American Mineralogist, Volume 77, pages 107–114, 1992

Direct observation on the formation of antiphase domain boundaries in pigeonite

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ABSTRACT

The formation of the antiphase domain structure in pigeonite was observed in situ using a heating stage in a transmission electron microscope. The phase transition between high and low pigeonite occurred athermal-martensitically over a range of about 100 °C. The observation showed that the APBs parallel to the **c** axis (L type) form at an earlier stage and the APBs transverse to the **c** axis (H type) form at the final stage of the $C2/c$ to $P2_1/c$ pigeonite transition interval during cooling. The observation suggests that the H APBs are formed as the traces of $C2/c$ pigeonite rather than as the original APBs. The formation of the H APBs is controlled by the movement of the interfaces between $P2_1/c$ and $C2/c$ pigeonite in the transition interval. $C2/c$ pigeonite grew from the H APBs, where there is expected to be Ca enrichment. The Ca enrichment seems to facilitate the transition to $C2/c$ pigeonite. Specifically, the Ca concentration locally lowers the transition temperature.

Based on the difference between the two types of APBs with regard to the formation stage and the Ca enrichment, the fact that the H APBs are commonly observed in slowly cooled pigeonite, whereas the L APBs are commonly found in rapidly cooled pigeonite, can be explained qualitatively.

INTRODUCTION

Ca-poor clinopyroxene, pigeonite, has been of interest to many researchers because it can retain information on thermal history. At high temperature, pigeonite possesses a structure with space group $C2/c$ (high pigeonite), in which all of the SiO_4 chains are symmetrically equivalent. On cooling, high pigeonite transforms displacively to the $P2_1/c$ structure (low pigeonite), which contains two symmetrically different silicate chains (designated as the A and B chains). During the transition, misarrangement in the distribution of the A and B chains generally takes place. Consequently, the antiphase domain (APD) structure, which consists of regions where the $P2_1/c$ structure is related across antiphase domain boundaries (APBs) by a translation of $1/2(\mathbf{a} + \mathbf{b})$, can be produced (Morimoto and Tokonami, 1969). The APDs were observed directly by transmission electron microscopy (TEM) in lunar and terrestrial pigeonite by Bailey et al. (1970), Christie et al. (1971), and Champness and Lorimer (1971). Carpenter (1978) studied the APB orientations in "001" pigeonite and classified them into two types: APBs crossing the **c** axis (H type) and APBs parallel to the **c** axis (L type). The former have a sigmoidal shape and are approximately parallel to $\{2\bar{1}\bar{1}\}$ and $\{211\}$, and the latter are straight and approximately parallel to $\{120\}$.

The APD structure in pigeonite is closely related to the cooling history. Many attempts have been made to use the size of APDs in pigeonite as a relative measure of cooling rate (e.g., Ghose et al., 1972; Lally et al., 1975; Carpenter, 1979). On the other hand, Fujino et al. (1988) suggested that the orientation of APBs, rather than the size of APDs, would be closely associated with the cooling rate. Fujino et al. (1988) performed annealing experiments, with different cooling rates, on "001" pigeonite lamellae in augite phenocrysts from Hakone-toge, Hakone Volcano, Japan. They showed that the APBs are nearly parallel to the **c** axis at fast cooling rates, whereas at slower cooling rates they are inclined to the **c** axis.

All of the previous observations of the APD structure were carried out at room temperature using natural specimens or the quenched products of annealing experiments. Formation of the APD structure in pigeonite has not been observed in situ at high temperatures. High-temperature transmission electron microscopy (HTTEM) is necessary for this purpose. In fact, formation processes of APD structures in other silicate minerals have already been observed in situ using HTTEM; for example, in anorthite by Van Tendeloo et al. (1989) at about 240 °C and in kanoite (Ca-poor magnesium manganese clinopyroxene) by Gordon et al. (1981) at about 330 °C. In the case of pigeonite, preliminary HTTEM work has also been carried out by Fuess et al. (1986), but they could not observe the formation of APD structure in situ. As part of the present series study, a double-tilt heating stage (Morimoto et al., 1989) was employed to study the $P2_1/c$-$C2/c$ phase transition of clinopyroxenes (Shimobayashi and Kitamura, 1990). The thermoelastic martensitic transformation of clinoenstatite and pigeonite is discussed in detail by Shimobayashi and Kitamura (1991). In the present paper, the in situ observation of the formation process of APD structure in "001" pigeonite is

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Fig. 1. Dark-field (**g** = 102) electron micrograph and electron diffraction pattern of the Hakone pigeonite with coarse, heterogeneously spaced "001" augite lamellae.

reported and discussed based on the athermal nature of the phase transition.

