Implication of Mössbauer spectra on the mixing model of eucrites and diogenites (resulting in howardites)

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In the present communication, we show that Mössbauer spectra of cumulate eucrites are characteristically different from those of non-cumulate eucrites. In cumulate eucrites, iron occupies only one site, i.e. the M2 site in pyroxene crystal lattice, while it is distributed in both M1 and M2 sites in non-cumulate eucrites. We discuss the importance of asymmetric doublet observed in the Mössbauer spectrum of cumulate body, especially in Vissannapeta cumulate eucrite (fallen in Andhra Pradesh, India), where an appreciable asymmetry was observed. The Mössbauer parameters of diogenite almost exactly replicate those of cumulate eucrites, suggesting that they originate from the same magma ocean. Howardites which are mixed eucrites and diogenites, show iron mineralogy which is different from what one would expect from a physical mixture model. The possible reasons for this difference are also discussed.

Keywords: Cumulate and non-cumulate eucrites, HED bodies, Mössbauer spectroscopy, meteorites.

VESTA-4 is the most investigated celestial body beyond the orbit of Mars. The recently completed DAWN mission of NASA acquired a wealth of information through its direct surface imaging from close proximity. However, the excitement about this asteroid started building up well over four decades ago when it was discovered that the reflectance spectra of Howardite–Eucrite–Diogenite (HED) meteorites resemble quite closely with those of Vesta-4 (refs 1–3). With every new sample of HED analysed using different techniques such as isotope shifts, petrography, reflectance spectroscopy, etc., new knowledge about Vesta-4 unfolded. With more than 200 HED meteorites available, we have a good opportunity of understanding this celestial body much better. Drake[4] has given a description of the series of studies establishing the connection between HED meteorites and Vesta-4.

Vesta-4 shows a big crater of diameter about 400 km, only somewhat smaller than the average diameter of about 506 km of the asteroid itself. The crater is almost 30 km deep. The material was thrown up supposedly during a big impact, forming a number of smaller bodies known as vestoids. More than 20 such vestoids have been identified, many of them between the orbit of Mars and 3 : 1 Jupiter orbit–orbit resonance[5]. The HED meteorites seem to come directly from the vestoids as this is consistent with meteoritic trajectories towards the earth[6]. These meteorites can be distinguished from other achondrites and igneous rocks from the restricted range of oxygen isotopic composition with Δ^{17}O values centred around –0.242 ± 0.016% per mil, showing the igneous isotopic homogenization of Vesta-4 (ref. 7).

According to the widely accepted model and experimental data, Vesta-4 had melted and differentiated due to the heat generated by radioactive decay, such as from ^{26}Al isotope, and then differentiated in core, mantle and crust[8]. Due to volcanic activities, a large amount of lava spread over the surface and solidified at the top surface, which corresponds to what we call non-cumulate basaltic eucrite. Below the surface, crystallization occurred whose rate varied with depth, the deeper layers crystallizing more slowly. The crystallization sequence is however still being debated as all experimental observations on mineral distribution and related geochemical trends in HED meteorites are difficult to explain based on any one particular model. In what is called partial melt model, eucrites are supposed to come from partial melt of an olivine–pigeonite–plagioclasespinel–metal source[9]. However, the siderophile element abundances of the eucrites do not favour this and better correspond to fractional crystallization where eucrites are seen to come from the residual melt after the crystallization of diogenites[10]. More recent findings have led to magma ocean models[11,12]. Whether eucrites and diogenites are cogenetic or not is an interesting issue to be settled. The processes that produce large and small-scale igneous units seem to be different[13].

The difference between eucrites and diogenites comes essentially from the geothermal history of the parent body. Indeed due to various geological activities, the mineralogy is mixed and complex. Cumulate eucrites are a sort of link between the ordinary non-cumulate eucrites and the diogenites. Howardites generally show a character representing mixed eucrite and diogenite. For example, the bulk composition of Lohawat howardite can be reproduced by projecting it as a mixture of 57% Juvinas eucrite and 43% Johnstown diogenite, or 59% Haraya eucrite and 41% Tatahonie diogenite[14]. Apparently, the big impact at the south pole mixed all the layers of eucrite/diogentites and this mixed mass gives the howardite meteorites (this is only a speculative hypothesis).

