



Sulfur-controlled iron isotope fractionation experiments of core formation in planetary bodies

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Abstract

A series of high pressure and temperature experiments were conducted to better constrain the Fe isotope fractionation during core–mantle differentiation in planetesimal and planetary bodies. Synthetic mixtures of oxides and metal having varying amounts of sulfur, approximating terrestrial and Martian compositions, were melted at 1–2 GPa and 1650 °C. Iron isotopic equilibrium between the resulting metal and glass run products was verified for all experiments using the three-isotope technique. Purified Fe from metal and glass was analyzed by multiple-collector ICP-MS in high resolution mode. Iron alloy and silicate glass show a well-resolved $\Delta^{57}\text{Fe}_{\text{metal-silicate}}$ of $+0.12 \pm 0.04\text{‰}$ in a sulfur-free system. Isotope fractionation increases with sulfur content to $+0.43 \pm 0.03\text{‰}$ at 18 wt.% sulfur in the metal. These results cannot be easily interpreted within the context of known Fe isotope ratios in most natural samples of planetary and asteroidal mantles and therefore suggest more complex processes affected the Fe isotope fractionation therein. However, to reconcile Martian meteorite iron isotopic signatures with geophysical models using this new experimental data requires a smaller amount of sulfur in the Martian core than previous estimates, with an upper limit of $\sim 8\text{ wt.}\%$.

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1. INTRODUCTION

Differentiation of a silicate mantle and metallic core occurred on the terrestrial planets, the moon and on many asteroids, as these objects were hot and big enough to melt the metal so it could percolate through the silicate (Taylor, 1992). The necessary heat largely comes from energy released during radioactive decay. The iron and siderophile elements sink to the center to form a core, while the silicates and lithophile elements form a mantle. Many details of differentiation processes are still unclear, but may be constrained by numerical modeling (e.g., Lodders, 2000;

Moskovitz and Gaidos, 2011; Elkins-Tanton, 2012), laboratory experimental work (e.g., Rushmer et al., 2005; Chabot et al., 2011) and analysis of natural samples (e.g., Kleine et al., 2005; Caro et al., 2006; Borg et al., 2011).

The distinct chemical bonding environments of elements in different phases are expected to induce isotope fractionation of these elements. For example, Fe in planets and planetesimals should manifest isotope fractionation resulting from core–mantle differentiation. In theory, the fractionation should constrain the bulk chemical composition of the planetary body and the composition and conditions of differentiation of unseen reservoirs like the core. In practice, interpretation of Fe isotope compositions of natural samples is less than straightforward. Iron in terrestrial abyssal peridotites (Craddock et al., 2013) has a similar

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isotope composition to most chondrites (Dauphas et al., 2009; Craddock and Dauphas, 2010). Shergotty–Nahkla–Chassigny (SNC) meteorites from Mars and eucrite–diogenite (HED) meteorites thought to originate on the asteroid Vesta, also have chondritic iron isotope values (Poitrasson et al., 2004; Wang et al., 2012). By contrast, Fe in peridotite xenoliths (Poitrasson et al., 2013), and in terrestrial basalts (e.g., Teng et al., 2013; Craddock et al., 2013) is enriched in the heavier isotopes by $\sim 0.1\%$ in $^{57/54}\text{Fe}$. These contrasts lead to uncertainties regarding the Fe isotope composition of the bulk silicate Earth, the isotopic effect of partial mantle melting in planet and planetesimals, and the extent of isotope fractionation during core–mantle differentiation.

Theoretical calculations (Polyakov, 2009) and laboratory experiments do not yet provide a consistent answer of whether iron should be isotopically fractionated during core–mantle differentiation. There are three previous experimental studies that constrain mass fractionation of Fe between Fe metal and magnesian silicate at high temperatures and pressures. Poitrasson et al. (2009) and Hin et al. (2012) suggested that fractionation was not resolvable between metal and silicate in their experiments, and Williams et al. (2012) inferred a large fractionation from their experimental results, which were conducted at higher pressure and oxygen fugacity conditions. We undertook a carefully-designed set of experiments to re-evaluate iron mass fractionation between metal and silicate during melting. In order to better simulate differentiation under natural conditions, we focus on the isotopic effect of adding a light element, in this case sulfur, to the metal.

