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Confirmation of the terrestrial occurrence of orthopyroxene with space group $P2_1ca$

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ABSTRACT

Based on $0kl$ electron diffraction patterns of two terrestrial orthopyroxene samples, all k-odd reflections violating a b glide are present. Such reflections are not caused by the overlap of diffractions from higher levels of orthopyroxene itself, or from the presence of $C2/c$ clinopyroxene exsolution lamellae or G.P. zones, by diffuse streaks along **a***, or by the multiple diffraction effects. Observations of a hypersthene sample with a TEM equipped with a heating stage indicate that a phase transformation is produced in which all $0kl$ diffraction spots with k-odd systematically became unobservable when the sample was heated to approximately 900 °C. The existence of a terrestrial orthopyroxene with space group $P2_1ca$ is therefore confirmed.

INTRODUCTION

The space group of orthopyroxenes in the enstatite-ferrosilite series has been regarded as being *Pbca* under normal pressure and temperature formation conditions from the time that the crystal structure of hypersthene was determined by Warren and Modell (1930). When Thompson (1970) investigated aspects of the amphibole structure, he indicated that orthorhombic pyroxenes, which obey the parity rule, should have space group $P2_1ca$. Papike et al. (1973), Matsumoto (1974), Pannhorst (1979), Law and Whittaker (1980), and Chisholm (1981, 1982) concluded that it is possible for orthopyroxene with space group $P2_1ca$ to exist. Smyth (1974) found k-odd reflections violating a b glide on $0kl$ precession photographs of bronzite from lunar troctolitic granulite. He interpreted the space group to be $P2_1ca$. Subsequently, Steele (1975), Harlow et al. (1979), Evensen et al. (1979), and Harlow (1980) reported space group $P2_1ca$ for one enstatite, four bronzites, and two hypersthenes. They occur in a lunar norite, a feldspar cumulate eucrite, the silicate inclusions of an iron meteorite, three achondrites, and one mesosiderite, respectively. Krstanović (1975) reported that there are $P2_1ca$ domains in enstatite occurring in harzburgite at Goleš Mountain, Yugoslavia. These domains are intimately intergrown with domains with space group *Pbca* that are richer in Mg. Harlow (1980) presented evidence that a terrestrial enstatite sample (AMNH G40403) had space group $P2_1ca$. This interpretation was subsequently reconsidered (Sasaki et al., 1984).

In a discussion of Smyth's (1974) report of $P2_1ca$ orthopyroxene, Nord et al. (1976) concluded that the appearance of diffractions violating the b glide was the result of multiple diffraction as is common in electron diffraction patterns. Sasaki and Matsumoto (1977) and Sasaki et al. (1981, 1984) subsequently proposed that the $0kl$ diffractions that appeared to violate the b glide were not caused by the lack of a b-glide plane. Recently, Smyth and Swope (1990) described further studies of lunar orthopyroxene 76535 and suggested a new explanation regarding the origin of the b-glide violations observed in that orthopyroxene.

Luo et al. (1987) ascertained that all of the k-odd diffraction spots are present on $0kl$ electron diffraction patterns of a hypersthene megacryst from Black Hill of Chengde, Hebei Province, China, thus confirming that pyroxene with space group $P2_1ca$ indeed exists. Luo et al. (1989) also derived the most probable structure topology, as shown in Figure 1. They indicated that it is possible to find domains related by twin plane (100). In such a structure, there are four kinds of tetrahedral chains, two M1 sites, and two M2 sites, all of which are symmetrically distinct. The ratio of O-rotated to S-rotated tetrahedral chains is 3:1, and the $++--$ repeat sequence of skew of octahedral strips along the **a** axis is the same as in orthopyroxene with space group *Pbca*. Such a structure obeys the parity rule suggested by Thompson (1970) and has **a** as a polar axis. If there are two neighboring domains whose **a** axes are in reversed orientations, a twin with {100} as twin plane could be formed. The structures of such domains are fully continuous at the join. Therefore, it would be difficult to distinguish such a twin from a single crystal in morphology, optical properties, and diffraction patterns. It can also be seen from Figure 1 that the twin plane serves as a b-glide parallel to {100}, causing the intensity of $0kl$ diffractions with k-odd to be weakened or even to become almost extinct. In this case, it would be more difficult to distinguish orthopyroxene with space group $P2_1ca$ from one with space group *Pbca*. There are indications that such twinning exists in both the orthopyroxenes from Black Hill and Bamble, Norway (Luo et al., in preparation). We believe that this kind of