The mineral composition of the HED meteorites possibly represents signatures of the thermal and pressure history of the parent body, Vesta-4 (refs 15, 16). Diogenites have more Mg content than eucrites. All these meteorites are rich in pyroxene with various kinds of distribution of Ca/Mg/Fe cations[17]. In pyroxenes of all these meteorites, there are two inequivalent sites, generally called M1 and M2 sites, which are preferentially

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occupied by the cations, M1 being more symmetric in its octahedral bondings than M2, which is a distorted, six-coordinated site[18–20].

Petrographic analysis of thin sections of the meteorites reveals several characteristics of mineral formation like shape, size, brecciation, etc. from which information on the crystallization environment can be inferred. However, these are not sensitive to relative occupation of cations in the available inequivalent sites in pyroxene crystals. This site occupancy is sensitive to environmental factors like temperature, pressure and cooling rate. Mössbauer spectroscopy can be used to get the site occupancy and hence can be developed as yet another tool to probe the history of mineral crystallization of the parent body. The pyroxene in meteorites that crystallize at high temperature and cool quickly, iron is distributed in both M2 and M1 sites. This indicates that there is highest degree of disorder in pyroxene. In this case Fe^{2+} is distributed in both M2 and M1 sites. Therefore, Mössbauer spectrum of pyroxene will exhibit the presence of two quadrupole doublets corresponding to iron in both M2 and M1 sites. On the other hand pyroxene that crystallizes at low temperature and cools slowly (e.g. pyroxene in cumulate bodies) is well-ordered and Fe^{2+} is preferentially distributed only in the relatively distorted M2 site. Under such conditions, the Mössbauer spectrum will exhibit only one quadrupole doublet corresponding to Fe^{2+} iron in the M1 site. The relative distribution of Fe^{2+} in the M1 and M2 sites is highly correlated with the cooling history of pyroxene in HED meteorites. Hence it is possible to differentiate between rapidly cooled and slowly cooled HED meteorites[21]. For further details regarding the cooling rates of HED meteorites, readers may refer to Burns *et al.*[18] and Klima *et al.*[21] and references therein.

While Mössbauer spectroscopy has been applied to derive many systematic trends in meteorite classification, e.g. in ordinary chondrites[22–25], to the best of our knowledge, till date no systematics has been developed based on Mössbauer parameters of HED meteorites. However, Klima *et al.*[21] have developed some systematics using near infrared spectroscopic study of HED meteorites.

In this communication, we report Mössbauer study of five eucrites and one diogenite together with the available Mössbauer spectroscopic data on HED meteorites in the literature. We propose systematics for the occupancy of Fe^{2+} in M1/M2 sites of pyroxenes.

Three cumulate eucrites (Vissannapeta, Talampaya and Serra de Magé), two non-cumulate basaltic meteorites (Sioux-Country and Millbillillie) and one diogenite (Ibbenbüren) sample were studied. A brief description of these meteorites, together with the references for their classification is given in Table 1. The eucrites are termed as cumulate or non-cumulate (basaltic) on the basis of petrographic studies reported. It is well known that all the diogenites have cumulate lithology.

To prepare the sample for Mössbauer spectroscopic study, about 500 mg of the meteorite fragment was ground to fine powder. About 70 mg of this powder was sandwiched between two transparent tapes to make a Mössbauer absorber of diameter about 12 mm. It was ensured that the thickness of the absorbers always remains within the thin absorber limit. Mössbauer spectra were recorded using conventional constant acceleration Mössbauer spectrometer with ^{57}Co in Rh matrix as the gamma-ray source. Spectra were computer-fitted using a least square routine and assuming each spectrum to be a sum of Lorentzian functions. During curve-fitting, the width and intensity of the two halves of a quadrupole doublet were constrained to be equal. The quality of fit was judged from the value of chi square, which is close to 1.0 per degree of freedom. The isomer shift (IS) is reported with respect to centroid of the spectrum of an alphairon foil. The reported values of IS, quadruple splitting (QS) and line width (LW) have an accuracy of about 0.01 mm/s. All spectra were recorded at room temperature.

