



Magnetic phases in hemo-ilmenite: Insight from low-velocity and high-field Mössbauer spectroscopy

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[1] We have studied natural samples of hemo-ilmenite (ilmenite with hematite exsolution lamellae) by use of Mössbauer spectroscopy. The samples are from the Allard Lake intrusion in Quebec. The stoichiometric compositions of the ilmenite host and the hematite lamellae are approximately $\text{Fe}^{2+}_{0.84}\text{Mg}^{2+}_{0.14}\text{Ti}^{4+}_{0.98}\text{Fe}^{3+}_{0.04}\text{O}_3$ and $\text{Fe}^{2+}_{0.17}\text{Mg}^{2+}_{0.01}\text{Ti}^{4+}_{0.18}\text{Fe}^{3+}_{1.64}\text{O}_3$, respectively. From low-velocity Mössbauer measurements, the Fe^{2+} and Fe^{3+} components of the Fe^{2+} -substituted hematite and Fe^{3+} -substituted ilmenite, formed by incomplete exsolution of hematite and ilmenite, have been suggested identified. High-field Mössbauer measurements, obtained at low-temperature, indicate the presence of a minor ferrimagnetic component. **Citation:** Frandsen, C., S. Mørup, S. A. McEnroe, P. Robinson, and F. Langenhorst (2007), Magnetic phases in hemo-ilmenite: Insight from low-velocity and high-field Mössbauer spectroscopy, *Geophys. Res. Lett.*, *34*, L07306, doi:10.1029/2006GL029063.

1. Introduction

[2] Hemo-ilmenite (ilmenite with exsolution lamellae of hematite) has recently attracted considerable attention, because its magnetic properties differ from those of pure ilmenite (FeTiO_3) and pure hematite ($\alpha\text{-Fe}_2\text{O}_3$). In particular, hemo-ilmenite-rich intrusions have been found to show a magnetic remanence, which cannot be explained by paramagnetic ilmenite, and which exceeds what can be explained by pure hematite that has only a small magnetization from its canted antiferromagnetism [see e.g., McEnroe *et al.*, 2001]. Understanding the magnetic properties of hemo-ilmenite, and especially revealing the magnetic components present, is considered important for understanding remanent magnetic anomalies on Earth as well as on other terrestrial planets such as Mars [see e.g., McEnroe *et al.*, 2002].

[3] Hemo-ilmenite forms from a solid solution of Fe_2O_3 and FeTiO_3 by slow cooling. Owing to immiscibility below ~ 1000 K between FeTiO_3 and $\alpha\text{-Fe}_2\text{O}_3$, exsolution lamellae form, with the most abundant phase being the host of the other phase. The exsolution does not produce pure end-member phases but near end-members through the substi-

tion $2\text{Fe}^{3+} \leftrightarrow \text{Fe}^{2+} + \text{Ti}^{4+}$. The exsolution lamellae are in multiple generations that range in thicknesses from micrometers down to 1–2 nanometers [see e.g., McEnroe *et al.*, 2002]. The phase diagram of the hematite-ilmenite series is described in detail by e.g., Ishikawa and Akimoto [1957], Burton [1991], Harrison *et al.* [2000], McEnroe *et al.* [2002], and Robinson *et al.* [2004].

[4] In order to understand the complex magnetic properties of hemo-ilmenite, it is relevant to apply different experimental techniques, including Mössbauer spectroscopy [Dyar *et al.*, 2004]. This technique allows study of Fe-containing minerals and is particularly useful for determination of specific Fe^{2+} and Fe^{3+} sites, their $\text{Fe}^{2+}/\text{Fe}^{3+}$ ratios, and magnetic properties. Here we demonstrate how Mössbauer spectroscopy can be used to reveal the Fe-bearing phases in hemo-ilmenite. In accordance with Dyar *et al.* [2004], we find that the Fe^{3+} -hematite and Fe^{2+} -ilmenite components dominate the spectra, but by use of low-velocity amplitude, low-temperature, and high-field measurements, we have resolved further details about the composition and the magnetic properties.

2. Samples

[5] We studied hemo-ilmenite samples from the Allard Lake intrusion in Quebec [Hargraves, 1959]. By Mössbauer spectroscopy we studied mainly two fractions of the AL23D sample [Hargraves, 1959], here named AL23D-1 and AL23D-2. Sample AL23B [Hargraves, 1959] comes from the same drill core as AL23D, and appears to be similar to AL23D. For AL23B we obtained chemical analyses by microprobe (EMPA) and transmission electron microscopy (TEM). We chose to use those results to compare with the Mössbauer results on the AL23D samples given in this paper.

