

Omphacites from Greece, Turkey, and Guatemala: composition limits of cation ordering

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Abstract

A combination of transmission electron microscopy with electron microprobe analysis has been used to define the composition limits of cation ordering in omphacites from the blueschist rocks of Syros, Greece, the Mihalliççik region of NW Turkey, and from jadeitic rocks of Guatemala. Systematic absences in reflections of selected-area diffraction photographs show that the probable space group for many of the ordered crystals is $P2/n$. Weak reflections violating the n -glide are also found in some ordered crystals which may have $P2$ symmetry. The occurrence of these extra reflections is not obviously related either to composition or to microstructure. Omphacite replacing sodic augite may have $P2/c$ symmetry. Increasing acmite content seems to allow a broader range of cation proportions to be accommodated by the ordered structures.

Fine-scale exsolution textures in omphacites with compositions close to the limits of the ordered field indicate that any region of immiscibility between ordered and disordered structures is narrow. A much broader miscibility gap has been confirmed for the range jadeite–omphacite by the presence of an ordered precipitate in impure jadeite from Guatemala, but this narrows rapidly with increasing acmite content.

Introduction

Jadeitic pyroxenes in the system acmite–augite–jadeite are common in rocks metamorphosed under the high-pressure, low-temperature conditions of the blueschist facies. The stability relations within this system, however, are not well known. Several crystal-structure refinements (Clark and Papike, 1968; Clark *et al.*, 1969; Curtis *et al.*, 1975; Matsumoto *et al.*, 1975) have shown that cation ordering can occur in pyroxenes with compositions close to Jd:Aug = 1:1 (omphacite), causing a symmetry change from C -face-centered to primitive. Dobretsov (1962) first postulated the presence of a solvus between jadeite and omphacite to explain the absence of naturally-occurring compositions in this range, and other authors have also suggested the existence of miscibility gaps. The summary of analyses by Onuki and Ernst (1969) shows that in fact there is a very wide range of compositions filling most of the system. Bell and Davis (1965, 1966, 1969) identified a broad solvus between jadeite and diopside on the basis of high-

pressure and -temperature experiments, but this has never been confirmed for geological conditions.

Traditional methods of determining the pyroxene/pyroxene relations meet with three problems: (a) direct experiments are too slow at the low temperatures involved; (b) although two or more pyroxenes with different compositions are sometimes found in the same rock they typically show reaction relations indicative of disequilibrium; and (c) the spread of compositions found may merely reflect the whole-rock chemistry or the composition of the fluid phase during metamorphism.

Other mineral systems have sometimes been studied by considering internal structures, such as exsolution. Because of the limited scale of diffusion possible at low temperatures, any such features are unlikely to be visible optically in blueschist pyroxenes. Transmission electron microscopy (TEM) provides the solution to this and has been used by Champness (1973) and Phakey and Ghose (1973) to image antiphase domains arising from the cation-ordering transformation in omphacite. Carpenter (in preparation) reports the presence of complex, fine-scale ($\sim 500\text{\AA}$) exsolution and ordering structures in a range of omphacites from Syros, Greece, which have

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Table 1. Representative electron-microprobe analyses of each pyroxene observed to be ordered

