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Glassy magnetic cronstedtite signatures in Mukundpura CM2 chondrite based on magnetic and Mössbauer studies

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Abstract–We have studied the Mukundpura CM2 meteorite for magnetic properties as a function of temperature and magnetic field, as well as its Mössbauer spectrum, at room and low temperatures (up to 5 K). We find that the high temperature paramagnetic phase is followed by two magnetic transitions: a weak transition near 125 K and a strong transition at 8 K. The weak (125 K) magnetic phase can be attributed to complex Fe^{2+}–Fe^{3+} constituents present in the meteorite. The absence of the characteristic sextet corresponding to magnetite in Mossbauer spectrum indicates that this magnetic phase is not magnetite, which, if present, must be in insignificant amount. The 8 K magnetic ordering is superimposed with weak ferromagnetic ordering, showing spin-glass transition. The Mössbauer spectrum taken at 5 K substantiates the observed spin-glassy nature, as very large hyperfine field ~32 T is recorded, causing localized subordering leading to spin-glass behavior. The Mössbauer spectra also confirm that iron is mainly present in serpentine-group minerals, both in ferrous and ferric states. The complete serpentinization of basic silicates indicates aggressive hydrous alteration. These results show that the observed spin-glass signature is a characteristic feature of the cronstedtite phase in CM meteorites. This feature is unique to carbonaceous CM chondrites and could be used for nondestructive, quick, and independent classification of this rare class of meteorites. Furthermore, the absence of olivine and the presence of cronstedtite in Mossbauer spectra show that the degree of aqueous alteration observed is the most severe in Mukundpura CM2 meteorite, as compared to many other CM2 meteorites. The degree of aqueous alteration in CM2 carbonaceous chondrites increases in the sequence: Paris, Murchison, Murray, Mighei, Nogoya, Cold Bokkeveld, and Mukundpura.

INTRODUCTION

Although observed falls of meteorites, especially carbonaceous chondrites, are rare, such a stone fell on June 6, 2017 at Bhankrota village, near Mukundpura (26°52′52.5″N and 75°39′53.7″E), Jaipur, Rajasthan, in India at about 5:15 AM (Indian Standard Time). The rock broke on impact, making a pit in dry alluvial farmland and the samples were quickly collected by Geological Survey of India (Geological Survey of India 2017). Based on mineral, elemental, thermal, and room temperature Mössbauer spectroscopic measurements, the meteorite was classified as CM type of carbonaceous chondrite by Tripathi et al. (2018). Furthermore, Rudraswami et al. (2018) and Ray and Shukla (2018), based on chemical and isotopic studies, showed that Mukundpura is dominated by phyllosilicates, with rare presence of olivine, calcite, and magnetite and classified it as a primitive CM2.0 chondrite. While Ray and Shukla (2018) proposed that Mukundpura is similar to both Murchison and Paris (CM2) meteorite (The Meteoritical Bulletin 2019), Rudraswami et al. (2018) found it to have undergone more severe aqueous alteration similar to cold Bokkeveld CM2.

To understand the ambient environment during evolutionary stages of the Mukundpura meteorite, we have now extended the study of magnetic minerals and their structural properties to Mössbauer measurements at low temperatures and also carried out temperature- and field-dependent magnetic studies on the same sample.

EXPERIMENTAL METHODS

A sample of Mukundpura meteorite was collected within a few hours of its fall from the impact site as described by Tripathi et al. (2018). The surface of the sample was cleaned and used for room temperature X-ray diffraction (XRD) measurements for identifying the different crystallographic phases present in the meteorite. The microstructural studies were carried out using scanning electron microscopy (SEM) and mineral identification was carried out using Oxford energy dispersive X-ray (EDX) system, equipped as an accessory with SEM, as reported earlier (Tripathi et al. 2018). The temperature- and field-dependent magnetic measurements were then carried out using the Quantum Design magnetic property measurement system (MPMS) (Quantum Design, USA). The room temperature Mössbauer studies (Tripathi et al. 2018) have now been extended to low temperatures, using a constant acceleration spectrometer with ^{57}Co source in a Rh matrix. The spectra are calibrated using alpha-iron as a standard before and after the sample measurement to ensure electronic stability during the measurements.

