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SCROLLING OF THIN CRYSTALS OF LIZARDITE: AN EXPRESSION OF INTERNAL STRESS

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ABSTRACT

An investigation using transmission electron microscopy (TEM) has shown that beneficiated vermiculite from an Ontario deposit includes thin crystals of lizardite. These thin crystals have a strong tendency to curl at their edges to form scrolls, possibly in response to the misfit between the larger sheet of octahedra and the smaller sheet of tetrahedra that make up the lizardite structure. The scrolling usually occurs about the *x* axis of the lizardite. As there are three equivalent *x*-axes, the scrolling produces spectacularly decorated lizardite plates with numerous scrolls at 60° to one another. The scrolls may well become detached from the parent plate of lizardite to superficially resemble chrysotile fibers. However, there are important differences between the lizardite scrolls and chrysotile. The lizardite scrolls do not have the well-defined central core found in chrysotile, but have either no visible core or a tapered central core, commonly with bubble-like structures. In many cases, they end in a minor scroll at 60° to the scroll axis (*x*-axis) and commonly display fragments of the parent, flat lizardite plate attached to their edges. The lizardite scrolls generally are torn and do not appear to have much mechanical competence. The electron-diffraction patterns of the scrolls have superficial similarities to those of chrysotile but are significantly different, reflecting their different origin. Lizardite scrolls are much more stable in the electron beam than chrysotile fibers; it is easy to record diffraction patterns of lizardite scrolls but difficult to record patterns of chrysotile. It is very important to be able to routinely distinguish lizardite scrolls from chrysotile fibers in evaluations of vermiculite.

Keywords: lizardite scrolls, vermiculite, lizardite, chrysotile, serpentine minerals, transmission electron microscopy, electron-diffraction patterns.

SOMMAIRE

Une étude en microscopie électronique en transmission (MET) montre que la vermiculite commerciale d'un gisement de l'Ontario contient de fins cristaux de lizardite. Ces fins cristaux ont une forte tendance à se courber sur les bords pour former des rouleaux, probablement en réponse à l'écart dimensionnel entre la couche d'octaèdres, plus grands, et la couche de tétraèdres, plus petits, qui constituent la structure de la lizardite. L'enroulement le plus courant a lieu autour de l'axe *x* de la lizardite. Etant donné qu'il y a trois axes *x* équivalents, l'enroulement produit des ourlets spectaculaires au bord des plaquettes de lizardite, constitués de nombreux rouleaux disposés à 60° les uns des autres. Souvent les enroulements se détachent des cristaux parents, et dans ce cas ils ressemblent à première vue au chrysotile. Pourtant il y a des différences importantes entre les enroulements de lizardite et le chrysotile. Les enroulements de lizardite n'ont pas de partie centrale bien définie comme dans le cas du chrysotile, mais plutôt une absence de cœur visible ou un cœur "en sablier" contenant souvent des structures analogues à des bulles. Ils se terminent en plusieurs cas par un enroulement secondaire disposé à 60° de l'enroulement principal et montrent des fragments de la plaquette de lizardite parente attachés à leurs bords. Les enroulements de lizardite semblent facilement déchirés et ne semblent pas être mécaniquement résistants. Les clichés de diffraction électronique des enroulements montrent des ressemblances avec ceux du chrysotile, mais en sont en fait assez différents, ce qui reflète leur origine distincte. Les enroulements de lizardite sont beaucoup plus stables que les fibres de chrysotile sous le faisceau électronique, de telle sorte qu'il est beaucoup plus facile d'acquérir ces clichés pour la lizardite enroulée que pour le chrysotile. Il est très important de pouvoir distinguer les enroulements de lizardite des fibres habituelles de chrysotile lors d'évaluations de vermiculite commerciale.

(Traduit par Alain Baronnet)

Mots-clés: enroulements de lizardite, vermiculite, lizardite, chrysotile, les minéraux de serpentine, microscopie électronique en transmission, diagrammes de diffraction électronique.

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Introduction

During examination of vermiculite samples from the Regis Resources vermiculite mine, near Peterborough, Ontario, a previously unnoticed phenomenon was observed. When the vermiculite was dispersed for observation with a transmission electron microscope (TEM), very thin crystals of lizardite were found to be present. These thin crystals have a strong tendency to scroll up into roughly cylindrical forms that superficially resemble chrysotile. The scrolling is controlled by the lizardite structure.

Several questions arise as a result of these observations. On the crystal-chemical level, why do lizardite plates roll up to form scrolls? Is this a natural, spontaneous stress-relief mechanism produced by the misfit between the sheet of octahedra and the sheet of tetrahedra in lizardite, or is it produced during the preparation of samples for TEM study? How are the lizardite scrolls related to chrysotile? On the practical level, how can lizardite scrolls be distinguished from chrysotile fibers? Several misidentifications have already been made in commercial laboratories, resulting in misidentifications of scrolled lizardite for chrysotile fibers. We attempt to answer some of these questions in this paper.

Background Information

Previous studies of vermiculite by Chatfield & Lewis (1979, 1980) have shown that it can scroll along the edges of plates. If these vermiculite scrolls become detached from the parent vermiculite plates, they superficially resemble chrysotile fibers. They do, however, have a different chemical composition, an SAED pattern of vermiculite rather than chrysotile, and a much lower contrast in electron micrographs compared to that exhibited by chrysotile.