| Locality | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 1 | 2 | 2 | 3 |
|---|--------|-------|-------|--------|--------|-------|-------|--------|-------|--------|--------|--------|
| Specimen No. | 97503 | 97614 | 97647 | 97615 | 97667 | 97636 | 97542 | 97506 | 97700 | 119432 | 119428 | 103484 |
| SiO ₂ | 56.72 | 57.69 | 56.64 | 57.66 | 55.90 | 56.60 | 56.71 | 56.22 | 56.21 | 58.24 | 56.22 | 54.92 |
| Al ₂ O ₃ | 12.01 | 10.68 | 9.33 | 9.96 | 7.37 | 12.57 | 11.19 | 12.61 | 8.39 | 13.42 | 10.19 | 6.16 |
| FeO * | 9.80 | 3.91 | 7.51 | 3.94 | 13.22 | 9.30 | 5.62 | 8.72 | 5.01 | 1.91 | 4.89 | 12.92 |
| MgO | 3.64 | 7.93 | 6.52 | 8.58 | 5.34 | 3.56 | 6.54 | 4.43 | 9.03 | 7.15 | 6.94 | 6.13 |
| CaO | 7.73 | 12.00 | 10.89 | 12.71 | 10.21 | 6.84 | 11.96 | 8.89 | 14.87 | 10.59 | 13.40 | 11.86 |
| Na ₂ O | 10.13 | 7.71 | 8.45 | 7.68 | 8.71 | 10.45 | 7.80 | 9.13 | 5.69 | 8.60 | 7.30 | 7.35 |
| Total | 100.03 | 99.92 | 99.34 | 100.53 | 100.75 | 99.32 | 99.82 | 100.00 | 99.20 | 99.91 | 98.94 | 99.34 |
| Cations per 2 Si ** | | | | | | | | | | | | |
| Si ⁴⁺ | 2.00 | 2.00 | 2.00 | 2.00 | 2.00 | 2.00 | 2.00 | 2.00 | 2.00 | 2.00 | 2.00 | 2.00 |
| Al ³⁺ | 0.50 | 0.44 | 0.39 | 0.40 | 0.31 | 0.53 | 0.46 | 0.53 | 0.35 | 0.54 | 0.42 | 0.26 |
| Fe ³⁺ *** | 0.19 | 0.08 | 0.19 | 0.11 | 0.29 | 0.18 | 0.07 | 0.10 | 0.04 | 0.03 | 0.08 | 0.26 |
| Fe ²⁺ | 0.10 | 0.04 | 0.04 | 0.01 | 0.11 | 0.09 | 0.10 | 0.16 | 0.11 | 0.03 | 0.07 | 0.13 |
| Mg ²⁺ | 0.20 | 0.41 | 0.34 | 0.44 | 0.28 | 0.19 | 0.35 | 0.24 | 0.48 | 0.36 | 0.36 | 0.33 |
| Ca ²⁺ | 0.29 | 0.44 | 0.41 | 0.47 | 0.39 | 0.25 | 0.45 | 0.34 | 0.57 | 0.39 | 0.51 | 0.47 |
| Na ²⁺ | 0.69 | 0.52 | 0.58 | 0.51 | 0.60 | 0.71 | 0.53 | 0.63 | 0.39 | 0.57 | 0.50 | 0.52 |
| EM | 3.97 | 3.93 | 3.95 | 3.94 | 3.98 | 3.95 | 3.96 | 4.00 | 3.94 | 3.92 | 3.94 | 3.97 |
| Jd | 51 | 46 | 39 | 41 | 31 | 55 | 47 | 55 | 37 | 56 | 42 | 26 |
| Ac | 19 | 8 | 19 | 11 | 29 | 19 | 7 | 10 | 4 | 3 | 8 | 26 |
| Aug | 30 | 46 | 42 | 48 | 40 | 26 | 46 | 35 | 59 | 41 | 50 | 48 |
| <i>Localities: 1=Syros, Greece; 2=Motagua fault zone, Guatemala; 3=Mihalliçik region, NW Turkey.</i> <i>Trace amounts of MnO, Cr₂O₃, NiO, V₂O₅, TiO₂ were often present but rarely exceeding 0.3 wt% in total. K₂O not detected.</i> <i>Oxide totals varied between 98.5 and 101.5, and cation totals between 3.89 and 4.01.</i> <i>* Total Fe given as FeO. ** Cation contents recast with Si=2.00. The silica totals may be slightly high, in which case the apparent non-stoichiometry is exaggerated. *** Fe³⁺ calculated as Na-Al.</i> | | | | | | | | | | | | |

evidently developed under metastable conditions.

