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Microtextural controls on the alteration of microcline perthites from the Spruce Pine District, North Carolina

Meyer Sheets, Julia Catherine, Ph.D.
The Ohio State University, 1994
MICROTEXTURAL CONTROLS ON THE ALTERATION
OF MICROCLINE PERTHITES FROM THE SPRUCE PINE DISTRICT,
NORTH CAROLINA

DISSERTATION

Presented in Partial Fulfillment of the Requirements for
the Degree Doctor of Philosophy in the Graduate
School of The Ohio State University

By

Julia Meyer Sheets, B.S., M.S.

*****

The Ohio State University
1994

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Approved by
Rodney T. Tettenhorst
Advisor
Department of Geological Sciences
To Rod, John, Ben, Mom and Dad
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I wish to express gratitude to Dr. Rodney T. Tettenhorst who enthusiastically provided guidance and support throughout all phases of my research.
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clinopyroxene and the role of deformation in the formation of pyroxene—Fe-Ti

of perthites from the Spruce Pine District, North Carolina, USA (abs.) EOS, 74.

Spruce Pine, North Carolina (abs.) Clay Minerals Society 30th Annual
Meeting, San Diego.

FIELDS OF STUDY

Major Field: Geological Sciences

Studies in mineralogy and crystallography (R.T. Tettenhorst and J.W. Downs),
and electron microscopy (W.T. Clark)
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CHAPTER I

INTRODUCTION

Overview and Significance

A dissolution/reprecipitation mechanism for the chemical weathering of feldspars is well established, and the transformation of feldspars to clays is thought to be nontopotactic (Eggleton, 1986). However, the influence of parent-mineral microtexture on dissolution and reprecipitation processes during the alteration of feldspar to clay is not well understood.

Defects in parent feldspars are believed to be important in the initial stages of alteration of feldspars to clays, but not at more advanced stages of the transformation (Eggleton and Buseck, 1980; Banfield and Eggleton, 1990). Parham (1969) discusses "flame structures" in artificially weathered K-feldspar with rolled morphology that he interprets as halloysite nucleating on reactive sites, such as dislocations in the feldspar crystal structure. Berner and Holdren (1977) note that experimentally etched feldspar surfaces are pitted in oriented channels, indicating crystallographic control on dissolution. These authors assume that etching occurs where dislocations intersect the surfaces of feldspar grains. Eggleton and Buseck (1980) describe holes in naturally altered feldspar bounded by (100), (101), (010) and (001) planes, and note the association of alteration material with dislocations.
Because processes of feldspar alteration such as dissolution, recrystallization, and formation of secondary minerals by magmatic (deuteric), hydrothermal, and meteoric fluids cannot be observed directly in nature, experimental evidence is extrapolated to natural situations. One example of this is the experimental determination of dissolution rates. When dissolution rates are measured for the same mineral in both laboratory and field settings, the field rate is typically slower by approximately two orders of magnitude (Swoboda-Colberg and Drever, 1993). A proposed explanation for differences between field and experimentally-derived rates is that the reacting surface area of the parent mineral undergoing dissolution is not well known and may be overestimated in the laboratory (Rowe and Brantley, 1993).

Experimental studies designed to determine feldspar dissolution rates indicate that feldspar dissolves congruently and that the reaction is surface controlled, as opposed to volume-diffusion controlled (Berner, 1981; Sverdrup, 1990). Dissolution-rate constants are determined from experimental runs on different size fractions of crushed feldspar grains. Crushed grains are used as starting material for dissolution experiments and do not necessarily reflect the physical conditions that exist during natural dissolution (Casey et al., 1993). Crushing in the experimental setting will induce breakage along cleavage directions, but natural specimens will not likely sustain the same mechanical changes prior to dissolution. If chemical reactions producing clay minerals from parent feldspar are surface-controlled, then the important surfaces for dissolution of natural specimens need to be determined to better understand the activated surface layer of a reacting feldspar grain in nature.

Experimentally determined mineral dissolution rates are used in soil studies to model cation release rates during weathering. Such models are important for judging the effect of weathering on the chemical properties of soils. Modeling the yield of base cations such as K, Na, Ca, Mg that are released during weathering helps determine natural
resistances to soil acidification (Sverdrup, 1990).

The influence of feldspar microtexture on argon diffusion kinetics has been studied by Zeitler and Fitz Gerald (1986), Burgess et al. (1992), Fitz Gerald (1993), Parsons (1993), and Fitz Gerald and Harrison (1993). Argon (40/39) incremental heating experiments were performed to try to extract complex cooling histories sustained by natural feldspar crystals (Lovera et al., 1989). These studies reveal that activation energies change with temperature; the latter are interpreted as arising from different microtextural domains in the feldspars, each of which may have a unique argon retentivity (Harrison et al., 1991). Incoherent or partially coherent grain boundaries between albite and microcline, which are regions of higher defect density in perthites, are potential sites for argon leakage into or out of feldspar (Burgess et al., 1992). Twin boundaries also have been proposed as sites for fast diffusion into and out of argon (Zeitler and Fitz Gerald, 1986). Determination of the preferential sites for feldspar alteration (the present study) may help to identify potential fast diffusion pathways for argon gas. Regions in feldspar microtextures that are susceptible to alteration should define the passageways for aqueous fluids in feldspars, and it is expected that these also are passageways for the transfer of argon gas to and from within the crystals (Fitz Gerald, 1993).

Objective

The objective of the present study is to evaluate the microtextural controls on the alteration of microcline perthites from a naturally weathered pegmatite. The texture of microcline perthites from granites and pegmatites varies considerably within a single crystal, and certain textural domains may be more susceptible to alteration. For instance, in such perthites, the microcline host, which is volumetrically the most abundant, contains twinning domains and albite exsolution and replacement lamellae (Smith, 1974). The character of these features may change on the scale of a μm or less, as revealed by
the present study and previous transmission electron microscopy studies (Akizuki, 1972; FitzGerald and McLaren, 1982). The present study attempts to determine the relative importance of intraphase boundaries (twin planes), phase boundaries (between host and albite lamellae), and other defects such as dislocations, as potential reaction sites for alteration of feldspar to clay.

Geologic Setting

Regional geology

The sample area lies within the Spruce Pine district, located in northwestern North Carolina in the Blue Ridge geologic province (Figure 1). The Spruce Pine District is located west of the axis of a southwestwardly plunging synclinorium (Kulp and Poldervaart, 1956). The area is mountainous and dissected by V-shaped drainage valleys (Brobst, 1962).

Granitic pegmatites such as those sampled intrude Precambrian amphibolite schists and gneisses (Brobst, 1962), and the pegmatites generally are concordant with metamorphic host rock structures (Hunter and Hash, 1949). Based on isotope studies and field relations, regional metamorphism that produced the country rocks is thought to have proceeded as two separate phases of plastic deformation (Kulp and Poldervaart, 1956). Metamorphism of Precambrian sedimentary sequences including an early isoclinal folding event occurred in the Precambrian (Butler, 1972). A subsequent late Paleozoic deformation event is recognized as large-scale gentle folding (Butler, 1972). Regional thrusting of the entire metamorphic sequence is thought to post-date the plastic deformation (Butler, 1972). A famous manifestation of the thrusting is located northeast of Spruce Pine (Figure 1), where the Grandfather Mountain window exposes upper Precambrian sedimentary rocks overlain by lower Precambrian metasediments.
Figure 1. Regional geologic map of a portion of North Carolina that includes the Spruce Pine district. A manifestation of thrust faulting on a regional scale is the Grandfather Mountain window (GMW), located northeast of Spruce Pine (after Butler, 1972).
Isotopic dating studies have been performed on minerals from Spruce Pine pegmatites and also on amphibolite country rocks, to date the pegmatites and to decipher the relative timing for the intrusion of pegmatites and regional metamorphism. Early U-Pb studies on minerals from the pegmatites yielded dates of 340 +/- 20 Ma (Kulp and Poldervaart, 1956). K-Ar studies of perthites from the pegmatites yield dates ranging from 300-350 Ma (Long and Kulp, 1956). These results are in accord, but both studies give a large spread in ages. A more recent Rb/Sr isotope study was undertaken on amphibolite country rocks collected from a portion of the Spruce Pine thrust sheet (Goldberg et al., 1992). Analyses of titanite, plagioclase, hornblende, and whole-rock analyses yield an isochron age of 395 +/- 6 Ma (Goldberg et al., 1992). This result agrees with field and isotope studies conducted by Butler (1973), who suggested that intrusion of pegmatites occurred during regional metamorphism 390 Ma ago. Goldberg et al. (1992) also conclude that intrusion of the Spruce Pine pegmatites was approximately contemporaneous with regional metamorphism, and occurred prior to thrusting. These ages indicate that intrusion of pegmatites in the Spruce Pine district occurred during the Acadian orogeny (Butler, 1973).

Periods of uplift exposed granitic pegmatite that was incised by the North and South Toe Rivers (Figure 2). This produced economically recoverable residual kaolin deposits averaging 40 ft in thickness from the top of the parent rock but locally exceeding 100 ft (Parker, 1949). In most kaolin mining districts, within the Spruce Pine area, stream terraces are present that contain old erosional surfaces capped by stratified sand and gravel stream deposits, and three or more terrace levels have been described that mark cycles of uplift and subsequent downcutting by streams (Parker, 1949). Exposures of entire sections that include unaltered parent rock at depth, followed sequentially by altered parent rock, an erosional surface, and stream sediments at the surface, indicate that the clay deposits formed by supergene alteration (Parker, 1949). Paleogeographic
Figure 2. Base map of North Carolina with drainage indicated. The sampling location (indicated by the "X") lies within the Spruce Pine district which includes portions of Mitchell, Yancy, and Avery (enlarged) counties. The area is incised by the North and South Toe rivers.
reconstructions place North Carolina near the equator after the formation of the pegmatites, indicating a humid tropical environment for the weathering reactions (Dietz and Holdren, 1970).

Geology of the sample collection site

Samples were collected from the Gusher Knob/Brushy Creek district, located in Avery County, approximately 5 miles northeast of Spruce Pine (Figure 3). The district was mined for feldspar and residual clays (Parker, 1949). The district is now owned by the Harris Clay Company but was first excavated in 1937 for “high-aluminum kaolin” by Kaolin, Incorporated (Parker, 1946). Gusher Knob, north of the sample collection site, contained commercial deposits of halloysite that were mined by the Harris Clay Company beginning in the early 1940’s (Hunter and Hash, 1949). Halloysite was used in the ceramic industry and was observed to impart a “glossy whiteness to porcelain” (Hunter and Hash, 1949).

The clay deposits in the Gusher Knob region are reported to result from the weathering of alaskite, a granitic pegmatite composed chiefly of oligoclase, microcline perthite, quartz and muscovite (Hunter and Hash, 1949). The term “alaskite” was first used by Spurr (1900) to describe granites containing a very low content of ferromagnesian minerals. Because pegmatites in the Spruce Pine district also contain a low volume of ferromagnesian minerals, the term was adopted for them (Hunter, 1940).

In descriptions of the weathered material from Gusher Knob and other halloysite deposits in northwestern North Carolina, Hunter and Hash (1949) report that the residual kaolin was produced mainly from the degradation of oligoclase. “Hard microcline” is a common mineralogical constituent contained in the clay matrix, and is present both at the surface and at depth, whereas plagioclase only persists at depth in the deposits (Hunter and Hash, 1949).
Figure 3. Large-scale map showing details of the sampling locations. Microcline perthites were collected from the area indicated by the arrow labelled "1." Extensively-altered pegmatite that did not contain unaltered feldspar was collected from the area indicated by the arrow labelled "2."
Powder X-ray diffraction (XRD) and chemical analyses of the clays from several mines in the Spruce Pine district reveal that they are composed of a mixture of both halloysite and kaolinite (Hunter and Hash, 1949). Percentages of halloysite and kaolinite in the mixtures are listed in Hunter and Hash (1949), but should be accepted with caution because distinguishing between kaolinite and halloysite by these methods is difficult, owing to their similar XRD patterns. These workers also describe properties of halloysite that allow it to be recognized in the field. These include "an unusually white color, its ability to roll when sliced with a knife, and strong adherence to the tongue", and in an attempt to explain the association of both halloysite and kaolinite in the clay, they suggest that the formation of halloysite may result from the weathering of plagioclase, and that kaolinite may form from microcline.

**Specimen Collection**

Specimens of microcline perthite were collected from a clay pit composed of altered pegmatite that is located south of Gusher Knob (Figure 3). Perthite crystals were selected by visibly estimating the intensity of their alteration to determine the role of parent mineral microtexture as alteration progresses. Relatively unaltered crystals have a vitreous luster and well-developed (001) and (010) cleavages. Perthites altered to a greater extent are chalky in appearance and the most altered specimens are friable and contain fracture or parting surfaces coated with clay. Fracture or parting controls the morphology of the most altered hand specimens; (001) and (010) cleavage planes are difficult to recognize in these specimens.

Precise characterization of the unaltered rock from which microclines sampled for the present study were derived is impossible, because a complete section, including unaltered parent, is not exposed in the sampling area. For this reason, unaltered pegmatite specimens were collected a few miles southeast of Gusher Knob. Previous studies
describing "alaskite granite and associated pegmatites" suggest that all of the silicic rocks from the Spruce Pine district are genetically related (Parker, 1949). The pegmatites are chemically and mineralogically similar, with the exception of variation in plagioclase composition from albite to oligoclase (Maurice, 1940). In fact, some workers opted to discontinue the term "alaskite" granite, preferring to use "fine-grained pegmatite", differentiating the alaskite from other pegmatites using grain size and mineralogical adjectives (Parker, 1946). Moreover, field observations of pegmatites in the vicinity of Gusher Knob indicate a gradual change from coarse grained pegmatite at the centers of the intrusives to "walls of finer grained alaskite" at the margins (Parker, 1949).

An extensively altered pegmatite was also sampled and is located a few thousand feet from the altered pegmatite from which the microcline perthite crystals were sampled. These predominantly clay samples were selected because they contain a texture indicative of relict twin planes from a parent plagioclase. The altered specimens were sealed in plastic bags to prevent contamination and excessive dehydration.

**Samples Selected for Detailed Study**

Samples collected from the Gusher Knob/Brushy Creek district include (1) microcline perthites with variable alteration, (2) clay matrix that is intensively altered, and (3) unaltered pegmatite that contains microcline perthite and plagioclase. Because this study is concerned with the influences of parent microtexture on the alteration of feldspar to clays, the author opted to examine three microcline perthites from the altered pegmatite, even though the crystals are not in place and do not represent a complete sequence from unaltered parent to saprolite. The rationale for this is two-fold: 1) all three perthites contain unaltered parent and this allows direct observation of the spatial relationships between parent microtexture and alteration to clays; and 2) neither the unaltered pegmatite samples nor the intensely altered clay-matrix samples allow direct
observation of the transformation of feldspar to clays. The unaltered pegmatite contains no appreciable clays. The intensively altered clay matrix from pegmatite was examined with a light microscope in an attempt to search for grains of parent feldspar but none were found.

The microcline perthite crystals selected for detailed study are named PW1, PW2, and PW3, which stands for “perthites weathered” to varying degrees. Sample PW1 contains a cream-colored, apparently unaltered K-feldspar crystal with well-developed (001) and (010) cleavages. Alteration to clay is detectable in sample PW2 which is the same color as PW1, but slightly chalky, with well-developed (001) and (010) cleavages. PW3 is the most highly altered of the study samples. The unaltered portions of crystal PW3 are also cream colored, but the specimen is whiter overall because of the presence of clay. PW3 is very chalky and the (001) and (010) cleavages are obscured by a fracture or parting plane coated with clay. All three microcline crystals are approximately 5-10 cm in dimension and contain millimeter-scale albite lamellae that are visible to the unaided eye. Major unaltered minerals present at the sampling site include microcline, muscovite, and quartz.

The term “alteration” as related to the microcline perthites in this study requires clarification. Pegmatites are water-rich and it was beyond the scope of this study to determine whether alteration to clays was produced by magmatic fluids. Moreover, (Goldberg et al., 1992) suggest that pegmatite intrusion was contemporaneous with regional metamorphism and, therefore, hydrothermal alteration cannot be ruled out. Nonetheless, it is likely that if microtexture imparts a control on the transformation of feldspar to clays, then the same reactive sites in the parent feldspar should be important, regardless of whether the alteration was by hydrothermal means or by surface weathering.
A general discussion of microcline twinning is deemed necessary here because twin textures are intimately related to all aspects of the present study. TEM studies of microcline twin textures have revealed that they are inhomogeneous on the micrometer scale (Fitz Gerald and McLaren, 1982; Tibballs and Olsen, 1977; Akizuki, 1972). The textures must reflect the post crystallization history of the feldspar and may influence subsequent alteration to clays.

Potassium-rich alkali feldspar crystallized at high temperature (sanidine) is disordered with respect to the distribution of aluminum and silicon atoms on the tetrahedral (T) sites in the atomic structure. The sanidine structure contains two nonequivalent tetrahedral sites (T1 and T2) in the asymmetric unit, and on each of these 1 Al per 3 Si from the formula unit KAlSi$_3$O$_8$ is disordered, i.e., randomly distributed (Taylor, 1933). This leads to an average composition for each T-site of 0.25 Al and 0.75 Si for fully disordered sanidine structure.

Ordering of Al on the T1 tetrahedral site occurs upon slow cooling to a temperature of approximately 480 °C (Nord, 1992). The ordering causes a symmetry reduction from C2/m to C1̅ that results in the loss of the (010) mirror plane and the [010] 2-fold axis. The end-member mineral names for potassium feldspars passing through progressively more ordered states are orthoclase and microcline. These names are used in this study because of their widespread use in the literature, however, a complete continuum
of ordered states is possible.

The microtextural manifestation of the ordering and reduction in symmetry is the development of twinning in four orientations which results in the cross-hatched twin pattern familiar to all who have examined microcline under the petrographic microscope. The four orientations are a pair each of symmetry-related twin lamellae for the albite and pericline twin laws. Albite twin pairs are related by reflection across (010), with (010) as the composition plane. Pericline twin pairs are related by an [010] two-fold rotation, and the composition plane is the irrational rhombic section (Smith, 1974).

The structural "change" required for the formation of an albite twin is a minor change in bond angle (Taylor et al., 1934), but if the material is ordered and the twinning occurred in the solid state, T-sites undergo Al and Si exchange which requires the breaking of T-O bonds (Smith, 1974).

The feldspar structure has minimal mismatch at the pericline twin boundary which is the rhombic section. The orientation of this plane changes with composition and temperature. The compositional control on the orientation of the rhombic section is significant and may be understood by considering the differences between this plane in anorthoclase and microcline. Figure 4 is a stereographic projection which shows that the rhombic sections for sodium-rich and potassium-rich alkali feldspars are almost at right angles to one another (Nord, 1992). The theoretical orientation of the rhombic section can be calculated from the unit cell parameters of the specimen of interest using the equation: \( \cot(s) = \cot(\alpha^*)/\cos(\gamma) \) (1), where \( s \) = the angle between the rhombic section and (001) (Smith, 1974). The orientation of the rhombic section can also be measured in (010) sections by observing the angle between the trace of the (001) cleavage and the trace of the pericline-twin composition planes. This angle is known as \( \sigma \), and can also be calculated with cell parameter data from the relation: \( \tan(\sigma) = \cos((\gamma)\sin(\beta))/[(\cos(\alpha)\cos(\beta))-\cos(\alpha)] \) (2) (Smith, 1974). For microcline, the average value of \( \sigma \) was
Figure 4. Stereographic projection showing the orientations of the rhombic sections (pericline-twin composition planes) for both Na-rich and K-rich alkali feldspars. The diagram includes the position of the [102] zone axis, which is the direction of projection for TEM images obtained during the present study. (After Nord, 1992).
calculated first by MacKenzie (1956) as 83-84 degrees. As noted by Smith (1974),
atoms in the T sites are misaligned across the twin boundary defined by the rhombic
section but no major changes in the feldspar structure are required for pericline twinning.
Two important points in terms of changes in the feldspar structure with cooling related to
the pericline twin orientation were noted by Smith (1974): 1) Remnant pericline twin
boundaries may persist in the twinning microtexture and conversion to a new pericline
orientation in accordance with the changes in structure with cooling only occurs with
major recrystallization; 2) If a pericline composition plane does not exactly coincide
with the rhombic section for a particular crystal, dislocations must be present to
accommodate the resulting structural mismatch.

Laves (1950) first proposed that single crystal XRD patterns containing two pairs
each of albite and pericline twin spots oriented at right angles to one another indicate that
microcline developed from an initially monoclinic feldspar. This is because \( \mathbf{b} \) and \( \mathbf{b}^* \)
must be parallel to produce this spot geometry, which is only possible in monoclinic
crystals. The triclinic phase subsequently inherits this geometry and the twin texture
produced is known as M-twinning (Laves, 1950). The diffraction spots observed by
Laves (1950) have a characteristic “Z-shaped” morphology, reflecting the four twin ori­
tentations.

Microcline twins are termed “transformation-induced,” because they occur with
changes in the crystal structure as the specimen cools (Nord, 1992). The twinning sub­
class is pseudo-merohedral, which describes a near-perfect structural match across the
twin composition plane (Smith, 1974). The mismatch of twin individuals increases
away from the composition plane and results in splitting of diffraction spots in a direc­
tion perpendicular to the twin boundary. This is shown diagrammatically by Nord
(1992), who illustrates the concept with theoretical electron diffraction patterns (Figure 5). As one follows the pattern of spots from low to high order (hkl) maxima, the distance between spots increases, representing the increasing structural divergence between twin individuals away from the twin composition plane (Figure 5).

Akizuki (1972) first used TEM to examine twinning textures in microclines from Spruce Pine, North Carolina, Amelia, Virginia, and two additional specimens from Japan. He noted that, although cross-hatched twinning is evident under the light microscope, pericline twin spots are not produced in electron diffraction patterns for all types of twin domains (Akizuki, 1972).

Akizuki showed two twin textures similar to those observed in the present study. One was imaged from the Spruce Pine specimen and it contains albite-twinned and untwinned lamellae. Diffraction patterns recorded from this texture did not produce pericline twin spots (Akizuki, 1972). The second twin texture, from the Amelia specimen, contained discrete pericline and albite-twinned regions that produced the characteristic "Z" spot geometry described by Laves (1950).

