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Study Of Olivines From Omolon And Seymchan Meteorites Using X-Ray Diffraction And Mössbauer Spectroscopy With A High Velocity Resolution

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Abstract. Study of olivine from Omolon and Seymchan meteorites was performed using X-ray diffraction and Mössbauer spectroscopy with a high velocity resolution. X-ray diffraction patterns were measured at room temperature while Mössbauer spectra were measured at 295 and 90 K. The orthorhombic crystal lattice parameters were evaluated for olivine from Omolon and Seymchan. These parameters appeared to be different for olivines from both meteorites. Mössbauer spectral components related to ^{57}Fe in crystallographically non-equivalent sites M1 and M2 in both olivines were determined and its Mössbauer hyperfine parameters were evaluated. Some differences in the tendencies of temperature dependence of spectral parameters and small variations of ^{57}Fe quadrupole splitting in both M1 and M2 sites of olivines from Omolon and Seymchan were found. On the basis of Mössbauer parameters and chemical data, the temperatures of equilibrium cation distribution were evaluated for both olivines.

Keywords: Mössbauer spectroscopy, olivine, pallasites, hyperfine parameters, ^{57}Fe in M1 and M2 sites.
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INTRODUCTION

Olivine $(Fe,Mg)_2SiO_4$ is a mineral widely distributed in terrestrial and extraterrestrial matter. Olivine is considered as a solid solution of forsterite (Mg_2SiO_4) and fayalite (Fe_2SiO_4). The olivine crystal structure demonstrates the presence of two crystallographically non-equivalent six-fold octahedral sites for Fe^{2+} and Mg^{2+} denoted as M1 and M2. Recently, a brief description of olivine structure was carried out by Dyar et al. [1]. These olivine sites are occupied by Fe^{2+} and Mg^{2+} ions in different ways. The Fe^{2+}–Mg^{2+} distribution between the two sites is of interest for mineral cooling history determination. Mössbauer spectroscopy was successfully used to distinguish M1 and M2 sites in pure synthetic olivines [1–3]. Recently, we

demonstrated new possibilities of Mössbauer spectroscopy with a high velocity resolution in the study of ordinary chondrites and other meteorites [4–6]. In particular, the differences of hyperfine parameters for ^{57}Fe in the M1 and M2 sites of olivines in different bulk ordinary chondrites were respectively revealed for the first time. Moreover, it was shown the possibility to evaluate the temperature of equilibrium cation distribution for olivines in ordinary chondrites, which were not shocked and reheated, on the basis of Mössbauer spectroscopy parameters. However, it would be better to study extracted meteoritic phases to increase the accuracy of obtaining data. Therefore, in the present work, we discuss results of the study of two olivines extracted from Omolon and Seymchan pallasites, using X-ray diffraction and Mössbauer spectroscopy with a high velocity resolution at 295 and 90 K.

EXPERIMENTAL DETAILS

Omolon and Seymchan are the main-group pallasites found in Magadan Region, Russian Federation. Olivine was mechanically extracted from Omolon and Seymchan fragments (see Fig. 1a and Fig. 1b; their sections are shown in Fig. 1c and Fig. 1d) and powdered, removing the remains of magnetic fraction with magnet. Olivine powder was glued on an aluminum foil, free from iron, with a diameter of 2 cm. Olivine samples had an effective thickness of ~6–8 mg Fe/cm^2 for Mössbauer study.
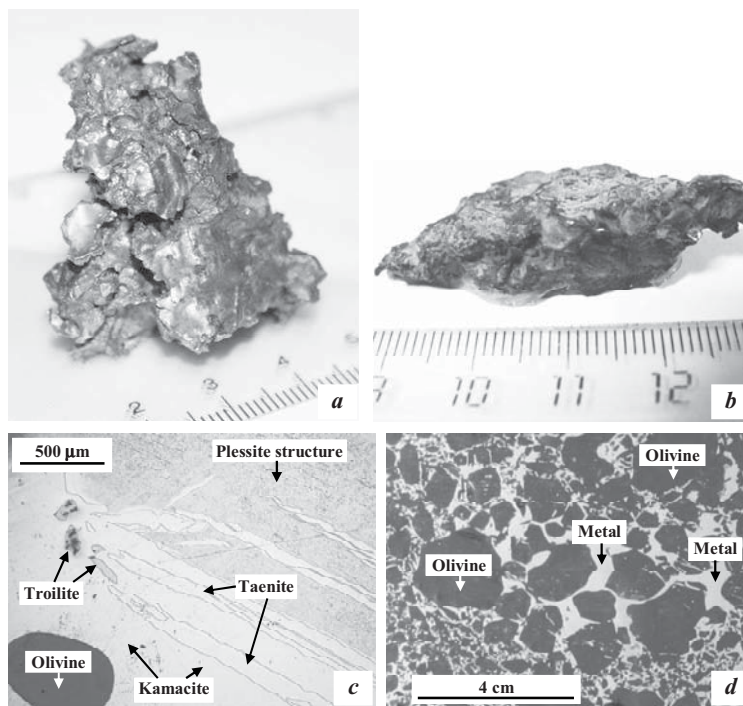

