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Microscale oxygen isotopic exchange and magnetite formation in the Ningqiang anomalous carbonaceous chondrite

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Abstract—We report in situ measurements of O-isotopic compositions of magnetite, olivine and pyroxene in chondrules of the Ningqiang anomalous carbonaceous chondrite. The petrographic setting of Ningqiang magnetite is similar to those in oxidized-CV chondrites such as Allende, where magnetite is found together with Ni-rich metal and sulfide in opaque assemblages in chondrules. Both magnetite and silicate oxygen data fall close to the carbonaceous-chondrite-anhydrous-mineral line with relatively large ranges in δ^{18} O in magnetite (-4.9 to +4.2‰) and in silicates (-15.2 to -4.5‰). Magnetite and silicates are not in O-isotopic equilibrium: the weighted average Δ^{17} O (= δ^{17} O - 0.52 × δ^{18} O) values of magnetite are 1.7 to 3.6‰ higher than those of the silicates in the same chondrules. The petrological characteristics and O-isotopic disequilibrium between magnetite and silicates suggest the formation of Ningqiang magnetite by the oxidation of preexisting metal grains by an aqueous fluid during parent body alteration. The weighted average Δ^{17} O of -3.3 ± 0.3‰ is the lowest magnetite value measured in unequilibrated chondrites and there is a positive correlation between Δ^{17} O values of magnetite and silicates in each chondrule. These observations indicate that, during aqueous alteration in the Ningqiang parent asteroid, the water/rock ratio was relatively low and O-isotopic exchange between the fluid and chondrule silicates occurred on the scale of individual chondrules. *Copyright* © 2003 Elsevier Ltd

1. INTRODUCTION

The origin of the observed heterogeneity of oxygen isotopes in solar system materials is one of the key unsolved questions regarding the origin and evolution of the solar nebula (Clayton, 1993; Wasson, 2000). The most ¹⁶O-rich components are preserved in the most refractory phases in unequilibrated chondrites, such as Ca-Al-rich inclusions (CAIs). Common and more volatile components (i.e., chondrules and matrix), are relatively depleted in ¹⁶O and fall near or just above the terrestrial mass fractionation (TF) line on three-oxygen-isotopic diagrams. Recent spot analyses show that some olivine grains in chondrules have ¹⁶O-enrichments similar to those in CAIs (Jones et al., 2002; Yurimoto and Wasson, 2002).

In unequilibrated chondrites, magnetite, formed by reaction between oxygen-free phases (Fe-Ni metal or FeS) and fluid (H₂O), is a good indicator of O-isotopic compositions of the oxidizing fluid present in asteroids and thus provides clues regarding the O-isotopic composition of the solar nebular gas. Previous studies showed that magnetite has the highest $\Delta^{17}O$ ($=\delta^{17}O - \delta^{18}O \times 0.52$) values in each chondrite group implying that $\Delta^{17}O$ in the low-temperature solar-nebula gas was higher than in refractory oxides and chondrule silicates (Choi et al., 1997, 1998, 2000).

As part of our continuing studies of the O-isotopic composition of magnetite in unequilibrated chondrites, we investigated the Ningqiang carbonaceous chondrite. Ningqiang is an anomalous carbonaceous chondrite, whose chemical composition and petrological characteristics are similar to those of CK and CV chondrites but not identical with them (Rubin et al., 1988; Kallemeyn et al., 1991). The classification of Ningqiang has meandered with time. It was classified as CV3-anomalous by Rubin et al. (1988) and as CK3-anomalous by Kallemeyn et al. (1991). Ningqiang has slightly lower Mg-normalized abundances of refractory lithophile elements and higher abundances of volatile elements than CK and CV chondrites. Thus Kallemeyn (1996) suggested that Ningqiang is best left as ungrouped.

Oxygen isotopic compositions of bulk and separated components such as chondrules and matrix in Ningqiang were measured by Mayeda et al. (1988) and Clayton and Mayeda (1999). No spot analyses for O-isotopic compositions of minerals in Ningqiang have been reported previously.