The ability of omphacite to order its cations will influence the partitioning of elements between it and other crystallizing phases. This may affect the mineralogy of rocks metamorphosed at high pressures, and must be considered when using geothermometers and geobarometers of the type developed by Råheim and Green (1975). To determine the compositional limits of this ordering, a combination of TEM observation and electron microprobe analysis has been used on pyroxenes from Syros (Greece), the Mihalliçik region of NW Turkey, and Guatemala.

Methods

Pyroxenes with a spread of compositions from the blueschist rocks of Syros were selected on the basis of original analyses made by Dixon (1968). All specimens, including the Turkish blueschist and the jadeitic rocks from Guatemala, are in the Harker collection, Department of Mineralogy and Petrology, Cambridge, and are identified in this paper by their

Harker numbers. The pyroxenes were analyzed by an electron microprobe with a Harwell Si(Li) detector and pulse processor (Statham, 1976). Correction procedures follow Sweatman and Long (1969).

Many authors, including Cawthorn and Collerson (1974), Hamm and Vieten (1971), and Essene and Fyfe (1967), have addressed the problem of recalculating Fe²⁺/Fe³⁺ ratios from electron microprobe analyses of pyroxenes. The commonest assumptions are that there are four cations and six oxygens per formula unit. Recent work by Wood and Henderson (1978) has shown that high-pressure pyroxenes may possess significant deviations from stoichiometric proportions, and Cawthorn and Collerson (1974) show that for natural samples the apparent deviations are greatest for jadeite-rich compositions. This makes recalculation of analyses rather difficult, especially as the usual assumptions in this case led to Si > 2.00 per six oxygens. The following scheme has therefore been adopted:

(a) Recast each analysis to Si = 2.00; ΣM (the

cation total) is then typically ≤ 4.00 .

(b) Jadeite = Al.

(c) Acmite = $\text{Fe}^{3+} = \text{Na} - \text{Al}$.

(d) $\text{Fe}^{2+} = \text{Fe}_{\text{total}} - \text{Fe}^{3+}$.

(e) Augite = Ca.

(f) Recalculate to $\text{Jd} + \text{Ac} + \text{Aug} = 100$. These three molecules account for 97 per cent of each analysis.

Representative analyses of each ordered pyroxene examined by TEM are given in Table 1.

To test the accuracy of the probe analyses, standard jadeite and olivine were checked regularly during each analytical session showing errors of $\sim \pm 1$ percent for the major elements. As a further check, the pyroxenes from four samples were separated and analyzed by wet-chemical techniques². In view of the strong zoning in the crystals, precise comparison of the wet-chemical and probe results is not possible; however, the $\text{Fe}^{2+}/\text{Fe}^{3+}$ ratios and all elements but silicon showed reasonable agreement. Silica contents in the probe analyses are consistently slightly high, in which case the apparent non-stoichiometry is exaggerated. It should be emphasized that the proportions of acmite, jadeite and augite are not significantly affected by any error in silicon, since the amount of Al substituted on tetrahedral sites in blueschist pyroxenes is known to be very small. Probably the greatest errors are introduced by the recalculation of $\text{Fe}^{2+}/\text{Fe}^{3+}$ ratios, since errors in Al and Na may be additive. For the purpose of delineating the composition range of cation ordering in the system Jd–Ac–Aug (Fig. 1) the method is adequate. It is unlikely that the ordered field could be more accurately defined using TEM and zoned minerals.

Specimens were prepared for electron microscopy by ion-beam thinning, and observations made with an AEI EM6G microscope operating at 100kV. Bright-field, dark-field, and selected-area diffraction techniques were used.

Observations

Petrology

Dixon (1968) describes the localities and petrology of the Syros blueschists in detail. The pyroxenes examined here are from metagabbros, eclogitic gneisses, and metasomatic blocks in serpentinite. In many cases two pyroxenes are present but they show reaction relations, with one rimming or being replaced by the other. All these rocks are thought to

have experienced the same metamorphic history, though in the metasomatic blocks the later pyroxenes crystallized when the pressure fell below its maximum value (Dixon, 1968).

