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Evolution of hematite and/or magnetite iron phases with thermal heating in ordinary chondrites: A generic characteristic

Ambesh Dixit[1,*], Beena Bhatia[2] and R P Tripathi[2]

[1]*Department of Physics, Indian Institute of Technology Jodhpur, Jodhpur 342 037, India.*
[2]*Department of Physics, JNV University, Jodhpur, Rajasthan 342 011, India.*
**Corresponding author. e-mail: ambesh@iitj.ac.in*

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We used ^{57}Fe Mössbauer spectroscopic measurements on untreated and controlled thermally treated Ararki, Didwana, and Bhawad ordinary chondritic meteorites to understand the evolution of different iron phases with temperature. The samples were subjected to different heating temperatures and room temperature Mössbauer spectra were collected after cooling down these samples. The untreated meteorite samples showed the presence of olivine, pyroxene, troilite, and Fe–Ni alloy (kamacite/taenite) minerals. The decomposition of such iron based minerals started at or above 600°C towards hematite iron phase for all these meteorites. More interestingly, at about 1000°C or above, all initial mineral phases got converted into either hematite and/or magnetite. The present study provides a clue to understand the mechanism of conversion of minerals in ordinary chondrites.

Keywords. Meteorites; ordinary chondrites; thermal treatment; Mössbauer spectroscopy.

1. Introduction

Meteorites are the solid fragments from interplanetary space that survive while passing through Earth's atmosphere, and generally only a small fraction of the initial body manages to reach the Earth without being ablated by atmospheric friction. Out of meteorites reached on Earth, a major fraction of these consists of ordinary chondrites (OCs). Depending on chemistry and mineralogy, ordinary chondrites are further divided into three groups, viz., H group, L group, and LL group. Among these, H group meteorites contain the highest (above 20%) weight percentage of iron with an appreciable amount in metallic Fe alloy kamacite in which iron has FeO state. LL group meteorites have the lowest weight percentage of iron with very meagre presence of metallic iron. Since ordinary chondrites contain appreciable amount of iron, ^{57}Fe Mössbauer spectroscopy is widely used in studying the chemical state of iron in ordinary chondrites (Cadogan *et al.* 2013; Maksimova *et al.* 2014). One of the reasons to use ^{57}Fe Mössbauer spectroscopy for characterizing iron-bearing minerals is that it can probe the local environment of iron nuclei quite sensitively. Further, it is a quick, reliable, and simple method for understanding iron-containing minerals and materials (Tominga and Minai 1984; Kuzmann *et al.* 2003; Galazka-Friedman *et al.* 2014, 2017; Guda *et al.* 2019). It is a non-destructive technique in the sense that the sample in powder form or thin slices is not getting altered during the experiment. Also, in a single run, we get information about all the iron minerals by proper deconvolution of the spectrum. The importance of ^{57}Fe Mössbauer spectroscopy has

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already been established by many workers in characterizing various iron minerals present in ordinary chondrites (OC) and other class of meteorites. Tripathi *et al.* (2000, 2018), Paliwal *et al.* (2000, 2002), Bhandari *et al.* (2008), Verma *et al.* (2002, 2003, 2008), Verma and Tripathi (2004), etc., demonstrated the usefulness of this technique in identifying and classifying the meteorites. On the basis of detailed Mössbauer spectroscopy of several ordinary chondrites, it is well established by Verma *et al.* that in unweathered ordinary chondrites, iron is mainly distributed in kamacite, troilite, olivine, and pyroxene. Meteorites, which are exposed to environment for very long time also exhibit iron in Fe^{3+} state in silicates and oxide phases, c.f. Bland *et al.* (2010).

