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Extreme oxygen-isotope compositions in magnetite from unequilibrated ordinary chondrites

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Primitive meteorites (such as the unequilibrated ordinary chondrites) have undergone only minor thermal processing on their parent asteroids, and thus provide relatively unaltered isotopic records from the early Solar System. For terrestrial materials, oxygen isotope compositions form a linear array called the terrestrial fractionation line[1]. In meteorites the oxygen isotopic composition commonly deviates from this line[2], the magnitude of the deviation being expressed by the quantity $\Delta^{17}O$. Such deviations, which cannot be explained by mass-dependent fractionation processes, are probably caused by the mixing of two or more nebular components having different nucleosynthetic histories, for example, solids and gas. But no direct evidence for the oxygen isotopic composition of the latter (which is the dominant oxygen reservoir) has hitherto been available. Here we report *in situ* oxygen-isotope measurements of magnetite grains in unequilibrated ordinary chondrites. Magnetite (which formed by aqueous alteration of metal in the parent asteroid) may serve as a proxy for nebular H_2O. We measured a value of $\Delta^{17}O \approx 5‰$, much higher than typical values of $0–2‰$ in ordinary-chondrite silicate grains. Our results imply that a nebular component of high-$\Delta^{17}O$ H_2O was incorporated into the parent asteroid of the unequilibrated ordinary chondrites.

The discovery of large oxygen-isotope variations both within[3] and among[4] the chondrite groups demonstrated the existence of significant isotopic heterogeneities in the solar nebula. At the places and times in the nebula where the chondrite groups (or sets of related groups) formed, nebular grains incorporated material from reservoirs having distinct oxygen-isotope compositions reflecting different nucleosynthetic histories. An important factor is the unique cosmochemistry of oxygen that simultaneously makes it the dominant rock-forming element as well as one of the principal components of the nebular gas phase. For this reason, disequilibrium oxygen-isotope distributions among meteorites are commonly modelled in terms of two reservoirs, one solid, the other gaseous (see, for example, Clayton and Mayeda[5]).

In minerals from unequilibrated ordinary chondrites (UOCs), major cations such as Mg^{2+} and Fe^{2+} have not exchanged appreciably since formation in the nebula, and in most silicates, O diffuses more slowly than these ions[6,7]. Thus most minerals retain their nebula compositions. However, fluid transported along grain surfaces in asteroids has produced some new, low-temperature minerals, for example magnetite[8] and phyllosilicates[9].

A minor subset of UOCs contain magnetite (Fe_3O_4)-carbide assemblages; Krot *et al.*[8] inferred that the magnetite resulted from oxidation of Fe–Ni metal, probably by H_2O in an asteroid. Because all the oxygen in magnetite comes from the oxidant, the study of magnetite can constrain the O-isotope composition of the oxidizing fluid.
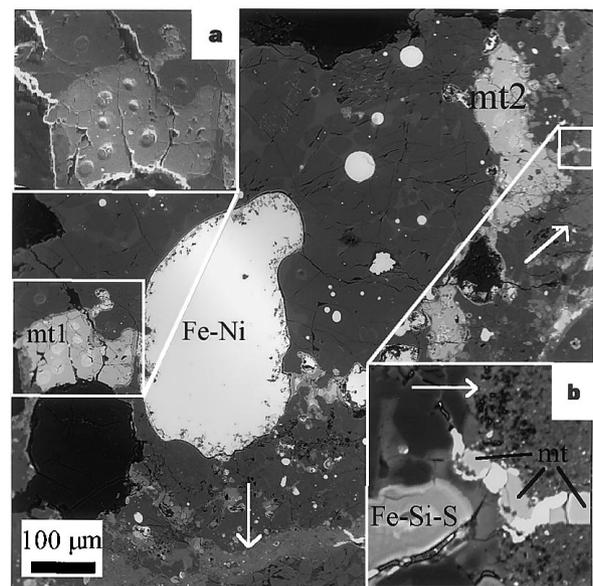
Figure 1 Back-scattered-electron image of part of low-FeO porphyritic olivine chondrule (PO1) in Semarkona USNM1805-5. Arrows indicate boundaries between the chondrule and matrix. In the chondrule white nodules are Fe–Ni metal with taenite, carbides and troilite. Light-grey objects with irregular shapes are magnetite (mt1 and mt2); the magnetite grains are surrounded by fayalite-like minerals ~5 μm thick. Dark areas on top of the image and at bottom of mt1 are plucked regions. **a**, Scanning electron microscopy image of mt1, showing seven craters formed by ion-microprobe sputtering. **b**, High-magnification back-scattered-electron image of chondrule–matrix boundary. A magnetite-bearing vein extends from the chondrule into the matrix implying that this vein formed after the chondrule and matrix were compacted together.