In another early study, Mifsud *et al.* (1977) found scrolling in vermiculite particles associated with silver-colored areas of alteration visible on the surface of vermiculite plates. TEM studies showed scrolling of vermiculite along plate edges, and along surface creases and pits. With increased folding back (scrolling) of the vermiculite, Mifsud *et al.* (1977) determined that it was altered to chrysotile tubes aligned at 60° and 120° to one another across the surface of the vermiculite. Infrared absorption spectra, powder XRD and SAED evidence were used to identify this material as chrysotile. However, the experimental evidence presented in the paper is not sufficiently detailed to distinguish between chrysotile and lizardite. The published TEM micrographs show a partially rolled and partially kinked structure, but not the tightly rolled structure of chrysotile. It is possible that these authors actually described lizardite scrolls similar to whose found in samples from the Regis Resources mine.

In all three of these examples of scrolling in vermiculite and serpentine, the $a = 5.3$ Å dimension forms the axis of scrolling. As all these structures are hexagonal or pseudohexagonal, the alternative a directions are aligned at 60° to one another.

The misfit between the larger sheet of octahedra and the smaller sheet of tetrahedra in the 1:1 layer serpentine structure has been cited as one of the reasons that cause serpentine to occur in three distinct structures: the flat planar structure of lizardite (Mellini 1982, Mellini & Zanazzi 1987, Wicks & O'Hanley 1988, Mellini & Viti 1994), the cylindrical structure of chrysotile (Whittaker 1953, 1956a, b, c, Wicks & Whittaker 1975), and the alternating wave structure of antigorite (Wicks & O'Hanley 1988, Capitani & Mellini 2004). In lizardite, the misfit is accommodated by small adjustments in the position of the atoms within the planar structure, and regular hydrogen bonding develops between the outer OH groups of the sheet of octahedra and the basal O of the adjacent sheet of tetrahedra.

Four structures of chrysotile have been found, chrysotile-2M_{c1}, chrysotile-2Or_{c1}, chrysotile-1M_{c1} and parachrysotile (Wicks & Whittaker 1975). All have cylindrical structures which produce the chrysotile's fibrous habit. The x axis is the fiber axis in three of the chrysotile structures, and the y axis is the fiber axis in parachrysotile. Parachrysotile and chrysotile 1M_{c1} are very rare and need not be discussed further. Chrysotile-2M_{c1} is the most abundant, followed by chrysotile-2Or_{c1}. The cylindrical structure produces rows and grooves formed from the outer OH groups of the sheet of octahedra running around the circumference of the cylinder. The basal atoms of oxygen of the sheet of tetrahedra are systematically protruded and withdrawn, so that the former fit into the grooves between OH rows, and the latter sit over the OH rows. This arrangement causes a shift between successive layers that is unique in serpentine minerals. Hydrogen bonding can develop between some OH groups and basal O atoms, but complete hydrogen bonding cannot develop because of the changing register between layers around the circumference of the structure.

Antigorite is curved about the y axis in an alternating-wave structure (Capitani & Mellini 2004). The wavelength is defined by m, the number of tetrahedra making up the wave. Samples of antigorite usually vary in m between 13 and 20, which is equivalent to 33 to 52 Å. The sheet of octahedra is continuous throughout the wave, as is the sheet of tetrahedra, but the latter reverses polarity where the wave changes its direction of curvature.

It is important to recognize that although the curvature relieves the misfit along the direction of curvature in the structures of chrysotile and antigorite, it does not relieve the misfit in the direction at right angles to the curvature. Thus, some shifts in atom positions similar to those in lizardite will also occur in these structures.

Cylindrical serpentine structures composed of 15 or 30 radial sectors of planar serpentine have been termed polygonal serpentine (Baronnet & Devouard 2005). It occurs in many non-fibrous serpentine veins and in some rock-forming serpentines. Occasionally, polygonal serpentine forms about a core of chrysotile, but commonly it forms on its own. The planar sectors appear to be formed of various lizardite polytypes, but more detailed work is required on this. Polygonal serpentine is essentially a unique and fascinating configuration of known serpentine structures, lizardite with or without chrysotile, not a different structure-type of serpentine. However, the curved segments that join adjacent polygonal sectors are structurally unique (Baronnet & Devouard 2005), so that polygonal serpentine does, in part, contain a new structural modification. It needs to be determined if the lizardite scrolls are in any way related to polygonal serpentine.

Experimental Procedures

All water used in the preparation of specimens for TEM study was distilled and filtered through a 0.1 μm porosity filter of mixed esters of cellulose (MEC). Approximately 50 grams of vermiculite ore were added to 800 mL of filtered distilled water in a 1000 mL glass beaker. The beaker was placed in an ultrasonic bath for 5 minutes. While the ultrasonic bath was still operating, aliquots of the suspension were withdrawn using a pipette, and the aliquots were filtered through 0.1 μm pore-size track-etched polycarbonate (PC) filters 25 mm in diameter.

TEM specimens with appropriate particulate loading were prepared from PC filters using the procedures of ISO 13794 (1999). The solvent used was a mixture of 1–2-diaminoethane and 1-methyl-2-pyrrolidone.

A Philips EM301 transmission electron microscope operated at 80 kV was used to acquire the images and electron-diffraction data presented here. The microscope is equipped with a Gresham Scientific Instruments Ltd. silicon X-ray detector with an area of 30 mm^2 and a resolution of approximately 143 eV and a Kevex 7000 analytical system.

Results

Morphology of attached scrolls

The TEM specimens are dominated by vermiculite, but a small amount of lizardite also is present. Vermiculite occurs as somewhat featureless plates. The lizardite occurs as very thin crystals with reasonably well-defined hexagonal outlines (Fig. 1). Semiquantitative energy-dispersion X-ray analyses of several plates and scrolls show that the vermiculite has a significant Al and Fe content, but that the lizardite has a very minor, but persistent Al and Fe content. Our study has concentrated on the features of the lizardite crystals.