EXPERIMENTAL PROCEDURE AND RESULTS

Specimen description

Specimens prepared for the present study are pigeonite phenocrysts in andesite from Hakone-toge, Hakone Volcano, Japan, which is also the locality of the augite phenocrysts that Fujino et al. (1988) used for their annealing experiments. The specimens were prepared from petrographic thin sections cut parallel to (010). They were mounted on 2-mmϕ Mo single-hole grids (0.1 mmϕ hole diameter), thinned further by ion milling and C coated on both sides to prevent charging effects. The specimens were imaged in dark field with a "b" type reflection using a 200 kV TEM (Hitachi H-700). The chemical compositions of the specimens were determined with analytical electron microscopy (AEM; Morimoto and Kitamura, 1981) using a Kevex Delta class energy-dispersive system (EDS).

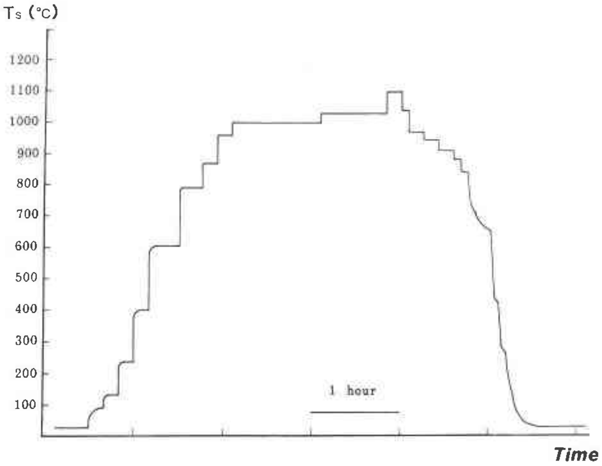

Fig. 2. Plot of temperature vs. time for the first heat treatment of the pigeonite.

The pigeonite specimen ($En_{58}Fs_{39}Wo_3$) contains "001" augite ($En_{42}Fs_{23}Wo_{35}$) lamellae, about 0.05–0.2 μm in thickness (Fig. 1). The APD structure is apparent in the pigeonite host. The H APBs are predominant in the specimen before heating in the TEM. In the central part of the thicker pigeonite lamellae (more than about 0.2 μm thick), fine augite plates are precipitated parallel to "001" at the APBs. In the marginal parts of these pigeonite lamellae, named "precipitate free zones" (PFZ) by Carpenter (1978), the L APBs are also found. The observed microtextures are quite similar to those of the slowly cooled pigeonite samples in coarse-grained dolerites described by Carpenter (1978).

Heating experiments

Heating experiments were carried out by using a double-tilt side-entry heating stage fitted with a special furnace (Morimoto et al., 1989). The heating stage can be tilted ±10° by pulling a Ta ribbon wound on the stage. A foil specimen is fixed with a C screw in the center of the furnace. The heater of the furnace is a W resistance coil (3.3 mm in diameter and 2 mm in height) embedded in an alumina insulator. The temperature is determined by the wattage of the coil heater. Specimen temperatures during the heating experiments could not be measured directly. Instead, the specimen temperature was measured indirectly by another thermocouple placed at the specimen position before the experiments and calibrated against wattage. All of the temperatures in the HTTEM experiments were derived from this calibration curve (Morimoto et al., 1989) and are regarded as the "specimen temperature." In the heating experiments, the temperatures were usually raised or lowered stepwise by about 10–100 °C at time intervals of 5–10 min (Fig. 2). It is very difficult to take photographs during the heating experiment because changes of texture often occur rapidly and because thermal drift of the stage usually takes place. Detailed textures during the transition were therefore observed on a TV monitor through an image processing system (Gatan model 622) and recorded on video tapes. Electron micrographs were hard copied from the video tapes by use of an image printer.