It is well known that when primary silicate minerals like pyroxenes, olivine and clay minerals are heated in the range 900°–1000°C, they decompose in oxide phases (Tripathi *et al.* 1978; Barcova *et al.* 2003; Guda *et al.* 2019). In the present investigation, we annealed three different ordinary chondrites at 950°C in a box furnace under open ambient conditions and studied these pre-heated samples using Mössbauer spectroscopy. Meteorites used in present investigation are (i) Didwana, which is classified as H5 by Paliwal *et al.* (2002), (ii) Ararki, which is collected from Thar desert of India, classified as L5 chondrite by Bhandari *et al.* (2008), and (iii) Bhawad or Jodhpur meteorite, which is classified as LL(6) by Bhandari *et al.* (2008). Out of these three meteorites, only H-chondrites (Didwana) show appreciable presence of Kamacite along with silicate phases. In other two, characteristic sextet corresponding to kamacite is not observed, indicating these meteorites contain very low kamacite fraction even beyond the detection limit of Mössbauer spectroscopy. All the three meteorites show the presence of troilite (FeS). In table 1, relative area of different phases observed in unheated sample are summarized.

Table 1. *The relative area of different meteoritic mineral phases in various unheated meteorites.*

Meteorite phases	Relative area of different phases in (%)		
	Didwana	Ararki	Bhawad
Kamacite	23	00	00
Troilite	22	16	15
Olivine	31	49	64
Pyroxene	24	36	21
Fe^{2+}-D	00	00	00

Here, one of the aims is to see if these meteorites show the same pattern of decomposition or is it different for H, L, and LL chondrites after heating at different temperatures. Apart from this, we have heated Ararki meteorite at different temperatures (this contains appreciable presence of olivine and pyroxene). The purpose of this is to get information on the thermal transformation mechanism of Fe^{2+} cations in the silicates structure.

2. Experimental details

The meteorites were ground first in a cleaned agate mortar to get the fine powder. About 70 mg cm^{-2} of this fine powder was sandwiched between two paper discs in a 2.5 cm diameter sample holder to prepare the Mössbauer study's absorber. Mössbauer spectrometer is M/S Fast Com Tech Germany make, containing a ^{57}Co in Rh matrix with initial strength of 10 mCi as the gamma-ray source and drive unit, function generator, high tension unit, preamplifier and multichannel analyser with analog to digital converter (ADC) unit to select 14.4 keV gamma-rays. The drive system used in this system is MA250. The source is moved towards and away from the sample, while varying velocity linearly with time. The proportional counter used was Kr filled and having 1024 channel analyzer. The calibration spectrum was recorded with pure α-iron sample before and after the experimental run to ensure electronic stability. The Mössbauer spectrum was collected in 512 channels using MCS mode of multichannel card host in PC slot. The stability of the system was checked regularly collecting standard six-line patterns of α-iron foil and stainless steel foil. After calibration, the Mössbauer spectrum of such powdered samples was recorded at 300 K using a conventional constant acceleration mode in transmission geometry coupled with function generator. Spectrum was fitted using a least-square routine assuming spectrum to be the sum of Lorentzian functions. Further, by using Meerwall's FORTRAN-based computer program, we resolved the various doublets and sextets (von Meerwal 1975). The width and intensity of a quadrupole doublet's two halves were constrained to be equal while fitting the data. Additionally, the areas of 1st and 6th peaks, 2nd and 5th middle peaks, and 3rd and 4th inner pair peaks were constrained to be equal in case of fitting the sextet spectra. The width of all the six peaks was also constrained to be

equal. The isomer shift (IS) is reported with respect to the centroid of a standard α-iron foil.

