Solution–redeposition and the orthoclase–microcline transformation: evidence from granulites and relevance to ^{18}O exchange

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Abstract

The Or-rich part of optically blebby to lamellar mesoperthite crystals from an Adirondack granulite has been shown by TEM to be a lamellar cryptoperthite, composed dominantly of tweed orthoclase. A fluid-absent, two-stage thermal history is proposed to explain the coarse and fine textures, with the cryptoperthite forming by coherent exsolution below ~350 °C, probably during uplift. The mechanism was most probably homogeneous coherent nucleation rather than spinodal decomposition. However, cutting the orthoclase cryptoperthite are thin (<1 μm) seams of tartan microcline with sharp boundaries, often defined locally by {110} planes, and micropores. The microcline has replaced orthoclase by solution–redeposition along narrow planes infiltrated by fluid during minor retrogression at $T < 350$ °C. Solution–redeposition is a common process in feldspars at $T < 500$ °C, potentially accompanied by ^{18}O exchange, because release of elastic strain energy in coherent perthite lamellar boundaries and twin-domain walls, followed by Si, Al ordering, provide driving forces for dissolution and reprecipitation of unstrained, more ordered phases.

KEYWORDS: orthoclase–microcline transformation, solution–redeposition, granulite, ^{18}O exchange, perthite, Adirondacks, low-temperature reactivity.

Introduction

IN this paper we present electron micrographs of granulite-facies potassium feldspars which suggest that the orthoclase → microcline, monoclinic → triclinic phase transformation may take place locally by an intracrystalline solution–redeposition process. Our observations are consistent with long-recognised circumstantial evidence that fluid–feldspar interactions are often essential if this transformation is to occur. We discuss evidence for solution–redeposition processes in feldspars in general, their significance in oxygen isotope exchange, and consider the nature of the thermodynamic driving forces in feldspar which are responsible for its remarkable low-T reactivity.

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The conversion of monoclinic to triclinic potassium feldspar has not been achieved experimentally, although the nature of the structural change is now reasonably well understood. As illustrated by Eggleton and Buseck (1980), orthoclase is characterised by a 'tweed' microtexture of ordered–antiordered twin domains on the scale of a few unit cells. This develops during the transformation from monoclinic low sanidine to pseudomonoclinic orthoclase which accompanies Si, Al ordering. Once the tweed texture has developed, further ordering cannot easily occur, and the tweed texture persists, because of the difficulty of reversing the ordering sense of ordered and antiordered domains. Eggleton and Buseck (op. cit.) proposed that the decrease in free energy obtained by Si,Al ordering was balanced by the increase in strain energy associated with the domains. The total driving force for ordering is at first very small and finally falls to zero, so that the partially ordered orthoclase tweed texture becomes energetically stranded. In their review of current ideas about ordering and phase transformation in alkali feldspars, Brown and Parsons (1989) favoured the view that orthoclase is not a truly stable phase at any *T*, although it may be considered to be in 'constrained' equilibrium where the constraint is conservation of the microtexture.

Low microcline is nearly fully ordered and is a stable phase below ~400 °C at low pressure. It is characterised optically by the irregular 'tartan' microtexture of coarse, intersecting Albite and Pericline twins. The status of intermediate and high microcline is somewhat controversial. Brown and Parsons (op. cit.) proposed that the low sanidine → high microcline transformation is thermodynamically continuous in character and that there is a narrow range of *T* over which intermediate microcline is stable. They suggested that intermediate microcline is rare because Si,Al ordering is not intrinsically slow and that once the barrier represented by tweed texture is overcome or sidestepped, ordering proceeds rapidly, leading to low microcline. An alternative view (Kroll and Ribbe, 1983), based on the common coexistence of orthoclase and microcline in rocks, is that the low sanidine → microcline transformation is thermodynamically discontinuous and first order.

