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Study of Chelyabinsk LL5 meteorite fragments with different lithology using Mössbauer spectroscopy with a high velocity resolution

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Abstract Study of four Chelyabinsk LL5 ordinary chondrite fragments with different lithology was carried out using optical and scanning electron microscopy, X-ray diffraction and ^{57}Fe Mössbauer spectroscopy with a high velocity resolution at 295 K. Components revealed from the Mössbauer spectra were related to the main iron-bearing crystals of minerals such as olivine, pyroxene, troilite, kamacite, taenite and chromite. However, the relative amount of these minerals appeared to be different in the studied fragments.

Keywords Chelyabinsk LL5 ordinary chondrite · Mössbauer spectroscopy · Iron-bearing minerals · Hyperfine parameters

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Introduction

Meteorites are of significant interest for various fields of knowledge as substances of extraterrestrial origin. Their formation process can be characterized with conditions of low temperature, extreme pressure, slow cooling, thermal, impact and other extreme effects. The structural states of meteorites substance formed in space as a result of these effects may not be reproducible under terrestrial conditions.

Ordinary chondrites were classified as stony meteorites. These meteorites are the most widespread among all meteorites found on Earth. The age of ordinary chondrites is considered close to that of the Solar system [1]. The mineral composition of these meteorites is similar to that in the majority of planets and asteroids in the Solar system. Ordinary chondrites consist of the crystals of iron-bearing minerals such as olivine (Fe, Mg)$_2$SiO$_4$, pyroxene (Fe, Mg, Ca)SiO$_3$, troilite FeS, chromite FeCr$_2$O$_4$, daubréelite FeCr$_2$S$_4$, kamacite α-Fe(Ni, Co), taenite γ-Fe(Ni, Co), etc. These meteorites contain 19–28 wt.% total iron including 10–18 wt.% iron in silicate minerals, 4–6 wt.% iron in troilite and 2–17 wt.% iron in the metallic grains consisted of kamacite and taenite [2]. Ordinary chondrites are divided into three groups H, L and LL which were determined by different total iron content and metallic iron content [1]. The high total iron content is in the H ordinary chondrites (High iron), the low total iron content is in the L ordinary chondrites (Low iron), while the low total iron and low metallic iron content is in the LL ordinary chondrites (Low iron, Low metal).

A big meteorite fall was observed on February 15, 2013 in Chelyabinsk Region, Russian Federation. On the next day the Meteoritical Expedition of the Ural Federal University went to the place of meteorite fall and collected more than 700 fragments of this meteorite matter. Later this meteorite was classified as ordinary chondrite of LL5 group with shock metamorphism grade S4 and terrestrial weathering grade W0 and named Chelyabinsk (Meteoritical Bulletin No. 102). The first studies of Chelyabinsk LL5 meteorite fragments demonstrated that these fragments had different lithology: light, dark and black (see, for instance, [3]). Therefore, a comparative study of four Chelyabinsk LL5 meteorite fragments with different lithology was carried out in the present work using metallography, scanning electron microscopy (SEM), X-ray diffraction (XRD) as well as Mössbauer spectroscopy with a high velocity resolution.

Mössbauer spectroscopy with a high velocity resolution permits to carry out precision measurements of Mössbauer spectra with substantially smaller step of the Doppler velocity increment for gamma-quanta energy modulation (the velocity scale discretization to form the velocity reference signal is 2^{12}, i.e. 4096 steps). As a result of this discretization, the instrumental (systematic) error for evaluation of the hyperfine parameters appeared to be almost one order smaller than that for conventional Mössbauer spectrometers with a low velocity resolution (i.e. when the velocity scale discretization does not exceed 512 steps), while Mössbauer spectra can be registered using 4096 channels of the analyzer. The latter permits also to carry out more reliable fit of complicated Mössbauer spectra [4]. Application of Mössbauer spectroscopy with a high velocity resolution, in particular, for the study of extraterrestrial matter demonstrated substantial advances in revealing new and more accurate information in comparison with similar studies carried out using Mössbauer spectroscopy with a low velocity resolution (see [5–7]). For example, various authors (see, for instance, [8–15]) revealed only a single spectral component related to each of such minerals as olivine, pyroxene or kamacite in the Mössbauer spectra of ordinary chondrites measured with a low velocity resolution. However, both olivine and pyroxene contain crystallographically non-equivalent sites M1 and M2 which are occupied by Fe^{2+} and Mg^{2+} cations in different proportions, which is why there are two quadrupole doublets in the Mössbauer spectra of natural and synthesized olivines and pyroxenes corresponding to these sites (see, for instance, [16, 17]). The metal grains in ordinary chondrites are inhomogeneous and contain regions of α- and γ-phases with variations in Ni concentration that may result in a superposition of several magnetic sextets (see, for instance, [18]). The spectral components related to the ^{57}Fe in the M1 and M2 sites in both olivine and pyroxene as well as those corresponding to kamacite and taenite with different Ni content were revealed exclusively from the fits of the Mössbauer spectra of ordinary chondrites measured with a high velocity resolution [5, 7, 19–21]).