The dominant features of the lizardite are its thinness and tendency to curl at the hexagonal edges of the crystal to form scrolls (Fig. 1). In contrast, the vermiculite is thicker and does not form scrolls. The scrolling of lizardite is, in general, crystallographically controlled, with angles between adjacent scrolls near 60°. However, although the initiation of scrolling is crystallographically controlled, once the scrolling begins, it may be deflected from the ideal angle by interference from other scrolls, by conical scroll development, or by the dynamics of rolling sending the scroll off in a slightly different direction (Figs. 1, 2).

The images in Figures 1 and 2 are two of the more spectacular. The scrolling is complex. In some cases, the entire thickness of lizardite rolls up to form a scroll (Fig. 1, top-right edges). On other edges, a few layers of lizardite roll up, leaving lower layers behind to roll up into successive, parallel scrolls (Fig. 1, bottom-left edge). Some edges remain flat (Fig. 1, bottom-left part of the crystal) and show the edges of the original hexagonal crystal. The lizardite scrolls appear to be fairly delicate, and some have visible tears and breaks (*e.g.*, Figs. 1, 2).

Not all scrolls are formed from the edges of the crystal. Near the center of the crystal in Figure 2, three scrolls have formed from a central crack or tear. One scroll has rolled up to the left, one to the right, and a third toward the bottom. The action of rolling the bottom scroll has caused thin lizardite layers to tear. This occurred at both ends of this scroll, but is particularly visible at the right end.

Some plates are less complex (Fig. 3) with only three major scrolls at near 60° to each other around a triangular, flat relic of the original particle. Some scrolls have irregular terminations (Fig. 3, smaller scroll at top left and the bottom end of the scroll on the right side), step-in-step terminations showing each individual roll of the scroll (Fig. 3, scroll at top left and Fig. 4) and some have a near-60° minor roll-over at the termination (Fig. 3 at top end of right scroll).

Scrolls may have parallel sides in a tight roll (Fig. 3, left scroll, and many scrolls in Fig. 1), be gently tapered toward the ends (Fig. 1, left scroll pointing to the top left), or be conical (Fig. 2, top and bottom scrolls on the left side, Fig. 3, right scroll). Some scrolls have no visible internal structure (Figs. 1, 2). Others are thinner at the ends and show successive turns of the scroll at 60° to the scroll axis (Fig. 3, right scroll). These scrolls must have started at a crystal edge and become longer than the crystal edge as they rolled into the body of the crystal, picking up new material. The central part of the scroll has the maximum number of layers, whereas the ends have fewer and fewer layers as they grow longer (Fig. 3, turns are visible at the top end of the right scroll). This is the opposite of the step-in-step terminations (Fig. 3, scroll at top left, and Fig. 4).

Some scrolls have internal structure. The scroll in Figure 4 is the most extreme example of this. A central

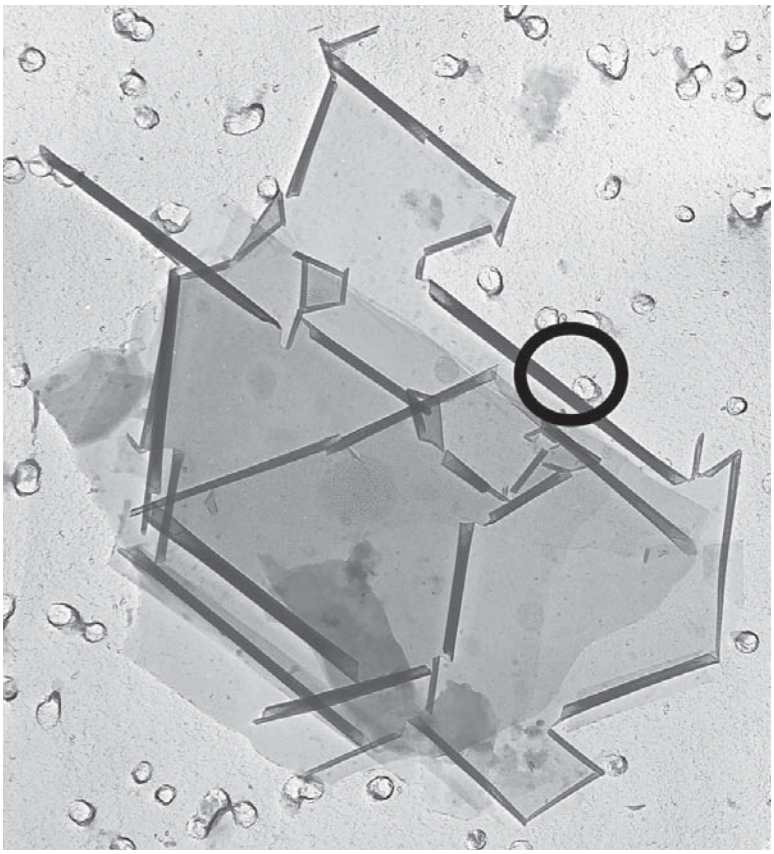


FIG. 1. A flat particle of lizardite with abundant scrolls arranged at 60° to each other. The scrolls on the top right side have formed by rolling of the complete thickness of the original particle of lizardite. The two scrolls on the bottom left side have formed successively from discrete thicknesses of the lizardite, leaving a thin flat portion of lizardite unrolled. The edges of the remaining flat particle of lizardite suggest that it was originally a euhedral or subhedral hexagonal crystal. The large scroll in the center of the image has broken, and the thin lizardite beneath it has been torn. Tearing associated with several scrolls also is visible. The circle marks an area of SAED pattern shown in Figure 10. The circular marks in the background are holes in the PC filter and are 0.1 μm in diameter.

opening tapers from the bottom toward the top of the scroll to end in a narrow zone with elongate bubble-like features. The bubble-like features occur in other scrolls (Fig. 3, all the scrolls).

It is possible that some of the scrolls are U-shaped, formed by curling from either side, but not closing across the top of the scroll. This is not proven but remains a possibility.