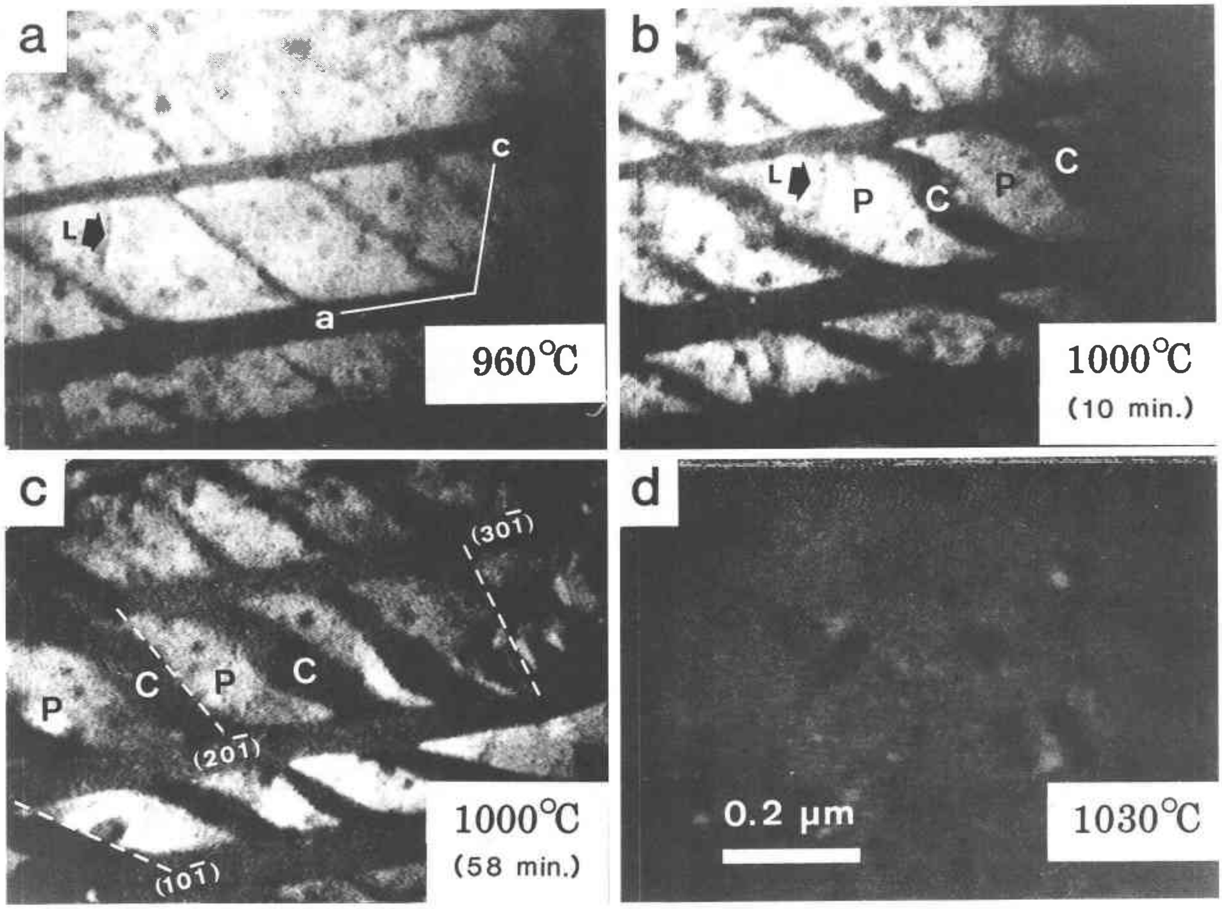

Fig. 3. A sequence of dark-field (**g** = 102) electron micrographs (**a**)–(**d**) hard copied from a video tape of the $P2_1/c$ to $C2/c$ pigeonite transition on heating in the first heat treatment. The letters P and C in **b** and **c** represent low pigeonite ($P2_1/c$) and high pigeonite ($C2/c$), respectively. $C2/c$ pigeonite nucleated not from the L APBs (shown by arrows) but from the H APBs.

In situ observations

During heating of the original specimen, when the temperature was raised to 960 °C from 870 °C, $C2/c$ pigeonite started to grow in the form of widening of the H APBs, whereas no significant change was observed in the L APBs (Fig. 3a). When the temperature was increased to 1000 °C, the $C2/c$ pigeonite regions grew wider (Figs. 3b and 3c). The growth of $C2/c$ pigeonite ceased within a few tens of minutes. The transition was not complete at this temperature (1000 °C) even after 1 h (Fig. 3c). On heating further to 1030 °C, however, the growth of $C2/c$ pigeonite resumed, and accordingly the $P2_1/c$-pigeonite regions became smaller and finally ceased to exist (Fig. 3d). In this transition, the amount of transformed $C2/c$ pigeonite is characteristic of temperature but not primarily of time, as discussed in detail by Shimobayashi and Kitamura (1991).