FIGURE 1. Fragments of (a) Omolon and (b) Seymchan pallasites and sections of (c) Omolon and (d) Seymchan pallasites.

X-ray diffraction (XRD) patterns were measured using an X'Pert PRO MRD diffractometer in vertical goniometry with CuK_α radiation in the Bragg-Brentano

geometry and β-filter (Ni) in the secondary beam. A solid-state PIXcel detector was used for recording the XRD patterns with an active length of 3.347°. Measurements were carried out at room temperature with 2Θ varying in the range from 16 to 110° with a step of 0.0066°. The total counting time per one step was 333 s. Firstly, the XRD patterns were measured for olivine samples prepared for Mössbauer measurements; then, the additional thin powder olivine sample from Omolon was prepared to exclude the effect of texture of larger size olivine particles in other samples. The thin powder olivine sample was measured using rotation in a horizontal plane at 0.5 rev/s. A Panalitical X'Pert High Score Plus (version 2.2c) computer program was used for XRD patterns fitting. The XRD data were further fitted with the least square procedure using the program of Rietveld full profile refinements and PDF-2 database. This Rietveld implementation is based on the source code of the LHPM program [7].

Mössbauer spectra were measured using an automated precision Mössbauer spectrometric system built on the base of an SM–2201 spectrometer with a high velocity resolution. Details and characteristics of this equipment and the system were given elsewhere [8–10]. The Mössbauer spectra were measured in transmission geometry with moving absorber in the cryostat at 295 and 90 K and recorded in 4096 channels. Spectra were measured in a velocity range of about ± 4 mm/s with a statistical rate ranging from $\sim 1.9 \times 10^5$ to $\sim 2.4 \times 10^5$ counts per channel and the signal-to-noise ratio varying from 63 to 74. The spectra were computer fitted with the least squares procedure using the UNIVEM-MS program with a Lorentzian line shape. The spectral parameters such as isomer shift (δ), quadrupole splitting (ΔE_Q), linewidth (Γ), relative subspectrum area (S), and statistical quality of the fit (χ^2) were determined. An instrumental (systematic) error for each spectrum point was ± 0.5 channel (the velocity scale), the instrumental (systematic) error for the hyperfine parameters was ± 1 channel while the instrumental (systematic) error for Γ evaluation was ± 2 channels. It should be noticed that spectrometer characteristics determined an integral velocity error (total mechanical and electronics systematic and random errors) which was several times smaller than a half of channel value in mm/s during spectra measurements using 4096 channels [8,10]. If an error calculated with the fitting procedure (fitting error) for these parameters exceeded the instrumental (systematic) error, we used the larger error instead. The velocity resolution (velocity per one channel) was ~0.002 mm/s per channel. The relative error for S did not exceed 10%. Differential spectra, χ^2 values and physical meaning of the spectral parameters were used as criteria for the best fit. Value of the standard deviation (σ) of χ^2 was 0.022 for 4096 channels spectra. Values of δ are given relative to α-Fe at 295 K.

