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Domain structure of low-symmetry vesuvianite from Crestmore, California

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ABSTRACT

A vesuvianite sample from Crestmore, California, exhibits violations of space group *P*4/*nnc* in selected-area electron diffraction (SAED) patterns. Within the errors of SAED, the patterns are consistent with space group *P*4/*n*. The violating diffraction spots are diffuse with a sharp center and are flattened in the [001] direction, suggesting fine-scale domains. Dark-field and high-resolution electron images reveal a pervasive domain structure that is interpreted to form by transformation twinning resulting from ordering of a disordered, high-temperature structure with space group *P*4/*nnc*. The domains in most regions are approximately 10–50 nm wide and are elongated parallel to [001], but larger domains in some areas exceed 1 μm in size. Rarely, the domain structure assumes a lamellar morphology, with the lamellae parallel to {100}. Convergent-beam electron diffraction patterns from adjacent domains and reversals in dark-field domain contrast with minor changes in orientation suggest that the true symmetry of this vesuvianite is *P*2/*n* or lower. The observation of a fine-scale domain structure in low-symmetry vesuvianite may help to explain difficulties encountered in some X-ray diffraction and TEM studies of these materials.

INTRODUCTION

Many vesuvianite samples possess the ideal space group *P*4/*nnc*, and successful X-ray structure refinements of some vesuvianite samples have been obtained in this space group (e.g., Coda et al., 1970; Rucklidge et al., 1975). As shown by Arem and Burnham (1969), however, other vesuvianite samples exhibit weak to intense reflections violating the various glide plane extinction rules. In addition, some vesuvianite samples are optically biaxial, indicating that they are not even tetragonal. Although relatively successful refinements of some low-symmetry vesuvianites have been obtained in space group *P*4/*n* (Fitzgerald et al., 1986, 1987; Allen, 1985; Giuseppetti and Mazzi, 1983), other attempts at refining non-*P*4/*nnc* vesuvianites in their low-symmetry space groups have been less successful (Arem and Burnham, 1969; Valley et al., 1985; Giuseppetti and Mazzi, 1983). All authors agree that violations of *P*4/*nnc* probably arise by ordering of the partially occupied B and C sites, but the precise ordering patterns are still not clear for the full range of ordered states. Thus, a complete understanding of the vesuvianite structure continues to elude crystal chemists.

Based on X-ray diffraction characteristics of low-symmetry vesuvianites and the observation of diffuseness of the glide-violating diffractions in some cases, most crystallographers agree that most of these specimens must possess some sort of domain structure (e.g., Rucklidge et al., 1975; Allen and Burnham, 1983; Allen, 1985; Giuseppetti and Mazzi, 1983; Valley et al., 1985; Fitzgerald et al., 1986). However, attempts to observe a domain structure directly in non-*P*4/*nnc* vesuvianites using TEM methods have been unsuccessful (Allen, 1985; Valley et al., 1985; unpublished work of Veblen, 1980).

The present TEM study of a vesuvianite specimen from Crestmore, California, shows that there is, indeed, a twin domain structure in at least some low-symmetry vesuvianite. The type of twinning observed also may provide some insight into the reasons why X-ray refinements of some low-symmetry vesuvianite structures have been problematical. A preliminary report of this study was presented by Veblen and Wiechmann (1988).

EXPERIMENTAL METHODS AND SAMPLE DESCRIPTION

Electron microscopy and analytical electron microscopy (AEM) were performed with a Philips 420ST instrument equipped with an Edax energy-dispersive X-ray spectrometer and a Princeton Gamma-Tech System 4000 analyzer, as described by Veblen and Bish (1988) and Livi and Veblen (1987). Samples were prepared by milling fragments of thin sections with Ar ions and then coating them with amorphous C.

The vesuvianite employed in this study is sample W2-85 of Wiechmann (in preparation), from the skarn at Crestmore Quarry, Crestmore, California. This sample was collected atop Sky Blue Hill, which borders the western edge of the quarry. The sample was quite close (approximately 5 m) to the quartz monzonite (QM) intrusion of Burnham (1959). It is thus possible that it experienced higher temperatures than vesuvianite samples obtained farther from the igneous contacts, although vigorous hydrothermal infiltration may have resulted in relatively small thermal gradients throughout the aureole (Wiech-