The specimen was cooled after further heating at 1100 °C for about 10 min. The cooling rate was about 200–300 °C/h in the present experiments. When the temperature was lowered to 940 °C from 970 °C, $P2_1/c$ pigeonite reappeared in the $C2/c$-pigeonite host (Fig. 4a). The growth of $P2_1/c$ pigeonite proceeded with further decrease in temperature (Figs. 4b and 4c). In the $P2_1/c$-pigeonite regions, newly formed L APBs were observed (Figs. 4a and 4b) during the cooling process. The $C2/c$ to $P2_1/c$ pigeonite transition was completed by cooling to 840 °C from 880 °C (Fig. 4d). H APBs, which were commonly observed before heating, formed again at the final stage of the phase transition (Fig. 4d). Some of the H APBs were found at almost the same positions as those observed in the original specimen before heating. The configuration of the H APBs, however, became complex in the cooled specimen because of the interaction with the L APBs. Figure 5 shows changes in the pigeonite texture in the first heat treatment before (Fig. 5a), during (Fig. 5b), and after (Fig. 5c) the heating experiment. Note the difference in the APB configurations between Figures 5a and 5c. After the first heat treatment, the H APBs became quite rare, and the APBs approximately parallel to the **c** axis were predominant (Fig. 5c). This difference in the

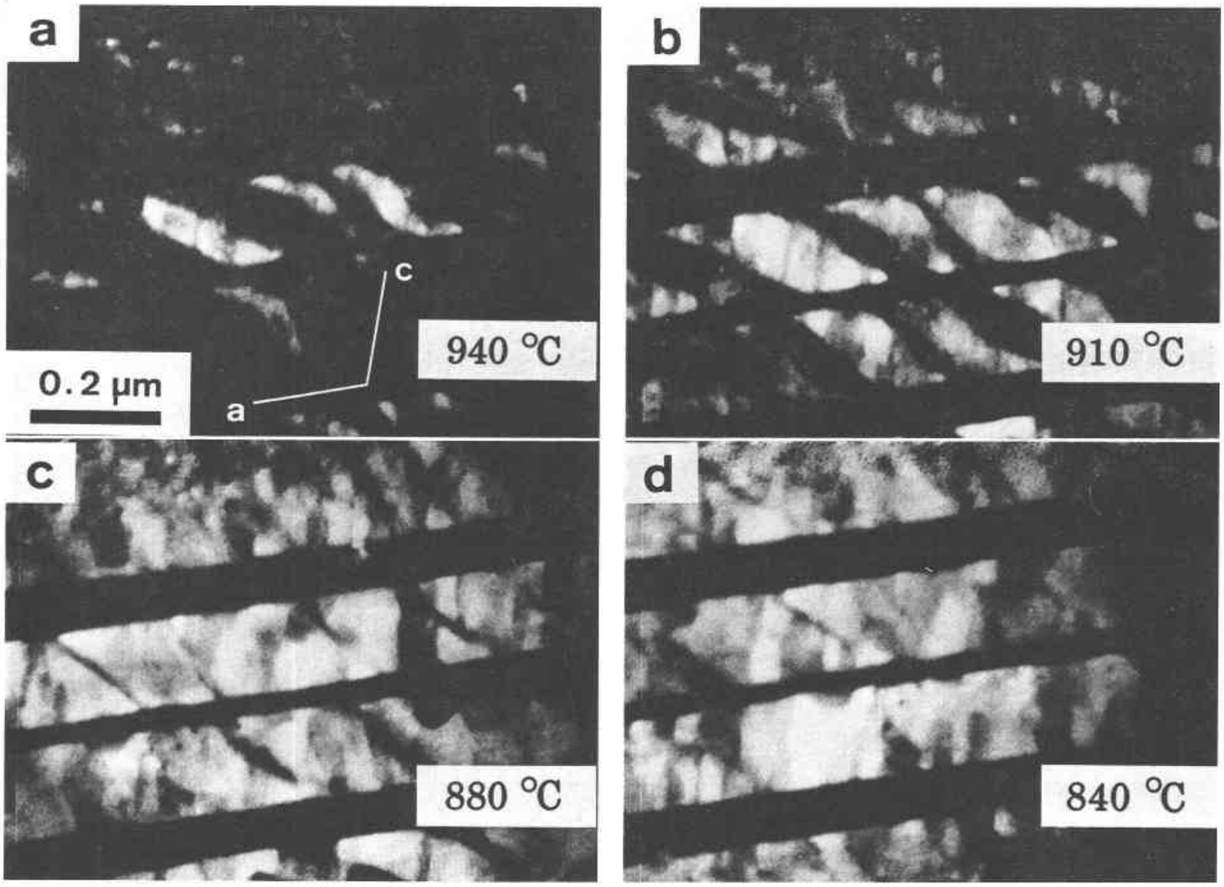

Fig. 4. A sequence of dark-field (**g** = 102) electron micrographs (**a**)–(**d**) hard copied from a video tape of the $C2/c$ to $P2_1/c$ pigeonite transition on cooling in the first heat treatment.