Blueschists from the Mihalliççik region of NW Turkey were metamorphosed at lower temperatures than the Syros rocks, and contain lawsonite rather than epidote. Minerals and textures belonging to earlier stages in their evolution are often preserved. The single specimen examined (103484) is a metadolerite with relict igneous augite crystals veined by sodic pyroxene. It is considered in detail elsewhere (Carpenter and Okay, in preparation), and it is no. 38/62 of Cogulu (1967), who outlines the general petrology of the Mihalliççik region.

The Guatemalan specimens are from included blocks in the serpentinite of the Motagua fault zone, and have been described by Hammond *et al.* (1977). Nos. 119432 and 119428 (US20 and HU8 of Hammond *et al.*) are massive granoblastic jadeite–omphacite–albite rocks in which pale-green omphacite forms rims on jadeite cores, the latter showing signs of replacement by albite. Foshag (1955, 1957) first described similar rocks in Guatemala. McBirney (1963) and McBirney *et al.* (1967) discuss the local geology of the area. Metamorphic rocks near the serpentinites are schists and gneisses belonging to the greenschist facies.

Microprobe analysis

The complete analyses of 15 specimens are plotted in terms of Jd, Ac, and Aug in Figure 1, which displays a considerable spread of compositions. Pyroxene/pyroxene relations in the Syros rocks are shown in Figure 2, and can be categorized as follows³.

(1) Impure jadeite cores zoning towards jadeite with a sharp compositional break before omphacite rims (97614). In 97503, which is richer in acmite, there is a narrower composition gap between core and rim. In 97496, the rim is of aegirine–augite.

(2) Chloromelanite cores with impure jadeite rims and only a narrow composition break between them (97636).

(3) Impure jadeite zoning to chloromelanite (97667). This specimen also has a second, later chloromelanite which is richer in aegirine–augite.

(4) Aegirine–jadeite zoning towards jadeite with a composition break before chloromelanite rims (97648).

² Analyst J. H. Scoon.

³ The mineral names are those used by Essene and Fyfe (1967).

