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THERMAL TRANSFORMATION OF TALC AS STUDIED BY ELECTRON-OPTICAL METHODS

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Abstract—The thermal decomposition of a fibrous talc was studied by transmission electron microscopy (TEM) and selected-area electron diffraction (SAD). Small changes in the lengths of a and b unit-cell parameters were noticeable at 500°C, but the talc laths did not change morphologically until 800°C. At this temperature striations began to appear in the TEM image, and the talc SAD reflections began to develop faint satellite streaks. At 900°C the striations appeared to be crystallites, and reflections of orthorhombic enstatite were noted. Both TEM and SAD evidence showed that the enstatite crystallites were formed in three orientations corresponding to the three pseudohexagonal a axes of the talc. Thus, triple superposition of the electron diffraction pattern at the three equivalent angles explains the high symmetry star-shaped pattern. At 1000° to 1100°C the enstatite crystallites were shorter and thicker, and the streaks in the SAD pattern were nearly absent. Above 1200°C only one orientation of the enstatite crystallites was found. Noncrystalline regions were also detected at 900°C and became progressively scarce at 1000° and 1100°C. They were not detected at 1200°C. At 1300°C cristobalite was detected in some SAD patterns. The crystallographic axes and unit-cell parameters of the talc and enstatite were also topotactically related as follows: a_t (5.3 Å)//c_e (5.2 Å); b_t (9.16 Å)//b_e (8.8 Å); d(001)$_t$ (18.84 Å)//a_e (18.2 Å). These results are compatible with an inhomogeneous decomposition mechanism as proposed by earlier workers.

Key Words—Enstatite, Morphology, Selected-area electron diffraction, Talc, Thermal transformation, Topotactic relation, Transmission electron microscopy.

Resumo—A decomposição térmica de um talco fibroso foi estudada por microscopia eletrônica de transmissão (MET) e por difração eletrônica de área selecionada (SAD). Foram observadas ligeiras modificações nos comprimentos dos parâmetros da cela unitária a e b a 500°C, mas as ripas de talco não apresentaram transformações morfológicas até 800°C, quando estriações começaram a aparecer nas micrografias e a desenvolver reflexões satélites em forma de traços muito tênues. A 900°C as estriações puderam ser caracterizadas como cristalitos e começaram a aparecer reflexões de enstatita ortorrômbica. As evidências obtidas por MET e SAD mostraram que os cristalitos de enstatita foram formados em três orientações correspondentes aos três eixos a pseudohexagonais do talco; assim a tripla superposição do diagrama nos três ângulos equivalentes explica a alta simetria do diagrama em forma de estrela. Entre 1000° e 1100°C os cristalitos se tornaram mais curtos e mais grossos, e os traços no diagrama SAD quase desapareceram; acima de 1200°C apenas uma direção dos cristalitos de enstatita foi encontrada. Regiões não cristalinas foram detectadas a 900°C se tornando progressivamente mais raras entre 1000° e 1100°C e desaparecendo a 1200°C. A 1300°C pôde ser detectada a cristobalita em alguns diagramas de SAD. Os eixos cristalográficos e os parâmetros das celas unitárias do talco e da enstatita são relacionados como se segue: a_t (5.3 Å)//c_e (5.2 Å); b_t (9.16 Å)//b_e (8.8 Å); d(001)$_t$ (18.84 Å)//a_e (18.2 Å). Os resultados do presente trabalho são compatíveis com um mecanismo de decomposição como aquele proposto por Taylor e por Brindley.

INTRODUCTION

The high-temperature reactions of individual clay minerals and other phyllosilicates are largely topotactic reactions (Brindley, 1963a). The reaction of talc to enstatite + cristobalite at high temperatures is represented by the equation:

$$\underset{\text{talc}}{Mg_6Si_8O_{20}(OH)_4} \longrightarrow \underset{\text{enstatite}}{6\,MgSiO_3} + \underset{\text{cristobalite}}{2\,SiO_2} + \underset{\text{water}}{2\,H_2O}. \quad (1)$$

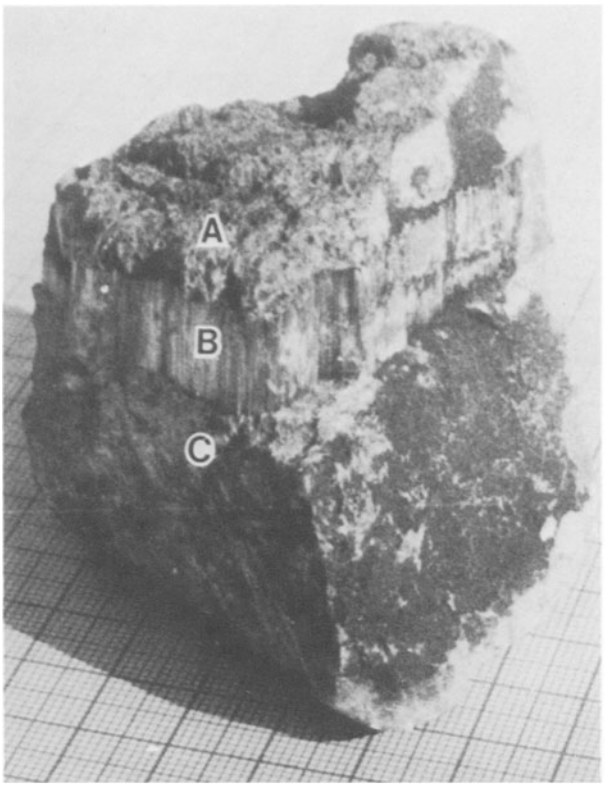
Nakahira and Kato (1964), using X-ray and electron diffraction to study dehydration mechanisms in talc and pyrophyllite, found that both enstatite and cristobalite formed topotactically with respect to the initial talc structure.