Experimental

Four Chelyabinsk LL5 meteorite fragments with different lithology denoted as No. 1 and No. 1a with a light lithology, No. 2 with a mixed light and dark lithology and No. 3 with a black lithology were chosen for comparative study. Preparation of these fragments slices was carried out by the standard metallography method for investigation of meteorite samples using optical and scanning electron microscopy. Analysis of the morphology and chemical composition of studied fragments was done using inverted optical microscope Axiovert 40 MAT (Carl Zeiss) and scanning electron microscope ΣIGMA VP (Carl Zeiss) with energy dispersion spectroscopy (EDS) device X-max (Oxford Instruments). Then powdered samples were mechanically prepared from the surface of fragments slices for X-ray phase analysis and Mössbauer spectroscopy. X-ray diffraction patterns were measured using XRD-700 diffractometer (Shimadzu) operated at 40 kV and 30 mA with CuK_{α} radiation and silicon monochromator. After this measurement the powdered samples were glued on iron free aluminum foil with a diameter of 2 cm for Mössbauer measurements. The sample thickness did not exceed 10 mg Fe/cm^2.

Mössbauer spectra were measured using automated precision Mössbauer spectrometric system with a high velocity resolution (registration in 4096 channels) built on the basis of Mössbauer spectrometer SM-2201. Detailed description and characteristics of this spectrometric system are given in [4, 22, 23]. A source of $\sim 1.8 \times 10^9$ Bq ^{57}Co in rhodium matrix (Ritverc GmbH, Saint-Petersburg) was used at room temperature. Measurements were carried out in transmission geometry with moving absorber at room temperature. A standard absorber α-Fe foil with a thickness of 7 μm was used for calibration of the velocity scale. Its spectrum lines had Lorentzian shape with a line width of 0.236 ± 0.008 mm/s. Each spectrum measurement time was in the range between one and 2 weeks. The measured Mössbauer spectra for their further fitting procedure were converted into 1024 channels by consequent summation of four neighboring channels to increase signal-to-noise ratio for the lowest intensity meteorite spectral components.

Mössbauer spectra were fitted with the least square method using UNIVEM-MS code (Research Institute of Physics, Southern Federal University, Rostov-on-Don) with Lorentzian line shape. Mössbauer parameters isomer shift, δ, quadrupole splitting, ΔE_Q, twice of the first order quadrupole shift for magnetic sextet components, 2ε, effective hyperfine magnetic field, H_{eff}, line width, Γ, relative subspectrum area, A were estimated for the spectra. The quality of the fit was evaluated using differential spectrum, statistical criterion χ^2 and physical meaning of parameters. Two types of errors, instrumental (systematic) and statistical, were taken into consideration for estimation of the errors of the obtained Mössbauer parameters. The instrumental (systematic) error in the velocity values attributed to the spectrum points, in the hyperfine parameters and in the line widths was taken to be equivalent to ±0.5, ±1 and ±2 channels, respectively [22]. From the statistical and instrumental (systematic) errors, it is the larger one that is indicated as the estimated error of the obtained parameters. All isomer shifts are given with respect to α-Fe at 295 K.

Additionally, the Mössbauer spectra of samples No. 1a (light lithology) and No. 2 (mixed light and dark lithology) were also fitted by using version 4.0Pre of the MossWinn program [24], in order to account for the spectral component of troilite more accurately, i.e. by the numerical diagonalization of the full static Hamiltonian of the corresponding magnetic and quadrupole hyperfine interactions for the excited and ground nuclear states. In this approach, apart from the corresponding A, δ, H_{eff} and Γ parameters, the troilite component was also determined by the V_{zz} (the main component of the electric field gradient tensor—EFG), η (asymmetry parameter of the EFG), β and α (respectively denoting the polar and azimuthal angle of H_{eff} in the eigensystem of the EFG) parameters. In order to alleviate the fit parameter ambiguity problem associated with the troilite spectrum component [26–28], as well as to promote comparison of the obtained results with those of previous studies [25], the asymmetry parameter η and the azimuthal angle of the magnetic hyperfine field vector in the EFG eigensystem, α, were fixed within this approach to 0.3 and 49°, respectively, in accordance with [25]. In addition, for the troilite component in the samples No. 1a and No. 2 the invariants S_0, S_1 and S_2 [26, 27] were determined as well.