Mössbauer spectra of all the samples studied are shown in Figure 1 and the best-fit parameters are given in Table 2. Mössbauer parameters of previously studied HED meteorites together with appropriate references are also given in Table 2. In Mössbauer spectrum of pyroxene, each of the two sites, M1 and M2, shows up as a doublet spectrum, the quadrupole splitting being different for the sites. The difference in the splitting depends on temperature. At room temperature or below, the M2 site presents a lower quadrupole splitting corresponding to more distortion from the octahedral symmetry. The site assignments in Table 2 are done according to the quadrupole splitting.

Mössbauer spectra of all the cumulate eucrites show two absorption peaks with slight asymmetry in intensity (Figure 1). The low-velocity peak in all these spectra is more intense than the high-velocity peak. One of the

Table 1. Description of the meteorites studied

Sample	Fall date	Description	Reference	Place
Vissannapeta	1997	Cumulate eucrite	28	Andhra Pradesh, India
Talampaya	1995	Cumulate eucrite	29	Argentina
Serra de Magé	1923	Cumulate eucrite	30	Brazil
Sioux-Country	1933	Non-cumulate eucrite	31	Nebraska, the United States
Millbillillie	1960	Non-cumulate eucrite	32	Western Australia
Ibbenbüren	1870	Cumulate diogenite	4	Nordrhein-Westfalen, Germany

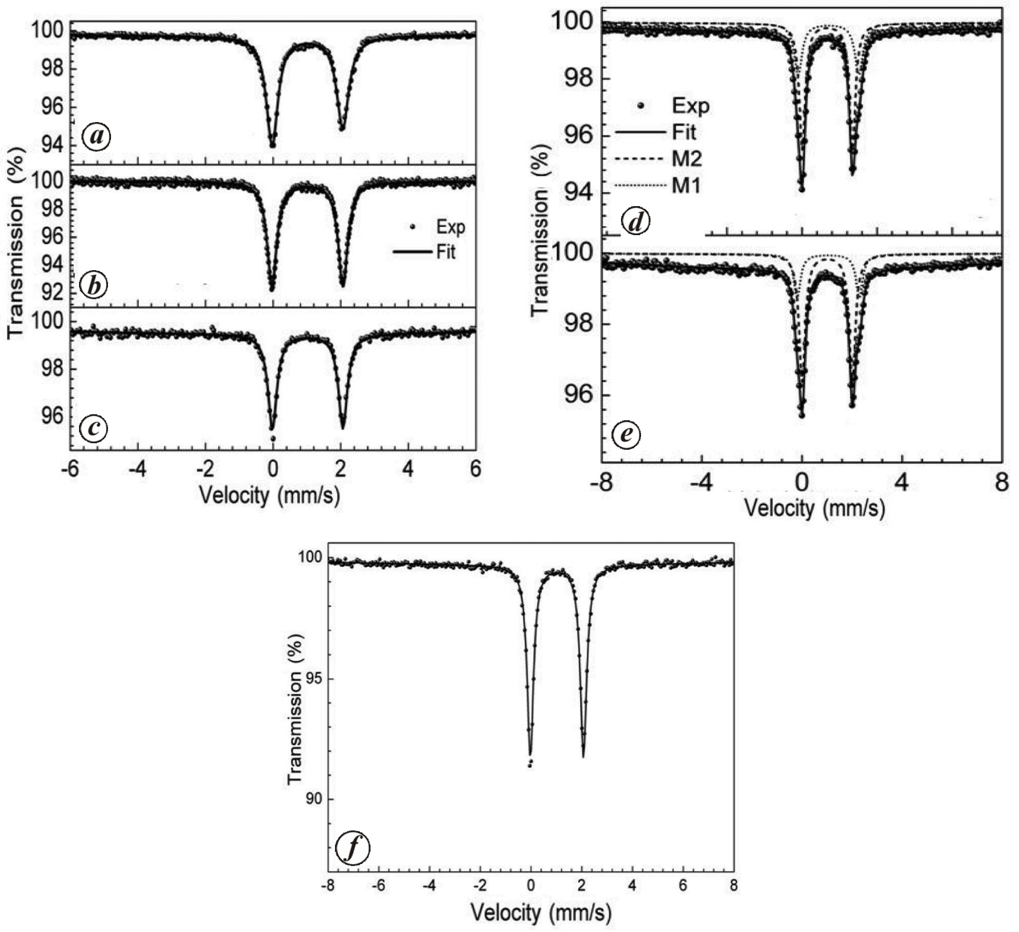

Figure 1. Mössbauer spectrum of (***a***) Vissannapeta, (***b***) Talampaya and (***c***) Serra de Magé eucrites, (***d***) Sioux-Country and (***e***) Millbillillie basaltic eucrites and (***f***) Ibbenbüren diogenite recorded at 300 K.