2. ANALYTICAL PROCEDURES

A polished thin section of Ningqiang (UCLA 421) was studied using optical microscopy and backscattered electron (BSE) imaging to find magnetite grains suitable for in situ measurements of oxygen isotopes. BSE images were obtained using the UCLA LEO 1430 scanning microscope before and after isotope measurements. Electron-probe microanalyses of magnetite, olivine and pyroxene along with Fe-Ni metal and sulfide were performed after isotope measurements using the Cameca 50X at the Korean Basic Science Institute and also using the JEOL JXA 8900R at National Center for Inter-University Research Facilities, Seoul National University. The accelerating voltage was 15 keV for silicates and magnetite and 20 keV for metals and sulfide, the beam current was 10–20 nA, the beam diameter was $\sim 1 \ \mu m$ and the count durations were 20 s.

Oxygen isotopic compositions of magnetite, olivine and pyroxene were measured using the UCLA Cameca ims 1270. A

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Table 1. Representative	electron	microprobe	analyses	(in wt%)	of mine	ral phases	in 1	Ningqiang	chondrules:	forsteritic-core	olivine	(ol-1),
favalitic-rim olivine (ol-2),	Ca-poor	pyroxene (py	c-1), Ca-rio	ch pyroxe	ene (px-2)	, magnetite	e (m	t), Ni-rich	metal (Fe-Ni	i), and troilite (I	FeS).	

Sample	ol-1	ol-2	px-1	px-2	mt		Fe-Ni	FeS
No. of analyses	38	31	41	12	29		16	16
SiO ₂	41.3	35.3	58.1	50.4	< 0.2	Fe	31.7	61.8
TiO ₂			0.2	0.7	< 0.2	Ni	65.9	0.2
$Al_2 \tilde{O}_3$	< 0.2	< 0.2	1.0	6.7	0.9	Co	2.1	
Cr_2O_3	< 0.2	< 0.2	0.2	0.6	1.5	Cr	< 0.2	< 0.2
FeO	4.5	36.0	0.8	0.7	89.3	S	_	36.2
MnO	< 0.2	0.3	< 0.2	0.2	< 0.2			
MgO	53.6	27.8	38.4	19.7	0.3			
CaO	0.2	0.2	0.7	20.3	0.2			
NiO	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2			
Total	100.1	100.0	99.9	99.5	92.6	Total	99.8	98.3
Phases	Fa _{4.5}	Fa _{42.1}	Wo _{1.3} , Fs _{1.1}	Wo _{42.0} , Fs _{1.1}		Phases	Ni ₂ Fe	FeS

defocused primary Cs⁺ beam of \sim 0.3 nA in intensity and \sim 15μ m in diameter was used to sputter the carbon coated sample surface and to produce secondary ions. Negatively charged secondary ions of ¹⁶O were measured by a Faraday cup, those of ¹⁷O and ¹⁸O by an electron multiplier. A high mass resolving power of ~ 6800 was used to separate ${}^{16}\text{OH}^-$ from the ${}^{17}\text{O}^$ peak. The contribution of the tail of ${}^{16}OH^-$ to the ${}^{17}O^-$ peak was monitored; the required correction was typically $\sim 0.1\%$ and always less than 0.4‰. A terrestrial magnetite, LP204 (almost pure Fe₃O₄ with δ^{18} O = +8.9‰, Valley and Graham, 1991) was used as magnetite standard, while San Carlos olivine $(Fa \approx 11 \text{ mol}\% \text{ and } \delta^{18}\text{O} = +5.25\%$, Eiler et al., 1995) was used as standard for silicates. Errors introduced by the correction procedures (mass fractionations, detector gains, and tail corrections) were all propagated into the final errors quoted: we use 1σ errors in text, tables and figures unless stated. However, matrix effects were neither monitored nor corrected. Based on our previous experiments, we estimate that the error due to a matrix effect is minor relative to our quoted analytical uncertainties. Analytical procedures are described in more detail in Choi et al. (1997).

3. MINERALOGY AND PETROLOGY

Ningqiang contains chondrules, chondrule fragments and amoeboid olivine inclusions (AOIs) with rare CAIs in a relatively abundant (\sim 50 vol%) fine-grained matrix (Rubin et al., 1988). Petrologically, Ningqiang closely resembles oxidized CV chondrites, such as Allende, except for the low abundance of CAIs.