Results and discussion

The slices microphotographs of Chelyabinsk LL5 meteorite fragments with different lithology are shown in Fig. 1. Their analysis demonstrated the presence of the metal grains and troilite inclusions in the silicate matrix of both olivine and pyroxene. The shock-melted troilite veins were additionally found in fragment No. 3 with a black lithology. A small amount of chromite inclusions was observed in these fragments. Chemical analysis by SEM with EDS showed that the metal grains contain kamacite, martensite α_2-Fe(Ni, Co), taenite and tetrataenite γ-FeNi. Martensite was observed in fragments No. 2 with a mixed light and dark lithology and No. 3 with a black lithology only. The Ni concentration was found to vary from 3 to 6 at.% in α-Fe(Ni, Co), from 8 to 25 at.% in α_2-Fe(Ni, Co), from 26 to 46 at.% in γ-Fe(Ni, Co), and from 46 to 50 at.% in γ-FeNi. The Co concentration in both α- and γ-phases was found up to 3.5 and 1.5 at.%, respectively. The troilite inclusions contained ~50–52 at.% of S and ~50–48 at.% of Fe. The Fe and Cr content in chromite was ~16–18 and ~24–28 at.%, respectively. The results of X-ray diffraction showed the presence of the main iron-bearing minerals such as olivine (from 53 to 63 wt.%), pyroxene (from 26 to 29 wt.%) and troilite (from 4 to 12 wt.%) in all fragments.

The Mössbauer spectra of four Chelyabinsk LL5 meteorite fragments with different lithology are shown in Fig. 2. These spectra were fitted first using UNIVEM-MS code. The best fit of spectra of the fragments studied revealed different number of components (magnetic and paramagnetic). Eight components related to the ^{57}Fe in kamacite, taenite and troilite (three magnetic sextets), in the M1 and M2 sites in both olivine and pyroxene (two pairs of quadrupole doublets) and in chromite (one paramagnetic singlet) were revealed for the Mössbauer spectrum of fragment No. 1 in accordance to earlier obtained results for ordinary chondrites [5] and chromite [29]. Meanwhile, a larger number of components related to the ^{57}Fe in kamacite, taenite and troilite (four magnetic sextets), in the M1 and M2 sites in both olivine and pyroxene (two pairs of quadrupole doublets), in chromite and paramagnetic taenite (two paramagnetic singlets) were revealed in the Mössbauer spectra of fragments No. 1a, No. 2 and No. 3. All spectral components associated with corresponding minerals on the basis of estimated hyperfine parameters are listed in Table 1. The iron content in the same minerals, which can be roughly evaluated using the corresponding relative subspectra areas, appeared to be different for four fragments of Chelyabinsk LL5 meteorite (see Table 1).

A comparison of the hyperfine parameters for Mössbauer spectral components related to olivine, pyroxene and metallic phases is shown in Fig. 3. It is interesting to point out that small differences in the hyperfine parameters beyond the error were observed for some crystals of the same iron-bearing minerals in Chelyabinsk LL5 meteorite fragments with different lithology. For example, the most visible differences in the hyperfine parameters for the ^{57}Fe in the M1 and M2 sites in olivine are clearly seen for fragment No. 1 in comparison with those for other fragments. The differences in the hyperfine parameters revealed for the spectral components corresponding to both olivine and pyroxene indicate small structural variations in the local environment of iron ions in these crystals for four different fragments. It should be noted that in the earlier