RESULTS AND DISCUSSION

Structural and Microstructural Studies

The structural and microstructural properties of Mukundpura are investigated using room temperature XRD and scanning electron microscopic measurements. The results are in agreement with earlier reports (Tripathi et al. 2018), showing the presence of higher volume fraction of Fe-cronstedtite phase, besides Mg serpentine, olivine, and calcite. The magnetite peak was also observed in this meteorite by Rudraswami et al. (2018), but the large variation in the intensity of this peak in different samples indicates that this component is not uniformly distributed within the meteorite and is a minor (≤~2%) phase.

Magnetic Studies

Based on the presence of Fe-cronstedtite, we anticipated the magnetic response of Mukundpura meteorite and carried out magnetic measurements under variable temperature and field conditions to understand the magnetic nature of the various components and their contribution to the observed magnetic properties. The temperature-dependent magnetic measurements were performed at $H = 100$ Oe and the results in 2–300 K temperature range are summarized in Fig. 1a. We observed one strong magnetic signature near ~8 K and another weak magnetic signature near ~125 K in M(T) data (Fig. 1a). The regions near these transitions are plotted as insets in more detail in Fig. 1a for easier identification. The origin of higher temperature (125 K) transition is usually attributed to magnetite (Fe_3O_4) or other mixed valence Fe complexes (Coey et al. 1981; Vaishnava et al. 2007). This transition is usually characterized as the Verwey transition in magnetite, although this is observed rarely or only in very high purity samples and is completely suppressed in nanostructured and amorphous materials (Rubin 1997; Walz 2002). The change in the magnetic moment at this transition is <0.001 emu g^{-1}, suggesting that the volume fraction of magnetite or other mixed-valence Fe complex is very small or negligible in the Mukundpura meteorite. There are some reports of spin-glass transitions in the gamma Fe_2O_3 system near this temperature (Aragon et al. 1985). In addition, we have observed another antiferromagnetic transition at ~8 K, as shown in the inset. The different magnetic transitions in Mukundpura meteorite suggest the presence of different Fe valence state complexes, suggesting alteration presumably on the parent body (Walz 2002; Parker et al. 2008; Barrat et al. 2012; Tripathi et al. 2018). We have calculated magnetic susceptibility (χ) and its inverse (χ^{-1}) is plotted against temperature in Fig. 1b for the lower temperature region, with inset showing higher temperature region. The inverse of magnetic susceptibility is fitted with a Curie–Weiss law $\chi^{-1} = C^{-1}\ (T{-}T_\theta)$, where C is the Curie–Weiss constant and T_θ is the Curie–Weiss temperature. The linear fit (red dashed line) suggests the paramagnetic behavior above 8 K magnetic transition with positive Curie–Weiss temperature T_θ ~4 K as marked in Fig. 1b, where χ^{-1} drops to zero. This small positive T_θ suggests weak ferromagnetic ordering below 8 K. This is consistent with the reported literature (Elmaleh et al. 2012). However, there is no direct evidence of long-range magnetic ordering, exhibiting ferromagnetic or antiferromagnetic behavior, as can be seen in the temperature-dependent magnetic measurements (Fig. 1a). In addition, zero field cooled and field cooled (FC) magnetic measurements show wide splitting below 8 K magnetic transition (Fig. 1c). This splitting is regarded as blocking temperature, similar to observations in magnetic nanostructures or spin-glass systems (Coey et al. 1981; Elmaleh et al. 2012). The magnetic moment in FC condition is increasing with decreasing temperature without showing any saturation down to 2 K. This is consistent with the observed positive $T_{\theta,}$ suggesting dominance of weak ferromagnetism below 8 K. We attribute the observed enhancement in magnetic moment to the smaller magnetic grains, that is, clusters of Fe-cronstedtite or their nanoclusters. This localized short-range magnetic ordering