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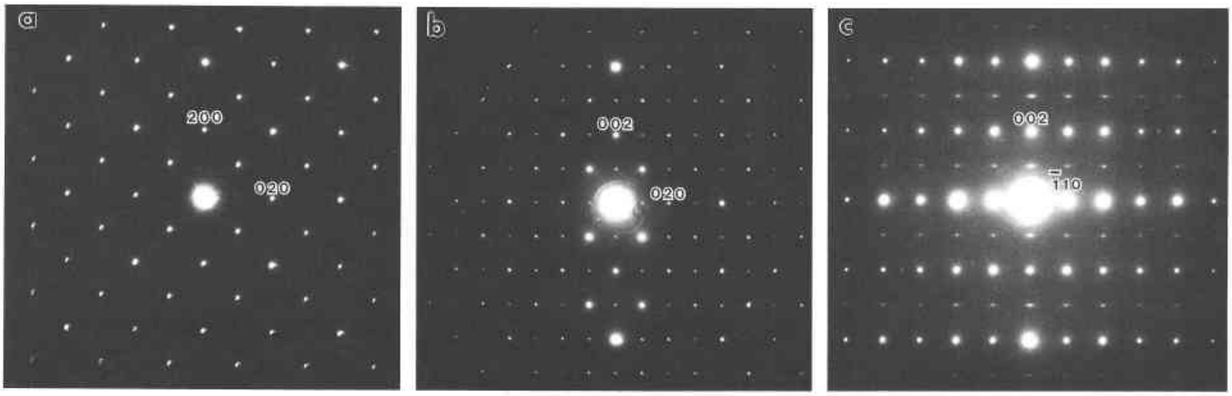

Fig. 1. SAED patterns from W2-85 Crestmore vesuvianite. **(a)** c-axis pattern, **(b)** a-axis pattern, **(c)** [110] pattern.

mann, in preparation). Note that the sample employed in the present study is not the same Crestmore sample as that described by Arem and Burnham (1969), which possessed the high-symmetry space group *P4/nnc*. That Crestmore vesuvianite was a well-crystallized museum specimen, and hence its relationship to the igneous bodies that formed the skarn is unknown.

Sample W2-85 contains vesuvianite, grossularite, wollastonite, a small amount of diopside, and calcite that is probably secondary. Peak metamorphic conditions are estimated to be 825 ± 10 °C, 1.25 ± 0.25 kbar, and X_{CO_2} was approximately 0.01 (Wiechmann, in preparation). Beause vesuvianite in this sample is texturally a second-stage metasomatic precipitate, the temperature of vesuvianite formation may have been somewhat lower than the peak temperature, perhaps in the 780–800 °C range.

The vesuvianite occurs as relatively drab, green crystals of irregular morphology that are approximately 1 mm in diameter. In thin section, the vesuvianite shows colorless to very pale yellow pleochroism and anomalous olive-gray to brown birefringence. It produces extremely poor interference figures, possibly as a result of heterogeneous optical properties, but is optically negative and appears to be weakly biaxial. The vesuvianite has a moth-eaten appearance, having been partially replaced by numerous rounded inclusions of grossularite.

Electron microprobe and AEM analysis show this to be an Fe-bearing vesuvianite. Electron microprobe analyses were acquired using the Carnegie Institution of Washington Geophysical Laboratory MAC probe, natural and synthetic silicate standards, and correction procedures of Bence and Albee (1968). Average microprobe analyses of vesuvianite from this outcrop yield the 50-cation structural formula $Ca_{18.99}(Mg_{2.64}Fe_{0.35}Mn_{0.01})(Al_{8.95}Fe_{0.82}Ti_{0.20})Si_{18.04}O_{69}(OH)_{8.24}$, assuming 3.00 divalent (Mg,Fe,Mn). Crestmore vesuvianite compositions vary along a Tschermak exchange vector $MgSiAl_{-2}$ (Wiechmann, in preparation), but the present sample is from a relatively silica-rich assemblage and does not show this substitution. AEM indicates that the vesuvianite is chemically homogeneous within the resolution and accuracy of the technique.

TEM OBSERVATIONS AND INTERPRETATION

Selected-area electron diffraction

Selected-area electron diffraction (SAED) patterns were obtained with the electron beam parallel to the **c** axis, the **a** axis, and [110]. The **c**-axis patterns (Fig. 1a) show no diffractions with $h + k = 2n + 1$, indicating the presence of a (001) *n*-glide plane. The **a**-axis patterns (Fig. 1b), however, exhibit numerous relatively strong diffractions with $k + l = 2n + 1$, consistent with violation of the {100} *n*-glide extinction rules that are present in space group *P4/nnc*. Similarly, [110] SAED patterns (Fig. 1c) contain numerous strong diffractions with $l = 2n + 1$, which violate the extinction rules for *c*-glides parallel to {110}.