RESULTS AND DISCUSSION

X-Ray Diffraction

XRD patterns of olivine samples from Omolon and Seymchan pallasites prepared for Mössbauer spectroscopy are shown in Fig. 2. The sample of olivine from Omolon contains a mixture of three phases: the main phase of about 94 wt.% of (Mg$_{1-}$

$_x$Fe$_x$)$_2$SiO$_4$, about 2 wt.% of FeS and about 4 wt.% of SiO$_2$. The sample of olivine from Seymchan contains two phases: the main phase of about 97 wt.% of (Mg$_{1-x}$Fe$_x$)$_2$SiO$_4$ and about 3 wt.% of SiO$_2$. Both olivines have orthorhombic crystal lattice with parameters determined as follows: a = 10.2337(7) Å, b = 5.9969(5) Å, and c = 4.7641(3) Å for olivine from Omolon and a = 10.2267(7) Å, b = 5.9939(4) Å, and c = 4.7623(3) Å for olivine from Seymchan. It is interesting that crystal lattice parameters a, b, and c appeared to be different for both olivines. This may be a result of different iron content in olivines from Omolon and Seymchan pallasites as well as different meteorites formation processes.
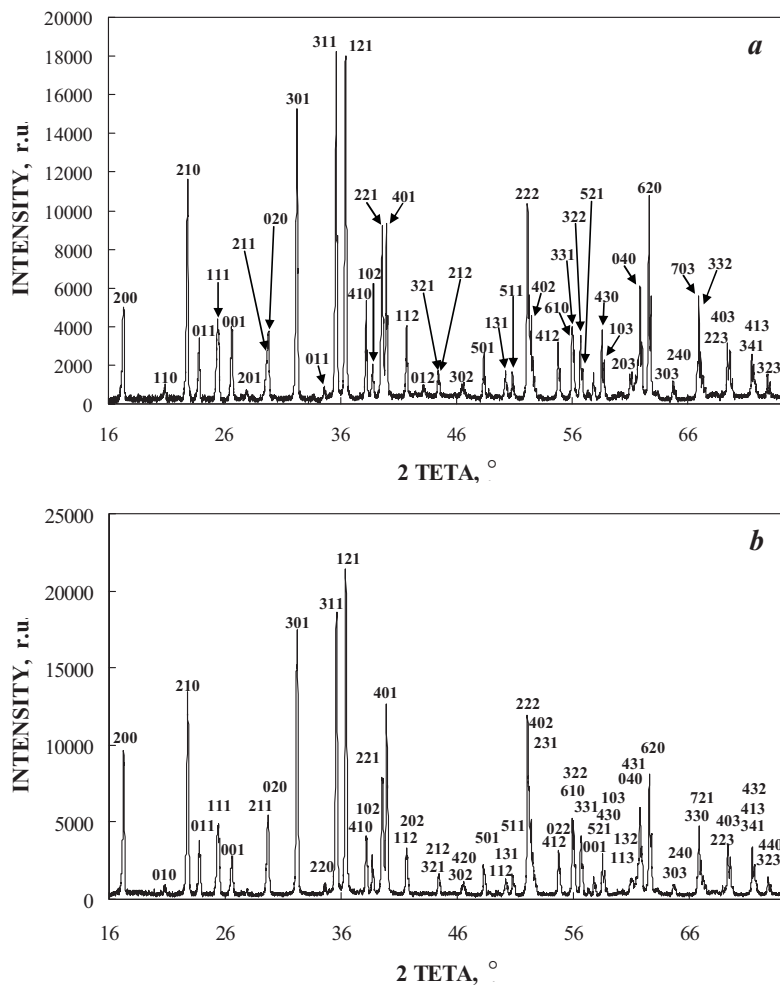

FIGURE 2. XRD patterns of olivine samples extracted from (a) Omolon and (b) Seymchan pallasites and prepared for Mössbauer measurements.

