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Thermal Studies of Some Minerals, No. 3.
—On Thermal Transformation of Antigorite in Air

By

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with 1 Table, 6 Text-figures, and 4 Plates

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ABSTRACT: The pulverized specimens of antigorite were subjected to the thermal treatments at various temperatures up to 1000°C in the air, and the effects of dehydration together with recrystallization were pursued by means of DTA, TGA, X-ray diffraction, infrared absorption spectroscopy and electron microscopy.

The results obtained evidently suggest that emergence of the shoulder-like endothermic and exothermic peaks on the DTA curve is ascribed to destruction of the super-lattice structure of the original antigorite and production of the "mosaic olivine" during dehydration respectively and that under electron microscopic observation the recrystallized olivines with various shapes showing the specific relationships to orientation of the original mineral, controlled by the position, are discernible.

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I. INTRODUCTION

On the basis of the previous data given by some authors such as ARUJA (1945), ZUSSMAN (1954) and KUNZE (1958) etc., it has been defined that antigorite is characterized with the wavy structure developed along *a*-axis, being distinguishable from the other serpentine minerals. The themes concerning the thermal transformation of various serpentine minerals to forsterite were inspected by BRINDLEY and ZUSSMAN (1957) and the mutual relationships were clarified to certain extent. The processes of dehydration and recrystallization of chrysotile under the dry and hydrothermal conditions were studied by BALL and TAYLOR (1963) and a sort of an "inhomogeneous mechanism" was proposed in contrast to an "alternative mechanism" suggested by BRINDLEY and HAYAMI (1965). On the other hand, that the rate of recrystallization of forsterite is in an inverse relation to that of its dehydration was also pointed out by them (1963a).

According to NAKAHIRA's data (1963) concerning the electric resistance of kaolinite and serpentine (uncertain variety) during dehydration in the air, that of the latter seems to show a rapid decrease, being interpreted by him as a result of increase of the cation mobility. In relation to this, the data for activation energy in dehydration of various kinds of serpentine minerals were given by SHIMODA (1967).

The purpose of this paper is to clarify the mechanism of thermal transformation of antigorite with the super-lattice structure especially in the complicated reactions during dehydration and in its transformation process to olivine and pyroxene by means of some ordinary procedures.

II. SPECIMENS PROVIDED FOR EXPERIMENTS

The specimens collected by Mr. H. FURUI of Shiraishi Lime Co. Ltd., silky in gloss, pale blue in colour and fibrous in habit (longer than 20 cm. in length), were provided for experimtns.

Those grinded to put the grain size in order were dispersed in the distilled water and the flaky ones remained to suspend after an hour were taken out. The X-ray diffraction data together with those given by HESS et al. (1952) are shown in Table 1.

III. EXPERIMENTS

A. DTA AND TGA

About 20 mg. of the specimens were treated at the heating rate of 10°C per minute with use of the DTA and TGA apparatus made by Rigaku Denki Co. .

As is shown in Fig. 1, two kinds of the endothermic peaks are recognized, in which the small one indicates the loss of water in a little amount at about 520°C and the larger appearing at about 600°C to 800°C probably reveals destruction of the antigorite structure in association with dehydration. As was pointed out

TABLE 1. DATA FOR X-RAY DIFFRACTION OF ANTIGORITE

h k l	HESS et al. (1952)*		authors (1972)	
	d(Å)	I/I ₀	d(Å)	I
001	7.30	400	7.21	vs
20 $\bar{1}$	6.95	24	6.85	w
30 $\bar{1}$	6.51	16	6.70	w
—	6.10	6		
401	5.78	8		
810	4.67	6		
020	4.62	7		
910	4.27	4	4.27	w
81 $\bar{1}$	4.01	6	4.00	vw
102,10 $\bar{2}$	3.63	300	3.61	vs
202,302	3.51	24	3.54	w
14.0 $\bar{1}$	2.88	2		
930	2.59	4	2.66	w
17.00	2.57	8	2.56	m
16.01	2.52	70	2.52	m
93 $\bar{1}$	2.46	9	2.45	w
18.00,003	2.42	38	2.42	ms
17.01	2.39	9	2.40	vw
403?	2.35	5		
15.02	2.237	6	2.23	vw
16.02	2.208	7	2.21	mw
83 $\bar{2}$	2.167	22	2.17	mw
16.02	2.150	20		
93 $\bar{2}$	2.126	4		
11.3 $\bar{2}$	2.035	4	2.040	vw
15.0 $\bar{3}$	1.886	3		
15.03	1.830	12	1.835	vw
004	1.815	23	1.815	m
93 $\bar{3}$	1.781	14	1.801	w
10.3 $\bar{3}$	1.755	4		
17.03	1.736	10		
21.31	1.688	2		
14.04	1.584	3		
24.30	1.560	12		
060	1.540	9	1.554	m
24.3 $\bar{1}$	1.535	9	1.538	mw
15.04	1.524	13		

Abbreviations: vs — very strong, ms — moderate strong, m — moderation, mw — moderate weak, w — weak, vw — very weak,

*: A. S. T. M. 7-417, a₀=43.5Å, b₀=9.25, c₀=7.26, β=91°23'

by SHIMODA (1971) the latter is likely to show some complicated patterns accompanied with somewhat shoulder-like peaks appearing at about 660°C and 730°C on the DTA curve. These complicated peaks may point to a faint transformation of the crystal, to which nothing has ever been alluded in the previous literatures.

The DTGA data obtained from the TG curve is illustrated in Fig. 1. The plateau at 650°C to 690°C almost corresponds to the peak at about 660°C on the DTA curve. At about 690°C to 750°C the rate of dehydration again increases and quickly diminishes. Consequently two shoulder-like peaks appearing on the DTA curve are probably ascribed