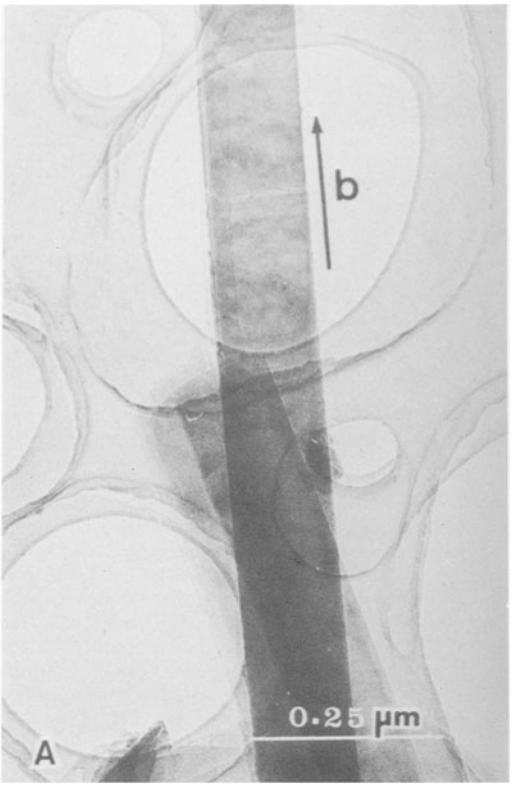
In their study of the decomposition of talc and the formation of enstatite by means of electron-optical and X-ray diffraction methods, Bapst and Eberhart (1970) found three orientations of enstatite crystals relative to the original talc.

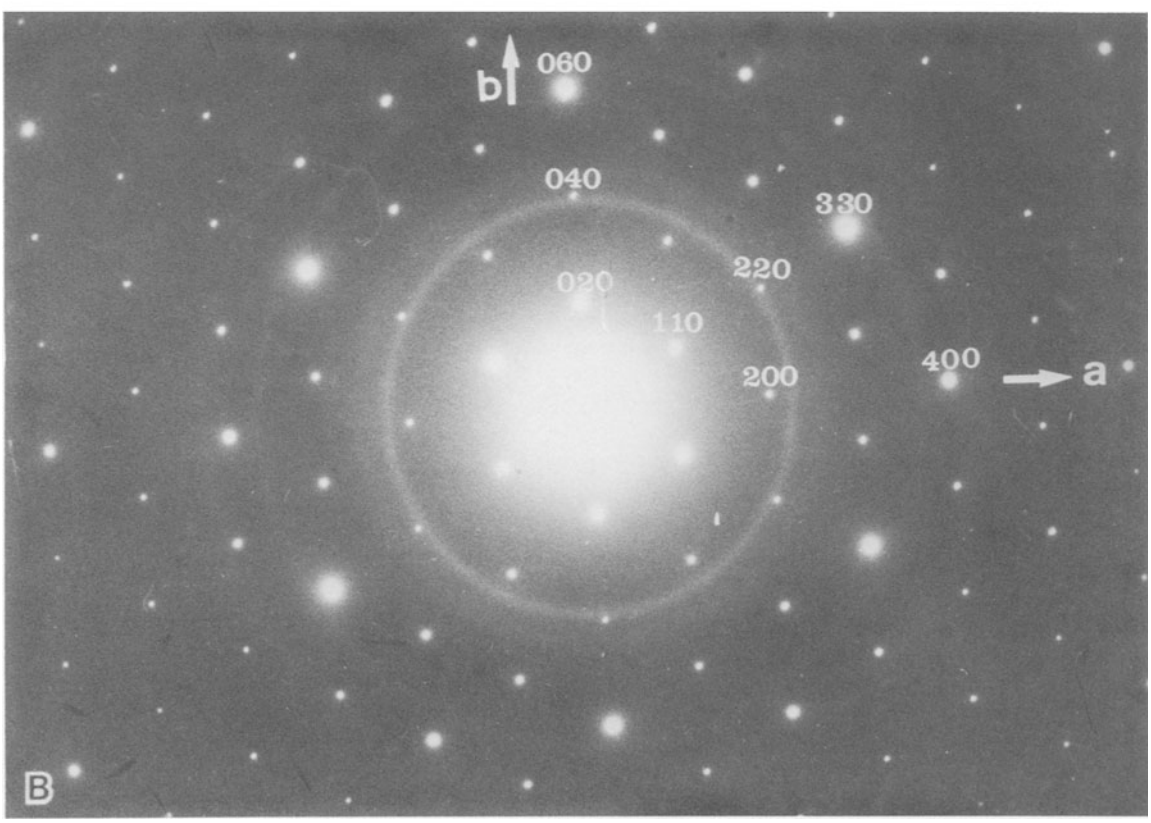
Daw *et al.* (1972) studied the transformation of talc by transmission electron microscopy and electron diffraction at high temperature and described the dehy-

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droxylation of talc and the development of enstatite. Bubbles and voids were observed during dehydroxylation, and enstatite crystals formed near the voids. The authors proposed that dislocations in talc served as preferential sites for nucleation of the bubbles and that the enstate "crystallites" formed topotactically.