Only a few minor examples of scrolling about the *y* axis were observed. At the bottom left side of Figure 2, a very thin scroll occurs at right angles to a major scroll. As the major scroll is rolled about the *x* axis, it follows that the scroll at right angles has rolled about the *y* axis. A second broad scroll, parallel to the first, occurs at the bottom right corner, and a very narrow one running NW–SE occurs halfway up the right-hand side.

Morphology of detached scrolls

When a scroll is still attached to a lizardite crystal, its origin is obvious. However, when a scroll becomes detached from the parent crystal of lizardite, it superficially resembles an individual fiber of chrysotile, or a bundle of fibers. In fact, misidentification has already been made during routine particle-analysis by a production-oriented technician relying on minimum

morphological features and automated identification of SAED patterns. Close examination of a scroll usually produces enough features to identify it as a lizardite scroll even without SAED confirmation.

Some detached scrolls are actually two scrolls that have rolled from either side of a lizardite crystal to form two adjacent, parallel scrolls (Fig. 5). However, there are still five areas of the original flat crystal of lizardite associated with these scrolls, and there are many minor scrolls related to these five areas.

A well-developed scroll, with little internal detail, is shown in Figure 6. A small second scroll occurs at the top end, and a larger secondary scroll at the bottom end of the main scroll. The main scroll and the larger

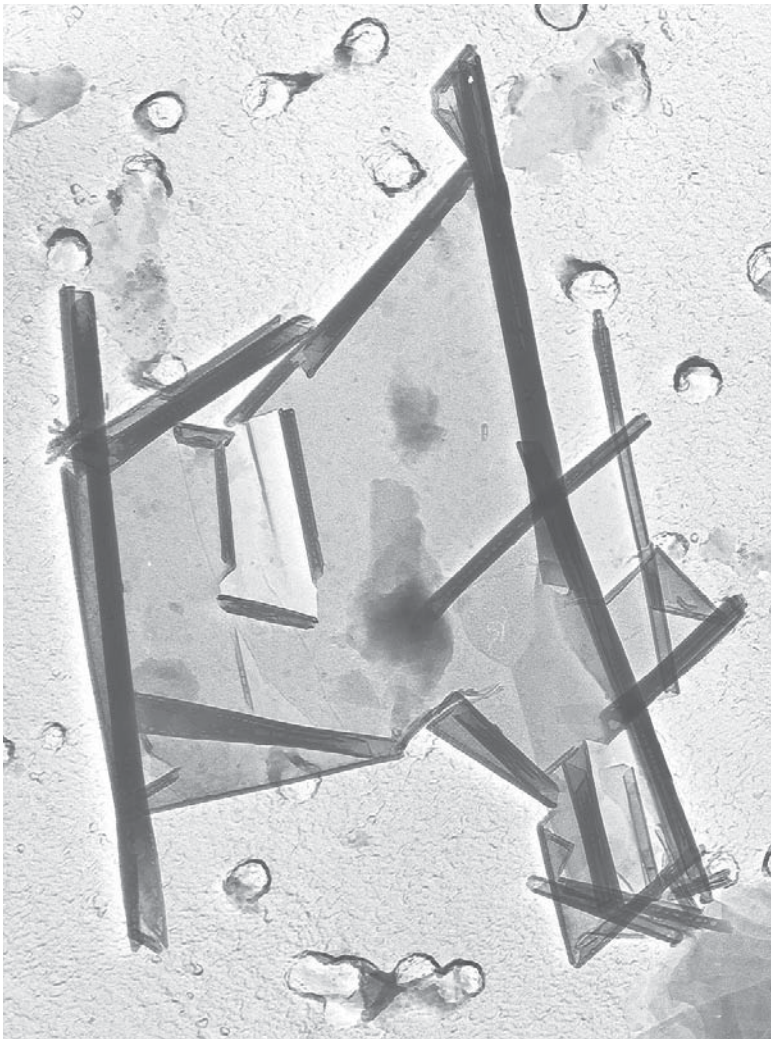


FIG. 2. A flat particle of lizardite with abundant scrolls. The scrolls in the center of the crystal have formed from a crack or tear in the lizardite particle, not from an edge. One scroll rolled to the left, one to the right, and a third toward the bottom. Tearing of the thin lizardite occurred as the third scroll formed. The scrolls to the top and bottom of the left side of the particle have a conical form. Three scrolls have rolled about the *y* axis, a very thin scroll at the bottom left edge of the particle, a broad scroll parallel to the first, at the bottom right corner, and a very narrow scroll running NW–SE halfway up the right-hand side. The detached scroll at the right edge is enlarged in Figure 4. The holes in the PC filter are 0.1 μm in diameter.