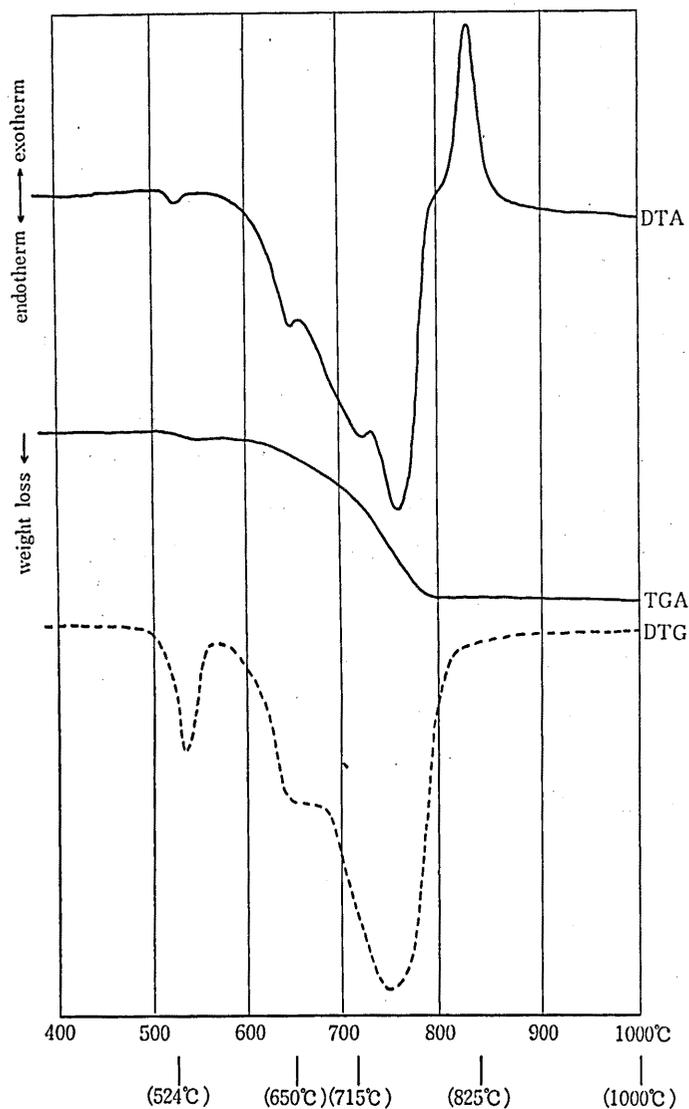


Fig. 1. Thermal variation of antigorite.

to the different kinds of reactions but not to dehydration of the structural water between OH ions on the surface of the octahedral layer in the structure and those jointing the octahedral layers with the tetrahedral ones. The peak at about 660°C may be caused from the endothermic reaction and that at about 730°C from the exothermic reaction occurring in the process of dehydration of the structural water. The last peak at 832°C is also exothermic.

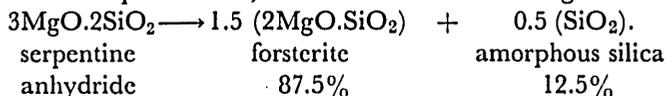
B. X-RAY DIFFRACTION

In order to research the decomposition of antigorite and recrystallization processes, X-ray powder diffraction method was used for the specimens heated respectively at 524, 650, 715, 825 and 1000°C in the furnace of differential thermal analyser and respectively allowed to cool. The results obtained are given in Fig. 2. Some of the same specimens were furthermore subjected to the infrared absorption and the electron microscopy, the data of which are mentioned later.

The diffraction intensity slightly decreases and the peaks reveal a little shift towards the higher-angle side in the case of heating at 524°C. At 650°C, the intensity considerably indicates the decrement and the tendency same as in the former case, where any patterns of olivine or pyroxene are not recognized. At 715°C, the peaks of olivine are observable together with some remainders of antigorite structure. These facts are ascertainable in the data of DTA. Before dehydration of the whole structural water a little exothermic peak appears on the DTA curve probably owing to emergence of the olivine structure in some part of the specimens. At 825°C, the peaks corresponding to antigorite entirely disappear while those of the olivine structure become more stronger and those of the pyroxene are recognized. At 1000°C, those of the latter become slightly strong.

C. INFRARED ABSORPTION

According to the view proposed by BALL and TAYLOR (1963) four stages are discerned in the processes of dehydration and recrystallization of serpentine in the air. The Mg- and Si-rich regions distinguished partially at the second stage assumed to be in relation to cation migration are combined with growth of olivine and that of pyroxene, respectively. On the basis of the opinion enlightened by BRINDLEY and HAYAMI (1965) almost all of MgO contained in the serpentine are consumed for formation of olivine accompanied with release of the amorphous silica, as is shown in the following formula:



The idea alluded to above was proved to be justifiable from the experimental data (formation of 78~80% forsterite).

In view of this, the infrared absorption spectroscopy for the heated specimens was put into operation by the present authors and its relation to the data obtained röntgenographically in the preceding was examined. The specimens dispersed in the liquid paraffin were provided for the experiments within the range from 4000 cm⁻¹ to 200 cm⁻¹ with use of the spectrometer of Perking Elmer 621 type.

As are drawn in Fig. 3, the absorption bands attributed to OH are observed at the temperature lower than 715°C with ascension of the temperature and their values indicate the shift from 3683 cm⁻¹ for the unheated specimen, through 3673, 3665 and to 3650 cm⁻¹

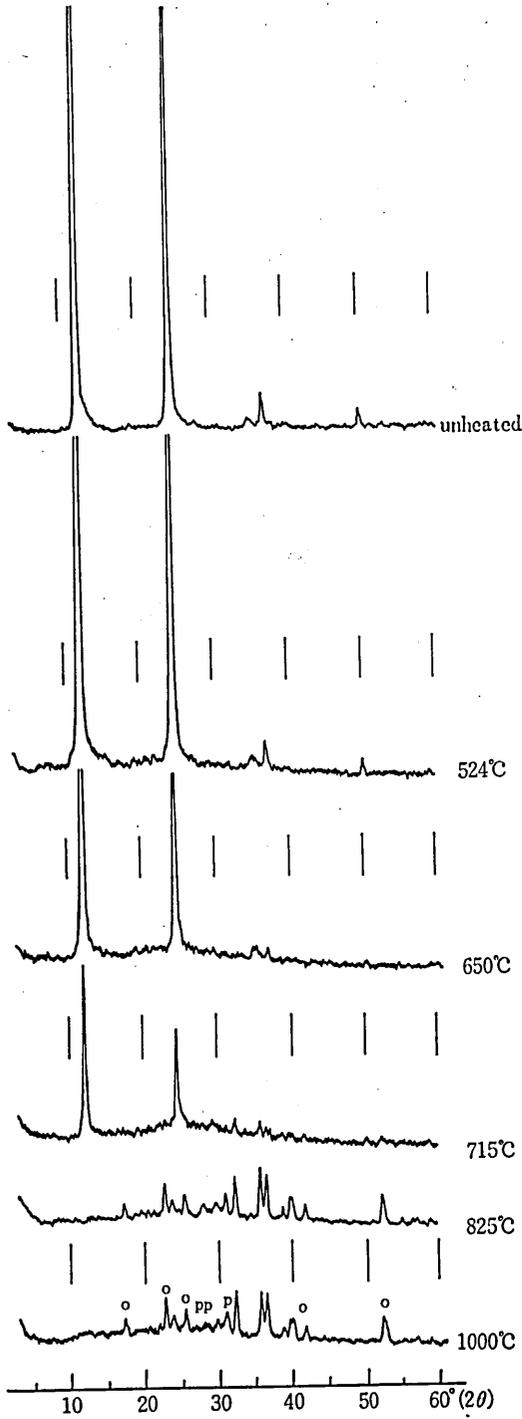


Fig. 2. X-ray diffraction patterns of the specimens obtained after heating.
o: olivine
p: pyroxene