The present work was undertaken to study thermal transformation of talc heated in dry conditions by high-resolution transmission electron microscopy (HRTEM), dark field imaging, and selected-area electron diffraction (SAD) in order to establish their precise topotactic orientation in relation to the two pseudo-axes of the initial talc structure.

MATERIALS AND METHODS

Talc for this study was hand picked from veins in a sample of serpentinite rock from the Jeffrey Mine, Asbestos, Quebec (Aumento, 1970). Fibrous talc was chosen because it consisted of lath-like particles, for which topotactical relations could be easily determined by TEM and SAD. Figure 1 is a photograph of the serpentine rock showing a central vein B of fibrous talc about 10 mm wide bordered in both sides by common massive talc (A and C), which has foliated hexagonal outlines when pulverized. Energy-dispersive X-ray microanalysis using a Kevex 7000Q system yielded the following composition (wt. %) for the central part of the sample studied, expressed in oxides and without H_2O- (Rayner and Brown, 1973):

$$MgO,\ 29.18;\ Al_2O_3,\ 2.53;\ FeO,\ 6.23;\ SiO_2,\ 62.09.$$

These values give the following calculated formula for the fibrous talc:

$$(Mg_{0.757},Fe^{2+}{}_{0.120},Al_{0.083})_6Si_8O_{20}(OH)_4. \qquad (2)$$

The fibrous talc was heated dry in an electric furnace for 8-hr periods at successively higher temperatures, from 600° to 1300°C. After heating for 8 hr at the desired temperatures, the samples were left to cool overnight. Samples heated in this manner were crushed and prepared by the suspension method on a microgrid (perforated film) reinforced with carbon for TEM. The particles spanning the holes, without a supporting film underneath, were observed and photographed.

A Siemens Elmiskop 101 electron microscope equipped with a pointed filament was used at 100 kV. The microscope was modified to decrease both spherical and chromatic aberrations by employing a "pole-piece-in-polepiece" system (Yada and Kawakatsu, 1976) and by increasing the excitation current of the objective lens. A lattice resolution of 2.4 Å could be achieved with these instrumental improvements.

RESULTS

The fibrous talc occurred as long and thin laths, typically 0.1–0.3 μm wide and 100–500 Å thick (Figure 2A). An indexed SAD pattern containing a well-defined hexagonal arrangement of spots is shown in Figure 2B. The crystal orientation excludes the basal reflection because the crystal is lying with its (001) plane on the support film. From the SAD patterns, the following unit-cell parameters were calculated: a = 5.26; b = 9.16 Å. From the SAD pattern of a lath tilted nearly perpendicular to the support film, an interplanar spacing d = 18.84 Å was obtained (using a β angle of 99°), which gave c equal to 19.06 Å.

At temperatures <700°C the talc laths did not change morphologically. In the 500°–700°C temperature interval, however, the b parameter decreased slightly and the a parameter increased slightly (see Figure 3), indicating some changes in the talc structure, reported by Mackenzie (1957) as "gradual breakdown."

After firing the sample at 800°C, very definite striations were noted in the image (short arrow in Figure 4A). The SAD pattern of this material showed faint streaks containing spots (small arrows in Figure 4B). As shown in Figure 3, the b value of talc clearly decreased and the a value increased slightly to maximum. These observations indicate that firing at 800°C resulted in an initial transformation of the talc. According to Mackenzie (1957), dehydroxylation of talc starts at 800°C and ends at 900°–1000°C, depending on the sample.

At 900°C the striations found in the crystal fired at 800°C, appeared to be crystallites (Figure 5A). The corresponding SAD pattern (Figure 5B) consisted of reflections from a newly formed phase (circles), in addition to those from talc (indexed). The new phase had the following unit-cell parameters: a = 18.2; b = 8.8; c = 5.2 Å, which are compatible with the orthorhombic structure of enstatite, the XRD data for which were reported by Brindley and Brown (1980, p. 405). The star-shaped electron diffraction patterns suggest that the laths, after firing at 900°C, were composite particles containing various proportions of undecomposed talc plus orthorhombic enstatite, and that a well-defined orientation relationship existed between these two

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Figure 1. (Upper left) Photograph of talc from Jeffrey Mine, Asbestos, Quebec, in which central vein B is fibrous talc and A and C are massive talc. Graph paper smallest grid = 1 mm.

Figure 2. (A) Transmission electron micrograph of long thin lath of talc. (B) Corresponding indexed, selected-area diffraction pattern showing well-defined hexagonal arrangement of spots.

