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Coupled ^{142}Nd–^{143}Nd evidence for a protracted magma ocean in Mars

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Resolving early silicate differentiation timescales is crucial for understanding the chemical evolution and thermal histories of terrestrial planets[1]. Planetary-scale magma oceans are thought to have formed during early stages of differentiation, but the longevity of such magma oceans is poorly constrained. In Mars, the absence of vigorous convection and plate tectonics has limited the scale of compositional mixing within its interior[2], thus preserving the early stages of planetary differentiation. The SNC (Shergotty–Nakhla–Chassigny) meteorites from Mars retain 'memory' of these events[3–5]. Here we apply the short-lived ^{146}Sm–^{142}Nd and the long-lived ^{147}Sm–^{143}Nd chronometers to a suite of shergottites to unravel the history of early silicate differentiation in Mars. Our data are best explained by progressive crystallization of a magma ocean with a duration of ~100 million years after core formation. This prolonged solidification requires the existence of a primitive thick atmosphere on Mars that reduces the cooling rate of the interior[6].

Metallic core formation in Mars is constrained to 7–15 Myr after solar nebula condensation from ^{182}Hf–^{182}W chronometry on SNC meteorites[5,7]. Core formation is thought to occur in conjunction with planetary-scale magma oceans that result from melting in the final stages of accretion[8]. Accretion, radioactive decay of short-lived isotopes such as ^{26}Al, and gravitational energy from core formation provided enough heat to melt a large portion of the martian mantle[1]. The rate of magma ocean solidification remains poorly constrained. Time intervals of magma ocean crystallization for Earth- and Mars-sized bodies from several thousand to a few hundred millions of years have been proposed[1,6,9].

Sm and Nd fractionate from each other during silicate differentiation because Nd is more incompatible than Sm during magmatic processes, resulting in greater Sm/Nd in residues than in partial melts. The ^{146}Sm–^{142}Nd chronometer with a half-life of 103 million years (Myr), coupled with the ^{147}Sm–^{143}Nd system with a half-life of 106 billion years (Gyr), can track early silicate differentiation events during extant ^{146}Sm of ~500 Myr. Anomalies in ^{142}Nd/^{144}Nd have previously been reported for SNCs[3,5,10–13]. These data are consistent with one or more silicate differentiation events at $4{,}525^{+19}_{-21}$ Myr ago, close to the time of core formation[5]. Variation in ^{142}Nd/^{144}Nd and the calculated ^{143}Nd/^{144}Nd for the mantle source compositions of SNCs presumably resulted from rapid crystallization of a martian magma ocean (MMO) that produced compositionally distinct materials within the mantle[4], and imply timescales of solidification of 40 Myr or less. However, the range of uncertainties in ^{142}Nd/^{144}Nd of ±12–31 p.p.m. (2σ) (1 p.p.m. corresponds to 10^{-6}) permits differentiation times as early as 4,567 Myr to younger than 4,000 Myr ago for individual rocks. Hence, the relationship between each rock reservoir and a single early magma ocean, the duration of this magma ocean, or whether there were multiple planetary-scale silicate differentiation events during the lifetime of ^{146}Sm, all remain unclear. In addition, earlier assessment was made assuming that the terrestrial standard value for ^{142}Nd/^{144}Nd was chondritic and represented the starting composition of the martian mantle. Precise ^{142}Nd/^{144}Nd measurements on present-day mantle-derived Earth samples show a clearly resolved difference with chondrites resulting from long-term differences in their Sm/Nd (refs 14–16). These differences affect the calculated ages derived from multi-stage mantle evolution models.