Our present study provides no new insights into the order of the phase transformation but is concerned with the mechanism by which the textural barrier to ordering represented by the tweed microtexture may be overcome in Or-rich feldspars. Brown and Parsons (1989) referred to this process as the 'unzipping' of orthoclase and, in line with many earlier proposals (e.g. Parsons and Boyd, 1971), suggested that it could occur either by feldspar–fluid interaction or as a result of external stress, or both. Many studies of Or-rich feldspars in plutonic igneous and metamorphic rocks, mostly using X-ray diffraction, have shown that the feldspar is either orthoclase, nearly fully ordered low microcline, or mixtures of the two. Transmission Electron Microscope (TEM) studies (Fitz Gerald and McLaren, 1982; Zeitler and Fitz Gerald, 1986; Bambauer *et al.*, 1989; White and Barnett, 1990) have shown that individual crystals may be intimate mixtures of tweed orthoclase and tartan microcline. The boundaries imaged between the two types of texture are either (*1*) continuous but sharp, or (*2*) gradational over a range of distances up to about 1 μm (e.g. fig. 11 in Fitz Gerald and McLaren, 1982; also fig. 2 in Bambauer *et al.*, 1989, but note that the micrograph has been exchanged with fig. 1) and (3) sometimes sharp and apparently incoherent (e.g. fig. 3 in White and Barnett, 1990). These micrographs do not, however, provide any clues as to how the conversion of tweed to tartan may have occurred.

Worden *et al.* (1990) used TEM to demonstrate pervasive structural rearrangements accompanying coarsening of strain-controlled cryptoperthite to patch microperthite, in an Ab-rich alkali feldspar ($Ab_{67}Or_{32}An_1$) from the Klokken syenite intrusion. This coarsening was associated with the development of turbidity, caused by large numbers of μm-scale micropores, and with the development of a subgrain mosaic. Perthite interfaces are complex, irregular and sometimes semi-coherent, and in places tartan microcline replaces tweed or diagonal microcline in the original coherent perthite. Growth of new feldspar, with {110} habit characteristic of Adularia, was shown to have occurred near micropores (their fig. 7C). Similar subgrain walls and dislocations were illustrated by Zeitler and Fitz Gerald (1986) and White and Barnett (1990) and we have found them to be characteristic of normally turbid feldspar from a wide range of rocks. Guthrie and Veblen (1991) also illustrated the relationship between turbidity and perthite coarsening, in high level granites from the Isle of Skye, and suggested that the textures record interactions with at least two fluids. Stable isotope work by Taylor and Forester (1971) and Forester and Taylor (1977) showed that the Skye rocks had suffered extensive interaction with meteoric fluids. Ferry (1985) showed that the scale of isotopic exchange could be correlated with the development of turbidity in the feldspars which accompanied breakdown of optically homogeneous alkali feldspar to irregular microperthite. He concluded that ^{18}O–^{16}O exchange occurred synchronously with this recrystallisation. It seems certain that in the Klokken syenite and in the Skye granites, pervasive fluid–feldspar interactions have occurred leading to major structural rearrangements within crystals that have not materially changed their outline. In the present paper we illustrate features in Or-rich feldspars in mildly retrogressed granulite-facies rocks which imply similar, but more subtle, interactions and which clearly lead to the orthoclase-microcline transition by a solution–redeposition process.

Geological setting and methods

Samples from the Adirondack Mountains were provided by J. M. McLelland (sample numbers 6-2-86-2 and 6-2-86-6). They are from a suite of charnockite/mangerite rocks (collected near Tupper Lake, NY) adjacent to massif anorthosites of the Adirondack Highlands. Charnockite/mangerite rocks and anorthosites are commonly found associated with one another in both space and time. Many workers believe the charnockite/mangerite suite and the anorthosite appear to have been emplaced together as a bimodal anorogenic complex (McLelland, 1991). Whatever the early history of the charnockite/mangerite suite, it was then metamorphosed at high pressure in the granulite facies, and has subsequently undergone a relatively mild phase of retrograde metamorphism.

Samples were prepared for TEM analysis from doubly-polished thin sections of suitably orientated feldspar crystals [(001) sections] which were identified and photographed using light microscopy. Final thinning of the samples was achieved with an Iontech atom mill using Ar as the sputtering agent. All TEM observations were made with a Philips CM12 microscope operating at 120 kV. Feldspar compositions were determined by wavelength dispersive microprobe analyses using a Cameca Camebax microprobe operating at 20 kV with a sample current of 10 nA. Na-loss was minimised by rastering the electron beam during analysis.