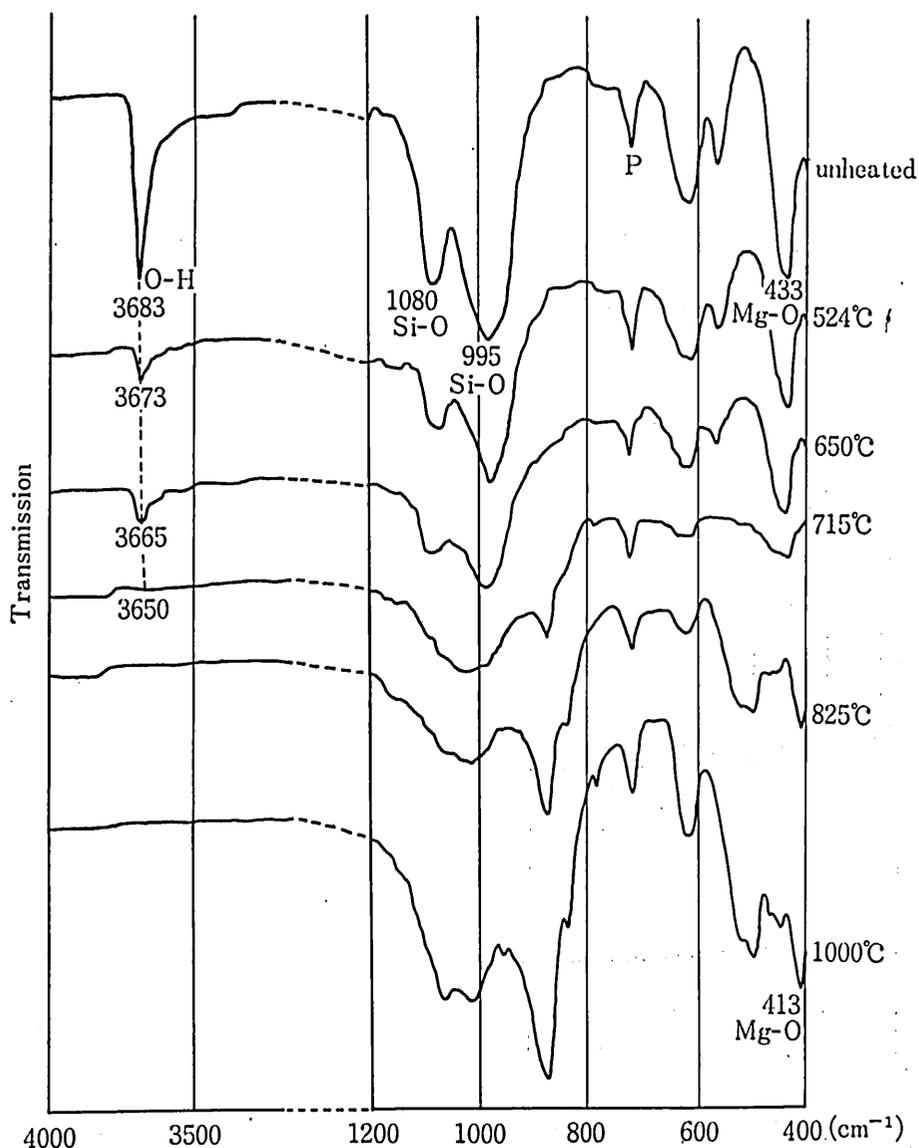


Fig. 3. Infrared absorption spectra of the specimens after heating.
p: paraffin

for those heated at the respective temperature as well as the decrease of intensity. The absorption bands concerned with Si-O appear at 995 and 1080 cm^{-1} for the unheated specimen, while the broad ones appear near 1020 cm^{-1} combined with the shoulder bands at about 995, 1060 and 1080 cm^{-1} for the specimen heated at 715°C. From the result of X-ray diffraction (Fig. 2), it is deducible that this specimen contains antigorite produced in the process of dehydration and olivine formed through recrystallization. Therefore, it seems possible that the shoulder bands at about 995 cm^{-1} together with 1080 cm^{-1} and

that at about 1020 cm^{-1} are related respectively to absorption by antigorite and by olivine, while that at about 1060 cm^{-1} is ascribable to the amorphous substance referred to already by BALL or BRINDLEY, although its form and composition are not yet distinct. At 825°C , 1020 cm^{-1} considered to be combined with formation of olivine at 715°C converts to 1013 cm^{-1} and 433 cm^{-1} related to Mg-O of the unheated antigorite to 413 cm^{-1} . At 1000°C the absorption band concerned with Si-O of enstatite appears at 1063 cm^{-1} .

D. ELECTRON MICROSCOPY

To investigate the complicated patterns on the DTA curve caused by dehydration and recrystallization of antigorite, the same specimens provided for the preceding experiments were observed through electron microscopy.

The mineral specimens in question show the rectangular forms extending along *b*-axis, as is shown in Plate 22-1. As their external forms are still preserved after heating, the variations are traceable. With the heating rate and duration or unevenness of the grain size several states are observable in the specimens heated at certain temperatures. Generally speaking, the results are summarized in the following.

It seems that the remarkable changes other than emergence of the extinction contours developing in the flakes are not recognized for the specimens heated at 524°C , but the lattice-like figures (about 45Å in spacing) derived from formation of the super-lattice structures are discernible, as is displayed in Plate 22-3. At 650°C , the extinction contours become conspicuous, as is evident in Plate 22-4, while the super-lattice images disappear in each flake. It is probable that the strains gradually spread in the flakes with the progress of dehydration. The shoulder-like peak at about 660°C on the DTA curve is ascribed to disappearance of the lattice structure or indicates the endothermic reaction caused by destruction of the wavy structure.

At 715°C , olivine is produced in the comparatively thick flake. As is observable in Plate 23-1, the minerals concerned are recognized nearly in the central part of the flakes and exhibit the "mosaic form" but are not found in the thin flakes or their marginal part. As is revealed in Plate 23-2, the selected area diffraction pattern does not indicate the characteristics of antigorite but apparently those of the lizardite-like structure having the diffused spots along *a**-axis of the original antigortie. The materials with the apparently lower absorption rate than that of olivine are found filling the interstices of the "mosaic olivine". The extinction contours cutting across the "mosaics" disappear in the part of the filling materials. As is shown in Plate 23-3, the diffraction patterns related to the "mosaic olivine" are imperfect. These facts well coincide with the results indicating appearance of the peak at about 735°C on the DTA curve and may clarify that the reaction is exothermic on account of production of the "mosaic olivine" before the complete dehydration of the structural water.

At 825°C , olivine is found formed in each flake and generally exhibits three different kinds of forms developed in the central part of a flake, in its marginal part and on the outside, as is clear in Plate 24-1.

The presence of pyroxene is obscure under the electron microscope but surely confirmable from the X-ray diffraction data. It may be that the mineral in question is produced in the central part and bears a lamellac-like texture. The relation between the "mosaic olivine" at 715°C and three kinds of olivine remains yet to be clarified.

At 1000°C, the forms almost same as those produced at 825°C are only obtainable.

IV. OLIVINE TRANSFORMED THERMALLY

Olivine transformed thermally from antigorite in the air at 825°C and 1000°C are classified into three kinds of forms connected with orientational relationships in relation to the positions of recrystallization such as:

A. central part: This area found in the most part of a relatively thick flake shows the lamellae-like texture with the scale of about 200~400 Å and the angle of few degrees to *a*-axis of the original antigorite, as is obvious in Plate 24-1. The lamellae with such a general trend as is alluded to above often indicate either combination or separation partially and variation in length. The portions with the absorption rate lower than that in the lamellae seem to be composed of pyroxene, though not accurately determined.