Fitz Gerald and McLaren (1982) described twinning microtextures in several microclines from granitic rocks and pegmatites. These authors examined differences in electron diffraction patterns taken from various twins and noted that evidence for pericline twinning is not present in diffraction patterns unless the texture includes discrete lenses of pericline twins contained in a matrix of albite twins or vice versa. Furthermore, in some regions albite twin lamellae cross over pericline twin lamellae, suggesting that the latter are converting to the former. Fitz Gerald and McLaren (1982) suggest that this is evidence that the stability of albite twinning increases relative to pericline twinning with cooling and ordering in the structure of microcline. Tibballs and Olsen (1977) also propose that pericline twin boundaries are inherently high energy planes relative to albite twin boundaries.
Figure 5. Model diagrams of theoretical [001] electron diffraction patterns of twinned microcline. The left diagram shows a twin spot pattern produced by albite-twinning, the right diagram shows the spot pattern for pericline-twinning, and the center diagram shows the spot pattern for the combination of these orientations, known as "M-twinning." (After Nord, 1992).
An analogous situation for the stability of pericline twins is discussed by Smith et al. (1987) for twinning in anorthoclase. The formation of pericline and albite twinning in anorthoclase is due to a displacive mechanism which is rapid and reversible, in contrast to the diffusive mechanism required for the migration of Al and Si and the breaking of T-O bonds as required for twinning due to ordering in microcline. Smith et al. (1987) observed the stability of albite twinning relative to pericline twinning directly in TEM with a specimen-heating stage. As predicted, albite twin lamellae were unstable and disappeared at elevated temperature (650°C), leaving only pericline lamellae visible in the micrographs. Likewise, albite twin spots vanished in corresponding electron diffraction patterns. Albite-twin composition planes and twin spots returned after cooling, and occasionally the boundaries between twinned and untwinned regions migrated to different locations than those of the original micrograph (Smith and McLaren, 1987).

The previous discussion suggests that cross-hatched twinning in microcline always occurs as the result of ordering of an initially high-temperature monoclinic structure. Cross-hatched twinning in microcline may also occur in feldspar specimens that initially crystallized with triclinic symmetry (Nord, 1992). One proponent for this case (Marmo, 1955b) studied microcline from a pegmatite from granitic rocks of Sierra Leone that did not contain remnants of orthoclase and was mostly untwinned. This author suggests that the twinning texture of these microclines results from replacement of albite by exchange of K for Na, and that stress was important in the formation of patches of cross-hatched twinning present in the specimens (Marmo, 1955b). Smith (1962) discusses the possibility that conversion of untwinned albite to form microcline could result in the latter showing no twinning; if the albite was twinned prior to replacement then the resulting microcline would show albite twinning.

A different diffraction-spot geometry arises when microcline initially crystallizes as a triclinic phase. The pericline \( b \) twin axis will not be parallel to the \( b^* \) albite twin
axis; the result is a three-spot configuration known as T-twinning (Nord, 1992).

Diffraction spots from twinned microcline also have been observed that represent twin individuals oriented midway between the "standard" orientations of pericline and albite twins (Smith and McLaren, 1983). This twin orientation is termed the diagonal association. Recall that the pericline twin operation is a 180 degree rotation about the b crystallographic axis, and the albite twin operation is a reflection about (010). The latter has also been described as a 180 degree rotation about b* (Smith and Brown, 1988). The author does not understand how a reflection across the (010) plane (an improper operation) can be described as a 180 degree rotation about the axis normal to the plane (b*).

The twin operation for diagonally associated individuals has been described as a rotation of 180 degrees about an axis midway between the b and b* axes (Smith and McLaren, 1983). The geometry of the diagonal association is understood through the use of stereographic projection, as illustrated by Smith and McKenzie (1959). These authors show the relative positions of the crystallographic axes for albite, pericline, and diagonally twinned crystal (Figure 6). In this projection, the crystallographic axes of diagonally associated microcline twins lie halfway between those of albite and pericline-twinned individuals.

The preceding discussion presents the types of twinning observed in microcline and the diffraction spot geometries arising from them. These data are used as a guide for the interpretation of twin textures and associated diffraction patterns observed in the Spruce Pine specimens of the present study. The observations of several workers suggest that albite twins are more stable than pericline twins (Smith et al., 1987; Fitz Gerald and McLaren, 1982; Tibballs and Olsen, 1977, Akizuki, 1972). If pericline twin composition planes are higher energy boundaries, then domains of pericline twinning may be more susceptible to alteration than domains of albite twinning.
Figure 6. Stereographic projection showing the relative positions of crystallographic axes for crystals twinned according to the albite and pericline orientations, plus an orientation midway between known as the diagonal association. The crystallographic axes are designated with the following subscripts: "A" for albite-twinned individuals, "P" for pericline-twinned individuals, and "D" for diagonally-associated individuals. (After Smith and MacKenzie, 1959).
CHAPTER III

EXPERIMENTAL TECHNIQUES

Specimen Preparation

Thin sections

Large microcline perthite crystals, approximately five to 10 cm in length, were collected from a weathered pegmatite near Gusher Knob, Spruce Pine district, North Carolina. Doubly-polished petrographic thin sections of the microcline perthite were made for examination by light-optical, electron probe microanalysis (EPMA), and TEM methods. Specimens are labeled as follows: unaltered perthite, judged by eye, is labelled PW1: intermediately altered perthite is denoted PW2: PW3 specimens represent the most altered perthite. All but two of the thin sections examined were prepared by Quality Thin Sections of Tucson, Arizona. The first perthite sections were prepared by the author at Ohio State University, and were made from the PW1 perthite. The methods described below are the procedures used by the author.

Well-developed (001) and (010) cleavage planes of the feldspar were used to orient the thin sections. Thin sections were made in lieu of grain mounts so that large areas could be produced that would retain textural relationships between mineral phases. Slices of specimen were cut parallel to the cleavage planes with a diamond saw to facilitate crystallographic orientation in the TEM. The cut crystals were trimmed to fit the dimensions of glass slides, and a flat surface with a 600 μm finish was worked onto one
side of each specimen. The 600 μm finish was achieved either by hand, on a rotating
diamond blade or by machine on a clean Logitech grinding lap (without grit) with
weights applied to the samples. Next, a small amount of Koldmount resin was applied
on the rough-cut side of each specimen. The resin was allowed to harden, and a small
hole was drilled into the resin at the centers of the specimens. The holes allowed mount­
ing arms of a Logitech auto-polishing device to fit securely to the specimens as they
machine-polished first to a 6 μm and then to a final 1 μm finish.

These singly-polished samples then were mounted to glass slides with the pol­
ished side down, using Buehler’s thermoplastic cement. This cement was chosen so that
TEM samples could be extracted easily from the final thin section with gentle heating.
Specimen mounting was a four-step process: 1) Frosted glass slides of known thickness
(to the nearest 5 μm) were placed on aluminum foil on a hot plate and heated to approxi­
mately 100-130 degrees C; 2) Small sticks of the Buehler cement were broken and
placed in disposable aluminum weighing pans on the hot plate and allowed to melt; 3)
A portion of the cement was transferred to the frosted side of each glass slide with a
toothpick when the cement was sufficiently liquid; the amount needed was determined
by trial and error and depended on the size of the individual sample; 4) Each specimen
was centered on a slide, removed from the hot plate, and quickly placed on aluminum
foil under a mounting jig at room temperature to even-out the interface of glue before the
adhesive hardened.

Next, the sections were parallel-cut with a diamond saw to remove excess sam­
ple, vacuum-mounted to a Logitech grinding jig set to a safe final thickness, and ground
using 600 μm tungsten carbide grit until the jig achieved a free-spin. Because the thick­
ness of the glue was unknown, the safest method was to periodically remove the jig to
check grinding progress.
After a satisfactory thickness was achieved (~50 µm), the sections were cleaned ultrasonically before mounting on the Logitech polisher for the second time. This time the sections were attached to the polisher using the vacuum mount designed for thin sections, and the second surface was polished to a 1 µm final finish using diamond polishing compound. Separate laps were used for each size of diamond compound to avoid contamination. Finished sections were observed in reflected light to ensure that a satisfactory polish was achieved.

**TEM samples**

Samples for the TEM studies were taken from the polished thin sections of oriented perthite described above. A Gatan ultrasonic disc cutter was used to drill discs 3 mm in diameter which are the correct dimension to fit into standard TEM specimen holders. The positions of the discs were selected at random. The main objective was to extract as many discs as possible from each thin section.

To estimate the drilling depth necessary to perforate the thin sections, the drill bit was allowed to rest directly on the glass slide, and then on the slide plus thin section. Micrometer readings were taken for both of these drill positions to estimate the thickness of perthite section. Slurry grit was placed on the area selected for drilling and a small amount of water was squirted on the area with a syringe mechanism attached to the bit. Next, the drill bit was gently lowered onto the area, turned on, and allowed to penetrate the specimen section and epoxy. Usually the frequency of vibration of the bit changed when the bit contacted epoxy, and this was used as a guide to stop drilling. Occasionally the drilled specimen disc dislodged from the slide and was sucked up into the hollow drill bit. When this happened, the specimen could be floated out gently into a glass dish by passing water through the bit via the syringe.
After drilling, the thin section containing drilled specimens that remained adhered to the glass slide was rinsed with distilled water. The rinsing was performed over a container to avoid loss of specimen disks. The slide was allowed to air dry and then placed on a hot plate heated to approximately 80-100 degrees C. After the Buehler cement liquefied a splintered toothpick was used to pry up and remove the drilled disks. The free disks were allowed to soak in acetone to remove excess epoxy and then were rinsed in distilled water followed by rinsing in methanol.

Copper support grids with 1mm x 2mm slot dimensions were glued to the clean specimen disks using Hillquist coverslip epoxy. Four small drops of epoxy, spaced evenly on the Cu grids, were applied using a splintered toothpick. Best results were achieved by using as little epoxy as possible. The specimen disks were centered on the Cu grids with the aid of a petrographic microscope. Cu-supported grids were allowed to air-dry overnight and were then cleaned with a cotton-tipped swab soaked with methanol to remove traces of epoxy.

Clean supported discs were placed in a Gatan dual ion mill for final thinning by argon ion bombardment. Operating conditions for the ion mill were 6 kV accelerating voltage, 14 degree angle of incidence, and gun currents of 0.5 mA per gun. After 6-8 hours, the settings were decreased to 4 kV, 0.5 mA gun currents, and a 10 degree angle of incidence to obtain a final polish. The kV was lowered in an attempt to minimize damage to the thin area; the angle of incidence was lowered to reduce the amorphous layer that develops on the surface of the specimen. The samples were allowed to thin to perforation: a hole was observed using a microscope attachment fitted to the specimen port cover of the ion mill. A petrographic microscope was used to examine the thin area. Thinned Cu-supported specimens were carbon coated using an Edwards/Penning bell jar vacuum apparatus.
Low-Resolution Transmission Electron Microscopy

A JEOL JEM 200CX transmission electron microscope, available in the Ohio State University Central Electron Optics Facility, was used for the majority of the data collection. The microscope is equipped with a double-tilt goniometer stage to facilitate specimen orientation and was operated at an accelerating voltage of 200 KV.

Selected area diffraction (SAD) patterns were obtained by placing a field-limiting aperture around regions of interest in the perthite samples. To facilitate indexing the experimental diffraction patterns, calculations of theoretical interplanar spacings and angles for microcline were carried out using a set of crystallographic programs written by the author. The programs DIFFRACT and DESKTOP MICROSCOPIST developed by H.L. Fraser were also used to calculate theoretical diffraction patterns for microcline in several zone-axis orientations. Cell parameters used in the computations were taken from Brown and Bailey (1964).

An algorithm was developed by the author early in the study to facilitate specimen orientation in the TEM. The routine calculates the angles necessary to bring a third zone axis parallel to the electron beam using tilt angles for two experimentally-observed zone axis diffraction patterns. Required input data are estimated unit cell parameters for the sample, two goniometer tilt angles for each of two experimentally-observed zones, and [uvw] for the third zone. Details of the algorithm and comparison of experimental and calculated tilts are given in Appendix B.

Qualitative chemical microanalyses were obtained using a Tracor Northern TN 2000 energy dispersive X-ray analyser (EDS) fitted to the TEM. Microanalysis was carried out in STEM mode (scanning transmission electron microscopy) to prevent specimen contamination that occurs when analyses are obtained using a focussed beam. The analyses helped to distinguish between twin domains in the K-rich host and fine-scale albite lamellae, and identified regions altered to clay minerals. Microprobe
analyses confirm that the host is nearly pure orthoclase (Or) in composition, while exsolution lamellae are nearly pure albite (Ab). Therefore, when a sodium peak was obtained from a lamellar-shaped region observed in the TEM image, it was identified as an albite exsolution lamella. The volume of sample scanned during an analysis varied with the morphology and size of a region of interest and was controlled by changing the dimensions of the scan raster. All chemical analyses were counted for at least 100 seconds.

**High Resolution Transmission Electron Microscopy (HRTEM)**

Lattice-fringe images were obtained from TEM specimens extracted from PW2, the intermediately altered perthite. The instrument used is a Hitachi H9000 high resolution transmission electron microscope, also available at the Central Electron Optics Facility. The specimen stage is equipped with a double-tilt goniometer allowing tilting to plus or minus 15 degrees, which was enough to obtain zone-axis fringe images from the (001)-oriented samples. This instrument was operated at 300 kV, with a gun bias setting of 5-8 microamperes.

**Electron Probe Microanalysis (EPMA)**

Electron probe microanalysis was performed using a Cameca SX-50 electron microprobe in the Department of Geological Sciences, The Ohio State University. The analyses were performed on a perthite specimen cut parallel to (010) that was not characterized by TEM, but is similar in hand specimen to PW1. An (010) orientation minimizes overlap between the Or-rich host phase and albite exsolution lamellae during an analysis, because the latter have an (h0l) habit plane. Analyses were carried out at a 15 kV operating potential and 20 nA beam current.
A spot size of 1 µm limited analyses of albite lamellae to those at least a few µm in width. A microcline standard from the Smithsonian Institution and an albite standard from the Taylor block were used for quantitative analyses. The Taylor block was obtained from the SEM laboratory in the Department of Geological Sciences. Feldspar compositions for the Or-rich host and albite exsolution lamellae were calculated using Cameca "GEO" software.

**X-ray Powder Diffraction**

A specimen of the clay from a weathered pegmatite, located a few thousand feet from the PW series microclines, was analyzed using a Philips powder diffractometer in the Department of Geological Sciences at The Ohio State University. The specimen was ground in a mortar and pestle and placed in distilled water for a few hours to settle out the majority of the coarse grain-size fraction. The supernatant was poured into ceramic dishes and allowed to air dry. The whole fraction of the supernatant (no attempt was made to isolate the <2 µm fraction) was analyzed using both back and side-loaded mounts. The samples were run at an operating voltage of 35 kV and 15 mA tube current.

In an attempt to obtain an X-ray analysis of the altered material in an individual microcline specimen, a microcline perthite similar in character in hand specimen to PW3 was crushed, placed in distilled water, and agitated using an ultrasonic probe. The supernatant was saved after each of several 5-minute runs with the probe, and was poured into ceramic dishes and dried in a convection oven at a temperature of 80 degrees C. The powder was removed from the ceramic dishes, front loaded in an aluminum holder, and X-rayed with a target voltage of 35 kV, 15 mA current.
CHAPTER IV

RESULTS

Light-optical Microscopy

Perthite terminology

A brief background on perthite terminology encountered in the literature is presented here to clarify light-optical descriptions of the PW-series perthites. Following standard nomenclature a “perthite” is a two-phase crystal that contains potassium feldspar as the dominant host phase and subordinate sodic plagioclase lamellae (Smith, 1974). The sodic feldspar in perthites may result from exsolution as the crystal cools, or from replacement by solid state diffusion or infilling of cracks (Smith, 1974). Perthites are classified broadly on the basis of texture as macroperthite, microperthite, or cryptoperthite. Macropertithites can be seen with the unaided eye or with a hand lens, microperthites require a light microscope, and cryptoperthites require electron optical microscopes for direct observation of lamellae (Smith and Brown, 1988).

Specimens PW1, PW2 and PW3 were examined with a petrographic microscope and were determined to be microcline perthites. They contain vein and film albite as the sodic plagioclase phase. The terms “vein” and “film” originated from an optical study of 300 perthites from granitic pegmatites (Andersen, 1928) and are useful to classify the plagioclase lamellae by size. Vein albite lamellae are large and can be detected with the unaided eye (macropertithite). They average 0.1 mm (100 μm) in width and may be
several millimeters in length (Smith, 1974). In comparison, the upper limit for the width of film albite lamellae is 0.01 mm (10 μm) for which the term microperthite has been used (Smith, 1974). Cryptoperthite lamellae are less than 0.0005 mm (0.5 μm) in thickness (Smith and Brown, 1988) and are not detected with a petrographic microscope.

**Thin section descriptions**

The scale bars on the light-optical micrographs presented below are given in μm to allow a more direct comparison with scales in TEM micrographs shown later, which are also given in μm. Thin sections for light-optical microscopy were prepared to be viewed subparallel to c*. Hence, all descriptions of photomicrographs refer to (001) sections unless otherwise specified.

A light-optical photomicrograph of a thin section prepared from perthite PW1 is shown in Plate I. Cross-hatched twinning is present in the microcline host (labelled "mic"). Untwinned regions are also present in the microcline, and are labelled "ut." Albite and pericline twin orientations in the host are indicated by lines parallel to the trace of (010) and the rhombic section (labelled "rhomb"). Vein albite lamellae are also present (labelled "vab") and are albite-twinned. These lamellae are irregular in outline and their maximum widths are of the order of 100 μm. In general, the widths of albite twins in vein albite as measured perpendicular to (010) are larger than the widths of albite twins in microcline. Moreover, the widths of albite twins in vein albite vary more than those in microcline. Some grain boundaries between vein albite and microcline are diffuse and appear black in the micrograph; this is attributed to clay mineral alteration and is labelled "alt." Alteration is also present in albite vein interiors. Film albite is not evident in this micrograph.
Plate I. Light-optical micrograph of a thin section prepared from perthite PW1. Cross-hatched twinned host microcline (labelled “mic”) and albite-twinned vein albite lamellae (labelled “vab”) are present. An untwinned area in host microcline is labelled “ut”. The trace of (010) and the rhombic section for the albite and pericline twin orientations in microcline are underscored and labelled “(010)” and “rhomb,” respectively. Alteration to clay (labelled “alt”) appears black and diffuse and occurs at grain boundaries between host microcline and vein albite.
A light-optical micrograph of a thin section prepared from specimen PW2 is shown in Plate II. Vein and film albite are both present and are labelled "vab" and "fab", respectively. The specimen is rotated to an orientation so that albite twin planes in vein albite are not in contrast and the veins appear white in the photo. PW2 microtexture resembles that of PW1, with vein albite lamellae approaching 100 μm in maximum width. Grain boundaries between cross-hatched host microcline (labelled "mic") and vein albite are irregular and sometimes contain the black alteration (labelled "alt") as described for PW1. Film albite approaches 10 μm in maximum width, and is concentrated in turbid, or cloudy regions between the large vein albite lamellae (outlined and labelled "turbid area"). The long dimensions of film albite are approximately parallel to the b-axis of microcline and the boundaries between film albite and microcline are more regular, i.e., they do not appear to change in crystallographic orientation compared to vein albite/microcline boundaries. In PW2 alteration appears black and diffuse and is concentrated within vein albite grains, at the grain-boundary margins between vein albite and host microcline, and also at microcline/film albite boundaries.

A photomicrograph of a thin section prepared from PW3 is shown in Plate III. The alteration to clay (labelled "alt") appears black in the photo and is substantially greater than that observed for PW1 and PW2. Vein albite is not clearly visible. In portions of the section where vein albite was identified, the albite twinning is obscured almost beyond recognition (not shown). Cross-hatched twinned microcline host (labelled "mic") is dissected by alteration channels (labelled "ch"). The position of the trace of (010) is not indicated in this micrograph because albite-twinneed vein albite was obscured and could not serve as a guide to determine the orientation of albite twin composition planes in the microcline.

Measurements of optic sign and the 2V angle were obtained from an (010) thin section cut from the same crystal, PW3. This section yielded an optic-axis figure with
Plate II. Light optical micrograph of a thin section prepared from perthite PW2. Cross-hatched host microcline is labelled “mic” and the (010) trace is underscored. Vein albite (labelled “vab”) and film albite (labelled “fab”) are present. The albite lamellae are in an orientation such that the twin planes show no contrast and appear white in the micrograph. A turbid region with a cloudy, pitted appearance is outlined and labelled “turbid area.” Alteration (labelled “alt”) appears black in the photo and occurs at vein albite/microcline boundaries and in the interiors of vein albite lamellae. Alteration also occurs in film albite lamellae and is associated with turbid zones.
Plate III. Light-optical micrograph of a thin section prepared from perthite PW3. Cross-hatching is still evident in microcline (labelled “mic”), but albite-twins in vein albite are obscured by alteration and the orientation of the (010) trace was not determined. Alteration (labelled “alt” appears black in the photo and is sometimes in the form of channels with sub-parallel boundaries (labelled “ch”).
negative sign and 2V angle of approximately 60 degrees. The 2V angle provides an estimate of the ordering of Al into T1 sites, and ranges from approximately 54 degrees to 88 degrees from disordered sanidine to fully ordered microcline.

The angle, $\sigma$, used to define the orientation of the rhombic section for pericline twin composition planes, was measured directly in two sections of PW3 cut parallel to (010). The $\sigma$ angles obtained were consistent with a microcline host phase in PW3. Ten angles were measured in the first section between (001) cleavage traces and the traces of pericline twin composition planes and gave an average for $\sigma$ of 84 degrees. Ten $\sigma$ angles were measured in the second (010) section, and yielded an average of 85 degrees. These measurements are in agreement with $\sigma$ values calculated for microcline using cell parameter data which ranged from 83-84 degrees (MacKenzie, 1956).

**TEM samples extracted from thin sections**

Light-optical photomicrographs were taken from ion-thinned TEM samples extracted from (001) thin sections PW1, PW2, and PW3. Some differences between the standard thin sections and ion-milled TEM samples are noted; the most notable differences are more clearly defined grain boundaries and the contrast of altered regions relative to unaltered regions is sharper in the ion-thinned samples. Small sample thicknesses minimize interference sustained by light traveling through inclined grain boundaries and make the boundaries more clearly visible. For instance, a turbid region in a thin section prepared from perthite PW2 (Plate II) that gives a cloudy or pitted appearance to the microcline was discussed above. This region contains lamellar film albite (labelled “fab”) with diffuse grain boundaries that are associated with alteration (black). Film albite/microcline grain boundaries are more clearly defined in an ion-thinned TEM sample extracted from PW2 (Plate IV).
Plate IV. Light-optical micrograph of an ion-thinned TEM specimen extracted from the PW2 thin section. Vein albite ("vab") and film albite ("fab") appear white in the photo and contain alteration to clay ("alt") which appears medium gray in the photo. Host microcline ("mic") is altered in the form of channels with subparallel boundaries ("ch").
Alteration to clay (labelled “alt”) appears medium gray in photos of ion-thinned TEM specimens (Plate IV). Identification of alteration in film albite and alteration channels in microcline (labelled “ch”) is convincing when compared to regions of altered vein albite. The vein albite in Plate IV is in an orientation such that albite twin planes are out of contrast and the veins appear white. Thus, the gray regions in vein albite represent alteration (compare with the black alteration of vein albite in Plate II). This is important when considering the alteration channels in microcline; the fact that they are gray suggests that they are infilled with clay and represent original microtexture. The specimen does appear to have fractured at the expense of altered regions in microcline during the sample preparation process, however.