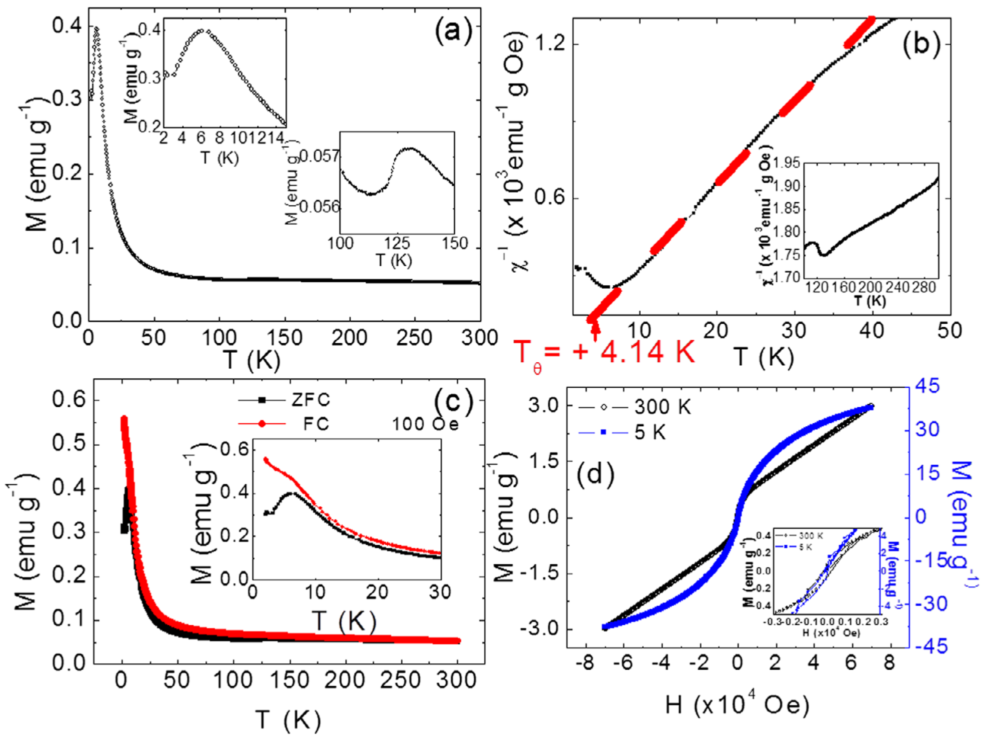

Fig. 1. Temperature dependent (a) zero field cooled (ZFC) plot with insets showing zoomed region near 8 and 125 K; (b) inverse susceptibility versus temperature plot with Curie–Weiss fit with inset showing higher temperature region; (c) ZFC and FC magnetic data with inset showing lower temperature region; and (d) field (H)-dependent magnetic moment (M), that is, magnetic hysteresis measurement at 300 and 5 K. The inset shows magnified M(H) near zero field regions.

due to Fe-cronstedtite spin-glass behavior in Mukundpura is consistent with the observation of magnetic spin-glassy nature in Fe-cronstedtite-based meteorites (Elmaleh et al. 2012). We have also carried out field-dependent magnetic hysteresis M(H) measurements at 300 and 5 K, which are summarized in Fig. 1d. M(H) measurement at 300 K shows magnetic hysteresis at lower fields. The measured magnetic moment becomes linear above 5000 Oe, until 8×10^4 Oe (Fig. 1d). The linear variation of the magnetic moment with field confirms the dominance of a paramagnetic phase at 300 K in Mukundpura meteorite. The low-field hysteresis coercivity is ~150 Oe and the magnetic moment is ~0.8 ± 0.05 emu g^{-1} at 0.5 T. This small magnetic moment may be attributed to the presence of Fe-cronstedtite in the meteorite. In contrast, the paramagnetic contribution may dominate with increasing fields due to the presence of Fe-cronstedtite, Mg-serpentine, and other paramagnetic phases (Elmaleh et al. 2012). The low temperature M(H) measurement was collected at 5 K, which is lower than the spin-glass blocking temperature of ~8 K. We have observed the relatively large magnetic moment, saturating at higher fields. The coercivity value is relatively low, ~150 Oe, which is consistent with the dominating weak ferromagnetic behavior observed below the blocking temperature and the small magnetic clustering becomes magnetically ordered with increasing magnetic field. However, the saturation magnetization value ~35 emu g^{-1} at 8 T magnetic field is much smaller than the magnetite superparamagnetic saturation magnetization, and thus, rules out the contribution of magnetite (Coey et al. 1989; Vaishnava et al. 2007). Furthermore, the blocking temperature of magnetite is much higher ~250 K (Coey et al. 1989), and thus, a large magnetic coercivity (~kOe or higher) is expected at temperatures below the magnetite blocking temperature. In Mukundpura, the coercivity value is only ~150 Oe and no widening in magnetic hysteresis is observed below blocking temperature. All these above-mentioned observations substantiate a spin-glass transition due to Fe-cronstedtite present in Mukundara meteorite and rule out the presence of magnetite.