We used the UCLA Cameca ims 1270 ion microprobe to make *in situ* measurements of O-isotope abundances in magnetite and olivine from two UOCs, LL3.0 Semarkona (the most primitive chondrite[10]) and LL3.6 Ngawi (type 3.0 indicates the least altered chondrites[11]; Ngawi mean subtype by A. Rubin, personal communication). Measurements were made at high mass-resolving power by using a defocused Cs^+ beam to sputter ~ 12–$25\,\mu m$ diameter craters (Fig. 1a) on carbon-coated polished thin sections. We measured $^{16}O^-$ ions in a Faraday cup, $^{17}O^-$ and $^{18}O^-$ on an electron multiplier. A more detailed description of the techniques is given elsewhere[12].

We studied 11 magnetite grains in Semarkona and 2 in Ngawi; sizes ranged from ~ 30 to $\sim 180\,\mu m$. Magnetite is relatively abundant in Semarkona; point-counting an area of $\sim 2\,cm^2$ yields an abundance of $40\,mg\,g^{-1}$ (corresponding to $11\,mg\,g^{-1}$ oxygen). Whereas kamacite and taenite nodules always show smooth, rounded and embayed surfaces, magnetite grains have ragged boundaries (Fig. 1) suggesting irregular growth. Some magnetite assemblages show strong textural evidence of *in situ* formation; for example, Fig. 1b shows a magnetite–cohenite vein filling a crack in a chondrule and adjacent matrix, implying that the vein formed after the chondrule and matrix were compacted together in the parent asteroid.

Oxygen-isotope compositions of magnetite grains are summarized in Table 1 and plotted in Fig. 2. Compositions of chondrule olivines of Semarkona measured at UCLA during the same period are also shown in Fig. 2; as expected, these are similar to the compositions of UOC chondrules[13]. They will be discussed in detail elsewhere. Four generalizations may be made: (1) magnetite compositions in Semarkona and Ngawi are similar; (2) the Semarkona data show a range of $\sim 13‰$ in $\delta^{18}O$; (3) a regression line through the magnetite compositions has a slope of 0.70; and (4) the mean $\Delta^{17}O$ is $\sim 5‰$.

The mean $\Delta^{17}O$ of $\sim 5‰$ is the highest measured in solid materials of Solar-System origin, although interstellar Al_2O_3 grains have more extreme compositions[14,15]. Our $\Delta^{17}O$ values of Semarkona magnetite are somewhat higher than those reported by Saxton *et al.*[16] (mean $\Delta^{17}O$ of $\sim 3‰$), but the data sets overlap within the uncertainties. Three measurements of two Ngawi magnetite grains plot close to the mean of Semarkona magnetite.

The $\delta^{18}O$ values in Semarkona magnetite span a wide range of $\sim 13‰$. Because there is the potential for such variations to be the result of sample contamination or a mass-dependent instrumental artefact[17], we examined these possibilities in detail. The data were corrected for instrumental drift in the standard magnetite; corrections were typically small, at most 1.5‰ in $\delta^{18}O$. The sputtered craters were examined, but no 'contaminating' inclusions of silicates or other oxides were observed. Although some craters have cracks, there is no systematic difference between data for cracked and crack-free craters. If variable instrumental fractionation were due to grain-boundary (that is, topographic) effects, we would expect that measurements on smaller grains would show more scatter than larger grains. We observed the reverse; the largest magnetite, 1805mt1, yielded an isotopic variation of 10‰ in $\delta^{18}O$, and several smaller magnetites yielded much smaller ranges (Table 1 and Fig. 2). These arguments make it unlikely that the large variation is an experimental artefact.

There is no resolvable correlation between $\delta^{18}O$ and position within an individual magnetite grain. This is not inconsistent with expectations regarding mineral growth and scaling during corrosion. Unfortunately, it has the consequence that we are not able to specify whether $\delta^{18}O$ increased or decreased during the course of magnetite growth.

Formation of the magnetite from a semi-infinite H_2O reservoir (for example, the solar nebula) would yield relatively constant $\delta^{18}O$ because the fractionation factor between magnetite and water is not very temperature sensitive in the range of likely formation temperatures for magnetite in chondrites[18]. The large (13‰) range

Figure 2 Oxygen-isotope measurements for Semarkona and Ngawi magnetite. Data are plotted on a $\delta^{18}O$–$\delta^{17}O$ diagram; 1σ error bars are shown. Ion microprobe data for Semarkona olivine are also shown; these agree well with previous whole-rock and chondrule data, which fall near the unequilibrated ordinary chondrite (UOC) line. The magnetite $\Delta^{17}O$ values are $\approx 4‰$ larger than those of olivine. The terrestrial-fractionation (TF) line and a regression line for our UOC magnetite data are shown for reference. The band bounded by dotted lines represents mass-dependent fractionation with a $\Delta^{17}O$ of $4.9 \pm 0.9‰$ (95% confidence interval for the mean of UOC magnetite $\Delta^{17}O$ values).