APB orientations is consistent with the results of the annealing experiments of Fujino et al. (1988), although they considered that the crystallographic orientation of APBs had actually changed. The present observations, however, show that the relative abundance of the two types of orientations changed in the first heat treatment, as discussed later.

The same specimen was heated again to determine whether the newly formed L APBs had an influence on the transition. The orientation of the interfaces between $P2_1/c$ and $C2/c$ pigeonite is unchanged between the first and the second heat treatment. The L APBs, which were newly formed in the first heat treatment, obviously did not have any effect on the orientation of the interfaces between $P2_1/c$ and $C2/c$ pigeonite (Fig. 6). At the beginning of the $P2_1/c$-$C2/c$ pigeonite transition in the second heat treatment, $C2/c$ pigeonite grew from the H APBs, but not from the L APBs, although the H APBs were reduced in number after the first heat treatment. Even in the absence of the H APBs, the nucleation of $C2/c$ pigeonite seemed to occur at the positions where the H APBs had been originally located before the first heating (Fig. 6).

DISCUSSION

Characteristics of the $P2_1/c$-$C2/c$ phase transition, such as its athermal nature and interface movement observed in the present HTTEM study, indicate that the phase transition belongs to a thermoelastic type of martensitic transformation, as discussed in detail by Shimobayashi and Kitamura (1991). The $P2_1/c$-$C2/c$ phase transition occurs athermally, and high- and low-pigeonite phases coexist over the transition interval (~100 °C). The different behaviors of the two types of APBs in pigeonite in the transition interval, revealed by the present HTTEM observation, are as follows: (1) H and L APBs are formed at different stages in the transition interval during cooling, and (2) $C2/c$ pigeonite grows from the H APBs but not from the L APBs at the beginning of the transition interval during heating. Based on these two characteristics, the nature and behavior of these two types of APBs are discussed below.

Formation of H and L APBs

The L APBs formed at the earlier stage of the transition interval, almost simultaneously with the formation of the

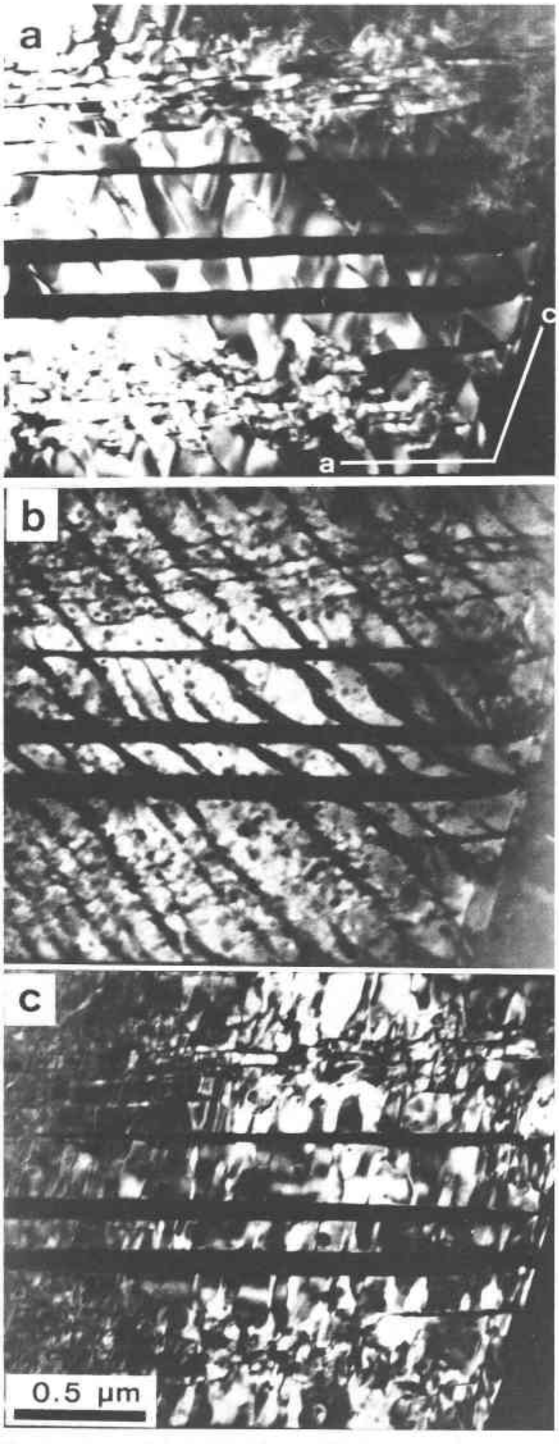

Fig. 5. A set of dark-field (**g** = 102) electron micrographs in the first heat treatment of the pigeonite (**a**) at room temperature before heating, (**b**) photographed in situ at 1000 °C, (**c**) at room temperature after cooling. The coexistence of low and high pigeonite is observed in **b**. The change in the orientation of APBs is shown between **a** (before) and **c** (after) the HTTEM heating experiment.