In chondrules, forsteritic olivines (fayalite content <10 mol%) are often surrounded by fayalitic olivine rims (up to Fa65). Fayalite contents of olivine grains in matrix are similar to those of the fayalitic olivine rims. Pyroxene has a relatively narrow range in FeO content (ferrosilite content <4 mol%). Wollastonite contents in high-Ca pyroxene in chondrules fall between 32 and 45 mol%.

Occurrences of magnetite in chondrules are very similar texturally to those in oxidized CV chondrites, such as Allende, Kaba and Mokoia (Choi et al., 1997; Krot et al., 1998), where magnetite replaced preexisting Fe-Ni metal and left Ni-rich residual metal (Ni₂Fe). Troilite is often associated with magnetite and Ni-rich metal. Ningqiang magnetite has relatively high Cr contents (Cr₂O₃ ~ 1.5 wt%; Rubin et al., 1988, this

study), which is also similar to magnetite found in opaque nodules with Ni-rich metal and sulfide in CV chondrites (Choi et al., 1997; Krot et al., 2000). Average chemical compositions of major mineral phases in Ningqiang are summarized in Table 1.

Seven magnetite-bearing chondrules (including chondrule fragments) were studied in detail for both petrologic characteristics and O isotopes. A brief petrological description for these chondrules is given in Table 2. BSE images of one of them are shown in Figure 1. All seven chondrules have high abundances of opaque-nodules that consist of magnetite, Ni-rich metal and troilite. They are either porphyritic olivine or porphyritic olivine-pyroxene chondrules in mineralogy and texture. Mean FeO contents are low (0.035 <FeO/(FeO + MgO) <0.08). With the exception of two chondrules (CHD-4 and 9), plagio-clase-normative mesostasis (or its alteration products) is rare or absent. Ca-rich pyroxene is also rare but occurs in most chondrules.

4. OXYGEN ISOTOPIC COMPOSITIONS

Oxygen isotopic compositions of magnetite, olivine and pyroxene grains in seven chondrules are summarized in Table 3 and plotted in Figure 2. Two measurements for magnetite in CHD-6 are from a single magnetite-bearing nodule, while all other measurements for magnetite and silicates are from individual grains. All of them fall close to carbonaceous chondrites anhydrous mineral (CCAM) line with relatively large δ^{18} O ranges.

Table 2. Petrological characteristics of the chondrules measured for O-isotopes. $^{\rm a}$

Chondrules	nondrules Texture		Fa	Fs	Wo	An	
	DOD	Irrogular	25.81	a.1.5	~ 20	a. 80	
CHD-4	FOF	ritegulai	3.3-6.4	1.5	.~30	~ 80	
CHD-5	PO	Circular	2.3 - 6.2	~ 1.0		_	
CHD-6	POP	Fragment	3.6–7.9	1.0 - 2.1	_		
CHD-7	POP	Irregular	3.9-8.5	0.5 - 1.0	38–43		
CHD-8	POP	Irregular	3.9–7.6	1.0 - 3.4	_		
CHD-9	PO	Circular	1.5 - 6.0	~ 1.5	_	~92	
CHD-10	POP	Fragment	6.0–14	~ 0.5	~36		

^a Fa = fayalite content in core of olivine; Fs = ferrosilite contents of pyroxene; Wo = wollastonite contents of Ca-rich pyroxene; An = anorthite contents of mesostasis; POP = porphyritic olivine pyroxene texture; PO = porphyritic olivine texture.



Fig. 1. Backscattered electron image of (a) a magnetite-bearing chondrule (CHD-9) in Ningqiang and (b) a more detailed view of the magnetite-rich area. CHD-9 is a porphyritic olivine chondrule with abundant opaque nodules. Opaque nodules consist of magnetite, Ni-rich metal and troilite. Ellipsoids mark the ion microprobe spots (a black one on magnetite and three white ones on olivine). Scale bar for (b) is 50 μ m.

Oxygen isotopic compositions of a dark inclusion, matrix and the bulk are reported in Clayton and Mayeda (1999), and of eight separated chondrules measured by Mayeda et al. (1988). They are plotted in Figure 2 together with our measurements. The weighted average of Δ^{17} O in our magnetite (-3.3 ± 0.3‰) is similar to those in the dark inclusion (-3.09‰) and matrix (-3.71‰) and somewhat higher than the bulk (-4.55‰). Our chondrule olivine and pyroxene Δ^{17} O values (from -4.3 to -7.7%) are more negative than observed in the separated chondrule data (from -2.9 to -5.6%). The two data sets may simply represent two chondrule populations differing in O-isotopic composition or, more likely, the bulk chondrules may consist of mixtures of primary, ¹⁶O-rich phases (silicates) and secondary, ¹⁶O-poor phases (e.g., magnetite).