Although these apparent violations theoretically could result from multiple diffraction involving the higher-order Laue zones (HOLZ) (Gjonnes and Moodie, 1965), their strong intensities and the fact that no such violations due to HOLZ effects are observed in the **c**-axis patterns argue against a dynamical diffraction origin. Furthermore, for most regions of the vesuvianite, the glide-violating diffraction spots are diffuse, whereas the other diffractions are sharp (see next paragraph). The diffuse parts of these violating diffractions could not result from dynamical diffraction involving only the sharp diffracted beams. We therefore conclude that the diffractions forbidden by the *c*-glide and the {100} *n*-glides are true violations of space group *P4/nnc* and are not produced by HOLZ involvement in multiple diffraction. Although some SAED patterns show subtle splitting of diffraction spots, the splitting does not form a consistent pattern that could be interpreted as resulting from twinning of a nontetragonal structure. The violations and geometry of the SAED patterns thus appear to be consistent with space group *P4/n*. It seems likely that the violations result from ordering of cations in the B and C sites of vesuvianite, as suggested by several authors (see references in the Introduction).

Figure 2 shows an enlarged portion of the diffraction

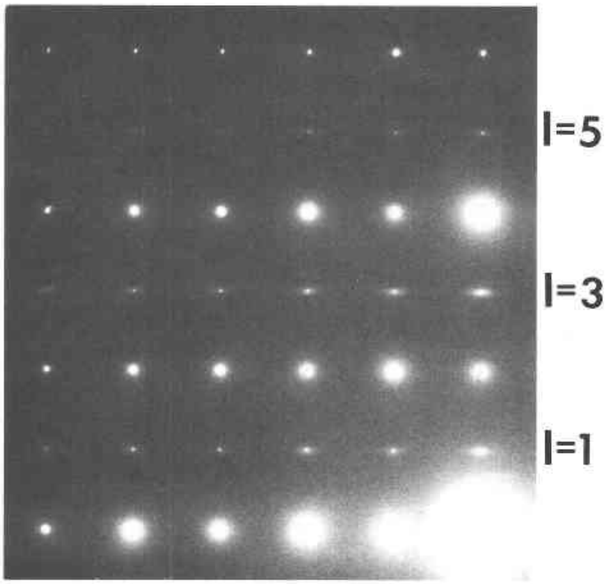

Fig. 2. Enlarged portion of [110] SAED pattern, showing diffuse $l = 2n + 1$ spots with sharp maxima at the ideal Bragg position.

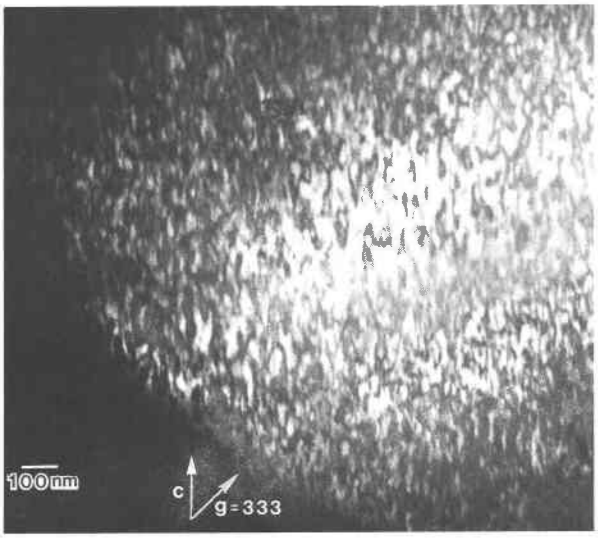

Fig. 3. Dark-field image (**g** = 333) showing fine-scale light and dark domains elongated parallel to the c-axis.

pattern from Figure 1c. The $l = 2n + 1$ spots consist of two parts, a diffuse halo that is elongated normal to $\mathbf{c}^*$ (and hence is normal to **c** in real space) and a sharp maximum at the Bragg position. The diffuse parts of these spots suggest that the glide-violating cation ordering occurs in small domains. Because the domain shape should approximate the Fourier transform of the three-dimensional spot shape (Hirsch et al., 1977, p. 97), this flattening of the spots implies that such domains should be elongated parallel to [001]. The sharp parts of the spots suggest that there are also some larger domains of ordered structure. These inferences from SAED can be tested with real-space TEM observations.

Amplitude-contrast TEM

Dark-field TEM images formed with glide-violating diffracted beams typically show a fine-scale microstructure consisting of light and dark domains (Fig. 3). The domains tend to be elongated parallel to **c**, consistent with the shapes of diffuse spots in SAED patterns. It should be noted that the microstructure is relatively subtle and would be very easy to overlook (it was first noticed only after 6 h of TEM work); failure to observe a domain structure in vesuvianite therefore may not prove that such a structure does not exist. AEM analyses from individual domains show no differences in chemistry, indicating that the microstructure is an antiphase or twin domain structure rather than a structure resulting from exsolution of two distinct compositions.