Photomicrographs of TEM specimens from crystals PW1, PW2, and PW3 are displayed in Plates V, VI and VII, respectively. Plate VI is a photo of the same region described in Plate IV, but the specimen is in an optical orientation such that albite lamellae appear black. Comparison of Plates V and VI shows slight, but possibly important, differences in microtexture. PW1, which appeared unaltered in hand sample, shows alteration of vein albite at grain boundary margins adjacent to host microcline (labelled “alt” in Plate V). Furthermore, PW2 contains lamellar film albite (labelled “fab”) located between vein albite lamellae (“vab”) in host microcline; the film albite lamellae are not present, at least not in large amounts, in PW1 (Plate V). Cross-hatched twinning appears to be more well-developed near the vein albite margins in PW2 whereas the cross-hatched twinning is more homogeneous in PW1.

Similarities between the TEM samples are most marked in terms of the geometry of the alteration. PW1, PW2, and PW3 all show that alteration is concentrated at vein albite/host microcline margins. Where alteration penetrates the microcline host, it is often in the form of channels with subplanar boundaries. Examination of these boundaries shows that they occur in several orientations with respect to unaltered microcline.
Plate V. Light optical micrograph of an ion-thinned TEM specimen extracted from the PW1 thin section. Vein albite ("vab") shows faint contrast of albite twin planes and is altered at vein interiors and along grain boundary margins with host microcline ("mic"). The alteration appears medium gray in the photo. Film albite is not present in the micrograph.
Plate VI. Light-optical micrograph of the ion-thinned TEM specimen described in Plate IV. The vein albite ("vab") and film albite ("fab") are in an orientation such that they appear black in the photo, and alteration ("alt") appears medium gray. Alteration is concentrated in vein and film albite at the lamellae interiors and at the margins with host microcline. Alteration in host microcline is in the form of channels ("ch") with subparallel boundaries ("spb") that are linear or curved ("curved"). Some channels in the microcline extend from altered regions in vein albite. The sample was thinned to perforation, and the hole appears black and is marked in the Plate. It appears that the specimen fractured at the expense of alteration channels in microcline during the specimen preparation process. However, intact channels filled with gray alteration are interpreted as original texture.
Plate VII. Light optical micrograph of an ion-thinned TEM specimen extracted from thin section PW3. Cross-hatching in microcline ("mic") is still evident, but the (010) trace was not determined because twinning in vein albite was obscured by alteration ("alt"), which appears medium gray in the micrograph. Alteration occurs as irregular masses and channels with subparallel boundaries; the latter are labelled "spb." The hole formed by ion thinning appears black and is indicated in the plate.
The planar boundaries between host microcline and alteration are labelled "spb" in Plates V, VI and VII. Occasionally, the alteration channels connect one vein albite lamella to another (Plate VI). Curved contacts between unaltered microcline and alteration to clay are also present; an example of such a contact is labelled "curved" in Plate VI.

Twin extinction angles measured from the trace of (010) in sections of alkali feldspar viewed parallel to c* provide an estimate of structural state, i.e., the degree of order of Al into T1 sites of the crystal (Smith, 1974). Extinction angles which were measured in TEM sections of all three samples yield an average of approximately 15 degrees. Another region of the same TEM sample shown in Plates IV and VI, was rotated so that the trace of (010) paralleled the N-S polarizer of the petrographic microscope (Plate VIII). The same specimen, rotated approximately 15 degrees to the extinction position, is shown in Plate IX. The extreme value for the extinction angle is 18 degrees for maximum microcline; the fact that the measured value is less may represent sub-microscopic twinning giving monoclinic optical properties, or may be due to exsolved albite, or both (Smith and Brown, 1988). Optic sign was determined as negative using flash figures.

**Low-Resolution Transmission Electron Microscopy**

Samples for TEM analysis were oriented with the electron beam nearly perpendicular to the c* crystallographic axis. The magnification for low resolution micrographs presented in the following plates ranges from 5000 to 73000x but, as a rule, the scale of the microtextures was of the order of one to several tens of μm, so 30000x was an effective "average" magnification. The term "unaltered" used in this section is defined as a region in the perthite that does not contain alteration to clay. It is not intended to imply that the perthite microtextures have not undergone substantial changes to their microtextures since the time of crystallization and, as will be developed later in
Plate VIII. Light optical micrograph of another region in the ion-thinned specimen described in Plates IV and VI. The specimen is rotated to the parallel position, i.e., the trace of (010) parallels the NS polarizer of the petrographic microscope, thus determining the albite twin extinction angles in the host microcline. Cross-hatched microcline ("mic"), vein albite ("vab") and alteration ("alt") are indicated in the plate. A hole produced by the thinning process is also indicated in the photo.
Plate IX. Light optical micrograph of the same region as in Plate VIII, rotated to the albite twin extinction position. Approximately 15 degrees of rotation was required to bring one orientation of the twin individuals to extinction. This is less than the maximum value of 18 degrees for fully ordered microcline and may result from sub-microscopic twinning or exsolution that yields pseudomonoclinic properties at the scale of the light-optical microscope.
the discussion chapter, some of the features described in this section may be intimately associated with alteration processes that these minerals have undergone.

The majority of the TEM work was performed on sample PW2 which was the first sample from which TEM specimens were prepared that contained adequate thin regions containing clay alteration. Two specimens of PW1 were examined primarily to establish the microtextural features of unaltered host microcline. These features were subsequently confirmed to be present in PW2 and PW3. One TEM sample was prepared from PW3. Observations from the TEM sample of PW3 were frustrated by the fact that the sample is extremely porous and most of the electron-transparent region is thick and thus difficult to interpret.

**Microtextural features of unaltered Spruce Pine perthites**

**Microcline host**

In general the microtexture of the host microclines can be described by four styles of twinning as observed with low resolution TEM. The four styles were used by the author as a guide; no absolute boundaries exist between the domains and they were observed to grade into one another. Also they are interrupted by untwinned regions that vary in scale and magnitude.

Type I domains (Plates X-XV) consist of alternating albite-twinned and untwinned lamellae whose long dimensions are approximately parallel to [010]. Type II domains (Plates XVI-XVII) contain zones of discrete lens-shaped pericline-twinned lamellae and these are often separated by albite-twinned zones. Albite twinning is more areally extensive as compared to pericline twinning in the Type II domains. Type III domains (Plate XVIII) are essentially the inverse microtexture of Type II; they are dominantly pericline-twinned with subordinate lens-shaped albite-twinned areas.
Plate X. Bright field image of a Type I twin domain in host microcline. Albite-twinned lamellae (labelled “a”) alternate with untwinned lamellae (labelled “ut”). The long dimensions of these lamellae are approximately parallel to the b axis.
Plate XI. Bright field image of a Type I domain in host microcline, comparable to the texture shown in Plate X but with narrower widths. Albite-twinned lamellae and untwinned lamellae are labelled “a” and “ut,” respectively.
Plate XII. Bright field image of a Type I domain in host microcline. The albite-twinned lamellae narrow to a few tenths of a μm in this region. Albite-twinned lamellae and untwinned lamellae are labelled “a” and “ut,” respectively.

1 μm
Plate XIII. Bright field image of a Type I domain showing albite twins extending into adjacent untwinned domains. Occasionally, the albite twins cross over and meet the next strongly albite-twinned lamella, indicated by “tc” in the micrograph. Narrow bands of wavy contrast, labelled “nb” parallel the lengths of albite twin bands (labelled “a”) and untwinned bands (labelled “ut”). The extension of albite twin composition planes appears to be blocked by the narrow contrast bands, indicated by “nbt.”
Plate XIV. Bright field image of a Type I domain showing the boundaries between directly adjacent, misoriented albite-twinned lamellae (arrowed). Holes are present at these boundaries, and appear white in the micrograph.
Plate XV. Bright field image of a Type I domain showing narrow bands of contrast (labelled “nb”) that occasionally inhibit the extension of albite twins into untwinned regions, marked “nbt.” Albite twinned lamellae and untwinned lamellae are labelled “a” and “ut,” respectively.
Plate XVI. Bright field image of a Type II domain in host microcline. Albite-twinned lamellae and untwinned lamellae are indicated by “a” and “ut,” respectively, and are occasionally separated by lens-shaped pericline-twinned lamellae, labelled “p.”
Plate XVII. An enlargement of Plate XV to illustrate the strong contrast features (labelled "sc") present where albite twins (labelled "a") overlap pericline twin individuals (labelled "p"). Untwinned regions are labelled "ut."
Plate XVIII. Bright-field image of a Type III domain in host microcline. These domains contain dominantly pericline-twinned regions (labelled "p") with subordinate lens-shaped albite-twinned areas (labelled "a").
Type IV domains (Plates XIX-XX) contain coarsely albite-twinned domains "decorated" with lens-shaped "nuclei"; the latter are approximately 100-1000 angstroms wide. These four major microtextural domains are described in detail below.

Type I domains are illustrated in Plate X. Albite-twinned, long, linear lamellae approximately parallel to the b axis of microcline (labelled "a") alternate with untwinned lamellae. The width of strongly albite-twinned lamellae is variable, ranging from approximately 1 to 0.1 μm (Plates X-XII). The albite twins occasionally extend into neighboring untwinned zones. In some areas these twins cross over and meet the next strongly-twinned albite zone (labelled "to" in Plate XIII). This type of twin interaction may ultimately produce directly adjacent, but slightly misoriented, albite-twinned lamellae; some of the boundaries between the misoriented albite-twinned lamellae are indicated with arrows in Plate XIV. Untwinned regions occasionally contain narrow bands of wavy contrast, oriented approximately parallel to the length of the albite-twinned/untwinned lamellae (marked "nb" in Plates XIII and XV). The trend of these bands is therefore approximately parallel to [010]. The ends of the albite twin lamellae sometimes terminate at the narrow bands (marked "nbt" in Plates XIII and XV), as though the bands inhibit the lengthwise extension of the albite-twin composition planes.

Type II domains include alternating strongly albite-twinned/untwinned regions interrupted by lens-shaped pericline-twinned domains (marked "p" in Plate XVI). The long axis of the pericline lenses is roughly parallel to the long axis of the albite-twinned/untwinned lamellae. The mutual orientation of pericline lenses and untwinned lamellae suggests that the untwinned lamellae may be large-scale pericline twins. Pericline lenses concentrate between albite-twinned lamellae. Where albite twins overlap with pericline lenses, strong contrast occurs (marked "sc" in Plate XVII, an enlargement of the region shown in Plate XVI). Where pericline twinning is more extensive, lens-shaped albite
Plate XIX. Bright field image of a Type IV domain in host microcline. These domains contain large-scale albite-twinned lamellae decorated by "nuclei" that may be sodic plagioclase exsolution lamellae. The nuclei form clusters that trend subparallel to the trace of (010).
Plate XX. Bright field image of a Type IV domain with clusters of nuclei present even where the traces of albite twin planes are absent, i.e., in untwinned zones. The clusters still trend subparallel to the trace of (010).
twinned regions result and the microstructure is termed here as Type III domains (Plate XVIII).

Type IV domains consist of large-scale albite-twinned regions "decorated" with lens-shaped nuclei of the order of 100-1000 angstroms wide (Plate XIX). The long axes of these small lenses are perpendicular to the trace of the (010) albite twin boundaries. The 100-1000 angstrom lenses also appear in areas where no albite twin boundaries are visible; however, they maintain their orientation, forming long clusters that trend parallel to the trace of (010) as indicated with the arrow in Plate XX.

**Albite lamellae**

Albite lamellae in the Spruce Pine specimens vary in scale by several orders of magnitude. Light-optical observations revealed two prominent size ranges. Large-scale vein albite lamellae, that may result from coarsening of smaller albite exsolution lamellae and/or replacement, average a few tenths of a mm wide (100 μm) and 1 mm long and are readily observed using polarized light microscopy. Film albite exsolution lamellae are just resolvable in the light microscope; these range in size from 1 to 10 μm in width and tens to hundreds of μm in length. These were discussed in the light-optical microscopy section above.

TEM, in contrast to the light microscope, shows a continuum of sizes of exsolution lamellae. These range from several μm to tenths of μm in length and tenths of μm to perhaps 100 angstroms in width.

However, observing zone-axis bright-field images does not allow one to easily distinguish between the long dimensions of pericline twin lenses and albite exsolution lamellae because both are parallel to [010]. Both of these lamellar features remain in strong contrast when imaged using BF zone-axis orientations nearly parallel to c*.

Therefore, two-beam images are formed, using reciprocal lattice vectors (g) which
"blank out" the contrast between individual pericline twin lamellae. For example, lamellar-shaped cryptoperthitic albite exsolution lamellae concentrate in transition regions between major microtextural domains. Plates XXI and XXII illustrate such a transition zone, between Type II (upper left) and Type IV (lower right) domains. Plate XXI is a bright field image recorded under two-beam conditions, using \( g = (20\bar{1}) \), so that pericline twin planes are out of contrast. \( (20\bar{1}) \) is the approximate composition plane of pericline twinning in Or-rich microcline. Thus, an image formed using a reciprocal lattice vector perpendicular to this plane, in this case \( g = (20\bar{1}) \), shows no contrast related to the twinning because all pericline twin lamellae diffract with equal intensity under these diffracting conditions (McLaren, 1991). This is convenient for recognizing albite exsolution lamellae because they remain in strong contrast with respect to host microcline for this orientation. Albite lamellae are shown by the arrows for both the bright field (BF) and dark field (DF) image conditions (Plates XXI and XXII, respectively). Out of contrast pericline lamellae are labelled "p" for comparison. Albite exsolution lamellae, arrowed and marked "ab1" in the plates, occur at the boundary between a lens-shaped pericline-twinned region in a Type II domain and a very coarsely albite-twinned region devoid of pericline twinning (Type IV domain). Albite exsolution lamellae (marked "ab2") occur at the intersection of misoriented albite-twinned lamellae in microcline (labelled "a").

The identification of long, lamellar, albite exsolution lamellae was confirmed using energy dispersive spectroscopy (EDS), and is discussed below in the qualitative EDS section.

The nuclei that decorate albite-twin composition planes in Type IV domains share the same contrast features and general orientation as the long-lamellar albite lamellae identified using EDS ("ab1" and "ab2" in Plates XXI and XXII). One interpretation is that they are smaller-scale albite exsolution lamellae, but this is difficult to prove chemically owing to their small grain size. The similarity in contrast and orientation to
Plate XXI. Two-beam bright field image recorded with (201) as the operating reflection. Pericline twin planes (labelled “p”) are out of contrast in this orientation. Cryptoperthitic albite exsolution lamellae remain in contrast and appear dark gray in the photo. A type II domain is shown in the left-center portion of the photo, and contains albite-twinned lamellae (marked “a”) that remain in contrast under these diffracting conditions. A type IV domain exists in the lower right portion of the photo, and contains nuclei that are similar in contrast (dark gray) and orientation (long axis parallel to b) as the larger albite lamellae. Some of these larger cryptoperthitic exsolution lamellae (labelled “ab2”) occur at the boundaries between albite-twinned lamellae and pericline lenses. Others occur at the transition between the Type II and Type IV domains (labelled “ab1”).
Plate XXII. Two-beam dark field image recorded with (20\bar{1}) as the operating reflection. The region is the same as described in Plate XXI. Cryptoperthitic albite exsolution lamellae and “nuclei” appear white in the photo.
the obvious exsolution precipitates makes this a compelling argument, but the possibility exists that they are pericline-twinned regions or some other defect not yet characterized.

Relatively large (1 to 5 μm) diamond-shaped albite exsolution lamellae are also observed in specimen PW-2. The long axis of the diamonds is approximately parallel to the \( \mathbf{b} \) axis of the host microcline. A BF electron micrograph showing two diamond-shaped albite lamellae (labelled “ab”) is shown in Plate XXIII. The oriented holes that parallel the boundaries of the diamonds with the host are noteworthy; dissolution may have been preferential along grain boundaries.

Na-rich vein albite lamellae were recognized in TEM images by the large width of their albite twins, as observed light-optically. Boundaries between vein albite and microcline are irregular, i.e., the orientation between the two phases changes, and host/vein albite boundaries are occasionally curved. Nonetheless, observed planar interfaces apparently prefer a limited number of orientations that can be assigned \( hkl \) indices with respect to microcline.

(010) is one common planar boundary orientation, obvious because its trace parallels the trace of (010) albite twin composition planes. Another common planar boundary occurs whose trace makes an angle of approximately 35 degrees with respect to the \( \mathbf{b}^* \) crystallographic axis. This measured angle corresponds to the (110) and/or (110) planes in microcline. Stereographic projection shows that the angles between the traces of these planes and the \( \mathbf{b}^* \) crystallographic axis, as measured in a plane perpendicular to the [102] zone, are approximately 35 and 36 degrees, respectively (Figure 7). Predicted angles only differ by a degree, so the boundary traces in this orientation are hereafter referred to as the \( hkl \) form \{110\}.

These orientation relationships apply for isolated islands of cross-hatched twinned microcline completely surrounded by vein albite. A [102] zone axis BF image showing the (010) and \{110\} grain boundary orientations between vein albite (labelled
Plate XXIII. Bright field image of a region containing two diamond shaped albite exsolution lamellae (labelled "ab"). The albite lamellae are albite-twinned and are bounded by holes adjacent to host microcline (arrowed).
Figure 7. Stereographic projection of the traces of (110) and (1\{\bar{1}\}0) planes down the [102] direction. The rotated [102] zone and pertinent plane normals are indicated by the "primes", e.g. "[102]'". The line of intersection between the traces of these {110} planes and the \(b^*\) axis is approximately 35 degrees, as measured in a plane perpendicular to [102], i.e. the image plane for [102] zone axis images.
"vab") and a microcline island (labelled "mic") is shown in Plate XXIV. Planar boundary traces follow {110} and (010) of microcline. Curved boundaries are also present, as well as a boundary that trends approximately parallel to pericline twin composition planes, or subparallel to the b axis (indicated by the arrow in Plate XXV). Dislocations (arrowed in Plates XXIV and XXV) are common in vein albite, and appear to interact with the microcline islands in the vicinity of grain boundaries.

Another vein albite/microcline contact is shown in Plate XXVI. This image was recorded in an orientation such that the specimen was tilted off the [102] zone axis, and contrast of pericline twin composition planes (labelled "P") is diminished. The (010) grain boundary trace was observed, and possibly the {110}, but the latter was not confirmed because the image was not recorded with the electron beam parallel to [102].

Cryptoperthite with small-scale albite twinning

A microtexture is observed in the K-rich host of specimen PW2 (labelled "host" in Plates XXVII and XXVIII) that resembles a tweed texture observed by Fitz Gerald and Harrison (1993). This microtexture contains very small-scale albite twinning; the width of twin individuals is on the order of 0.01 to 0.1 μm. The texture does not appear to be a true tweed in that no alternating light and dark linear bands are observed perpendicular to (010). Linear contrast perpendicular to (010) was observed by Fitz Gerald and Harrison (1993) but these authors noted that the (010) twin component was more prominent in the perthite they studied.

Lens-shaped cryptoperthitic albite exsolution lamellae (labelled "ab" in Plate XXVII) averaging a few μm long and a few tenths of a μm wide are present in the fine-scale albite twin domains. The length of the albite lenses parallels the b-axis which is consistent with an (h0l)-type boundary plane with the K-rich host. The precise orientation relationship between these lamellae and the host phase was not determined.
Plate XXIV. [102] zone axis bright field image of a microcline island (labelled "mic") completely surrounded by vein albite (labelled "vab"). Some planar phase boundaries parallel directions that are consistent with the traces of (010) and {110} of microcline. Dislocations are located in the vicinity of grain boundaries (arrowed).
Plate XXV. [102] zone axis bright field image containing the same region as depicted in Plate XXIV. The field of view of this micrograph contains a phase boundary sub-parallel to the trace of pericline twin composition planes (arrowed).
Plate XXVI. Bright field image of a microcline ("mic")/vein albite ("vab") boundary. This image was recorded so that the pericline twin planes (marked "p") are out of contrast, i.e. the specimen is tilted off the [102] zone. (010) vein albite/microcline contacts were noted, as well as a contact similar to the {110} orientation, but the latter is not conclusive because the image plane is not perpendicular to [102]. Curved phase boundaries are also present.
Plate XXVII. (102) bright field image of small-scale albite twinned cryptoperthitic texture. Albite-twinned “host” contains lens-shaped albite exsolution lamellae of sub-μm scale thickness (labelled “ab”) whose lengths trend subparallel to the b-axis. Albite-twinned host alternates with lamellar shaped untwinned regions (labelled “ut”).
Plate XXVIII. Same region described for Plate XXVII that includes a cryptoperthitic albite exsolution lamella (indicated by the arrow) extending across an untwinned zone in the "host." The exsolution lamella is untwinned (according to the albite law) where it overlaps untwinned ("ut") host.
because this requires observations from samples viewed parallel to [010]. However, the exsolution microtexture is very similar to that shown by Fitz Gerald and Harrison (1993) who viewed TEM samples which contain cryptoperthite lamellae parallel to the [010] zone and determined that the lamellar phase boundaries parallel (601) of the K-rich host. These authors imaged their microtexture under bright field conditions with zone axis vectors [106] and [102] parallel to the electron beam. The former yields an edge-on view of the albite lamellae; the latter yields an inclined view of the lamellae on the image plane. The Spruce Pine cryptoperthite lamellae were imaged under bright field conditions parallel to [102], and the shapes of exsolution lamellae in the images are similar to the [102] bright-field images of tweed texture observed by Fitz Gerald and Harrison (1993). In the Spruce Pine sample, long lamellae containing fine-scale albite twinning alternate with long strips of untwinned K-rich host (labelled “ut” in Plates XXVII and XXVIII). Occasionally, albite lamellae extend over the boundary between small-scale albite twinned and untwinned host. When this occurs the lamellae are sometimes untwinned (with respect to the albite law). An albite lamella exhibiting this behavior is indicated with the arrow in Plate XXVIII.

Holes

Holes are a common feature of altered feldspars, and a number of terms used to describe them appear in the literature. For instance, Eggleton and Buseck (1980) termed them “negative crystals” and “vacuoles” in their TEM study of weathered feldspar. Burgess et al. (1992) termed holes observed in deuterically altered perthites as “micropores.” The present work uses these terms interchangeably for holes interpreted to have been present in the perthites prior to sample preparation. Holes also are produced by ion milling, but these are bounded by non-planar outlines that bear no geometric relation to the microtexture of the sample.
Holes bounded by principal crystallographic directions that average a few tenths of a µm in dimension are observed in TEM images of specimens PW1, PW2, and PW3. The holes are bounded by linear edges that represent the traces of crystallographic planes as projected on the image plane. They are associated with misoriented twin domains, microfractures, dislocations, and regions containing alteration to clay. Some large holes with irregular boundaries also occur that likely represent original microtexture but these were not all positively identified as such because ion-thinning also produces holes with irregular outlines. Irregular holes intimately associated with alteration to clay are interpreted to be original.

Three holes in PW1, oriented with their long dimension (as observed in this projection) parallel to the trace of (010), are shown in Plate XXIX. These holes and all holes discussed later appear white in the TEM images. Dislocations are associated with holes in Plate XXIX; this bright-field image recorded nearly parallel to the [102] zone axis shows that holes have developed along a band of dislocations that is indicated by arrows in the micrograph. Other holes in PW1 are observed at the contact between adjacent misoriented albite-twin lamellae (Plate XIV, above), and at boundaries between diamond-shaped albite exsolution lamellae (labelled “ab”) and host microcline (Plate XXX).