[6] Our microprobe measurements show molar compositions of the ilmenite host ranging between Ilm91 and Ilm92, where Ilm = mol% ($\text{FeTiO}_3 + \text{MgTiO}_3$), and of the hematite lamellae ranging between Ilm26 and Ilm29. Analytical measurements made by energy-dispersive X-ray microanalysis (EDX) on TEM allows for finer spatial resolution than the microprobe [Langenhorst *et al.*, 1995]. Our EDX measurements show compositions of the ilmenite host in the range Ilm97.8–Ilm101 and hematite lamellae being Ilm15.2–Ilm19.7. The smallest lamellae, a few unit cells wide, are still beyond our analytical abilities to obtain accurate compositions by EDX. The analyses further show that ilmenite and to much smaller extent hematite contains substituted Mg^{2+} -ions corresponding to geikielite (MgTiO_3) components of 13–14% and 0–2%, respectively. The abundances of other cations are negligible and not considered here. We find the approximate respective compositions

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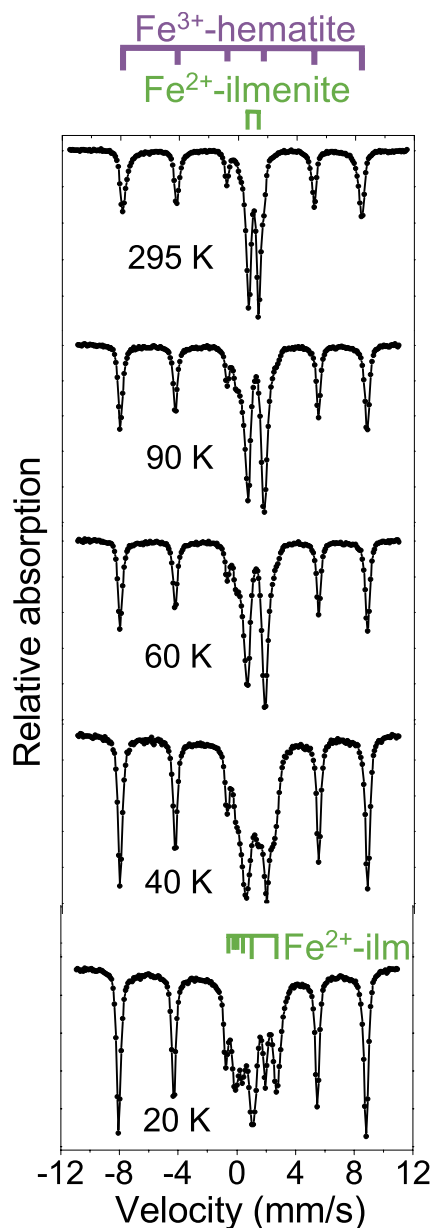


Figure 1. Mössbauer spectra of AL23D-1 obtained at the indicated temperatures. The bar diagrams indicate the line positions of the Fe^{3+} -hematite and Fe^{2+} -ilmenite sextets and doublet. The solid lines connect data points.

of the host and the lamellae are $\text{Fe}^{2+}_{0.84}\text{Mg}^{2+}_{0.14}\text{Ti}^{4+}_{0.98}\text{Fe}^{3+}_{0.04}\text{O}_3$ and $\text{Fe}^{2+}_{0.17}\text{Mg}^{2+}_{0.01}\text{Ti}^{4+}_{0.18}\text{Fe}^{3+}_{1.64}\text{O}_3$.*

3. Mössbauer Spectroscopy

[7] Mössbauer spectra were obtained using conventional constant acceleration spectrometers with sources of ^{57}Co in rhodium. Low-temperature spectra were obtained using temperature-controlled liquid nitrogen and helium cryostats. Measurements were obtained with velocity amplitude of either ~ 12 mm/s or ~ 4 mm/s. High-field measurements (6 T) were obtained at 6 K by use of a superconducting coil.

*The composition is correct here. The article as originally published is online.

The instruments were calibrated by use of a $12.5 \mu\text{m}$ foil of $\alpha\text{-Fe}$ at room temperature. Velocities and isomer shifts are given relative to the centroid of the calibration spectrum. The Mössbauer spectra were fitted with the program “mfitt” (available at <http://www.raunvis.hi.is/~kj/mfitt/>).