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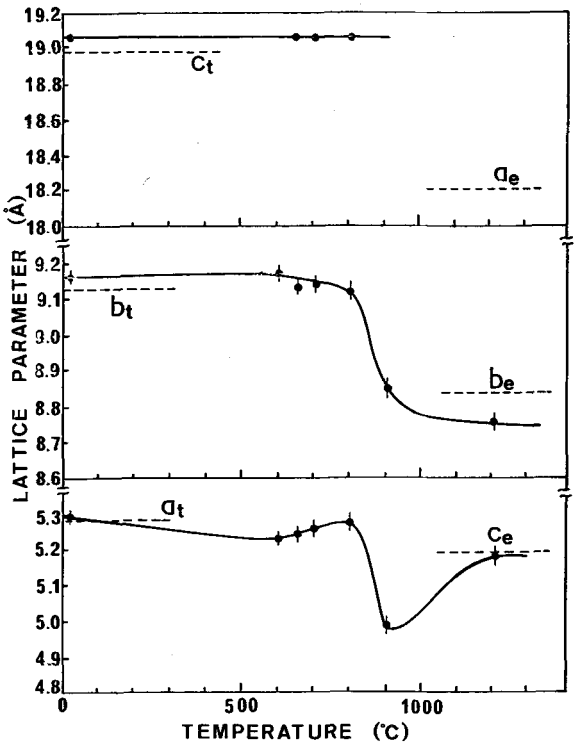


Figure 3. Lattice parameters of talc vs. temperature. Subscript "t" = talc; subscript "e" = enstatite. The b_t value of the original talc parameter maintains its individuality to 700°C; at >800°C, it shows an accentuated decrease, mainly between 800° and 900°C; at 900°C the b_t parameter decreases to the value of the b_e parameter of enstatite. The a_t parameter expands slightly (at 700°C) followed by an accentuated decrease from 800° to 900°C, to rise to the value of the c_e parameter. The c_t parameter is nearly unchanged to 800°C, transforming to the a_e of enstatite. Dashed lines = literature values (Brindley and Brown, 1980, p. 40).

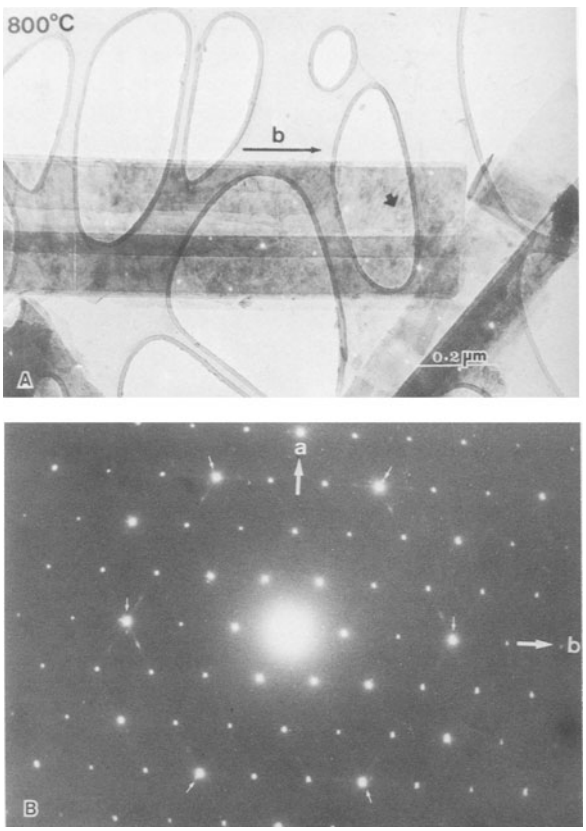


Figure 4. (A) Transmission electron micrograph of talc showing that at 800°C definite striations are present in the laths (short arrow). (B) Corresponding selected-area electron diffraction pattern showing faint streaks and spots (small arrows).

minerals. The crystallites of enstatite present at this temperature (900°C) were disposed in three directions at 60° to each other (Figure 6A) within noncrystalline regions, as indicated in dark field images. Individual crystallites were about 50–100 Å in diameter and 1000–2000 Å in length; the corresponding SAD pattern (Figure 6B) shows the crystallinity of such particles.

At 1000°C the crystallites were wider and shorter, forming strips that tended to coalescence (arrow in Figure 7A); accordingly, the SAD pattern contained weaker streaks (Figure 7B). Figure 7C clearly shows the lattice image of the crystallites (Cr) among noncrystalline regions (Am). Figure 8A (1100°C) shows thick crystallites inside the laths and scarce noncrystalline regions. The lattice image of one crystallite (Figure 8C) clearly shows 4.4-Å fringes, corresponding to the 020 reflections of enstatite. In the corresponding SAD pattern (Figure 8B) the streaks are faint and the pattern is formed almost exclusively by the reflections of enstatite. At 1200°C the crystallites were relatively thick, and noncrystalline regions were not observed (Figure 9A); the three-directional crystallites of enstatite were unified to a single direction (a axis). Figure 9B shows the corresponding SAD pattern.