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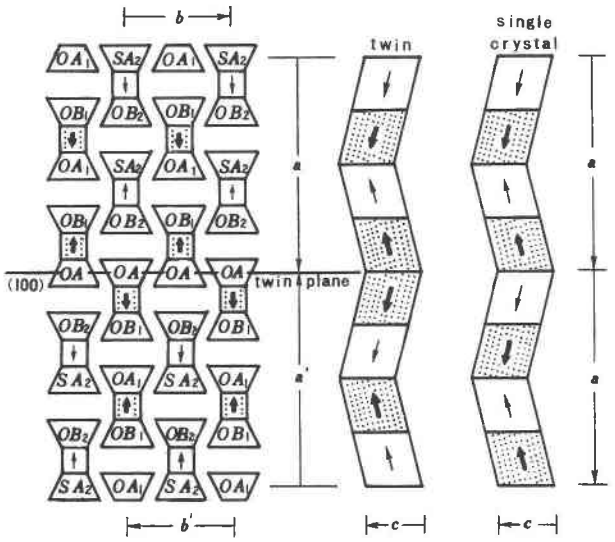

Fig. 1. Schematic I-beam model of the most probable structure and the structural relationship between the two domains in a twin on {100} of $P2_1ca$ orthopyroxene. The polar **a** axes are oppositely oriented in the twin domains. The skew of octahedral strips is symbolized with arrows with opposite directions instead of the conventional + and − to allow direct comparison with the skew of octahedral strips in the twin domains. The light and heavy arrows indicate octahedral strips that are symmetrically nonequivalent (after Luo et al., 1989).

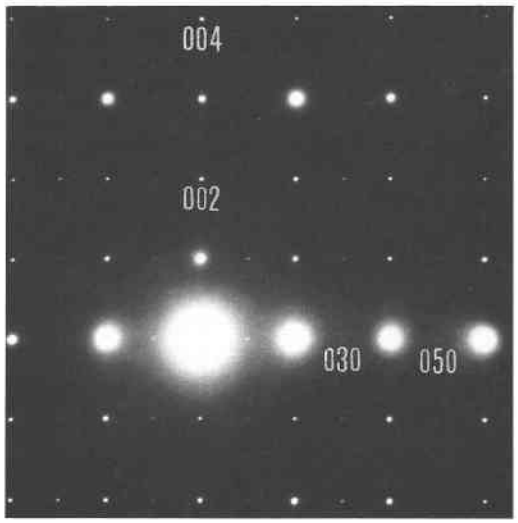

Fig. 2. A [100] electron diffraction pattern of the Chengde hypersthene (sample 1). The $0kl$ diffraction spots with $k = 2n + 1$ systematically appear but are weaker than those with $k = 2n$.

twin is the cause of ambiguities regarding the space group of orthopyroxene. In this paper, the relations for the diffraction intensities violating the b glide in orthopyroxene are discussed.

Samples and experiments

Sample 1 is a hypersthene megacryst (average composition: $En_{56}Fs_{41}Wo_3$; $a = 18.310$, $b = 8.900$, $c = 5.223$ Å) from the Presinian noritic pegmatite from Black Hill, Chengde, Hebei Province, China. The sample contains exsolution lamellae of augite (approximately $En_{45}Fs_{30}Wo_{25}$; $a = 9.76$, $b = 8.91$, $c = 5.22$ Å, $\beta = 105.2°$) and G.P. zones parallel to {100}. It always occurs as anhedral crystals over 2 cm in size, bronze in color, and with {100} parting. Sample 2 is an enstatite megacryst ($En_{96.6}Fs_{3.4}$; $a = 18.224$, $b = 9.815$, $c = 5.179$ Å) from Bamble (collection of Department of Earth Sciences, Nanjing University). It is green in color and over 10 cm in length, with {100} parting.

The samples were thinned by ion milling and coated with a C film. Selected area diffraction patterns were obtained using a JEM-200CX transmission electron microscope (TEM) operated at 200 kV, with camera lengths of 137 and 55 cm. Before the samples were examined by TEM, they were studied by polarized light microscopy, X-ray powder diffraction, and precession single crystal X-ray diffraction. Long exposure (120 h) [100] X-ray precession photographs revealed that $0kl$ diffraction spots with k-odd, such as 011, 031, 033, 051, 054, and 074, are clearly observable, although they are weaker than those with k-even.