Parts of the specimen that produce sharp Bragg maxima in the centers of diffuse glide violations show a mixture of fine-scale and larger domains (Fig. 4). SAED experiments show that the sharp maxima arise from the coarse domains, whereas the diffuse violations come from parts of the specimen possessing the finer-scale domain structure. Within some of the larger domains, speckled contrast suggests that there are small domains corresponding to another ordering pattern, although the bulk of each large domain presumably possesses a single ordering scheme in the B and C sites. Although the domains are elongated to parallel to [001] in most parts of the specimen, they do not form a rigorously oriented microstructure. However, over very small regions the domain structure occurs as lamellae elongated parallel to {100}, as shown in Figure 5.

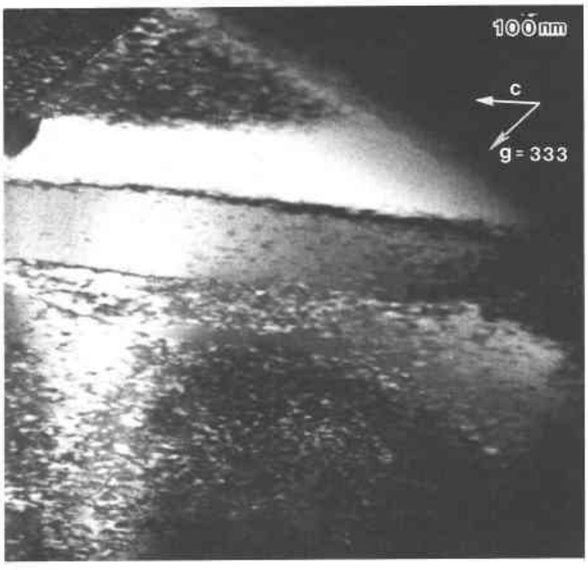

Fig. 4. Dark-field image (**g** = 333) showing coexistence of both fine-scale domain structure and larger domains.

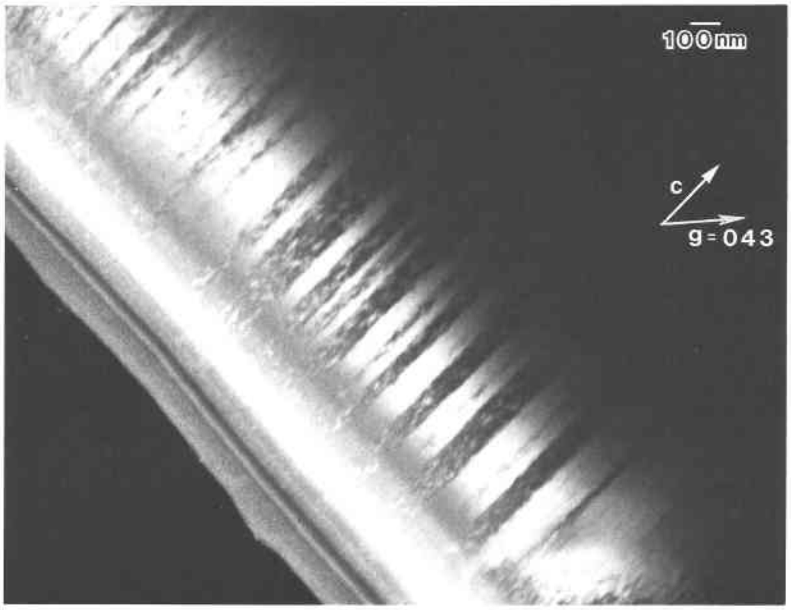

Fig. 5. Dark-field image ($\mathbf{g}$ = 043) showing lamellar domain structure with lamellae parallel to {100}.

Very small (<0.1°) changes in the orientation of the specimen can cause reversal of the contrast in dark-field images (Fig. 6). This suggests that the reciprocal lattice points of the imaged reflections are very slightly split, even though a consistent pattern of splitting could not be observed in SAED patterns. Thus, when the reciprocal lattice point corresponding to one set of domains is in the exact Bragg diffracting condition, that set appears bright in the dark-field image. When very slight rotation of the crystal brings the reciprocal lattice point corresponding to the other set of domains into strong diffracting condition, then that set becomes bright, and the first set of domains becomes dark. The implied, very slight orientation difference between the reciprocal lattices of the domains indicates (1) that the observed microstructure is a twin domain structure, rather than an antiphase domain structure, since the domains have different crystallographic orientations, and (2) that the twin operation does not simply involve a perfectly merohedral overlapping of two tetragonal lattices. A more likely interpretation is that the individual domains possess a symmetry that is lower than $P4/n$ and is most likely a subgroup of that space group, such as $P2/n$, as discussed further below.