Mössbauer Spectroscopy

Mössbauer spectra of olivine extracted from Omolon and Seymchan meteorites measured at 295 and 90 K in 4096 channels are shown in Fig. 3. These spectra consist of asymmetrical doublets with inverse asymmetry of peak intensities at 295 and 90 K.
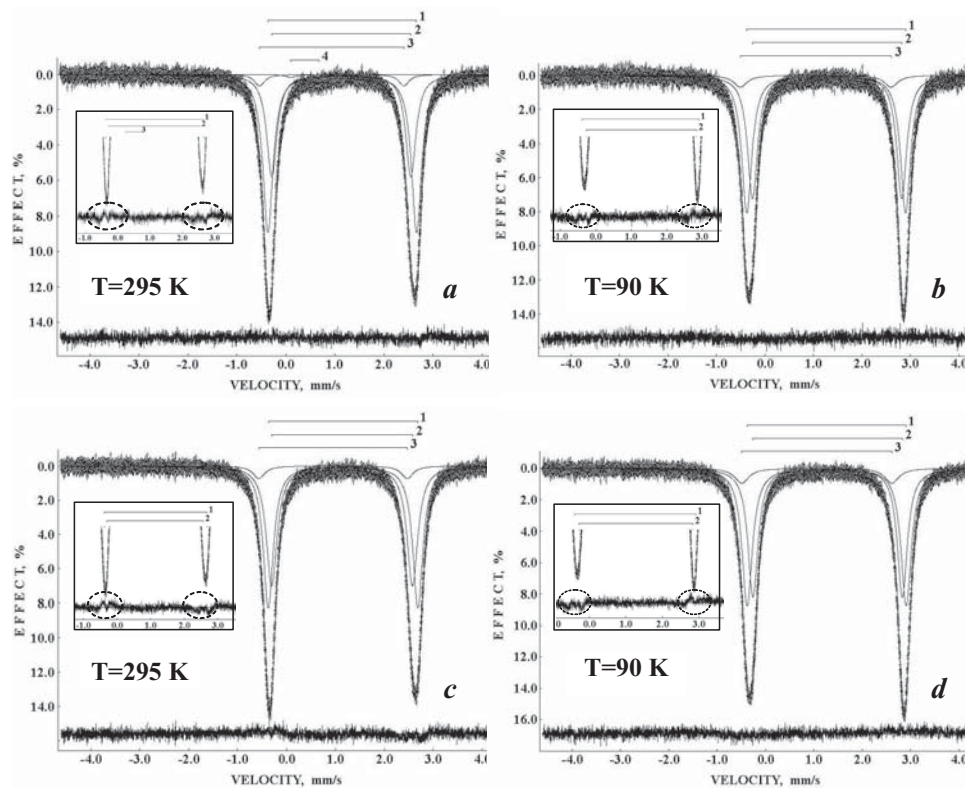
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FIGURE 3. Mössbauer spectra of olivines measured with a high velocity resolution (in 4096 channels) at 295 and 90 K: (a, b) olivine extracted from Omolon and (c, d) Seymchan pallasites. Indicated components are the result of the best fit (see text). Differential spectra are shown below.

It should be noted that in the spectra of olivine from Omolon, we did not observed component related to FeS that was detected by XRD. However, in the preliminary study of olivine from Omolon, we detected a weak magnetic sextet (S = 2.3%) with hyperfine parameters corresponding to troilite in the room-temperature spectrum measured in a large velocity range [11]. However, intensities of internal sextet lines were within the spectral noise. Therefore, we neglected this magnetic component in the spectra measured in a small velocity range. The best fit of the room-temperature spectrum of olivine from Omolon demonstrated the presence of 4 components related to three ferrous (1–3) and one ferric (4) compounds (the Omolon spectrum measured in a large velocity range at room temperature and shown in [11] demonstrated also the same ferric compound). The values of χ^2 for this spectrum with different fits were 1.541 (1 doublet fit, deviation of χ^2 was 24.5 σ), 1.078 (2 doublets fit, deviation of χ^2 was 3.5 σ), 1.067 (3 doublets fit, deviation of χ^2 was 3.0 σ), and 0.975 (4 doublets fit, deviation of χ^2 was 1.1 σ). In these fits, the differential spectra clearly indicated the necessity of 4 doublets for the best fit (see comparison with differential spectrum for 3 doublets fit in Fig. 3a). 90 K Mössbauer spectrum of olivine from Omolon was better fitted using 3 components related to three ferrous compounds (1–3) only. It is possible that the minor ferric paramagnetic component after cooling down to 90 K transferred to magnetically split component with intensities of internal sextet lines within the spectral noise; this is why we did not found this component in a small velocity range. The values of χ^2 for 90 K spectrum with different fits were 1.444 (1 doublet fit,

deviation of χ^2 was 20.2 σ), 1.100 (2 doublets fit, deviation of χ^2 was 4.5 σ), 1.020 (3 doublets fit, deviation of χ^2 was 0.9 σ). The differential spectra clearly indicated the necessity of 3 doublets for the best fit in comparison with 2 doublets fit (see Fig. 3b).