The composite SAD pattern shown schematically in Figure 10 represents the patterns of talc and talc heated at 900° and 1100°C. This pattern shows that the crystallographic axes and unit-cell parameters of the talc and enstatite are topotactically related as follows:

$$a_t \text{ (5.3 Å)} \;//\; c_e \text{ (5.2 Å)}$$
$$b_t \text{ (9.16 Å)} \;//\; b_e \text{ (8.8 Å)}$$
$$\text{d}(001)_t \text{ (18.84 Å)} \;//\; a_e \text{ (18.2 Å)},$$

where t and e signify talc and enstatite, respectively.

The changes in lattice parameters are summarized in Figure 3. The b_t parameter maintained its individuality as high as 700°C; above 800°C, b_t decreased sharply, and at 900°C it was the same as b_e. The a_t parameter increased slightly to 700°C and decreased sharply from 800° to 900°C. At 900°C, $a_t = c_e$. The c_t parameter was nearly unchanged <800°C, at which temperature it was nearly equal to a. At 1300°C, two

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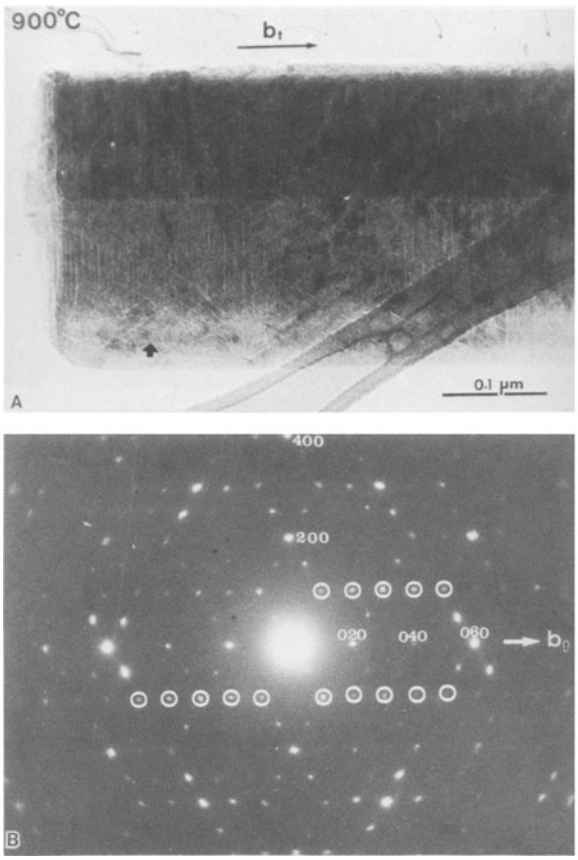


Figure 5. (A) Transmission electron micrograph of talc heated at 900°C showing crystallites inside laths (arrow). (B) Corresponding selected-area electron diffraction consists of reflections from talc (indexed) and enstatite (circles).

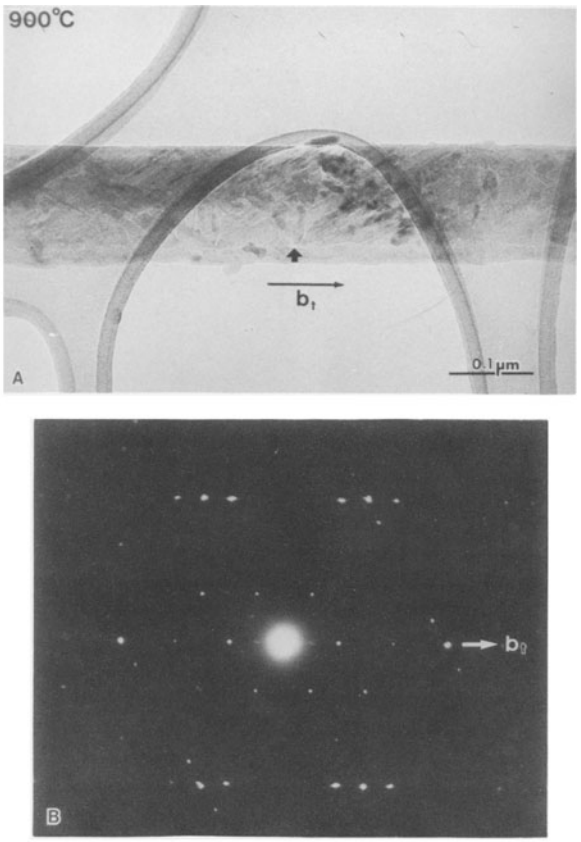


Figure 6. (A) Transmission electron micrograph of talc lath heated at 900°C. Note crystallites inside the original lath in three orientations, making 60° angles with each other (short arrow). (B) Corresponding selected-area electron diffraction pattern demonstrating the crystallinity of such particles.

crystalline phases were present: enstatite and cristobalite. According to Eq. (1) the molar amount of enstatite should be three times that of cristobalite. Actually, the cristobalite reflections were fewer than those of enstatite and were rather difficult to detect; however, they could be seen in some SAD patterns. Daw *et al.* (1972) experienced the same difficulties.

DISCUSSION AND CONCLUSIONS

Thermal transformation

At temperatures <800°C, the talc laths did not change morphologically. Their SAD patterns were almost undistinguishable from those of the original unheated particles. At 900°C, enstatite formed from the talc. Inasmuch as dehydroxylation occurred between 800° and 900°C, dehydroxylation and the enstatite formation were not simultaneous, but occurred without the formation of an anhydrous form of talc as an intermediate phase.