Table 1 Oxygen isotope compositions of Semarkona and Ngawi magnetites

Sample	$\delta^{18}O \pm 1\sigma^*$† (‰)	$\delta^{17}O \pm 1\sigma^*$†‡ (‰)	$\Delta^{17}O$§ (‰)	Site‖
15 July 1996 Semarkona UCLA229				
1-1¶	−2.8 ± 0.6	3.1 ± 1.0	4.6	Matrix
2-1	−4.0 ± 0.8	2.3 ± 1.3	4.4	PP1
2-2	−2.4 ± 0.7	3.0 ± 1.1	4.3	PP1
14 October 1996 Semarkona USNM1805-5				
1-1	9.4 ± 1.2	10.4 ± 1.3	5.5	PO1
1-2	−1.4 ± 1.4	3.3 ± 1.3	4.1	PO1
1-3	4.4 ± 1.2	7.3 ± 1.3	5.0	PO1
1-4	0.8 ± 1.3	4.9 ± 1.2	4.5	PO1
2-1	4.9 ± 1.2	6.7 ± 1.2	4.2	PO1
2-2	5.0 ± 1.5	8.4 ± 1.5	5.8	PO1
2-3	6.6 ± 1.7	9.4 ± 1.6	6.0	PO1
3	1.2 ± 1.1	5.4 ± 1.3	4.8	Matrix
14 November 1996 Semarkona UNM312				
1	5.0 ± 1.7	9.5 ± 1.0	6.9	PP2
2	5.0 ± 1.6	8.2 ± 1.2	5.6	PP2
3-1	−2.0 ± 1.6	3.6 ± 1.2	4.7	Matrix
3-2	0.3 ± 1.5	4.6 ± 1.0	4.5	Matrix
3-3	2.1 ± 1.5	6.6 ± 1.1	5.5	Matrix
4¶	2.1 ± 1.5	5.7 ± 0.7	4.6	Rim
5-1	1.2 ± 1.6	5.6 ± 1.1	5.0	Matrix
5-2	2.5 ± 1.6	5.9 ± 0.9	4.6	Matrix
15 November 1996 Ngawi USNM2483				
1-1	1.0 ± 0.9	6.0 ± 1.5	5.5	Matrix
1-2	0.7 ± 0.9	4.3 ± 1.5	4.0	Matrix
2	1.6 ± 1.1	5.4 ± 1.5	4.6	Rim
17 November 1996 Semarkona USNM1805-5				
1-5	6.5 ± 0.9	8.8 ± 0.9	5.4	PO1
1-6	3.8 ± 0.7	8.8 ± 0.9	6.9	PO1
1-7	4.3 ± 0.9	7.2 ± 1.0	4.9	PO1
15 January 1997 Semarkona UCLA229				
1-2¶	−0.7 ± 0.9	3.5 ± 1.0	3.9	Matrix
3	−2.8 ± 0.9	0.8 ± 1.4	2.3	Rim

* $\delta^{18}O = \{[(^{18}O/^{16}O)_{sample}/(^{18}O/^{16}O)_{SMOW}] - 1\} \times 10^3$, where $\delta^{18}O$ is in units of per mil, SMOW indicates standard mean ocean water value and $\delta^{17}O$ is defined analogously.
† Errors include the standard deviations of the standard during each analytical session.
‡ OH^- interferences were ≤0.5‰, typically ≤0.1‰.
§ $\Delta^{17}O \equiv \delta^{17}O − 0.52\delta^{18}O$.
‖ PO, porphyritic olivine chondrule; PP, porphyritic pyroxene chondrule; PO1 and PP1 are low-FeO; PO2 is high-FeO; rim indicates rim around chondrules.
¶ Weighted means of two or three successive measurements on the same spot.

observed therefore suggests that a limited supply of oxidant was largely consumed during magnetite formation (resulting in Rayleigh-type fractionation). This reinforces the conclusion of Krot et al.[8] that the magnetite formed in an asteroidal setting.