3. Results and discussions

3.1 *Assignment of phases*

Room temperature Mössbauer spectra collected for fresh or unaltered samples exhibit the presence of iron in (i) olivine, (ii) pyroxene, (iii) troilite, and (iv) Fe–Ni alloy (kamacite/taenite) as a major iron-containing phase. Out of these four phases, olivine and pyroxene are silicate minerals and others are opaque. Mössbauer parameters such as isomer shift, quadrupole splitting, and hyperfine magnetic field are characteristics for a given system and can be easily used to identify the materials/minerals. For example, Mössbauer spectrum of chondrites consists of two slightly overlapping strong doublets, each having a centre shift of around 1.1 mm s^{-1}. The splitting of the outer doublet (corresponding to iron in olivine) is around 3 mm s^{-1} and that of the inner one (corresponding to iron in pyroxene) is around 2.0 mm s^{-1}. In addition to these, there are two sextets of small amplitudes corresponding to troilite (B_{HF} centred around 31T), and kamacite (B_{HF} centred around 33T). The outermost peaks of the two sextets are quite distinct on the negative velocity side, but they often merged into a single broad peak on the positive velocity side. In some ordinary chondrites, minor iron phases are also present. Though in H, L, LL chondrites, iron phases are the same, their relative distribution is significantly different. While H chondrites show appreciable relative absorption corresponding to kamacite, it is very small in LL/L chondrites. The details about the distribution of iron phases in such chondrites are described by Verma *et al.* (2003).

Apart from this, we identified oxide phases on the basis of Mössbauer parameters, provided in tables 2–5 likely to be formed due to the heating of various iron phases present in meteorite. Due to the complex structure of meteorite, inclusion of impurities may also differ/alter, showing some variation in IS and QS values, those observed for ideal composition. But the magnetic hyperfield values can be used to distinguish between various oxide phases. In the present work, also hyperfine field, i.e., B_{HF} was used to characterize the oxidized phases formed during the high temperature thermal treatment of these meteorite samples. B_{HF} for hematite iron oxide (Fe_2O_3) is always above 51T while magnetite/spinal phases show a wider range hyperfine magnetic field (45–48T) depending on the distribution of iron in different sites as well as on and Fe^{2+}/Fe^{3+} ratio. As there is no other iron oxide phase in this range of magnetic hyperfine fields except hematite or magnetite phases, the values of B_{HF} can be used to characterize these meteorites. The fitted Mössbauer spectra are shown in the respective figures with properly assigned doublet and sextets with respective parameters in tables.

3.2 *Temperature-dependent Mössbauer spectra*

Mössbauer spectra for all three unheated and annealed at 950°C samples are given in figures 1–3 with respective Mössbauer parameters in tables 2–4. The respective areas of different kamacite, troilite, olivine and pyroxene meteorite phases are summarized in table 1 for all pristine, i.e., untreated samples. It can be seen from table 1 that unheated Didwana meteorite shows presence of olivine, pyroxene, troilite phases and also the appreciable presence of metallic iron alloy kamacite. But Mössbauer spectra for L and LL samples (i.e., Ararki and Bhawad meteorites) are devoid of kamacite, however, silicate minerals are present in substantial amount in L and LL chondrites along with troilite.

It can be seen that the Mössbauer spectrum of all three meteorite (DM (figure 1b), AM (figure 2b), and BM (figure 3b)) samples annealed at 950°C lost all the information about initial phases. Instead, these spectra exhibit the presence of sextet/sextets and doublet/doublets. If we compare Mössbauer spectrum of Didwana meteorite (DM) Mössbauer pattern (figure 1), this meteorite is very much different from other L/LL (i.e., AM, figure 2 and BM, figure 3) meteorites. Mössbauer spectrum for 950°C preheated Didwana meteorite (figure 1b) shows the presence of characteristic sextet corresponding to hematite along with an intense doublet corresponding to smaller size hematite grains of hematite showing superparamagnetism. A doublet with similar parameters is also observed by Guda *et al.* (2019) while studying thermal heating of natural olivine and attributed this doublet to the superparamagnetic behaviour of iron oxide grains (Guda *et al.* 2019). Though silicate-rich L and LL chondrites show iron oxide (hematite) as dominant phase, they also exhibit appreciable magnetic

Table 2. *The different Mossbauer parameters for (a) unheated and (b) preheated at 950°C Didwana meteorite (DM).*

