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Mössbauer and infrared spectrometry of lizardite-17 from Monte Fico, Elba

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Abstract A well crystallized and homogeneous specimen of lizardite from Monte Fico, Elba, Italy, has been studied by Mössbauer and Fourier transform infrared (FTIR) spectrometries. One of the aims was the determination of the oxidation state and the distribution of iron in the structure of this reference sample. Mössbauer data indicate the presence of octahedral ferrous iron, octahedral ferric iron and tetrahedral ferric iron (59.9, 31.3 and 8.8% of total iron, respectively). The existence of only one octahedral site, previously suggested by X-ray structure refinement, is confirmed. The occurrence of tetrahedrally coordinated iron is indicated also by FTIR spectrometry, in particular by the presence of an absorption band at 790 cm⁻¹. Based also on new electron microprobe data, the improved crystal chemical formula for lizardite from Monte Fico is:

$$\begin{split} (Mg_{2.74}Fe^{2+}{}_{0.10}Fe^{3+}{}_{0.05}Al_{0.11})_{\Sigma=3.00} \\ & \cdot \ (Si_{1.94}Al_{0.05}Fe^{3+}{}_{0.01})_{\Sigma=2.00}O_{5.05}(OH)_{3.95}. \end{split}$$

Introduction

Lizardite, ideally Mg₃Si₂O₅(OH)₄, is the archetype model for the serpentine crystal structures. In recent years, specimens of lizardite good enough for X-ray diffraction single-crystal refinement have been reported (Mellini 1982; Mellini and Zanazzi 1987; Mellini and Viti 1994), and

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M. Mellini (🖂) Dip. Scienze Terra, Universita', Via delle Cerchia 3, 53100 Siena, Italy e-mail: mellini@unisi.it also aluminium-rich compositions intermediate between lizardite and amesite (Brigatti et al. 1997). In addition the lizardite structure has been studied at high pressure in the diamond anvil cell (Mellini and Zanazzi 1989), as well as at low temperature using the Rietveld refinement of neutron powder diffraction data (Gregorkiewitz et al. 1996).

Therefore, the structure of the trioctahedral T-O layer is now defined as far as the main geometrical features are concerned (octahedral and tetrahedral bond geometry, ditrigonal distortion, hydrogen bonding, layer-to-layer interaction). The studies mentioned progressively emphasized the importance of trivalent cations; in particular, the coupled substitution of two M³⁺ for M²⁺ octahedral and T⁴⁺ tetrahedral cations promotes thermal stability (Caruso and Chernosky 1979) and crystal order (Mellini and Viti 1994). The increasing layer polarization leads to increasing hydrogen bonding between adjacent layers, and overcomes the intrinsic instability otherwise determined by the two-dimensional nature of chemical bonding in the T-O layer. Hydrogen bonding is furthermore able to modify the layer configuration by increasing the ditrigonal distortion, as indicated both by the numerous experimental data and the ab-initio periodic Hartree-Fock calculations (Smrcok and Benco 1996).

It is therefore important to quantify the amount and distribution of trivalent cations (namely, aluminium and iron). This target can be reached, at least in part, by using different absorption and resonance spectroscopies. In fact, Mössbauer (e.g., Blaauw et al. 1979; Rozenson et al. 1979; Stroink et al. 1980; O'Hanley and Dyar 1993; Votyakov et al. 1993), EPR (Sharrock 1982) and IR spectroscopies (Luce 1971; Heller-Kallai et al. 1975; Serna et al. 1979; Velde 1980; Velde and Martinez 1981) have been applied to serpentine minerals long ago.

Serpentine often occurs as an association, or even a fine intergrowth, of different minerals, not only chrysotile, lizardite or antigorite, but also brucite and magnetite. Therefore, a poor crystal order and the multiphase nature of the mineral assemblage, producing contaminated data, may constitute a difficult obstacle to producing reliable

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results. An abundant occurrence of exceptionally well crystallized, homogeneous lizardite-*1T* has recently been reported and used for different studies: structural analysis (Mellini and Viti 1994; Gregorkiewitz et al. 1996), chemical, physical and microstructural determinations (Viti and Mellini 1997). We now extend the knowledge of that reference sample by supplying consistent spectroscopic data (Mössbauer and infrared). The sample comes from the Monte Fico North quarry on the Island of Elba, Italy; it occurs as a dark-green vein of recrystallized, granoblastic lizardite, occasionally embedded in a fibrous matrix of chrysotile and minor polygonal serpentine.