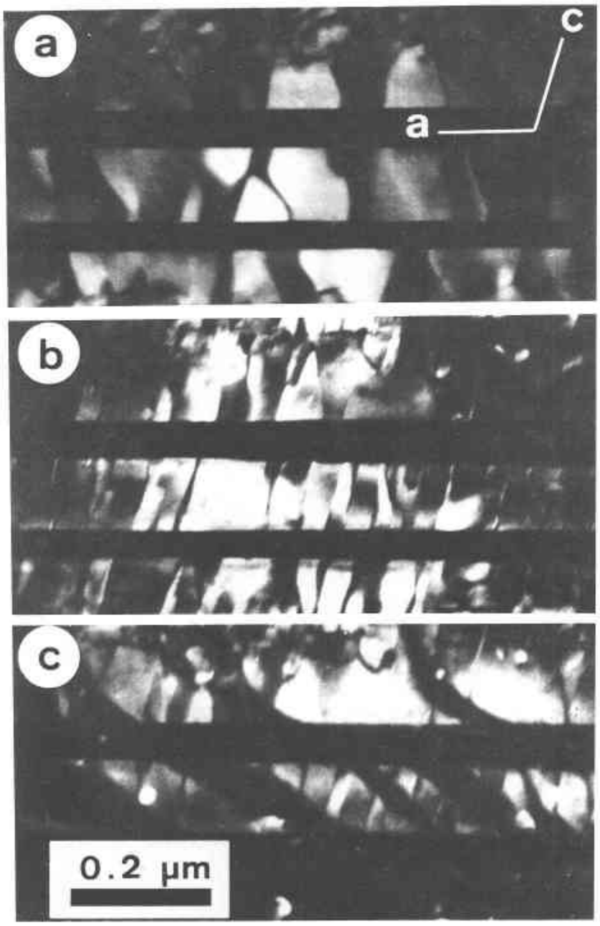

Fig. 6. A set of dark-field (**g** = 102) electron micrographs (**a**) at room temperature before heating, (**b**) at room temperature after the first heat treatment, (**c**) photographed in situ at 960 °C in the second heat treatment. The orientation of the interfaces is almost the same as in the first heat treatment. The growth of *C*2/*c* pigeonite seems to occur from the trace of the original H APBs but not from the L APBs.

$P2_1/c$-pigeonite regions during cooling. That is, each $P2_1/c$-pigeonite region, bounded by two coarse augite lamellae and two $C2/c$-pigeonite regions, was generally composed of several APDs of low pigeonite separated by L APBs (Figs. 4a and 4b). This type of APB, which should be formed by the nucleation and thermally activated growth process, is common in alloys and minerals. The number of L APBs would depend upon the nucleation frequency of $P2_1/c$ pigeonite.

The H APBs have also been considered to be formed by the same nucleation and growth process of $P2_1/c$ pigeonite as the L APBs. However, the present observations clearly show that the H APBs are controlled by the movement of interfaces between $P2_1/c$ and $C2/c$ pigeonite in the transition interval. The H APBs formed at the final stage of the $C2/c$ to $P2_1/c$ pigeonite transition interval, apparently as the "traces" of these interfaces (Fig.