Morphologically, the enstatite formed from talc at 900°C occurred as narrow strips (about 50–100 Å in width) of crystallites (ec) parallel to the *a* axis, as well as parallel to the two pseudo-*a* axes (pa) (Figure 11). This fact explains the appearance of three directional streaks in the star-shaped electron diffraction patterns. SAD patterns of samples heated between 900° and 1100°C were, therefore, the product of the superposition of narrow enstatite crystallites disposed in three directions rotated by 60° to each other.

Ball and Taylor (1963), using X-ray diffraction techniques to study talc single crystals, found that enstatite formed in only one of these three possible orientations. Nakahira and Kato (1964) suggested that the pronounced symmetry of the diffraction patterns of thermally treated talc was due to an equal probability of the *a* and the pseudo-*a* axes of talc being transformed to the *c* axis of enstatite. Daw *et al.* (1972) also reported that enstatite produced by heating talc occurred in only one of the three possible orientations and suggested as a cause the lattice misfit in relating the parent and product phases. In contrast with the findings of Ball and Taylor (1963), Nakahira and Kato (1964), and of Daw *et al.* (1972), the present study found that the enstatite crystallites were disposed in all three possible

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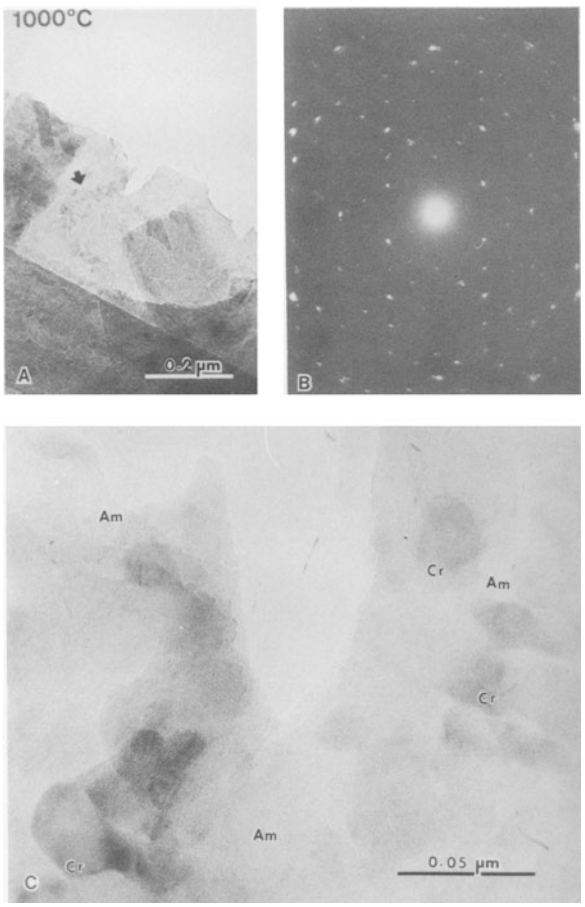


Figure 7. (A) Transmission electron micrograph of talc heated at 1000°C. Enstatite crystallites are wider and shorter (arrow) than those shown in Figure 6. (B) Corresponding selected-area electron diffraction pattern shows weaker streaks. (C) Figure (A) at higher magnification showing the crystallites (lattice image Cr) among noncrystalline ("amorphous") regions (Am).

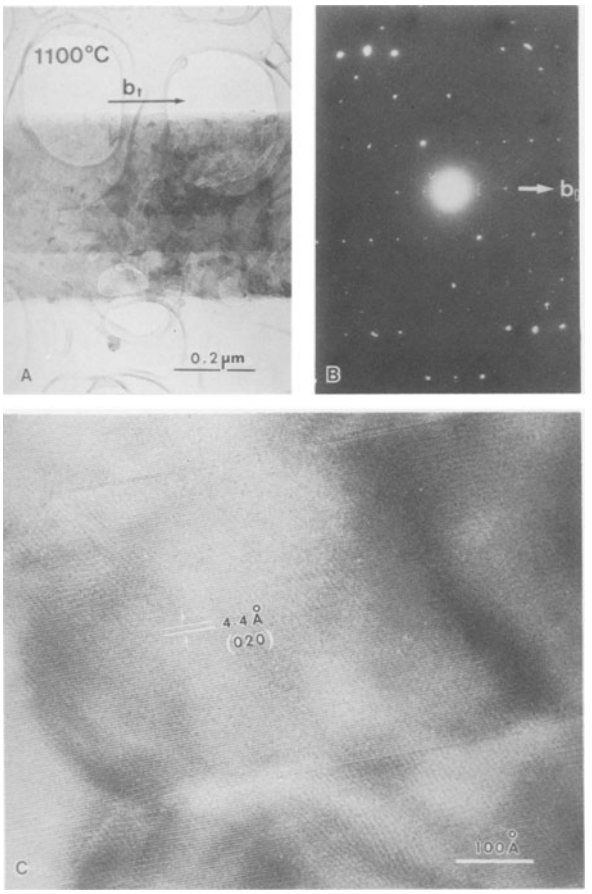


Figure 8. (A) Transmission electron micrograph of talc lath heated at 1100°C. Enstatite crystallites are present among scarce noncrystalline regions. (B) Corresponding selected-area electron diffraction, showing faint streaks. Pattern is formed almost exclusively by enstatite reflections. (C) Lattice image of one crystallite showing 4.4-Å fringes corresponding to the enstatite 020 reflection.

orientations, probably due to the high resolution of our electron micrographs.