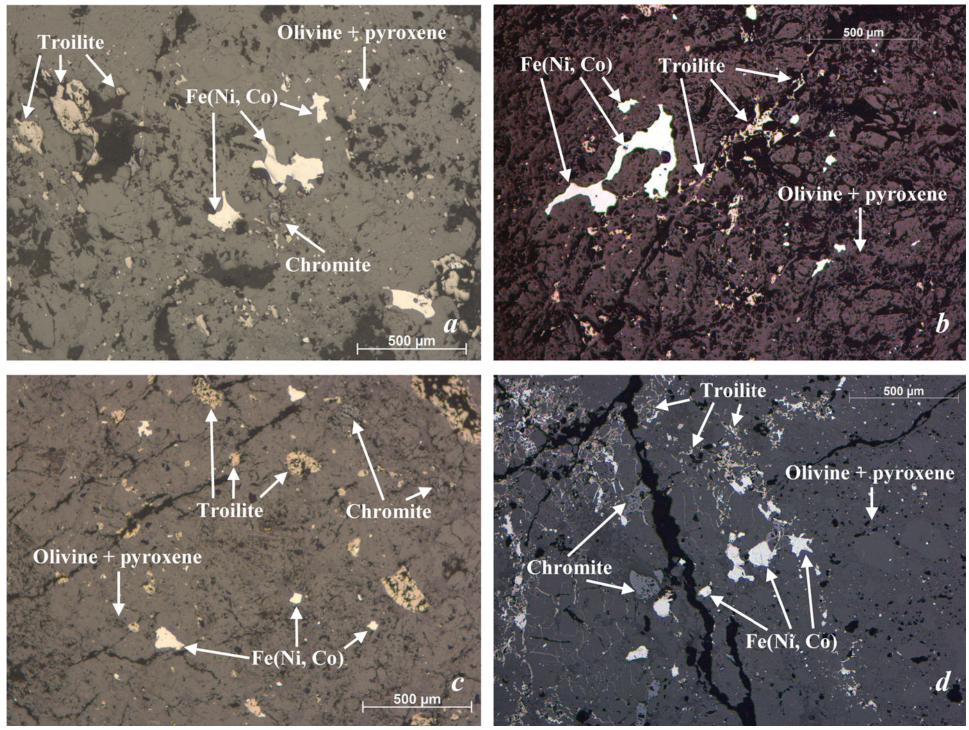

Fig. 1 Microphotographs of the slices of Chelyabinsk LL5 meteorite fragments with different lithology: No. 1, light lithology (**a**), No. 1a, light lithology (**b**), No. 2, mixed light and dark lithology (**c**), No. 3, black lithology (**d**)

