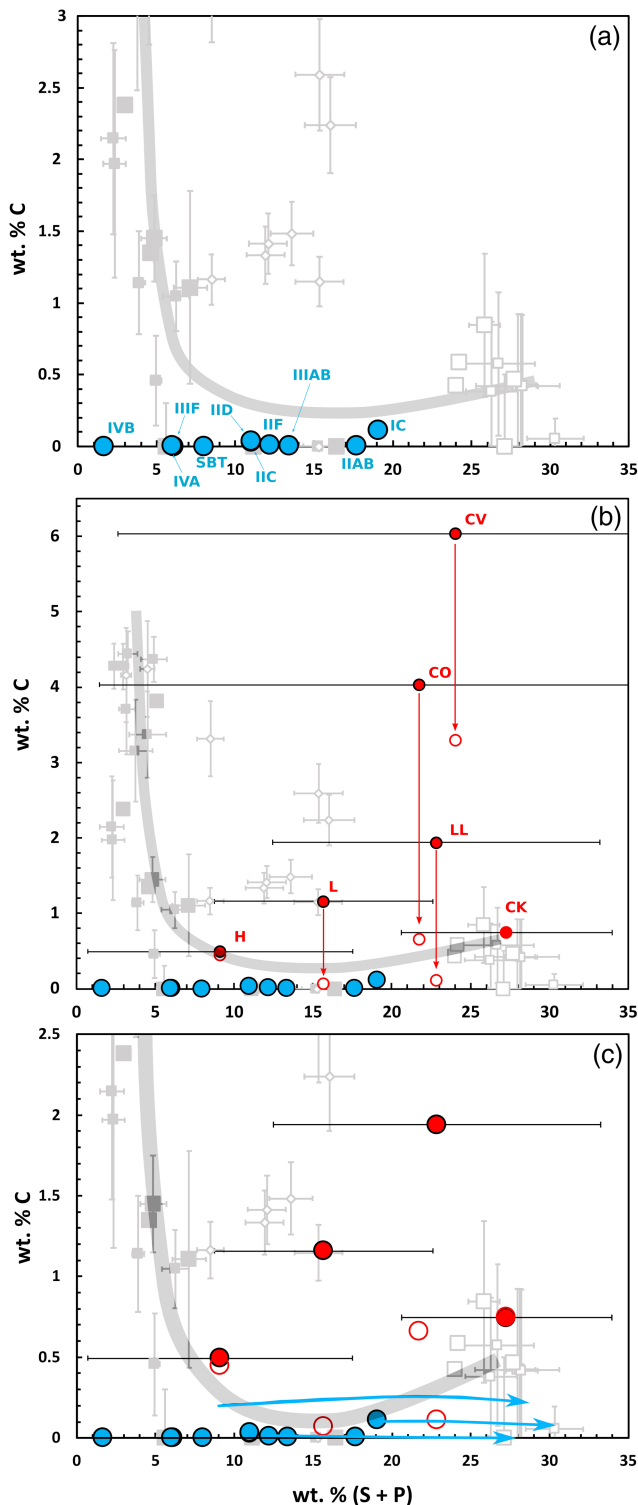


Figure 2 Model planetesimal core compositions; data from Figure 1b shown in light grey for clarity. **(a)** Blue circles: magmatic iron meteorite 'parent core' compositions from Hilton *et al.* (2022) with C contents from Grewal and Asimow (2023). **(b)** Red circles: modelled planetesimal core compositions based on metal/silicate differentiation of averaged chondrite compositions, after Bercovici *et al.* (2022). Error bars are propagated from uncertainties in S and P contents. Open circles: corresponding 'minimum' C core compositions for each chondrite group based on variability in chondrite C contents for petrological types 3–6 (Fig. 3). **(c)** Blue arrows: modelled liquid compositions with progressive crystallisation of solid Fe/Ni.