Here we report Sm–Nd isotope systematics for eight martian shergottites that span the range of known compositions. Neodymium from the samples was purified using cation column chromatography and Ln-Spec resin (di(2-ethylhexyl) orthophosphoric acid (HDEHP) saturated on teflon beads). Cerium was removed from the Nd cuts by solvent extraction[17]. The high-precision Nd isotope measurements were made on a ThermoFinnigan Triton thermal ionization mass spectrometer as positive metal ions in multidynamic mode. Details of analytical techniques are presented in the Methods and in the Supplementary Information. The ^{142}Nd/^{144}Nd was measured to precisions of ±2–6 p.p.m. (2σ) on these samples, allowing for a more precise evaluation of the timescales and number of early silicate differentiation events. The Nd isotope data are presented in ε notation (Fig. 1, Table 1), defined as deviation in parts per 10^4 from the

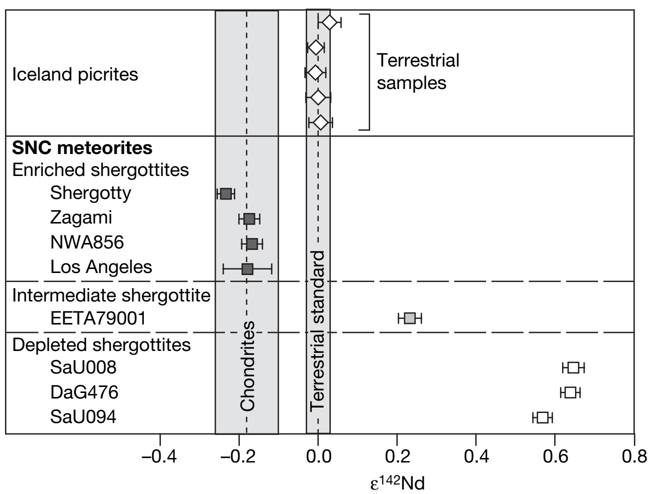

Figure 1 | ε^{142}Nd measured for martian meteorites in this study. **Terrestrial samples (Iceland picrites) measured during the same analytical campaign are added for comparison. ε^{142}Nd is defined as:**

$$\varepsilon^{142}\mathrm{Nd} = \left(\frac{(^{142}\mathrm{Nd}/^{144}\mathrm{Nd})_{\mathrm{sample}}}{(^{142}\mathrm{Nd}/^{144}\mathrm{Nd})_{\mathrm{terrestrial\ standard}}} - 1\right) \times 10{,}000.$$

Shaded areas represent the chondrite value (-0.18 ± 0.08[15]), and the terrestrial standard (0.0 ± 0.03). Error bars represent $\pm 2\sigma$ for each measurement.

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terrestrial standard for ε^{142}Nd and the chondrite uniform reservoir (CHUR) for ε^{143}Nd (ref. 14).

The average chondrite value for ε^{142}Nd is -0.18 ± 0.08 (ref. 15), while that for the terrestrial standard is 0 ± 0.03 (refs 14–16) (Fig. 1). The depleted shergottites SaU008, SaU094, and DaG476 have ε^{142}Nd values from $+0.57 \pm 0.04$ to $+0.65 \pm 0.05$ (2σ), and initial ε^{143}Nd values from +36.2 to +39.1, indicating derivation from an incompatible-trace-element-depleted source formed during the first 500 Myr evolution of Mars. The intermediate (EETA79001) and enriched shergottites (Shergotty, Zagami, Los Angeles, NWA856) show a progressive decrease in ^{142}Nd/^{144}Nd and ^{143}Nd/^{144}Nd, from incompatible-trace-element-depleted to enriched with ε^{142}Nd from $+0.23 \pm 0.03$ to -0.23 ± 0.02, and initial ε^{143}Nd from +16.9 to −7.0, respectively.

The evolution of source reservoirs for shergottites is modelled with a bulk Mars that is initially chondritic for Sm/Nd with present-day $\varepsilon^{142}\text{Nd} = -0.18$ and $\varepsilon^{143}\text{Nd} = 0$. The ε^{142}Nd and ε^{143}Nd for each shergottite is calculated using a two-stage model (Fig. 2), in which ε^{143}Nd is calculated from ^{147}Sm/^{144}Nd ratios for time-integrated sources at t_1 and projected in time to 150 Myr ago:

$$\left(\frac{^{142}\text{Nd}}{^{144}\text{Nd}}\right)_{t_2} = \left(\frac{^{142}\text{Nd}}{^{144}\text{Nd}}\right)^{\text{CHUR}}_{\text{present}} - \left(\frac{^{146}\text{Sm}}{^{144}\text{Sm}}\right)^{\text{CHUR}}_{T_0} \times \left(\frac{^{144}\text{Sm}}{^{147}\text{Sm}}\right)^{\text{CHUR}} \times \left[\left(\frac{^{147}\text{Sm}}{^{144}\text{Nd}}\right)^{\text{CHUR}} \times e^{-\lambda_{146}(T_0 - t_1)} + \left(\frac{^{147}\text{Sm}}{^{144}\text{Nd}}\right)^{\text{source}}_{t_1} \times \left(e^{-\lambda_{146}(T_0 - t_2)} - e^{-\lambda_{146}(T_0 - t_1)}\right)\right] \quad (1)$$

$$\left(\frac{^{143}\text{Nd}}{^{144}\text{Nd}}\right)^{\text{source}}_{t_2} = \left(\frac{^{143}\text{Nd}}{^{144}\text{Nd}}\right)^{\text{CHUR}}_{\text{present}} + \left(\frac{^{147}\text{Sm}}{^{144}\text{Nd}}\right)^{\text{CHUR}} \times \left(1 - e^{\lambda_{147} t_1}\right) + \left(\frac{^{147}\text{Sm}}{^{144}\text{Nd}}\right)^{\text{source}}_{t_1} \times \left(e^{\lambda_{147} t_1} - e^{\lambda_{147} t_2}\right) \quad (2)$$

where $T_0 = 4.567$ Gyr ago, the onset of solar nebula condensation, t_1 is the time of Sm/Nd fractionation, and t_2 is the crystallization age of each meteorite (Table 1). $\lambda_{146} = 6.74 \times 10^{-9}\,\text{yr}^{-1}$ and $\lambda_{147} = 6.54 \times 10^{-12}\,\text{yr}^{-1}$ are the decay constants for ^{146}Sm and ^{147}Sm respectively. In this two-stage model, samples plotting inside the field defined by isochrons for t_1 (Fig. 2) give information on the timing of shergottite source formation. Shergottites plot inside the isochron field, and define a line with an R^2 coefficient of 0.997. It has been proposed that this linear array represents both a mixing line and an isochron, implying near-simultaneous formation of enriched and depleted shergottite end-members[5,10,12,18,19]. Our new high-precision data show that this line misses the origin point (Fig. 2) and is not an isochron. Instead, the coupled ^{142}Nd–^{143}Nd systematics for the depleted and enriched shergottites constrain earlier and later Sm/Nd fractionation events, respectively. Thus, the formation age of the enriched end-member is not simultaneous with the depleted shergottite source. This observation still holds if Mars, instead of being initially chondritic, had an initial composition similar to that of the Earth's depleted mantle, characterized by present-day $\varepsilon^{142}\text{Nd} = 0$ and $\varepsilon^{143}\text{Nd} = +10.69$ (ref. 16). The shergottite mixing line still misses the origin point and is independent of these starting parameters.

A straightforward interpretation of the two-stage model for the depleted shergottite source is that it marks the time for early-formed cumulates in an MMO[4]. Solving equations (1) and (2) for depleted shergottites leads to a source formation age of $4{,}535^{+7}_{-7}$ Myr ago, which is ~32 Myr after Solar System condensation (Fig. 2).

The origin of the enriched shergottites is at present debated, and two scenarios may explain the observed coupled ^{142}Nd–^{143}Nd systematics. One (scenario A) is that the depleted shergottite source underwent variable and progressive enrichment at some time after development of this source but before production of the first shergottite magma at 474 Myr ago (Table 1). This could result from migration of metasomatic fluids or silicate melts in the martian mantle[12,20]. A second (scenario B) is that the regression line corresponds to mixing between a depleted source and a distinct enriched source that formed separately. The distinct enriched source could be late-stage quenched residual melt from the MMO formed within the martian mantle[4,12] or the martian crust[10,19,21,22]. This enriched source remains separated from the depleted reservoir until the time of melting that results in shergottite magmatism.