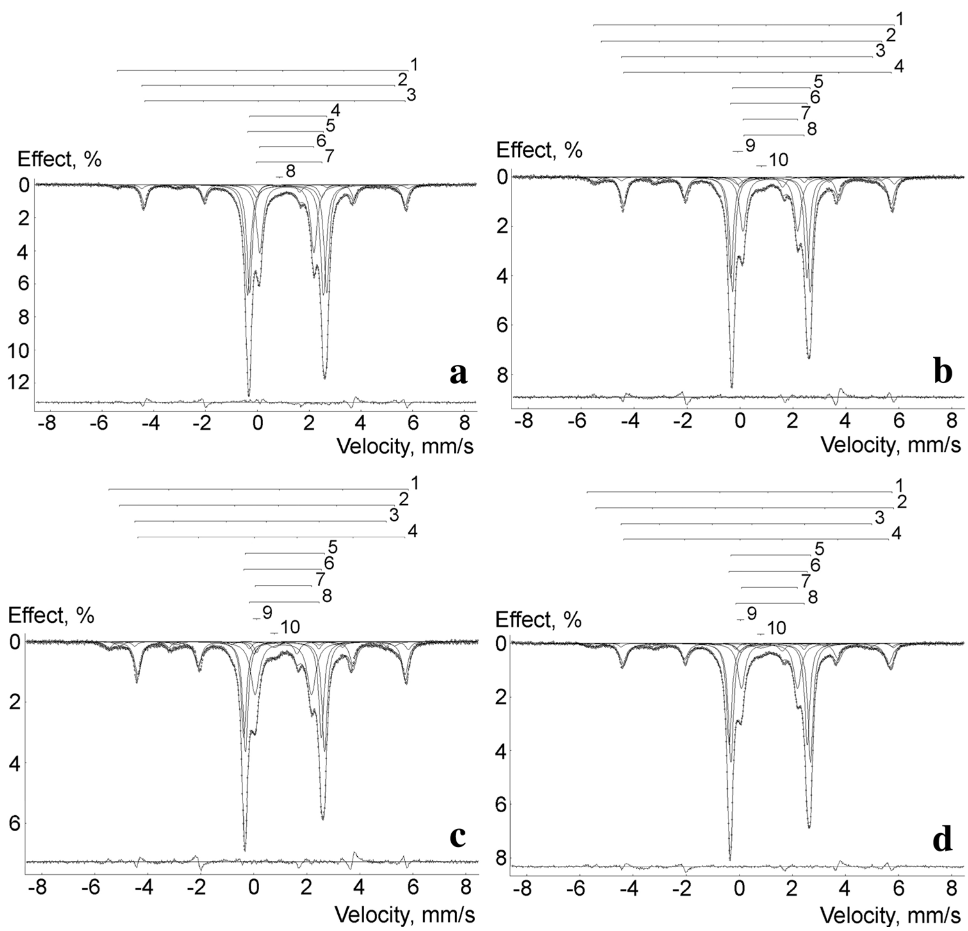

Fig. 2 Mössbauer spectra of Chelyabinsk LL5 meteorite fragments with different lithology measured with a high velocity resolution at room temperature: No. 1, light lithology (**a**), No. 1a, light lithology (**b**), No. 2, mixed light and dark lithology (**c**), No. 3, black lithology (**d**). Indicated components are the results of the best fits using UNIVEM-MS code. The differential spectra are shown below

Table 1 Components of the Mössbauer spectra of Chelyabinsk LL5 meteorite fragments with different lithology and their relative areas

Chelyabinsk LL5 meteorite fragment	No of component[a]	Phase/mineral	*A* (%)
No. 1, light lithology	1	α-Fe(Ni, Co)	2.57
	2	γ-Fe(Ni, Co)	2.16
	3	Troilite	11.98
	4	Olivine (M1 sites)	30.86
	5	Olivine (M2 sites)	27.62
	6	Pyroxene (M2 sites)	20.70
	7	Pyroxene (M1 sites)	3.76
	8	Chromite	0.34
No. 1a, light lithology	1	α-Fe(Ni, Co)	4.09
	2	α-Fe(Ni, Co)	1.68
	3	γ-Fe(Ni, Co)	1.93
	4	Troilite	18.51
	5	Olivine (M1 sites)	28.12
	6	Olivine (M2 sites)	24.88
	7	Pyroxene (M2 sites)	17.28
	8	Pyroxene (M1 sites)	1.24
	9	Paramagnetic γ-Fe(Ni, Co)	1.62
	10	Chromite	0.65
No. 2, mixed light and dark lithology	1	α-Fe(Ni, Co)	4.15
	2	γ-Fe(Ni, Co)	2.87
	3	γ-Fe(Ni, Co)	2.15
	4	Troilite	19.96
	5	Olivine (M1 sites)	24.94
	6	Olivine (M2 sites)	21.83
	7	Pyroxene (M2 sites)	19.87
	8	Pyroxene (M1 sites)	1.63
	9	Paramagnetic γ-Fe(Ni, Co)	1.24
	10	Chromite	1.36
No. 3, black lithology	1	α-Fe(Ni, Co)	1.70
	2	α-Fe(Ni, Co)	2.32
	3	γ-Fe(Ni, Co)	1.86
	4	Troilite	16.52
	5	Olivine (M1 sites)	30.20
	6	Olivine (M2 sites)	25.79
	7	Pyroxene (M2 sites)	16.29
	8	Pyroxene (M1 sites)	1.52
	9	Paramagnetic γ-Fe(Ni, Co)	1.82
	10	Chromite	1.98

[a] Component numbers correspond to the numbers of component in Fig. 2

studies of synthetic and meteoritic olivines [16, 30] as well as synthetic pyroxene [31] with varied composition of Fe^{2+} and Mg^{2+} the small differences in the values of quadrupole splitting were observed also (for instance, for various olivines quadrupole splitting varied from ~2.84 mm/s till ~2.99 mm/s in the corresponding fayalite range from 100 down to 10 [30]). However, taken into account different accuracy of the present Mössbauer spectrometric system with a high velocity resolution and other conventional spectrometers with a low velocity resolution, it is not possible to do direct comparison of the present and earlier observed differences. Nevertheless, on the basis of previous results [16, 30, 31] we can conclude that the present small differences in quadrupole splitting for both olivine and pyroxene in different Chelyabinsk LL5 fragments may be also related to small variations of the Fe^{2+} and Mg^{2+} partitioning in both silicates (in spite of fayalite and ferrosilite for various fragments of Chelyabinsk LL5

