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An electron-microscopic and X-ray study of complex exsolution textures in a cryptoperthitic alkali feldspar

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The configurations and geometrical relations of the exsolution† lamellae from a cryptoperthite from the Wausau, Wisconsin syenite have been studied by transmission electron microscopic and X-ray methods. By using bright and dark field techniques it has been possible to relate the lattice geometries, the configurations of the exsolved regions and their composition planes. Two major configurations were found of alternating sodic and potassic lamellae: (a) albite-twinned low albite and maximum microcline in diagonal configuration with the boundary plane near $(\bar{8}\bar{6}1)$: (b) pericline-twinned low albite and finely twinned M-type microcline with the boundary plane near $(\bar{8}01)$. The second configuration appears to be more abundant. The two configurations do not appear to alternate periodically and the numbers of lamellae in each configuration appears to vary from grain to grain. The twins in the low-albite lamellae are periodic and give rise to satellite spots.

The lattice angles appear to be nearly normal for the various regions, whereas the cell edges are strained. The diffraction spots are joined by streaks in some cases. The boundaries are thus essentially coherent. The b^* axis for low albite in configuration (a) is parallel to b in configuration (b), although they occur in different areas.

1. Introduction

Alkali feldspars are tectosilicates of general formula $(K,Na)AlSi_3O_8$. At high temperature the equilibrium state is a complete series of solid solutions between $NaAlSi_3O_8$ (albite, *Ab*) and $KAlSi_3O_8$ (sanidine or orthoclase, *Or*) with space-group $C2/m$. On cooling three processes may occur.

(1) A reversible displacive monoclinic/triclinic $(C\bar{1})$ symmetry inversion in $NaAlSi_3O_8$-rich (*Ab*-rich) feldspars, the temperature of inversion decreasing from near 1000°C for albite to room temperature for $Ab_{40}Or_{60}$.

(2) The atoms of sodium and potassium segregate into lamellar regions in the crystal.

(3) The atoms of aluminium and silicon, which are disordered at high temperature, become ordered at lower temperature. This ordering gives rise to a diffusive [1] symmetry change $(C2/m \rightarrow C\bar{1})$ in *Or*-rich feldspars, producing (triclinic) *microcline*.

The first process is instantaneous, the second very rapid (hours to days), whereas the third is very slow (years to thousands of years). The microtextures produced on cooling in rocks thus depend on the bulk composition and on the cooling-rate, and a simplified cooling sequence can be established for a feldspar of intermediate composition: a homogeneous monoclinic feldspar; two monoclinic regions; monoclinic/triclinic regions, the *Or*-rich regions being monoclinic, the *Ab*-rich ones triclinic; triclinic regions. The exsolution textures may be coarse or fine, the scale depending on the cooling rate. The fine-scale textures studied here are called *cryptoperthites*, and this paper describes in detail an example of a complex configuration with only triclinic regions.

†"Exsolution" is the mineralogical term for precipitation.

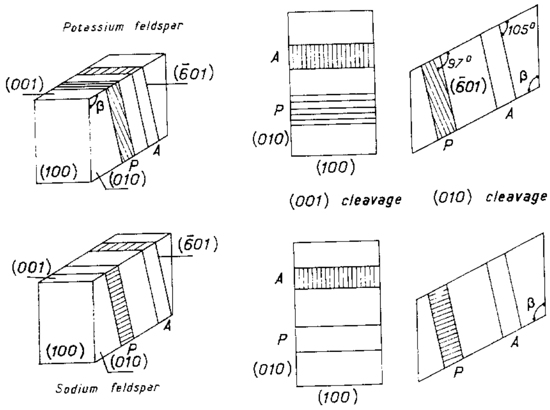

Figure 1 Schematic representations of the orientations of the exsolution lamellae and the twin domains for potassium and sodium feldspar as a block diagram and on (001) and (0 1 0) cleavages. Above: albite- (A) and pericline- (P) twinned K-rich lamellae in an Na-rich matrix. Below: albite- (A) and pericline- (P) twinned Na-rich lamellae in a K-rich matrix. The pericline twin planes shown correspond to high albite with σ small and positive.