Terrestrial seismic data supports the presence of light elements in addition to Fe within the core of the Earth. The main candidates considered are carbon, sulfur, oxygen, silicon and hydrogen. The elements with which molten Fe metal bonds during core–mantle differentiation is a function of the conditions attending the core formation. For example, at high temperature under reducing conditions, Si is likely to alloy with iron (Ricolleau et al., 2011); under more oxidizing conditions, however, sulfur is likely to enter the metal (Sanloup and Fei, 2004). The presence of substantial sulfur has long been inferred for the Martian core (Wänke and Dreibus, 1988). Trace element contents of iron meteorites that require sulfur during crystallization imply its importance in many asteroidal cores (e.g., Chabot, 2004). For these reasons, we chose to evaluate the effect of sulfur on the mass fractionation of Fe during laboratory experiments similar to differentiation.

2. METHODS

2.1. Experimental setup

Experiments were conducted in a piston cylinder apparatus at the Geophysical Laboratory at 1 and 2 GPa. The $\frac{1}{2}$ " piston cylinder assembly consisted of a graphite tube heater insulated with a Pyrex glass sleeve surrounded by a talc shell. The samples were contained in BN capsules or MgO capsules. Due to interaction with the capsule, discussed below, only one experiment in an MgO capsule

was found to have equilibrated and therefore that is the only experiment in an MgO capsule for which we provide the isotopic analyses. The temperature was measured with a W/Re thermocouple in contact with the top of the sample capsule. All runs were conducted at 1650 °C. Run durations were varied from 5 to 150 min. The quench times were measured and a temperature of 200 °C was obtained in less than 10 s after power was cut from the experiment.

Two different starting materials were used: Mars-like and Earth-like. The Earth experiments consisted of 69 wt.% of an oxide mix and 31 wt.% of pure Fe metal. These experiments were conducted in MgO capsules. However, as noted above, only one of these experiments came to isotopic equilibrium. The Mars-like experiments consisted of 78 wt.% of an oxide mix and 22 wt.% of a mixture of FeS, Fe, and Ni. The metal contained 17, 10, 5 or 0 wt.% S. The starting silicate compositions are given in Table 1. Duplicate experiments with 10 wt.% sulfur were done to double check equilibrium conditions and to evaluate whether a pressure difference of 1 and 2 GPa affects the iron isotopic fractionation.

2.2. Chemical analyses of the experimental charges

After each experiment, the capsule was broken in half. One side of the experiment was polished and analyzed for its chemical composition using instrumentation at the Carnegie Institution of Washington discussed below. The pieces of glass and metallic beads chosen for sample characterization were set in epoxy and polished for analysis (Fig. 1). Results are provided in Table 2.

Metal was analyzed using a JEOL 6500F Field Emission scanning electron microscope (SEM) outfitted with an Oxford X-Max 80 mm² Si Drift Detector combined with an Aztec software package. Use of the SEM provided high accuracy for the metal compositions, because the metal quenched to a dendritic pattern. Analyses were made over large representative areas of the quenched metal for 50 s, using an accelerating voltage of 15KeV and a beam current of 1 nA. Standards were pure Fe metal, pure Ni metal, pyrite, and BN. The Si-drift detector provides higher sensitivity and lower detection limits (to 0.05% for Fe (John Armstrong, personal communication)) compared to standard EDS. Oxygen detected in the metal samples resulted from oxidation of the metal surface during preparation for analysis.

Table 1
Starting compositions of the silicate fractions.

Composition 1 "Earth-like"	Oxide (wt.%)	Composition 2 "Mars-like"	Oxide (wt.%)
SiO ₂	47.9	SiO ₂	46.5
MgO	36.2	MgO	29
Fe ₂ O ₃	6.4	FeO	17.6
Al ₂ O ₃	3.8	Al ₂ O ₃	2.8
CaO	5.6	CaO	2.4
⁵⁴ FeO	~ 0.1	⁵⁴ FeO	~ 0.1
		Na ₂ O	1.0

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