Re-examination of a larger dataset of chondritic compositions casts doubt on this interpretation (Fig. 3). There are marked trends in $\log C/S$ versus $\log C$ in both carbonaceous and non-carbonaceous (ordinary) chondrite compositions, but no systematic trends with petrological type, a proxy for peak metamorphic temperature. In contrast, (1) there is greater variability within petrological type than between types for ordinary chondrites, (2) type 3 chondrites, which represent the mineralogically most 'pristine' material, span the full range of C and C/S, and (3) variations for types 3–6 across both populations are statistically indistinguishable from a 1:1 relationship, *i.e.* a 2 order of magnitude variation in C at constant S. Instead, variability of C contents in chondrites is largely independent of variability in S, and, therefore, not reflective of the extent of degassing during metamorphism.

Volatile concentrations in chondrites partly mirror variability in moderately volatile element (MVE) contents, which reflect nebular processes such as incomplete condensation and mixing between distinct reservoirs (Braukmüller *et al.*, 2018). Subsequent variation in C reflects low temperature rather than high temperature degassing processes. This is supported by step heating experiments (Court and Sephton, 2014) which demonstrate that substantial C loss and minor S loss only occurs at low temperatures. Similarly, correlations between bulk C and N in chondrites and peak metamorphic temperatures have been argued to imply that C loss occurred at low temperatures (<200 °C) (Grewal, 2022; Grewal *et al.* 2022).

Chondrite compositions cannot be used to infer a single high temperature degassing process. By extension, although IMPLs have low C/S compared to chondrites, no simple inferences can be drawn regarding the relative timing of volatile loss. Iron meteorites evidence a family of light element-poor, largely single liquids. However, cooling rates, isotope systematics and MVE contents suggest that, at least, some groups evidence degassing after core segregation and following planetesimal disruption (*e.g.*, Yang *et al.*, 2007; Matthes *et al.*, 2018; Hunt *et al.*, 2022; Rubin *et al.*, 2022; Zhang *et al.*, 2024). Absolute volatile contents in IMPL might not, therefore, fully reflect initial planetesimal core compositions.

Volatile-rich planetesimal cores: modelling chondrite differentiation. Variability in the C content of chondrites reflects low temperature degassing processes. However, as some ordinary chondrites evidence peak metamorphic temperatures up to the onset of FeS melting close to 1000 °C (Mare *et al.*, 2014), chondrite compositions are useful in assessing total volatile content in planetesimals prior to/at the onset of core formation. This can be more meaningfully compared to IMPL compositions by calculating hypothetical planetesimal core compositions based on chondrite data. Bercovici *et al.* (2022) modelled core/mantle differentiation for 8 averaged chondritic compositions to constrain S content of differentiated planetesimal cores. Here, we use the same approach to calculate S, C and P contents of planetesimal cores based on average chondrite compositions. However, as C contents of chondrites are highly variable and reflect low temperature processes, we additionally calculate core compositions based on minimum C contents in chondrites; *i.e.* we calculate a lower bound of C for each average chondrite composition based on the full variability of C contents shown in Figure 3 (see Supplementary Information for full details).

Calculated core compositions based on averaged chondrite data (filled red circles, Fig. 2b) have S contents of 9–37 %, and P core contents of 0.5–2.5 % (Table S-4). 'Minimum C' models give a more meaningful assessment of core C contents, as the large variability in C contents in chondrites