The triclinic feldspars are pseudo-monoclinic, the obliquity ($b \wedge b^*$) being less than about 4°, so that twinning by pseudosymmetry is frequent (Fig. 1) according to Albite Law ((0 1 0) is the twin and composition plane) or the Pericline Law ([0 1 0] is the twin axis and the composition plane is the rhombic section, an irrational plane of indices (h 0 l) whose position depends on the lattice angles at the moment of twinning. The rhombic section orientation is defined by the σ angle between the a-axis and the trace of the rhombic section on (0 1 0).)

The exsolution domains are lamellar in form and their orientation is determined by minimum elastic energy in the boundary [2]. In general, at the beginning of exsolution the boundary is near ($\bar{6}$0 1) (Fig. 1 and [3]), but when potassic lamellae become triclinic this boundary may be near ($\bar{6}\bar{6}$1) [4, 5].

The textures and structures obtained are highly complex and have been studied in detail only by X-ray methods. Mackenzie and Smith [6] have established a more complete cooling sequence. The lattice parameters of the albite- or pericline-twinned sodic feldspar are not generally the same, as was first pointed out by Chao and Taylor [7], and it is now known that the first is generally close to low-albite and the second to high-albite though more complex cases occur [6]. Pericline-twinned high-albite cryptoperthites are easily homogenized, whereas albite-twinned low-albite cryptoperthites are more difficult to homogenize.

2. Experimental methods

The cryptoperthites were studied by the X-ray precession method in order to obtain the orientation relations of the regions and their lattice parameters. They were also studied by electron microscopy with a 100kV Philips EM 300 microscope fitted with a goniometer stage. Samples were prepared by MacLaren and Phakey's technique [8] by crushing small crystals between two glass slides. Fortunately, feldspars have two very good cleavages, {0 0 1} and {0 1 0}, so that thin flakes up to several microns in diameter were easily obtainable and the textures looked for can be seen on these cleavages by transmission.

3. Examples of simple cryptoperthites configurations

In rapidly cooled rocks it is possible to find cryptoperthites with two monoclinic regions. Fig. 2 shows a micrograph of an (0 1 0) cleavage of a sanidine cryptoperthite from Samothraki Island, Greece. In places pericline-twinned regions occur in the sodic feldspar. This is borne out by X-ray diffraction–long-exposure photos show traces of pericline twins, contrary to the observations of Soldatos [9] on similar material.

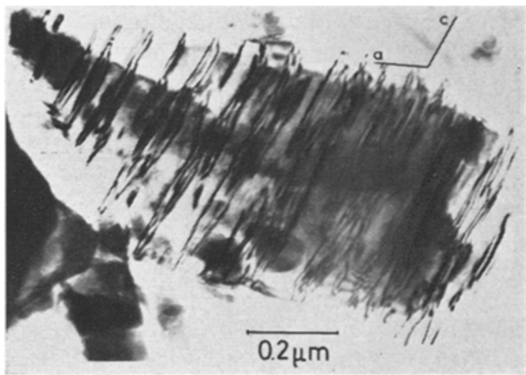

Figure 2 (0 1 0) transmission photograph of sanidine cryptoperthite from Samothraki Island, Greece showing two monoclinic regions, the more abundant being K-rich σ for lamellar boundary near 117°.

Cryptoperthites with only pericline-twinned or only albite-twinned sodic regions (Fig. 3) associated with monoclinic potassic regions are very common and the latter were first studied by

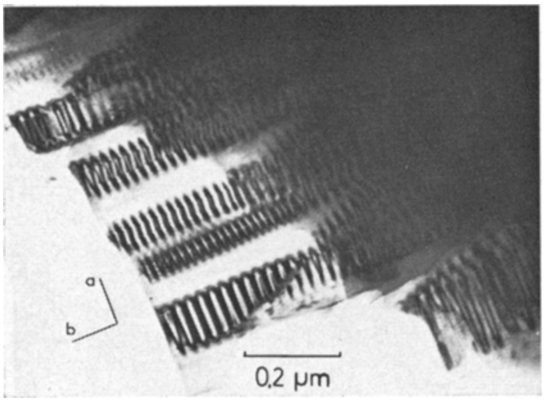

Figure 3 (001) transmission photograph of cryptoperthite F99 from near Larvik, Norway with periodic albite twins in low albite lamellae and monoclinic potassium feldspar.