Doublet/sextet	IS (mm s^{-1})	QS (mm s^{-1})	LW (mm s^{-1})	HMF (kOe)	Area (%)
(*a*) *Unheated*					
Doublet-1 (O)	1.15	2.95	–	0	31
Doublet-2 (P)	1.11	2.11	–	0	24
Sextet-1 (K)	0.00	0.00	–	332	23
Sextet-2 (T)	0.70	–0.17	–	314	22
(*b*) *Preheated*					
Doublet-1 (Fe^{2+}-D)	0.18	0.35	0.84	–	26.18
Sextet-1 (H)	0.50	–0.19	0.48	506.64	74.02

Table 3. *The different Mossbauer parameters for (a) unheated and (b) preheated at 950°C Ararki meteorite (AM).*

Doublet/sextet	IS (mm s^{-1})	QS (mm s^{-1})	LW (mm s^{-1})	HMF (kOe)	Area (%)
(*a*) *Unheated*					
Doublet-1 (O)	1.14	2.98	–	–	48.57
Doublet-2 (P)	1.14	2.14	–	–	35.62
Sextet-1	0.57	–0.16	–	287	11.43
Sextet-2 (K)	0.69	–0.19	–	313	4.30
(*b*) *Preheated*					
Doublet-1 (Fe^{2+}-D)	1.43	2.57	0.58	–	7.90
Doublet-2 (Fe^{2+}-D)	1.12	1.84	0.52	–	6.94
Sextet-1 (H)	0.45	–0.21	0.40	519.15	45.74
Sextet-2 (M)	0.32	0.11	0.37	496.32	23.12
Sextet-3 (M)	0.41	–0.28	0.33	463.40	16.04

Table 4. *The different Mossbauer parameters for (a) unheated and (b) preheated at 950°C BM/Jodhpur meteorite (BM).*

Doublet/sextet	IS (mm s^{-1})	QS (mm s^{-1})	LW (mm s^{-1})	HMF (kOe)	Area (%)
(*a*) *Unheated*					
Doublet-1 (O)	1.14	3.0	–	0	64
Doublet-2 (P)	1.18	2.13	–	0	21
Sextet1 (T)	0.70	0.15	–	314	15
(*b*) *Preheated*					
Doublet-1 (Fe^{2+}-D)	1.44	2.44	0.51	–	6.34
Doublet-2 (Fe^{2+}-D)	1.12	2.38	0.45	–	5.90
Sextet-1 (H)	0.44	−0.17	0.34	515.61	42.44
Sextet-2 (M-MF)	0.36	−0.03	1.09	476.23	45.36

Here IS = isomer shift, QS = quadrupole shift, LW = line width, HMF = hyperfine magnetic field Mössbauer parameters and O = olivine, P = pyroxene, H = hematite, M = magnetite, Fe^{2+}-D = Fe in 2+ state in distorted octahedra, M-MF = magnetite/magnesioferrite, K = kamacite, T = troilite. Note: Bhawad meteorite was initially reported as Jodhpur meteorite (Verma *et al.* 2003) and the room temperature data for this meteorite is adopted from Verma *et al.* (2003).

phases with Mössbauer parameters very close to magnetite/magnesioferrite. Our results indicate that the transformation of iron phases in oxides for H, L, and LL meteorites is different and depends on the silicate/opaque mineral ratio of meteorite when heated in similar conditions. We believe that information obtained in the present investigation will be useful while studying the iron mineralogy of samples collected from small craters formed due to the impact of meteorites.

Table 5. *Mössbauer parameters, extracted from figure 4, for different temperature annealed Ararki meteorite samples corresponding to fitted respective doublet/sextets.*