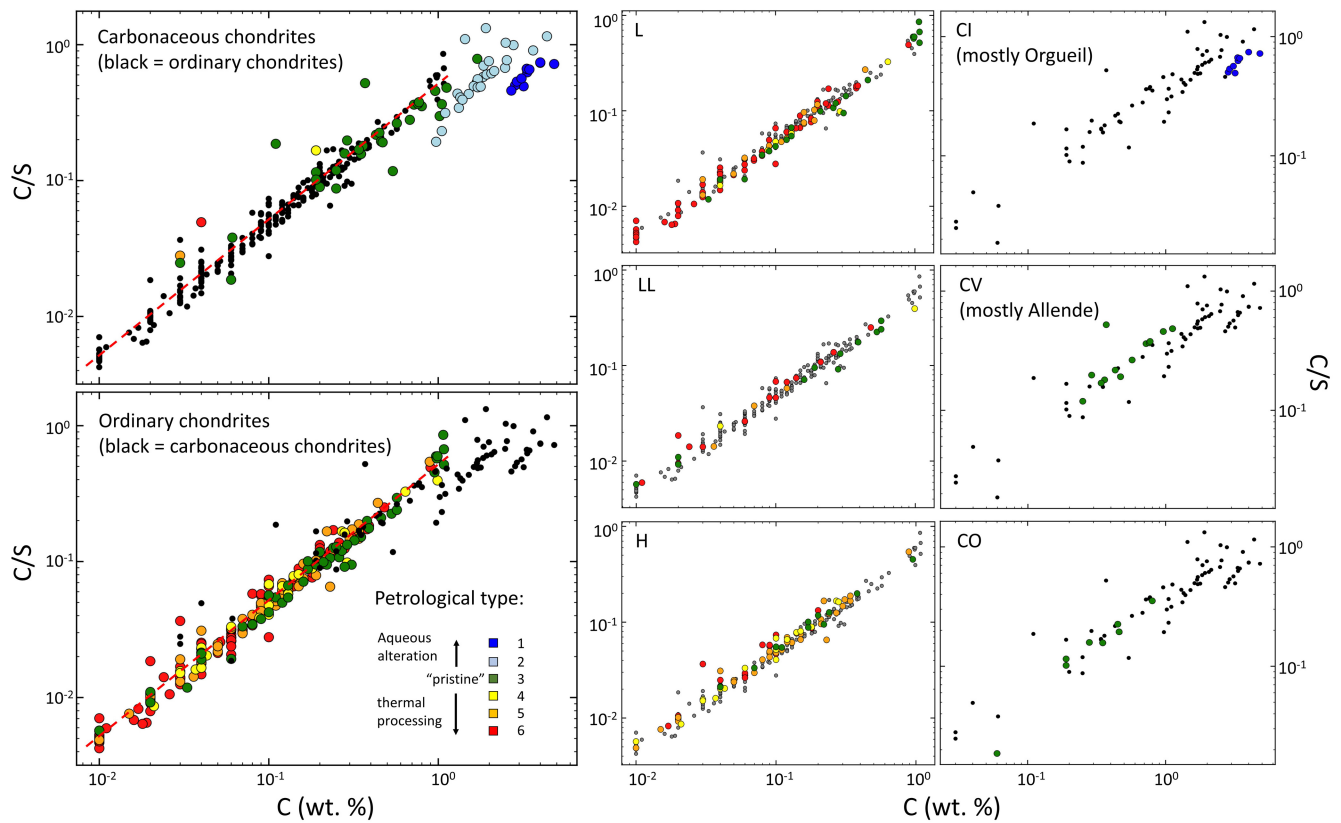


Figure 3 Compositions of carbonaceous and ordinary chondrites as a function of petrological type (left), and for specific groups (right), after Hirschmann *et al.* (2021). Within each group, colour symbols denote petrological type. Small, black symbols are all other ordinary and carbonaceous chondrite data, for comparison. Red dashed line is a 1:1 fit to data (types 3–6). Compositions from the Metbase (2024) database for samples with combined bulk C and S, and clear petrologic type (<http://www.metbase.org>, GeoPlatform UG, Germany).

could indicate later C-enrichment in parent bodies; minimum C core contents (open circles) range from <0.1 to 3.3 %. Core compositions extend across the miscibility curve in Figure 2b; however, S and P contents of all except those based on average H and L chondrites are high enough to result in immiscibility excluding any influence of C (Fig. S-2b). Large uncertainties in modelled core compositions arise from significant variations in volatile contents in chondrite compositions. Regardless, in comparison IMPL compositions are volatile-poor (Hilton *et al.*, 2022; Grewal and Asimow, 2023), and record significant C loss, and moderate S and P loss compared to model chondrite core compositions. This is consistent with multiple processes of element depletion, both before and after differentiation and core formation.