The shergottites plot as a mixing line to the right of a 1:1 line for time-integrated ^{147}Sm/^{144}Nd for their sources versus those measured in the lavas (Fig. 3). This means that the ^{147}Sm/^{144}Nd source ratio calculated from the initial ^{143}Nd/^{144}Nd at the time of crystallization for each rock is less than that measured in the lavas, which is the opposite of what occurs during partial melting. This can be explained by a first episode of partial melt extraction (first stage) in the shergottite source just before a second episode of partial melting (second stage) that produced shergottite magma (ref.10; Supplementary Fig. 2). The first stage results in increasing Sm/Nd in the residue because Nd is more incompatible than Sm. The second-stage shergottite magma produced from the first-stage residue has Sm/Nd > initial Sm/Nd before first-stage partial melt extraction and plots to the right of the 1:1 line (Fig. 3). Because the ^{143}Nd/^{144}Nd in the source does not have time to evolve between these two events[10], the calculated source Sm/Nd is that before the first-stage partial melting, and is thus lower than the Sm/Nd measured. Shergottites have a range of crystallization ages from 165 to 474 Myr ago (Table 1). If their parental magmas issued from a single depleted source that underwent progressive degrees of metasomatism as in scenario A above, then the cycle of first-stage melting that predates each second stage of shergottite magma production was repeated multiple times. Given these systematics, scenario A requires that variable degrees of partial melting in multiple melting stages and variable degrees of metasomatism result in measured and calculated source Sm/Nd for each shergottite in a

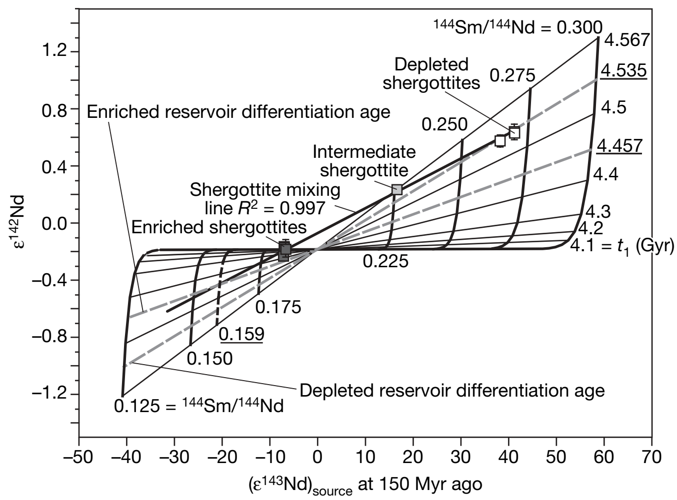

Figure 2 | A two-stage coupled ^{142}Nd–^{143}Nd evolution model for a chondritic martian magma ocean projected to 150 Myr ago. Symbols and error bars (comprised in symbol size) as in Fig. 1. The black bold line represents the mixing trend between all shergottites and misses the origin point. Black lines are loci of equal differentiation ages. Dashed grey lines are the differentiation ages for depleted and enriched shergottite sources, respectively. Black bold curves are loci of equal ^{147}Sm/^{144}Nd ratios in the source, which are identical at t_1 and the present-day. The dashed bold curve represents the maximum ^{147}Sm/^{144}Nd value of 0.159 (underlined) for the enriched end-member. Parameters used in calculations are: $\left(^{147}\text{Sm}/^{144}\text{Nd}\right)^{\text{CHUR}} = 0.1966$; $\left(^{143}\text{Nd}/^{144}\text{Nd}\right)^{\text{CHUR}}_{\text{present}} = 0.512638$; $\left(^{146}\text{Sm}/^{144}\text{Sm}\right)^{\text{CHUR}}_{T_0} = 0.008$ (see discussion in ref. 27); $\varepsilon^{142}\text{Nd}_{\text{CHUR}} = -0.18$; and $\left(^{147}\text{Sm}/^{144}\text{Sm}\right)^{\text{CHUR}} = 4.88899$.