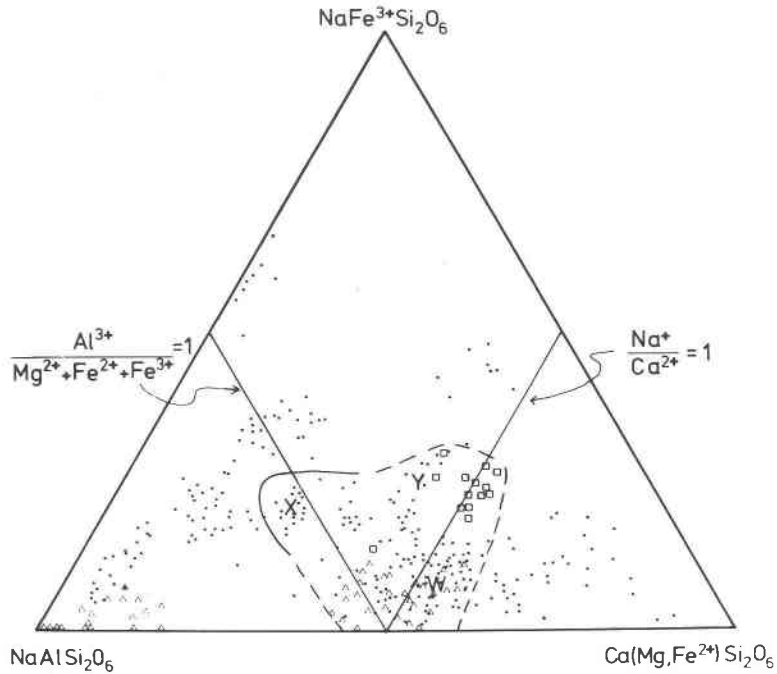


Fig. 1. Electron-probe analyses of 15 pyroxenes from Syros (dots), Turkey (open squares), and Guatemala (open triangles), plotted in terms of jadeite, acmite, and augite. Each point represents one analysis. The field of cation ordering is indicated by a continuous line where it has been determined by the observation of ordered compositions on one side and disordered on the other. A broken line is an inner limit to the ordered field, located by the observation of ordered compositions only. Complex exsolution textures were found in the composition range around W. At X, exsolution is of a disordered phase from an ordered one, and near Y it is of two ordered phases.

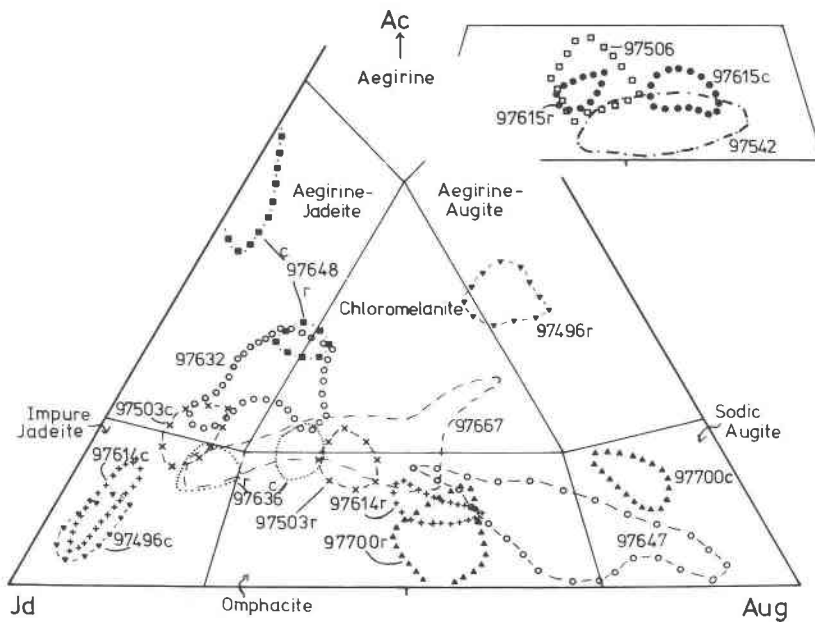


Fig. 2. Pyroxene/pyroxene relations in the Syros blueschists. Each encircled area represents the limits of the pyroxene compositions in each rock. The mineral names are those defined by Essene and Fyfe (1967). c = core, r = rim. Inset is field of omphacite.

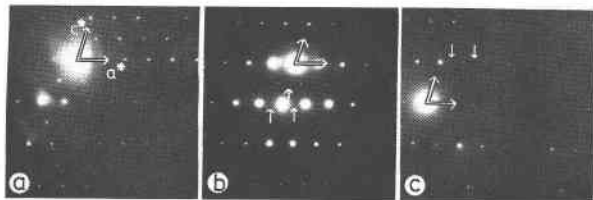


Fig. 3. Selected-area diffraction patterns from ordered omphacite showing a^*c^* sections of the reciprocal lattice. (a) Absences of the type $h+l = \text{odd}$ in $(h0l)$, $?P2/n$. (b) All reflections present with those violating the n glide weak (e.g. arrowed), $?P2$. (c) Absences of the type $l = \text{odd}$ in $(h0l)$, $?P2/c$. $h+k = \text{odd}$ with $l = \text{even}$ are weak (e.g. arrowed).

(5) Sodic augite cores (corroded) with omphacite rims (97700). This specimen has a sharp composition break between core and rim, whereas 97647 is inconsistently zoned back and forth between the two compositions.