electron microscopy by Fleet and Ribbe [10]. In such albite-twinned cryptoperthites, the twin period depends on the width of the sodium-rich regions [11] and a similar result is predicted for pericline twins.

In one case of a cryptoperthite with only triclinic regions, the potassium feldspar is in the diagonal association [12] and the sodium feldspar is albite-twinned. Fig. 4 shows an aspect of the geometry of such a feldspar. Zig-zag or lozenge-shaped zones of sodic feldspar are separated by albite-twinned microcline [4, 5].

4. Description of a complex multi-domain cryptoperthite

A systematic X-ray examination of many perthites by MacKenzie and Smith [6] has shown the existence of albite and pericline-twinned

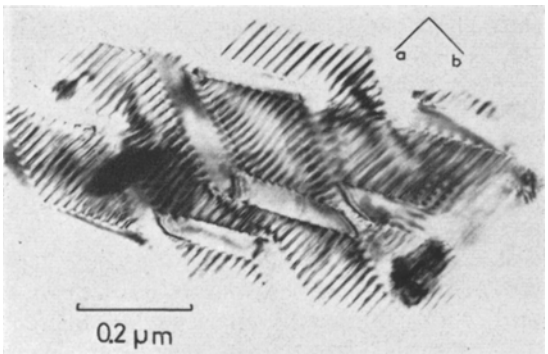

Figure 4 (001) transmission photograph of cryptoperthite L29 from Wausau, Wisconsin showing diagonal association. Composition plane near $(\bar{6}\bar{6}1)$.

sodium feldspar associated with twinned potassium feldspar, though they were unable to determine the configurations of the regions. This paper describes in detail a complex cryptoperthite L31 of composition Or_{35} Ab_{62} An_3 from microprobe analysis, from a syenite from Wausau, Wisconsin. This is a different sample from the one previously studied [4, 5]. Only by combining X-ray and bright and dark-field electron microscopy with electron diffraction was it possible to determine the configuration of this cryptoperthite.

4.1. Observations with bright-field electron microscopy

The exsolution configurations vary markedly in different preparations from the same crystal. Indeed, some grains observed up to a few micrometers across did not show any exsolution, but were composed of albite and pericline-twinned microcline.

Exsolution textures are well seen on (001) and (010) cleavages of most fragments. It is clearly seen on (001) that there are at least two distinct exsolution configurations intimately associated on the same cleavage fragment (Figs. 5 and 6). The number of adjacent zig-zag or lozenge-shaped sodic bands varies from grain to grain; three in Fig. 5 and only one in Fig. 6. The structure is more clearly developed when the number of bands increases. The centre portion of the composite band in Fig. 5 is very typical and the boundary approaches $(\bar{6}\bar{6}1)$ for the potassic bands [4, 5]. In Fig. 6 the sodic bands are more wavy than angular. The other type of configuration is also seen in Figs. 5 and 6 and appears as straight bands with a boundary surface near $(\bar{6}01)$. A periodicity at the scale of the grain in the arrangement of these two types of configuration was looked for in many grains but not found.