Results and discussion

Smyth (1974) first described the existence of pyroxene with space group $P2_1ca$ in a lunar sample. However, Nord et al. (1976), Sasaki and Matsumoto (1977), and Sasaki et al. (1981, 1984) questioned the existence of orthopyroxene with space group $P2_1ca$ for three reasons. They suggested that the presence of reflections incompatible with a b glide could be explained as (1) overlapping of diffractions from $C2/c$ clinopyroxene exsolved on {100} of the host orthopyroxene, (2) diffuse streaks of orthopyroxene or clinopyroxene from levels separated from the $0kl$ net, or (3) multiple diffraction. Using our own experimental results, we discuss our interpretations of the origin of the diffractions violating a b glide for the orthopyroxenes as follows.

Relations for overlapping diffraction patterns of opx host and cpx lamellae

If the diffraction pattern of clinopyroxene {100} lamellae completely overlaps that of orthopyroxene with space group *Pbca,* cell dimensions of both phases must satisfy specific conditions because **c*** of host and guest crystals are not parallel to each other. The reciprocal lattice translation of clinopyroxene that is approximately parallel to **c*** of orthopyroxene is $[\bar{1}02]^*$ (Sasaki et al., 1984). Cell dimensions of orthopyroxene and clinopyroxene can have values consistent with $S_{\bar{1}02(cpx)}$ being almost parallel and equal to $S_{002(opx)}$ if the interfaces are coherent. Even so, the diffraction spots of clinopyroxene with space group $C2/c$ that appear on the $0kl$ net of orthopyroxene, as pointed out by Sasaki et al. (1984), are limited to indices with k-odd, $l = \pm 2, \pm 6$, and so on, indexed on the

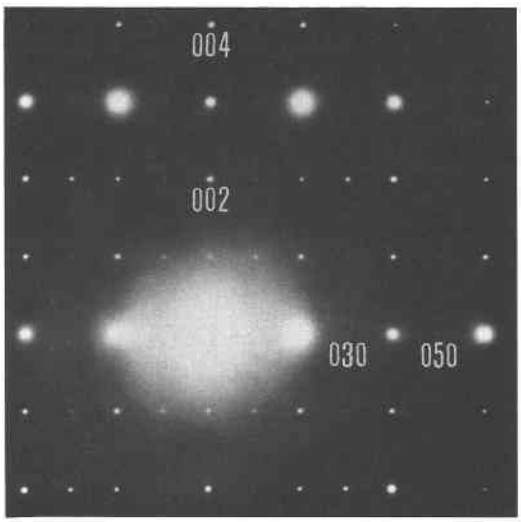

Fig. 3. A [100] electron diffraction pattern of Bamble enstatite (sample 2). The $0kl$ diffraction spots with $k = 2n + 1$ occur but are weaker than those with $k = 2n$.

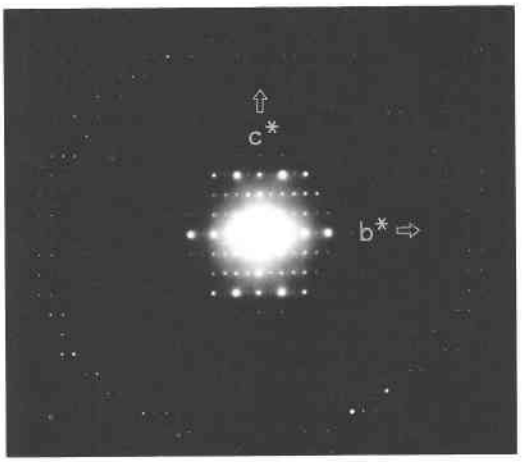

Fig. 4. A [100] electron diffraction pattern of Chengde hypersthene (sample 1) including reflections from the first level. It can be clearly seen that there is a gap between the two Laue zones.

cell of orthopyroxene. This is because the reciprocal net of clinopyroxene that is parallel to the $0kl$ net of the orthopyroxene is of hkl type with $h + k = 2n$ and $l = \pm 2h$; therefore, when k is odd, l must be equal to $4n + 2$. For instance, the allowed diffractions of clinopyroxene should be $\bar{2}04$, $\bar{4}08$, $\bar{1}12$, $\bar{3}16$, . . . , and they would overlap with 004, 008, 012, 016, . . . of orthopyroxene, respectively. Accordingly, it can be concluded that the $0kl$ spots with k-odd and $l \neq 4n + 2$ cannot be caused by the clinopyroxene lamellae. Selected area electron diffraction patterns of the $0kl$ net for samples 1 and 2 (Figs. 2 and 3) contain, in addition to k-even spots, k-odd spots (including those with $l \neq 4n + 2$) that are very sharp.