The better fit of the room-temperature spectrum of olivine from Seymchan demonstrated the presence of 3 components related to ferrous compounds (1–3). There was no ferric compound detected in this spectrum in comparison with that for olivine from Omolon. The values of χ^2 for this spectrum with different fits were 1.637 (1 doublet fit, deviation of χ^2 was 29.0 σ), 1.208 (2 doublets fit, deviation of χ^2 was 9.5 σ) and 1.106 (3 doublets fit, deviation of χ^2 was 4.8 σ). In these fits, the differential spectra clearly indicated the necessity of 3 doublets for the better fit (see comparison with differential spectrum for the 2 doublets fit in Fig. 3c). 90 K Mössbauer spectrum of olivine from Seymchan was better fitted also using 3 components related to 3 ferrous compounds (1–3). The values of χ^2 for 90 K spectrum with different fits were 1.684 (1 doublet fit, deviation of χ^2 was 31.1 σ), 1.214 (2 doublets fit, deviation of χ^2 was 9.7 σ), 1.058 (3 doublets fit, deviation of χ^2 was 2.6 σ). The differential spectra clearly indicated the necessity of 3 doublets for the better fit in comparison with 2 doublets fit (see Fig. 3d). However, we cannot consider 3 doublets fit of Mössbauer spectra of olivine from Seymchan as the best fit. We chose 3 doublets fit because the fit using 4 doublets demonstrated very different parameters for the 3rd and the 4th components in 295 and 90 K spectra in spite of smaller standard deviation of χ^2 (1.1 σ and 1.7 σ, respectively). Taking into account the lack of physical meaning for unusual variation of Mössbauer parameters for the 3rd and the 4th components with temperature decrease, we exclude this fit from consideration. Parameters of Mössbauer spectra of olivine from both Omolon and Seymchan meteorites obtained from the best fits are summarized in Table 1.

TABLE 1. Mössbauer parameters of olivines from Omolon and Seymchan meteorites.

Sample	T (K)	Γ (mm/s)	δ (mm/s)	ΔE_Q (mm/s)	S (%)	Component[a]
Olivine from Omolon	295	0.252 ± 0.004	1.156 ± 0.002	3.020 ± 0.002	51	M1 (1)
		0.312 ± 0.004	1.117 ± 0.002	2.849 ± 0.015	43	M2 (2)
		0.253 ± 0.025	1.007 ± 0.010	3.108 ± 0.022	5	X (3)
		0.266 ± 0.046	0.389 ± 0.018	0.570 ± 0.034	1	Fe^{3+} (4)
	90	0.254 ± 0.004	1.264 ± 0.002	3.275 ± 0.002	50	M1 (1)
		0.252 ± 0.004	1.284 ± 0.002	3.076 ± 0.002	44	M2 (2)
		0.334 ± 0.026	1.047 ± 0.012	3.105 ± 0.011	6	X (3)
Olivine from Seymchan	295	0.252 ± 0.004	1.158 ± 0.002	3.058 ± 0.002	50	M1 (1)
		0.269 ± 0.004	1.137 ± 0.002	2.876 ± 0.002	45	M2 (2)
		0.292 ± 0.029	0.952 ± 0.010	3.032 ± 0.010	5	X (3)
	90	0.262 ± 0.004	1.266 ± 0.002	3.287 ± 0.002	49	M1 (1)
		0.252 ± 0.004	1.286 ± 0.002	3.088 ± 0.002	44	M2 (2)
		0.354 ± 0.019	1.061 ± 0.010	3.103 ± 0.009	7	X (3)

[a] Numbers in parenthesis correspond to the spectral components in Fig. 3.

The tendencies of Mössbauer parameters temperature dependencies for ferrous compounds are shown in Fig. 4. Based on our previous data for olivine study [4–6,11–13], we related the component 1 with the larger ΔE_Q and S values to ^{57}Fe in the M1 sites while the component 2 with the smaller ΔE_Q and S values was related to ^{57}Fe in the M2 sites in olivine. The component 3 had parameters which we cannot relate to any impurity. This suggests that this component may be a result of some sites