PW2 also contains holes; these were observed in association with dislocations and microfractures in host microcline. An example of such a hole, that occurs directly on dislocations, is shown in Plate XXXI. The dislocations are oriented along a narrow band and are indicated by the arrows in the micrograph.

Holes in the PW3 TEM sample are seen in [102] BF images (Plates XXXII and XXXIII). Some linear hole boundaries parallel (010) of the host microcline. Others form linear contacts that are subparallel to pericline-twin composition planes (indicated by the single arrows in Plates XXXII and XXXIII). Another linear hole boundary makes
Plate XXIX. [102] BF image of host microcline in a PW1 TEM sample containing three holes with planar boundaries. These holes and all holes discussed subsequently appear white in the micrographs. These holes are associated with dislocations (indicated by the arrows) and their boundaries terminate in relatively untwinned regions of microcline.
Plate XXX. Hole in PW1 that occurs along a boundary between a diamond-shaped albite exsolution lamella (labelled "ab") and host microcline. Hole is located in the upper right hand portion of the micrograph.
Plate XXXI. Hole in PW2 that occurs in association with a band of dislocations (indicated by the arrows) in host microcline.
Plate XXXII. Holes in PW3 with planar and irregular traces. Planar contacts are oriented subparallel (010), subparallel to pericline twin contacts (indicated by the single arrow), and also form an angle of approximately 35 degrees with $b^*$ consistent with $\{110\}$ (indicated by the double arrows). Irregular boundaries are also present, indicated by “irr.”
Plate XXXIII. Holes in another region of PW3 forming planar contacts subparallel to (010), pericline twin planes (indicated by the single arrow), and {110} (indicated with the double arrows). An irregular hole boundary is indicated by "irr."
an angle of approximately 35 degrees with the $b^*$ axis of microcline, i.e., parallel to the trace of {110} planes (indicated by the double arrows in Plates XXXII and XXXIII). Some holes were observed to have irregular, curved boundaries that change in orientation (labelled “irr” in Plates XXXII and XXXIII). A thinner, more electron-transparent region of host microcline in the same specimen that does not contain holes is shown in Plate XXXIV, so that hole geometry can be compared to host microtexture. Plate XXXIV shows twin composition planes and crystallographic directions more clearly than Plates XXXII and XXXIII. Albite-twinned and pericline-twinned regions are labelled “a” and “p” in Plate XXXIV.

**Microfractures**

Healed (?) microfractures in host microcline are observed in TEM samples of specimen PW2, and these are associated with holes having linear boundaries. Some microfractures trend approximately parallel to the trace of {110} planes (Plate XXXV). Albite twins in microcline (labelled “a”) are occasionally displaced along the trace of the cracks, as along a fault. Negative crystals sometimes occur within a crack; the traces of the planes forming hole boundaries parallel (010) and {110} of microcline. The fracture shown in Plate XXXV was followed for a distance of several μm. Eventually, the fracture grades into a band of dislocations aligned in a sub-parallel “en echelon” arrangement (indicated by the arrows in Plates XXXVI and XXXVII). The angle between the “trace” of the band of dislocations and $b^*$ decreased to approximately 25 degrees, in comparison to 35 degrees observed for the angle between the fracture trace and $b^*$. 

Plate XXXIV. (102) BF image of unaltered microcline from the same specimen (PW3) shown in Plates XXXII and XXXIII. This plate was obtained from a thinner region of the TEM specimen and clearly shows the microtexture; albite twinning is marked “a”, pericline twinning is marked “p.”
Plate XXXV. Microfracture in PW2 host microcline showing albite ("a") and pericline ("p") twinning. Albite twin planes are displaced across the fracture, as along a fault. The fracture trace forms an angle of approximately 35 degrees with $b^*$, consistent with $\{110\}$. Holes are developed along the trace of the fracture with boundary traces parallel to $\{010\}$ and $\{110\}$. 
Plate XXXVI. A microfracture in host microcline of specimen PW2 was followed for several μm in the specimen and appeared to grade in to a band of dislocations, as shown by the arrow in this micrograph. The trace of the dislocation band forms an angle of approximately 25 degrees with $b^*$, compared with 35 degrees for microfracture. Albite twinning is labelled “a,” pericline twinning is labelled “p.”
Plate XXXVII. Band of dislocations in PW2 microcline that extend from the upper left of the micrograph shown in Plate XXXVI (Plates XXXVI and XXXVII form a composite image). The dislocation contrast lines are more clearly seen in the upper left portion of this plate (indicated by the arrow). Albite twinning is labelled “a.”
Electron diffraction analysis of microcline twin textures

A systematic study of twin domains in the microcline host was performed for specimen PW2 to observe the effect of twin texture on diffraction patterns parallel to [102]. A selected area aperture was placed around a particular twin texture and the image, including aperture outline, was recorded. This defines that portion of the image contributing to the corresponding diffraction pattern. All patterns in this section were oriented with [102] parallel to, or nearly so, the electron beam.

A bright-field image of a Type I domain and the corresponding [102] zone axis diffraction pattern is shown in Figures 8a and 8b. The selected area image region contains alternating albite-twinned (labelled “a”) and untwinned (labelled “ut”) microcline lamellae (Figure 8a). The resulting diffraction pattern shows spot doubling along b* in accord with albite-twinned microcline, plus a third spot oriented farther away from the transmitted spot and positioned off center with respect to the (0k0) doublets (indicated by the arrow in Figure 8b). When a smaller selected area aperture was placed over an albite-twinning band only, the third spot was not observed in the pattern; only albite-twin doublets along (0k0) were recorded in this case (indicated by the arrows in Plate XXXVIII). Unfortunately, the image corresponding to this diffraction pattern was overexposed.

An area enclosing albite-twinned microcline (labelled “a”) plus pericline-twinned lenses (labelled “p”) is shown in Figure 9a. This type of texture is representative of that found in Type II or Type III domains discussed above. The diffraction pattern recorded for this region includes spot doublets along b* for albite-twinned microcline plus a third spot (indicated by the arrow), farther from the transmitted beam spot and positioned between the (0k0) doublets (Figure 9b). In this case the diffraction pattern was oriented slightly off [102]. When the crystal was tilted to bring [102] parallel to the beam, the same image area yielded a very complex diffraction pattern (Figure 9c) whose
Figure 8. Electron diffraction analysis of Type I domain in host microcline—

a) [102] BF image of Type I domain in PW2 microcline. Albite twinned region is labelled “a”; untwinned region is labelled “ut.” The curved outlines at the perimeter of the photo indicate the projection of the image of a selected area aperture used to obtain the diffraction pattern shown in Figure 8b.

b) [102] zone axis diffraction pattern from region shown in Figure 8a. Albite-twinned microcline spot doublets are present parallel to b*; the spacing of the doublets increases away from the transmitted spot in the direction of (h0l). A third spot is also present (indicated by the arrow), is located farther from the transmitted spot (in an (h0l) direction) than the albite-twinned microcline doublets, and lies between them.
Plate XXXVIII. [102] zone axis diffraction pattern obtained from a single albite-twinned lamella of microcline in the region depicted in Figure 8a (Type I domain). Only albite-twinned microcline doublets were identified in this pattern, indicated by the double arrows.
Figure 9. Electron diffraction analysis of host microcline containing discrete regions of albite and pericline twinning—

a) [102] BF image of a region including both albite twinned ("a") and pericline twinned ("p") microcline. The selected area aperture is not in position,

b) Diffraction pattern for region in Figure 9a, tilted slightly off the [102] zone axis. The “third spot” discussed in Figure 8b was identified (indicated by the arrow), as were albite-twinned microcline spot doublets,

c) [102] zone axis diffraction pattern obtained from region of Figure 9a. The pattern is complex and is not understood,

d) [102] BF image of a single pericline twinned lens ("p") in microcline from the region shown in Figure 9a. The selected area aperture was placed around the p-twinning, but some albite twinning is also present along the aperture margin,

e) [102] zone axis diffraction pattern from regions shown in Figure 9d. Although the aperture was placed around a pericline twinned lens, no evidence of spot doubling was observed to indicate pericline twinning in the diffraction pattern. Slight streaking was observed parallel to $b^*$ and (h0l) directions; a spot exhibiting the streaking is indicated by the arrow,

f) [102] BF image of selected area region (actually a portion of Figure 9a) containing albite-twinned ("a") and pericline-twinned ("p") microcline, and a boundary between them,

g) [102] zone axis diffraction pattern for image shown in Figure 9f. Albite twinned microcline spots are present along with the third spot similar to that found in Figures 8b and 9b,

h) [102] BF image showing the entire region shown in Figures 9a, 9d, and 9f. Beam damaged microcline in the form of circular holes (arrowed) is concentrated where albite and pericline twins overlap.
Figure 9.
Figure 9, continued
Figure 9, continued
Figure 9, continued
nature is not understood. To test which portions of the microstructure contributed to the
diffraction spots, a smaller aperture was placed around a region containing only pericline
twin lamellae (labelled “p” in Figure 9d). The pattern obtained from this portion of the
texture contained no obvious spot doubling that would indicate the existence of albite or
pericline twinning, but slight streaking was observed in the $b^*$ and (h0l) directions
(arrowed in Figure 9e). Finally, the same size aperture was placed around a portion of a
pericline-twinned lens (labelled “p”) and an adjacent albite-twinned area (labelled “a”),
including the boundary between them. The image and diffraction pattern for this texture
condition are shown in Figures 9f and 9g. The [102] diffraction pattern contains (0k0)
spot doubling and the third spot (arrowed) similar to that described above. After this
twin analysis beam damage in the form of circular holes was observed where albite and
pericline twins intersect (indicated by the arrow in Figure 9h).

Type IV domains were investigated to observe the twinning pattern and also to
check for the existence of albite exsolution lamellae. “Nuclei” observed along the (010)
composition planes of coarsely albite-twinned microcline should yield diffraction spots
for albite exsolution lamellae. An image formed and enclosed by the larger aperture and
the associated diffraction pattern are presented in Figures 10a and 10b, respectively. The
small aperture was also used in the area and the image and diffraction pattern are illus-
trated in Figures 10c and 10d. The “nuclei” are indicated by the arrows in Figures 10a
and 10c. The diffraction patterns show only (0k0) spot doubling resulting from albite-
twinned microcline; the identification of the nuclei as albite exsolution lamellae was
therefore not confirmed using this method.

Diffraction patterns for albite-twinned vein albite in the vicinity of the microcline
twin analysis region were recorded for comparison with albite-twinned microcline. The
image and associated [102] pattern for vein albite (labelled “vab”) are illustrated in Fig-
ures 11a and 11b. The diffraction patterns for the two phases appear identical (compare
Figure 10. Electron diffraction analysis of Type IV domain in host microcline—

a) [102] BF image of a Type IV region in microcline containing large-scale albite twins decorated by "nuclei." Nuclei are indicated by the arrow and the projected image of the selected area aperture used to obtain the diffraction pattern in Figure 10b is shown in this micrograph,

b) [102] zone axis diffraction pattern for the region shown in Figure 10a. Albite-twinned microcline spots are present in the pattern, but no extra spots were observed to identify the "nuclei" shown in Figure 10a,

c) [102] BF image containing a portion of the image shown in Figure 10a (Type IV domain in microcline). A smaller selected area aperture was used to obtain the pattern given in Figure 10d. "Nuclei" are indicated by the arrow,

d) [102] zone axis diffraction pattern obtained from the region shown in Figure 10c. As in Figure 10b, albite twinned microcline spots are evident but no trace of extra spots that would identify the nuclei are present in this pattern.
Figure 10, continued
Figure 11. Electron diffraction analysis of albite-twinned vein albite—
a) [102] BF image of albite-twinned vein albite ("vab") located near the microcline twin analysis region (see text). This image and associated diffraction pattern were taken to compare the geometry of albite-twinned microcline spot patterns with those of albite-twinned vein albite,
b) [102] zone axis diffraction pattern for albite-twinned vein albite (Figure 11a). This pattern contains (0k0) spot doubling in the direction of b* similar to microcline (compare with Figures 10b and 10d). The spots are farther away from the transmitted spot in the direction of (h0l) as expected for (h0l) spacings in albite, but otherwise the patterns appear identical.
with Figures 10b and 10d), but close inspection shows that albite spots are farther away from the transmitted beam than the microcline spots as expected, because albite has larger (h0l) d-spacings. Microcline and vein albite are similar in orientation. Only a few degrees of tilt are required to move from an albite [102] to an adjacent microcline [102] zone axis.

Microtexture of altered regions in Spruce Pine perthites

Characterization of clay mineral alteration

Halloysite, the only alteration product confirmed to be present in PW2, was recognized in TEM by its characteristic rolled layer morphology (Figure 12a). Halloysite is present in void spaces detached from feldspar and also forms grain-boundary interfaces with host microcline and vein albite. The fact that halloysite is often detached is most likely a relict of the sample preparation procedure. Halloysite is prone to thin much more rapidly than the host feldspar when subjected to argon ion bombardment. Therefore, mineral phase boundaries that include halloysite in adequately thin regions (i.e., electron-transparent as opposed to thicker regions that produce dark images due to absorption) are comparatively rare.

A rolled halloysite morphology dominates the altered regions. The rolled morphology may represent either spheres or tubes viewed lengthwise; no clay mineral concentrates from the slightly altered specimens were made to distinguish between these morphologies by TEM. Halloysite particles are occasionally polygonal in outline and show well-developed contrast lines that separate the individuals (arrowed in Figure 12a). These lines may result, in part, from electron-beam dehydration (Kohyama et al., 1978). However, it is likely that the samples were dehydrated under natural conditions and during sample preparation before the specimen was ever brought in contact with the electron
Figure 12. TEM characterization of halloysite—
a) BF image of halloysite ("H") showing a rolled layer morphology. White contrast lines occasionally separate halloysite layers (indicated by the arrows). Rolled layers are circular, oval, or polygonal in form,
b) Representative electron diffraction pattern typical of those obtained from halloysite. The ring patterns contain a 7 angstrom layer spacing (indicated by the arrow).
beam (Gard, 1972). Identification of halloysite was supported by qualitative EDS and SAD. The latter produced ring patterns indicative of 7-angstrom halloysite (Figure 12b). The intensity distribution of the individual rings is similar to experimental and calculated diagrams of XRD lines of halloysite (Brindley, 1951). Corresponding to the calculated XRD lines for halloysite, individual SAD rings are most intense nearest the transmitted beam and the intensity diminishes with increasing \( \sin(\theta)/\lambda \) (Figure 12b).

**Alteration of vein albite**

Halloysite is mostly detached from albite in thin, electron transparent regions, often to the point where it is difficult to determine if the halloysite has moved from the albite contact during ion milling. Where albite/halloysite contacts occur, the boundary is often non-linear and irregular. However, portions of vein albite/halloysite interfaces are observed in one TEM sample. An altered region in vein albite with partially preserved albite/halloysite grain boundaries is shown in Plates XXXIX and XL. These plates form a composite image; a dislocation is arrowed in both plates to show where the regions overlap. Vein albite and halloysite are labelled “vab” and “H,” respectively. Planar albite/halloysite contacts in several orientations are observed. The planar traces make angles of approximately 56, 78, and 90 degrees with \( b^* \). Curved boundaries, indicating a continuous change in orientation, are also present. A hole in vein albite is noted in the vicinity of this altered region that has “negative crystal faces” along (010), plus another orientation whose trace makes an angle of approximately 35 degrees with \( b^* \), which is consistent with the trace of \{110\).

**Alteration of film albite**

No TEM data were obtained for regions of altered film albite that contain intact boundaries with halloysite. However, an area is observed that includes an albite/microcline grain boundary (labelled “gb” in Plates XLII and XLIII) subparallel to (h0l), in
Plate XXXIX. BF image of an altered region of albite-twinnepd vein albite ("vab") recorded nearly parallel to c*. Partially intact planar vein albite/halloysite boundaries form angles of approximately 90, 78, and 56 degrees with respect to b* of vein albite (indicated by numbers in the micrograph). This image forms a composite with Plate XL; a dislocation is arrowed in both plates to show where regions overlap.
Plate XL. BF image of an altered region of albite-twinneed vein albite, including a portion of the image shown in Plate XXXIX (a dislocation is arrowed to show where regions in the micrographs overlap). Some boundaries in Plates XXXVIII and this plate are curved indicating a change in orientation with respect to \( b^* \) of vein albite.
Plate XLI. BF image of a hole in untwinned vein albite ("vab"). This region was recorded near Plates XXXIX and XL, but at a higher magnification, so the orientation of $b^*$ is rotated with respect to its position in Plates XXXIX and XL. Planar hole boundary traces parallel the trace of (010). Another planar trace (arrowed) forms an angle of approximately 35 degrees with $b^*$, consistent with the trace of {110}. 

![Image of a hole in untwinned vein albite](image-url)
Plate XLII. BF image of an albite-twinneal albite lamella whose length is subparallel to (h0l) consistent with film albite observed light optically. The image includes a grain boundary ("gb") between untwinned ("ut") host microcline ("mic") and the albite-twinneal film albite lamella ("fab"). Alteration to halloysite ("H") is shown in the lower left portion of the micrograph and is largely detached from the film albite lamella.
Plate XLIII. BF image of the same lamella described in Plate XLII (these form a composite) and including more of the alteration to halloysite shown in the lower left of Plate XLII.
accord with orientations for film albite observed light-optically. These BF images form a composite in which an albite-twinned albite lamella (labelled "fab") trends diagonally from upper-left to lower-right in the center of Plate XLII. Untwinned ("ut") microcline is in the upper right, and halloysite ("H") is in the lower left of the plate. Halloysite occurs over a distance of several μm as shown in the lower left portion of Plate XLII and throughout Plate XLIII and is mostly detached from the albite interface.

**Alteration of microcline host**

Regular contacts between microcline and halloysite are observed more frequently than for vein albite and halloysite. Halloysite (labelled "H") in a cavity in microcline (labelled "mic") exhibits both planar and sub-planar boundaries (marked "pb" and "spb," respectively in Plate XLIV). In this region, planar boundaries parallel the trace of the (010) planes of microcline (hereafter abbreviated (010)mic), and alteration terminates in relatively untwinned feldspar (labelled "ut"). The right-hand boundary between halloysite and microcline contains negative crystals in the microcline; these holes are bounded by planar "faces" including one orientation parallel to (010) microcline (indicated by the rightmost arrow in Plate XLV).

An elongate channel of halloysite (labelled "H") bounded by (010)mic is shown in Plate XLVI. The microtexture of host microcline (labelled "mic") in this region includes an untwinned zone (labelled "ut") to the left and a pericline-twinned zone (labelled "p") to the right of the halloysite channel. Faint contrast lines parallel to (010) in the pericline-twinned zone (indicated by "a") may represent pericline twins partially converted to albite twins. Albite-twinned albite exsolution lamellae would also produce lines of contrast parallel to (010) but the image was not recorded under the 2-beam diffracting conditions necessary to distinguish them. Halloysite rolls are well developed adjacent to the pericline-twinning. The right-hand boundary between halloysite and
Plate XLIV. BF image of a cavity in host microcline ("mic") containing alteration to halloysite ("H"). Planar microcline/halloysite boundaries ("pb") parallel the trace of (010) and the microcline adjacent to the planar traces tends to be untwinned ("ut"). Subparallel boundaries ("spb") are also present associated with more strongly albite-twinned regions in microcline.
Plate XLV. Enlargement of a portion of the BF image shown in Plate XLIV that includes holes that appear white (indicated by the arrows) positioned along a microcline/halloysite boundary. The holes have planar traces; one was identified that trends parallel to the trace of (010) (indicated by the arrow located at the margin of host microcline).
Plate XLVI. BF image of an elongate channel of halloysite ("H") in host microcline ("mic"). Long planar microcline/halloysite traces parallel (010) and abut untwinned areas ("ut") in microcline. This halloysite channel widens in the vicinity of pericline twinning "p." The halloysite/microcline boundary is more irregular at the contact with pericline twinning, but close inspection shows that some small boundaries with halloysite in this area also parallel (010) (indicated by arrows). The pericline-twinned microcline region contains faint contrast lines parallel to (010) (indicated by "a") that represent albite-twins overlapping pericline twins. Albite exsolution lamellae may also be in the p-twinned vicinity, but this was not confirmed with 2-beam imaging or EDS.
pericline-twinned microcline is much less regular; however, close inspection of this boundary reveals some small-scale planar contact lines parallel to the trace of (010)mic that are indicated by the arrows at the halloysite margin (Plate XLVI). Similar textural relationships are shown in Plate XLVII. All of the feldspar in this region was confirmed to be microcline ("mic") using EDS. Halloysite ("H") is continuous with the halloysite shown in Plate XLVI but the latter was overexposed and appears white in the upper central portion of Plate XLVII. The alteration channel is widest where it is adjacent to more strongly albite-twinned microcline (labelled "a" in Plate XLVII). Halloysite bounded by untwinned microcline (labelled "ut") forms planar contacts parallel to (010)mic, except for one irregular boundary which is indicated by the arrow.

Halloysite channels also are observed in the vicinity of microfractured microcline. An example of such a fracture, whose trace trends subparallel to \{110\} microcline, is shown in Plate XLVIII. A negative crystal is located on the fracture trace that has boundaries parallel to (010) and \{110\} of microcline (indicated with arrows in Plate XLVIII). A 1-2 μm wide halloysite channel (labelled "H") bounded by \{110\} of microcline was observed within several μm of this microfracture in the same TEM specimen (Plate XLIX).

Alteration channels in microcline appear to show no preference for development with respect to specific twinning microtextures. A BF image of a halloysite channel ("H") that cuts across various twin textures is shown in Plate L. Planar (010)mic/halloysite contacts abut albite-twinned (labelled "a"), pericline-twinned (labelled "p"), and untwinned (labelled "ut") microcline. The traces of planar channel boundaries do not always parallel the traces of (010)mic; other angles between \(b^*\) and planar halloysite/microcline contacts were estimated to be near 40 and 70 degrees (labelled "40" and "70" in Plate L).
Plate XLVII. BF image of host microcline ("mic") altered to halloysite ("H"). Planar halloysite/microcline boundary traces parallel (010) and abut untwinned host microcline ("ut"). and irregular contact is also present, indicated by the arrow. The halloysite extends into more strongly albite-twinned host microcline ("a"). The overexposed area that appears white in the upper central portion of this micrograph contains the region shown in Plate XLVI. Phase identification was confirmed using EDS.
Plate XLVIII. [102] BF image of a microfracture in PW2 host microcline. The microfracture trends subparallel to the trace of \{110\}. The fracture contains a hole with planar boundaries parallel to \((010)\) and \{110\} (indicated by the arrows).
Plate XLIX. [102] BF image of microcline altered to halloysite ("H") in the vicinity of the region shown in Plate XLVIII. Microcline/halloysite boundaries are subparallel to \{110\}.
Plate L. BF image of halloysite channel ("H") in host microcline. The channel cuts across albite twinned ("a"), pericline twinned ("p"), and untwinned ("ut") microcline. Planar boundaries form angles of 40 and 70 degrees with b (shown by the numbers on the micrograph) in addition to the planar boundaries that parallel the trace of (010).
The trace of another planar contact makes an angle of approximately 42 degrees with \( b^* \) of microcline (labelled "42" in Plate LI). Microcline adjacent to the halloysite channel in Plate LI contains another textural feature. The lower central and mid-central regions in this micrograph show twin texture in microcline that has been apparently "degraded" to an untwinned state (labelled "ut"). These untwinned regions lack the contrast lines between twin composition planes and contain dislocations (indicated with the arrow). They do not appear to be "original" untwinned zones because their boundaries with respect to the surviving twin texture are irregular. Originally untwinned regions characterized in unaltered microcline are lamellar in shape and form right-angle intersections with twinned regions.