Description

The rock samples examined contain mesoperthitic alkali feldspar, quartz and minor amounts (<10 modal %) of hornblende and retrograde chlorite/biotite intergrowths. The mesoperthite crystals are irregularly shaped and 1–3 mm in diameter. The perthites are generally pristine, with turbidity restricted to diffuse areas and bands that crosscut the exsolution textures. Exsolution features visible with light microscopy in (001) sections (Fig. 1) comprise rounded Ab-rich blebs (10–30 μm wide and up to 2 mm long) with oblique tails which intersect fine (1–2 μm wide), straight Ab-rich lamellae set in featureless Or-rich feldspar. Elemental imaging in the electron probe showed areas with three compositions: blebs are oligoclase with negligible K (maximum Or_1, average composition $\sim Ab_{85}An_{15}$); the tails are close to pure albite ($Ab_{98}An_1Or_1$), and (on the basis of their appearance in the elemental images) are probably similar in composition to the fine lamellae in the Or-rich areas, although the fine lamellae could not be measured directly; Or-rich areas, containing fine Ab-rich lamellae, which have bulk compositions (obtained by rastering the probe beam over a 15 × 15 μm area) of $\sim Ab_{15}Or_{85}$ [maximum $(An + Cs)_1$]. The composition of the Or-rich phase in these areas could not be estimated reliably because TEM showed all areas to be cryptoperthitic. The most extreme composition obtained from the Or-rich areas was $Ab_8Or_{92}An_0$ so that the Or-rich phase is $\geq Or_{92}$.

In the cryptoperthite (darkest grey on Fig. 1), very fine (0.05–0.10 μm wide, up to 1 μm long), straight lenses of Albite-twinned albite, with long axes parallel to *b*, are coherently intergrown with tweed-textured orthoclase (Fig. 2A) Lattice fringes, visible on the original micrograph, show this intergrowth to be fully coherent. To the best of our knowledge, this is the first reported occurrence of cryptoperthitic feldspar in a granulite-facies rock although optical mesoperthite, often on a coarse scale, has been reported many times (see Addendum).

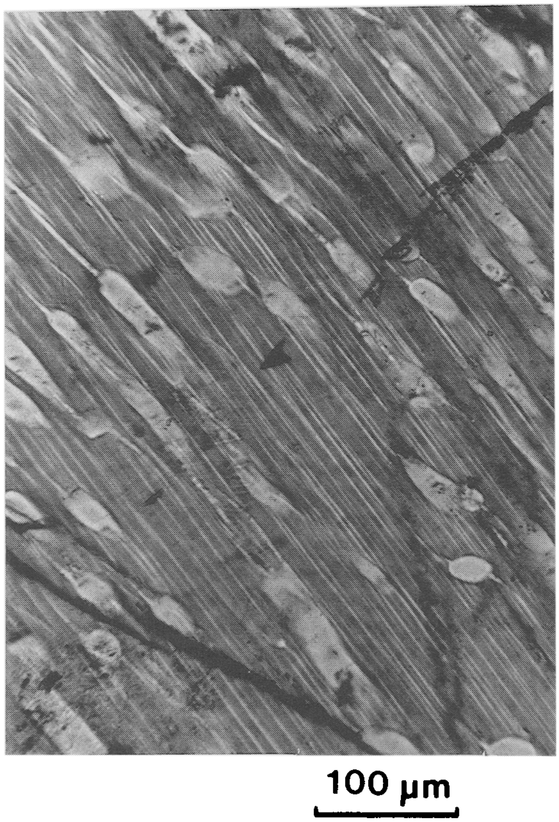

FIG. 1. Optical micrograph of mesoperthite in sample 6-2-86-6. Two sets of exsolution features are visible: coarse, round blebs and find, straight lamellae. Visible turbidity is restricted to diffuse areas (lower left, and top). The section is roughly parallel to (001) and the fine lamellae are parallel to *b*. Microcline seams (Fig. 2) are in the optically featureless, medium grey areas. Black lines are fractures.