possibilities for the asymmetric doublet could be the presence of a single line component closely overlapping with the low velocity peak. In meteorites, such a component might come from gamma phase of Fe–Ni metal. We used a single-line component superposed on a symmetric doublet to the Vissannapeta spectrum to look for such a phase. The singlet with an intensity of about 7% was unphysical, as the line width was unreasonably low. We also fitted two single lines to the spectrum with unconstrained intensity and line width. Any presence of a third component is likely to result in broadening of one of the lines. But the line width of the two peaks in the two-line fitting was found to be equal. All these do not support an iron phase other than in pyroxene. Since pyroxenes are sensitive to the application of pressure, one can argue that this asymmetry may arise due to preparation of absorber in the laboratory. But this is ruled out because under similar experimental conditions, Verma *et al.*[25] have reported Mössbauer spectroscopic study of several ordinary chondrite samples, but none of the samples exhibited asymmetry in Mössbauer peaks of pyroxene doublet. Our results indicate that only cumulate bodies show asymmetry in Mössbauer peaks. This asymmetry indicates preferential orientation of pyroxene crystals in cumulate meteorites. Application of pressure may result in this type of orientation. Since preferential orientation is the characteristic of cumulate bodies, it is possible that it has some extraterrestrial origin. It suggests that this should have some correlation with the cumulate bodies originating from the Vesta asteroid.

Recently Chandra *et al.*[26] have reported ^{57}Fe Mössbauer spectroscopic study of Lohawat howardite at different pressures. The main findings of their study are:

(1) The Mössbauer spectrum of uncompressed sample of Lohawat howardite exhibits two quadrupole doublets corresponding to M2 and M1 sites without any asymmetry. They observed almost identical Mössbauer spectrum and Mössbauer parameters as Verma *et al.*[25] for this meteorite in transmission geometry. They found that just by applying a small pressure (~2 GPa), Mössbauer pattern becomes asymmetric and this asymmetry is irreversible and increases with increase in pressure (c.f. Figure 2 *a*).

Table 2. Mössbauer parameters of HED meteorites. All spectra are recorded at room temperature. Fe^{2+} is written for pyroxene sites

Sample meteorite	IS (mm/s)	QS (mm/s)	LW (mm/s)	Area (%)	Assignment	Reference
Vissannapeta (Cumulate eucrite)	1.10	2.11	0.42	100	M2	Present study
Talampaya (Cumulate eucrite)	1.11	2.12	0.34	100	M2	Present study
Serra de Magé (Cumulate eucrite)	1.11	2.09	0.32	100	M2	Present study
Sioux-Country (Basaltic eucrite)	1.10	2.01	0.28	65	M2	Present study
	1.15	2.42	0.36	35	M1	
Millbillillie (Basaltic eucrite)	1.10	2.01	0.29	73	M2	Present study
	1.14	2.49	0.32	27	M1	
Piplia Kalan (Basaltic eucrite)	1.08	1.94	–	73	M2	24
	1.13	2.40	–	22	M1	
	0.26	0.93	–	5	Fe^{3+}	
Ibitira (Basaltic eucrite)	1.17	1.94	–	–	M2	33
	1.15	2.54	–	–	M1	
Stannern (Basaltic eucrite)	1.13	1.99	–	–	M2	34
	1.16	2.46	–	–	M1	
	1.13	1.96	–	–	M2	35
	1.17	2.08	–	–	M2	
	1.18	2.53	–	–	M1	
	1.07	0.68	–	–	Fe^{2+}	
Cachari (Basaltic eucrite, glass)	1.10	2.02	0.43	56	M2	36, 37
	1.13	2.55	0.54	20	M1	
	1.00	1.52	0.51	24	Fe^{2+}	
Cachari (Basaltic eucrite)	1.16	2.46	–	64	M2	
	1.13	2.00	–	36	M1	
Ibbenbüren (Diogenite)	1.11	2.10	0.30	100	M2	Present study
ALH77256-121 (Diogenite)	1.10	2.10	–	100	M2	25
Tatahonie (Diogenite)	1.11	2.12	–	100	M2	25
Bilanga (Diogenite)	1.11	2.11	–	100	M2	25
Lohawat (Howerdite)	1.18	2.03		73	M2	24
	1.22	2.52		27	M1	
Kapoeta (Howardite)	1.13	1.95	–	59	M2	38
	1.14	2.26	–	41	M1	