Magnetite grains in each chondrule have similar O-isotopic compositions, not resolvable within our analytical uncertainty.

Table 3. Oxygen isotopic compositions (in ‰) of magnetite, olivine and pyroxene and mineralogical compositions (in %) of the ion-probed silicates in Ningqiang.

Hosts	Minerals	δ^{18} O	1σ	$\delta^{17}O$	1σ	$\Delta^{17}O$	1σ	Comments
CHD-4	mt	-2.2	0.9	-4.1	1.0	-3.0	1.1	
		-1.6	1.0	-2.8	0.8	-2.0	1.0	
		-0.4	0.8	-2.3	0.9	-2.1	1.0	
	ol	-4.5	1.5	-7.3	1.0	-5.0	1.1	Fa = 3.5
		-5.2	1.5	-6.9	1.0	-4.2	1.1	Fa = 4.5
CHD-5	mt	-3.0	0.9	-5.0	0.9	-3.4	1.0	
	ol	-7.1	2.2	-9.2	1.1	-5.5	1.3	Fa = 2.3
		-5.6	1.9	-8.8	1.4	-5.9	1.3	Fa = 6.0
CHD-6	mt	-3.8	1.4	-3.9	1.0	-1.9	1.1	
		-4.6	1.5	-4.5	1.0	-2.1	1.1	
	ol	-4.9	2.0	-8.2	1.4	-5.6	1.3	Fa = 3.9
CHD-7	mt	1.1	0.9	-4.1	0.7	-4.6	0.9	
	ol	-8.7	1.4	-12.2	1.4	-7.7	1.3	Fa = 4.0
	px	-9.9	1.8	-13.6	1.2	-8.4	1.2	Wo = 37.8; Fs = 0.8
		-9.0	1.1	-9.9	0.9	-5.1	1.0	Wo = 1.0; Fs = 0.7
CHD-8	mt	4.2	1.2	-0.1	0.8	-2.2	1.0	
		3.4	0.9	-1.4	0.9	-3.1	1.0	
	ol	-9.7	1.8	-10.8	0.9	-5.8	1.0	Fa = 3.9
CHD-9	mt	-3.0	0.9	-5.6	0.8	-4.0	1.0	
		-4.9	1.1	-7.4	0.6	-4.9	0.9	
	ol	-13.4	1.5	-15.3	1.0	-8.4	1.1	Fa = 4.2
		-15.2	1.8	-16.8	1.1	-8.9	1.2	Fa = 3.8
		-9.2	1.5	-10.9	1.0	-6.1	1.1	Fa = 3.3
CHD-10	mt	2.6	0.8	-0.9	0.7	-2.2	0.9	
		2.7	1.0	-1.8	0.9	-3.2	1.0	
	ol	-5.7	1.4	-7.3	1.1	-4.3	1.1	Fa = 13.2



-5

 $\delta^{18}O_{SMOW}$ (‰)

-10

Bulk measurements (Clavton and coworkers)

▲ matrix

whole rock

chondrules

dark inclusion

5

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One olivine in CHD-9 has different O-isotopic composition with the other two olivine grains in the same chondrule. Even though we cannot rule out that it could simply due to analytical uncertainty (they are barely resolvable within 2σ errors), it is possible that there is O-isotopic heterogeneity in olivine grains in CHD-9. Much larger O-isotopic heterogeneities for chondrule olivine have been reported recently (Jones et al., 2002; Yurimoto and Wasson, 2002).