The diffraction patterns in the central part are illustrated in Plate 25-1 and its duplicate in Fig. 4. Although these results are remained yet to be analysed, it appears difficult

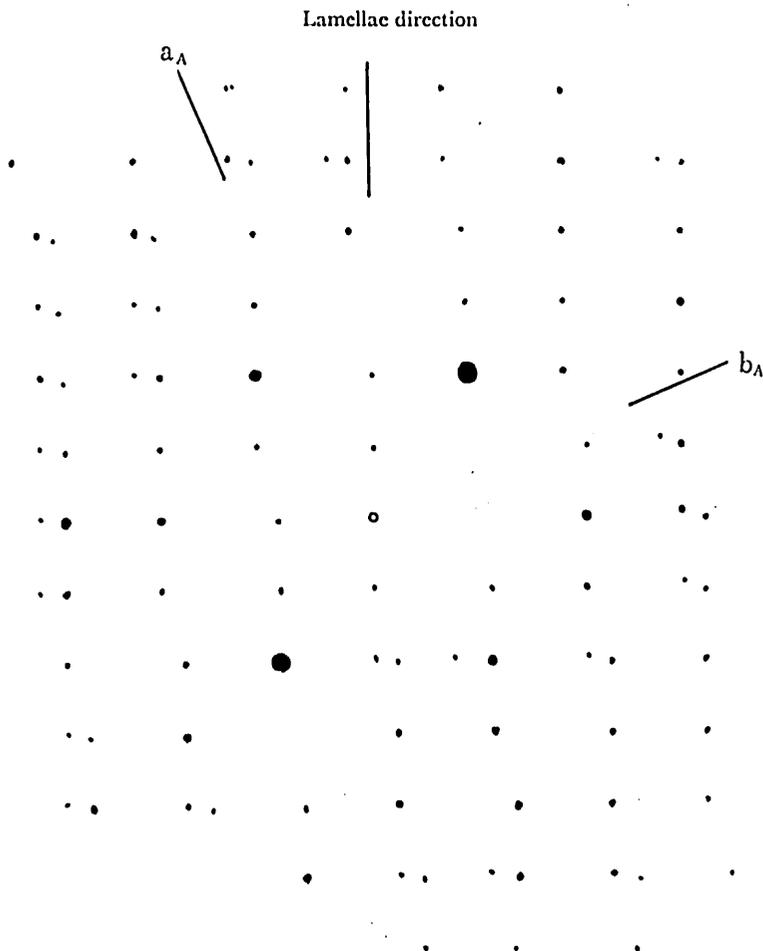


Fig. 4. The selected area diffraction pattern in the central part.

to explain only with the relation of orientation of serpentine to that of its transformed products as were pointed out by BRINDLEY and ZUSSMAN (1957) and BALL and TAYLOR (1963) etc., since one axis of the product, for instance, is not parallel to a - or b -axis of the original antigorite but to the general direction of the lamellae.

B. middle part: This is observed in the confined area on the outside of the central part, bears the texture more homogeneous than that of the central part, as is shown in Plate 24-2, and comprises many of the ameoba-like or subround spots with the size generally becoming smaller from the center to the outside of a flake and with a tendency of the absorption rate lower than that of the matrix.

The electron diffraction pattern of this part is given in Plate 25-2, indicating the reciprocal lattice points on the b^*-c^* plane of olivine, in which the odd ordered lines of k are not recognizable. Orientationally the relation of the olivine in this part to the original antigorite are as follows:

$$a_A // b_O \text{ and } b_A // c_O$$

and in consequence

$$c_A^* // a_O.$$

The subscripts A and O noted above will hereunder be used for the indices referring to antigorite and olivine, respectively.

C. marginal part: Granular olivines with the size of about several hundred Å in diameter are formed on the outermost side or in the relatively thin flake as is observed in Plates 24-1 and 2. The selected area diffraction pattern in this part is shown in Plate 25-3 and the reciprocal lattice points on the b^*-c^* plane of olivines (forsterite) disposed per 60° around a -axis are in Plate 25-4. These two figures closely resemble each other. It is possible to consider that each individual of the granular olivine is formed through rotation per 60° around a -axis and preserves such relation to the original antigorite as:

$$a_A // c_O \text{ and } b_A // b_O$$

and accordingly

$$c_A^* // a_O.$$

This result coincides with that obtained for clino-chrysotile heated under the hydrothermal condition by BALL and TAYLOR (1963).

In the case of the granular olivines produced in the thinner and smaller flakes, it seems common that the minerals in question are arranged mutually with inclination of 60° in angle and concerned with the original antigorite with such relation as:

$$a_A // b_O \text{ and } b_A // c_O$$

and it thus follows that:

$$c_A^* // a_O.$$

The result mentioned above are well agreeable with that given for the same mineral heated in the air by the same authors.

Orientation in the case of formation of olivines in the middle and marginal parts or in the relatively thin flakes is presented in Fig. 5 illustrated by the stereographic projection.

V. DISCUSSION AND CONCLUSIONS

It has become clear that the dehydration process of antigorite in the air is essentially complicated. Although there is a room for finding out the factors, a little amount of dehydration is ascertained before the main dehydration, in the course of which destruction

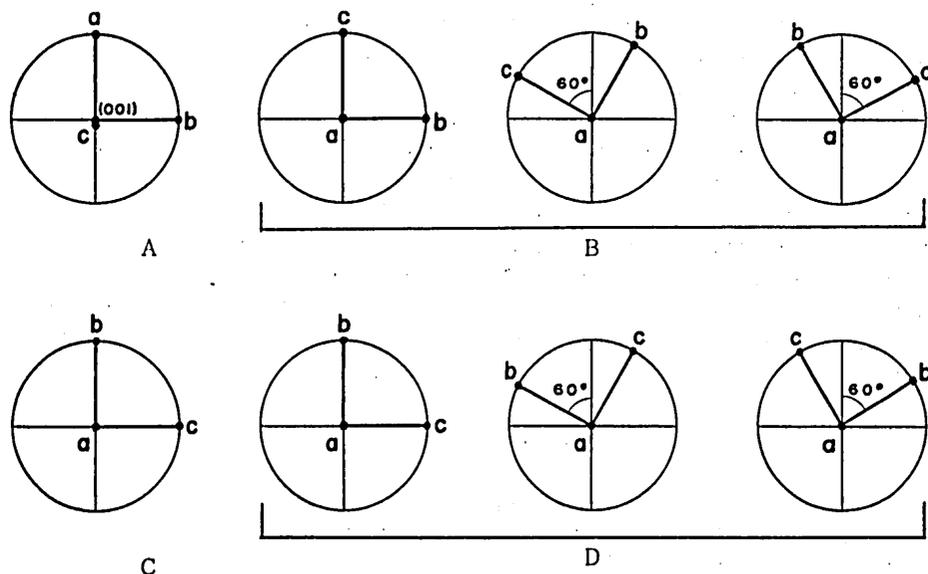


Fig. 5. Stereographic projection of orientation, illustrating the relation of olivine transformed thermally to the original specimen of antigorite.