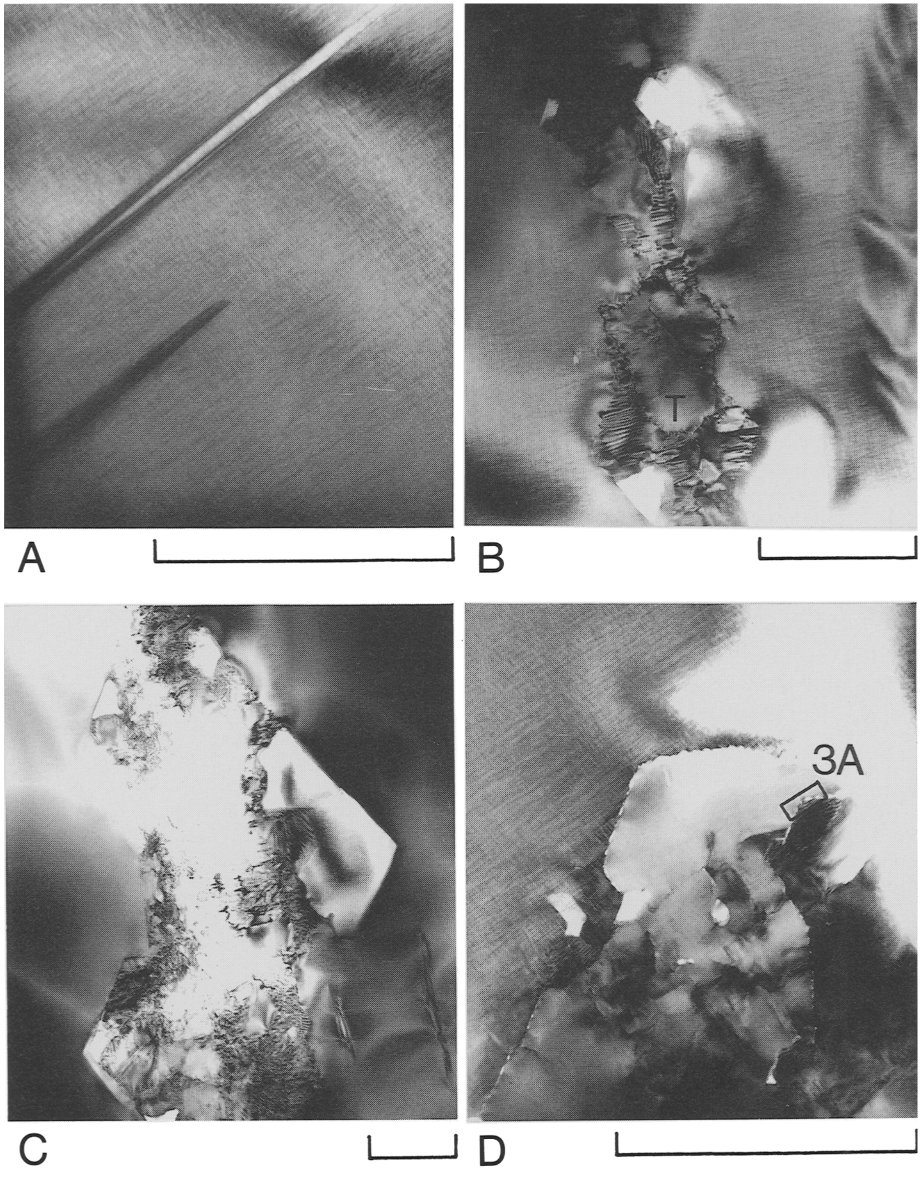

FIG. 2 Bright-field electron micrographs, beam approximately parallel to *c*. All scale bars are 500 nm. (A) Elongate lenses of albite parallel to *b*, in tweed orthoclase. (110) lattice fringes are visible on the original micrograph, and indicate perfect coherency. Note the absence of strain shadows in the region of the wedgeshaped end of the lower lamella. (B) Mostly Albite-twinned microcline seam in tweed-textured orthoclase. The microcline/tweed interface, subgrain boundaries within the seam, and the edges of micropores are delimited by {110}. An area of tweed orthoclase (T) also occurs within the seam. (C) Cryptoperthitic tweed orthoclase containing a microcline seam. Two coherent Ab-rich lenses parallel to *b* (bottom right) are set in orthoclase, and the microcline seam is parallel to the lenses. Portions of the microcline/orthoclase interface are made up of {110} planes. Subgrain boundaries (some parallel to {110}), Albite and Pericline twins, and micropores are visible within the seam as bright areas at the lefthand edge. (D) Edge of portion of microcline seam. The microcline/tweed interface (running from lower left to top right) has steps with the {110} form. The interface (running from lower left to top right) has steps with the {110} form. The interface and subgrain boundaries within the seam (bottom right) are marked by somewhat periodically spaced beam-damage holes. Boxed area is enlarged in Fig. 3A.