Although strong contrast from the domain microstructure is observed with glide-violating diffracted beams, it was found that the microstructure could also be observed in dark-field images formed with certain substructure reflections (the substructure reflections are those that are present for space group $P4/nnc$; i.e., those with $h + k = 2n$, $h + l = 2n$, and $k + l = 2n$). The strongest contrast was achieved by imaging beams that showed large differences in the structure factors for the hkl and $h\bar{k}\bar{l}$ X-ray reflections as determined in the refinement of a low-symmetry vesuvianite structure by Fitzgerald et al. (1986). For example, the magnitude of F_{calc} for 133 of this vesuvianite is 333, whereas F_{calc} for $1\bar{3}\bar{3}$ is 1497 (Sharon Fitzgerald, personal communication, 1988), and strong contrast was observed when dark-field images were formed with the 133 diffracted beam (Fig. 7). This pair of structure factors is required by symmetry to be equal in space group $P4/nnc$ and fortuitously shows a large difference due to the ordering of B and C cations. However, most pairs of substructure reflections that are symmetrically related in $P4/nnc$ have similar observed and calculated structure factors as determined in the refinement of Fitzgerald et al. (1986), and dark-field images formed with these beams show less contrast. In these cases, most of the contrast presumably arises simply from splitting of the reciprocal lattice points due to nonmerohedral twinning. This consistency between contrast observed in our Crestmore vesuvianite and structure factors for the vesuvianite refined by Fitzgerald et al. (1986) suggests that their refinement accurately reflects the ordering in at least some low-symmetry vesuvianite structures and that the single crystal used for their refinement was either a single-domain crystal or contained a large volume proportion of one domain orientation.

In addition to the typical pattern of fine-scale and coarser twin domains, Figure 7 shows another type of defect that was observed rarely in W2-85 Crestmore vesuvianite (indicated by white arrows). These defects appear to be two dimensional and have contrast similar to that from antiphase boundaries. They were never observed to terminate and hence do not appear to be dislocations. Although the twin domain structure can be understood easily in terms of the ordering of B and C cations, it is not clear what feature of the vesuvianite structure could give rise to these additional features.