- A: Original antigorite (monoclinic)
- B: Granular olivine transformed thermally in the marginal part of the antigorite flake (orthorhombic)
- C: Olivine in the middle part of the antigorite flake (orthorhombic)
- D: Granular olivine transformed in the thinner flake (orthorhombic)

of the super-lattice structure of antigorite and recrystallization of olivine ("mosaic olivine") are included. The former may point to destruction of the long-period structure along *a*-axis of antigorite (wavy structure) and conversion of the transitional phase to olivine.

From the data of electron microscopy it is deducible that the dehydration begins first along the periphery of antigorite flakes or specifically in the relatively thin flakes, while the recrystallization takes place in the inner part or in the relatively thick flakes. These facts are consistent with the results proved röntgenometrically for the disc- and block-shaped specimens of serpentine by BRINDLEY and HAYAMI (1963a).

On the other hand, since it seems sure that the portions with the lower rate of absorption for the electron beam are composed of pyroxene, the mineral concerned is likely to be produced neither in the marginal part of antigorite nor in its thinner flakes containing only the granular olivines, but rather in the inner part or in the relatively thick ones. SHIMODA (1967) alluded to no reflections for pyroxene in spite of heating at 1000°C of antigorite indicating its disappearance merely by grinding. According to his view, destruction of the antigorite structure before heating is able to bring about its transformation to the olivine-like structure but not to the chain-like one concerned with pyroxene. In the present study no pyroxene is also found formed in the marginal part or in the thinner flakes, while the olivines produced in both cases show the similarity in shape and orientation. But the latter case is hardly interpretable after SHIMODA's idea, although the relations of olivine to antigorite in orientation are not referred to in his paper.

According to BALL and TAYLOR (1963), the manners of orientation in forsterite trans-

formed from serpentine under the dry condition are controlled by oxygens packed in the octahedral layer of chrysotile and under hydrothermal condition by those in the tetrahedral layer. With respect to orientation the relation of the granular olivines formed in the marginal part to those only found in the thinner and tiny flakes coincides with that of those transformed from the chrysotiles under the hydrothermal condition to those obtained through heating of the latter in the air.

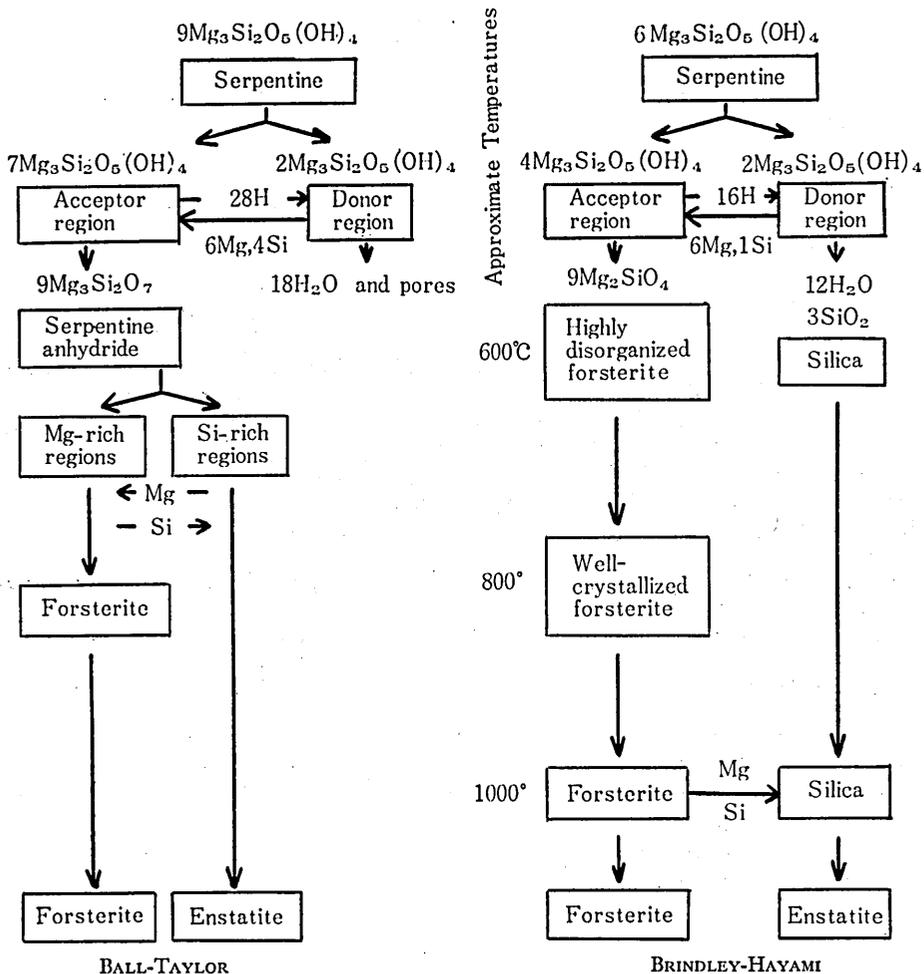


Fig. 6. Comparison of the reaction schemes proposed By BALL and TAYLOR with those given by BRINDLEY and HAYAM*.

As is shown schematically in Fig. 6, two concepts concerning the recrystallization mechanism for serpentine on heating are in contrast to each other. But the disagreement of two opinions may be caused from disregarding the position of recrystallization. The process of recrystallization occurring in the central part of a large antigorite flake almost agrees with the "inhomogeneous mechanism" proposed by BALL and TAYLOR (1963) and

the Mg- and Si-rich regions are formed in antigorite, where the former changes into olivine and the latter into pyroxene. On the other hand, recrystallization in the marginal part or the thinner flakes may be explained by the "alternative mechanism" proposed by BRINDLEY and HAYAMI (1965), because olivine is merely formed up to 1000°C. Moreover, the recrystallization mechanism of antigorite on heating is also considered to be concerned with dehydration of the structural water.

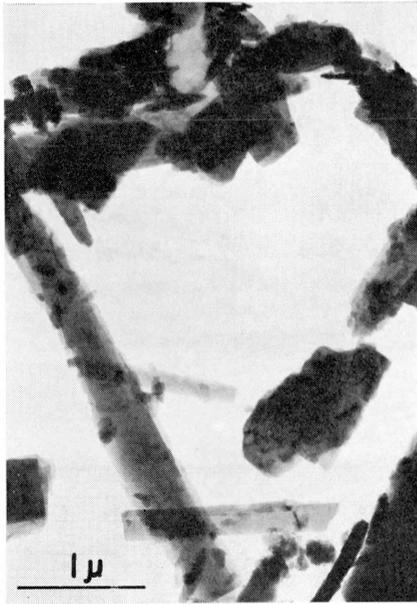
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EXPLANATION OF PLATE XXII

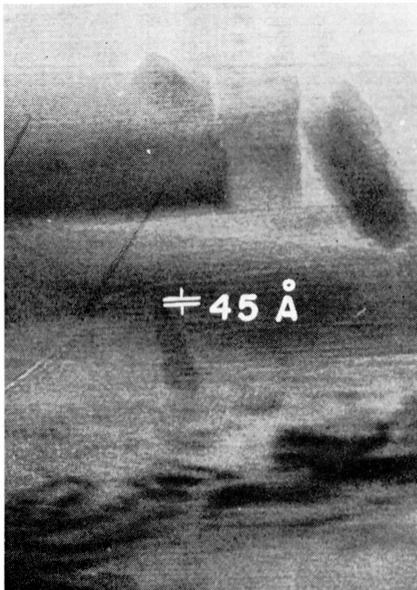
1. Photomicrograph of the unheated specimen.
2. Electron diffraction pattern of the single crystal of antigorite.
3. Photomicrograph of the specimen heated at 524°C. The extinction contours as well as the lattice fringes with the width of about 45 Å are observable.
4. Photomicrograph of the specimen heated at 650°C. Abundance of the extinction contours accompanied with no periodic images are recognizable.