(6) Rocks with one pyroxene only: 97615, 97506, 97542 (omphacite); 97632 (aegirine-jadeite). The omphacite in 97615 is occasionally rimmed by a slightly jadeite-richer omphacite.

The Guatemalan pyroxenes are low in acmite content and have impure jadeite cores which zone away from pure jadeite, with a sharp compositional break before omphacite rims. The cores achieve compositions richer in omphacite than their Syros equivalents. Replacement veins in relict igneous augite from the Turkish sample have compositions which lie in the augite-rich corner of the chloromelanite field.

Electron microscopy

Cation ordering was inferred from the presence of diffraction maxima with $h+k = \text{odd}$, indicating a primitive lattice. These reflections could be used to image antiphase domains in dark-field. The precise composition of each area examined was not measured, but the general core/rim relations were known from the microprobe analysis and therefore composition limits for the ordered structures could be placed. These limits are shown in Figure 1, marked as a continuous line where compositions from both sides have been examined. A broken line represents the boundary where it has been assumed for observations and compositions on the ordered side only. The ordered field overlaps from omphacite into chloromelanite compositions.

A number of noteworthy facts emerge:

(1) Ordering was not detected in aegirine-augite with composition $\text{Ac:Aug} \approx 1:1$ or in aegirine-jadeite with $\text{Ac:Jd} \approx 1:1$.

(2) Several different ordering schemes may have been used, as evidenced by systematic absences in a^*c^* selected area diffraction patterns. Absences of the type $h+l = \text{odd}$ in $(h0l)$ are the most common, indicating the existence of n -glide planes perpendicular to $[010]$ in the ordered structure (Fig. 3a). Sharp but weak reflections violating the n glide are also common, however, and all reflections may be present in $(h0l)$ (Fig. 3b). The intensity of these reflections varies, but they have been observed in some areas of almost all the omphacites, and their occurrence bears no obvious relation to the type of microstructure locally. In the area of 97700 where ordered material was replacing disordered sodic augite, and in the finest veins of omphacite in augite of 103484, absences of the type $l = \text{odd}$ in $(h0l)$ were found, indicating c -glide planes perpendicular to $[010]$ (Fig. 3c).

None of the weak reflections could be due to double diffraction in these patterns, because any secondary diffraction from strong reflections would lie in primary reflections of the same type (Gard, 1976).

(3) Exsolution in the Syros pyroxenes was always found associated with ordering structures. Fine, coherent or semicoherent precipitates occur in augite-rich compositions (W in Fig. 1), showing the approximate limit of the ordering field in this range (Carpenter, in preparation). Less complicated structures are present in omphacites with compositions towards aegirine-jadeite (X in Fig. 1), in which one phase was ordered and the other disordered. Chloromelanites just within the ordered field (Y in Fig. 1) contain patchily-distributed composition modulations, approximately parallel to (100) in most cases, in which both components are ordered (dark-field images with $h+k = \text{odd}$ revealed antiphase boundaries in them both, see Fig. 4a,b).

In each of these cases of exsolution the cell dimensions, and therefore the compositions, of host and precipitate are quite similar.

(4) Near the core/rim interface of the impure jadeite regions of 119428 (from Guatemala) precipitation approximately parallel to (010) was found on a scale of $\sim 300\text{\AA}$ (Fig. 4c). The minor component has distinctly larger cell dimensions than its host and also gives $h+k = \text{odd}$ diffraction maxima. This microstructure is interpreted as having formed by the precipitation of ordered omphacite from impure jadeite.

(5) No evidence for a miscibility gap was found to explain the composition break in the zoning of the aegirine-jadeite in 97648.



Fig. 4. Electron micrographs of exsolution textures. (a) (100) exsolution of two ordered phases (both with antiphase domains); dark-field, $g = 101$, 97667. (b) Same area as (a); dark-field, $g = 202$. (c) Patchy exsolution of omphacite in impure jadeite, approximately parallel to (010); bright-field, 119428.