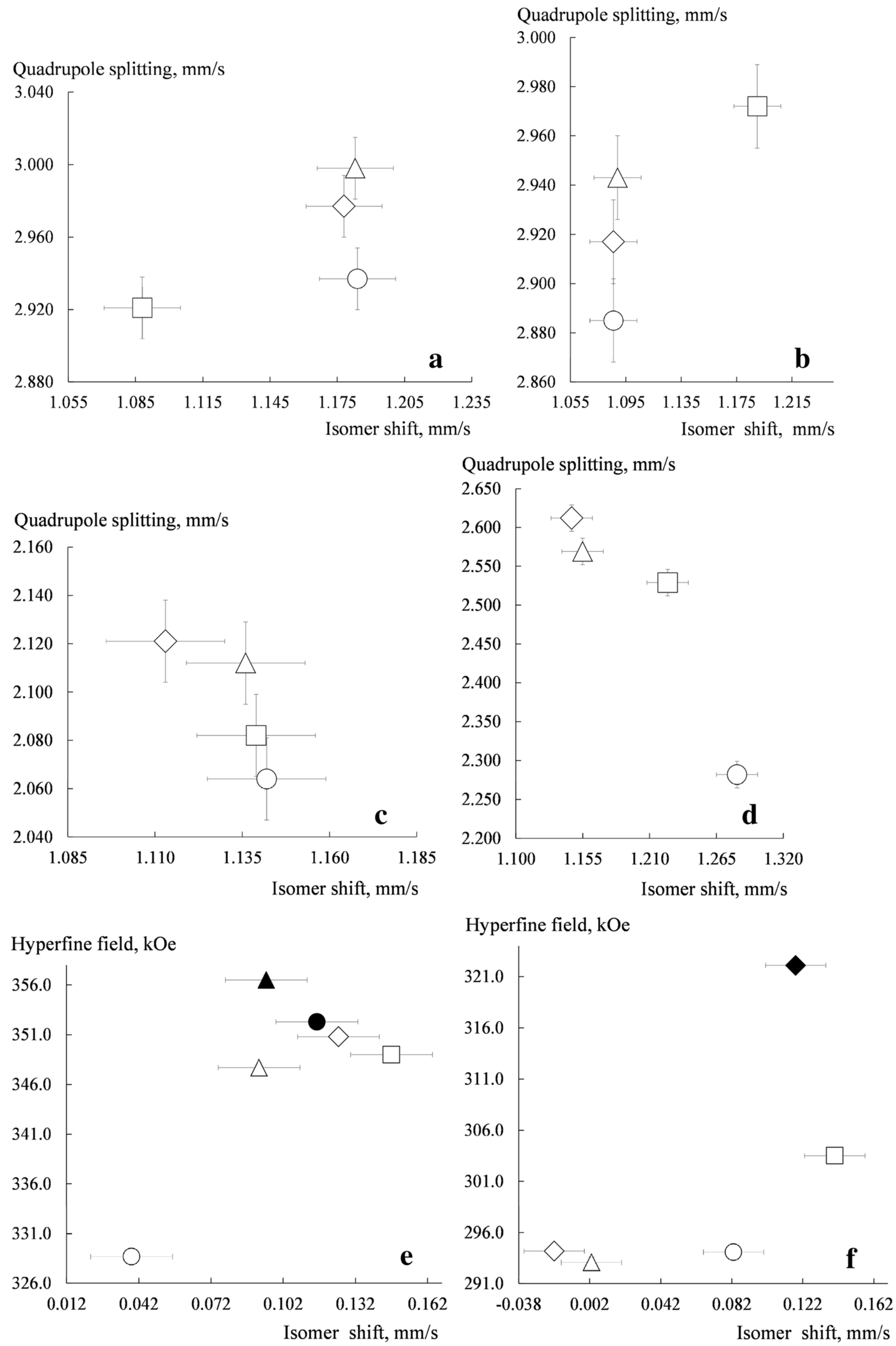

Fig. 3 The differences of the hyperfine parameters for the components in the Mössbauer spectra of Chelyabinsk LL5 fragments with different lithology: No. 1, light lithology (*open square*), No. 1a, light lithology (*open circle*, *filled circle*), No. 2, mixed light and dark lithology (*open diamond*, *filled diamond*), No. 3, black lithology (*open triangle*, *filled triangle*); M1 sites in olivine (**a**), M2 sites in olivine (**b**), M2 sites in pyroxene (**c**), M1 sites in pyroxene (**d**), kamacite and/or martensite (**e**), taenite (**f**)

meteorite were determined by chemical analysis as ∼28 and ∼23, respectively [32]). The observed differences in the hyperfine parameters for kamacite and taenite can be related to small variations in Ni and Co concentrations in these phases in different fragments of Chelyabinsk LL5 meteorite. Earlier it was shown that the average hyperfine field at the ^{57}Fe versus Ni concentration in the range between 0 and 25 at.% for α-Fe(Ni) varied from ∼330 till ∼340 kOe while that versus Co concentration in the range between 0 and 30 at.% for α-Fe(Co) varied from ∼330 till ∼370 kOe [33]. In spite of the authors determined the average values of hyperfine field and used the low velocity resolution Mössbauer spectroscopy these data may be used to indicate the possibility of the hyperfine field variation