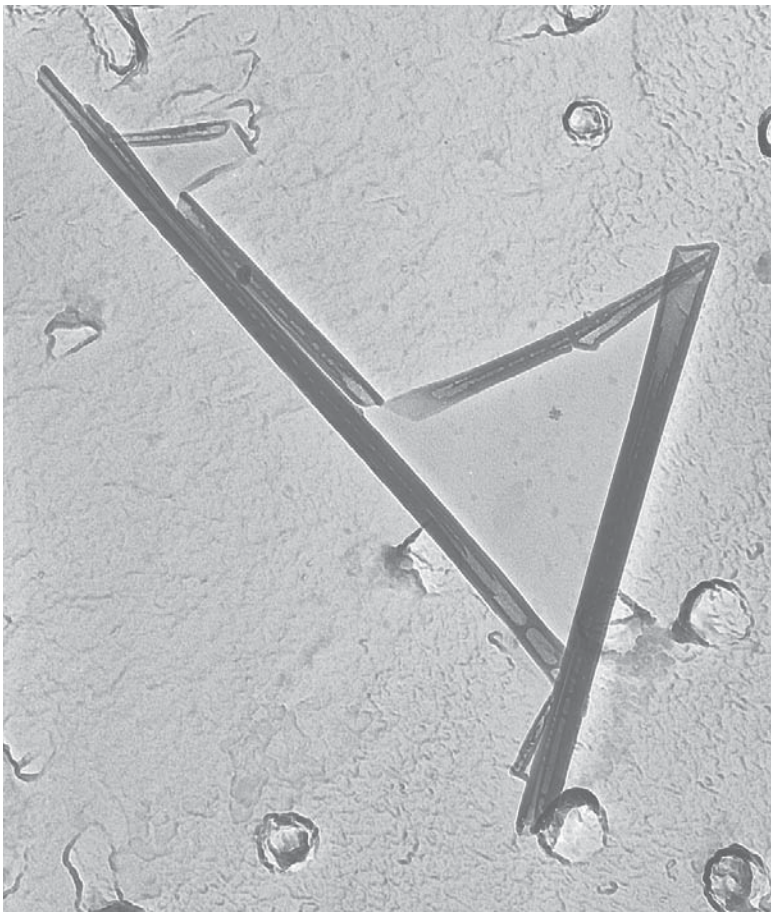


FIG. 3. A relict flat particle of lizardite with three scrolls at 60° to each other. The right-hand scroll is slightly conical and has a 60° roll-over at the top end. It is also thinner at that end and shows successive turns of the scroll at 60° to the scroll axis. All the scrolls have a fine central zone with bubble-like features. The holes in the PC filter are 0.1 μm in diameter.

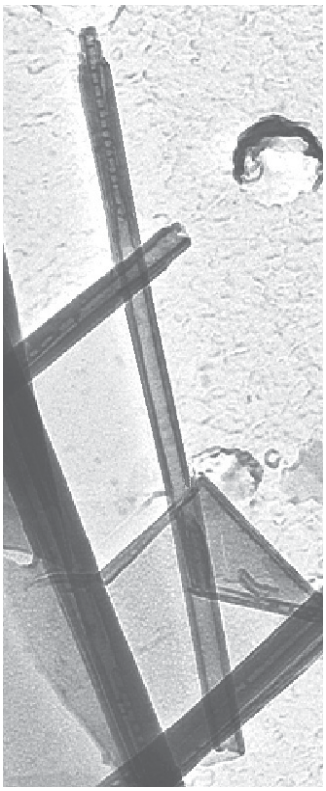
secondary scroll have a joint 60° roll-over termination. At the other end of the secondary scroll, faint successive layers of lizardite are visible within the end of the scroll at 60° to the scroll axis. There is also a small residual part of the original lizardite crystal, with secondary scrolls, still attached to the main scroll.

Groups of scrolls could be mistaken for a bundle of chrysotile fibers by inexperienced operators. However, close examination of Figure 7 shows that adjacent scrolls have formed from the same thin crystal of lizardite by rolling toward each other until they are almost in contact. A narrow area of the original flat lizardite is visible between the scroll that has rolled from the left and the two scrolls from the right. There are elongate bubble-like features along the central zone in the right-hand scroll, and similar features are significantly offset from the center of the left-hand scroll.

Even in isolated scrolls that do not show most of the details described above, there are usually small clues, such as the small projection of flat lizardite on the scroll in Figure 8, that identify the lizardite scroll.

Selected-area electron diffraction

The SAED patterns from the lizardite scrolls show that the *x* axis is the scroll axis. Only three examples of small scrolls formed about the *y* axis were observed (Fig. 2). Because the *x* axis is the fiber axis of chrysotile-2M_{c1} and chrysotile-2Or_{c1}, the diffraction patterns of chrysotile fibers bear a general similarity to those



of lizardite scrolls (Zussman *et al*. 1957, Wicks 1979, Wicks & O'Hanley 1988). However, there are important differences between the two types of diffraction patterns, which reflect the differences between the two rolled structures. The SAED patterns of the lizardite scrolls are complex and more variable than chrysotile patterns because variations in the scrolling produce variations in the diffraction patterns. Variations in intensities between the top and bottom and left and right sides of the SAED patterns are common.

The thin, flat crystals give a lizardite SAED pattern (Fig. 9). This is the basic hexagonal pattern upon which other diffraction patterns of lizardite, produced by the scrolling, are superimposed. This pattern is indexed with an orthohexagonal cell for easy comparison with the diffraction patterns of the scrolls.

The diffraction patterns of two scrolls formed from rolling of the edge of this flat lizardite plate are superimposed on the main pattern. The diffraction pattern of the first scroll is aligned with the diffraction pattern of

FIG. 4. A long scroll of lizardite broken one-third of the way from the bottom end, with step-in-step termination at the top end. The scroll appears to have a central core that tapers from the bottom toward the top. There is fine bubble-like structure in the core at the upper end. The holes in the PC filter are 0.1 μm in diameter.