4. Results and Discussion

[8] Figure 1 shows Mössbauer spectra of the hemo-ilmenite sample AL23D-1 at temperatures between 20 K and 295 K. The spectra are dominated by the contributions from Fe^{3+} in hematite and Fe^{2+} in ilmenite. At low temperatures (20 K) the Fe^{3+} -hematite sextet has relatively narrow lines, a hyperfine field of ~ 52.3 T, an isomer shift of ~ 0.49 mm/s, and a quadrupole shift of -0.11 mm/s. In pure $\alpha\text{-Fe}_2\text{O}_3$ the quadrupole shift is $+0.20$ mm/s below the Morin transition temperature, $T_M \sim 260$ K [Ericsson *et al.*, 1986]. The quadrupole shift of about -0.10 mm/s at low temperature of hematite in the Allard Lake sample shows that the Morin transition is absent. This can be explained by the presence of titanium in the hematite structure since as little as 1% Ti^{4+} can suppress the Morin transition below 10 K [Ericsson *et al.*, 1986]. The magnetic hyperfine field in the hemo-ilmenite sample is about 1–2% less than that of pure hematite, even when compensating for the absence of a Morin transition. It is likely that the reduced hyperfine field is caused by substitution of non-magnetic cations such as Ti^{4+} (and Mg^{2+}) in the hematite. With increasing temperature (Figure 1) the lines of the Fe^{3+} -hematite sextet broaden asymmetrically, especially for the outer lines. Non-magnetic neighbours like Ti^{4+} (and Mg^{2+}) lead to a faster decrease in magnetic hyperfine field with increasing temperature, and the line broadening observed for Fe^{3+} -hematite is presumably caused by a distribution in cations surrounding Fe^{3+} .

[9] Below ~ 40 K, the spectrum of Fe^{2+} -ilmenite is a sextet, with a magnetic hyperfine field of up to 5.2 T, a quadrupole shift of 0.84 mm/s, and an isomer shift of 1.13 mm/s, indicating that ilmenite is below its Néel temperature. (There may be minor uncertainties in the parameters obtained for the sextet of magnetically ordered ilmenite. The reason is that while the magnetic hyperfine field of ilmenite is rather small, the fitting programme uses the approximation that the magnetic hyperfine interaction should be large compared to the quadrupole interaction.) At temperatures above ~ 40 K, the Mössbauer component of ilmenite is a doublet, indicating that ilmenite is paramagnetic (at room temperature, the isomer shift is 1.06 mm/s and the quadrupole splitting is 0.70 mm/s). The Néel temperature (~ 40 K) found here is in accordance with low-temperature susceptibility measurements (S. A. McEnroe *et al.*, Mineral chemistry, phase relations and magnetic properties of hemo-ilmenite ores with micron- to nanometer-scale exsolution lamellae from Allard Lake, Quebec: Implications for planetary magnetization, submitted to *Journal of Geophysical Research*, 2007, hereinafter referred to as McEnroe *et al.*, submitted manuscript, 2007), but it is much lower than that for pure ilmenite (55 K, [Ishikawa and Akimoto, 1957]). We suggest this depression is caused by the substantial substitution of Mg^{2+} for Fe^{2+} , and it may also be influenced by the small substitution of Fe^{3+} as shown by Ishikawa *et al.* [1985].

[10] Incomplete exsolution should lead to ilmenite containing Fe^{3+} . Fitting for instance the room temperature

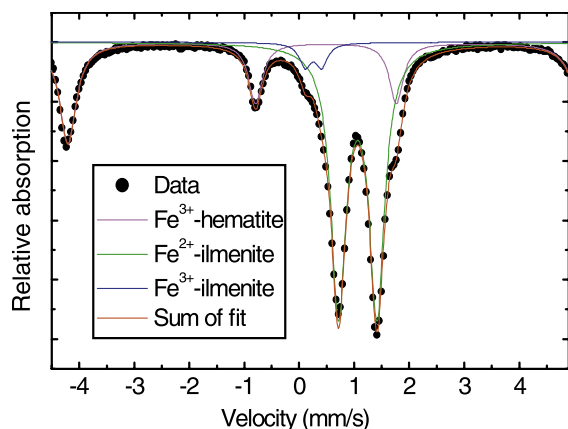


Figure 2. Room temperature spectrum of AL23D-1 obtained with small velocity amplitude. Fit to data is shown.