Magnetite and silicates (olivine and pyroxene) are not in O-isotopic equilibrium. Magnetite grains have higher Δ^{17} O values than the associated silicates: weighted averages of Δ^{17} O values are $-3.3 \pm 0.3\%$ for all magnetite data and $-6.1 \pm 0.4\%$ for silicates. In the same chondrules, the average Δ^{17} O values of magnetite are 1.7 to 3.6‰ higher ($2.5 \pm 0.5\%$ on weighted average) than those of olivine, and there is a positive correlation between Δ^{17} O values of magnetite and of silicates in the same chondrules (Fig. 3); the slope of a weighted regression using 2σ error bars on both δ^{17} O and δ^{18} O has a slope of 0.96 \pm 0.03 and MSWD (mean square weighted deviation) of 1.4.

5. FORMATION OF MAGNETITE AND MICROSCALE OXYGEN EXCHANGE IN THE NINGQIANG PARENT BODY

Although the origin of Ningqiang magnetite has not been discussed in detail previously, the origin of magnetite in other unequilibrated chondrites such as Allende, Mokoia, Kaba (oxidized-CV3 chondrites), Semarkona and Ngawi (LL3 chondrites) has been examined by Choi et al. (1997, 1998, 2000). They found O-isotopic disequilibrium between magnetite and associated silicates that suggests secondary origin of magnetite. Petrological characteristics of magnetite in these chondrites indicate in situ formation of magnetite by replacing preexisting



Fig. 3. A correlation diagram showing the average $\Delta^{17}O$ values in magnetite and silicates in the same chondrules. The $\Delta^{17}O$ values of magnetites average 2.5 \pm 0.5% higher than those of silicates and there is a positive correlation between these quantities. The solid line marks the position of $\Delta^{17}O_{magnetite} = \Delta^{17}O_{silicates}$, and the dashed line is a weighted regression line.

phases. Thus, Choi et al. (1997, 1998, 2000) conclude that the magnetite formed by oxidation of metallic phases during parent body aqueous alteration. Since Ningqiang magnetite is also in O-isotopic disequilibrium with associated silicates and has petrological characteristics similar to those of oxidized-CV chondrites, we conclude that Ningqiang magnetite also formed during parent body alteration.

The mean Δ^{17} O value of magnetite in Ningqiang is the lowest measured in unequilibrated chondrites. The second lowest Δ^{17} O values are found in magnetite in Allende (Choi et al., 1997) and the highest in the ordinary chondrites, Semarkona and Ngawi (Choi et al., 1998). We compare the average Δ^{17} O values of magnetite in carbonaceous chondrites and those of the host bulk chondrites in Figure 4; the two quantities show a positive correlation. Positions on Figure 4 could reflect: (1) the original compositions of unaltered silicates and water, (2) the water/rock ratio, and (3) the degree of isotopic exchange between water and silicates.

For carbonaceous chondrites, we assume that the original Δ^{17} O values of bulk dry low-FeO chondrules before the formation of magnetite (i.e., before the aqueous alteration) were more or less similar to each other and were $\leq -5\%$ based on the lowest bulk chondrule values measured in carbonaceous chondrites (Clayton et al., 1977; Mayeda et al., 1988). It seems likely that the original Δ^{17} O value of the asteroidal water was larger than the values observed in CI magnetite and possibly even higher than that in Semarkona magnetite. The final oxygen isotopic compositions of silicates and water would be decided by the water/rock ratios and the degree of isotopic exchanges. It seems reasonable to assume that the magnetite records the Δ^{17} O of the H₂O at the time of magnetite formation. Of course, there could be a sizable compositional range within each magnetite grain that cannot be resolved by the ca. $15-\mu m$ ion-probe spots, but we will limit our discussion to mean $\Delta^{17}O$ values.

An arbitrary initial point for H₂O and average silicates in

 $\delta^{17}O_{SMOW}(\%0)$

-15

-20

-20

Spot analyses (this study)

olivine, pyroxene

magnetite

-15

0



Fig. 4. A correlation diagram showing the average $\Delta^{17}O$ values in magnetite in carbonaceous chondrites and their hosts. The solid line is for $\Delta^{17}O_{magnetite} = \Delta^{17}O_{whole\ rock}$, and the dashed lines are for $\Delta^{17}O_{magnetite} = \Delta^{17}O_{whole\ rock} + 1, +2$, and +3‰. The $\Delta^{17}O$ values of magnetite are higher than those of the host chondrites and there is a positive correlation. An arbitrary initial composition of water and average silicates before aqueous alteration in carbonaceous chondrites is shown as point I. The composition would move toward $\Delta^{17}O_{magnetite} = \Delta^{17}O_{whole\ rock}$ line by O-isotopic exchange during the alteration. The vector F_A is for a infinitely large water/rock ratio, and F_B for an infinitely small ratio. The higher the lowest position among unequilibrated chondrites on this diagram, suggesting that it had the lowest water/rock ratio. Sources of data: Choi et al. (1997, 2000), Clayton and Mayeda (1999), Rowe et al. (1994).