Chemical composition

The chemical composition of the Monte Fico lizardite (on the basis of electron microprobe, titrimetry and loss on ignition; Viti and Mellini 1997) was given as:

$(Mg_{2.79}Fe^{2+}{}_{0.09}Fe^{3+}{}_{0.07}Al_{0.07})_{\Sigma=3.02}(Si_{1.91}Al_{0.09})_{\Sigma=2.00}O_5(OH)_{3.90}.$

When proposing that formula, it was assumed that only aluminium was substituting for silicon in the tetrahedral site, and the LOI (loss on ignition) value was used to calculate the water content.

The chemical analysis has been now repeated using a Cameca SX-100 electron microprobe, operating at 15 kV, with a specimen current of 20.1 nA, fully equipped with wavelength-dispersive spectrometers using high sensitivity TAP, PET and LiF crystals. The analyses were calibrated using periclase for magnesium, albite for aluminium, olivine for silicon, and the corresponding oxydes for chromium, iron and nickel. By averaging fifteen different points, and assuming the Fe²⁺/Fe³⁺ ratio from previous titrimetry, the following formula:

$$(Mg_{2.74}Fe^{2+}_{0.09}Fe^{3+}_{0.07}Al_{0.10})_{\Sigma=3.00}(Si_{1.94}Al_{0.06})_{\Sigma=2.00}O_{5.05}(OH)_{3.95}$$

was finally achieved, with the hydroxyl content calculated to assure full oxygen occupancy. The two formulas match closely, notwithstanding the use of different microprobes; the only minor difference is constituted by less magnesium and more silicon in the more recent one. If meaningful, this small difference may either be due to the different technical procedures (e.g., thickness of carbon coating, choice of standards), or minor compositional heterogeneity in the sample.

The latter formula must be considered as provisional, as the Mössbauer analysis is expected to improve it, by supplying an independent estimate of the oxidation state of iron, as well as its actual distribution among tetrahedral and octahedral sites.

Mössbauer spectrometry

Mössbauer studies were carried out at room and low temperature (300 K, 77 K and 4.2 K, respectively), using approximately 200 mg of powdered specimen embedded in an iron free aluminium foil. Mössbauer experiment at 300 K was performed twice: first with the absorber perpendicular to the \mathbf{k} vector (direction of gamma rays) then with the absorber oriented 54° to the k vector (magic angle technique). The spectrum obtained using the magic angle technique is the very same as the one obtained with the sample oriented perpendicularly to the direction of the gamma rays, showing that texture effects can be ruled out (Figs. 1a,b). Transmission spectra (Fig. 1a-c) were recorded on a 512 channels analyzer, in a constant acceleration mode, using a source of ⁵⁷Co diffused into a Rh matrix. Folding of the data was applied, reducing the number of channels to 256. Data were calibrated using $\alpha\mbox{-}\mbox{Fe}.$ The $^{57}\mbox{Fe}$ hyperfine parameters (Table 1) were derived using the MOSFIT program (Varret 1981) and Lorentzian peak shape. Voigt-based method for fitting of



Fig. 1a–c Mössbauer spectrum of the Elba lizardite at **a** 300 K; **b** 300 K with the absorber oriented 54° to the direction of gamma-rays, **k** vector (magic angle technique); **c** 77 K (magic angle technique)

Mössbauer spectra as proposed by Rancourt and Ping (1991) was also attempted but brought no significant improvement to the fitting for this sample.

In the +1 to +2 mm/s region the fitting is not complete (Fig. 1a,b). This residual is thought to be due to poorly crystalline magnetic impurities. Actually, weak absorption bands present in the 4.2 K spectrum at a velocity of 10 mm/s indicate the occurrence of magnetic particles with tiny dimensions. Rare opaque particles were also observed in the lizardite optical thin section under the microscope.

