

MICROTEXTURAL AND POWDER-DIFFRACTION STUDY OF ANALCIME PHENOCRYSTS IN VOLCANIC ROCKS OF THE CROWSNEST FORMATION, SOUTHERN ALBERTA, CANADA

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ABSTRACT

We present results of powder X-ray-diffraction and microstructural studies of analcime from the volcanic rocks of the Crowsnest Formation, Alberta, Canada. The powder X-ray diffractogram of only one of the analcime samples contains a peak inconsistent with the cubic symmetry that has been attributed to conversion from leucite. We re-evaluate this criterion, and suggest that primary igneous analcime may be cubic as well; caution thus is required in deducing paragenesis from crystal symmetry. A more compelling argument for the primary nature of the analcime is that fresh fracture-surfaces are clean and planar, the characteristics suggested by previous workers to be indicative of crystallization from a melt or fluid. The fracture surfaces lack the porous microstructure previously attributed to the conversion of leucite to analcime.

Keywords: analcime, microtextures, Crowsnest Formation, Alberta.

SOMMAIRE

Nous présentons les résultats d'études en diffraction X (méthode des poudres) et microstructurales de l'analcime des roches volcaniques de la formation de Crowsnest, en Alberta, Canada. Un des trois échantillons contient une raie en violation de la symétrie cubique, antérieurement attribuée à une transformation de l'analcime à partir de la leucite. Nous ré-évaluons la validité de ce critère, et nous préférons l'hypothèse que la symétrie cubique pourrait aussi caractériser l'analcime ignée primaire. Il est donc nécessaire d'utiliser ce critère paragénétique avec circonspection. Il semble plus sûr de se fier sur la microstructure des phénocristaux. Les surfaces de fractures sont propres et planaires dans le cas de l'analcime fraîche et primaire, indication d'une croissance à partir d'un bain fondu ou bien d'une phase fluide. Les surfaces de fractures sont dépourvues des microstructures poreuses attribuées dans la littérature à la transformation de la leucite en analcime.

(Traduit par la Rédaction)

Mots-clés: analcime, microtextures, Formation de Crowsnest, Alberta.

INTRODUCTION

The origin of analcime "phenocrysts" in the volcanic rocks of the Cretaceous Crowsnest Formation has been controversial for over a century. There are two competing hypotheses. Analcime may have crystallized from a trachytic melt as a primary magmatic mineral, or it may have been produced by ion exchange from pre-existing leucite. The first hypothesis is based on textural and field observations (MacKenzie 1915, Pearce 1970, 1993, Peterson *et al.* 1997) and on experimental studies (Peters *et al.* 1966, Liou 1971, Kim & Burley 1971, Roux & Hamilton 1976). The second is equally permissible, based both on observations of leucite par-

tially converted into analcime in natural samples (Luhr & Giannetti 1987) and experimental ion-exchange studies (Gupta & Fyfe 1975, Taylor & MacKenzie 1975). The debate has been ongoing since the first detailed study of MacKenzie (1914), who proposed that the analcime is primary. In less than a year came the first proposal of a replacive origin of the analcime (Pirsson 1915) and the reply by MacKenzie (1915), with the final sentence "With the facts of the original paper in mind, and their further explanation here given, the primary nature of the analcime of the Crowsnest volcanics can not [*sic*] be open to any reasonable doubt." (MacKenzie 1915, p. 574). Despite this statement, the controversy has yet to be resolved. More recent contri-

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butions to the debate include those of Pearce (1970) (primary), Ferguson & Edgar (1978) (primary), Karlsson & Clayton (1991) (secondary), and Peterson *et al.* (1997) (primary).

As illustrated by the ongoing debate [*e.g.*, Pearce (1993) *versus* Karlsson & Clayton (1993)], the current evidence is permissive of either origin. In light of recent work on crystal structures, degree of Si–Al order, and phase transitions in leucite and analcime, we re-evaluate the criterion used by Gottardi & Galli (1985) that analcime formed by ion exchange of leucite should have a disordered Si–Al framework, and therefore be cubic, hence optically isotropic and lacking specific peaks in the powder X-ray diffractogram. In support of this idea, synthetic analcime produced by ion exchange of leucite was reported to be cubic (Line *et al.* 1996).

We also examine microstructures of freshly fractured surfaces, using the approach and criteria of Putnis *et al.* (1994). These authors examined microtextures observable on freshly fractured surfaces of analcime by scanning electron microscopy (SEM). They found distinctively different microtextures in hydrothermally crystallized analcime (H-type, crystallized from a fluid) compared to those in X-type analcime, formed by experimental or natural ion-exchange. The fracture surfaces of the hydrothermal analcime have smooth, planar surfaces; in contrast, fracture surfaces of the X-type analcime have a porous microtexture. Putnis *et al.* (1994) suggested that analcime grown from a melt should have planar surfaces analogous to those of the H-type analcime.