Daw *et al.* (1972) reported that heating talc between 875° and 900°C produced numerous small voids, which were disposed preferentially along dislocations in the talc crystals. On further heating crystallites formed adjacent to the voids, they concluded that the dislocations formed preferential sites for the nucleation. Voids or pores were not observed in the present work; however, after the talc had been heated at 800°C definite striations were noted in the TEM images. Following Daw *et al.* (1972) the appearance, nucleation, and development of enstatite crystallites with streaking in the corresponding SADs seem to have been directly related to striations in the talc laths.

At temperatures >1200°C, the enstatite crystallites grew and their three directions of growth were unified to just one. The individual enstatite crystallites (50–100 Å) were much smaller than those (500 Å) reported by Daw *et al.* (1972) at the same temperature. The results of the present work agree with those of Daw *et al.* (1972) about finding an orthorhombic structure of enstatite for samples heated to 1300°C and cooled to room temperature and also with Foster's results (1951) in which an enstatite crystal heated to 1250°C retained the enstatite structure, even after cooling to room temperature.

Noncrystalline regions among crystallites of enstatite inside the laths were observed in the TEM of samples heated to 900°C. These regions disappeared after the sample was heated at 1200°C, and only enstatite crystallites were observed. Kedesdy (1943) found that between 800° and 900°C talc decomposes, forming enstatite, noncrystalline silica, and water. As enstatite was clearly identified, and the water was eliminated as vapor, it was probable that the noncrystalline regions

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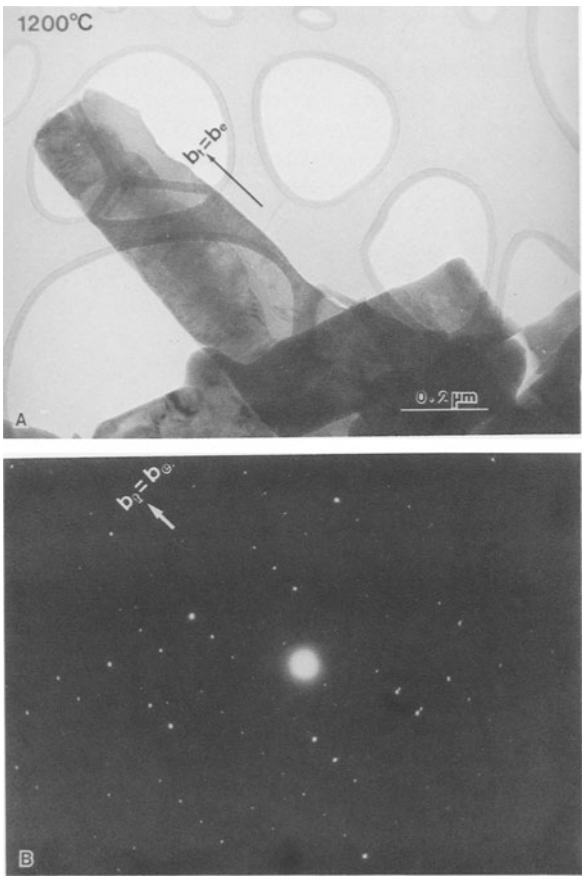


Figure 9. (A) Transmission electron micrograph of talc heated at 1200°C. Enstatite crystallites are much thicker than those shown in Figure 8 and present only one direction; noncrystalline regions are absent. (B) Corresponding selected-area electron diffraction of well-crystallized enstatite.

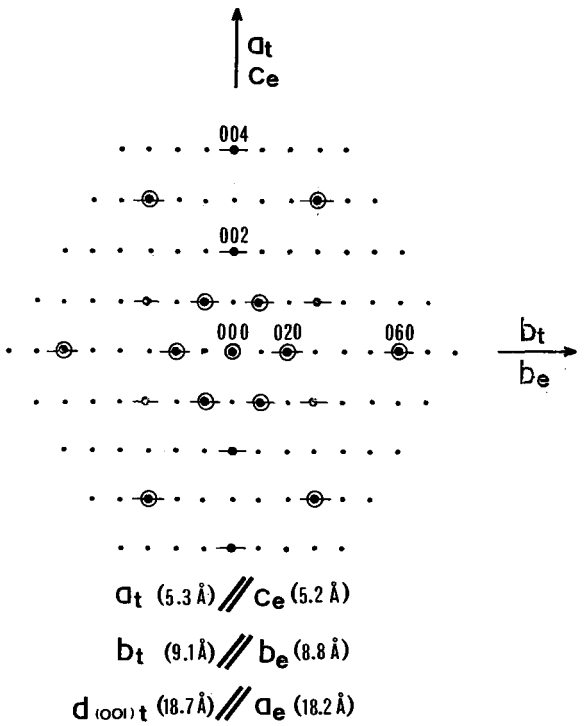


Figure 10. Schematic representation of composite selected-area electron diffraction pattern of talc unheated and heated to 900° and 1100°C. Various topotactical relationships are shown.

were noncrystalline silica, which began to be exsolved at 900°C.