Of particular interest are areas within the Or-rich cryptoperthite where the predominant regular tweed microtexture is but by 'seams' of microcline. Microcline seams are 0.1–1.5 μm wide and extend for several μm in a direction parallel to *b* (and thus to the cryptoperthite lamellae). Within the seams, microcline occurs as irregular polygonal subgrains. Adjacent subgrains have slightly different crystallographic orientation (Figs. 2B,C,D) and/or are defined by curving dislocations forming subgrain walls. Some subgrains are featureless, but most are twinned and are similar to the 'irregular microcline' described by Bambauer *et al.* (1989). Most twins are related by the Albite law, but have irregular, slightly wavy composition planes. Pericline twins are not as abundant as Albite twins. Where both are present, cross-hatched or 'tartan' microtexture results.

The edges of microcline seams (the microcline/tweed interface) are very sharply defined. Segments of the interfaces appear to have simple indices such as {110} (Figs. 2B,C,D). They appear to have been 'negative' crystal faces with the {110} Adularia habit. The 'Adularia-shaped' areas are partially to completely filled with microcline. Where microcline occupies only part of these areas, voids, or micropores (0.1–1.5 μm in diameter), occur (Figs. 1B, C, D and 2A). Micropores tend to have sharp, straight edges, often related by the {110} form, and resemble the micropores associated with turbidity and subgrain formation reported by Worden *et al.* (1990). They are often connected by a network of subgrain walls (see particularly Fig. 2D). Micropores are distinct from the smaller (0.01 μm), rounded holes that appear and enlarge during prolonged exposure to the electron beam.

Lattice fringes show that there is little misorientation of the two feldspar structures across the microcline/tweed interface (Fig. 3A). Holes caused by beam damage often form along subgrain boundaries and microcline/tweed interfaces and may mark the sites of dislocations. Away from these areas of beam damage, some lattice rows run continuously from the microcline to the orthoclase (Fig. 3A).

Discussion

The discovery of cryptoperthite (Figs. 1 and 2A) in the Or-rich phase of a typical granulite-facies mesoperthite was unexpected. The complex textures (blebs, tails and lamellae, Fig. 1) indicate at least a two-process exsolution history which does not involve a solution–redeposition stage, as follows:

(*1*) Formation and coarsening of the coarse blebby mesoperthite texture, which from its shape is likely to be incoherent or semi-coherent, leading to phase compositions on the strain-free solvus. These textures are so coarse (10^3 times coarser than the fine lamellae, Fig. 2A) that it is doubtful whether any evidence of the initial mechanism of formation is preserved. The bleb–matrix pair (oligoclase–bulk Or-rich feldspar) might represent an equilibrium pair at some low *T*. The low Or in the plagioclase and low An in alkali feldspar imply a low *T* (calculated to be ~480 °C using MTHERM3, Fuhrman and Lindsley 1988) but the shape of the ternary solvus is poorly constrained at low An contents and the thermometer cannot be used for ordered feldspars.