Mössbauer Studies

Iron-bearing minerals form an important and differentiating component of all classes of meteorites (Dodd 1981). Mössbauer spectroscopy is a powerful technique for characterizing Fe containing minerals and is extensively used in investigating meteorites (Verma et al. 2010; Bhatia et al. 2015). In previous studies by

Verma et al. (2010) and Bhatia et al. (2015), different classes of meteorites exhibited distinguishing and characteristic Mössbauer parameters and shapes, and thus, Mössbauer analysis can be used as a "fingerprint" for quick identification and classification of meteorites (Kuzmann et al. 2003). In CM chondrites, due to severe aqueous alteration, Fe is mainly distributed in "poorly characterized phases (PCP)." The PCP consists of an intimate mixture of minerals comprising phyllosilicates dominated by opaque, mixed valence Fe^{2+}–Fe^{3+} serpentine minerals, cronstedtite (Fe^{2+}, Fe^{3+}, $Mg_3[SiFe^{3+}]_2O_5[OH]_4$), hydrous Fe-Mg-rich serpentine, and hydrous iron sulfide minerals related to tochilinite ($Fe_{1.3}Ni_{0.1}SO_{1.4}$) (Elmaleh et al. 2012). Considering the presence of these minerals, Mössbauer measurements were carried out on Mukundpura at different temperatures to understand the localized magnetic structures. The room temperature Mössbauer measurements were reported in previous studies by Tripathi et al. (2018), where two quadrupole doublets of nearly equal intensity were reported. The inner doublet is attributed to Fe^{3+} and the outer doublet to Fe^{2+} states, similar to serpentine minerals, with isomer shift (IS) and quadrupole splitting (QS) quite different from that of olivine, suggesting complete serpentinization of iron-bearing olivine phases in Mukundpura meteorite, reflecting the ambient severe hydrous conditions prevailing on the parent body. The absence of sextet indicates that the magnetite or troilite phases may occur in low abundance (≤2%), that is, below the detection limit of Mössbauer spectroscopy. In comparison, these magnetic phases are prominent in CK and CI chondrites. These results substantiate the conversion of olivine phases into serpentine-groups. Furthermore, the observed room temperature spectrum of Mukundpura meteorite is similar to that of other CM-type meteorites like Cold Bokkeveld (CM), suggesting similar history of mineral evolution, as suggested by Tripathi et al. (2018).

We have now carried out new Mössbauer studies at 100 and 5 K and these spectra are shown in Fig. 2. The 100 K Mössbauer spectrum is identical to that at 300 K, reported in the earlier studies (Tripathi et al. 2018). The spectra were fitted with NORMOS least square routine using Lorentzian function and the fitted Mössbauer spectra were used to estimate IS and QS for both 100 and 5 K.

The IS and QS values are listed in Table 1. The two doublets are the best fit for 100 K Mössbauer spectra, showing nearly the same intensity as observed at 300 K. The inner and outer doublets correspond to Fe^{3+} and Fe^{2+} iron states; the absence of any sextet indicates that the abundance of magnetite or olivine systems is below the detection limit. However, there is a very small signature near 125 K in the magnetic moment variations versus temperature, as shown in Fig. 1a (inset), suggesting the presence of trace amounts of magnetite in the sample. The Mössbauer technique is sensitive for probing the local magnetic structure and thus, the absence of sextet in Mössbauer spectra (Fig. 2a) puts a stringent limit, below the detection limits of Mössbauer measurements on the abundance of magnetite in Mukundpara. An interesting observation is the onset of a broad sextet in 5 K Mössbauer spectra (Fig. 2b) in conjunction with two paramagnetic components, similar to that in the Mössbauer spectra at 100 K (Fig. 2a) and 300 K (Tripathi et al. 2018). The distribution of hyperfine fields is used to fit the broad sextet and the probability distribution of hyperfine fields is plotted as an inset in Fig. 2b.