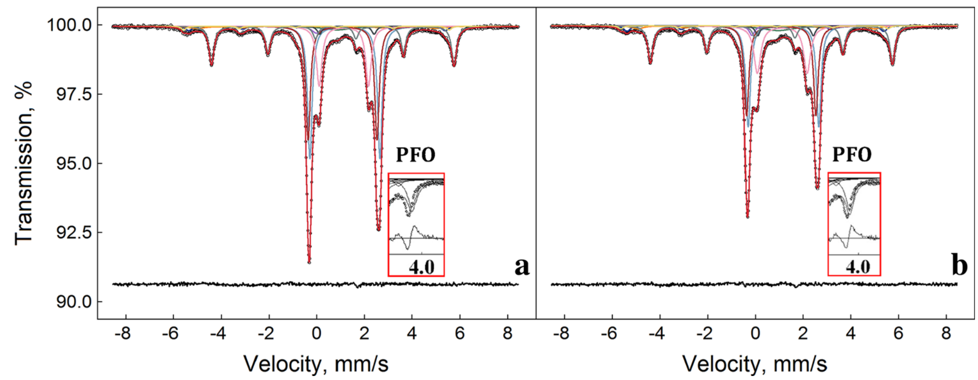

Fig. 4 Mössbauer spectra of Chelyabinsk LL5 meteorite fragments fitted using MossWinn code with the full static Hamiltonian approximation applied for troilite subspectrum: No. 1a, light lithology (**a**), No. 2, mixed light and dark lithology (**b**). The differential spectra are shown below

Table 2 Mössbauer parameters of troilite in Chelyabinsk LL5 meteorite fragments with a light lithology (No. 1a) and mixed light and dark lithology (No. 2) obtained from the fit of the corresponding spectra with the MossWinn program by accounting for the troilite component with the consideration of the full static Hamiltonian in the ground and excited nuclear states of the corresponding ^{57}Fe nuclides

Fragment	No. 1a	No. 2
A (%)	18.0 ± 0.2	19.8 ± 0.2
δ (mm/s)	0.736 ± 0.017	0.747 ± 0.017
H_{eff} (kOe)	311.5 ± 0.5	311.4 ± 0.5
V_{zz} 10^{21} (V/m^2)	−5.13 ± 0.04	−4.81 ± 0.04
η	0.3[a]	0.3[a]
β (°)	48.2 ± 0.1	47.1 ± 0.1
α (°)	49[a]	49[a]
Γ	0.251 ± 0.034	0.257 ± 0.034
S_0 (mm/s)	2.12 ± 0.02	1.99 ± 0.02
S_1 (mm/s)	−0.263 ± 0.005	−0.296 ± 0.005
S_2 (mm/s)	1.33 ± 0.01	1.26 ± 0.01

[a] Fixed parameter

versus Ni and/or Co concentration, however, it takes to carry out new measurements using the high velocity resolution Mössbauer spectroscopy for clarification. For instance, recent results of the high velocity resolution Mössbauer spectroscopy of several iron meteorites with different Ni concentration [34] demonstrated the possibility to reveal several magnetic components with larger differences of the values of hyperfine field related to different Ni concentrations than those in [33] while correlated with the present results for metallic iron grains in Chelyabinsk LL5 fragments.

It should be pointed out that fitting of the Mössbauer spectra using UNIVEM-MS code with the perturbation of the first order approximation (PFO) for magnetic components did not allow to fit troilite subspectrum correctly. It takes to use the full static Hamiltonian (FH) to fit this subspectrum correctly (see [21] and references therein). Therefore, additionally the Mössbauer spectra of Chelyabinsk LL5 meteorite fragments No. 1a and No. 2 were fitted with this approach by using the MossWinn program. The results of these fits are shown in Fig. 4. The obtained corresponding parameters of troilite in fragments No. 1a and No. 2 are given in Table 2.