distortion in olivine which was not previously observed. We denoted these unknown sites as X. It was interesting to observe an increase in δ for ^{57}Fe in both M1 and M2 sites with temperature decrease (see Fig. 4). This fact can be explained as a result of the second order Doppler shift. Recently, similar dependence was observed for synthetic olivines [1]. Moreover, a crossing of the tendencies of δ temperature dependences for ^{57}Fe in the M1 and M2 sites indicated that Debye temperature for ^{57}Fe in the M1 and M2 sites was different, i.e., dynamics of ^{57}Fe nuclei in these sites was different. The values of ΔE_Q for ^{57}Fe in both M1 and M2 sites increased with temperature decrease while S values were the same within the error (see Fig. 4). It was also interesting to observe slight differences of Mössbauer hyperfine parameters for ^{57}Fe in both M1 and M2 sites for olivines from Omolon and Seymchan at 295 and 90 K (see Fig. 5). These differences indicate small variations in the iron microenvironment in both M1 and M2 sites of olivines in Omolon and Seymchan meteorites. This result is in agreement with XRD data showing that the crystal lattice parameters are different for both olivines.
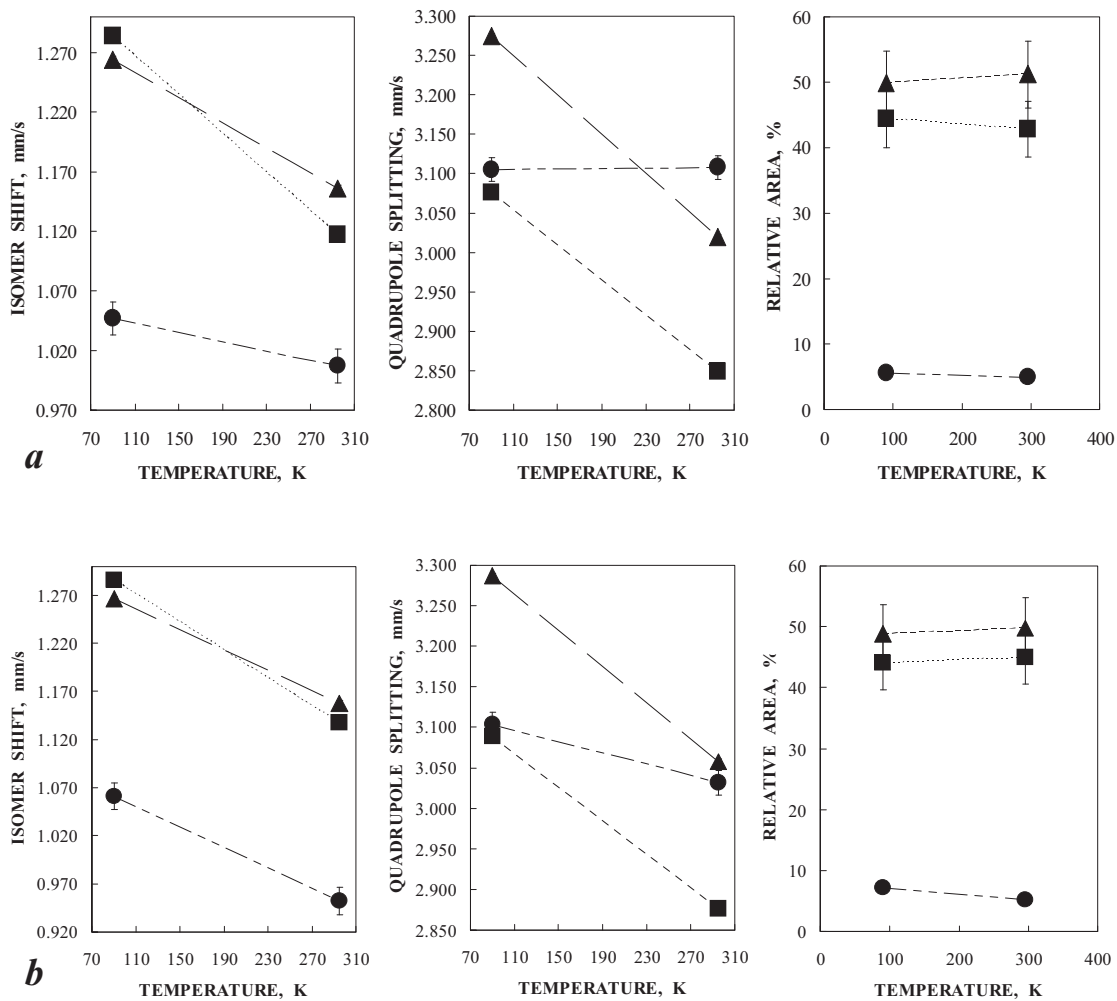