4.2. Determination of the nature of the domains by dark-field electron microscopy

As the width of the exsolution bands is only of the order of 0.2µm, it was impossible to obtain selected-field microdiffractions from only one. The diffraction patterns of the various grains are quite complex and (001) cleavages may contain superposition of the different types as well as microcline with microcline-type twinning (M-type) [13]. Satellite spots may also occur because of the periodic twinning; for albite

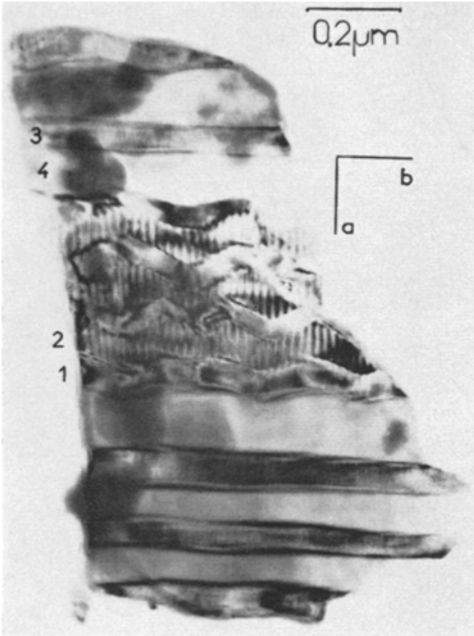

Figure 5 (001) transmission photograph of two configurations in L31 (a) diagonal configuration between 1 and 2 (b) M-type twinned microcline (3) with pericline-twinned low albite, 4. The M-type twinning in 3 is seen as fine cross-hatching, whereas the pericline twins are not visible in 4.

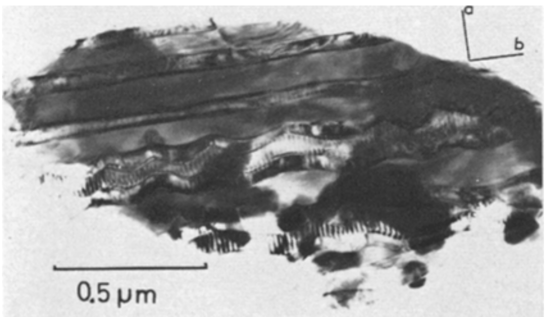

Figure 6 (001) transmission photograph of L31 showing similar configurations as in Fig. 5. Note the different lamellar sequence and the fact that the albite-twinned sodic lamellae are wavy.

twins the satellites are in the ($hk0$) plane, whereas this is not the case for pericline twins. Thus by tilting the specimen with pericline twins it is possible to illuminate the different spots successively.

The dark-field technique can be used to determine the correlation between the different diffraction spots and the regions. The angle between the 200 spots for potassium-rich and sodium-rich bands is about 1.5′ of arc, while that between satellite spots corresponding to either band is less than 1′ of arc. The angular aperture is about 10′ with a 10μm objective aperture. Thus the dark-field images are obtained under poor conditions: astigmatism due to the fact that the satellite spot selected cannot be at the centre of the aperture. Images could also be taken with second, third or higher order reflections but with such a long exposure time the specimen suffered beam damage. Nevertheless by combining dark-field techniques with sample tilting, it was possible to determine the nature of the different regions.

On both (001) and (010) cleavages, potassium and sodium-rich bands alternate and each band has only one texture on the scale of several μm irrespective of its composition. The regions may be associated to form composite textures. Dark-field images of the zig-zag regions on (001) cleavages (1 on Fig. 5) show that they correspond to diagonally associated microcline; between two such regions is found lozenge-shaped albite-twinned albite (2 on Fig. 5). They thus resemble the diagonal configuration found in another sample from the same rock [4] and shown in Fig. 4.

The wide sodic lamellae (4 on Fig. 5) show no diffraction contrast on (001) bright-field photos. On (010) cleavage flakes they are seen to consist of periodically pericline-twinned albite (Fig. 7). The angle between the twin composition plane and the (001) cleavage is about 35° and that between the lamellar boundary and the trace of (001) is 108° corresponding approximately to ($\bar{8}01$). The narrow intervening potassic lamellae (3 on Fig. 5) show the typical contrast of albite and pericline-twinned microcline on dark-field photos. The albite twins are not visible on (010) cleavages, whereas the pericline twins show up clearly. The composition plane of pericline twins in potassic feldspars is nearly perpendicular to the (001) cleavage plane, explaining why they are visible on (001). Neither twin is periodic. The (001) and (010) cleavage images do not necessarily correspond as they are made on different fragments and may have different sequences and scales of textures.