spectra of AL23D-1 with one doublet due to Fe^{2+} in ilmenite and a sextet due to Fe^{3+} -hematite does not provide a complete fit to the central part of the spectrum since there is an extra absorption at around 0.1 mm/s, which has not been covered hereby. *Dyar et al.* [2004] suggested that a doublet corresponding to tetrahedrally coordinated Fe^{3+} could explain this kink seen on the low-velocity side of the Fe^{2+} -ilmenite doublet. The problem that remains, however, is to determine which mineralogic phase, or feature of a phase, this component can be ascribed to [*Dyar et al.*, 2004]. Most of our room-temperature hemo-ilmenite spectra can be fitted well by including a doublet with an isomer shift of 0.26 mm/s and a quadrupole splitting of 0.32 mm/s (see fit in Figure 2). These parameters are similar to those found by *Dyar et al.* [2004]. Recently, *Seda and Hearne* [2004] have reported that Fe^{3+} in ilmenite can have isomer shifts and quadrupole splittings close to the values found by *Dyar et al.* [2004] and by us. It is therefore likely that Fe^{3+} in ilmenite explains this absorption. At temperatures around and below the Néel temperature of the ilmenite phase, we further observe a sextet with very broad lines and a hyperfine field of up to around 50 T appearing in the spectra (in Figure 1 it is seen as a hanging background at 40 K and 20 K) presumably due to magnetic splitting of the spectral component of Fe^{3+} -ilmenite.

[11] Exsolution of hematite and ilmenite should also give rise to hematite containing Fe^{2+} , which compensates for the charge of substituted Ti^{4+} . In one sample, AL23D-2, we have a strong indication of what could be the Mössbauer signature of Fe^{2+} in hematite. It is difficult to resolve this component in the Mössbauer spectra obtained with the large velocity amplitude (as in Figure 1), but spectra obtained at room temperature with small velocity amplitude make it apparent (Figure 3). As can be seen from Figure 3, one can fit a sextet with a hyperfine field of 3.33 T, a quadrupole shift of 0.90 mm/s, and an isomer shift of 1.05 mm/s to the spectrum. The parameters of this sextet are similar to those of ilmenite below the Néel temperature. The electronic surroundings of Fe^{2+} in hematite are similar to those of Fe^{2+} in ilmenite, and it is therefore expected that the Mössbauer parameters should be similar. In contrast to the ilmenite, the spectrum of Fe^{2+} in hematite is magnetically split at room temperature, because hematite is magnetically ordered at this

temperature. It is therefore likely that the sextet can be ascribed to Fe^{2+} in hematite. The sextet of Fe^{2+} -hematite is less visible in the spectra of other hemo-ilmenite samples, which we have studied. This variation in signal may be a consequence of different abundances of the lamellae.

[12] Fe^{3+} in ilmenite, as well as phases like pyrite and pyroxene, have absorption lines, which may overlap in part with the absorption lines of Fe^{2+} -hematite. One could argue that perhaps such phases could account for the Fe^{2+} -hematite spectrum. However, in case of the Allard Lake AL23 samples, microprobe analyses show that phases like pyrite and pyroxene are rare ($\leq 1\%$, if any). The only non-oxide impurity seems to be plagioclase, which has no Mössbauer signature. Hence we do not find that such phases can explain the spectrum of Fe^{2+} -hematite in Figure 3.

[13] The results presented above show that by taking into account the Fe^{2+} in hematite and Fe^{3+} in ilmenite, produced by exsolution, in addition to Fe^{3+} -hematite and Fe^{2+} -ilmenite, it is possible to explain the main features of the Mössbauer spectrum of hemo-ilmenite. Especially, kinks on the low velocity side of the Fe^{2+} -ilmenite doublet, are suggested to be explained by this.

[14] From the relative areas of the absorption lines of the different components it is possible to estimate ratios of Fe^{3+} and Fe^{2+} in hematite and ilmenite. For instance, in hematite the $\text{Fe}^{2+}/\text{Fe}^{3+}$ ratio is ~ 0.19 (Figure 3) corresponding to $\sim \text{Ilm}28$, and in ilmenite the $\text{Fe}^{3+}/\text{Fe}^{2+}$ ratio is found to be ~ 0.06 (Figure 2) corresponding to $\sim \text{Ilm}97$. These ratios, obtained from the Mössbauer spectra, may be slightly overestimated due to saturation of the most intense lines (especially Fe^{2+} -ilmenite) and due to spectral overlap of minor components. In case of hematite, the Fe^{2+} -component is only clearly resolvable in the spectra of AL23D-2, hence in general the average composition of the hematite lamellae is expected to be closer to end-member composition, i.e. \sim below Ilm20, as also indicated by EDX. For the ilmenite spectra the estimate is in agreement with the low end of the EDX results at Ilm98. The correspondence between results from Mössbauer spectroscopy and the chemical analyses (EDX and EMPA), all showing significant solid solution, supports the existence of the suggested components in the Mössbauer spectra.