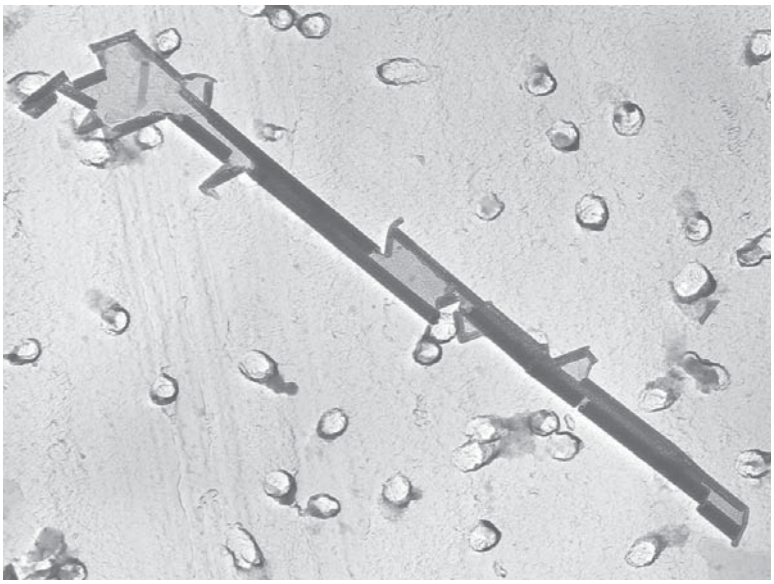


FIG. 5. Lizardite scrolls that have formed by rolling from either side of a lizardite plate. There are five relic areas of the initial crystal of lizardite along the length of the scrolls. Each area has its own secondary scrolls. The holes in the PC filter are 0.1 μm in diameter.

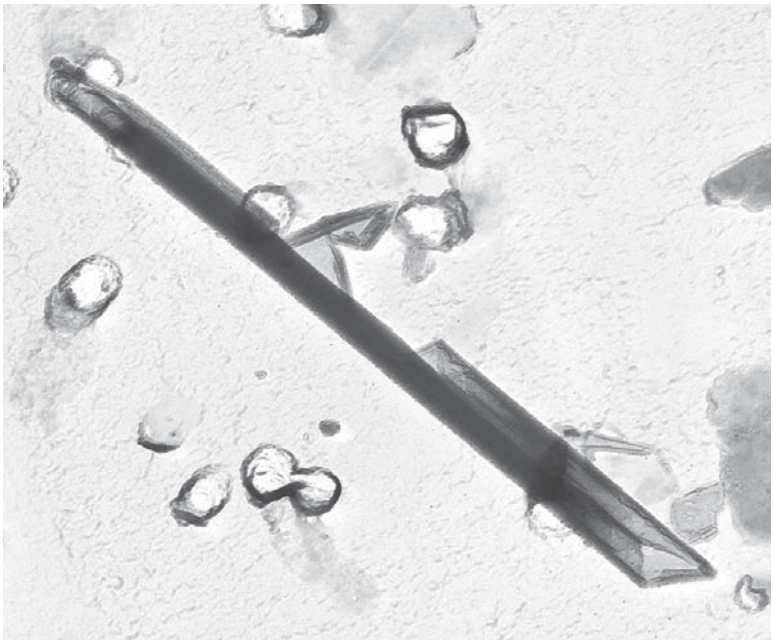


FIG. 6. A lizardite scroll with little visible internal structure. The top termination is irregular, and a minor secondary scroll has rolled up against the top right side of the main scroll. A larger secondary scroll has rolled against the bottom end of the main scroll, and the two scrolls share a 60° roll-over termination. A small area of the original planar lizardite with its own secondary scrolls projects from near the midpoint of the main scroll. The holes in the PC filter are 0.1 μm in diameter.

the flat portion and shows up as 00*l* reflections in addition to the 0*k*0 reflections along the zero-layer line. The diffraction pattern of the second scroll is very weak and inclined at 13° to the first scroll and the main pattern.

The SAED pattern in Figure 10 was recorded from the scroll identified by the circle in Figure 1. The series of 20*l* reflections on the upper second-layer line identify the serpentine to be lizardite-1*T*, the most commonly occurring polytype of lizardite. This scroll, or at least part of this scroll, is well ordered. This is unusual, as most SAED patterns from scrolls show streaking of intensity along the second-layer lines, suggesting significant disorder.

In many of the diffraction patterns of the scrolls, the 00*l* reflections lie along the zero-layer line, but the 0*k*0 reflections are commonly spread perpendicular to the zero-layer line or split into reflections on either side of the line (Fig. 10). The 1*k*0 reflections are similarly split about the first-layer line (Fig. 11) and are occasionally followed by what appears to be 1*kl* reflections (Fig. 11). These features suggest that there is a helical element in the rolling of the scrolls (Whittaker 1955, Whittaker & Zussman 1971, Yada 1971), so that one side of the scroll is in a different position to the electron

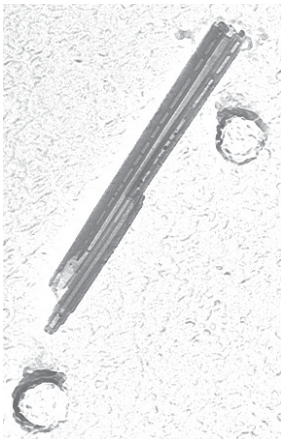


FIG. 7. Two scrolls of lizardite have formed by rolling from either side of a lizardite plate that is visible in the area between the scrolls. There is an edge of the original crystal at 60° to the irregular bottom end of the left-hand scroll. A third scroll lies against the bottom end of the right scroll that has step-in-step terminations. The right scroll has a near-central zone of bubble-like structure, but in the left scroll, the zone is offset to one side of the scroll. The holes in the PC filter are 0.1 μm in diameter.

FIG. 8. A lizardite scroll with little visible internal structure, an irregular termination at the bottom end, and a 60° roll-over termination at the top end. A small relic of the original planar lizardite protrudes from this end of the scroll. The holes in the PC filter are 0.1 μm in diameter.

beam than the opposite side of the scroll. This situation does not affect the 00*l* reflections, which remain on the zero-layer line.