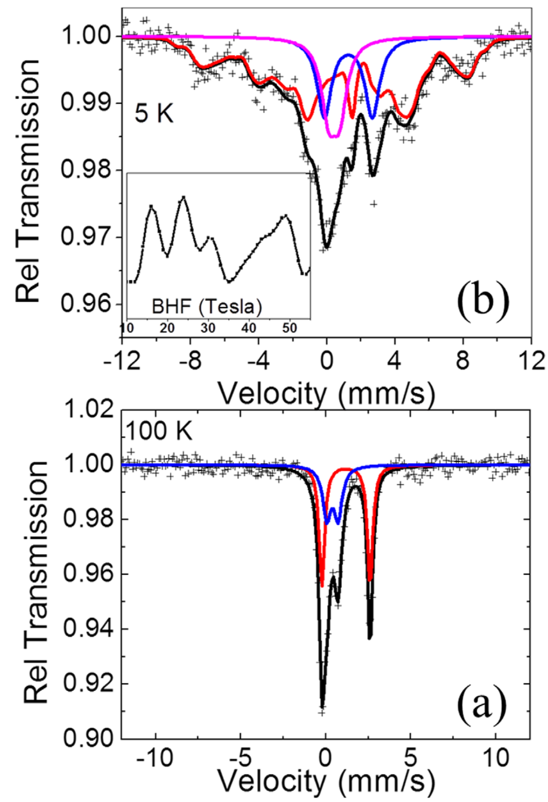

Fig. 2. Mössbauer spectra of Mukundpura meteorite at (a) 100 K with two doublets (blue line), for Fe^{3+} and red line, for Fe^{2+} states, and total fitted spectra (black solid line) and (b) 5 K temperatures with two paramagnetic components (magenta line and doublet blue line correspond to Fe^{3+} states), one broad sextet (red line) and total fitted spectra (black solid line) in conjunction with inset showing the probability distribution of hyperfine fields for broad sextet.

The phases corresponding to these different doublets and sextet are identified in Table 1 as (a) Fe^{2+} phyllosilicates (serpentine), (b) Fe^{3+} phyllosilicates (serpentine), (c) Fe^{2+} phyllosilicates (serpentine), (d) Fe^{3+} phyllosilicates (serpentine), (e) Fe^{2+}–Fe^{3+} cronstedtite.

Table 1. IS and QS for Mukundpura meteorite samples, estimated from 100 and 5 K Mössbauer spectra.

Measurement temperature (K)	FWHM (mm s^{-1})	IS (mm s^{-1})	QS (mm s^{-1})	Nature of the best fit	BHF (T)
100	0.38 ± 0.01	1.18 ± 0.01	2.84 ± 0.0	Doublet[a]	–
	0.58 ± 0.01	0.38 ± 0.01	0.67 ± 0.01	Doublet[b]	–
5	1.02 ± 0.01	1.25	2.82 ± 0.01	Doublet[c]	–
	1.02 ± 0.01	0.41 ± 0.04	0.64 ± 0.01	Doublet[d]	–
	0.55	0.79 ± 0.05 (Avg)	0.33 ± 0.01 (Avg)	Sextet[e]	32.8 (Avg)

Avg = average; FWHM, full width at half maximum.

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The cronstedtite shows magnetic ordering below 10 K (Walz 2002) where other serpentine-group minerals still exhibit paramagnetic contributions. The onset of sextet at 5 K is attributed to cronstedtite phase in Mukundpura meteorite, which also showed magnetic ordering at 8 K (Fig 1a). These results are consistent with the Mossbauer spectra of CM below 10 K (Burns and Fisher 1990), who observed a complex sextet, corresponding to the cronstedtite in conjunction with two intense doublets for Fe^{2+} and Fe^{3+} in Fe-Mg serpentine and a very weak intensity doublet corresponding to iron in tochilinite (Bhatia et al. 2015). However, no explanation was provided for the observed complex sextet due to cronstedtite.