Intensity of $0kl$ reflections in relation to reflection shape

Differentiation of overlap from neighboring levels. Each reciprocal lattice point has a rodlike shape whose length increases with decreasing thickness of the sample. Nonzero level reflections can therefore overlap the zero level. However, our diffraction patterns show that the spots violating the b glide in the $0kl$ pattern of orthopyroxene are not caused by such overlap. Figure 4 is a [100] electron diffraction pattern of sample 1 that includes reflections from a higher-order Laue zone. The distance between levels is calculated to be 0.0546 Å^{-1}, which corresponds to $1/a^*$ of orthopyroxene (0.0549–0.0543 Å^{-1}). The higher-order Laue zone in Figure 4 must therefore correspond to reflections with indices $1kl$. A gap is evident between the first and zero order Laue zones, also apparent in sample 2 (Fig. 5); this could not occur if $1kl$ reflections overlapped the zero level. Thus, none of the $0kl$ diffractions, including those with k-odd, result from overlap with higher-level reflections.

Streaking along a*. The [010] and [001] electron diffraction patterns of sample 1 (Figs. 6 and 7) show that, as observed by other authors, all diffraction spots are diffuse in the **a*** direction. However, the intensities of the diffuse streaks are not strong enough to form a sharp diffraction spot on the $0kl$ net. If the forbidden $0kl$ diffractions of a b glide were caused by diffuse streaks extending between the $1kl$ and $\bar{1}kl$ diffractions, there should have been diffraction spots between the first- and zero-order Laue zones in Figures 4 and 5, but there are no such reflections. Furthermore, $h = 2n$ for $hk0$ reflections of $Pbca$ and $P2_1ca$, and $1k0$ and $\bar{1}k0$ diffractions are systematically extinct. Thus, $0k0$ diffractions with k-odd would have to be caused by diffuse streaks between reflections $2k0$ and $\bar{2}k0$. Such diffraction relations are highly unlikely because of the large separation between pairs of reflections. In fact, no streaking with intensity commensurate

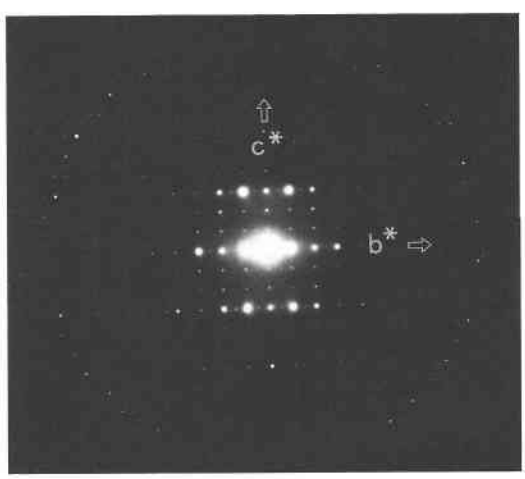

Fig. 5. A [100] electron diffraction pattern of Bamble enstatite (sample 2) including reflections from the first-order Laue zone. There is a gap between reflections from the two Laue zones.

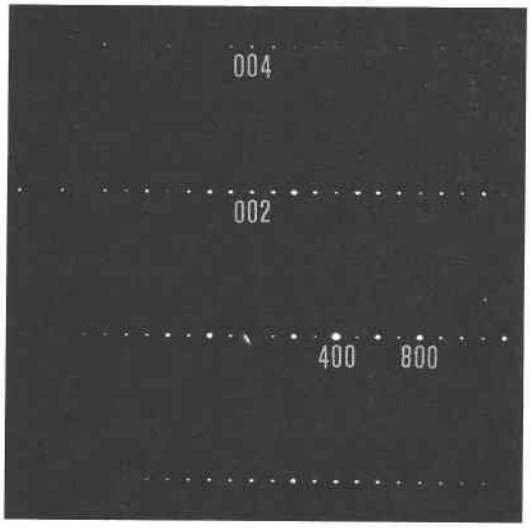

Fig. 6. A [010] electron diffraction pattern of Chengde hypersthene (sample 1).