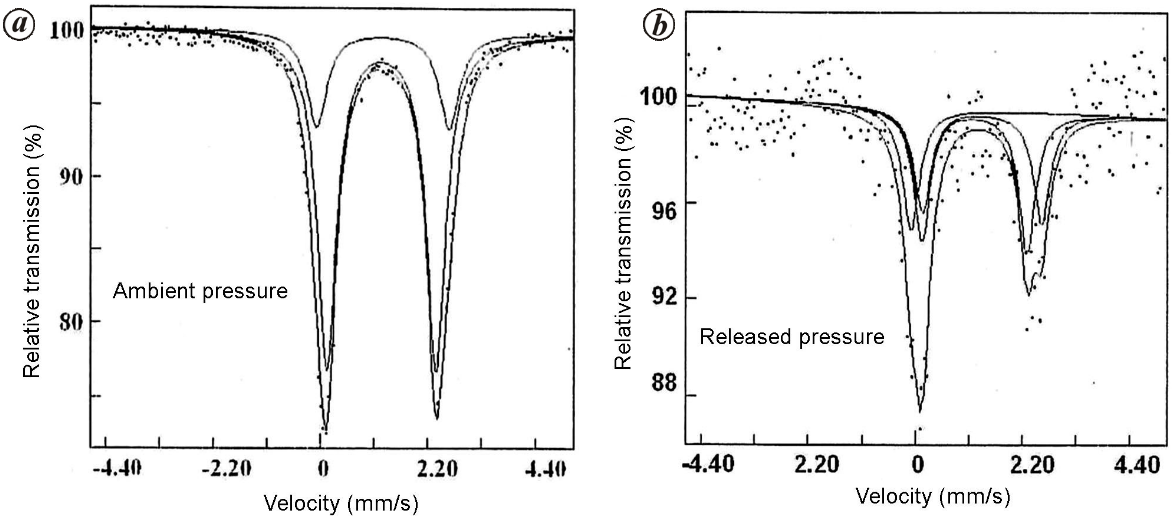

Figure 2. Mössbauer spectrum (*a*) of the uncompressed sample and (*b*) at ambient pressure after decompression (reproduced from Chandra *et al.*[26]).

(2) Mössbauer spectrum of samples recorded at different pressures shows considerable asymmetric pattern.

(3) Lohawat howardite sample was first compressed at 8.4 GPa and then after decompressing it, the Mössbauer spectrum was recorded at ambient pressure. It can be seen from Figure 2 *b* that this asymmetry still persists. This confirms the irreversibility of asymmetry. Apart from this, they have also shown that

> there is considerable change in the site occupancy of Fe^{2+} in M2 and M1 sites and these are more equally populated in decompressed samples compared to uncompressed samples. This distribution is almost similar as observed in Kapoeta howardite. For the sake of continuity, Figure 2 is reproduced from Chandra *et al.*[26].

The study carried out by Chandra *et al.*[26] suggests that when pressure is applied on meteorite samples, there is irreversible change in crystal orientation and site occupancy of pyroxene. To check that if the crystals are orientated in Vissannapeta, we recorded the Mössbauer spectrum with the sample plane rotated at 45° in gamma direction. It can be seen from Figure 3 that the area ratio of two peaks has changed significantly and becomes more symmetric.

On the basis of the above discussion, the appreciable asymmetry observed in Mössbauer spectrum, particularly in Vissannapeta and in general in cumulate bodies of HED meteorites can be explained only if we assume that these bodies must have experienced appreciable pressure (possibly through an impact event) from where they originated. There are two possibilities; either this pressure is due to overburden, if they are formed at depth, or during their extrusion from the interior to the surface. However, using Mössbauer technique it is not possible to distinguish between these two possibilities.