(*2*) The initial coarsening was followed by a second phase of exsolution which led to the fine, lamellar micro- and cryptoperthitic intergrowths. It is possible, in principle, that this occurred during a continuous cooling process in which the phase compositions left the strain-free solvus leading to a stranded diffusion profile between the coarse albite blebs. The fine lamellae could then form subsequently either by coherent nucleation or by spinodal decomposition when the stranded phase had cooled through the coherent solvus. However, in view of the extraordinarily long time-scales likely if these granulite facies rocks had cooled monotonically, and in view of the probable complexity of their thermal and deformational history, it seems more probable that the textures represent a multistage thermal history. Indeed, the presence of granulite facies rocks at the Earth's surface implies at least a two-stage cooling history. There is other mineralogical and isotopic evidence that a relatively rapid uplift phase followed Grenville metamorphism at 2.5 GPa (McLelland and Chiarenzelli, 1990; Chiarenzelli and McLelland, 1991). This being the case we suggest that the compositions of the phases in the coarse textures left the strain-free solvus in response to cooling at the relatively faster rate experienced during uplift. The hypothesis is supported by the marked compositional difference between blebs and tails/lamellae, as well as by the morphological differences. There is no tendency for the late, fine albite lamellae to be concentrated in the centres of the regions of the Or-rich feldspar phase, away from the albite blebs (Fig. 1). Such concentrations would be expected if a stranded diffusion profile had developed between the blebs during monotonic cooling, but the observed even distribution is consistent with the development of compositionally stranded phase following a change in cooling rate.

At some lower T, the compositionally stranded Or-rich phase intersected the coherent solvus leading to the fine coherent lamellar textures. From the measured bulk composition ($Ab_{15}Or_{85}$) and fig. 8b from Brown and Parsons (1989), the coherent solvus would be intersected at ~350 °C. It is not possible to deduce the mechanism of exsolution from the textures alone, but it is unlikely to have been spinodal decomposition because of the low T of the coherent spinodal (in the region of 100 °C) for the Or-rich bulk composition of the stranded phase. Whatever the exact cooling history of these deep-seated metamorphic rocks this implies a relatively long period of time in the temperature interval between coherent solvus and spinodal, during which coherent nucleation could have occurred. The absence of features in the orthoclase which could have provided sites for heterogeneous nucleation, the perfect coherency of the intergrowths and the lack of visible strain in the orthoclase (Fig. 2A) are all consistent with homogeneous coherent nucleation, close to the coherent solvus. The lack of strain is consistent with a low T of formation so that readjustments in the cell dimensions of the phases during cooling to surface T were minor. Although diffusion below 350 °C is extremely slow, long periods of annealing in a cooling granulite-facies terrain could produce such textures. Small amounts of An may have a large effect on exsolution temperatures, but An is extremely low (<1%) in these alkali feldspars.

In contrast, the association of the microcline seams with development of micropores is consistent with involvement of a fluid in the process of microcline formation (Worden *et al.*, 1990). The {110} habit of newly grown feldspar is also observed repeatedly in feldspars which have