Sl. no.	Sample name/Mössbauer spectra no.	Doublet/sextet	IS (mm s^{-1})	QS (mm s^{-1})	LW (mm s^{-1})	HMF (kOe)	Area (%)
1	Ararki meteorite preheated at 600°C	Doublet-1 (Olivine)	1.20	2.94	0.43	–	21.06
		Doublet-2 (Pyroxene)	1.22	2.36	0.60	–	20.78
		Doublet-3 (Fe^{2+}-D)	0.29	1.13	0.93	–	22.28
		Sextet-1 (Hematite)	0.74	–0.19	0.21	519.40	17.84
		Sextet-1 (Hematite)	0.56	–0.16	0.83	498.53	18.04
2	Ararki meteorite preheated at 830°C	Doublet-1 (Olivine)	1.12	2.76	0.61	–	16.14
		Doublet-2 (Pyroxene)	1.01	1.76	0.51	–	13.0
		Doublet-3 (Fe^{2+}-D)	0.36	0.62	0.82	–	11.38
		Sextet-1 (Hematite)	0.64	–0.16	0.38	520.76	38.56
		Sextet-2 (Magnetite)	0.90	0.11	0.72	476.01	20.92
3	Ararki meteorite preheated at 1100°C	Doublet-1 (Fe^{2+}-D)	0.18	0.43	0.58	–	6.54
		Sextet-1 (Magnetite)	0.19	–0.04	0.51	471.33	52.70
		Sextet-2 (Magnetite)	0.47	0.01	0.38	473.27	26.42
		Sextet-3 (Magnetite)	0.90	0.14	0.54	470.80	14.36
4	Ararki meteorite preheated at 1500°C	Doublet-1 (Fe^{2+}-D)	0.42	0.47	0.34	–	89.48
		Doublet-2 (Fe^{3+}, glassy flakes)	0.41	1.18	0.27	–	10.52

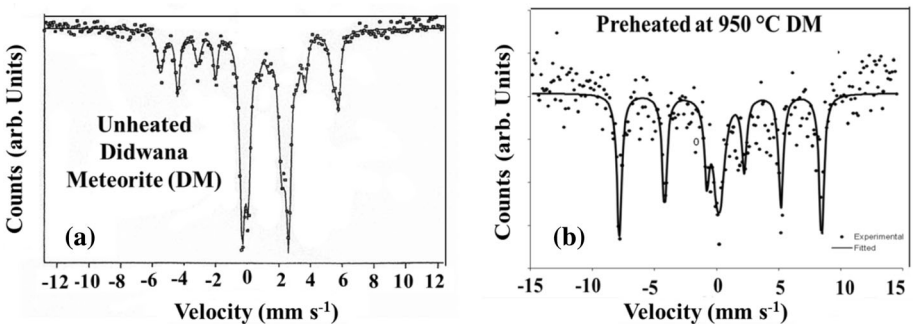

Figure 1. Mossbauer spectra of (**a**) unheated and (**b**) preheated at 950°C Didwana meteorite (DM).

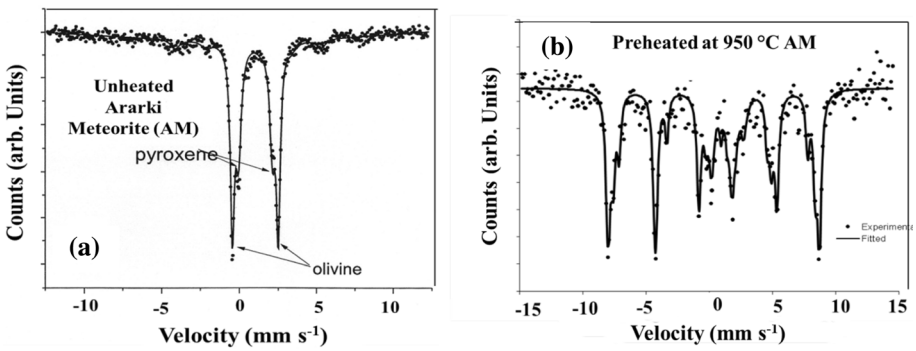

Figure 2. Mossbauer spectra of (**a**) unheated and (**b**) preheated at 950°C Ararki meteorite (AM).