EXPERIMENTAL METHODS

In the more friable units of the Crowsnest Volcanic Suite, it is possible to pluck the analcime phenocrysts, which can be >3 cm in diameter, out of the matrix by hand. The samples studied here were collected in the summer of 2002 during fieldwork for the second author's B.Sc. thesis research. They were sampled from outcrops in the Pipeline Road locality of Peterson *et al.* (1997), which is located just southwest of Coleman, Alberta.

Two intact phenocrysts of analcime, 7 and 9 mm in diameter, and a fractured half of a crystal 9.5 mm in diameter, were selected for this study. All three are honey-brown in color. Pearce (1993) previously described the brown-colored analcime as the freshest, with red, orange, and green varieties produced by alteration. The crystals were broken up such that the largest fragments measure ~2 mm in diameter. The fragments were ultrasonically cleaned in deionized water for 10 minutes, and then rinsed with fresh deionized water. The ultrasonic cleaning was repeated twice, followed by rinsing the fragments with ethanol and drying in a drying oven for 30 minutes at 75°C.

A portion of each crystal was ground in an agate mortar and pestle under ethanol, and fragments were

extracted for an examination in immersion oils with a petrographic microscope. Another portion of each crystal was further ground in an agate mortar and pestle under ethanol and mounted on a quartz plate as a slurry in acetone. The powder X-ray diffractogram for each was obtained with a Rigaku Geigerflex diffractometer at 40 kV and 30 mA using CoK α radiation. The scan was run from 2° to 90° 2 θ . The data were processed using Jade6 software (MDI Corp.).

Selected fragments were mounted on a standard SEM stub with double-sided tape, coated with gold, and examined on a JEOL 6301-FE scanning electron microscope (SEM) with an accelerating voltage of 5 kV.

RESULTS

Under the petrographic microscope, the grains of all three crystals of analcime are optically isotropic, in agreement with the observation of Pearce (1993) that the brown Crowsnest analcime is isotropic.

The powder X-ray diffractograms for each of the analcime crystals (Fig. 1) can be indexed on a cubic cell, when compared to calculated patterns (*e.g.*, PDF 71–0811) that account for peaks with relative intensities less than 1%. One sample has a peak at the appropriate location ($d = 6.90 \text{ \AA}$) to be the 200 reflection, which is not consistent with the *Ia3d* symmetry. That peak has been cited by previous investigators as the hallmark of non-cubic symmetry in analcime (Knowles *et al.* 1965, Gottardi & Galli 1985). The relative intensity of this peak in our diffractogram is low (0.6%). By comparison, the 200 peak of the orthorhombic analcime listed on ICDD PDF19–1180 has a relative intensity of 2%. This difference, and the low intensity in our diffractogram, poses the question of the reliability of using the 200 peak as being diagnostic of non-cubic character. To investigate this issue further, we used XPOW (Downs *et al.* 1993) to produce powder XRD patterns from data on the analcime structure in the American Mineralogist Crystal Structure Database (Downs & Hall-Wallace 2003). The structures from Mazzi & Galli (1978) produce diffractograms with 200 peaks having relative intensities ranging from 0.15 to 4.26% for the five *I4₁/acd* structures and from 0.55 to 0.91% for the two *Ibca* structures. To ensure that we did not overlook a peak of even lower intensity, we reran our other two samples at a smaller step-scan and longer dwell-time to ensure that no peak exists at $d \approx 6.90 \text{ \AA}$. No peak was present in this region in either diffractogram. We conclude, therefore, that these two samples of analcime are cubic, whereas the third is potentially non-cubic. Given the optically isotropic nature of all three samples, the inferred symmetry of this third sample may be questionable.

Examination with the SEM revealed that the outer surface of the analcime crystals is etched and partially dissolved (Fig. 2). The porous microtexture characteristic of analcime formed by ion exchange and hydration