Triple superposition of the electron diffraction pattern in Figure 11 at the three equivalent angles explains the actually observed star-shaped pattern. Figure 11 shows also the topotactic relationship between the talc and enstatite structures: the *c* axis of enstatite and the *a* axis of talc are parallel, the *b* axis of the two structures are parallel, and the *c* (talc) and *a* (enstatite) axes makes a small angle. These results are in agreement with the evidence presented by Nakahira and Kato (1964) and Daw *et al.* (1972). Furthermore, the transformation of talc to enstatite takes place topotactically in relation to the *a* and the pseudo-*a* axis of talc.

Dehydroxylation mechanism

The idea of an inhomogeneous mechanism for the dehydroxylation of minerals was developed independently by Ball and Taylor (1961, 1963), Taylor (1962), and Brindley (1961, 1963b). According to Taylor (1962) the dehydroxylation by an inhomogeneous mechanism proceeds in three stages: (1) cation migration, (2) formation of water molecules in a donor region and their subsequent expulsion, and (3) repacking of the oxygen framework within an acceptor region. The electron-

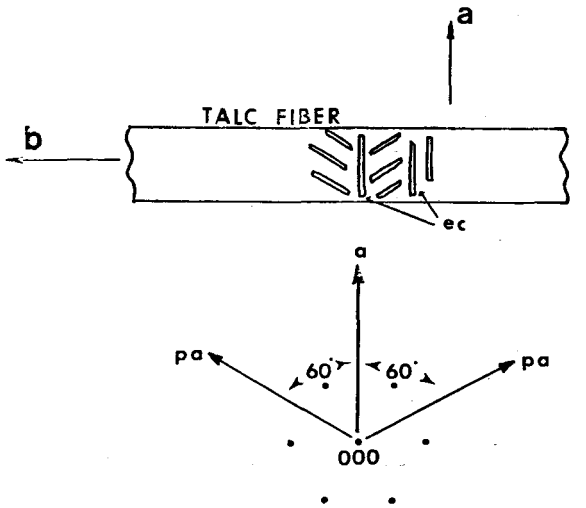


Figure 11. Schematic representation of enstatite crystallites (ec) aligned along the *a* axis and along the two pseudo-*a* axes (pa). A selected-area electron diffraction pattern is thus formed as the superposition of the crystallites disposed in three directions rotated by 60° to each other.

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optical results of the present study are in agreement with such an inhomogeneous mechanism.

Taylor (1962) and Ball and Taylor (1963) stated that the decomposition of talc probably occurs by an inhomogeneous mechanism and that the initial stage of dehydroxylation takes place by the expulsion of water from the donor regions and formation of a partially disordered product. In the scheme suggested by Taylor (1962), this donor region changed into pores. The presence of striations at 900°C (which is the dehydroxylation temperature) in the micrographs of the present study and the absence of pores suggest that these striations are the place from which the high-temperature water vapor is expelled from the dehydroxylated laths. After dehydroxylation at 800°–900°C favorable sites probably develop at the surface of the laths in the striations for the nucleation of enstatite.

The basic assumption of the inhomogeneous mechanism is that no oxygen atom is lost from those parts of talc (acceptor regions) where enstatite is to be formed. The orderly reaction of talc to enstatite reported by Nakahira and Kato (1964) takes place crystallographically with $V_t \cong V_e$, i.e., the number of oxygen anions remains unchanged, and the cationic interchange is represented by $8H \rightleftharpoons 4Mg$. Mg^{2+} cations migrate towards the acceptor region, in which the oxygen framework is maintained practically intact. Therefore, in the transformation of talc to enstatite, almost all the enstatite formed should be topotactically related to the original talc structure. In the present study, not only were all of the topotactical relationships stated by Nakahira and Kato (1964) confirmed, but the enstatite that formed was found to be disposed along the three possible directions of the pseudohexagonal form of talc.

According to Brindley (1983a), in magnesian silicate reactions dehydroxylation and recrystallization proceed simultaneously and the donor regions are left with excess of silica. Whether the loss of water results in a porous texture is not known. In the present study, the dehydroxylation and the enstatite formation were observed within a small temperature interval, which may account for the necessary rearrangement of the migrating cations. The donor regions were not completely destroyed (see Taylor, 1962), but a residue of silica was left and tended to remain disordered, which, under suitable conditions, recrystallized to give cristobalite at >1000°C. Noncrystalline regions among crystallites were observed by the TEM and SAD in the present study, from 900° (the temperature just above the dehydroxylation temperature), to 1100°C. Noncrystalline regions also became progressively scarce. Thus, the liberation of noncrystalline silica probably started at 900°C, but occurred mainly from 1000° to 1100°C. At 1200°C noncrystalline regions were not found; probably the noncrystalline silica was used to form cristobalite, which was detected only at 1300°C.

Thus, the development of the chain structure of enstatite from the layer structure of talc takes place with the orientational relations first reported by Nakahira and Kato (1964). Furthermore, the present work clearly showed that the formation of enstatite follows the directions of the three equivalent axes of talc.

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