Table 1 | Sm–Nd isotope values for shergottites

	Crystallization age t_2 (Myr)	Measured $^{147}Sm/^{144}Nd$	$\varepsilon^{142}Nd$ (2σ)		$\varepsilon^{143}Nd$ at t_2
Enriched shergottites					
Shergotty	165 (ref. 28)	0.2250	−0.233	±0.022	−6.9
Zagami	177 (ref. 28)	0.2247	−0.174	±0.026	−7.0
NWA856	186 (ref. 29)	0.2218	−0.167	±0.026	−6.8
Los Angeles	170 (ref. 28)	0.2224	−0.179	±0.061	−6.5
Intermediate shergottite					
EETA79001	173 (ref. 28)	0.3971	0.233	±0.029	16.9
Depleted shergottites					
SaU008	446 (ref. 30)	0.4863	0.647	±0.054	39.1
DaG476	474 (ref. 28)	0.4924	0.639	±0.031	38.9
SaU094	446 (ref. 30)	0.4629	0.569	±0.042	36.2

$^{147}Sm/^{144}Nd$ are the directly measured values in the samples. $\varepsilon^{143}Nd$ are calculated at t_2 (age of crystallization) relative to CHUR. See Supplementary information for more details.

manner that forms a well-defined line. This complex scenario is fortuitous and highly unlikely.

A simpler explanation is scenario B, in which all shergottites are derived from a unique depleted source having first undergone one partial melting event before shergottite parental magma production. The enriched and intermediate shergottites are then explained by mixing different proportions of melts derived from the depleted source and a distinct enriched source. In this case, the enriched source has Sm/Nd smaller than the most enriched measured lava, and falls to the left of the 1:1 line. This corresponds to $^{147}Sm/^{144}Nd \leq 0.159$ (Fig. 3). For this $^{147}Sm/^{144}Nd$ value, the enriched source formed at 4,457 Myr ago for a chondritic Mars (Fig. 2) or ~100 Myr after core formation. These Sm–Nd systematics could reflect either the final stages of MMO solidification, or the crust that contaminates the depleted shergottite magmas.

The well-defined mixing lines projecting away from the depleted shergottites in Figs 2 and 3 are inconsistent with the scatter expected where individual magma batches are contaminated by crust. This is supported by the chondritic initial $^{187}Os/^{188}Os$ of all shergottites[23]. Ancient basaltic or felsic crust on Mars will have superchondritic Re/Os and evolve to very high $^{187}Os/^{188}Os$ in hundreds of Myr, such that very small amounts of crust would drive contaminated magmas to much greater $^{187}Os/^{188}Os$ ratios than observed[23]. Also, NWA1068 is an enriched shergottite with a more primitive major-element composition (that is, molar (Mg/Mg+Fe) = 0.59) than those studied here (molar (Mg/Mg+Fe) = 0.25–0.52), but they all have nearly identical $\varepsilon^{143}Nd$ values of −6.5 to −7.0 (Table 1; ref. 24). This is inconsistent with models for progressive crustal contamination during fractional crystallization[20].

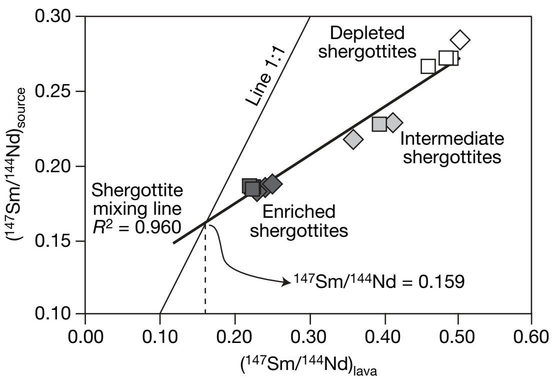

Figure 3 | The $^{147}Sm/^{144}Nd$ time-integrated ratios for mantle sources versus the measured ratios in lavas. The $^{147}Sm/^{144}Nd$ source ratio calculated using equation (2) and considering that the source differentiated at T_0 and evolved with constant parent/daughter ratios until the sample being measured was extracted from the mantle at t_2. All of the shergottites plot to the right of the 1:1 line. This has been explained by two partial melting stages close in time in the shergottite source. Data from this study: square symbols as in Fig. 1. Data from the literature[10,20,31,32]: diamond symbols, with the same greyscale as the data from this study.