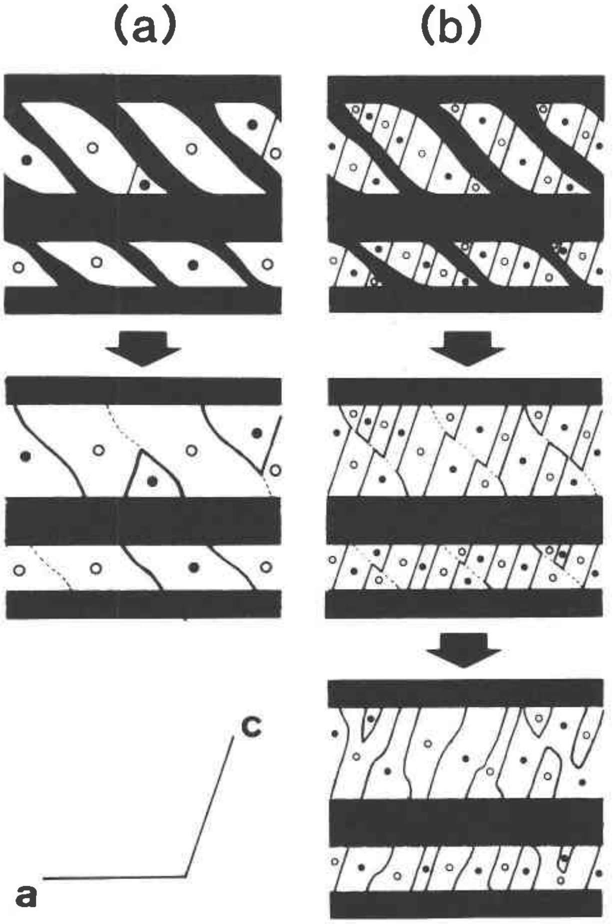

Fig. 7. Schematic illustration of the formation of APBs in "001" pigeonite for (**a**) the case in which L APBs are scarce (i.e., during very slow cooling), (**b**) the case in which L APBs are abundant (i.e., result of quenching). Hollow and solid circles represent the phase of domains. If the adjacent domains with the same phase (○-○ or ●-●) grow into contact, they coalesce into one domain; while between two domains with opposite phase (○-●), an APB is produced.

4d). Shimobayashi and Kitamura (1991) carried out the calculation of the energy for the interface on the basis of a coherent elastic model (Willaime and Brown, 1974) combined with the lattice parameter data from the high-temperature X-ray study of clinohypersthene of Smyth (1974). They pointed out that the orientation of these interfaces is almost consistent with the direction for one of the energy minima. That is, the orientation of the H APBs crossing the **c** axis can be explained by the orientation of low-energy interfaces in the athermal transition, although Carpenter (1978) considered that the H APBs behaved as high-energy dislocations. The curvature of the interfaces seems to be affected by a strain field associated with the coarse "001" augite lamellae and the Ca concentration gradient in the pigeonite lamellae.

If two $P2_1/c$-pigeonite regions on opposite sides of a $C2/c$-pigeonite region in the transition interval are in antiphase relation, the trace (shown by broken lines in Fig. 7) remains as an H APB; if not, it cannot remain as an APB and vanishes without leaving any trace of the $C2/c$ pigeonite. In the present case, however, the mechanism of the APB formation is complicated because of the presence of the L APBs in the $P2_1/c$-pigeonite regions, i.e., L APBs interfere with the formation of H APBs. At slower cooling, since the L APBs seem to be sparse (i.e., size of the APDs is large) due to the coarsening of the APDs, this interference process is less effective and H APBs are retained after cooling (Fig. 7a). In the case of fast cooling, on the contrary, the L APBs are abundant (i.e., size of the APDs is small) because the coarsening of the APDs could not fully occur during the transition interval. At the end of the transition, the preexisting L APBs cut the trace of the $C2/c$ pigeonite into segments, some of which were removed. A mixed type of APB, connected with the remaining short segments (H APB) and L APBs, was formed as a necessary consequence. This APB cannot migrate because of the drag effect of the Ca ions (Carpenter, 1978) but may change its shape by reorienting itself to minimize the surface free energy during further cooling (Fig. 7b). Therefore, the mixed APB is inclined to the **c** axis at a smaller angle than the H APBs. The angle between this APB and the **c** axis seems to decrease with the decreasing number of the L APBs, i.e., the angle depends upon the cooling rate.

This above deduction from the present HTTEM observations confirms the proposition experimentally derived by Fujino et al. (1988), that "the preferred orientation of APBs in '001' pigeonite depends on the cooling rate; at fast cooling rates the APBs are nearly parallel to the **c** axis, whereas at slower cooling rates they are inclined to the **c** axis." The orientation of APBs has the capability of providing an estimate of cooling rates.

Nucleation and growth from APBs

The APBs in pigeonite are thought to locally have the $C2/c$ structure (Morimoto and Tokonami, 1969) where Ca^{2+} is concentrated. Carpenter (1978) proposed simple structural models to suggest that the H APBs provide more suitable sites for Ca^{2+} ions than the L APBs. Consequently, the present original specimen of natural pigeonite would be expected to have Ca^{2+} ions concentrated more on the H APBs than on the L APBs. Based on this assumption of the Ca concentration, the observed behavior of the APBs can be explained as follows.