Our modelled chondrite core compositions provide upper bounds on volatile content of planetesimal cores, as chondrite compositions do not record degassing during planetesimal differentiation. The extent of planetesimal degassing will depend on thermal history and differentiation processes (*e.g.*, Kaminski *et al.*, 2020). Significant volatile loss would occur on planetesimals which underwent largescale melting, particularly for those with external as opposed to internal magma oceans (Hirschmann *et al.*, 2021; Grewal *et al.*, 2022). However, some thermal modelling suggests that silicate melting in planetesimals would be limited in extent and duration (Kaminski *et al.*, 2020; see Supplementary Information), and it is argued that core formation in some planetesimals could proceed without large-scale silicate melting (*e.g.*, Bromiley, 2023). As such, the extent of degassing in planetesimals during core formation could have been variable. In turn, the importance of immiscibility in core-forming liquids, which is a function of degassing and planetesimal thermal history, is

difficult to constrain. However, based on meteorite data (Figure 2 and S-2), it is clear that evidence for or against immiscibility can provide useful constraints on the extent of degassing prior to and during planetary differentiation, and immiscibility probably played an important role in the evolution of some differentiating bodies. One pertinent illustration of this is segregation processes in icy/rocky bodies in the outer solar system. It has been suggested (*e.g.*, Melwani Daswani *et al.*, 2021) that some icy moons have bulk compositions close to those of carbonaceous chondrites, and formed following slow heating and segregation of rock/ice/metal over 10s–100s Myr timescales. Slow differentiation and retention of volatiles in these bodies explain the presence and composition of thick cryospheres. Under these conditions, immiscibility in core-forming liquids may be inevitable for bodies with internal pressures <5–8 GPa.

Immiscibility during core solidification. It is often assumed that solidification of planetesimal cores would progressively enrich remaining liquid in light elements, eventually resulting in immiscibility (*e.g.*, Chabot and Drake, 2000; Corgne *et al.*, 2008; Goldstein *et al.*, 2009; Bercovici *et al.*, 2022). Although there is a lack of experimental data on solid/liquid element partitioning in complex systems, compositional controls on light element partitioning can be used to assess changes in liquid composition during core solidification. Here, we use data from Grewal and Asimow (2023) and Zhai *et al.* (2022) to model incremental extraction of small batches of solid Fe, Ni from single liquid planetesimal core-forming liquids (Fig. 2c; see Supplementary Information for details). Blue arrows denote modelled liquid compositions during crystallisation of 3 core-forming liquids. Due to the highly incompatible nature of S (*e.g.*, Dasgupta *et al.*, 2009), liquids become progressively S-rich with crystallisation.

However, as P and C partitioning is strongly dependent on liquid S content, concentrations of both initially increase in liquids during crystallisation, but ultimately decrease at high liquid S contents (Fig. 2c). Consequentially, liquid compositions evolve towards FeS rather than becoming light-element enriched and moving into the 2-liquid field. Progression in liquid compositions is model dependent, and the approach used here assumes no control of temperature or cross-compositional terms for C and P. Regardless, an inevitable consequence of non-ideal mixing of C, P and S is that liquids must progress towards S-rich, C-poor and P-poor compositions during crystallisation. Some single liquid compositions remain in the 1-liquid stability field. Other liquids can progress into the 2-liquid field, and depending on extent of liquid segregation, may then eventually evolve back towards single liquids. As such, immiscibility is not simply ascribable to core solidification, but, again, is largely a function of planetesimal composition, size and differentiation processes.

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Additional Information

Supplementary Information accompanies this letter at <https://www.geochemicalperspectivesletters.org/article2445>.



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