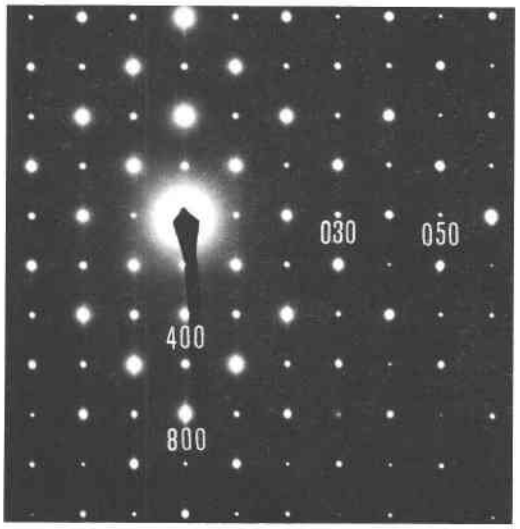

Fig. 7. A [001] electron diffraction pattern of Chengde hypersthene (sample 1). The $hk0$ diffraction spots with $h + 2k = 4n + 2$ occur but are weaker than those with $h + 2k = 4n$.

with that of the $0k0$ spots is observed in the [001] electron diffraction pattern (Fig. 7); formation of sharp $0k0$ spots due to diffuse streaks between $2k0$ and $\bar{2}k0$ diffractions is therefore not possible.

Multiple diffraction

Conditions for multiple diffraction. In order for multiple diffraction to occur, the indices of the secondary diffraction hkl and the two cooperating diffractions $h_1k_1l_1$ and $h_2k_2l_2$, which cause the diffraction hkl, should be $h = h_1 + h_2$, $k = k_1 + k_2$, $l = l_1 + l_2$. If the space group of orthopyroxene were *Pbca,* the nonextinct diffractions would have indices with values of k equal to $2n$ for $0kl$ diffractions. Obviously, the sum of even numbers cannot equal an odd number, so the k-odd diffractions on the $0kl$ pattern of the orthopyroxene cannot result from secondary diffractions originating from two $0kl$ diffractions of orthopyroxene with space group *Pbca.*

Multiple diffraction from higher levels. No matter whether the space group of orthopyroxene is *Pbca* or $P2_1ca$, the following diffraction conditions will exist: $h0l$: $l = 2n$, no condition for h; $00l$: $l = 2n$; $hk0$: $h = 2n$; $h00$: $h = 2n$; $0kl$: no condition for l. Experimental observation shows that, as expected, all $h0l$ diffractions with l-odd and $hk0$ diffractions with h-odd are extinct as shown by Figures 6 and 7. However, a few $00l$ diffractions with l-odd that appear on the [100] electron diffraction patterns (Figs. 2 and 3) are due to multiple diffraction for $0kl$ diffractions. The $00l$ diffractions with l-odd on the [010] pattern (Fig. 6) are extinct as a result of the absences of all $h0l$ diffractions with l-odd. Similarly, because the $hk0$ diffractions with h-odd are extinct but h-odd indices are permitted for $h0l$ diffractions, the forbidden diffractions $h00$ with h-odd appear on the [010] pattern (Fig. 6) but not on the [001] pattern (Fig. 7). Therefore, at least in the [010] and [001] diffraction patterns, there are no diffraction spots that are caused by multiple diffraction involving higher levels. Further experimentation (see below) shows that this also is true for the [100] pattern. In other words, the multiple diffractions all result from the diffractions for the zero level. We have already shown that the $0kl$ diffractions with k-even cannot produce any secondary $0kl$ diffractions with k-odd. Therefore, we conclude that the k-odd violations are not the result of multiple diffractions.

Distinguishing multiple diffraction. Multiple diffractions can be distinguished by tilting the sample and moving the source of multiple diffraction away from the Ewald sphere. For the [100] diffraction pattern, the forbidden diffractions of $00l$ become unobservable when the sample is tilted about **c***. This indicates that these diffractions are caused by multiple diffraction. The $0k0$ diffractions with either k-even or k-odd do not become unobservable when the sample is tilted about **b***. To examine further whether the k-odd spots on $0kl$ diffraction patterns are caused by the multiple diffraction from higher levels, the (100) section of sample 1 was tilted about the reciprocal vector $\mathbf{S}_{032}$, and the change in intensity for 032, an intense spot on the $0kl$ net, was observed. There was no perceivable change in its intensity. Seven selected-area diffraction patterns were obtained from $+2°10'$ to $-3°50'$ to confirm this observation. Therefore, the presence of $0kl$ diffractions with k-odd on the [100] pattern is not the result of multiple diffraction.