FIGURE 4. Tendencies of temperature dependences of Mössbauer parameters (isomer shift, quadrupole splitting, and relative area) for olivines extracted from (a) Omolon and (b) Seymchan pallasites: ▲ – M1 sites, ■ – M2 sites, and ● – X sites.
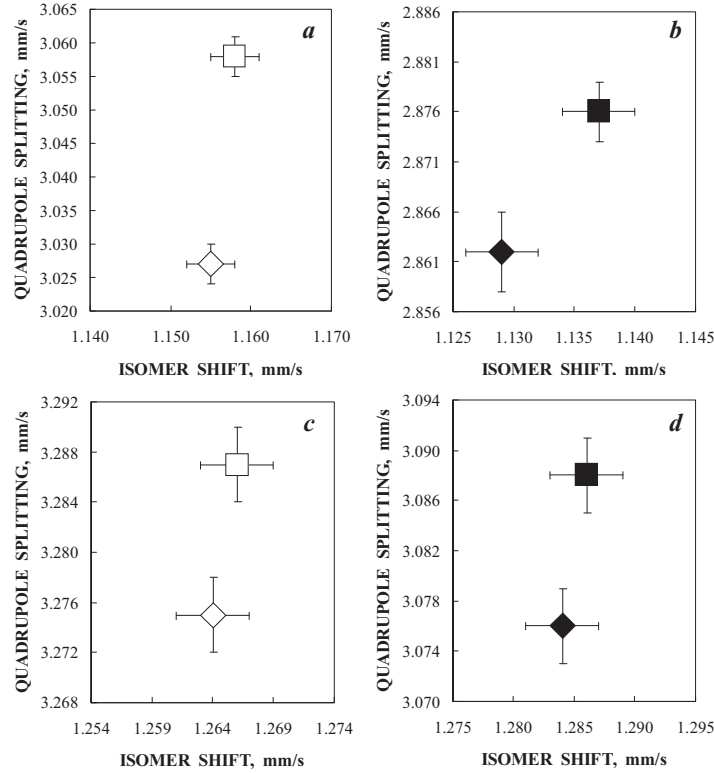
FIGURE 5. Differences of Mössbauer hyperfine parameters for ^{57}Fe in the (a, c) M1 and (b, d) M2 sites of olivines from Omolon ($\diamond$, $\blacklozenge$) and Seymchan ($\square$, $\blacksquare$) pallasites at (a, b) 295 and (c, d) 90 K.

Evaluation of the Temperature of Equilibrium Cation Distribution

We used the Rietveld method, which was recently applied for determination of cation distribution in (Co,Mg)-olivine from XRD by Müller-Sommer et al. [14], to evaluate the temperature of Fe^{2+}–Mg^{2+} equilibrium distribution in olivine from Omolon. For this evaluation, XRD data acquired for the thin powder olivine sample from Omolon were used. The Fe content $x = 0.123$ in $(Mg_{1-x}Fe_x)_2SiO_4$ olivine from Omolon (fayalite abundance) was taken from [15]. The Fe^{2+}–Mg^{2+} distribution coefficient between the M1 and M2 sites (K_D) and the temperature of equilibrium cation distribution (T_{eq}) were determined using equations (1) and (2), respectively (see [4]):

$$K_D = \frac{X_{Fe}^{M1} \cdot X_{Mg}^{M2}}{X_{Fe}^{M2} \cdot X_{Mg}^{M1}}, \tag{1}$$

$$-\Delta G = R \cdot T_{eq} \cdot \ln K_D, \tag{2}$$

where X_{Fe}^{M1} is the mole fraction of Fe^{2+} in the M1 sites, X_{Mg}^{M1} is the mole fraction of Mg^{2+} in the M1 sites, X_{Fe}^{M2} is the mole fraction of Fe^{2+} in the M2 sites, X_{Mg}^{M2} is the mole fraction of Mg^{2+} in the M2, sites ΔG is the Gibbs energy (20935 J for olivine), $R = 8.31$ $JK^{-1}mol^{-1}$. Using the Rietveld method, we evaluated the molar fractions: $X_{Fe}^{M1} = 0.140$, $X_{Mg}^{M1} = 0.860$, $X_{Fe}^{M2} = 0.106$, and $X_{Mg}^{M2} = 0.894$. The values of K_D and T_{eq} were evaluated to be equal to 1.39 and 795 K, respectively, for olivine from Omolon.