The elongate, diffuse 200 reflections from scrolls commonly occur on either side of the central sharp 200 reflection from the flat areas of the lizardite (Figs. 9, 11). The 20*l* reflections are broad, and intensities are spread out along the second-layer line. This produces an apparent similarity to the second-layer line of chrysotile-2M_{c1}, but in detail the reflections from the scrolls are in different positions and have different intensities than those expected from chrysotile-2M_{c1} and chrysotile-2Or_{c1}. The streaking-out of intensity means that there is disorder within a scroll. As the lizardite rolls are composed of reasonable well-crystallized lizardite 1*T,* the streaking suggests that successive rolls of lizardite within a scroll are not well aligned with each other, and this introduces disorder. Hydrogen bonding thus is not well-developed between successive rolls of the scroll.

The lizardite scrolls are very stable in the electron beam, regardless of the degree of irradiation in the TEM. This generally allows intense SAED patterns of lizardite to be recorded. In contrast, chrysotile fibers are unstable, and degrade rapidly in the electron beam, so that the SAED patterns fade rapidly during irradiation, in some cases even before they can be photographically recorded. Once one is aware of this characteristic, it is a convenient aid in identification.

DISCUSSION AND CONCLUSIONS

It is appealing to think that, where it is able to, lizardite rolls up to help to relieve the misfit between the sheets of octahedra and tetrahedra. In fact, in this study, we show that lizardite does roll up into scrolls. The force involved in the scrolling is fairly strong, as the tearing of the thin crystal of lizardite during the formation of scrolls demonstrates (Fig. 2). But scrolling is not universal, and flat plates of lizardite remain after other areas have rolled into scrolls (Fig. 1). This finding suggests that stress produced by the misfit is not the only factor influencing the structural morphology. More research is required to understand the interplay of forces between planar and scrolled lizardite, and the possible effect of sample preparation on scrolling.

It is important to realize that, in spite of their superficially similar forms, lizardite scrolls and chrysotile fibers are fundamentally different, both mechanically and at the atomic scale. Lizardite scrolls are mechanically weak, commonly broken, and tend to break rather than bend. They appear to have a maximum length to approximately 4 μm because they are limited to the size of the parent crystal of lizardite. In contrast, chrysotile fibers are mechanically strong, with a high tensile strength, usually in the range of 1,100 to 4,400 MN m^{-2} (Hodgson 1986), and are usually very flexible. Chrysotile fibers are continuously grown crystals

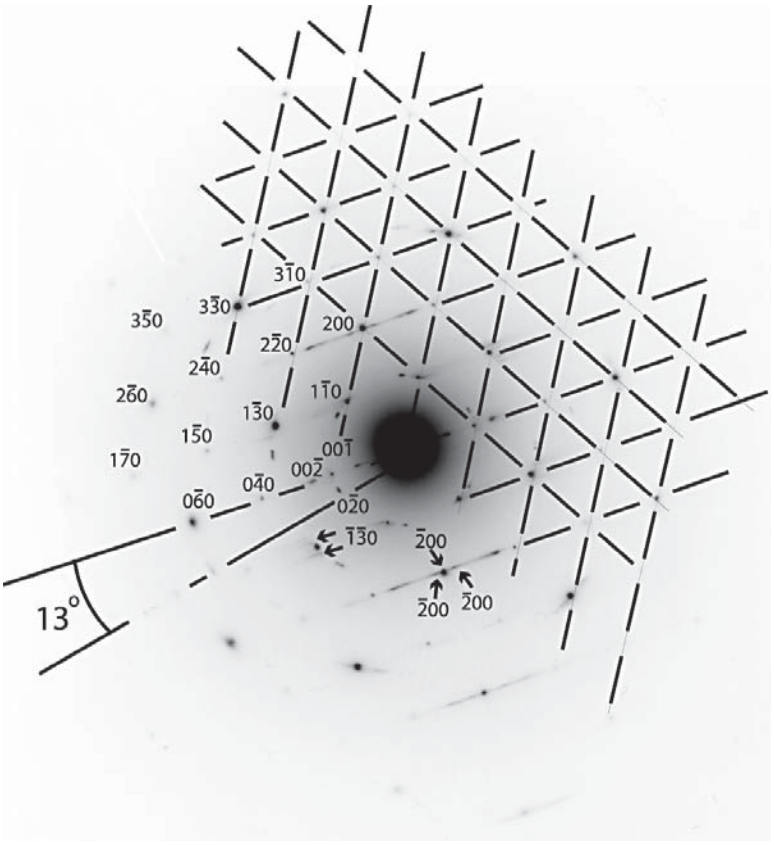


FIG. 9. An SAED pattern from a lizardite crystal composed of *hk*0 reflections in a hexagonal array. A series of 00*l* reflections from an attached scroll lie along the zero layer line. Diffuse 200 reflections from the scroll are present on either side of the sharp 200 reflection from the crystal. Weak 00*l* reflections lie on a zero-layer line from a second scroll, aligned at 13° to the first scroll.

from their core through to their outer margin, and their strength and flexibility are related to this. Although the successive rolls of lizardite scrolls also are crystalline, the scrolls have not grown as a coherent crystal, but simply have rolled up about the *x* axis. As the conical or tapered shape scrolls, and those that have rolled significantly off the *x* axis demonstrate, there is no strict crystallographic control between rolls. This limits systematic hydrogen bonding between successive rolls. Thus the scrolls are not crystalline in the way a chrysotile fiber is crystalline, and this difference would make the lizardite scrolls mechanically weak.

In the electron beam of the TEM, the behavior of the two materials is reversed. Chrysotile fibers are unstable in the beam, but the lizardite scrolls are very stable and can withstand a high degree of irradiation. It seems that the irregular hydrogen bonding in chrysotile leaves a large proportion of underbonded hydrogen throughout the structure that is disturbed by the electrons and results in the breakdown of the structure. In contrast, most of the hydrogen in lizardite approximates the ideal configuration for hydrogen bonding because the unsatisfied hydrogen bonds at the interface between successive rolls of lizardite are relatively insignificant compared to the mass of the lizardite in the successive rolls. Even if this underbonded hydrogen is disturbed by the electrons, it is not essential to the mechanical structure of the lizardite scroll, so the scroll remains stable.