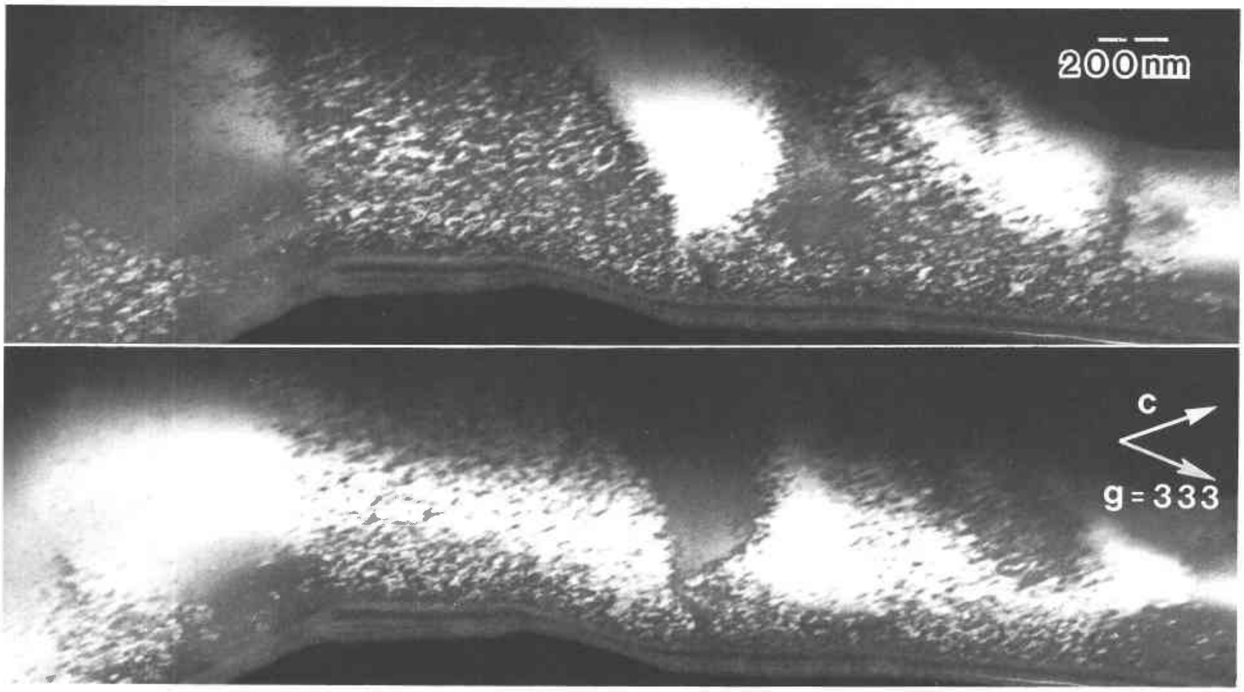

Fig. 6. Dark-field images (**g** = 333) showing contrast reversal that occurs with very slight change in the orientation of the specimen.

Convergent-beam electron diffraction

Numerous convergent-beam electron diffraction (CBED) patterns were observed from adjacent, individual domains in the Crestmore vesuvianite. By moving a fine probe (diameter ≅ 10 nm) across the domain boundaries, very slight differences in orientation were observed between the light and dark domains; the effect was noted for both the fine-scale and coarser domains. Figure 8 shows CBED patterns obtained from adjacent domains without changing the orientation of the specimen. The pattern in Figure 8 (left) is in fairly good zone-axis orientation (i.e., the intensities of hkl and $\bar{h}\bar{k}\bar{l}$ diffracted beams are similar), whereas the pattern from the adjacent domain shown in Figure 8 (right) is clearly out of orientation. Thus, the reciprocal lattices of the two domains have different orientations; this finding is consistent with the imaging experiments discussed above and with the interpretation of the domain structure as resulting from nonmerohedral twinning.

In addition to showing very slight orientation differences between domains, CBED patterns obtained with the domain in good zone-axis orientation exhibit systematic

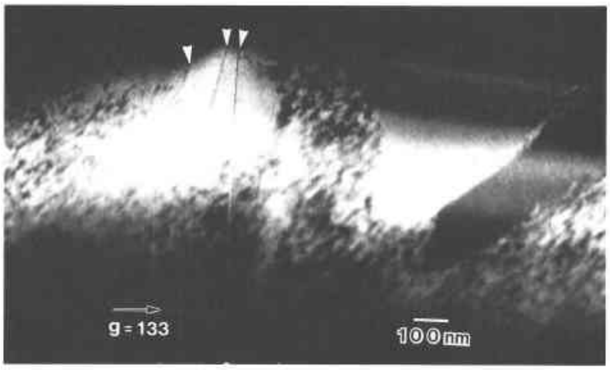

Fig. 7. Dark-field image formed with a substructure diffracted beam having a large difference in structure factor for the two domain orientations (**g** = 133), showing both fine-scale and larger white and black domains. In addition, the arrows indicate fine lines that have contrast similar to that formed by antiphase boundaries.

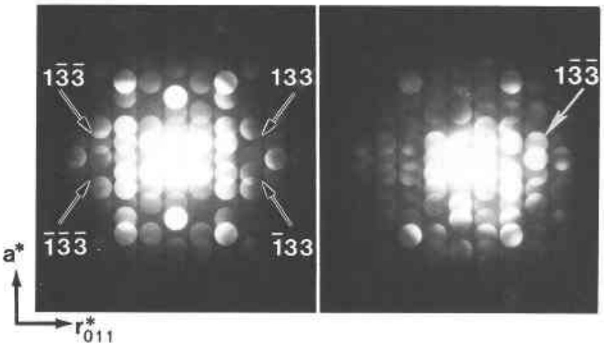

Fig. 8. CBED patterns obtained from adjacent domains, indicating a difference in orientation.