All the three cumulate eucrites, namely Vissannapeta, Talampaya and Serra de Magé, show a single quadrupole doublet. This means that Fe^{2+} ions occupy largely one of the two inequivalent sites M1 and M2 in pyroxene crystal structure. The quadrupole splitting of around 2.10 mm/s shows these ions occupy the M2 site. On the other hand, the two basaltic eucrites, Sioux-Country and Millbillillie, studied here, show two quadrupole doublets, one with quadrupole splitting around 2.01 mm/s and the other around 2.45 mm/s (Figure 1). The basaltic eucrites Stannern and Cachari, Piplia Kalan and Ibitira show similar characteristic as depicted in Table 2. The ratio of Mössbauer area in M2 to that in M1 for some basaltic eucrites varies between 1.7 and 3.2, as shown in Table 3. This is exactly similar as predicted by Burns[18]. It can be seen that even in the basaltic eucrites the M2 site is preferentially occupied compared to the M1 site. As we have already mentioned, the ratio of relative distribution of Fe^{2+} in M2 and M1 sites depends on cooling history of meteorite and Fe^{2+}/Mg^{2+} disorder. The observed ratio of basaltic eucrites and preferential occupation of Fe^{2+} in cumulate eucrites in the present study is inconsistent with that reported in the literature for many other basaltic eucrites. We can infer that cooling history of Vissannapeta eucrite and Sioux-Country (basaltic eucrite) must be different. Pyroxene in Vissannapeta eucrite crystallizes at low temperature and cool slowly. On the other hand, pyroxene in Sioux-Country (basaltic eucrite) may have crystallized at a very high temperature and cooled rapidly[21]. It is not possible to provide accurate range of cooling history and cooling rate of the meteorite on the geological timescale. However, in the near infrared study of a HED body, Klima *et al.*[21] have used several synthesized pyroxene samples at different temperatures and with different cooling rates. They have also studied some terrestrial samples containing pyroxenes. Their cooling history was known from their origin. They have also reported excellent comparison and correlation between cooling history and cooling rate of synthesized pyroxenes and those present in the meteorites. However, a crude estimate can be given. Accordingly, basaltic rocks are formed at temperatures above 800°C and cumulate rocks much below this.

The diogenite sample Ibbenbüren studied here, also shows a single quadrupole doublet of IS = 1.10 mm/s and QS = 2.10 mm/s (Figure 1). The other diogenites

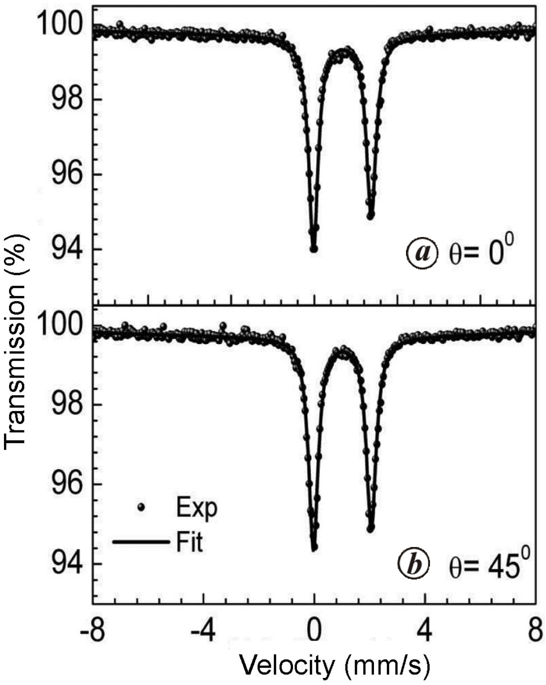

Figure 3. Mössbauer spectrum of the Vissannapeta eucrite recorded with the sample normal at an angle (***a***) 0° and (***b***) 45° to the incident gamma ray.