Extra extinction conditions

Chisholm (1981) has indicated, in his derivation of pyribole structure types, that in the case of projection down **c**, the point ¼,½ has the same environment as the origin 0,0 for pyroxene with space group $P2_1ca$, and this symmetrical relation gives rise to an additional diffrac-

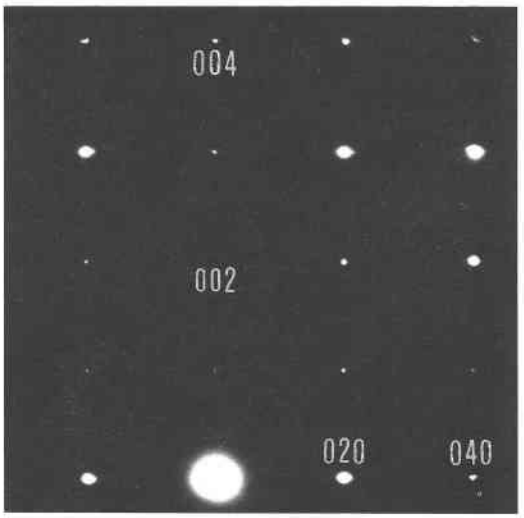

Fig. 8. A [100] electron diffraction pattern of Chengde hypersthene (sample 1) when heated to more than 900 °C. The $0kl$ diffraction spots with $k = 2n + 1$ are systematically extinct, and the others are diffuse along **b***.

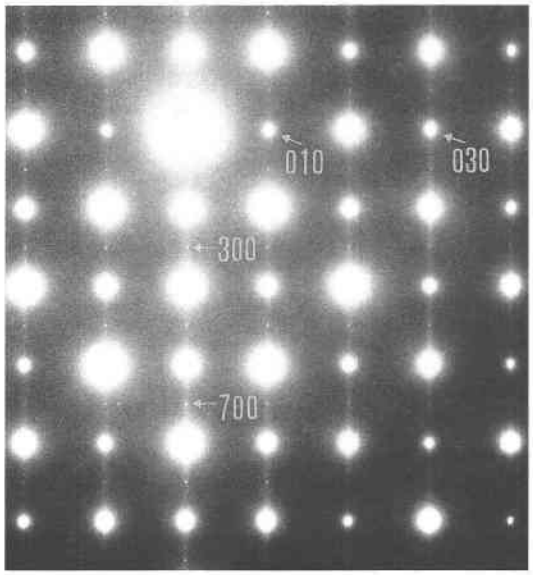

Fig. 9. A [001] electron diffraction pattern of Chengde hypersthene (sample 1) obtained from an area with abundant G.P. zones. A set of extra $hk0$ spots with h-odd is present.

tion condition $2h + k = 4n$ for $hk0$ diffractions. The additional diffraction condition $2h + k = 4n$ should instead be $h + 2k = 4n$, however. Nevertheless, in contrast to Chisholm's structure model of pyroxene with space group $P2_1ca$, the environment of the point ¼,½ is different from, although similar to, that of the origin 0,0 in the model of $P2_1ca$ orthopyroxene (Fig. 1) proposed by Luo et al. (1989). Thus, it is expected that there would be no extra extinction condition for $hk0$ diffractions, but the intensities of the $hk0$ diffractions with $h + 2k = 4n + 2$ would be systematically weaker than those with $h + 2k = 4n$. This relation is evident in Figure 7.

Heating experiment

Heating of sample 1, carried out by means of a heating stage in the TEM (Xu et al., 1989), showed that there was no change in the [100] diffraction pattern until the sample was heated to approximately 900 °C. The $0kl$ spots (including $0k0$ spots) with k-odd became systematically unobservable after the sample was held at 900 °C for 1 h. The remaining spots were streaked along **b*** (Fig. 8). This suggests that a phase transformation may occur at approximately 900 °C, and the observation of all $0kl$ diffractions at room temperature depends on the intrinsic symmetry of crystals and not on factors such as multiple diffraction.