Regions of halloysite completely enclosed by feldspar are most often observed in comparatively thick regions in the TEM specimens. One of these regions is shown in Plates LII-LIV. A \([102]\) BF image of the entire area is shown in Plate LII. The area was also imaged under two beam conditions with \((20\bar{1})\) as the operating reflection; this orientation shows halloysite/feldspar contacts more clearly. The 2-beam bright-field image is presented in Plate LIII and the corresponding dark field image is presented in Plate LIV. Unfortunately, the area is too thick to positively identify the enclosing feldspar (labelled "FELD") as albite or microcline based on twin texture alone and no EDS data were obtained. The author suspects that the feldspar is vein albite because of the large thickness of albite twin individuals located in the upper portion of Plate LIV. Halloysite/feldspar boundaries are either planar (marked "pb") or irregular (marked "irr"); the planar contacts parallel \((010)\) and \((110)\) of the feldspar (Plates LIII and LIV). Linear contrast features (labelled "c") parallel to \((010)\) are present at irregular boundaries and these resemble fine-scale albite twinning. Dislocations also are present in the irregular boundaries (labelled "dis" in Plate LIII). The prominent planar contact parallel to the trace of
Plate LI. BF image of host microcline ("mic") altered to halloysite ("H"). Planar microcline/halloysite contacts parallel (010) plus another planar orientation whose trace makes an angle of approximately 42 degrees with $b^*$ (indicated by "42"). Twin texture in microcline appears to be "degraded" to untwinned areas ("ut") in the central, and lower central portions of the micrograph. The untwinned regions contain dislocations (indicated by the arrow). These untwinned areas do not appear to be "original" because their boundaries with respect to the "surviving" twin texture are irregular. Originally untwinned regions characterized in unaltered microcline are lamellar in shape and form right-angle intersections with twinned regions (compare with untwinned microtextures described previously).
Plate LII. [102] BF image of a halloysite region ("H") completely enclosed by feldspar (FELD). No EDS data are available to confirm the identity of the feldspar, and the area is too thick to determine the identity on the basis of twin texture alone.
Plate LIII. 2-beam BF image of a portion of the Plate LII region recorded with (20\bar{1}) as the operating reflection. Note that the angles between planar halloysite/feldspar boundaries ("pb") and (010) of the feldspar are the same as those measured in the [102] zone axis image. Planar halloysite/feldspar boundaries thus form orientations consistent with (010) and {110} of the feldspar. Irregular boundaries ("irr") are also present and contain fine contrast lines ("c") indicative of small-scale albite twinning as well as dislocations ("dis").
Plate LIV. 2-beam DF image of the same area described for Plate LIII. The same features were identified and labelled as in Plate LIII. The upper left and upper central portions of this micrograph show the large thickness of albite twinning in the feldspar that is similar to albite twin thicknesses observed for vein albite.
in the upper-right portion of the micrograph (arrowed) does not contain these defects (Plates LIII and LIV).

A region is observed in which alteration to halloysite (labelled “H”) completely surrounds “relict” microcline (labelled “mic” in Plate LV). Phase identification in this area was confirmed with EDS. This plate is presented to emphasize that not all feldspar/halloysite boundaries are planar and that in this case irregular contacts dominate. However, a planar phase boundary does exist that parallels the trace of (010)mic (labelled “pb”). Dislocations are observed in a microcline/halloysite phase boundary located in the center-right portion of the photo (indicated by the arrow).

High Resolution Transmission Electron Microscopy (HRTEM)

[102] lattice-fringe images were recorded from albite and pericline-twinned host microcline in specimen PW2. Lattice images show coherent crystal structure across twin composition planes. Dislocations, that would arise to accommodate structural misfit across a twin boundary if the orientation difference could not be achieved by slight distortion of the atomic structure, are not observed in the twin composition planes. Features that resemble edge-dislocations are observed in some areas along albite-twin boundaries, but McLaren (1991) indicates that these features can arise from the slight misorientation and overlap of individuals across the boundary.

A [102] diffraction pattern and lattice fringe image of an albite-twinned region in microcline is shown in Plate LVI. A twin boundary is indicated by the arrow in Plate LVI; twin boundaries separate two zones of different contrast on the TEM photo.
Plate LV. Region of "relict" microcline ("mic") surrounded by alteration to halloysite ("H"). This plate illustrates that halloysite/microcline phase boundaries are not always parallel, and irregular boundaries may dominate. One planar boundary ("pb") was identified in the area parallel to the trace of (010). Dislocations were identified in a halloysite/microcline grain boundary (indicated by the arrow). Phase identification was confirmed using EDS.
Plate LVI. HRTEM image of albite-twinned region in microcline (specimen PW2), [102] zone-axis projection. The lack of dislocations and continuous crystal structure across twin boundaries, which separate twin individuals that alternate in contrast, suggests coherent crystal structure across the (010) twin composition planes. One such twin boundary is indicated by the arrow. Lattice-fringe spacing perpendicular to (010) is approximately 6.5 angstroms.
Electron Microprobe data

Electron microprobe analyses were obtained for host microcline and Na-rich lamellae in a perthite similar in character to PW1. The Na-rich lamellae analyzed are larger than the 10-µm spot size employed for the analyses and therefore represent film or vein albite. The thin section of perthite was oriented with (010) approximately parallel to the glass slide. In this orientation, plagioclase lamellae with (h0l)-type habit planes are oriented perpendicular to the electron probe, which minimizes chemical contribution from the adjacent K-rich host phase. The microprobe analyses are quantitative and give confidence to mineral phase identification using EDS, which are qualitative. EDS data confirm, for instance, the identity of cryptoperthite lamellae that are smaller than a fully converged beam diameter of an electron microprobe.

The results of the microprobe analyses are given in Table 1 in the form of end-member chemical formulae; “Or” represents percent orthoclase (K-content), “Ab” represents percent albite (Na-content), and “An” represents percent anorthite (Ca-content). A sample calculation of an end-member chemical formula from weight percent oxides is presented in Table 2. The weight-percent oxide data used in the calculation are those obtained from analysis number 10 (Table 1). The chemical analyses were performed for individual host and plagioclase lamellae. Broad beam analyses, often used to obtain reintegrated bulk chemistry for cryptoperthites, were not attempted. This is because the large grain sizes of film and vein albite lamellae in the Spruce Pine perthites would require very large sample areas to obtain accurate bulk analyses, which was not possible.

Host microcline averaged >90% Or in composition and vein albite averaged >90% Ab (Table 1). A host phase of essentially pure KAlSi₃O₈ (Or-rich) and a plagioclase phase of essentially pure NaAlSi₃O₈ (Na-rich) simplified phase identification using qualitative EDS. For example, pericline twin lamellae and cryptoperthitic albite lamellae are very similar in size, shape and orientation; they are both long-lamellar in shape.
Table 1. Results of microprobe analyses for host microcline and Na-rich lamellae in a perthite.

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<td>88.73</td>
<td>11.17</td>
<td>0.10</td>
<td>host</td>
</tr>
<tr>
<td>3</td>
<td>94.00</td>
<td>6.00</td>
<td>0.00</td>
<td>host</td>
</tr>
<tr>
<td>4</td>
<td>0.73</td>
<td>97.69</td>
<td>1.58</td>
<td>lamella</td>
</tr>
<tr>
<td>5</td>
<td>91.31</td>
<td>8.69</td>
<td>0.00</td>
<td>host</td>
</tr>
<tr>
<td>6</td>
<td>95.53</td>
<td>4.47</td>
<td>0.00</td>
<td>host</td>
</tr>
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<td>0.38</td>
<td>98.76</td>
<td>0.86</td>
<td>lamella</td>
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<tr>
<td>8</td>
<td>0.70</td>
<td>97.54</td>
<td>1.77</td>
<td>lamella</td>
</tr>
<tr>
<td>9</td>
<td>0.44</td>
<td>97.63</td>
<td>1.94</td>
<td>lamella</td>
</tr>
<tr>
<td>10</td>
<td>0.32</td>
<td>98.64</td>
<td>1.03</td>
<td>lamella</td>
</tr>
<tr>
<td>11</td>
<td>0.44</td>
<td>98.53</td>
<td>1.03</td>
<td>lamella</td>
</tr>
<tr>
<td>12</td>
<td>0.36</td>
<td>98.35</td>
<td>1.29</td>
<td>lamella</td>
</tr>
<tr>
<td>13</td>
<td>0.94</td>
<td>97.52</td>
<td>1.53</td>
<td>lamella</td>
</tr>
<tr>
<td>14</td>
<td>90.84</td>
<td>9.12</td>
<td>0.04</td>
<td>host</td>
</tr>
<tr>
<td>15</td>
<td>94.99</td>
<td>4.85</td>
<td>0.16</td>
<td>host</td>
</tr>
<tr>
<td>16</td>
<td>0.52</td>
<td>98.15</td>
<td>1.33</td>
<td>lamella</td>
</tr>
<tr>
<td>17</td>
<td>93.69</td>
<td>6.22</td>
<td>0.09</td>
<td>host</td>
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<tr>
<td>18</td>
<td>94.25</td>
<td>5.75</td>
<td>0.00</td>
<td>host</td>
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Table 2. Sample calculation of an end-member chemical formula from weight percent oxides.

<table>
<thead>
<tr>
<th>Oxide</th>
<th>wt. % oxide</th>
<th>mol. prop. (1)*</th>
<th>Cation</th>
<th>wt. % cation (2)*</th>
<th>Cation prop. (3)*</th>
<th># of oxygens (4)*</th>
<th>CATIONS (5)*</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiO2</td>
<td>67.9279</td>
<td>1.1305</td>
<td>Si</td>
<td>31.7526</td>
<td>2.2610</td>
<td>2.9859</td>
<td></td>
</tr>
<tr>
<td>Al2O3</td>
<td>19.7759</td>
<td>0.1939</td>
<td>Al</td>
<td>10.4666</td>
<td>0.5819</td>
<td>1.0245</td>
<td></td>
</tr>
<tr>
<td>MgO</td>
<td>0.0003</td>
<td>0.0000</td>
<td>Mg</td>
<td>0.0002</td>
<td>0.0003</td>
<td>0.0000</td>
<td></td>
</tr>
<tr>
<td>CaO</td>
<td>0.0212</td>
<td>0.0038</td>
<td>Ca</td>
<td>0.1516</td>
<td>0.0038</td>
<td>0.0038</td>
<td></td>
</tr>
<tr>
<td>FeO</td>
<td>0.0736</td>
<td>0.0010</td>
<td>Fe</td>
<td>0.0572</td>
<td>0.0010</td>
<td>0.0027</td>
<td></td>
</tr>
<tr>
<td>Na2O</td>
<td>11.1941</td>
<td>0.1806</td>
<td>Na</td>
<td>8.3045</td>
<td>0.3612</td>
<td>0.1806</td>
<td></td>
</tr>
<tr>
<td>K2O</td>
<td>0.0558</td>
<td>0.0006</td>
<td>K</td>
<td>0.0464</td>
<td>0.0012</td>
<td>0.0006</td>
<td></td>
</tr>
<tr>
<td>TOTAL</td>
<td>99.2379</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

(1)* entry = (wt. % oxide)/(mol. wt. oxide)

(2)* entry = (atomic wt. cation/atomic wt. oxide) x (wt % oxide)

(3)* entry = (wt. % cation)/(atomic wt. cation)

(4)* entry = (mol. prop. oxide) x (# of oxygens in oxide)

(5)* entry = (cation prop.) x (oxygen factor)**

** Oxygen factor = 8/total O, in this case = 2.6412.

The oxygen factor assumes 8 O per formula unit, as in (K,Na)AlSi3O8

Each cation proportion is multiplied by the oxygen factor to obtain the total number of each cation per formula unit.
and have (h0l)-type habit planes. When a Na peak was observed in the EDS X-ray spectrum for such a lamellar phase, it was assumed to be cryptoperthitic albite.

**Qualitative Energy Dispersive Spectroscopy (EDS)**

Qualitative EDS was performed to verify the identities of mineral phases observed in the TEM specimens of the Spruce Pine perthites. The detection of Na in cryptoperthitic lamellae required long counting times (400 seconds). This was possibly due to the orientation of the lamellae with respect the TEM samples; the latter are approximately parallel to (001). In (001) TEM specimen orientation, lamellae with (h0l)-type habit planes are inclined to the electron beam; this as well as the small size of the lamellae may have rendered low count rates for Na.

The EDS results were obtained from both altered (to secondary clay) and unaltered regions in the perthites, and are listed in Table 3. The data in the table include, for each analysis, a region number that corresponds to an EDS spectrum which is presented in Appendix 1, a description of the texture analyzed, the counting time for the analysis (in seconds), characteristic peaks identified in order of decreasing intensity, and figure and plate numbers (where applicable).

Some of the analyses in Table 3 were obtained from regions for which there is no corresponding image shown in the present chapter. The region numbers for these analyses are not marked with an asterisk in the table. They include analyses of twin textures in microcline in which no qualitative difference in chemistry was observed for different twin domains observed in microcline host (regions 11-1106 through 16-1106). Other analyses presented without images include those that were obtained from halloysite alteration and adjacent unaltered feldspar (regions 19, 23, 27-35). Some of the clay analyses contained Na along with Si and Al (regions 20-22; 24-26) and the corresponding texture descriptions for these areas (Table 3) include the term “tubular” to describe the
Table 3. EDS results obtained from altered (to secondary clay) and unaltered regions in the perthites.

<table>
<thead>
<tr>
<th>Region</th>
<th>Description</th>
<th>t (s)</th>
<th>Peaks</th>
<th>Plate</th>
</tr>
</thead>
<tbody>
<tr>
<td>10-1107*</td>
<td>host microcline</td>
<td>100</td>
<td>Si, K, Al</td>
<td>XXIII, XXX</td>
</tr>
<tr>
<td>11-1107*</td>
<td>diamond albite lam.</td>
<td>100</td>
<td>Si, Al, Na, small K</td>
<td>XXIII, XXX</td>
</tr>
<tr>
<td>12-1107*</td>
<td>diamond albite lam.</td>
<td>100</td>
<td>Si, Al, Na, small K</td>
<td>XXIII, XXX</td>
</tr>
<tr>
<td>13-1107*</td>
<td>diamond albite lam.</td>
<td>200</td>
<td>Si, Al, Na, small K</td>
<td>XXIII, XXX</td>
</tr>
<tr>
<td>14-1107</td>
<td>small lam., overlap host</td>
<td>200</td>
<td>Si, K, Al</td>
<td></td>
</tr>
<tr>
<td>15-1107</td>
<td>host plus several lam.</td>
<td>100</td>
<td>Si, K, Al</td>
<td></td>
</tr>
<tr>
<td>16-1107</td>
<td>&quot;degraded&quot; host</td>
<td>100</td>
<td>Si, K, Al</td>
<td></td>
</tr>
<tr>
<td>17-1107</td>
<td>&quot;degraded&quot; host</td>
<td>100</td>
<td>Si, K, Al</td>
<td></td>
</tr>
<tr>
<td>18-1107</td>
<td>&quot;degraded?&quot; host</td>
<td>100</td>
<td>Si, K, Al</td>
<td></td>
</tr>
<tr>
<td>19</td>
<td>clay</td>
<td>100</td>
<td>Si, Al</td>
<td></td>
</tr>
<tr>
<td>20</td>
<td>tube form in clay</td>
<td>100</td>
<td>Si, Al, Na</td>
<td></td>
</tr>
<tr>
<td>21</td>
<td>host near clay bound.</td>
<td>100</td>
<td>Si, K, Al</td>
<td></td>
</tr>
<tr>
<td>22</td>
<td>tube forms in clay</td>
<td>100</td>
<td>Si, Al, Na</td>
<td></td>
</tr>
<tr>
<td>23</td>
<td>clay without tubes, central</td>
<td>100</td>
<td>Si, Al</td>
<td></td>
</tr>
<tr>
<td>24</td>
<td>slightly tubular</td>
<td>100</td>
<td>Si, Al, small Na</td>
<td></td>
</tr>
<tr>
<td>25</td>
<td>host</td>
<td>100</td>
<td>Si, K, Al, v. small Na</td>
<td></td>
</tr>
<tr>
<td>26</td>
<td>clay</td>
<td>100</td>
<td>Si, Al, v. small Na</td>
<td></td>
</tr>
<tr>
<td>27</td>
<td>clay, to left of tube</td>
<td>100</td>
<td>Si, Al</td>
<td></td>
</tr>
<tr>
<td>28</td>
<td>another tube</td>
<td>100</td>
<td>Si, Al</td>
<td></td>
</tr>
<tr>
<td>29</td>
<td>host</td>
<td>100</td>
<td>Si, K, Al</td>
<td></td>
</tr>
<tr>
<td>30</td>
<td>host just adj. to clay</td>
<td>100</td>
<td>Si, K, Al</td>
<td></td>
</tr>
<tr>
<td>31</td>
<td>host adj. to clay</td>
<td>100</td>
<td>Si, K, Al</td>
<td></td>
</tr>
<tr>
<td>32</td>
<td>scan on host/clay bound.</td>
<td>100</td>
<td>Si, K, Al</td>
<td></td>
</tr>
<tr>
<td>33</td>
<td>clay just adj. to clay</td>
<td>100</td>
<td>Si, small K, Al</td>
<td></td>
</tr>
<tr>
<td>34</td>
<td>clay</td>
<td>100</td>
<td>Si</td>
<td></td>
</tr>
<tr>
<td>35</td>
<td>clay</td>
<td>100</td>
<td>Si</td>
<td></td>
</tr>
<tr>
<td>36*</td>
<td>&quot;relict&quot; host</td>
<td>100</td>
<td>Si, K, Al</td>
<td>LV</td>
</tr>
<tr>
<td>37</td>
<td>gibbsite?</td>
<td>100</td>
<td>Al, small Si</td>
<td></td>
</tr>
<tr>
<td>38</td>
<td>clay adj. to hi-Al area</td>
<td>100</td>
<td>Al, Si</td>
<td></td>
</tr>
<tr>
<td>39</td>
<td>more gibbsite?</td>
<td>100</td>
<td>Al</td>
<td></td>
</tr>
<tr>
<td>40</td>
<td>clay in twinned host</td>
<td>100</td>
<td>Si, Al</td>
<td>XLVII</td>
</tr>
<tr>
<td>41</td>
<td>clay in twinned host</td>
<td>100</td>
<td>Si, Al</td>
<td>XLVII</td>
</tr>
<tr>
<td>42*</td>
<td>clay in twinned host</td>
<td>100</td>
<td>Si, small K, Al</td>
<td>XLVII</td>
</tr>
<tr>
<td>43*</td>
<td>clay in twinned host</td>
<td>100</td>
<td>Si, small K, Al</td>
<td>XLVII</td>
</tr>
<tr>
<td>44*</td>
<td>clay in twinned host</td>
<td>100</td>
<td>Si, v. small K, Al</td>
<td>XLVII</td>
</tr>
<tr>
<td>45*</td>
<td>clay in twinned host</td>
<td>100</td>
<td>Si, small K, Al</td>
<td>XLVII</td>
</tr>
<tr>
<td>46*</td>
<td>twinned host below clay</td>
<td>100</td>
<td>Si, K, Al</td>
<td>XLVII</td>
</tr>
<tr>
<td>47*</td>
<td>host to right of clay</td>
<td>100</td>
<td>Si, K, Al</td>
<td>XLVII</td>
</tr>
<tr>
<td>48*</td>
<td>crack in host</td>
<td>100</td>
<td>Si, K, Al</td>
<td>XLVII</td>
</tr>
<tr>
<td>49*</td>
<td>rolls above tubes</td>
<td>100</td>
<td>Si, small K, Al</td>
<td>XLVII</td>
</tr>
<tr>
<td>50*</td>
<td>twinned host adj. to clay</td>
<td>100</td>
<td>Si, K, Al</td>
<td>XLVII</td>
</tr>
<tr>
<td>11-1106</td>
<td>&quot;spine&quot; twin texture</td>
<td>100</td>
<td>Si, K, Al</td>
<td></td>
</tr>
<tr>
<td>12-1106</td>
<td>host adj. to spine</td>
<td>100</td>
<td>Si, K, Al</td>
<td></td>
</tr>
<tr>
<td>13-1106</td>
<td>strong contrast lines</td>
<td>100</td>
<td>Si, K, Al</td>
<td></td>
</tr>
<tr>
<td>14-1106</td>
<td>pericline lens</td>
<td>100</td>
<td>Si, K, Al</td>
<td></td>
</tr>
<tr>
<td>15-1106</td>
<td>pericline lens</td>
<td>100</td>
<td>Si, K, Al</td>
<td></td>
</tr>
<tr>
<td>16-1106</td>
<td>pericline lens</td>
<td>100</td>
<td>Si, K, Al</td>
<td></td>
</tr>
<tr>
<td>17-1106</td>
<td>Type IV &quot;nuclei&quot;</td>
<td>400</td>
<td>Si, K, Al</td>
<td></td>
</tr>
<tr>
<td>18-1106</td>
<td>same analysis as 17</td>
<td>100</td>
<td>Si, K, Al</td>
<td></td>
</tr>
<tr>
<td>19-1106</td>
<td>larger lamella than nuclei</td>
<td>400</td>
<td>Si, K, Al</td>
<td></td>
</tr>
<tr>
<td>20-1106</td>
<td>same analysis as 19</td>
<td>100</td>
<td>Si, K, Al</td>
<td></td>
</tr>
<tr>
<td>21-1106</td>
<td>larger lam. than in 19</td>
<td>300</td>
<td>Si, K, Al, v. small Na</td>
<td></td>
</tr>
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</table>
morphism of grains. The presence of Na in these areas is interpreted by the author as most likely representing some relict albite within the halloysite alteration. Two altered regions were analyzed that only produced Al in their spectra (regions 38 and 40). It is possible that these areas contain gibbsite, but in the absence of other data the author regards this interpretation as tentative. Small "nuclei" in Type IV domains did not produce a Na peak to verify their identity as albite exsolution lamellae (regions 17-1106, 18-1106). Moreover, small cryptoperthitic lamellae did not produce significant Na, possibly because of orientation problems described above (regions 19-1106, 20-1106). However, Na was detected in an analysis of a larger lamella nearby with the same morphology, orientation, and contrast as smaller lamellae (region 21-1106). The latter were therefore interpreted as cryptoperthitic exsolution lamellae.