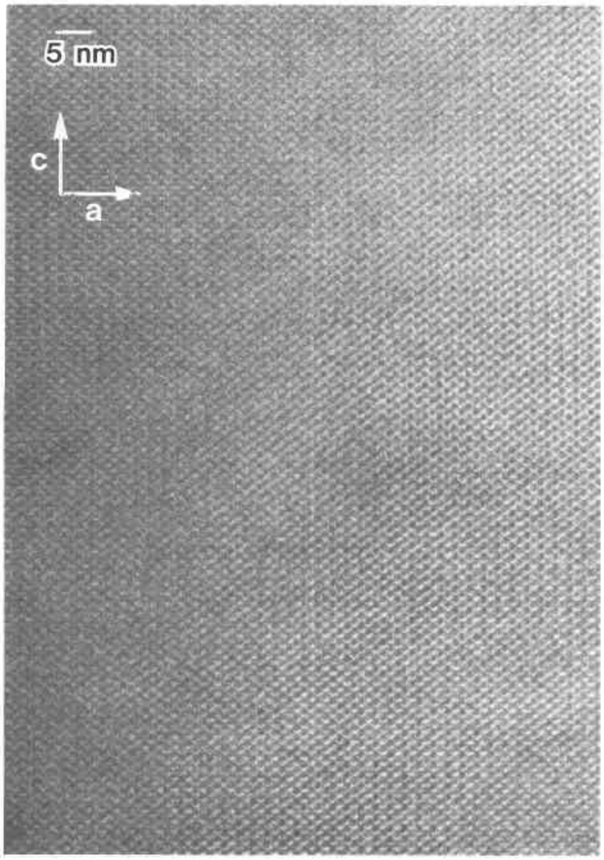

Fig. 9. HRTEM image showing modulations due to ordering and offsets in these modulations between domains. The offsets are best seen by viewing at a low angle parallel to the **c**-axis.

differences in the intensities of certain substructure diffractions that would be symmetrically identical in space group *P4/nnc.* For example, in the pattern of Figure 8 (left), the 133 and $\bar{1}\bar{3}\bar{3}$ spots have relatively low intensity, whereas $1\bar{3}\bar{3}$ and $\bar{1}33$ are more intense. This observation is consistent with the relatively large difference noted above between the 133 and $1\bar{3}\bar{3}$ structure factors as determined in the low-symmetry vesuvianite refinement of Fitzgerald et al. (1986). Note that the intensity of the diffracted beam labelled $1\bar{3}\bar{3}$ is much greater in the CBED pattern from the adjacent domain (Fig. 8, right), but this difference may largely be due to the change in orientation between the domains.

High-resolution TEM

It was also possible to image the domain structure in high-resolution TEM (HRTEM) mode. Figure 9 is an **a**-axis HRTEM image showing modulations in the intensity of the image due to contributions of the $h + l = 2n + 1$ diffracted beams. If the image were formed from only $h + l = 2n$ beams, then the vertical rows of dark spots in the image would all have the same intensity. Instead, alternating rows of dark spots are lighter and darker. This modulated contrast occurs in patches corresponding to the ordered domains in the crystal. By viewing at a low angle parallel to **c**, it can be seen that the modulations are offset in going from one domain to another. This can be interpreted as indicating an offset in the projected ordering pattern, caused by the twinning that forms the domain structure.

DISCUSSION

Symmetry and the crystallographic nature of the domain structure

The above observations and interpretations show that the Crestmore vesuvianite we studied has space-group symmetry lower than *P4/nnc* and possesses a pervasive domain structure, presumably due to cation ordering in the B and C crystallographic sites. This ordering gives rise to observable effects in several different types of TEM experiments: (1) violations of extinction rules for *P4/nnc* and diffuseness of the violating diffractions in SAED patterns; (2) contrast in dark-field TEM images that reveals the pervasive development of both very fine-scale and coarser domains, which typically are irregular in shape but in some small areas form lamellae parallel to {100}; (3) differences in the orientation shown by CBED patterns from adjacent domains; and (4) offsets across domain boundaries in modulations observed in HRTEM images.

The combined evidence from all of these experiments strongly suggests that the domain structure is formed by twinning, since adjacent domains clearly have different crystallographic orientations. Although all diffracted beams produced some contrast from the domain structure in dark-field images, the strongest contrast occurred for beams that violate extinction rules for space group *P4/nnc* or that have large differences in structure factor compared to diffractions that would be symmetrically identical in that space group. This suggests that the twin laws that relate the ordering patterns in adjacent domains are the mirror components of the {100} *n*-glide planes and the {110} *c*-glide planes that are absent in this low-symmetry vesuvianite.

If this low-symmetry vesuvianite were truly tetragonal, or even only metrically tetragonal (e.g., triclinic, but with $a = b$ and $\alpha = \beta = \gamma = 90°$; or monoclinic, with $a = c$ and $\beta = 90°$), then both {100} and {110} mirror twins would result in perfect overlapping of the reciprocal lattice points from adjacent domains, i.e., the twinning would be perfectly merohedral. That this is not the case is shown clearly by both imaging and CBED experiments. It thus appears that the lattice of this Crestmore vesuvianite is not metrically tetragonal, although the deviation from tetragonal shape is too small to be observed directly from distortions or doubling of spots in electron diffraction patterns. This deviation from tetragonal unit-cell shape is consistent with the apparent biaxial optics, although biaxiality requires only a nontetragonal point group, not a unit cell distorted from tetragonal shape.