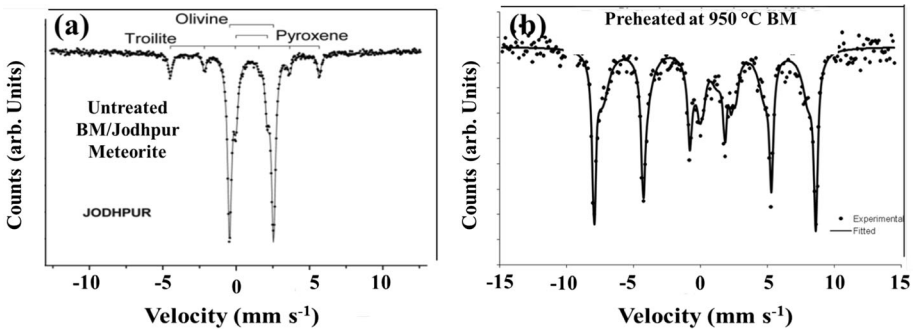

Figure 3. Mossbauer spectra of (**a**) unheated and (**b**) preheated at 950°C BM/Jodhpur meteorite (BM).

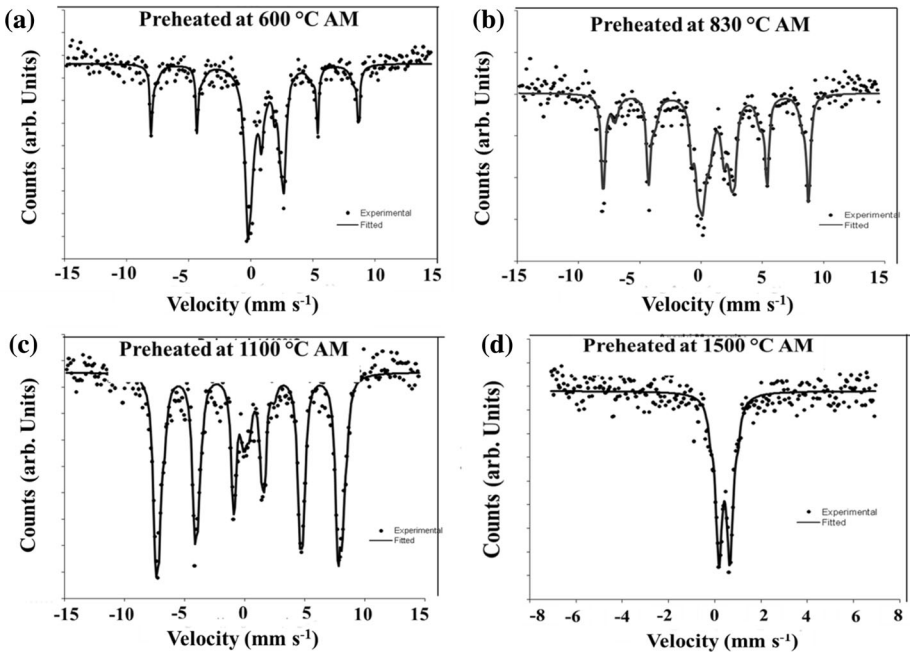

Figure 4. The Mössbauer spectra of Ararki meteorite subjected to different temperatures under ambient conditions, (**a**) 600°C, (**b**) 830°C, (**c**) 1100°C, and (**d**) 1500°C.

3.3 *Mechanism of transformation of Fe^{2+} in silicate minerals*

Mössbauer spectrum collected for unheated Ararki meteorite (figure 2a) shows that relative area of olivine and pyroxene is approximately comparable and iron present in Fe^{2+}. Ararki meteorite (AM) is subjected to 600°, 830°, 950°, 1100°, and around 1500°C for heat treatment under normal ambient conditions to understand the evolution dynamics of different iron oxide phases in this meteorite sample. The Mössbauer spectra for unheated and 950°C heated Ararki meteorite are shown in figure 1(a and b), respectively. In contrast, Mössbauer spectra for heat-treated samples at other temperatures are shown in figure 4. The respective parameters extracted from these Mössbauer spectra are summarized in table 5 and respective doublet and sextet assignments.