Previously, we showed that it was possible to evaluate K_D and T_{eq} using Mössbauer subspectra relative areas S and data of olivine chemical analysis (see [4]) using equations (1) and (2) as well as formulas

$$\frac{X_{Fe}^{M1}}{X_{Fe}^{M2}} = \frac{S_{M1}}{S_{M2}} = r \,, \tag{3}$$

$$X_{Mg}^{M1} = 1 - \frac{r \cdot Y_{Fe}}{1+r} \,, \tag{4}$$

$$X_{Mg}^{M2} = 1 - \frac{Y_{Fe}}{1+r} \,, \tag{5}$$

where S_{M1} and S_{M2} are the relative areas of components in Mössbauer spectra related to ^{57}Fe in the M1 and M2 sites, respectively, and $Y_{Fe} = 2x$. We tried to evaluate K_D and T_{eq} using $x = 0.123$ for olivine extracted form Omolon meteorite [15] and using $x = 0.113$ for olivine extracted from Seymchan meteorite [16] as well as S values for the components 1 and 2 obtained at both 295 and 90 K. In this case, values of T_{eq} appeared to be unrealistically large: 1549 and 1929 K for Omolon at two temperatures, respectively, and 2215 and 2198 K for Seymchan at two temperatures, respectively. Then, we suggested that the so-called component X may be a result of either M1 or M2 sites distortion. Therefore, the S value for the component 3 should be taken into account. In the case of X sites representing distorted M1 sites ($S_{M1} = S_1 + S_3$), we calculated realistic values: $K_D = 1.40$, $T_{eq} = 743$ K and $K_D = 1.29$, $T_{eq} = 997$ K using the data for Omolon obtained from 295 and 90 K Mössbauer spectra, respectively, as well as $K_D = 1.25$, $T_{eq} = 1118$ K and $K_D = 1.31$, $T_{eq} = 938$ K using the data for Seymchan obtained from 295 and 90 K Mössbauer spectra, respectively. In other case, the values of T_{eq} were unrealistic again. It was interesting that the values of T_{eq}, obtained for olivine from Omolon using the room-temperature XRD and Mössbauer data, appeared to be similar (795 and 743 K, respectively). Therefore, we can denote X sites as, for instance, M1a. It should be noticed that taking into account different Debye temperatures and ^{57}Fe nuclei dynamics in the M1, M2, and M1a sites, the values of K_D and T_{eq}, evaluated from 90 K Mössbauer spectrum parameters, seems to be more realistic. Therefore, we can conclude that the most reliable values of temperatures of equilibrium cation distribution for olivines from Omolon and Seymchan are 997 and 938 K, respectively.

CONCLUSIONS

A comparative study of olivines extracted from Omolon and Seymchan pallasites using X-ray diffraction and Mössbauer spectroscopy with a high velocity resolution demonstrated some differences in the two olivines. The crystal lattice parameters were slightly different for these olivines that may be a result of different Fe content and processes of meteorites formation. The Mössbauer spectra measured at 295 and 90 K demonstrated the presence of two components related to ^{57}Fe in the M1 and M2 sites in olivines as well as a new unknown minor component 3 obtained from the spectra fit and related to distorted M1 sites (i.e., M1a). The tendencies of temperature dependences of Mössbauer parameters were obtained for both olivines. The behavior

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of temperature dependences of the isomer shift for ^{57}Fe in M1 and M2 sites in olivines from Omolon and Seymchan demonstrated different Debye temperatures and dynamics for ^{57}Fe in the M1 and M2 sites. Small differences of corresponding Mössbauer hyperfine parameters for ^{57}Fe in both M1 and M2 sites of the two olivines were revealed and demonstrated small variations in Fe environment in the same sites of olivines, being in agreement with XRD data. Finally, the temperatures of equilibrium cation distribution were evaluated for olivines from Omolon and Seymchan using the room-temperature XRD data and 295 and 90 K Mössbauer spectroscopic data. The low temperature evaluations seem to be more reliable due to decrease of dynamics differences for ^{57}Fe in the M1 and M2 sites.

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