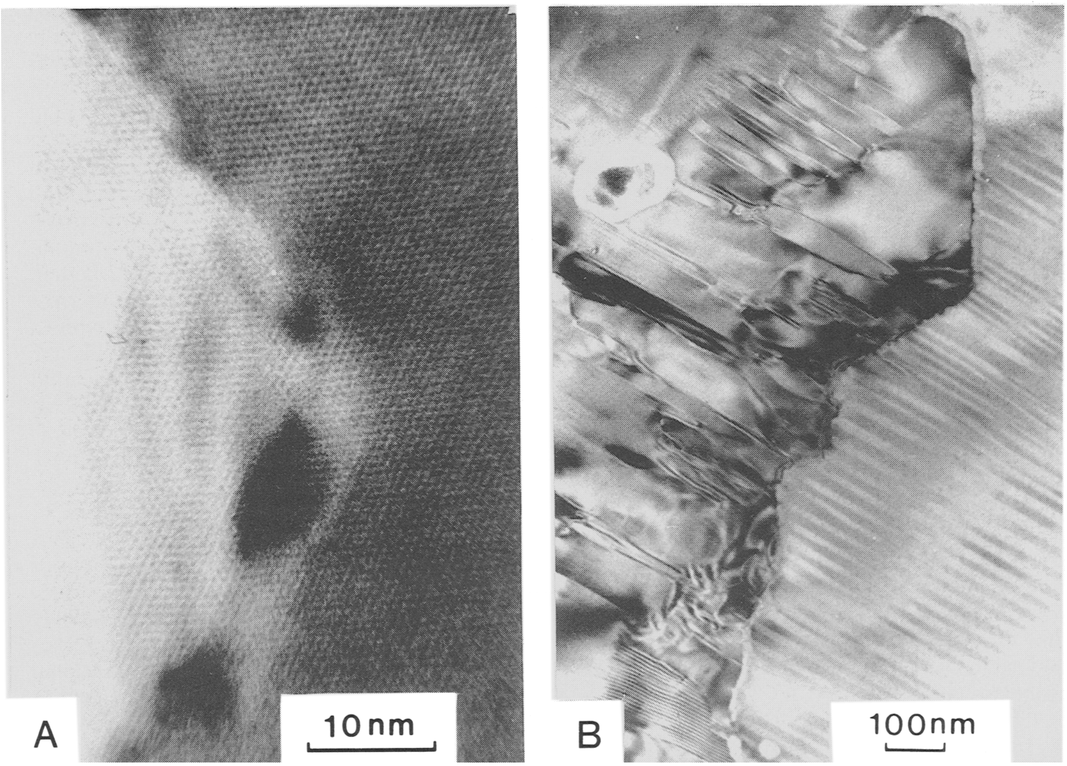

FIG. 3 (A) Subgrain boundary within seam, detail of Fig. 2D. The image above is a photographic negative compared with Fig. 2D. Dark patches are beam-damage holes. Away from the holes, some lattice fringes [(020) fringes run from left to right] are continuous across the boundary, with little misorientation between the subgrains. Note that the subgrain to the left is bounded by {110} planes. (B) An example of the development of a {110} interface in a deuterically coarsened perthite from the Klokken syenite (Worden *et al.*, 1990). Albite-twinned albite (right) shows a jagged, incoherent {110} interface against dominantly Albite-twinned microcline (left) (micrograph taken by R. H. Worden).

suffered interactions involving fluid. Worden *et al.* (1990, figs. 5 and 7c) illustrated euhedral outgrowths of Or-rich feldspar with this habit in micropores and Walker (1991) showed that pores are often so defined. Planes close to {110} are also common at the interfaces of incoherent perthites. Fig. 3B (taken by R. H. Worden) shows an albite–microcline interface in a patch perthite in the Klokken syenite. Although the overall perthite interface is roughly parallel to the average position of *b*, it is made up of sub-μm steps of {110}. The texture in Figure 3B represents a complete reconstruction of what was originally a coherent braid cryptoperthite (see Worden *et al.*, 1990, fig. 6), in the same way that the microcline seams in Figure 2 represent reconstruction of orthoclase. Subgrains in the microcline seams sometimes have the {110} outline (Fig. 2B). Adularia is the characteristic habit of feldspar growing in low-*T* veins or in sediments, and the same habit appears to develop during *in situ* solution–redeposition in alkali feldspar. In the Klokken example the maximum *T* for breakdown of the original texture was ~450 °C based on the presence of low microcline in the unaffected coherent intergrowths. The upper *T* limit for the development of the cryptoperthite in the present rocks is ~350 °C and we infer that the microcline-forming microtextures illustrated in Fig. 2 result from a solution–redeposition process associated with an aqueous fluid at some lower *T*. The minimum *T* is unknown, but it should be noted that the {110} habit is also characteristic of feldspars growing at diagenetic temperatures and that replacement of feldspar at near-surface temperatures has been shown to occur at buried unconformities (Lidiak and Ceci, 1991).

The microcline seams are semi-coherent with respect to the enclosing tweed domain texture, but in places are structurally continuous with the matrix orthoclase (Fig. 3A). Complete coherency between a large region of microcline (in a single orientation), with a pseudomonoclinic tweed structure, is geometrically unlikely but partial coherency would be possible with tweed domains of appropriate sense of order or antiorder. Short-period tweed modulations in orthoclase could be accommodated by local strain at the interface. The roughly periodic beam damage (Figs. 2D and 3A) is consistent with periodic misfit dislocations between the tweed texture and the microcline, and the continuous boundaries are consistent with an expitaxial relationship between orthoclase and new microcline. The new microcline may adopt, by epitaxy, an orientation reflecting a predominant orientation of the structure in the tweed modulations. Otherwise, it is by no means clear why Albite-twinned microcline should develop by solution–redeposition. In places, new growth appears to exhibit tweed texture (Fig. 2B).