We therefore examined models involving asteroidal oxidation by H_2O. There are no clear constraints on the number of independent oxygen reservoirs in the ordinary-chondrite part of the solar nebula. In ordinary chondrites, mean whole-rock $\Delta^{17}O$ (ref. 13) correlates with degree of oxidation measured by bulk $FeO/(FeO + Mg)$ ratios[19]. Although it is possible that the oxygen in UOC magnetite is fully independent of the oxygen in silicates, we investigated models that could explain not only the high $\Delta^{17}O$ of magnetite but also the correlation between $\Delta^{17}O$ and degree of oxidation. We defined a UOC line having a slope of 0.78 (Fig. 2) based on O-isotope data for whole-rock falls and for chondrules[13]. According to this picture, the high-$\Delta^{17}O$ oxidant that formed UOC magnetite should have a composition on or near this UOC line.

We do not try to define the asteroidal source of the water. To produce the observed slope in the magnetite data (0.70) requires exchange between the oxidant and another reservoir having lower $\Delta^{17}O$; we suggest that UOC chondrule silicates are a plausible choice. The observed fractionation is produced by simultaneously forming magnetite and phyllosilicates, with the latter consuming 2.5 times as much oxygen as the former. This amount of phyllosilicate is somewhat higher than that obtained by a comparison of our estimate abundance ($11 \, mg \, g^{-1}$) of O in magnetite with the H_2O concentration of $16.2 \, mg \, g^{-1}$ ($= 14.4 \, mg \, g^{-1}$ O) measured in Semarkona[19], but some phyllosilicate water may have been lost during mild metamorphism. We assume that the H_2O was present as a liquid, that the equilibrium fractionation between magnetite and $H_2O(l)$ is $-11‰$ in $\delta^{18}O$ and that between phyllosilicate OH and H_2O (l) is $+12‰$ (roughly corresponding to $T = 300 \, K$ based on estimated bond-water O-isotope fractionations[20]), and that, after formation, magnetite and phyllosilicate remained isolated

Figure 3 A model calculation for formation of magnetite and phyllosilicates. This was made to reproduce the O-isotope trend of Semarkona magnetite having a slope of 0.70. Initial $H_2O(l)$ was assumed to have $\delta^{18}O = 21.0$ and $\delta^{17}O = 17.5‰$ ($\Delta^{17}O = 6.6‰$). The equilibrium fractionation factors between magnetite and $H_2O(l)$ of $-11‰$ and between hydroxyl O in smectite and $H_2O(l)$ of $+12‰$ were used; points show increments of 10% in fraction of water reacted. It was assumed that 2.5 times as much as oxygen goes into forming phyllosilicates as into magnetite. To reproduce the trend of decreasing $\Delta^{17}O$ with decreasing $\delta^{18}O$, we assume that after each 0.5% fractionation, 0.15% of the $H_2O(l)$ oxygen is replaced with silicate oxygen having the composition of average UOC chondrules.

from the remaining H_2O. To account for a slope of 0.70, we further assume a starting $\Delta^{17}O$ of 6.6‰ and that, after each 0.5% of the $H_2O(l)$ reacted, 0.15% of the oxygen remaining in this phase was replaced by chondrule oxygen with $\delta^{18}O \approx 5.0‰$, $\delta^{17}O \approx 3.5‰$ (ref. 13).

The first magnetite to form has $\delta^{18}O = 10‰$, thus the $\delta^{18}O$ of the $H_2O(l)$ was 21‰. As shown in Fig. 3, the magnetite oxygen tracks that of the remaining H_2O. The trend of the magnetite composition curves slightly but the mean slope is close to that of the data array, 0.70. This simple model of magnetite formation can account for the observed trend by interpreting the oxidant to have been H_2O with an O-isotope composition relatively close to the extrapolation of the UOC trend to $\Delta^{17}O \approx 7‰$. In principle, the model could be tested by analysing the O-isotope composition of the H_2O evolved from phyllosilicates at low temperatures; although the $\Delta^{17}O$ of this H_2O would include a substantial contribution from the anhydrous precursor, it should still have a $\Delta^{17}O$ appreciably higher than the bulk meteorite.

An interesting question is whether the nebular $H_2O(g)$ could have had such a high $\Delta^{17}O$ value while the silicate precursors of the chondrules were forming. Cosmochemical evidence seems best understood in terms of near-complete volatilization of pre-solar materials in the nebula[21] followed by recondensation. If the $\Delta^{17}O$ of the nebular gas were $\geq 6‰$ when chondrule precursors were forming, it is remarkable that chondrule $\Delta^{17}O$ values are 0–2‰. This might suggest that $\Delta^{17}O$ values in the UOC region were initially near 0‰, but that subsequently infalling materials had higher $\Delta^{17}O$ values, and that the $\Delta^{17}O$ of the nebular gas gradually increased. □

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