Table 1 ⁵⁷Fe Mössbauer least-squares fitted parameters for Elba lizardite (in mm/s), with estimated standard deviation given in parentheses

T Velocity	Assignment	Lizardite 300 K 4 mm/s	Lizardite 77 K 4 mm/s
δα-Fe		-0.135(1)	-0.136(1)
$\delta^a \Delta \Gamma^b \%$	Fe ²⁺ in octahedral site	1.129(2) 2.692(3) 0.165(2) 59.9	1.366(1) 3.296(2) 0.167(2) 59.8
$\delta^a \Delta \Gamma^b \%$	Fe ³⁺ in octahedral site	0.38(1) 0.71(2) 0.236(9) 31.3	0.480(6) 0.91(1) 0.257(9) 28.9
$\delta^a \Delta \Gamma^b \%$	Fe ³⁺ in tetrahedral site	0.170(2) 0.30(3) 0.15(2) 8.8	$\begin{array}{c} 0.303(6) \\ 0.38(1) \\ 0.145(9) \\ 11.3 \end{array}$

^a Isomer shift values are relative to metallic iron at room temperature

^b Γ is the linewith at the half height of the Lorentzian line

The values of the isomer shift (δ) for the first doublet (Table 1) are consistent with Fe²⁺ and the values of the quadrupole splitting (Δ) allow as to attribute this doublet to an octahedral site. The δ values of the second and third doublets represent Fe³⁺. The ratios of the peak areas corresponding to the three doublets are 59.9, 31.3 and 8.8%, thus indicating a slight but significant dominance of ferrous iron over ferric.

On the basis of its δ and Δ values the second doublet can be attributed to Fe³⁺ iron in an octahedral site. The attribution of the third doublet to ^[4]Fe³⁺ is based on the values of δ and Δ and on the comparison of the Mössbauer spectra obtained at room and liquid N₂ temperature. The δ and Δ values obtained at room temperature agree with those of O'Hanley and Dyar (1993), i.e., δ =0.21±0.04 and Δ =0.36±0.14 mm/s. The spectra exhibit also a special feature characteristic of minerals known to contain Fe³⁺ in tetrahedral coordination (Hogarth et al. 1970, Rancourt et al. 1992): at 300 K a shoulder occurs at 0.41±0.02 mm/s with respect to α -Fe on the high energy side of the absorption peak of Fe²⁺ located at -0.1 mm/s (Fig. 1b). At 77 K it becomes sharper and moves close to 0.5 mm/s (Fig. 1c).

The present results are only in partial agreement with those of Rozenson et al. (1979), as regards the assignment of the different doublets to octahedral and tetrahedral sites. In particular, Rozenson et al. (1979) distinguished two different doublets for octahedral Fe²⁺ in two of three specimens (i.e., HU4050, lizardite from Cyprus, and HU11759, lizardite from Lizard), and concluded that lizardite has two differently distorted octahedral sites.

Our attempts to fit the spectra with more than one doublet for ferrous iron in octahedral coordination did not improve the least squares fit, and the amount of Fe^{2+} of this hypothetical second site was not higher than 2.5%. The best fit was obtained by assuming only one doublet for octahedral Fe^{2+} . A similar interpretation has been also put forward by Votyakov et al. (1993), for lizardites from the Nizhniy Tagil dunites and from the Kempirsay dunites and harz-burgites and by O'Hanley and Dyar (1993) for Canadian serpentinites.

We can therefore assume that the assignment by Rozenson et al. (1979) was likely biased by an unknown contamination in the original specimens. For instance, the chemical composition of HU4050, with (Mg,Al,Fe)_{2.78}Si_{2.06} ratios (Heller-Kallai et al. 1975), resembles antigorite more than lizardite, in agreement with the provenance from Cyprus.

The amount of ${}^{[4]}Fe^{3+}$ is moderately high in the Monte Fico lizardite. Rozenson et al. (1979) did not find ${}^{[4]}Fe^{3+}$ peaks in their sam-



Fig. 2a, b FTIR spectrum of the Elba lizardite in the range: **a** 4000–3000 cm⁻¹; **b** 1200–400 cm⁻¹

ples. However, O'Hanley and Dyar (1993) describe an elevated percentage of Fe^{3+} (up to 51% of total Fe) located in the tetrahedral site in the lizardite they studied.

A final point of this section comes from a comparison with chrysotile. In particular, the Mössbauer parameters for the Monte Fico lizardite match closely the parameters reported by Blaauw et al. (1979) for Canadian chrysotiles, and by Stroink et al. (1980) for Canadian and Rhodesian UICC standard reference chrysotile (on the average, δ and Δ values of 1.12 and 2.65, 0.34 and 0.75, 0.20 and 0.34 for ^[6]Fe²⁺, ^[6]Fe³⁺ and ^[4]Fe³⁺, respectively). In our opinion, the similar spectral features mean that, independently of the different long range arrangements of the T-O layer, the coordination polyhedra of chrysotile and lizardite do not largely deviate in their bonding pattern.