During the first heating of the present study, the transition started from the H APBs which acted as nuclei of $C2/c$ pigeonite. Because of the slowness of Ca diffusion, the Ca^{2+} ions in the pigeonite specimen could not fully migrate from their locations during the present HTTEM experiments. During cooling, the nucleation of low pigeonite occurred at points away from these Ca-rich regions. $C2/c$ pigeonite remained, therefore, until the end of the transition interval at these Ca-rich regions. As described before, two types of APBs interact at the end of the transition interval. Without the interaction with L APBs, some of the *C*2/*c*-pigeonite regions would remain at their original positions as H APBs. In fact, previous HTTEM work on anorthite (Van Tendeloo et al., 1989) and on kanoite (Gordon et al., 1981) showed that both the orientation and position of the APBs were essentially the same before and after heating through the transition temperatures. This behavior, called the "memory effect," has been explained as due to the stabilization of APBs by the concentration of Ca or by some defect at the APBs (Gordon et al., 1981). The memory effect, however, could not be confirmed for the APBs of pigeonite in the present study.

In subsequent heating, the growth of *C*2/*c* pigeonite occurred from traces of preexisting, but no longer present, APBs of the former *C*2/*c*-pigeonite regions as well as from the H APBs. On the other hand, the L APBs showed no association with the growth of *C*2/*c* pigeonite. The fact that H APB but not L APB acted as a nucleus for *C*2/*c* pigeonite should be interpreted in conjunction with the Ca distribution in the pigeonite specimen. The Ca enrichment seems to lower the transition temperature locally, so that regions where Ca^{2+} ions concentrated were easily transformed from $P2_1/c$ to *C*2/*c* pigeonite on heating but were difficult to transform from *C*2/*c* to $P2_1/c$ pigeonite on cooling.

Application to APBs in natural pigeonite

Starting specimens used in the annealing experiments of Fujino et al. (1988) and the present HTTEM study are thought to be compositionally heterogeneous, especially with respect to the Ca distribution, because of the APD texture. On the other hand, APBs in natural pigeonite are thought to have been formed during the cooling from "homogeneous" *C*2/*c* pigeonite, free from local Ca enrichment either on the APBs or elsewhere. Homogeneous *C*2/*c* pigeonite could not be obtained in the present heating experiments chiefly because of the slowness of Ca diffusion. In addition, the cooling rates in the heating experiments are generally much faster than those in nature. Consequently, the APB configurations in slowly cooled natural pigeonite could not be reproduced in the laboratory. Combining the present HTTEM observation with suggestions of Carpenter (1978), a hypothesis regarding the behavior of APBs under natural conditions is proposed as follows.

During slow cooling, a small number of $P2_1/c$-pigeonite domains nucleate in the *C*2/*c*-pigeonite host. If two $P2_1/c$-pigeonite domains in antiphase relation grow into contact, an L APB is produced. The $P2_1/c$-pigeonite regions, including L APBs, coexist with *C*2/*c*-pigeonite regions in the transition interval. With very slow cooling, the pigeonite remains at high temperatures long enough for Ca^{2+} ions to diffuse to the *C*2/*c*-pigeonite regions and concentrate there. Carpenter (1978) suggested that the L APBs, which are less affected by the Ca^{2+} ions, can migrate faster than the H APBs. Therefore, L APBs may migrate and be lost (coarsening the APDs) during cooling in the transition interval. Consequently, the traces of the *C*2/*c*-pigeonite regions are rarely cut by L APBs at the end of the interval (Fig. 7a). If two domains on both sides of one *C*2/*c* pigeonite trace are in antiphase relation, the trace remains as an H APB with Ca enrichment. The H APBs may be fixed by the concentration of Ca ions whose diffusion rate becomes more sluggish as the cooling proceeds. Consequently, H APBs are predominant in the slowly cooled pigeonite.

In the case of rapid cooling, on the other hand, the coarsening of APDs cannot fully occur in the $P2_1/c$-pigeonite regions. The L APBs formed at an earlier stage of the transition interval may be preserved in $P2_1/c$ pigeonite. Because the traces of the *C*2/*c*-pigeonite regions interact with the L APBs at the end of the interval, the resulting APB configurations are quite complicated as discussed above (Fig. 7b). As a result, L APBs or APBs nearly parallel to the **c** axis predominate in quenched pigeonite.

According to the above hypothesis, the fact that the H APBs are observed in slowly cooled pigeonite, whereas the APBs nearly parallel to the **c** axis (L type or its analogues) are found in quenched pigeonite, is explained qualitatively.

ACKNOWLEDGMENTS

I would like to thank Masao Kitamura for his numerous discussions and his critical reading of this manuscript. I also would like to thank Nobuo Morimoto for his continuing suggestions and helpful comments. Critical reviews by Tamsin C. McCormick and Roy G. Christoffersen were very helpful and aided in improving this manuscript.

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Manuscript received December 27, 1990
Manuscript accepted August 18, 1991