Regions listed in Table 3 that are presented as figures or plates (above) in the present chapter are indicated by an asterisk on the region number. These include: 1) The identification of unaltered diamond-shaped albite lamellae was confirmed by the presence of Na (Regions 10-1107 through 13-1107; Plates XXIII and XXX). Adjacent, smaller lamellae with similar contrast to that of the diamond lamellae did not indicate Na after counting for 100 seconds (regions 14-1107, 15-1107). 2) The identification of "relict" feldspar completely surrounded by halloysite was confirmed as microcline, indicated by an intense K-peak and Na not detected (region 36; Plate LV). 3) The identification of albite-twinned feldspar partially replaced by halloysite was confirmed as microcline, indicated by an intense K-peak (regions 42-50; Plate XLVII).
X-ray Powder Diffraction

*Altered pegmatite*

Two powder mounts were made for mineral-phase identification of coarse and clay-size fractions of pegmatite in which feldspar was completely altered to clay. The X-ray diffraction pattern for an oriented mount of the coarse fraction shows muscovite, quartz, and halloysite are present (Figure 13).

An X-ray pattern from a side-loaded mount of the clay-size fraction shows the presence of gibbsite, halloysite, and quartz, and possibly muscovite (Figure 14).

*Altered microcline*

A perthite specimen similar in intensity of alteration to sample PW3 was crushed and treated ultrasonically in distilled water in an attempt to extract the altered material. An oriented mount made from the supernatant liquid after ultrasonic treatment shows no evidence for the presence of significant clay in the supernatant but yields a pattern for microcline (Figure 15).
Figure 13. X-ray powder pattern of coarse-fraction of extensively altered pegmatite. Mineral phases identified are muscovite, quartz, halloysite, and gibbsite.
Figure 14. X-ray powder pattern of clay-size fraction of extensively altered pegmatite. Mineral phases identified include gibbsite, halloysite, quartz, and a 10 angstrom phase that is likely to be muscovite.
Figure 15. X-ray powder pattern of microcline perthite specimen similar in character to PW3 and collected in the same area as the PW-series perthite.
CHAPTER V
DISCUSSION

Interpretation of twin textures in Spruce Pine perthites

Four general styles of twinning in host microcline are observed in specimens PW1 and PW2, plus a fine-scale albite-twinned cryptoperthitic texture in specimen PW2. Type I domains include alternating albite-twinned and untwinned lamellae approximately 1 μm in width (Plate IX). Akizuki (1972) observed a twin texture comparable to Type I domains in his study of a microcline perthite from Spruce Pine. Type II domains consist of albite-twinned domains occasionally separated by pericline-twinned lenses (Plate XVI). Type III domains are essentially the inverse texture of Type II and are dominated by pericline twinning that is occasionally interrupted by albite-twinned lenses (Plate XVIII). Type IV domains consist of large-scale albite twins decorated with “nuclei” (Plate XIX). The fine-scale albite twinned domains in PW2 (Plate XXVII) resemble a “tweed” texture observed for intermediately-ordered K-feldspar (Fitz Gerald and Harrison, 1993). These authors observe that \( \gamma^* \) deviated from 90 degrees (by less than 1 degree) in [001] electron diffraction patterns of tweed textures, which indicates at least some triclinicity. They attribute the texture to partial ordering during slow cooling (Fitz Gerald and Harrison, 1993).

SAD patterns of specimen PW2 show that albite twinning dominates the microcline twin domains in agreement with studies of Or-rich microclines by Akizuki (1972),
Tibballs and Olsen (1979), and Fitz Gerald and McLaren (1982). The absence of pericline twin spots in a SAD pattern formed from a pericline-twinned lens (Figures 9d and 9e) suggests that pericline twin composition planes may be "relict," as proposed by McLaren (1978). Albite twins in Spruce Pine specimens are observed to cross over pericline-twinned lenses repeatedly (Plate XVII and XVIII), suggesting that the former are more stable at low temperatures and increased ordering.

Interpretation of a third diffraction reflection between albite-twinned microcline reflections noted in PW2 diffraction patterns is not certain (Figure 10b). It could represent "T-twinning," described for microclines crystallized directly with triclinic symmetry (Nord, 1992). Alternatively, the extra reflection could result from cryptoperthitic albite-exsolution lamellae. The twin analysis was performed with the [102] zone axis of the specimen parallel to the electron beam; and under these conditions cryptoperthitic lamellae are difficult to distinguish from pericline twins.

Microtextural relationships between twinning and Na-rich cryptoperthitic lamellae in Spruce Pine perthites

Cryptoperthitic albite lamellae having (h01)-type habit planes with respect to host microcline are identified on the basis of contrast in two-beam images. These lamellae show strong contrast and appear dark gray in bright field and white in dark field images formed with (201) as the operating reflection (Plate XXI and XXII, respectively). Na was detected using EDS performed on a relatively large cryptoperthitic lamella showing the 2-beam contrast described above, confirming its identity as albite (region 21-1106). EDS spectra from the majority of the cryptoperthitic lamellae analyzed did not contain a Na peak; this could be due to their small size and the orientation of the TEM specimens, which is parallel to (001). In (001) sections the lamellae are oriented so that their widths are not perpendicular to the specimen plane but are inclined instead. EDS spectra
obtained from lamellae so oriented likely contain a chemical contribution from overlapping host microcline.

There appears to be a spatial relationship between cryptoperthitic exsolution lamellae and twin textures in the Spruce Pine specimens. Cryptoperthitic albite lamellae occur where pericline and albite-twinned microcline lamellae intersect and where mutually misoriented albite-twinned microcline lamellae meet (indicated by the two arrows labelled “ab2” in Plates XXI and XXII). They are also observed in the transition region between Type II (upper left) and Type IV (lower right) domains (labelled “ab1” in Plates XXI and XXII). Cryptoperthitic lamellae also occur in fine-scale albite-twinned K-rich host and concentrate in twinned relative to untwinned regions, although they occasionally extend into adjacent untwinned host (Plates XXVII and XXVIII). “Nuclei” in Type IV domains that the author believes to be the smallest phase of Na-rich exsolution often occur in contact with (010) composition planes in coarse-scale albite-twinned microcline (Plate XIX). EDS spectra of Type IV regions did not contain a Na peak but this could be due to the small size and orientation of the nuclei as discussed above. Diffraction patterns obtained from Type IV domains do not include reflections for albite; the presence of these reflections would prove the nuclei to be exsolution lamellae (Figures 11a and 11b). However, the contrast of larger cryptoperthitic exsolution lamellae imaged in two-beam conditions is the same as nearby Type IV nuclei (see lower right portion of Plates XXI and XXII); therefore they both share a common orientation (because of similar contrast) with respect to the microcline, an (h0l)-type habit plane, which suggests that these nuclei are also cryptoperthitic exsolution lamellae. If they do represent exsolution, the tendency for the nuclei to occur along albite twin planes suggests that the nucleation was controlled in part by the twin planes. A similar texture is discussed in Smith and Brown (1988), who observed nuclei of sanidine in a perthite containing Ab-rich host; they attribute the formation of K-rich lamellae on
albite-twinned albite composition planes to a heterogeneous nucleation mechanism.

Controversy exists concerning the influence of albite exsolution lamellae on the development of twinning in slowly cooled K-rich perthites. For example, Fitz Gerald and McLaren (1982) believe that twinning occurs independent of exsolution, although they did observe the tendency for exsolution to occur in strongly-twinned regions relative to untwinned regions. In contrast, Tibballs and Olsen (1979) suggest that cryptoperthitic exsolution provides the stress that initiates albite twinning in microcline. The latter also suggest that the symmetry reduction resulting from ordering in alkali feldspars promotes exsolution by reducing the ability of Na and K to remain in solid solution (Tibballs and Olsen, 1979).

TEM images of Spruce Pine specimens show that cryptoperthitic albite concentrates in fine-scale albite-twinned domains in the K-rich host (Plate XXVII) or at the intersections of microcline lamellae twinned according to either the albite or the pericline laws (Plates XXI and XXII). These observations suggest that exsolution influences twin development; i.e., albite twinning is favored in microcline lamellae where the latter abut cryptoperthitic albite lamellae. Alternatively it is possible that a phase of exsolution preferentially develops along the intersections of twin domains in microcline. In evaluating these two possibilities it is useful to consider TEM studies of perthites with well-established cooling histories.

For example, alkali feldspars from rapidly-cooled volcanic rocks (and perthites produced experimentally) show a fine-scale exsolution microtexture containing alternating nanometer-scale Na and K-rich lamellae that are untwinned and monoclinic (Brown and Parsons, 1984). This texture is attributed to spinodal decomposition which was described first by Cahn (1968) as “a chemical modulation small in magnitude and large in extent.” The spinodal mechanism can operate when the composition of a chemical solid solution falls within a region bounded by the inflection points on a free
energy versus temperature curve (Putnis, 1992). More slowly cooled perthites show lamellar coarsening and fine-scale twinning in the Na-rich phase (which becomes triclinic), while the K-rich phase remains untwinned and monoclinic (Lorimer and Champness, 1973). The Klokken syenite in Greenland contains perthites that represent a sequence of textures that were controlled by changing cooling rates in a plutonic environment (Brown and Parsons, 1984b). The most rapidly-cooled Klokken specimens, collected at the margins of the pluton, contain alternating K-rich and Na-rich lamellae that are twinned and triclinic (Brown and Parsons, 1984). Twinning is more pronounced in the Na-rich lamellae. The Klokken also contains some perthites that have been deuterically coarsened with the aid of a fluid, and the K-rich phase is twinned according to the albite and pericline twin laws. Twinning in the coarsened K-rich lamellae resembles that described for maximum microcline by several authors (Fitz Gerald and McLaren, 1982; Akizuki, 1972; Tibballs and Olsen, 1977).

The models described above for the textural evolution of perthites originated from studies of specimens with intermediate bulk compositions. Williame et al. (1976) discuss a progression of textures for Or-rich perthites. The first step in this sequence produces a monoclinic K-rich host and exsolved monoclinic Na-rich lamellae; nucleation is the mechanism thought to produce the exsolved lamellae (Williame et al., 1976). The spinodal mechanism cannot operate for specimens with compositions that do not intersect the spinodal curve on a temperature versus composition diagram. Therefore nucleation is the mechanism attributed to exsolution at more extreme bulk compositions (Putnis, 1992). At later stages the Na-rich phase transforms to triclinic, and, subsequently, twins in response to the misfit between monoclinic host and triclinic lamellae. The lamellae coarsen and the K-rich host twins according to the albite and pericline laws (Williame et al., 1976).
With these models in mind, cryptoperthitic exsolution in the Spruce Pine specimens most likely preceded twinning in microcline and, therefore, exsolution influenced the observed twin textures in the host. The exsolution lamellae labelled “ab2” in Plates XXI and XXII support this view. The “ab2” lamellae occur along boundaries between albite-twinned microcline lamellae, and therefore stress associated with the host/lamellar boundary may have initiated the albite twinning in the host. Moreover, the termination of these albite-twinned lamellae along the cryptoperthitic exsolution lamellae also suggests that the latter may be a barrier to the extension of the albite-twin composition planes in the microcline.

Nuclei observed in Type IV domains probably reflect the reverse scenario; albite twinning seems to influence the development of small nuclei. The prevalence of nuclei in Type IV domains along albite-twin composition planes in the microcline suggests that the former nucleated heterogeneously on the latter (Plate XX). Putnis (1992) discusses heterogeneous nucleation of exsolution lamellae in which nuclei of the latter form preferentially along defect sites in the parent crystal. He lists possible parent defects in order of increasing energy as: point defects, line defects (dislocations), plane defects, grain boundaries, and free surfaces (Putnis, 1992). Albite-twin composition planes apparently served as planar defects to lower the free energy for nucleation of small lamellae in the Spruce Pine perthites.

**Origin and evolution of perthitic textures**

Perthitic textures form by exsolution during subsolidus cooling when host and minor phase unmix. The host and minor phases adopt mutual orientations at their boundaries which minimize the misfit between the two structures that have slightly different unit cell parameters.
For instance, the phase boundary orientation was observed to change with depth (and cooling rate) in perthites from the Klokken syenite. The most rapidly cooled perthites sampled, from the margin of the intrusion, are thought to have been achieved with cooling by spinodal decomposition (the perthites have an "intermediate" composition within the limits of the spinodal) or by homogeneous nucleation. A spinodal texture (without coarsening) is not preserved in the Klokken. The rapidly cooled perthites contain host and exsolution lamellae that are oriented parallel to (601) (Brown and Parsons, 1984b). The Na-rich phase has ordered, has undergone a symmetry inversion to \( \text{CI} \), and albite-twinned, and the K-rich phase is monoclinic and untwinned (Brown and Parsons, 1984b). The plane (601) is the phase boundary predicted theoretically for alkali feldspar exsolution (Bollmann and Nissen, 1968). It is also the phase boundary observed in laboratory studies and in specimens from rapidly cooled volcanic rocks (Parsons and Brown, 1984). Samples from greater depths in the Klokken coarsen and lamellar boundaries become wavy; eventually the albite/microcline lamellar boundary becomes (661).

A deuteric coarsening event that is described as catastrophic and abrupt is also preserved in some perthites from the Klokken; light-optical micrographs of single perthite grains show sharp boundaries dividing coherent braid microperthite and turbid, coarsened regions (Parsons and Brown, 1984). TEM images show a change in the host/lamellar phase grain boundary from (661) to (010) in the coarsened regions (Worden et al., 1990). Moreover, the texture of the K-rich phase changes from untwinned to a twinned texture that includes albite and pericline orientations. A proposed mechanism for deuteric coarsening is one of fluid-induced step-wise dissolution and reprecipitation (O'Neil and Taylor, 1967). The fluid source responsible for the coarsening of lamellae in Klokken perthites is thought to be either magmatic or due to exsolution of "dissolved" water from the feldspar crystal structure (Worden et al., 1990). These authors state that
the coarsened texture in the Klokken perthites occurs in specimens that are the most
"chemically evolved", and that they therefore crystallized from a more water-rich
magma than that which produced the strain-controlled perthites in the same syenite. The
water-rich magma could generate feldspar with a higher dissolved water content to pro-
duce the source of the fluid for catastrophic coarsening (Worden et al., 1990).

Because the PW-series perthites from Spruce Pine occur in pegmatites intruded
in metamorphic country rocks, a large number of physical variables may have had an
effect on the observed exsolution microtextures. Therefore, it is not possible to correlate
each type of albite lamella in the Spruce Pine perthites with a specific cooling rate as was
done for the Klokken syenite (Brown and Parsons, 1984). However, studies of microtex-
tural evolution of perthites from the Klokken and of perthites with high Or-contents
(Williame et al., 1976) provide a guide for probable formation mechanisms of the albite
lamellae observed in the Spruce Pine specimens.

The cryptoperthitic albite lamellae in the Spruce Pine perthites likely formed by
exsolution via a nucleation mechanism. A spinodal mechanism requires rapid cooling
rates where there is not ample time for nucleation and growth (Putnis, 1992), which is
geologically unrealistic for perthites formed in a pegmatite. Moreover, a nucleation
mechanism is consistent with Or-rich perthite bulk compositions (Putnis, 1992), such as
the one measured from a perthite specimen from Spruce Pine that is Or7gAb22An0 (Aki-
zuki, 1972).

Williame et al. (1976) discuss the development of cryptoperthitic lamellae in per-
thites with bulk compositions greater than Or70. For these compositions the K-rich host/
Ab-rich exsolved phase share the (601) phase boundary. The precise albite/microcline
phase boundary was not directly determined for the Spruce Pine specimens, but is an
(h01)-type. Moreover, [102] images of these regions (Plates XXVII and XXVIII) are
very similar in form and contrast to those observed by Fitz Gerald and Harrison (1993),
who determined the orientation of their observed lamellae to be (601) by viewing (010) sections directly. The author believes that the cryptoperthitic phase boundary in the studied Spruce Pine samples is likely to be (601), but examination of (010) TEM samples is necessary to prove this orientation. This was not done because the present evaluation of microtextures concentrated on (001) sections; in this orientation both albite and pericline twin composition planes are viewed directly and can be related to exsolution and alteration in the samples.

The small size of the Type IV nuclei suggests that they represent the latest stage of cryptoperthitic albite exsolution preserved in the Spruce Pine perthites. These regions may have formed in response to annealing and uplift during metamorphism. Heterogeneous nucleation can form at a smaller degree of undercooling than homogeneous nucleation, i.e., a smaller difference between the actual exsolution temperature and the temperature at which the crystal unmixes (Putnis, 1992).

Regions that contain fine-scale albite-twinned K-rich host and cryptoperthitic albite lamellae (Plates XXVII and XXVIII) suggest that the finest-scale albite twinning observed in TEM images of Type I and Type II domains post-dated exsolution and was preferred in areas containing cryptoperthitic albite exsolution lamellae. However, some finely albite-twinned lamellar bands in the host also contain small nuclei that appear to orient along the albite twin composition planes, suggesting heterogeneous nucleation along the twin boundaries, as described above for Type IV nuclei.

The formation of film and vein albite may have involved subsolidus replacement with the aid of a fluid. It is not possible to determine whether the fluid was derived from the “parent” magma or originated from outside the system. In general, the formation of coarse grained plagioclase lamellae in perthites has been attributed to coarsening of pre-existing lamellae or by fracture filling (Smith and Brown, 1988). Because pegmatites form from water-rich magmas, the fluid involved in lamellar coarsening may be
magmatic in origin. Alternatively, fluids may have been derived from metamorphic
country rocks surrounding pegmatites that contain the Spruce Pine perthites. Moreover,
deformation has been attributed to the formation of some perthitic textures (Debat et al.,
1978), and the possibility cannot be ruled out that deformation played a role in the
development of the Spruce Pine perthite textures, although the specimens are not
obviously deformed.

**Lamellar perthite textures as possible crystal structure defects**

Phase boundaries in cryptoperthites may be mostly coherent, as exemplified by
high resolution transmission electron microscopy (Brown and Parsons, 1984b). Criteria
used to define a coherent boundary include a continuous crystal structure across it, i.e.
minimal disruption of crystal structure when crossing the phase boundary, and a subse­
quent lack of dislocations in the boundary (Parsons and Brown, 1984). They have been
termed "strain-controlled" because the orientation adopted by host and exsolved phases
minimizes strain at the phase boundary (Parsons and Brown, 1984). Strain-controlled
exsolution microtextures have been observed in natural perthites (Brown and Parsons,
1984b), have been predicted theoretically (Bollmann and Nissen, 1968; Williame and
Brown, 1974) and have been synthesized experimentally (Yund, 1983). HRTEM images
of the Klokken perthites show coherency across cryptoperthitic phase boundaries even
after coarsening and rotation parallel to (651) planes (Brown and Parsons, 1984b).

In contrast, coarsened lamellae have been shown to be incoherent. For instance,
a TEM study of a deuterically altered perthite from the Klokken syenite showed that
within a single TEM sample, the progression between perthite with coherent lamellae
and deuterically coarsened perthite with incoherent lamellae, could be followed over an
extremely sharp contact only a few μm across (Worden et al., 1990). Associated with the
change to deuteritic texture was a notable increase in the population of micropores along
with a change in albite grain boundary orientation from (661) to (010) of the microcline. Secondary mineralization including clays and Fe-oxides was also observed in the deuterically altered regions (Worden et al., 1990).

A relationship between the size of plagioclase lamellae in perthites and argon retentivity has been shown; coarse-grained lamellar regions retain less argon, while fine-grained lamellar regions are essentially defect-free and retain argon (Parsons, 1993). This is supported by Parsons et al. (1988) who attempted to model argon loss using the dimensions of, and spacing between, small-scale cryptoperthitic lamellae to define the argon diffusion dimension (i.e., the effective grain size). This model shows that cryptoperthitic lamellae retain most argon, compared to deuterically coarsened areas where argon was lost more readily. Burgess et al. (1992), in a study to determine the relationship between microtexture and argon retentivity in perthites, observed that regions containing cryptoperthitic lamellae were “pristine,” i.e., exsolution lamellae were coherent and did not contain holes. In contrast, these authors observed subgrains associated with a large microporosity in deuterically coarsened regions of the perthites and an associated loss of argon of approximately 40%. These studies indicate that feldspars are most defect-prone in regions containing large lamellae.

Alteration of lamellar albite to halloysite in Spruce Pine perthites studied occurs primarily in the vicinity of vein and film albite (Plates XXXIX-XLIII). Small-width (tenths of µm) cryptoperthitic albite exsolution lamellae are not observed to have been replaced by clay. This does not prove that cryptoperthitic lamellae are not altered in these specimens because a limited volume of sample is observed in TEM work. However, my light-optical and TEM data suggest that larger lamellae are more susceptible to alteration.
Relationship between microtextures and dissolution

Early arguments favoring defect-controlled feldspar breakdown resulted from observations using the polarizing light microscope. Feldspar “turbidity” was defined by the cloudy appearance noted by petrographers for feldspars that were then characterized as “altered.” The fact that the turbidity was prevalent in grain interiors as well as at grain boundaries led to hypotheses suggesting that structural defects are responsible for the observed turbidity. But the question as to whether feldspar breakdown is random or structurally controlled remained unanswered until feldspars were observed using electron-optical methods. Dissolution experiments of Berner and Holdren (1977) show that feldspar grains etched in acids dissolve in a crystallographically controlled manner. SEM images of both experimentally etched and naturally weathered feldspars from soils reveal oriented etch pits on (001) that are elongate parallel to <010> (Berner and Holdren, 1977; Berner and Holdren, 1979). Eggleton and Buseck (1980) observed that the traces of “negative crystal” boundaries in weathered feldspar correspond to rational crystallographic planes (001), (010), (101), and (100). Banfield and Eggleton (1990) also describe holes bounded by (010) planes in weathered feldspar.

Micropore geometry observed in (001) sections of some deuterically altered (but not weathered) perthites from the Klokken syenite show that traces of holes were commonly irregular but some were bounded by (010) and {110}; this was observed both in ion-milled TEM and (001)-cleaved SEM images, so hole geometry was not an artifact of the thinning process (Worden et al., 1990). David and Walker (1990) conducted oxygen diffusion experiments on Klokken perthite grains that contain both coarsened, turbid regions and “pristine” (i.e., not deuterically altered) cryptoperthite. After treating cleavage fragments of the perthites in 99% H₂¹⁸O at 0.1 Ga and 700 degrees C, they observed, by ion-microprobe imaging, that the ¹⁸O preferentially penetrated into the turbid regions containing more holes, thereby demonstrating that coarsened regions are
permeable to solutions (David and Walker, 1990). These authors also note that while holes imaged in feldspars have been attributed to formation during weathering (Eggleton and Buseck, 1980), the Klokken perthite micropores are part of the original (not weathered) texture (David and Walker, 1990). However, these authors do discuss the importance of holes and microcracks as potentially susceptible sites for alteration.

Microcline perthites from Spruce Pine also contain "negative crystals" (Eggleton and Buseck, 1980) with sharp linear boundaries. Hole geometry in the Spruce Pine specimens of the present study is similar to that found in the Klokken. In bright-field images close to [102], i.e., with $c^*$ nearly parallel to the electron beam, the angle between $b^*$ and the traces of some of the planes observed to bound holes (approximately 35 degrees) is consistent with those traces corresponding to {110} (Plate XXXV). Holes were also bounded by (010) traces (Plate XXIX). Other orientations are present that have not been "indexed," including an orientation that appears to parallel an (h0l)-type planar trace (Plate XXXIII).