In summary, the linear arrays observed for Sm–Nd isotope systematics of the shergottites are consistent with mixing between two distinct source reservoirs formed at ~4.535 Gyr and ~4.457 Gyr ago. The origin of the incompatible-trace-element-enriched reservoir is not likely to be found in ancient crust or metasomatism of the mantle. Instead, the most probable origin for this reservoir is late-stage quenched residual melt from the MMO. For a $^{147}Sm/^{144}Nd$ ratio between 0.150 and 0.159, comparable to the value of 0.164 inferred for the lunar KREEP (that is, strongly enriched in potassium (K), rare-earth elements (REE) and phosphorus (P)) component[25] and to the value of 0.152–0.154 for the trapped melt at the end of MMO crystallization[4,10], the formation time interval of the depleted and enriched reservoirs record progressive MMO crystallization to ~100 Myr after core formation. This is in agreement with the survival of shallow magma oceans for 100–200 Myr without any heating processes in the presence of a transient atmosphere[6]. It implies that a primitive atmosphere was present on Mars during this time interval, first acting as an insulating blanket and slowing down the solidification process[6], but later removed within the first billion years[26].

METHODS SUMMARY

Coarse fractions of three desert meteorites (Los Angeles, SaU008 and DaG476) were leached in 50% acetic acid (see 'Terrestrial contamination' in the Supplementary Information). Powders were dissolved in Teflon beakers using ultrapure and concentrated $HF\text{-}HNO_3\text{-}HClO_4$ (10:2:1) at 160 °C for 1 week. After evaporation, concentrated HCl was added and residues were redissolved at 160 °C for 1 week. After re-dissolution in HCl, a 5% aliquot of each solution was spiked with a mixed ^{150}Nd–^{149}Sm spike. Rare-earth elements (REE) were purified by cation exchange chromatography using AG50-X8 resin, followed by Ce, Sm and Nd separation with Eichrom Ln-Spec resin. The Nd cut was then purified of Ce using solvent extraction[17] (see 'Analytical techniques' in the Supplementary Information). Total procedural blanks are ≤30 pg and ≤60 pg for Nd and Sm, respectively.

Spiked Sm and Nd aliquots were analysed on a multi-collector inductively coupled plasma mass spectrometer (MC-ICP-MS, Nu Plasma) at the University of California in Davis. The high-precision $^{142}Nd/^{144}Nd$ measurements of unspiked Nd aliquots were performed on a nine-faraday-cup Triton thermal ionization mass spectrometer at the Johnson Space Center (Supplementary Tables 1 and 2), as positive metal ions in multidynamic mode with rotating amplifiers between blocks[27]. The data are normalized to $^{146}Nd/^{144}Nd = 0.7219$ to correct for instrument mass fractionation. The interference of ^{142}Ce on ^{142}Nd was monitored with ^{140}Ce. Corrections for this interference to $^{142}Nd/^{144}Nd$ ranged from 0.2 to 9.5 p.p.m., and for all but one standard run and four Iceland picrite runs, were ≤4 p.p.m. and within 2σ internal and established 2σ external precisions on $^{142}Nd/^{144}Nd$ (Supplementary Table 2). Sm interferences on Nd were monitored with ^{147}Sm, which was never detectable on the faraday cups above background noise. Scans on the electron multiplier showed that ^{147}Sm was never above 1,000 counts s^{-1}, and therefore negligible. Internal precisions of ±3 p.p.m. were achieved for 600 ng standard aliquots for measuring times of 5 to 7 h. The average for these standards was $^{142}Nd/^{144}Nd = 1.1418402 \pm 0.0000034$ ($n = 14$, 2σ) and is used as the reference value of $\varepsilon^{142}Nd = 0$ for calculating $\varepsilon^{142}Nd$. The 2σ external precision for these 14 standard runs for $\varepsilon^{142}Nd$ is ±0.03 (±3 p.p.m.) (see 'Mass spectrometry' in Supplementary Information).

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Supplementary Information is linked to the online version of the paper at www.nature.com/nature.

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