It should be noted that in all cases illustrated (Fig. 2), the microcline seams are at the limit of optical resolution. Elsewhere, the feldspars are sometimes slightly turbid (Fig. 1) but they are (in common with other granulite-facies mesoperthites) generally pristine. The orthoclase–microcline transformation was facilitated by infiltration of fluid on a remarkably fine scale at a *T* well below the upper stability of low microcline (~420 °C, fig. 8*a* in Brown and Parsons, 1989). It is not clear whether any pre-existing structural features, such as cracks or dislocations produced by external stress, guided the infiltration.

General implications

Previous TEM work on perthites (Worden *et al.*, 1990; Guthrie and Veblen, 1991), and the present study, illustrate the role of solution–redeposition in modifying feldspar microtextures at relatively low temperatures. The rather chaotic microtextures seen in the microcline seams (Fig. 2) are common in Or-rich feldspars from plutonic rocks (Fitz Gerald and McLaren, 1982; Zeitler and Fitz Gerald, 1986; White and Barnett, 1990; and our own work in progress) and this suggests that pervasive recrystallisation of alkali feldspars commonly occurs without significantly affecting the crystal outlines. The microtextures in feldspars provide a wealth of information on possible reactions with fluids, and these have important geochemical implications, particularly for $^{18}O/^{16}O$ exchange. The present study shows that solution–redeposition, without change in crystal outlines, is an important process at temperatures at least as low as 350–400 °C, and the study of Lidiak and Ceci (1991) shows that this can continue to near-surface temperatures.

Giletti (1985) reviewed the processes which might lead to changes in $\delta^{18}O$ in minerals. The free-energy changes associated with oxygen exchange are minuscule and cannot drive the exchange reaction. Giletti recognised that, as well as occurring by volume self diffusion, isotopic exchange between fluid and crystals could be achieved by solution–redeposition, in response, for example, to non-hydrostatic stresses, or accompanying a chemical reation, where fluid and solid are out of chemical (as opposed to isotopic) equilibrium.

In feldspars, several types of driving force for solution and redeposition are possible. Worden *et al.* (1990) proposed that the driving force for dissolution during perthite coarsening was a decrease in total free energy by the release of elastic strain energy in coherent cryptoperthites, coupled with decrease of perthite interfacial energy on coarsening. Compositional change was not a driving force because phase compositions were already close to end-member. In the orthoclase–microcline transformation there is no significant compositional change, but there is a decrease in free energy because of release of strain energy in the tweed domains and because of a small increase in Si,Al order in the newly grown microcline (Brown and Parsons, 1989, fig. 4). The low-T reactivity of many feldspars in the presence of aqueous fluids can thus be ascribed to the presence of stored energy in the microtextures resulting from processes unable to go to completion at higher temperatures. Brown and Parsons (1993) estimate that the stored elastic strain energy amounts to 2–3 $kJmol^{-1}$ in orthoclase and ~2.5–4 $kJmol^{-1}$ in cryptoperthites. Turbid feldspar, which may make up a minor (as in the present example, Fig. 1) or major (as in the Klokken or Skye examples) part of individual crystals, remains reactive because it is characterised by a permeable network of subgrain walls (Walker, 1990), which render the crystals highly sensitive to further interactions with fluids.

We conclude that oxygen isotopic exchange in alkali feldspars at temperatures below 500 °C is likely to be readily accomplished by dissolution–redeposition and that this process certainly occurs at T <350–400 °C and may continue to near-surface temperatures. In our present example, dissolution–redeposition occurred on a suboptical scale. Oxygen exchange by volume diffusion may occur at near-solidus temperatures. It remains to be demonstrated that exchange can occur at intermediate temperatures without destruction of coherent exsolution and domain microtextures.

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Addendum: When the present paper was in proof, Evangelakakis *et al.* reported cryptoperthite in granulites from Sri Lanka.

Evangelakakis, C., Kroll, H., Voll, G., Wenk, H-R., Meisheng, H. and Köpeke, J. (1993) Low-temperature coherent exsolutions in alkali feldspars from high-grade metamorphic rocks of Sri Lanka. *Contrib. Mineral. Petrol.*, **114**, 519–32.