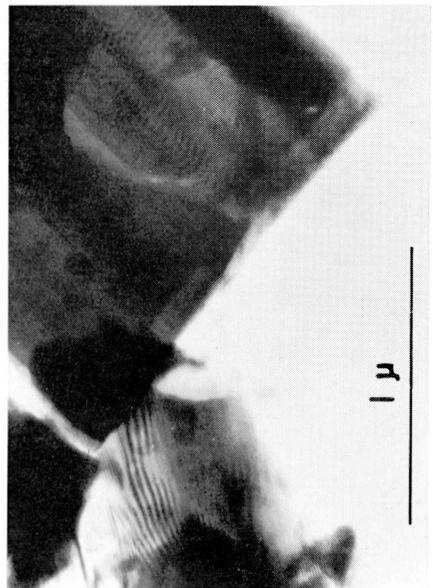
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2



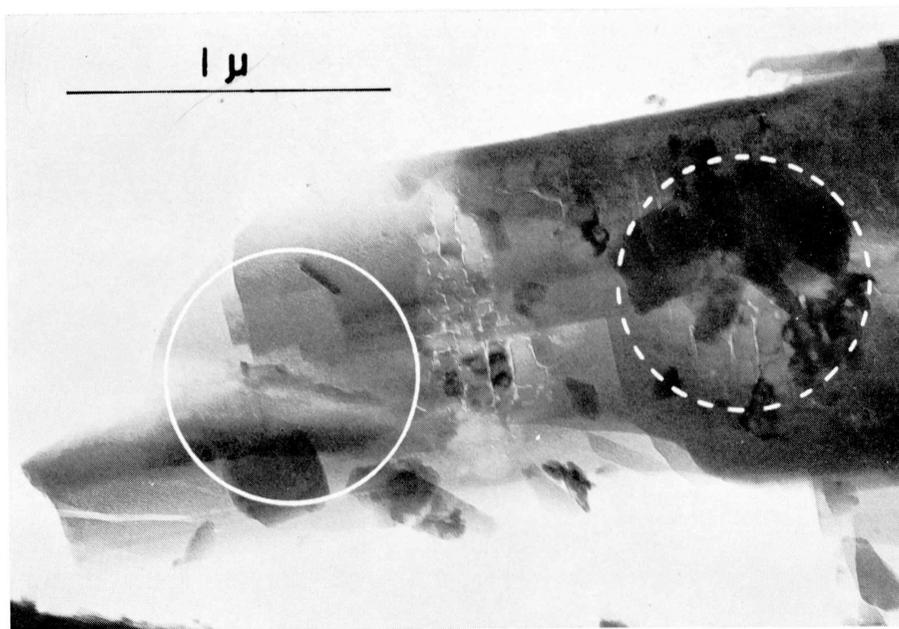
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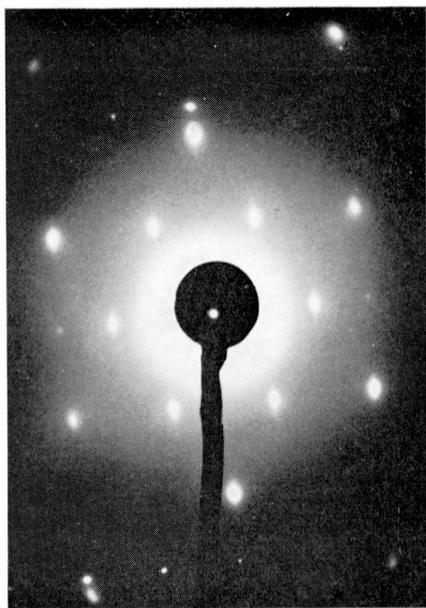
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EXPLANATION OF PLATE XXIII

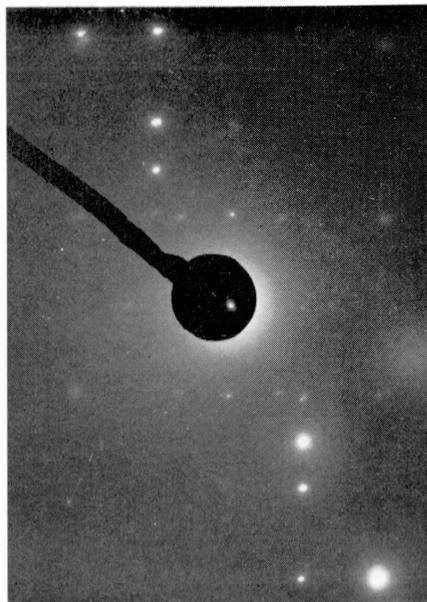
1. Photomicrograph of the specimen heated at 715°C. Formation of the "mosaic olivine" is discernible. The circles of the actual and broken lines respectively point to the parts of the selected area diffraction given in 2 and 3.
2. The selected area diffraction pattern for the part confined within the actual line shown in the photomicrograph. The points of the reciprocal lattice of the lizardite-like structure diffusing along a^* -axis of the original antigorite and the diffraction spots of olivine are also confirmable.
3. The selected area diffraction pattern for the part limited within the broken line. The points of the reciprocal lattice on the b^* - c^* plane of olivine are roughly arranged along the definite orientation.