Ten diffraction spots are thus observable in different cleavage flakes from this cryptoperthite

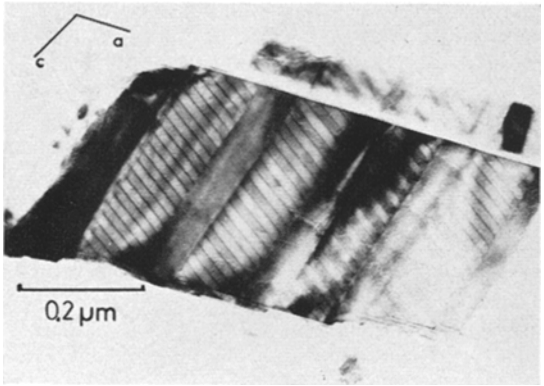

Figure 7 (010) transmission photograph of L31 with σ for lamellar boundary near 108° and for pericline twins in low albite near 35°.

for a given *hkl* (the numbers refer to regions on Fig. 5).

Potassic regions	1 diagonal associated microcline	2 orientations
	3 M twinned microcline	4 orientations
Sodic regions	2 albite-twinned albite	2 orientations
	4 pericline-twinned albite	2 orientations

Superstructure spots arise because of the periodicity of the twins in 2 and 4. Streaks join the spots in 1 and 3.

4.3. X-ray diffraction

X-ray diffraction on a single crystal gives a volume average of that which may be seen on many electron transmission photographs. Precession photographs were carefully taken of the different reciprocal lattice planes in order to obtain their relative orientations and to obtain the corresponding lattice parameters. Unit cell parameters were measured for both sodic domains and for the diagonally associated microcline and are given below.

Domain	*a*(Å)	*b*(Å)	*c*(Å)
1 potassic	8.652	12.895	7.186
2 sodic	8.113	12.820	7.176
4 sodic	8.127	12.845	7.176

Domain	α	β	γ	Vol. (Å³)
1 potassic	91.15°	116.35°	87.40°	716.4
2 sodic	93.87°	116.71°	88.27°	665.3
4 sodic	93.85°	116.75°	87.80°	667.4

The lattice parameters for bands 1 and 2 are nearly the same as those for similar material, L29, only showing the diagonal configuration [4]. They are perhaps slightly more strained and the texture is perhaps more coherent [5]. The pericline-twinned bands, 4, have almost identical lattice angles, whereas the cell edges differ more; they are more strained if the regions have the same composition as 2 or more potassic if they are equally coherent. Both 2 and 4 are nearly pure low albite, while 1 is maximum microcline. It was not possible to measure the parameters of the M-type microcline (region 3) as it corresponds to microcline of very low obliquity and only gave streaks on X-ray photographs.

The orientation relations for the albite- and pericline-twinned low albite (2 and 4) were of M-type [14] i.e. *b* for the pericline twins was parallel to *b** for the albite twins as originally found for cross-hatched microcline by Laves [14]. and also found for 3. In addition all these directions coincided. 1 had the typical diagonal orientation [4, 12]. Streaks joined the spots corresponding to each of the regions.

5. Conclusions

The configurations and the geometrical relations of the various lamellae in simple or complex cryptoperthites can best be studied by combining X-ray and transmission electron microscopy. The complex cryptoperthite from the Wausau syenite has two major configurations. (a) Albite-twinned low albite and maximum microcline in the diagonal configuration, the boundary plane approaching ($\bar{8}\bar{6}1$); the number of sodic lamellae varies from one to four or five and only the intervening potassic lamellae are albite-twinned maximum microcline. (b) Pericline-twinned low albite associated with M-type twinned microcline with a plane near ($\bar{8}01$) as boundary surface. This configuration appears to be more abundant. The geometrical relations of the lattices of albite and pericline twinned low albite is of M-type in spite of the fact that they are in different configurations. The lattices are strained and the lamellae are probably essentially coherent.

No detailed explanation can be given at present for this association of two major configurations in this cryptoperthite.

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