FTIR spectrometry

Infrared spectrometry of lizardite was carried out to verify the results of the Mössbauer spectrometry. Approximately 2–3 mg of powdered lizardite were mixed with 300 mg of KBr and pressed to a disk. The spectrum was recorded using a 5DX type Nicolet FTIR spectrometer, operating between 400 and 4000 cm⁻¹.

The IR spectrum of lizardite shows absorption bands in the $3700-3400 \text{ cm}^{-1}$ region (Fig. 2a). They correspond to the stretching vibrations of the hydroxyl groups bonded to octahedral cations. The 3684 cm^{-1} band can be attributed to hydroxyl bonded to magnesium atoms and the 3575 cm^{-1} band to hydroxyl bonded to ferrous iron atoms; the wide absorption band near 3400 cm^{-1} is normally reported as due to hydroxyl bonded to trivalent cations (i.e., Al or Fe³⁺) in octahedral coordination (e.g., Heller-Kallai et al., 1975; Serna et al. 1979). The existence of a shoulder on the lower wavenumber side of the Mg-OH stretching band represents heterogeneous cation distribution in the trimeric octahedral clusters. A possible higher heterogeneity might explain the broadening of the Fe²⁺ -OH at 3575 cm^{-1} and of the R³⁺-OH bands at 3400 cm^{-1} .

The absorption bands in the $1100-800 \text{ cm}^{-1}$ (Fig. 2b) are related to the vibrations of the different Si-O bonds in the tetrahedron. The

1086 cm⁻¹ absorption band is attributed to Si-O bonds linking the tetrahedra (Si-O-Si, Jenkins 1989). The apical Si-O bond is theoretically located at 1000 cm⁻¹ but can be slightly shifted to lower wave numbers in sheet silicates (Jenkins 1989). In the studied sample, it corresponds to a band at 992 cm⁻¹. Bands located between 960 and 940 cm⁻¹ can be attributed to Si-O bonds linked with R^{3+} cations (i.e., Al, Fe³⁺). Complexity of possible combinations explain the width of this band. Further major bands occur at 610 and 432 cm⁻¹ but their assignment remains uncertain.

The weak band present at 790 cm⁻¹ was already present (near 805 cm⁻¹) in the infrared pattern reported by Viti and Mellini (1997); however, they could not identify its origin. As suggested by the Mössbauer evidence, it is now possible to attribute this band to tetrahedrally coordinated ferric iron. In fact, based upon the theoretical value of the Si-O at 1000 cm⁻¹, the expected value of the apical Fe³⁺-O bond in the tetrahedron would be 784 cm⁻¹. Therefore, Mössbauer and FTIR data seem to agree in demonstrating the presence of ^[4]Fe³⁺.

Discussion and conclusions

Improved crystal chemical formula

On the basis of the spectrometrical results, it is possible to improve the previous structural formula for this lizardite to:

$$\begin{split} (Mg_{2.74}Fe^{2+}{}_{0.10}Fe^{3+}{}_{0.05}Al_{0.11})_{\Sigma=3.00} \\ & \cdot \; (Si_{1.94}Al_{0.05}Fe^{3+}{}_{0.01})_{\Sigma=2.00}O_{5.05}(OH)_{3.95}, \end{split}$$

by specifying the actual nature and distribution of iron cations.

As regards the oxidation state of iron in the Monte Fico lizardite, the Mössbauer determination leads to the same values already obtained by titrimetric determination (Viti and Mellini 1997). Therefore, we can confidently conclude that approximately one half (more precisely, 59.9%) of total iron there occurs in the reduced state. Fe²⁺ occurs in only one octahedral site (in agreement with all the previous three-dimensional structure refinements). Fe³⁺ occurs mostly in replacement for Fe²⁺ in the octahedral site (31.3% of total iron); however, a significant amount (8.8%, or about one fifth of total Fe³⁺) substitutes for silicon in tetrahedral coordination.