1



2



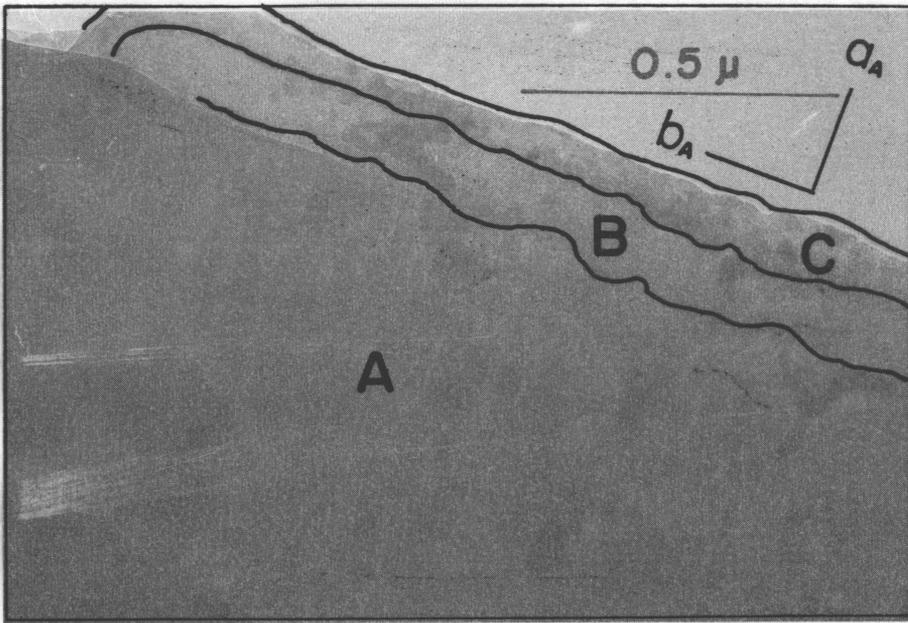
3

EXPLANATION OF PLATE XXIV

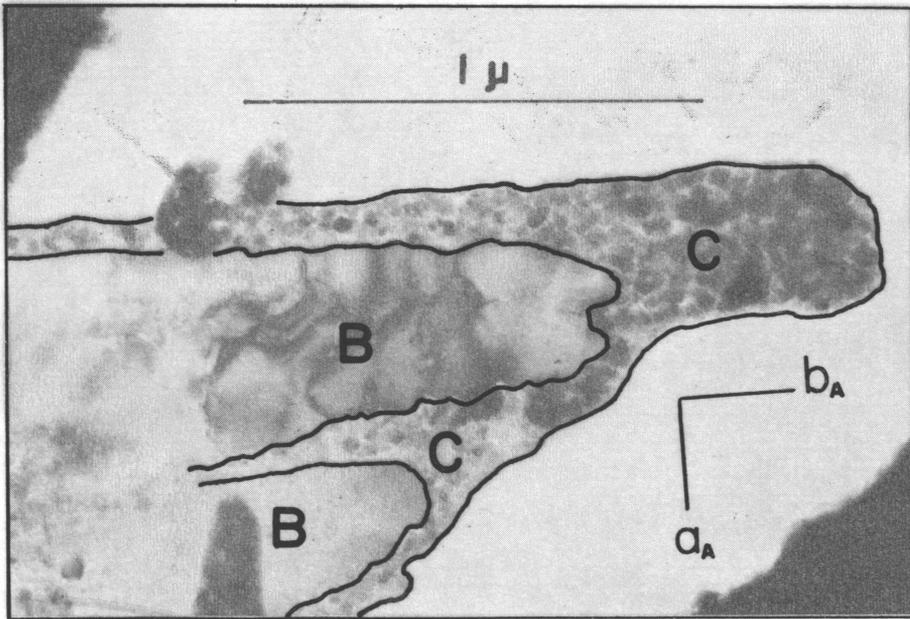
1. Photomicrograph of the comparatively thick flake heated at 825°C.
2. That of the thin flake heated at 1000°C.

In the cases of those heated at 825°C and 1000°C the textures are classifiable into three parts such as A, B and C.

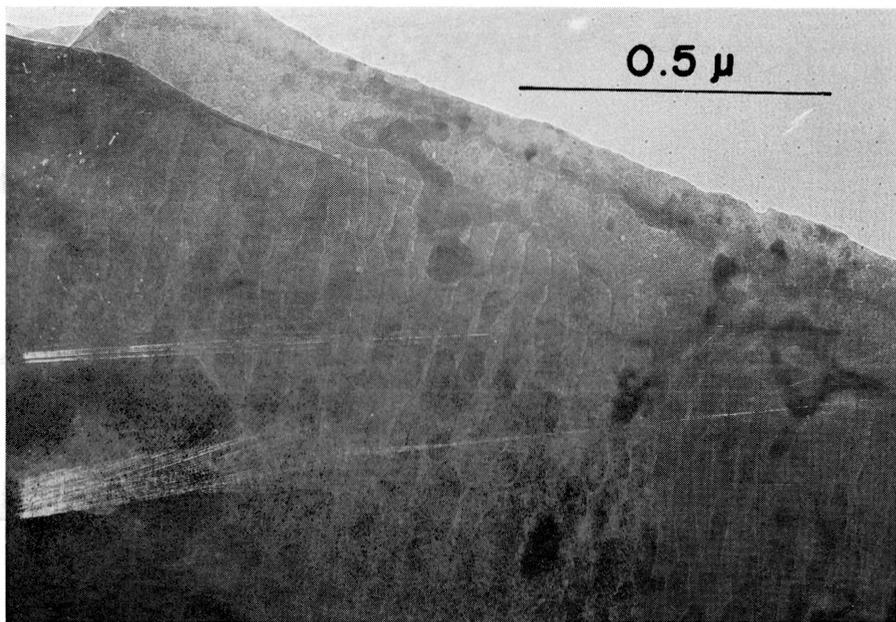
- A: the lamellae-like one elongated with a slight inclination for the *a*-axis of the original antigorite and divided into two parts with the higher and lower rate of absorption for the electron beam, occupying the most and central area of a flake.
- B: the dotted or amoeba-like ones with the lower rate of absorption for the electron beam, found in the homogeneous matrix and developing in the middle part around the area of A.
- C: the granular one observed along the margin of a flake.