Discussion and conclusions

Peak P and T estimates for the Syros metamorphism are ~ 13 kbar and 450 – 500°C , based on the coexistence of zoisite + paragonite + quartz (\equiv lawsonite + jadeite) (personal communication, Dr. J. E. Dixon, Grant Institute of Geology, University of Edinburgh). Clearly, under these conditions there is a wide range of pyroxene solid solution. The presence of antiphase domains in ordered compositions implies that the ordering transformation occurred after crystallization, though the ordering temperature is not known. Exsolution is invariably associated with ordering, and the two processes are probably closely related. A significant miscibility gap between jadeite and omphacite appears to have operated during metamorphic recrystallization, though it narrows rapidly with increasing Ac content, as noted by Dobretsov *et al.* (1971). This gap is confirmed for lower pressures and temperatures by the exsolution of omphacite from impure jadeite across it in the Guatemalan sample. Any miscibility gap for other compositions surrounding the ordered field is rather narrow, a view that is supported by Yokoyama *et al.* (1976) for the omphacite-to-diopside range.

From the shape of the ordered field (Fig. 1) it appears that increasing the acmite content broadens the range of cation proportions which can be accommodated by the ordered structures. Four M-site cations with significantly different ionic radii must be present before symmetry reduction from the usual pyroxene space group can be achieved, since neither aegirine–augite (Clark and Papike, 1968; this study) nor aegirine–jadeite (this study) shows signs of ordering. The ordered field does, however, extend a significant way towards these compositions, following lines where $\text{Na/Ca} = 1$ and $\text{Al}/(\text{Mg}+\text{Fe}) = 1$, which suggests that they represent the ideal end members of ordered solid solutions.

Ordered omphacite has been reported as having $P2$

symmetry (Clark and Papike, 1968; Clark *et al.*, 1969) and $P2/n$ symmetry (Matsumoto *et al.*, 1975; Curtis *et al.*, 1975). The structures proposed by these authors are all very similar, involving complete Al/Mg order but, because of local charge-balance requirements, only partial Na/Ca order. Evidence for both is provided by electron diffraction patterns either with n -glide absences ($P2/n$) or all reflections present ($P2$). In addition, c -glide absences were occasionally found, which would fit $P2/c$. This space group is theoretically possible for pyroxenes (Brown, 1972), and an ordering scheme can be devised to account for it. It is unlikely that all three space groups represent stable ordered states, and the observation of them in different areas of ordered crystals implies disequilibrium. The variety of exsolution and ordering microstructures (Carpenter, in preparation), sometimes involving two ordered phases (Fig. 4a,b), is also not typical of systems developing under equilibrium conditions. It would be unwise to interpret the observations in terms of equilibrium phase diagrams until possible differences between real and ideal behavior have been evaluated. The ordered field merely represents the composition limits within which cation ordering may occur on some basis under blueschist-facies conditions.

Note added in proof

Two of the omphacite samples discussed above come from the same rocks as omphacites studied recently by other workers. JD 951 and JD 1013 of Fleet *et al.* (*Am. Mineral.*, 63, 1100–1106, 1978) and Aldridge *et al.* (*Am. Mineral.*, 63, 1107–1115, 1978) are the same as 97615 and 97647 respectively in this paper. Both contain exsolution lamellae on an electron optical scale.

Acknowledgments

My thanks are due to Dr. M. G. Bown, Dr. S. O. Agrell, Dr. J. D. C. McConnell, and Mr. A. Okay for reading and commenting

on this paper. I am also grateful for advice from Dr. J. E. Dixon, who kindly permitted me to use unpublished information from his Ph.D. thesis. Constructive criticisms in a review by Dr. J. R. Clark were much appreciated. I acknowledge the receipt of a research studentship from the Natural Environment Research Council, which also provided the electron microscope facilities.

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Manuscript received, December 13, 1977;
accepted for publication, August 1, 1978.