We investigated the probability distribution of hyperfine fields of this broad sextet for 5 K Mössbauer spectra. This probability distribution shows the presence of different components with various fields (considering peak values), and thus, the average value of hyperfine fields is found to be about 32.8 T (Table 1). The observed high hyperfine field of about 32.8 T supports the spin-glass behavior of cronstedtite, consistent with the spin-glass magnetic signature observed in magnetic studies.

Mössbauer spectroscopy reflects only the chemical states of iron in the sample. Olivine $(Fe,Mg)SiO_4$ in room temperature Mössbauer spectrum shows a characteristic doublet, in which QS is centered around 3.0 mm s^{-1} and IS is centered around 1.20 mm s^{-1}. Furthermore, as aqueous alteration increases, the area corresponding to the olivine decreases, and may be completely absent in highly altered CM2 chondrites. As far as Mukundpura meteorite is concerned, the characteristic peaks of olivine are not observed in Mössbauer spectrum at room temperature (Tripathi et al. 2018) or even at low temperature (Fig. 2). However, the presence of olivine reported in the literature may correspond to $MgSiO_4$ and it should be in a very small amount, present only at the microscopic level, if at all (Bhatia et al. 2015). Similarly, the room temperature Mössbauer spectrum is expected to exhibit a characteristic sextet having HFMF (hyperfine magnetic field) about 480 KOe for magnetite. We have not observed any such sextet in room temperature Mössbauer spectra for Mukundpura meteorite (Tripathi et al. 2018) indicating that magnetite is below the Mössbauer detection limit. We were also unable to observe the characteristic doublet corresponding to sulfur-containing minerals, that is, tochilinite, further indicating that sulfur was dissolved before combining with any metallic system in the meteorite.

Thus, on the basis of the distribution of organic matter (polyaromatic hydrocarbons) and their alkylation, the degree of aqueous alteration in various CM2 chondrites is shown to increase in the order: Paris < Murchison < Murray < Mighei < Nogoya < Cold Bokkeveld (Browning et al. 1996; Elsila et al. 2005; Martins et al. 2015). Burns and Fisher (1990) reported the absence of olivine in CM based on their room and low-temperature Mössbauer studies, as we have observed for Mukundpura sample, implying similar aqueous alteration in Mukundpura and CM. However, the absence of sulfur-containing minerals in Mukundpura suggests that the severity of aqueous alteration may be a little higher than that in CM. Thus, Mukundpura meteorite shows the highest degree of aqueous alteration among all the CM2 chondrites studied so far. The ratio of the area under the peaks for (olivine + tochilinite)/(cronstedite + iron containing serpentine) is an important parameter to understand the degree of aqueous alteration in CM2 chondrites. In Mukundpura, this ratio is close to zero, suggesting the maximum aqueous alteration of all such chondrites studied. Cronstedtite is a major phase only in CM chondrites and does not occur in any other group of meteorites. Therefore, Mössbauer and magnetic studies can provide an easy, nondestructive, and quick way of identifying and classifying this group of meteorites, and also estimate the extent of aqueous alteration in case of CM2 chondrites.

CONCLUSION

Magnetic and Mössbauer studies on Mukundpura meteorite show that the room temperature phase is due to the paramagnetic contribution of the meteorite constituents. Magnetic transitions observed at low temperatures are due to different valence iron orderings. An interesting feature is a low-temperature antiferromagnetic transition at 8 K. The Curie–Weiss fit to the low-temperature data shows a positive Curie–Weiss temperature of ~4.14 K, suggesting the superposition of weak ferromagnetic ordering on the antiferromagnetic phase. This leads to magnetic frustration, causing spin-glass transition. This is confirmed by 5 K Mössbauer measurement, showing a very large hyperfine field. This is a characteristic feature of cronstedtite phase, which is only observed in CM chrondrites. Thus, the presence of cronstedtite may provide a way of identifying and classifying CM chondrites and their degree of aqueous alteration. Using this criterion, we find that Mukundpura was subjected to severe hydrous activity resulting in the most altered CM2 chondrite, among seven CM2 chondrites studied so far.

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