Table 3. Mössbauer area ratio of two doublets (M1 and M2) of basaltic eucrites

Sample	M1 and M2 Mössbauer area ratio of basaltic eucrites
Sioux-Country	1.8
Ibitira	1.9
Millbillillie	2.7
Cachari	2.8
Piplia Kalan	3.3

ALH77256-121, Tatahouine and Bilanga, studied earlier, also show similar Mössbauer spectra[27]. All these samples differ in their terrestrial history. For example, Bilanga is a relatively recent fall (1999), Tatahouine is about 80-years-old fall, whereas ALH77256 is a find from Allens Hills, Antarctica. The Fe occupancy in pyroxene sites of diogenites shows the same behaviour – that of cumulate eucrites. This suggests that the preferential occupancy of Fe^{2+} in M2 site of pyroxene both in cumulate eucrites and diogenites is in consistent with magma ocean model and suggests that cumulate eucrites and diogenites are genetically related. Interestingly, diogenites also show slightly asymmetric doublet.

Table 2 gives Mössbauer parameters of two howardite meteorites, namely Lohawat and Kapoeta. As howardites show a lithology representing mixed eucrites and diogenites, one would expect that in howardites relative distribution of Fe^{2+} in M2 and M1 sites in pyroxenes should depend on the statistical and weightage average of diogenite and eucrite. The cumulate eucrites and diogenites show iron occupancy only in the M2 site, while the basaltic eucrites show 65–75% absorption area in the M2 site. On a physical mixture model, the absorption area in M2 sites in Mössbauer spectrum of a howardite may be expected to be more than 75%. This area is 59% for Kapoeta, whereas for Lohawat it is 73%.

The large variation in the site occupancy of M2 and M1 cannot be explained only on the basis of physical mixture model. Further it can be seen that quadrupole doublet in Mössbauer spectrum in howardite always shows, sharp line width. But in physical mixture model, we expect considerable broadening in the line width of the doublets due to large variation in the chemical composition of pyroxene grains. Hence we also expect a large fluctuation in the symmetry around iron atom. This should result in larger line width in the Mössbauer spectrum. It appears that the impact may be responsible for mixing of eucrite and diogenite layers in Vesta-4, which might have changed the iron mineralogy. This inference is also in agreement with the results obtained by Chandra *et.al.*[26].

In all the cumulate eucrites and diogenites (which are also cumulate), Fe^{2+} occupies dominantly in the M2 site of pyroxene. The basaltic eucrites show Fe^{2+} occupation in both M1 and M2 sites, with M2 having larger absorption area. Mössbauer parameters for these HED meteorites do not support a physical mixture model of howardites. It also appears that the local temperature and pressure showed a rise during the big impact on Vesta-4, which has changed the iron mineralogy while mixing eucrites and diogenites.

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A new direct retrieval method of refractive index for the metamaterial

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With the advent of metamaterial the approaches of automated extraction of effective parameters for a metamaterial have recently attracted considerable attention among researchers. Evaluation of refractive index has received huge importance especially for the left-handed metamaterial. This communication presents a new simplified equation for the direct retrieval of refractive index from the transmission and reflection co-efficient. Refractive index is calculated from the new equation for two recently published metamaterials. The resultant curve for the proposed method shows good conformity with the classical NRW-method and shows a result closer to that achieved using the TR-method. In addition, a comparative study of the three methods is presented.

Keywords: Metamaterials, Nicolson–Rose–Weir method, transmission–reflection method.

METAMATERIAL is an artificially engineered material which exhibit some extraordinary electromagnetic properties; it does not exist naturally. These extraordinary electromagnetic properties of the metamaterials have extended their demand in many important applications like antenna design, electromagnetic cloaking, electromagnetic absorption reduction, etc.[1–3]. A metamaterial may be either single-negative (SNG) or double-negative (DNG). A DNG or left-handed (LHM) or negative refractive index metamaterial is one that is characterized by its effective parameters, i.e. permeability and permittivity. For the left-handed material or negative-refractive index metamaterial the effective parameters have to be negative simultaneously, which never happens naturally. Parameter extraction is one of the important tasks for characterizing the metamaterial. Due to the growing interest on metamaterial research, the effective parameter extractions as well as direct calculation technique of refractive index have received much attention by the researchers.

Different methods for effective parameter extraction of metamaterials are available[4–14]. Among them, a class exist based on the transmission and reflection parameters, like TR (transmission–reflection) method, direct-retrieval method, Nicolson–Ross–Weir (NRW) method, etc.

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