The oxidation state of Monte Fico lizardite

With respect to the range of values (Fe²⁺ ranging from 13.7 to 57.0%) found by Votyakov et al. 1993, the Monte Fico lizardite represents a more reduced specimen. Similarly, only four among 24 lizardite specimens (with total range of Fe²⁺ from 0 to 78% of total iron) in the tabulation by O'Hanley and Dyar (1993) are more reduced than the one from Monte Fico. Therefore, we can conclude that the formation of the Monte Fico lizardite took place under conditions (e.g., oxygen fugacity, temperature) leading to the crystallization of a more reduced lizardite; these relatively reducing conditions are matched by an almost total absence of magnetite, that was not detected by X-

rays but confirmed optically and by Mössbauer spectroscopy.

Al and Fe partitioning

The crystallochemical formula of the Monte Fico lizardite indicates that Fe³⁺ and Al are preferentially partitioned into the octahedral site, with an octahedral vs. tetrahedral partitioning coefficient of three. Evidently, the analytical error does not justify a reliable quantitative estimate of this value; for instance, if we compare the microprobe data given by Viti and Mellini (1997) differing from the present one only in having 1.91 versus 1.94 silicon atoms, the octahedral/tetrahedral partitioning coefficient for aluminium drops from 2.2 down to 1.4. Similarly, the data by Votyakov et al. (1993), (octahedral ferric iron ranging from 41.9 to 75.4% of total iron, versus tetrahedral ferric iron from 3.2 to 26.7), confirm the preference of ferric iron for octahedral coordination in lizardite. This preference is justified in terms of the general crystallochemical behavior of iron, which has an ionic radius more akin to octahedral than tetrahedral coordination.

Trivalent cation and/or H vacancies?

The ideal Tschermak-like substitution in lizardite (M_3T_2 -> $M_{3-x}Z_xT_{2-x}Z_x$, with Z indicating trivalent cations) would require an equal number of trivalent cations in tetrahedral and octahedral coordination. If, as it now seems, this is not the case, we have to admit the existence of another mechanism able to reduce the positive charge (O'Hanley and Dyar 1993). Two main possibilities exist:

a. The number of cations sums to less than 5.0. If we use the observed density of 2.611 g cm⁻³ and the lattice parameters derived by powder X-ray diffraction (Viti and Mellini 1997), we obtain a calculated formula weight of 281.36. Assuming a full occupancy of oxygen and hydrogen positions, the total number of vacancies would be 0.05, namely in the order of the expected value. The calculated vacancies reduce to 0.03 if the unit cell volume from single crystal X-ray diffraction is used.

b. The number of tetrahedral and octahedral cations does not differ significantly from 5.0. In that case, an alternative explanation is a partial deprotonation, already evident in the crystallochemical formula based on a full occupancy and charge balance. In particular, deprotonation would be related to the excess trivalent cations in the octahedral sheet: when ferric iron or aluminium occupy the site, some hydroxyls are lost.

This point has already been addressed by Votyakov et al. (1993). They reported that quadrupole splitting for octahedral ferric iron of 0.72–0.85 mm/s (0.71(2) for Monte Fico) would correspond to highly defective specimens, with numerous vacancies in the octahedra. Low-defects lizardite would have quadrupole splitting between 0.58 and 0.70 mm/s. Inasmuch the Monte Fico lizardite $(\Delta=0.71)$ is intermediate between those two ranges, further experimental work on the topic of deprotonation vs. octahedral vacancies is planned.

Polyhedra in chrysotile

Whereas the details of the bonding pattern in lizardite are well known, the current knowledge about the same features in chrysotile is far from satisfactory. On one side, the two minerals are definitely different on the "long range" scale, as regards the flat or curved arrangement of the TO layer. On the other side, there is no reason for having a significantly different arrangement on the "short range" scale, namely as far as the main features of polyhedral coordination are concerned. A first piece of evidence for a similarity in coordination polyhedra comes from the comparison of Mössbauer hyperfine parameters beween lizardite (present work; Votyakov et al. 1993; O'Hanley and Dyar 1993) and chrysotile (Blaauw et al. 1979; Stroink et al. 1980). A second piece of evidence may be found when analyzing polygonal serpentine (Baronnet et al. 1994; moving from the center of the fiber to the rim, the progressive transition from definitely curved structures to definitely polygonalized arrangement is observed, with no major discontinuity inbetween.

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