The similarity in hole geometry of Spruce Pine specimens with that of the Klokken perthite is noteworthy because the Klokken is quite different chemically, with an "intermediate" bulk composition of $\text{Or}_{32}\text{Ab}_{67}\text{An}_1$ (Brown and Parsons, 1984b). Bulk chemical data for the Spruce Pine microcline perthites of the present study were not obtained. However, bulk analyses of the microcline perthite sample from Spruce Pine studied by Akizuki (1972) yielded $\text{Or}_{78}\text{Ab}_{22}\text{An}_0$. Therefore, the orientation of micropores in alkali feldspars may not be a function of composition.

The location of dissolution pits in the Spruce Pine perthites appears to be in specific microtextural regions. Negative crystals observed in TEM images of Spruce Pine perthites occur in vein albite (Plates XXXIX-XLI) along dislocations near the intersection of twinned and relatively untwinned regions of host microcline (Plate XXIX), at the boundary between slightly misaligned albite-twinned regions in host
microcline (Plate XIV), at the boundaries between diamond-shaped albite lamellae a few µm in dimension (Plate XXX), and along microfractures (Plates XXXI and XXXV).

The sub-µm scale cryptoperthitic albite exsolution lamellae are not observed to be associated with negative crystals. These observations are in agreement with studies of deuterically altered perthites of intermediate compositions (Worden et al., 1990). These authors showed that micropores occur only in areas where lamellae have coarsened and twinning in K-feldspar was extensive (Worden et al., 1990).

The potential of dislocations and microfractures as sites for alteration

The planes (010) and {110} are prominent cleavage and parting directions in alkali feldspar, respectively (Smith and Brown, 1988). These planes also define slip systems and fracture planes in alkali feldspars. Slip systems can produce plastic deformation parallel to a crystallographic plane along a crystallographic direction in that plane. The crystallographic direction is also known as the Burgers vector for the dislocation. In feldspars slip generally is achieved only at high temperatures experimentally, but may occur in nature at lower temperatures and strain rates over geologic time (Gandais and Willaime, 1984). Water may also encourage ductile behavior at lower temperatures (Gandais and Willaime, 1984). Defect analyses of experimentally deformed sanidine using TEM led to the identification of the most prominent slip systems as (010)[001], (010)[101], and (12\overline{1})[101] at low experimental temperatures (T=700 degrees C). The less important system (1\overline{1}0)1/2[\overline{1}1\overline{2}] was also observed along with others (Gandais and Willaime, 1984). A light-optical study of naturally deformed microcline perthite crystals from Arize, France showed that the prominent slip planes were (001) and {110} at an estimated temperature of 550 degrees C (Debat et al., 1978). Tension gashes infilled with sigmoidally shaped albite lamellae were observed bounded by {110} slip planes. These authors hypothesize that (010) could be a key glide plane for deformation. Debat
et al. (1978) noted an increase in the amount of perthite for more strongly deformed specimens and postulate that perthite formation has a mechanical origin in this case. A light-optical study of microfracturing of plagioclase in a meta-anorthosite, which was naturally deformed under brittle conditions, shows that (001), (010), (110), and (110) are important fracture planes (Brown and Macaudiere, 1984).

TEM images of {110}-trending microfractures in microcline from the Spruce Pine specimens show a progression from microfracture, with displacement of albite twins on either side of the fracture, to bands of dislocations (Plate XXXVI and XXXVII). Holes bounded by the traces of (010) and {110} occur directly on these microfractures (Plate XXXV). Halloysite channels bounded by {110} planes of host microcline (Plate XLIX) occur within a few μm of the {110}-trending microfractures (Plate XLVIII). These observations indicate that halloysite formation may occur along microfractures.

The development of holes along a line of dislocations indicate that the latter are important as sites for dissolution in Spruce Pine perthites. Moreover, dislocations always are observed in vein albite in the vicinity of vein albite/microcline boundaries and sometimes directly on these boundaries (Plate XXIV). Dislocations are also observed at the interface between feldspar and halloysite (Plate LIII and LV). Therefore, dislocations may be important in processes of albite lamellar coarsening, dissolution, and reprecipitation of halloysite. The projection of the images of some dislocation lines on the plane of the specimen shows that they are aligned parallel to the trace of (010) twin planes in vein albite (Plate XXIV). This indicates that dislocations in (010) may have been important in the formation of vein albite.

Dislocation lines are present in other orientations in the Spruce Pine feldspars, but the directions of these relative to crystallographic directions in the feldspar were not determined. Moreover, a determination of the Burgers vectors for dislocations in vein
albite and microcline was not performed. Information on defects involved in transfor-
mations in alkali feldspars may be useful for feldspar dissolution models and is an
important subject of future work.

The development of perthite/halloysite boundary planes along (010) and {110} of
the parent material suggests that these surfaces are important for the development of
alteration. It is likely that cleavage and parting planes allow fluid migration and aid the
formation of µm-scale halloysite “channels.” Linear defects within (010) and {110}
planes may define preferred directions for the transformation of feldspar to clays. For
instance, the slip system (010)[001] was cited by Gandais and Williame (1984) as the
most prominent in deformed feldspar. The [001] direction may therefore be preferred for
alteration processes, but this is speculation until detailed defect analysis is performed.

Optical micrographs of TEM specimens from crystals PW2 and PW3 show that
vein albite lamellae are occasionally connected via alteration channels (Plate VI). It is
possible that stress induced by the development of vein albite lamellae leads to cleavage
or fracture and ultimately to alteration of some regions in host microcline. This is con-
sistent with the observation that halloysite alteration is non-uniform throughout the
microcline. It is also possible that microfracture occurred in the Spruce Pine perthites
after coarsening and the positions of the fractures depend on the orientation of stresses
imparted to the crystals later on.

**Halloysite: Structure and Distinguishing Characteristics**

The major chemical difference between kaolinite, Al$_2$Si$_2$O$_5$(OH)$_4$, and
halloysite, Al$_2$Si$_2$O$_5$(OH)$_4$.2H$_2$O, is the presence of interlayer water in the hydrated
form of the latter, and the fact that halloysite shows more stacking disorder than
kaolinite. Halloysite and kaolinite usually also have different particle morphologies,
which is likely at least partially, a function of their different chemistries, and they are
commonly distinguished from one another by this characteristic. In general, halloysite tends to exhibit rolled or spherical particle shapes while kaolinite is platy or sheet-like.

TEM is deemed the most accurate method of distinguishing between kaolinite and halloysite (Dixon, 1989), because particle morphology can be observed directly.

The diagnostic rolled-tube and spherical morphologies of halloysite have been attributed to surface tension between layers (Hope and Kittrick, 1964). Dehydration of 10-angstrom halloysite with a platy morphology caused the development of rolled crystallites after air drying (Hope and Kittrick, 1964). Rolling of initially platy kaolinite (Robertson and Eggleton, 1991) and dislocation-assisted spiral growth (Kirkman, 1981) have also been proposed as possible mechanisms of formation for halloysite. Halloysite crystals may also be thinner and less extensive in the ab-plane which may allow the polar structure to curl into a rolled morphology.

**Halloysite: Discussion of its significance in the weathering environment**

The occurrence of halloysite as an alteration product in saprolites and soils derived from a wide range of parent-rock or parent-soil mineral compositions is well documented. Halloysite has been observed in electron microscopy studies of naturally weathered granites (Tazaki and Fyfe, 1987; Robertson and Eggleton, 1991; Banfield and Eggleton, 1990; Churchman and Gilkes, 1989; Singh and Gilkes, 1992), weathered rhyolitic tephras (Kirkman, 1981), weathered trachytic pumice (Quantin et al., 1988), weathered basalts including pyroclastics (Banfield et al., 1991), and weathered soils derived from basaltic volcanics (Delvaux et al., 1990; Delvaux et al., 1992; Sudo, 1953; Sudo and Takahashi, 1956; Pecro et al., 1962). Halloysite also was observed in an electron microscopy study of laboratory altered K-feldspar crystals (Parham, 1969). Parent-rock mineralogy, composition, and rock texture (grain size) are, therefore, not the only controls on the formation of halloysite in saprolites and soils. In fact, Grim (1968) notes
that the same parent rock can produce very different clay mineral weathering products; likewise, the same clay mineral assemblage can be formed from parent rocks of variable composition and mineralogy.

Several studies of changes in soil mineralogy as a function of soil profile depth indicate that halloysite decreases with a concurrent increase in kaolinite with distance from the parent rock (Churchman and Gilkes, 1989; Delvaux et al., 1992). Delvaux et al. (1992) investigated the changes in particle morphology as a function of depth for several clay fractions of weathered basaltic soils. The number of clay particles with a rolled or spherical morphology decreased upsection, i.e. halloysite was shown to decrease in favor of platy kaolinite at more advanced stages of weathering.

In general, halloysite is less stable than kaolinite in the soil environment (Dixon, 1989). Very few soil types have been classified as halloysitic. Perhaps frequent periods of wetting and drying sustained in the soil environment also influence the degradation of halloysite in favor of kaolinite (Dixon, 1989). Saprolites containing halloysite likely avoid the wet/dry cycles sustained closer to the surface. Halloysite formation has been associated with tropical climates with no definite dry season (Beutelspacher et al., 1961).

TEM studies of feldspar weathering have focussed on determining reaction mechanisms taking place in the transformation from parent feldspar to product clay minerals. As noted by Banfield and Eggleton (1990), the importance of quasi-crystalline intermediate reaction products as precursors to product clay minerals is still uncertain. Several authors suggest that such a precursor is not important and that transformation proceeds directly via dissolution and re-precipitation (Keller, 1978; Petrovic, 1976b; Tsuzuki and Kawabe, 1983). In contrast, TEM studies of K-feldspar weathering indicate that a primitive precursor to clay product may be important (Banfield and Eggleton, 1990; Tazaki and Fyfe, 1987) and that halloysite may preferentially precipitate from this intermediate.
Tazaki and Fyfe (1987) studied what they termed "primitive precursor" phases on naturally-weathered microcline perthites. The perthites came from a specimen surrounded by an iron-rich weathering rind. Spherical halloysite apparently formed from Fe-rich precursors in the following sequence: K-feldspar, to fibers with Fe spots, to bundles of fibers, to circular forms, to halloysite (Tazaki and Fyfe, 1987).

Banfield and Eggleton (1990) note a similar sequence outward from the K-feldspar interface; in this case the authors observe a "cell-textured" material abutting K-feldspar grains, intergrown with and then followed by smectite, a second cell-textured material, followed finally by halloysite (Banfield and Eggleton, 1990). The cell-textured material is defined by the authors as tiny, equant grains lacking long-range order that are not completely amorphous, with a Si:Al ratio similar to allophane which ranges from 1.3 to 2.0 (Banfield and Eggleton, 1990). Chemical data showed an increase in iron content for the cell-textured material and electron diffraction data indicated that the material was intermediate between an amorphous and a completely crystalline phase.

Both studies described above discuss the formation of halloysite in terms of timing and intensity of weathering; material farther from unaltered feldspar interfaces (i.e., halloysite) is assumed to have been subjected to the alteration process for a longer time. Tazaki and Fyfe (1987) note that well crystallized halloysite forms from primitive clay precursors "in even a more intense stage of weathering." Banfield and Eggleton (1990) observed tubular halloysite "in cavities in more weathered samples."

In the present study, halloysite rolls are observed to abut unaltered microcline, suggesting that reaction intermediates are not important in the Spruce Pine perthites. However, HRTEM imaging of product halloysite/feldspar boundaries were not successful, and it is possible that intermediate phases are present but are beyond the resolution limit of conventional TEM (Banfield, personal communication). No definite orientation relationship exists between parent feldspar and halloysite, i.e., alteration is
non-topotactic. However, SAD patterns indicate that the halloysite tubes (or spheres) are oriented to some degree. If the halloysite rolled-layer morphology represents tubes viewed end on it is possible that the crystallographic axis defining the axis of the tube may preferentially align along the feldspar planes observed to abut halloysite, namely (at least) (010) and \{110\}.

It must be emphasized that although the Spruce Pine perthites were collected from a weathered profile, the alteration to halloysite may be of hydrothermal origin. However, it is expected that fluids of deuteric, hydrothermal, or meteoric origin would prefer similar pathways for transformation of feldspar to clay.
CHAPTER VI

CONCLUSIONS

Interpretation of Twin Textures

Twinning microtextures observed in the microcline host of Spruce Pine perthites are in accord with those observed in other feldspars from granites and pegmatites similar in composition, i.e., those with potassium-rich bulk compositions. SAD analysis of Spruce Pine textures indicates that the albite twin orientation is dominant in microcline. This was proposed also by Akizuki (1972), FitzGerald and McLaren (1982) and Tibballs and Olsen (1977) for perthites from granitic rocks and pegmatites. Bright-field images of PW-series perthites contain pericline-twinned domains that apparently are converting to albite twins; this is indicated by the extension of albite twin composition planes over pericline-twin lamellae (marked “sc” in Plate XVII).

Akizuki (1972) presented an image of microcline twinning from a perthite collected from the Spruce Pine district that resembles the Type I domains (Plates X, XI, and XII) observed in the present study. He describes the twin domains as bands perpendicular to the albite twin orientation with sharp boundaries, with some albite twins projecting from the boundaries. Akizuki also states that pericline twin reflections are not always produced in diffraction patterns obtained from such twin domains, although the bands are described as having a “contrast similar to the pericline twin.” These data and the data from the PW-series perthites suggest that bands resembling pericline twins are adopting
the albite-twin orientation. The formation of adjacent long lamellar misoriented albite-twinned regions observed in the present study (Plate XIV), like those observed by Fitz Gerald and McLaren (1982), may represent the complete conversion of bands that were at one time (at least morphologically) pericline twins.

Pericline-twinned regions in Type II and Type III domains of the PW-series also support conversion of pericline to albite twinning. Some images of pericline-twinned lenses are apparently crossed by albite-twin composition planes (Plates XVII and XVIII).

Domains of fine-scale albite twinning are also present in the PW-series perthites (Plate XXVII). A similar type of twin domain in an Or-rich specimen has been attributed to “partial ordering with slow cooling in nature” in specimens observed by Fitz Gerald and Harrison (1993). The albite-twin component dominates the pericline-twin orientation present in this “tweed” texture (Fitz Gerald and Harrison, 1993). Akizuki (1972) also reports perthite specimens from Japan containing “fine cross-hatching” indicative of orthoclase textures, and SAD analysis revealed that the albite-twin orientation dominates the cross-hatching. Akizuki (1972) suggests that fine cross-hatching develops in response to incipient ordering resulting in the monoclinic to triclinic inversion, and that pericline and albite twins coarsen and form discrete domains with increasing structural order. The author believes that the fine-scale albite twinning in the Spruce Pine perthites may also represent a “less-ordered state” because the twinning resembles textures that show less triclinicity (FitzGerald and Harrison, 1993).
Interpretation of Na-rich Lamellae

Na-rich lamellae cover an extreme range of sizes in the PW series samples. From coarsest to finest texture, these include macroscopic vein albite (tenths of mm to several mm in dimension), microscopic film albite (up to tens of μm in dimension), and cryptoperthitic albite that range from a few tenths of a μm to perhaps 100 angstroms in lamellar width. The cryptoperthitic lamellae appear to represent more than one stage of exsolution. Cryptoperthite in the 0.1-0.5 μm width range was observed in two types of microcline twin-domain textures. In one of these, cryptoperthitic albite occurs where pericline-twinned lenses meet albite twinned lamellae, or where two albite-twinned lamellae intersect (Plates XXI and XXII). Cryptoperthitic albite juxtaposed along and separating these twinned regions suggests that the former may have influenced the development of albite and pericline twinning in discrete regions. Cryptoperthitic albite in the 0.1-0.5 μm size range also occurs in fine-scale albite-twinned K-rich host (Plates XXVII and XXVIII). The fine-scale albite twinning is homogeneous in scale and extent in this texture and does not appear to have been affected by the presence of exsolution lamellae. If the fine-scale texture represents feldspar in a “less-ordered state” than feldspar containing both pericline and albite twins in discrete regions, then perhaps cryptoperthitic albite only influences the twin textures developed with increased ordering in microcline.

The nature of the narrow bands of wavy contrast, oriented approximately parallel to the b-axis in untwinned microcline lamellae (labelled “nb” in Plate XV) has not been determined. A similar texture was observed in a two-perthite alkali feldspar crystal studied by Brown and Parsons (1983). These authors describe “speckled contrast” in the Or-rich lamellae of a cryptoperthite and state that they did not determine the precise nature of the contrast (Brown and Parsons, 1983). However, they believe that the speckled contrast represents thin, oriented platelets of exsolution lamellae formed by homogenous
nucleation. Albite twinning in the PW-series microclines is blocked or retarded at the bands of contrast (marked “nbt” in Plate XV). This suggests that whatever the cause of the bands’ contrast, they are important in inhibiting the extension of the albite twinning. If these bands are fine-scale “nuclei,” then they may represent another phase of exsolution that influences the formation of discrete regions of albite or pericline twinning, as described for cryptoperthitic lamellae discussed above.

The “nuclei” observed in Type IV domains are believed by the author to represent a separate stage of exsolution, possibly resulting from regional uplift and a subsequent increase in cooling rate. The nuclei share the same orientation with larger cryptoperthitic lamellae that were confirmed to be albite by the presence of Na in EDS spectra. They also have a lamellar habit typical of exsolution lamellae. The tendency of the nuclei to align in clusters directly on albite-twin composition planes suggests that the exsolution mechanism was one of heterogeneous nucleation. However, some clusters of nuclei are present where no albite twin plane is observed (Plate XX). A similar texture was reported in Smith (1988) for an antiperthite; in this case K-rich nuclei are occasionally aligned along albite-twin planes in a Na-rich host. Large-width albite-twinned domains from a perthite from Japan, similar in scale as those observed in the Type IV domains of the present study, were described by Akizuki (1972) but did not contain “nuclei.”

The formation of film and vein albite in perthites has been attributed to coarsening of pre-existing cryptoperthite, or to replacement; the latter mechanism may be “mutual” replacement or replacement by introduction of components from outside the crystal, i.e., “open-system replacement” (Smith, 1988). Either mechanism is considered to be aided by the presence of a fluid that originates from a water-rich differentiated magma (deuteric) or from hydrothermal fluids originating from outside the crystal (Smith, 1988). In pegmatites, either mutual replacement or open-system replacement is
possible (Smith, 1988). An optical microscope study of vein albite textures from giant perthite crystals in pegmatites suggests that vein albite forms from the coalescence of patch perthite (Sanchez et al., 1993). Patch perthite "lamellae" are similar in size to vein albite but are more variable in morphology than the latter (Smith and Brown, 1988). However, patch perthite formation in pegmatites has been attributed either to mutual replacement or to open-system replacement (Smith, 1988), and, therefore, this mechanism does not appear to resolve the question as to whether replacement was intracrystalline or occurred by introduction of chemical components from outside the crystal.

Deuteric coarsening by mutual replacement has been suggested for the formation of coarsened lamellae in some Klokken syenite perthites (Worden et al., 1990). These authors observed that the deuteronically-altered regions cross-cut unaltered regions. Cross-cutting of cryptoperthitic regions by coarsened vein or film albite was not observed in the Spruce Pine specimens studied. Therefore, establishing the relative timing of the lamellar textures is not possible. Nor is it possible to establish whether Na was brought into the crystal from the outside. If open system replacement to produce vein albite did occur, a possible source of external Na is through the weathering of coexisting plagioclase in Spruce Pine pegmatites. Coexisting plagioclases from these pegmatites range from albite (Ab\textsubscript{100}An\textsubscript{0} - Ab\textsubscript{90}An\textsubscript{10}) to oligoclase (Ab\textsubscript{90}An\textsubscript{10} - Ab\textsubscript{70}An\textsubscript{30}) in composition (Maurice, 1940).
Interpretation of Defect and Alteration Textures

TEM data for the studied Spruce Pine perthites show that traces of planes bounding negative crystals, microfractures, microcline/vein albite contacts, and halloysite-infilled channels are similar in orientation. Planar interfaces for all of these microtextural components parallel (010) and {110} of the microcline host. Other orientations also were noted. Penetration of a feldspar crystal by a fluid phase has been proposed as being essential for the formation of vein albite (Smith, 1988), the formation of holes (Worden et al., 1990), and alteration to secondary minerals (Worden et al., 1990). Some of the negative crystals observed may have been present prior to alteration to clay (David and Walker, 1990). Nonetheless, the association of holes with microfractures and with clay/feldspar interfaces suggests that even if some are primary in origin, they are also important as reactive sites for the development of alteration. Therefore the present observations lead to the hypothesis that the (010) and {110} planes impart at least control for fluid-induced changes of the crystal structure: (010) is a prominent cleavage direction that intersects (001) sections of feldspar; and {110} are prominent parting directions, especially in perthites (Smith and Brown, 1988).

Berner (1981) discusses the possible mechanisms for dissolution and replacement of minerals during alteration reactions. These include surface-controlled, transport-controlled, and a combination of these two mechanisms. According to Berner (1981), a surface-controlled reaction produces dissolution pits in the parent mineral that are bounded by planar interfaces, which implies crystallographic control on the part of the parent mineral. A surface reaction is thought to proceed preferentially at high energy sites, such as at the “outcrops” of dislocations along the fluid/crystal interface (Berner, 1981). A transport-controlled mechanism, on the other hand, produces irregular dissolution surfaces on the reacting parent crystal, implying the loss of crystallographic control on the alteration process (Berner, 1981). The planar nature of clay/feldspar boundaries
observed in the Spruce Pine perthites indicate that the reaction is at least in part surface-controlled, and that important surfaces include (010) and {110}. The most extensively altered specimen, PW3, contained negative crystals bounded by (010) and {110}, as well as others. However, holes in the PW3 specimen were also noted with irregular boundaries, which indicates that the nature of the reaction may change to transport-controlled as alteration becomes more extensive.

Unaltered, twinned regions that include all of the observed twin domains in microcline are cross-cut by approximately 1 μm-wide channels of halloysite. This suggests that microfracture and cleavage planes provide important pathways for solutions that produce clays. Twin boundaries alone do not appear to have rendered the Spruce Pine perthites permeable.

With the present TEM data, no distinctions are made between pericline and albite twins in terms of susceptibility to alteration. Channels of halloysite were observed to cross-cut all twin domain types without preference of one domain over another. However, in some regions of host microcline, alteration to halloysite is favored in more extensively twinned areas (Plates XLVI and XLVII). This suggests that once alteration to clay begins, dissolution and replacement of microcline by clay occurs more readily in more strongly-twinned areas, i.e., those regions containing a greater percentage of twin composition planes per volume of crystal.

Dislocations were observed at the contacts between vein albite and microcline, in association with negative crystals and halloysite alteration in microcline, and in some feldspar/halloysite boundaries. Dislocation lines are observed to approximately parallel (010) in vein albite, and sometimes occur at (010) twin contacts. These observations suggest that dislocations are important in the coarsening of vein albite, in the formation of holes, and in the formation of halloysite in perthites from Spruce Pine.
Non-planar reaction boundaries were observed between host microcline and halloysite and vein albite and halloysite. Thus, it is possible that crystallographic control is minimized after a certain point in the reaction to clays, as suggested by Banfield and Eggleton (1990).