[15] In the hematite-ilmenite series, magnetic cation interchange, as found above from Mössbauer spectroscopy, is

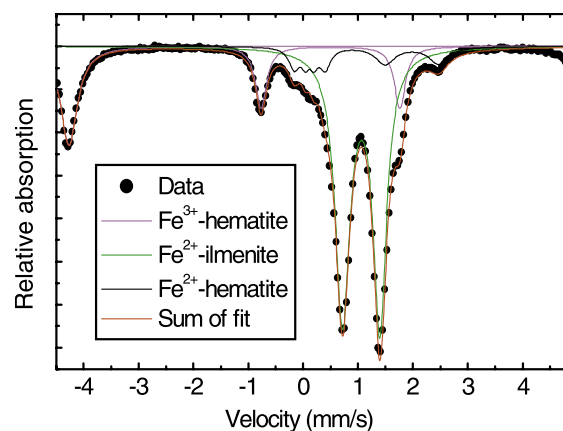


Figure 3. Room temperature Mössbauer spectrum of AL23D-2. Fit to data is shown.

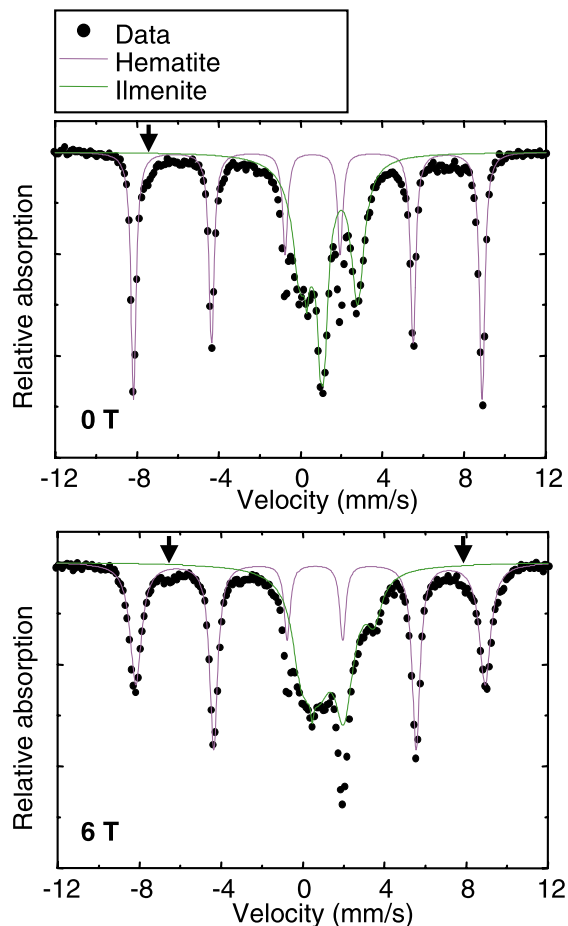


Figure 4. Mössbauer spectra of AL23D-1 obtained at 6 K, in zero-field and in an applied field of 6 T parallel to the γ -rays. Fits with Fe^{2+} -ilmenite and Fe^{3+} -hematite are shown. The arrows indicate positions of lines due to a ferrimagnetic component, as explained in the text.

well known and it should not itself lead to the intense remanent magnetizations found in hemo-ilmenite samples: The cation substitution in hematite is disordered [Ishikawa and Akimoto, 1957] and will not result in a ferrimagnetic phase. Fe^{3+} substitution in ilmenite might lead to a ferrimagnetic phase [see e.g., Ishikawa and Akimoto, 1957], but the Allard Lake ilmenite compositions are far too poor in Fe^{3+} to exhibit such ferrimagnetism. Instead, it has been suggested that the strong magnetic remanence of hemo-ilmenite is due to “lamellar magnetism” - a ferrimagnetic substructure - mainly originating from uncompensated magnetic spins at the interfaces of hematite lamellae that have odd numbers of magnetic cation layers [Robinson et al., 2004]. The cation layers at the interfaces may contain a mixture of Fe^{2+} and Fe^{3+} [Robinson et al., 2002, 2004]. Calculations suggest that the magnetization of such “lamellar” ferrimagnetic material could be as much as 32 times larger than that of hematite if all the lamellae were perfectly in-phase, a situation not expected in natural samples [Robinson et al., 2002, 2004].