Extra diffractions resulting from G.P. zones

Recently, in a further study of the origin of space-group violations in lunar orthopyroxene 76535, Smyth and Swope (1990) showed that forbidden diffractions for a b glide are not caused by diffractions from clinopyroxene lamellae, by diffuse streaks, or by multiple diffraction effects, based on automated four-circle X-ray diffractometer data. This corresponds with our above-described observations. They also suggested that the most reasonable explanation for the observed space-group violations is that both the a- and b-glide violations result from ordering of Ca into {100} G.P. zones.

In the electron diffraction patterns of sample 1, diffractions violating not only the b but also the a glide are present. Figure 9 is an [001] diffraction pattern that was obtained from an area that has abundant G.P. zones. Besides the streaks along **a***, extra $hk0$ spots with h-odd appear, but their intensities are much weaker than those with h-even. The dark-field image formed using the 300 diffraction shows that the extra diffractions violating the a glide result from G.P. zones. Since the G.P. zones are much smaller than the thickness of the selected area of sample and the diameter of the electron beam, the volume ratio of G.P. zones to host for any sample orientation is constant. However, if the intensities of $0k0$ diffractions in different patterns are taken as a reference, it is found that the intensity ratio of $hk0$ (h-odd) to $0k0$ in the [001] pattern (Fig. 9) is much smaller than that of $0kl$ (k-odd) to $0k0$ in the [100] patterns (Fig. 2). In other words, the intensities of $hk0$ (h-odd) diffractions are obviously incommensurate with those of $0kl$ (k-odd) ones. These relations imply that the b-glide violations are caused by another factor. The dark-field image formed using the 010 diffraction (Fig. 10) indicates that the 010 diffraction intensity is due to the host orthopyroxene and not to G.P. zones or clinopyroxene lamellae. Therefore, the appearance of $0kl$ (including $0k0$) diffractions with k-odd should mainly be attributed to the host orthopyroxene and not to the G.P. zones.

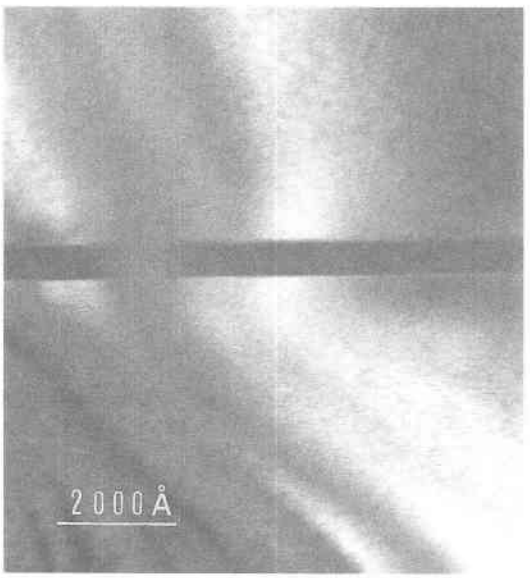

Fig. 10. Dark-field image of the Chengde hypersthene (sample 1) formed using the 010 diffraction. Only the host orthopyroxene is in bright contrast. The horizontal dark strip is an augite lamella.

Conclusions

Based on the diffraction relations for the orthopyroxene samples, the following conclusions can be drawn.

1. For the two samples studied, the diffractions violating a *b* glide on [100] electron diffraction patterns are true violations. Their presence is not caused by overlap of diffractions from higher levels of orthopyroxene, from augite exsolution lamellae, or by diffuse streaks along **a***, and they are neither the result of multiple diffraction nor contributed by G.P. zones. The presence of diffractions violating a *b* glide is caused by the inherent symmetry of the crystal. Observations for sample 1 during heating tests support this view.

2. The conditions of diffraction for the two orthopyroxenes studied are: *hkl*: no condition; *hk*0: $h = 2n$; 0*kl*: no condition; *h*0*l*: $l = 2n$; *h*00: $(h = 2n)$; 0*k*0: no condition; 00*l*: $(l = 2n)$. The corresponding possible space groups are *Pmca* or $P2_1ca$. However, the true space group is most likely $P2_1ca$ (Luo et al., 1987) because there is no mirror plane in the crystal structure of any pyroxene. Therefore, we conclude that orthopyroxene with space group $P2_1ca$ can exist on the Moon, the Earth, and in meteorites.

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