It was found that the values of V_{zz}, angle β and invariants S_0, S_1 and S_2 were different for troilite crystals in these fragments that may be a result of small structural variations and different degree of non-stoichiometry. Owing to the possible changes of parameters of other subspectra due to more correct fitting of the troilite subspectrum by FH, a comparison of Mössbauer parameters were done by given an example of two fits using UNIVEM-MS and MossWinn codes for fragment No. 2. The results of this comparison are given in Table 3. It is interesting to point out that the values of parameters for olivine (M1 and M2) and pyroxene (M2) appeared to be the same for both fits while parameters for other minor components were different probably due to stronger correlation of parameters.

Conclusions

A comparative study of four Chelyabinsk LL5 meteorite fragments with different lithology using Mössbauer spectroscopy with a high velocity resolution revealed spectral components corresponding to crystals of the main iron-bearing minerals such as olivine, pyroxene, troilite, kamacite, taenite and chromite in these fragments. However, the iron content in these minerals appeared to be different in four fragments. Small variations were found in the hyperfine parameters of the Mössbauer spectral components corresponding to ^{57}Fe nuclei in the M1 and M2 sites in both olivine and pyroxene, in troilite, kamacite and taenite for different fragments. This result may be related to small microstructural variations in relevant crystals of silicates and troilite as well as to variations in Ni and Co concentrations in the metal grains. These differences could be a result of the breccia structure of Chelyabinsk LL5

Table 3 Comparison of the results of two fits of the Mössbauer spectrum of Chelyabinsk LL5 meteorite fragment with mixed light and dark lithology (No. 2) using the full static Hamiltonian (FH) and its first order approximation (PFO) applied to troilite subspectrum (using MossWinn and UNIVEM-MS codes, respectively)

	Γ (mm/s)	δ (mm/s)	2ε/ΔE_Q (mm/s)	H_{eff} (kOe)	*A* (%)	Phase/mineral
PFO	0.261 ± 0.034	0.125 ± 0.017	0.108 ± 0.017	350.8 ± 0.5	4.15	α_2-Fe(Ni, Co)
FH	0.230[a]	−0.015 ± 0.017	−0.048 ± 0.019	348.3 ± 0.5	1.46	
PFO	0.338 ± 0.035	0.118 ± 0.017	0.014 ± 0.017	322.1 ± 0.7	2.87	α-Fe(Ni, Co)
FH	0.321 ± 0.034	0.009 ± 0.017	−0.017 ± 0.017	334.8 ± 0.5	3.77	
PFO	0.233 ± 0.034	−0.018 ± 0.017	0.529 ± 0.017	294.2 ± 0.5	2.15	γ-Fe(Ni, Co)
FH	0.527 ± 0.063	−0.027 ± 0.017	0.136 ± 0.031	308.8 ± 0.5	3.50	
PFO	0.307 ± 0.034	0.745 ± 0.017	−0.161 ± 0.017	312.6 ± 0.5	19.96	Troilite[b]
FH	0.257 ± 0.034	0.747 ± 0.017	−0.812 ± 0.017	311.4 ± 0.5	19.81	
PFO	0.233 ± 0.034	1.178 ± 0.017	2.976 ± 0.017	–	24.94	Olivine (M1 sites)
FH	0.230[a]	1.178 ± 0.017	2.973 ± 0.017	–	24.61	
PFO	0.233 ± 0.034	1.086 ± 0.017	2.917 ± 0.017	–	21.83	Olivine (M2 sites)
FH	0.230[a]	1.084 ± 0.017	2.916 ± 0.017	–	21.91	
PFO	0.388 ± 0.034	1.113 ± 0.017	2.121 ± 0.017	–	19.87	Pyroxene (M2 sites)
FH	0.343 ± 0.034	1.125 ± 0.017	2.097 ± 0.017	–	17.17	
PFO	0.233 ± 0.034	1.146 ± 0.017	2.612 ± 0.021	–	1.63	Pyroxene (M1 sites)
FH	0.230[a]	1.263 ± 0.017	2.340 ± 0.019	–	2.39	
PFO	0.233 ± 0.034	0.115 ± 0.017	–	–	1.24	Paramagnetic γ-Fe(Ni, Co)
FH	0.269 ± 0.034	−0.149 ± 0.017	–	–	1.35	
PFO	0.481 ± 0.048	0.780 ± 0.017	–	–	1.36	Chromite
FH	1.579 ± 0.171	0.956 ± 0.041	–	–	4.02	

[a] Fixed parameter

[b] For PFO value of 2ε and for FH value of ΔE_Q are given

meteorite formed in the space by reiterated collisions of the parent bodies. Results of a more accurate fit of the troilite component using the full static Hamiltonian showed different EFG for troilite in two fragments with different lithology.

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