[16] We have looked for traces of ferrimagnetic components in the Mössbauer spectra. Such components may be

identified by recording spectra both in zero-magnetic-field and in large applied fields. In Figure 4, spectra of sample AL23D-1 at 6 K in zero-field and in a field of 6 T, applied parallel to the gamma-rays in the Mössbauer spectrometer, are shown. If the hematite lamellae were ferrimagnetic due to uncompensated spins, the zero-field six-line spectrum of hematite would split into two sextets by the applied field because the hyperfine field of the two sublattices now differ [Bahl et al., 2006]. From the spectra in Figure 4, it can be seen that in general the hematite sextet does not split in two sextets with applied field. Thus, overall the hematite behaves like an antiferromagnet. This is also expected because most of the hematite is present as micrometer-sized lamellae. However, a close inspection of the hematite spectrum shows that the intensities of lines 2 and 5 increase with applied field, but only by $\sim 55\%$, not $\sim 100\%$ as ideally expected for hematite above T_M [Bødker et al., 2000]. For small lamellae, the moment from uncompensated spins may be comparable in size to or larger than that from canted antiferromagnetism, and such lamellae could account for the reduced line intensities.

[17] Additionally, we note from Figure 4 that there appear to be small “shoulders” in the outer lines. The most visible features are the shoulder at -7.4 mm/s, on the low-velocity side of line 1 in the zero-field spectrum, and the distinct absorption at -6.4 mm/s and the shoulder at $+7.7$ mm/s, both seen in the 6-T-spectrum, all indicated by the arrows in Figure 4. In order to account for these features, we have fitted the spectra by including a ferrimagnetic component: a zero-field-sextet, which, at 6 T, splits into two sextets with hyperfine fields 6 T smaller and larger than the zero-field-sextet. This ferrimagnetic component may be due to a cation arrangement of Fe^{2+} and Fe^{3+} at the interface (a contact layer), as has been suggested in the model of “lamellar magnetism” to provide strong remanent magnetization [Robinson et al., 2002]. However, the uncertainty of the fit is large and we have therefore omitted to show it.

[18] From the present data it is not possible to identify the ferrimagnetic component further. It is possible that it may be due to another ferrimagnetic phase than that of “lamellar magnetism”, but it cannot be explained by the presence of magnetite in the samples. Room-temperature spectra with very good statistics show that the characteristic B-site components of magnetite were absent. Neither does superparamagnetic magnetite appear to be present in the spectra. Also, high-temperature magnetization measurements indicate a Curie temperature between 595 and 620°C [McEnroe et al., submitted manuscript, 2007], i.e., well above that of magnetite, supporting its absence.

[19] We have seen the same high-field behaviour in Mössbauer spectra of different hemo-ilmenite samples. Furthermore, Mössbauer measurements obtained by application of a field of 6 T perpendicular to the gamma-rays show changes in intensities of lines 2 and 5 of the ferrimagnetic component, which support the interpretation of the spectra obtained in parallel field. The abundance of the indicated ferrimagnetic feature is unfortunately extremely low in all the samples and this leads to significant uncertainty to the results. Further investigations are clearly needed to elucidate the nature of this component, which could be a direct experimental signature of the feature

responsible for the magnetic remanence in hemo-ilmenite-rich rocks.

[20] In conclusion, the present study shows that detailed information about chemical variation in hemo-ilmenite samples can be obtained from Mössbauer spectroscopy. We have identified the Fe^{2+} and Fe^{3+} components of near-end member hematite and ilmenite. In particular, the Mössbauer signatures of Fe^{2+} -hematite and Fe^{3+} -ilmenite have been suggested. Including the Fe^{2+} and Fe^{3+} components resulting from exsolution provides the possibility to fully fit the Mössbauer data of hemo-ilmenite samples. In addition, high-field Mössbauer measurements show the presence of a minor ferrimagnetic contribution with implications for the intriguing rock-magnetic properties of hemo-ilmenite.

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