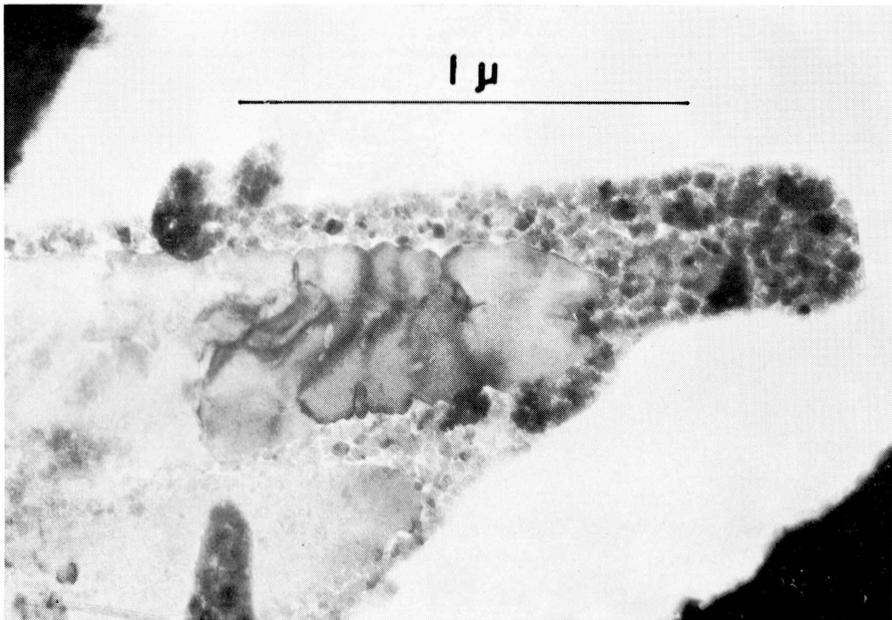
1



2



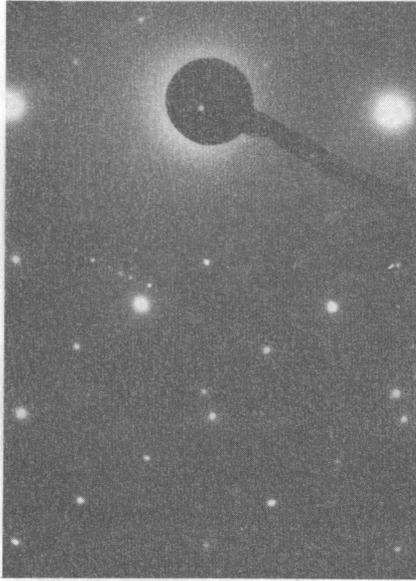
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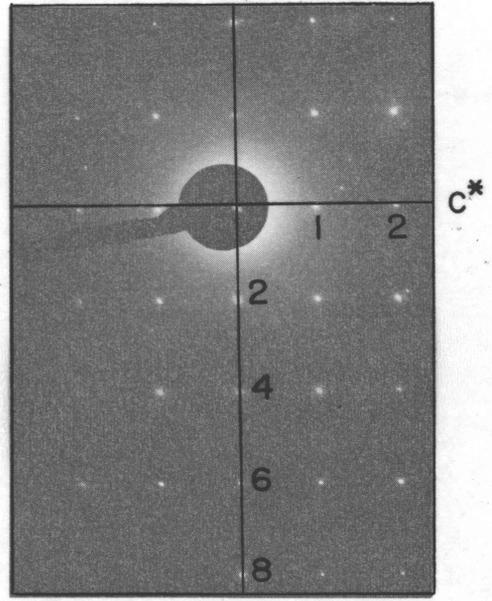
2

EXPLANATION OF PLATE XXV

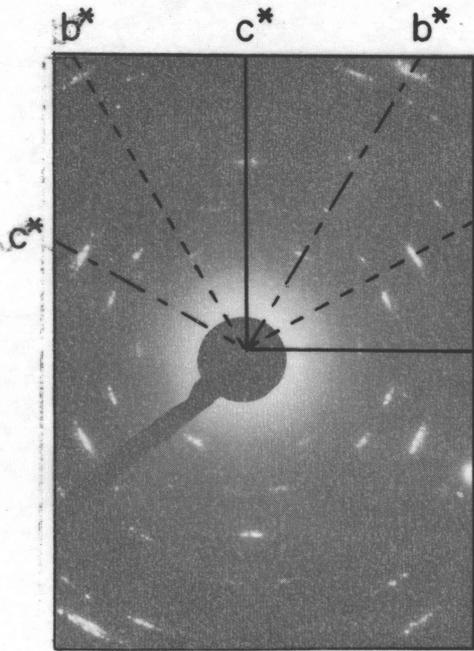
1. The selected area diffraction pattern of the central part.
2. That of the middle part, revealing the points of the reciprocal lattice on the b^*-c^* plane of olivine without the odd-order lines for b^* -axis.
3. That of the marginal part, indicating the points of the reciprocal lattice on the b^*-c^* planes of three individuals of olivine along a^* -axis inclined mutually with about 60° in angle.
4. The points of the reciprocal lattice on the b^*-c^* plane, drawn through arrangement of three individuals of olivine along a^* -axis with inclination of 60° in angle.



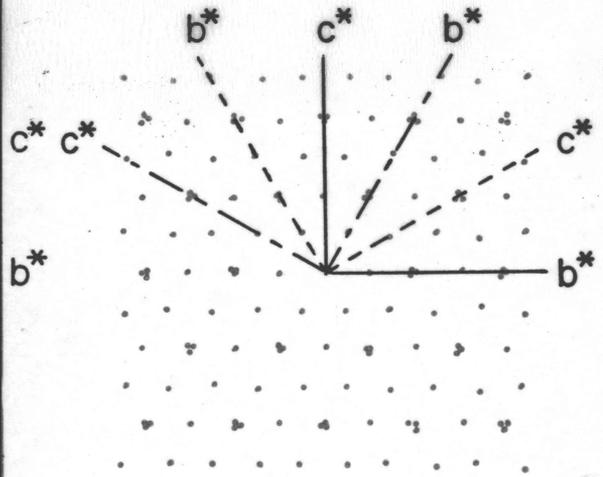
1



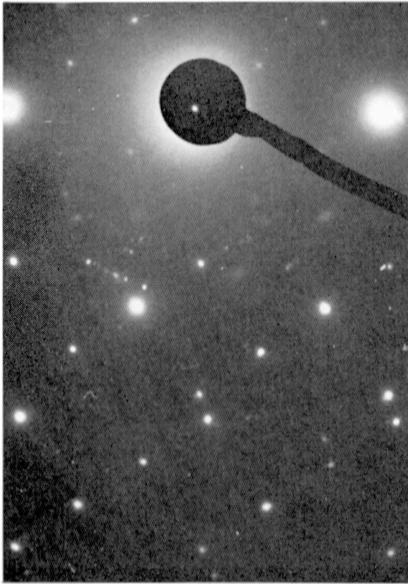
$2b^*$



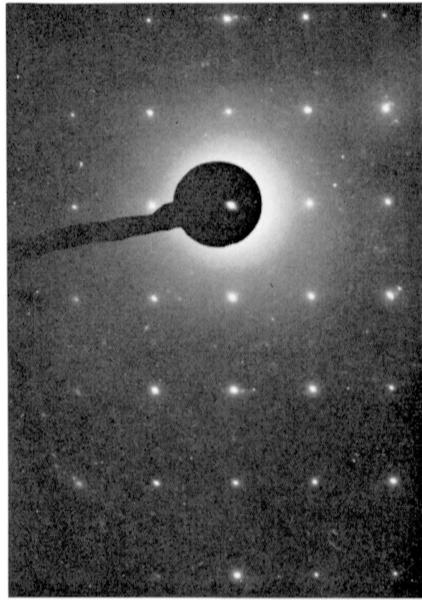
3



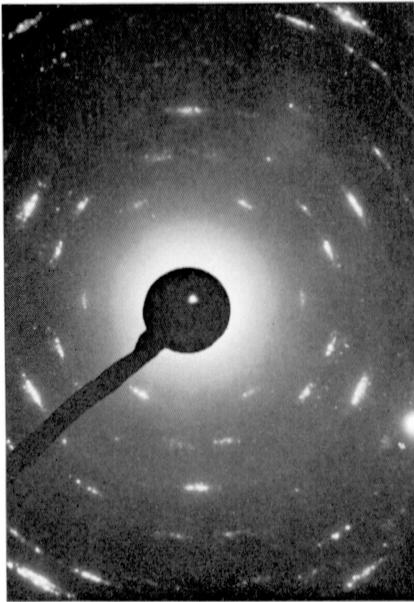
4



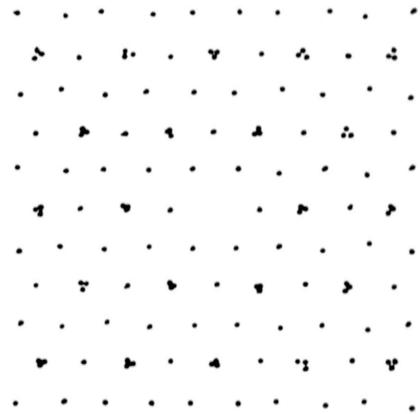
1



2



3



4