carbonaceous chondrites before aqueous alteration is shown as point I in Figure 4. Isotopic exchange would cause the H₂O composition to move toward the $\Delta^{17}O_{magnetite} = \Delta^{17}O_{whole rock}$ line; extensive isotopic exchange would bring compositions close to this line, as observed in CK4 chondrites, Karoonda and Y6903 (Fig. 4). Thus, the distance between the original composition and the final composition would reflect the degree of isotopic exchange (during aqueous alteration and thermal metamorphism). The angle of the line connecting the original and final compositions (θ in Fig. 4) reflects the water/rock ratio at the time of the isotopic exchange. If the water/rock ratio is very large, only the Δ^{17} O value of silicates would move toward the $\Delta^{17}O_{magnetite} = \Delta^{17}O_{whole rock}$ line (arrow F_A in Fig. 4). If the water/rock ratio is very small, only the Δ^{17} O value of H₂O (and hence magnetite) would move (arrow F_C in Fig. 4). Any direction of isotopic exchange with a water/rock ratio between these two extreme cases would generate a different angle such as the arrow F_B in Figure 4; larger water/rock ratios would result in smaller angles. Among the data in Figure 4 the CI chondrites form the smallest angles implying the highest water/rock ratios. Magnetite in CI chondrites is commonly interpreted to have formed at high water/rock ratios (Rowe et al., 1994).

Since Ningqiang (and the equilibrated CK chondrites) form the largest angle in Figure 4, we suggest that Ningqiang magnetite formed in an environment having a smaller water/rock ratio than was present in the parent asteroids of the other carbonaceous chondrite groups. A smaller water/rock ratio could explain the positive correlation between $\Delta^{17}O$ values of magnetite and silicates in each chondrule shown in Figure 3. It is likely that, if H₂O was abundant, the isotopic composition of H₂O was more or less homogenous at any given time during isotopic exchange with silicates. However, if the water/rock ratio was very low, small amounts of H₂O infiltrated into each chondrule would remain isolated from any nearby H₂O reservoirs. In each chondrule, the $\Delta^{17}O$ values of H₂O were reduced by local isotopic exchange; the lower the initial bulk $\Delta^{17}O$ of the chondrule, the lower the $\Delta^{17}O$ of the H₂O at the time of magnetite formation.

6. SUMMARY AND CONCLUSIONS

Petrological characteristics of Ningqiang magnetite grains are similar to those of oxidized-CV chondrites and magnetite grains are in O-isotopic disequilibrium with associated silicates, implying that the magnetite formed by similar processes, i.e., the oxidation of preexisting metallic (or possibly FeS) phases by water during parent body alteration. Our in situ measurements show that magnetite, olivine and pyroxene have O-isotopic compositions close to CCAM line with relatively large ranges of δ^{18} O. Magnetite and silicates in the same chondrules are not in O-isotopic equilibrium and differ in Δ^{17} O values. However, there is a positive correlation between $\Delta^{17}O$ of magnetite and olivine (and pyroxene) in the same chondrules, with magnetite values 2.5 \pm 0.5‰ on average higher than those of silicates. Both Δ^{17} O values of magnetite and of bulk Ningqiang are the lowest among magnetite-bearing unequilibrated chondrites. Assuming that the initial water and silicates before aqueous alteration in carbonaceous chondrite parent bodies had similar Δ^{17} O values, the lower Δ^{17} O in Ningqiang magnetite implies that the magnetite formed in an environment having a lower water/rock ratio than present in the other magnetite-bearing unequilibrated carbonaceous chondrites. Due to the low water/rock ratio, H₂O permeating each chondrule would have been largely isolated and experienced local (microscale) O-isotopic exchange with chondrule silicates producing the positive correlation between Δ^{17} O values of magnetite and silicates in the same chondrules.

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