We also investigated Mössbauer spectrum for preheated AM samples at 600°C (figure 4a). A sextet corresponding to troilite has disappeared entirely, and the intensity of doublet corresponding to olivine doublet has decreased considerably (figure 4a). Similarly, the intensity of pyroxene doublet has also reduced. It appears that at the expense of sulphide and silicate minerals, two new sextets appeared in the spectrum (figure 4a), with almost comparable intensity and B_{HF}, characteristic of hematite. Here only one sextet is observed, which corresponds to hematite from the extracted Mössbauer parameters. Thus, Mössbauer spectrum for preheated AM at 600°C is confirming

the decomposition of iron phases at or even below 600°C.

Further, Mössbauer spectra for preheated samples at 830°C (figure 4b), indicating the decomposition of silicate phase and one more additional sextet appeared in the spectra for AM and WM samples. The onset of this additional sextet is attributed to the magnetite. In fact, magnesium ferrite was observed by Barcova *et al.* (2003), while studying the decomposition of terrestrial olivine heated in air. Now the characteristic doublets, corresponding to the iron in olivine and pyroxene, are not present, instead, a considerable reduction in QS values for doublets is observed for the Ararki meteorite sample. The corresponding QS values for doublets are 2.76 and 1.76 mm s^{-1} for 830°C preheated AM sample (table 5). This reduction indicates that in this sample, Fe^{2+} iron is present in a relatively more distorted octahedral site compared to that of unheated AM samples (figure 2a).

It is well known that silicate octahedra suffer irreversible distortion after heating (Tripathi *et al.* 1978). As we have already mentioned above, in the case of preheated AM samples at 950°C (figure 1), no significant changes are observed in respective sextets, but now olivine and pyroxene peaks are absent suggesting complete decomposition of initial silicate phases present in untreated Ararki meteorite sample. Ararki sample exhibited three sextets in Mössbauer spectrum when heated at 830°C (figure 4b) and 950°C (figure 2b) (corresponding to hematite, magnetite and/or magnesioferrite). We could do further heat treatment at 1100°C and 1500°C for the Ararki meteorite sample only. Mössbauer spectrum for Ararki sample preheated at 1100°C (figure 4c), showed complete disappearance of hematite and showing only sextets corresponding to magnetite and spinal phases. When Ararki is heated around 1500°C, a blue-coloured glassy slag was formed and thus, showed Fe^{3+} iron in two sites (figure 4d). It suggests that the meteorite material is getting converted into glassy flakes at very high temperatures.

Thus, we observed that the initial iron phases in untreated OC meteorite samples decompose in oxide phases after heating around 1000°C and completely losing information about initial iron phases in untreated samples. Here it is worth pointing that all the samples after heating brought near the magnet showed attraction, indicating that the magnetic oxides are present in these heat-treated meteorite samples. These studies substantiate the presence of magnetic components in preheated OC samples. Barcova *et al.* (2003) showed that olivine doublet is persisting in the Mössbauer spectrum even after 1200°C heating. However, the decomposition mechanism of extra-terrestrial olivine present in the meteorite is markedly different from that of terrestrial olivine. In OC meteorites, olivine decomposes at lower temperatures, i.e., about 600°C or less (figure 4a).

4. Conclusion

The present study is the first investigation demonstrating the decomposition behaviour of OC meteorites. The findings suggest that decomposition starts even at a relatively lower temperature ∼600°C and ends up with only magnetic oxide phases for thermally treated OC meteorites at higher temperatures about 1000°C or more, losing the information about initial iron phases completely. Thus, the evolution of various iron phases is explained against the thermal treatment. Our results suggest that the mechanism of transformation of iron-containing minerals is distinctly different for H chondrite (containing kamacite) compared to L/LL chondrites (devoid of Kamacite). Though all show presence of iron oxide, L and LL chondrites favour the formation of magnetite. It may be due to a difference in initial composition and different Fe/Mg ratios.

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Author statement

AD: Data management and processing, writing and reviewing. BB: Data collection and processing. RPT: Data analysis, writing and reviewing.

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