Both light-optical and TEM data suggest that there is textural control on the alteration of microcline perthites from Spruce Pine. The most important avenues for the passage of fluids are grain boundaries formed between vein and film albite and host microcline, microfractures, and negative crystals. Moreover, vein albite lamellae are observed to be interconnected by halloysite channels. Vein albite/microcline grain boundaries, microfractures, and negative crystals all are along (010) and {110}, which suggests that these are very important surfaces in the reaction of feldspars to clay. Strongly-twinned regions appear to be favored over untwinned areas once the crystal has become permeable. Specific twin domains and cryptoperthitic exsolution lamellae do not appear to be as susceptible to alteration as do large-lamellar albite, fractures, and negative crystals.

Algorithm Developed for TEM Specimen Orientation

An algorithm was developed for TEM specimen orientation, as discussed above in Chapter III. This was done early in the study when the author was learning about electron diffraction patterns obtained from feldspars. What follows are comments on the course of study leading to the development of the algorithm, discussion of how the algorithm was used in the present study, and discussion of the merits of its use in future studies. Details of the algorithm are presented in Appendix B, which includes a computer program and sample calculations using experimental data from isometric, tetragonal, and triclinic crystals.
The first TEM specimens examined were prepared from thin sections cut approximately parallel to the (010) cleavage directions. The focus of the study at this stage was on the development of the algorithm, in anticipation of identifying defects whose characterization would require the use of various diffracting vectors. The use of different diffracting vectors depends upon finding the zone axes containing these vectors, and a computer program was developed that would calculate the tilt angles necessary to view any third zone axis, given that two other zones were successfully identified and their goniometer tilts were determined experimentally. Some success was met at calculating the third zone axis, but the program did not always predict the tilts observed experimentally for the third zone axis.

The next TEM specimens were cut from thin sections of PW1 oriented parallel to the (001) cleavage direction. Preparation of these specimens yielded the first extensive thin areas containing twinning microtextures. The focus of the study at this stage changed to obtaining images of various twin domains, and also to comparing these observations with the observations of TEM images of microcline made by other workers. [102] electron diffraction patterns were found and indexed based on d-spacings and angles calculated by estimated unit cell parameters (Brown and Bailey, 1964). This orientation and others close to [102] was maintained for consistency in comparing observations. Development of the algorithm was abandoned during this stage.

The next set of specimens contained alteration to halloysite (samples from PW2 and PW3) in the thinned regions, and the focus shifted to determining the spatial relationship between microtextural domains and alteration. The [102] orientation was maintained for comparison with previous observations. The algorithm was revisited near the end of the study and calculations were made using zone axes that the author considers to be indexed with confidence. The algorithm predicted the positions of third zone axes for PW-series microclines only in some cases. The program was tested using tilt data for
isometric and tetragonal crystals obtained from Cindy Wise Barnicky and Dan Evans, respectively. Good agreement was found between experimental and calculated third zone axes of the isometric and tetragonal crystals (Tables 4 and 5, Appendix B). Data from one tilt analysis of the tetragonal crystal (bottom of Table 5, Appendix B) yielded internally consistent results for four out of five observed zone axes. The calculated tilts for one of these five zones did not agree with experimental observations, so several zones of the same crystallographic form were calculated, and again, the agreement was poor. However, a zone axis located close to the experimentally observed zone (when their positions are compared in stereographic projection) yielded tilts comparable to those observed experimentally.

The author considers problems in predicting zone axes in the microclines to be due to the following: 1) The unit cell parameters of microclines are large and the crystals have low symmetry; therefore, a large number of closely-spaced zone axes whose diffraction patterns are similar in geometry are present. This can lead to incorrectly indexed patterns. 2) Experimentally-determined tilt-angles are only accurate to +/- a few degrees of tilt, and so small errors in tilt (i.e., the observer may not have the crystal oriented precisely parallel to the zone) may cause problems in predicting the positions of other zones. The data for microcline (Table 6) show that the calculations are sensitive to small changes in tilt.

The algorithm was successful in predicting tilt angles for higher symmetry crystals whose zone axes are not close together, and thus has merit for at least higher-symmetry crystals as a rapid check for specimen orientation. Defect analysis may proceed more efficiently because the TEM operator can calculate tilt angles without the use of stereographic projection.
Suggestions for Future Work

(1) TEM observations of perthite sections cut parallel to other principal crystallographic directions are necessary to fully characterize the geometry of alteration. For instance, (001) is the dominant cleavage direction and also are cited as a prominent direction for microfracture (Brown and Macaudiere, 1984). Sections of feldspar cut parallel to (010) may reveal (001) as a prominent boundary for alteration channels.

(2) A detailed characterization of dislocations including a determination of the Burgers vectors was not performed. This needs to be done for dislocations associated with the development of negative crystals, dislocations at or near vein and film albite/microcline boundaries, and dislocations observed at feldspar/halloysite margins.

(3) Studies concerned with modeling the rate of dissolution of feldspars are concerned with the reactive surface area. Field-dissolution rates may be slower than laboratory rates by several orders of magnitude, and this has been attributed to an overestimation of the physical surface area involved in the reaction (Rowe and Brantley, 1993). A first approximation that might prove useful for modeling dissolution in microcline would be to estimate accurately the surface area formed at vein and/or film albite contacts, possibly by image analysis of light-optical micrographs.

(4) Stable isotope work on halloysite may be useful for determining the origin(s) of the reacting fluids.
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APPENDIX A

ENERGY DISPERSIVE SPECTROSCOPY
Figure 16. EDS spectra obtained from host microcline and albite lamellae—
a) Region 10-1107 (Plates XXIII and XXX),
b) Region 11-1107 (Plates XXIII and XXX),
c) Region 12-1107 (Plates XXIII and XXX),
d) Region 13-1107 (Plates XXIII and XXX),
e) Region 14-1107,
f) Region 15-1107,
g) Region 16-1107,
h) Region 17-1107,
i) Region 18-1107.
Figure 16, continued

**REGION 11-1107**

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**REGION 12-1107**

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Figure 16, continued

**REGION 13-1107**

**REGION 14-1107**

**ENERGY** (KeV)
Figure 16, continued
Figure 16, continued

**REGION 17-1107**

- Si
- Al
- K
- Na
- Cu

**ENERGY (KeV)**

**REGION 18-1107**

- Si
- Al
- K
- Na
- Cu

**ENERGY (KeV)**
Figure 17. EDS spectra obtained from clay and adjacent host microcline—
a) Region 19, b) Region 20, c) Region 21, d) Region 22, e) Region 23,
f) Region 24, g) Region 25, h) Region 26, i) Region 27, j) Region 28,
k) Region 29, l) Region 30, m) Region 31, n) Region 32, o) Region 33,
p) Region 34, q) Region 35, r) Region 36 (Plate LV), s) Region 38,
t) Region 39, u) Region 40.
Figure 17, continued

REGION 22

REGION 23
Figure 17, continued

**REGION 24**

**REGION 25**

Energy (KeV)
Figure 17, continued
Figure 17, continued

**REGION 28**

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**REGION 29**

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**ENERGY (KeV)**
Figure 17, continued
Figure 17, continued

REGION 32

REGION 33
Figure 17, continued
Figure 17, continued

REGION 38

REGION 40

[Graphs showing energy spectra with peaks labeled AL, SI, BI, and CU]
Figure 18. EDS spectra obtained from altered region in host microcline shown in Plate XLVII—a) Region 42, b) Region 43, c) Region 44, d) Region 45, e) Region 46, f) Region 47, g) Region 48, h) Region 49, i) Region 50.
Figure 18, continued

REGION 43

REGION 44

INTENSITY

ENERGY (keV)

0.00 2.00 4.00 6.00 8.00 10.00
Figure 18, continued

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**REGION 45**

**REGION 46**
Figure 18, continued

REGION 48

REGION 50

h

i
Figure 19. EDS spectra obtained from various twin textures in microcline—a) Region 11-1106, b) Region 12-1106, c) Region 13-1106, d) Region 14-1106, e) Region 15-1106, f) Region 16-1106.
Figure 19, continued

**REGION 12-1106**

**REGION 13-1108**

**ENERGY (KeV)**
Figure 19, continued

REGION 14-1108

REGION 15-1108

ENERGY (KeV)
Figure 19, continued

ENERGY (KeV)
Figure 20. EDS spectra obtained from areas containing Type IV “nuclei” and cryptoperthitic albite—
a) Region 17-1106, b) Region 18-1106, c) Region 19-1106, d) Region 20-1106, e) Region 21-1106.
Figure 20, continued

**REGION 1B-140S**

Energy (KeV)

**REGION 19-110B**

Energy (KeV)
Figure 20, continued

REGION 20-1108

ENERGY (KeV)

REGION 21-1108

ENERGY (KeV)
APPENDIX B

ALGORITHM FOR CALCULATING TILTS REQUIRED TO BRING A ZONE AXIS PARALLEL TO THE ELECTRON BEAM
This algorithm was developed to expedite orienting thinned, single crystal specimens in a TEM fitted with a double-tilt goniometer specimen stage. It is often necessary to accurately orient a TEM specimen to determine crystallographic relationships within and between mineral phases, and for defect analysis. These relationships require knowledge of the location of crystallographic directions and planes in the sample. Specific reflecting vectors (g=hkl) are used in defect analysis; the location of zone-axes which contain these must be determined. Electron-beam-time is expensive and thinned crystal specimens are often beam-sensitive. Zone axes are located by tilting the goniometer stage, and this must be done as quickly as possible. This is often difficult especially for crystals with low symmetry which contain numerous closely-spaced zone axes that produce diffraction patterns that are similar.

The algorithm developed for the present study requires experimental identification of two zone axes. Following this the tilts necessary to bring any other zone axis parallel to the electron beam can be calculated. The routine is general and works for any symmetry. The author developed the routine for use with microcline, a triclinic mineral with pseudomonoclinic cell parameters. The program was developed early in the course of the TEM study of Spruce Pine perthites, when the author was not familiar with the diffraction patterns obtained from the feldspars. The routine was tested for an isometric crystal (stainless steel) using tilt data obtained from Cindy Wise Barnicki, and for a tetragonal crystal using tilt data obtained from Dan Evans. Both of these workers are former graduate students in the Department of Materials Sciences and Engineering, The Ohio State University. The data they supplied, as well as the data for microcline, was obtained from the JEOL 200CX TEM located in the Central Electron Optics Facility, The Ohio State University.

The work of Busing and Levy (1967), who developed an algorithm from four-circle X-ray diffractometers was considered, and a matrix inversion routine from the
program MATOP (Boisen and Gibbs, 1985) was used in the program written for the present work. Five lines of input, in the form of a data file, are required. The first line contains $a$, $b$, $c$, $\alpha$, $\beta$, and $\gamma$, separated by blanks. These estimated cell parameters are required to calculate the metric tensor, $G$, the inverse metric tensor, $G^{-1}$, a Cartesian basis tensor, $C$, and the inverse of the Cartesian basis tensor, $C^{-1}$. The second line of input contains $T_1$ for zone 1, $T_2$ for zone 1, $T_1$ for zone 2, and $T_2$ for zone 2 which are the tilt angles (in degrees) required to orient two experimentally observed zone axes parallel to the electron beam. $T_1$ is defined as a rotation about a horizontal axis extending to the right of the TEM operator. $T_2$ is defined as a rotation about a horizontal axis pointing toward the observer, and forms a right angle with $T_1$. Tilt angles are in degrees, and a counterclockwise (ccw) rotation is taken as positive. The electron beam is vertical and is perpendicular to the plane containing $T_1$ and $T_2$. Together the three axes define a Cartesian coordinate system in the TEM. In this coordinate system, the $T_1$ axis is defined as [010], the $T_2$ axis is defined as [100], and the electron beam parallels [001], to form a right-handed system. Figure 21 is a sketch showing the geometry of the instrument coordinate system.

![Figure 21. Geometry of the instrument coordinate system.](image)
The third line of input contains \( u, v, \) and \( w \) of experimentally observed zone 1, in the form of integers separated by blanks. The fourth line contains \( u, v, \) and \( w \) of experimentally observed zone 2, integers separated by blanks. The fifth line contains \( u, v, \) and \( w \) of the desired zone axis in the form of integers, separated by blanks.

Two lines of reasoning were used in the development of the algorithm. First, the natural coordinate system for a crystal (not necessarily Cartesian) can also be transformed to a Cartesian basis, and the geometrical relationships between these two crystal coordinate systems do not change when the crystal is rotated. Second, this crystal Cartesian basis that is fixed with respect to the natural coordinate system of the crystal can be rotated into coincidence with the Cartesian basis of the TEM. Once the Cartesian basis of the crystal has been established from tilt data for any two experimentally observed zone-axes, the orientation of any other zone axis is theoretically fixed, and one should be able to calculate the tilt angles about \( T_1 \) and \( T_2 \) necessary to orient the desired zone axis parallel to the electron beam.

First, a matrix is set up for each experimentally determined tilt angle required for the two observed zone axes. These are denoted \( T_1 \) and \( T_2 \) for each zone for a total of four tilt matrices. Let \( \theta \) and \( \phi \) define the rotation angles (in degrees) about the axes \( T_1 \) and \( T_2 \), respectively. Referring to the diagram in Figure 21, the form of the \( T_1 \) and \( T_2 \) rotation matrices is:

\[
\begin{bmatrix}
\cos \theta & 0 & \sin \theta \\
0 & 1 & 0 \\
-\sin \theta & 0 & \cos \theta
\end{bmatrix} = T_1; \quad
\begin{bmatrix}
1 & 0 & 0 \\
0 & \cos \phi & -\sin \phi \\
0 & \sin \phi & \cos \phi
\end{bmatrix} = T_2
\]
Next, a subroutine multiplies matrices T1 and T2 for each zone axis to obtain a rotation matrix for each, denoted ROT. Because the electron beam parallels vector $\mathbf{k}_1$ of the TEM Cartesian coordinate system (Figure 21), ROT re-orients a zone axis vector such that it parallels [001]. Therefore:

$$\text{ROT}\begin{bmatrix} x \\ y \\ z \end{bmatrix} = \begin{bmatrix} 0 \\ 0 \\ 1 \end{bmatrix}$$ (4)

The vector xyz in (4) represents the $x$, $y$, and $z$ coordinates of an experimentally observed zone axis in terms of the TEM cartesian coordinate system. This zone axis may have any orientation depending on how the specimen was prepared and placed in the TEM specimen stage. To establish the crystal coordinate system in terms of the TEM basis, the values $x$, $y$ and $z$ for two zone axes are determined by computing the inverse of both respective rotation matrices, denoted $\text{ROT}^{-1}$. Both sides of equation (4) are multiplied by $\text{ROT}^{-1}$ to obtain:

$$\begin{bmatrix} x \\ y \\ z \end{bmatrix} = \text{ROT}^{-1}\begin{bmatrix} 0 \\ 0 \\ 1 \end{bmatrix}$$ (5)

Finally, the complete coordinate system of the crystal is defined by computing the cross product of the two vectors containing $x$, $y$, and $z$ for each zone axis, giving an hkl vector perpendicular to the plane containing the two zone axes. These three vectors are made unit vectors, and are designated: $[xyz]$zone 1, $[xyz]$zone 2, and $[xyz](hkl)$.

Next, the two observed zone axes and the plane perpendicular to them are defined in terms of a standard crystal Cartesian system: $[[100], [010], [001]]$. This is done by multiplying the two [uvw] directions by $C^{-1}$ and by multiplying the (hkl) plane by C. The
angles between these three transformed vectors and each standard Cartesian axis are determined by taking three dot products, for a total of nine angles. For example, the first set of three angles is calculated by taking the dot products of $[uvw]$ for zone 1 (transformed to standard Cartesian coordinates) and the standard axes $[100]$, $[010]$, and $[001]$. The cosines of these three angles define a $3 \times 1$ column vector $b_1$. $b_2$ and $b_3$ are similarly calculated by taking the dot products of $[uvw]$ for zone 2 and $(hkl)$ with the standard Cartesian axes.

Because in any given Cartesian system the angles between specific vectors will not change, a Cartesian system that relates crystal Cartesian to microscope Cartesian can now be determined. Let $i_2$, $j_2$ and $k_2$ denote this final Cartesian system. The $xyz$ coordinates of this system are calculated using the following equations:

$$A \begin{bmatrix} x \\ y \\ z \end{bmatrix} = b_1 \quad (6)$$
$$A \begin{bmatrix} x \\ y \\ z \end{bmatrix} = b_2 \quad (7)$$
$$A \begin{bmatrix} x \\ y \\ z \end{bmatrix} = b_3 \quad (8)$$

where $A$ is a matrix whose rows contain the coordinates of zone 1, zone 2, and $hkl$ in terms of the microscope coordinate system: $([xyz]_{zone \ 1}$, $[xyz]_{zone \ 2}$, and $[xyz](hkl)$) determined previously. The angles between the vectors of a crystal in standard orientation are the same as the angles between these same vectors in a crystal whose orientation is not standard (such as a typical TEM sample), so the real $xyz$ coordinates of
the vectors in the TEM sample are the row vectors in A. The xyz's for \(i_2, j_2, \text{ and } k_2\) are solved using:

\[
Ax = b
\]  

(9)

with \(x\) = a vector containing the xyz's for \(i_2, j_2, \text{ or } k_2\).

Equation (9) is solved by inverting the A-matrix, designated \(A^{-1}\):

\[
x = A^{-1}b
\]  

(10)

This is performed three times to solve for the xyz coordinates of \(i_2, j_2, \text{ and } k_2\). These become the column vectors for the orientation matrix, T. Any xyz for a desired zone is found by multiplying T by the desired zone-axis [uvw]. Tilts are extracted from the new xyz coordinates, obtained from:

\[
\begin{bmatrix}
x \\
y \\
z
\end{bmatrix} = T
\begin{bmatrix}
u \\
v \\
w
\end{bmatrix}
\]

(11)

The required tilts are obtained by analyzing the new x, y, and z coordinates for the desired zone axis obtained from equation (11). For instance, the tilt matrix for T2 describes a rotation, \(\phi\), about the [100] axis of the TEM. The action of this rotation places the desired zone axis vector in the \(i_1, k_1\) plane of the instrument coordinate system (Figure 22). The resultant zone axis vector coordinates are therefore in the form of
The matrix equation describing the geometry of a T2 tilt is:

\[
\begin{bmatrix}
1 & 0 & 0 \\
0 & \cos \phi & -\sin \phi \\
0 & \sin \phi & \cos \phi
\end{bmatrix}
\begin{bmatrix}
x \\
y \\
z
\end{bmatrix}
= 
\begin{bmatrix}
x \\
y \\
z'
\end{bmatrix}
\tag{12}
\]

From the second row of matrix equation (12), one obtains the equality:

\[
\phi = \arctan \frac{y}{z}
\tag{13}
\]

Once the T2 angle, \( \phi \), has been solved it can be substituted into equation (14), which is obtained from the third row of (12):

\[
z' = y \{ \sin (\phi) \} + z \{ \cos (\phi) \}
\tag{14}
\]

Finally, the T1 angle, \( \theta \), is solved with:

\[
\theta = \arctan \frac{x}{z'}
\tag{15}
\]
The geometry of the action of tilts $T_2$ and $T_1$ on the desired zone axis is given below in figures 22 and 23.

Tables 4, 5, and 6 include observed and calculated tilt data for isometric, tetragonal, and monoclinic crystals. The FORTRAN computer code and sample input and output files follow these tables. A brief discussion of the calculations and the sources of tilt data for isometric and tetragonal crystals are discussed above in Chapter V.

Figure 22. Rotation about $T_2$ brings vector into $i_1$, $k_1$ plane.

Figure 23. Rotation of $T_1$ brings $[x0z']$ vector parallel to $k_1$. 
Table 4. Observed and calculated tilt data for an isometric crystal.

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<tr>
<th>zone 1</th>
<th>T1 (z1)</th>
<th>T2 (z1)</th>
<th>zone 2</th>
<th>T1 (z2)</th>
<th>T2 (z2)</th>
<th>zone 3</th>
<th>T1 (z3)</th>
<th>T2 (z3)</th>
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<td>0 0 1</td>
<td>-12.7</td>
<td>-6.5</td>
<td>1 1 2</td>
<td>22.7</td>
<td>-12.5</td>
<td>0 1 1</td>
<td>13.3</td>
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<td></td>
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<tr>
<td>obs.</td>
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<td>13.3</td>
<td>-40.8</td>
<td>0 0 1</td>
<td>36.2</td>
<td>-2.5</td>
<td>-1 1 1</td>
<td>-18.5</td>
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<td></td>
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<td></td>
</tr>
<tr>
<td>obs.</td>
<td>0 1 1</td>
<td>13.3</td>
<td>-40.8</td>
<td>0 0 1</td>
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<td>1 1 1</td>
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<td></td>
</tr>
<tr>
<td>obs.</td>
<td>1 1 2</td>
<td>-10.5</td>
<td>15.8</td>
<td>1 1 1</td>
<td>-23.2</td>
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</tr>
<tr>
<td>obs.</td>
<td>0 1 1</td>
<td>0.7</td>
<td>-18.8</td>
<td>0 0 1</td>
<td>-27.8</td>
<td>14.8</td>
<td>1 2 1</td>
<td>30.2</td>
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<td>calc.</td>
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</table>
Table 5. Observed and calculated tilt data for a tetragonal crystal.

<table>
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<th>zone 1</th>
<th>T1 (z1)</th>
<th>T2 (z1)</th>
<th>zone 2</th>
<th>T1 (z2)</th>
<th>T2 (z2)</th>
<th>zone 3</th>
<th>T1 (z3)</th>
<th>T2 (z3)</th>
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<tbody>
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<td>obs.</td>
<td>-3 3 1</td>
<td>21.5</td>
<td>-2.3</td>
<td>0 1 0</td>
<td>-30.5</td>
<td>-6.3</td>
<td>-1 5 1</td>
<td>-31.0</td>
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<tr>
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</tr>
<tr>
<td>obs.</td>
<td>-3 3 1</td>
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<td>-2.3</td>
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<td>-6.3</td>
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</tr>
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<td>obs.</td>
<td>-5 3 -1</td>
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<td>-13.3</td>
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<td>-30.5</td>
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<td>-13.3</td>
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<td>obs.</td>
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</table>
Table 6. Observed and calculated tilt data for a PW-series microcline (pseudomonoclinic).

Monoclinic* :  $a = 8.56$  $b = 12.96$  $c = 7.21$  $\alpha = 90$.  $\beta = 115.83$  $\gamma = 90$

Crystal PW3, Spruce Pine, N.C.

<table>
<thead>
<tr>
<th>Zone 1</th>
<th>T1 (Z1)</th>
<th>T2 (Z2)</th>
<th>Zone 2</th>
<th>T1 (Z2)</th>
<th>T2 (Z2)</th>
<th>Zone 3</th>
<th>T1 (Z3)</th>
<th>T2 (Z3)</th>
</tr>
</thead>
<tbody>
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<td>0 1 2</td>
<td>12.0</td>
<td>12.3</td>
<td>2 0 3</td>
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<td>12.3</td>
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Table 6, continued

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Table 6, continued

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