Because this vesuvianite is not tetragonal, it cannot possess any of the tetragonal space groups in which crystal structure of refinements have been performed for low-symmetry vesuvianites, such as $P4/n$ (Fitzgerald et al., 1986; Allen, 1985), $P4nc$ (Giuseppetti and Mazzi, 1983), or $P\bar{4}$ (Allen, 1985). Since the (001) n-glide plane appears to be present in our specimen, $P2/n$ may be a likely space group, as suggested by Allen and Burnham (1983) and Allen (1985). However, the present data are not capable of resolving the issue of space group definitively, other than ruling out tetragonal groups.

Origin of the domain structure

It is conceivable that the observed domain structure formed during crystal growth. However, the small domain size and relatively homogeneous domain size distribution observed in the regions of fine-scale twinning (Fig. 3) are much more suggestive of transformation twinning. If this interpretation is correct, it seems likely that this Crestmore vesuvianite grew in a disordered state with space group $P4/nnc$. During cooling, one or more phase transitions took place, transforming the vesuvianite to its present, relatively low-symmetry state. At the ordering phase transition(s), the mirror components of the lost glide planes became twinning elements that now relate the observed domains to each other. In addition, the lattice became slightly distorted out of tetragonal shape at some point in the cooling history.

Based on the symmetry elements lost in the phase transition from point group $\frac{4}{m}\frac{2}{m}\frac{2}{m}$ to $\frac{4}{m}$, there should be two domain types. Assuming that a second transition took place from $\frac{4}{m}$ to $\frac{2}{m}$, these two sets should have been further divided into four domain types. Due to the small size of the domains in this Crestmore sample, however, the TEM experiments were incapable of differentiating multiple domain types. In addition, the actual space group of the final, low-symmetry vesuvianite is not known for certain, so that the exact number of domain types and their exact structural relationships remain unresolved.

The coarser twin domains may represent places where the initially fine-scale twins coarsened during continued slow cooling. Alternatively, they may be areas where the crystal grew initially in a partially ordered state. Finally, it is possible that the coarser domains formed during the phase transition(s) and are simply larger for some reason we do not understand.

It is clear from the work of Arem and Burnham (1969) that some vesuvianite from Crestmore possesses space group $P4/nnc$; however, sample W2-85 clearly has lower symmetry. This demonstrates that vesuvianite from the same skarn can occur in both ordered and disordered forms. One possible explanation for the occurrence of the different space groups is that the specimens experienced different thermal histories. Our sample was collected very close to the quartz monzonite intrusion (Burnham, 1959) and, as a result, may have experienced higher temperatures, a slower cooling rate, or a greater fluid flux than other Crestmore samples. Thus, it grew in a disordered state but cooled under the right conditions to order into a low-symmetry space group. Vesuvianite farther from the igneous bodies at Crestmore presumably grew under different conditions and may not have experienced as protracted a thermal history. Since the W2-85 vesuvianite apparently grew at approximately 800 °C, our study suggests that the critical temperature for ordering in vesuvianite is below that temperature.

Previous X-ray and TEM studies of low-symmetry vesuvianites

The present study may help to explain why structure refinements of certain low-symmetry vesuvianite samples have not been entirely successful. If a vesuvianite specimen is pervasively twinned on as fine a scale as our Crestmore material, it clearly will be impossible to obtain an untwinned crystal suitable for single-crystal X-ray diffraction experiments. X-ray intensity data obtained with a twinned crystal will average intensities from the different domains, and the resulting data will not faithfully represent the structure of the individual domains. However, some low-symmetry vesuvianite samples produce intensity distributions and resulting refinements that suggest they may form reasonably good, relatively untwinned single crystals (e.g., Fitzgerald, 1986; the Eden Mills material of Allen, 1985).

Previous TEM studies of low-symmetry vesuvianites have not demonstrated the presence of a domain structure. This may be explained in two ways. First, some low-symmetry vesuvianite samples may grow as ordered, single crystals with little or no twinning. Second, the twinning may occur with an extremely small domain size that is difficult to image in the best of circumstances and may be impossible to observe with some TEM instruments. As noted above, the domain structure elucidated in the present study was not even detected during many hours of initial TEM observations. It is thus possible that some studies in which a domain structure was not found (e.g., Allen, 1985; Valley et al., 1985; Lee Groat, personal communication, 1989) may simply have not detected extremely fine-scale twinning. On the other hand, our own recent attempts to image a domain structure in Eden Mills vesuvianite suggest fine-scale variations in degree of ordering, but no detectable fine-scale twinning. Nonetheless, the present study shows that some low-symmetry vesuvianite samples can possess a fine-scale domain structure, and similar microstructures in other samples may explain some of the difficulties encountered in past studies of vesuvianite.

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