One similarity between lizardite scrolls and chrysotile fibers is the rarity of curvature about the *y* axis. Parachrysotile, the chrysotile formed by curvature about the *y* axis, occurs infrequently. Curvature about the *y* axis also is rare in lizardite scrolls. Curvature about the *x* axis is preferred in these structures, but it is interesting that curvature about the *y* axis is essential to the antigorite alternating-wave structure.

There are many morphological details that can be used to distinguish detached scrolls of lizardite from

chrysotile fibers. The scroll may have parallel sides, but are commonly gently tapered, or are slightly or strongly conical. Careful examination of a scroll almost always reveals an attached fragment of the initial flat crystal of lizardite. The termination of the scrolls is commonly diagnostic, with step-in-step, 60° roll-overs, visible successive turns of the scroll at 60° to the scroll axis near the end of the scroll, or an irregular opening. Lizardite scrolls generally have no visible internal details. Others have a tapered or irregular central zone, commonly with bubble-like features. Although this is usually central, it can be off-center with respect to the scroll. All these features are different from those found in chrysotile fibers. Chrysotile fibers have smooth parallel sides, blunt featureless terminations, and a uniform central core.

The SAED diffraction patterns of lizardite scrolls and chrysotile fibers have the same basic configuration because both are rolled about the *x* axis. However, the details of the patterns are quite different. In rare cases, the scroll produces a lizardite 1*T* diffraction pattern. More commonly, the lizardite scrolls produce a more complex diffraction-pattern. The second layer line is complex, with many reflections recorded, but they do not match, in terms of intensities or positions, the diffraction patterns of either the chrysotile-2M_{c1} or chrysotile-2Or_{c1}. Splitting of 0*k*0 and 1*k*0 reflections about their layer lines is common in patterns from lizardite scrolls, and a few have 1*kl* reflections following 1*k*0 reflections. Lizardite scrolls are stable in the electron beam, and SAED patterns are easy to record. Chrysotile fibers are unstable in the electron beam, and SAED patterns are more difficult to record.

This paper is a report of work in progress, but it is important to publish it now so that serpentine researchers are aware of lizardite scrolls and can distinguish between lizardite scrolls and chrysotile fibers. Several features need to be determined. Are these lizardite scrolls a natural feature, or did they form during specimen preparation? Is the lizardite interlayered with the vermiculite? It is assumed that lizardite scrolls form with the sheet of octahedra on the outside, but this needs to be confirmed. Does the fact that lizardite is a polar structure have any role to play in the scrolling mechanism? The thickness of the initial crystals of lizardite and the thickness of units forming scrolls need to be determined. The bubble-like features along the length of some of the scrolls need to be studied in detail to see how they are related to the scrolling. Whether or not scrolling produces U-shaped, as well as cylindrical, scrolls needs to be determined. The variations in the selected-area electron-diffraction patterns of the scrolls, particularly parallel-sided and conical scrolls, need to be studied in detail. It is important to learn more on the details of the scrolling and if the lizardite scrolls have any relationship to polygonal serpentine. To do this, cross-sections of lizardite scrolls need to be observed.

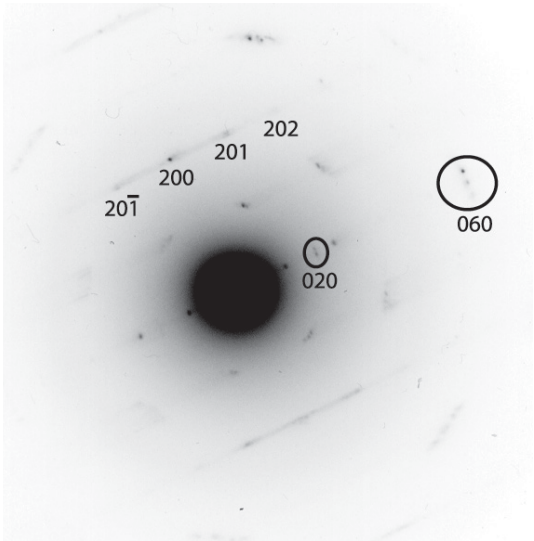


FIG. 10. An SAED pattern from a lizardite-1*T* scroll. The series of 20*l* reflections on the second layer line are diagnostic. The 0*k*0 reflections (circled) are split about the zero layer line, suggesting a helical twist to the scroll.

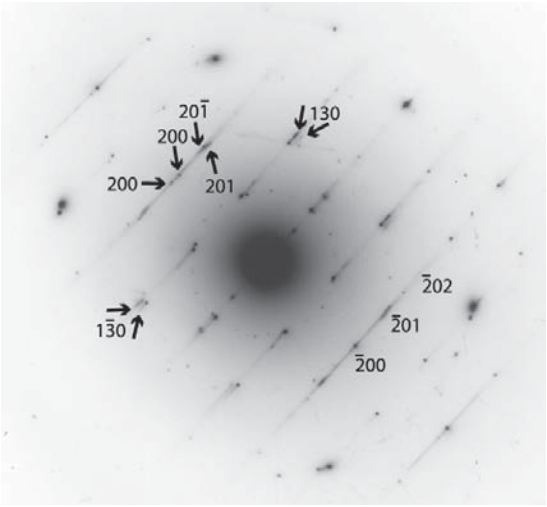


FIG. 11. An SAED pattern from a lizardite scroll. Two sets of 20*l* reflections are present, one from either side of the scroll. The 1*k*0 reflections occur in two positions about the first layer line, and 13*l* reflections follow some of the 130 reflections.

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