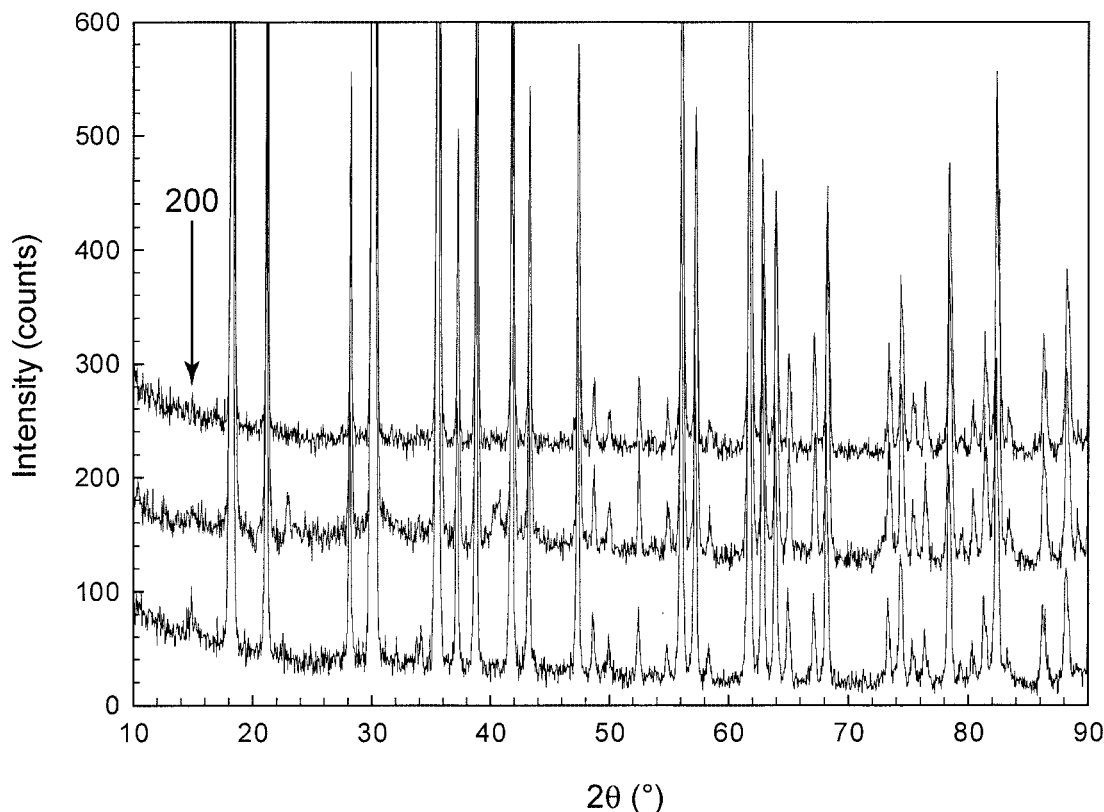


FIG. 1. Powder X-ray diffractograms of analcime from the Crowsnest Volcanic Suite. To expand the scale, the portion of the diffractograms at $2\theta < 10^\circ$ are omitted, as it does not contain any peaks. The baselines for the upper two diffractograms are offset for clarity. The intensity scale has been magnified to show the low-intensity peaks; the maximum intensity in the three scans (from top to bottom) is 4870, 7300, and 9383 counts, respectively. Arrow indicates the location of peaks inconsistent with cubic symmetry. Peaks at 22.9 and $40.6^\circ 2\theta$ in the middle scan are tentatively attributed to chloritoid.

of leucite (Figs. 1b, c of Putnis *et al.* 1994) was not observed, either on these weathered faces or on freshly fractured surfaces (Figs. 3a, b). The clean, planar surfaces we observe are similar to those observed by Putnis *et al.* (1994) for analcime that crystallized from hydrothermal solutions (their Fig. 1a).

DISCUSSION

The X-ray results for our two cubic samples disagree with previous work on analcime from the Crowsnest Volcanic Suite. Gottardi & Galli (1985) cited the study of Aurisicchio *et al.* (1975), who reported optically anisotropic analcime and XRD patterns that contain the 200 peak. These investigators studied red-colored analcime, and their results may be reconciled with those of the present study by noting that Pearce (1993) observed that altered analcime is commonly red and optically anisotropic.

In order to evaluate the significance of the symmetry of these analcime samples as a monitor of the mode of their formation, some background on the Gottardi & Galli (1985) criterion is required. These authors reference Galli *et al.* (1978), who based their argument on the "fully disordered (Si, Al)-distribution in leucite". They stated that all samples of analcime were generated by Na-for-K exchange of leucite should be fully disordered, and hence cubic. This statement assumes that non-cubic analcime must have an ordered Si-Al distribution. The ordering of Si and Al in both leucite and analcime has been the topic of a number of studies since that of Galli *et al.* (1978).

At high temperature, leucite is cubic (space group $Ia\bar{3}d$; Peacor 1968). It contains a single tetrahedrally coordinated site, which requires a long-range disordered distribution of Si and Al. At lower temperatures ($<665^\circ\text{C}$), leucite adopts a tetragonal structure (space group $I4_1/a$, Mazzi *et al.* 1976), after passing through an intermediate phase with space group $I4_1/acd$ (Heaney

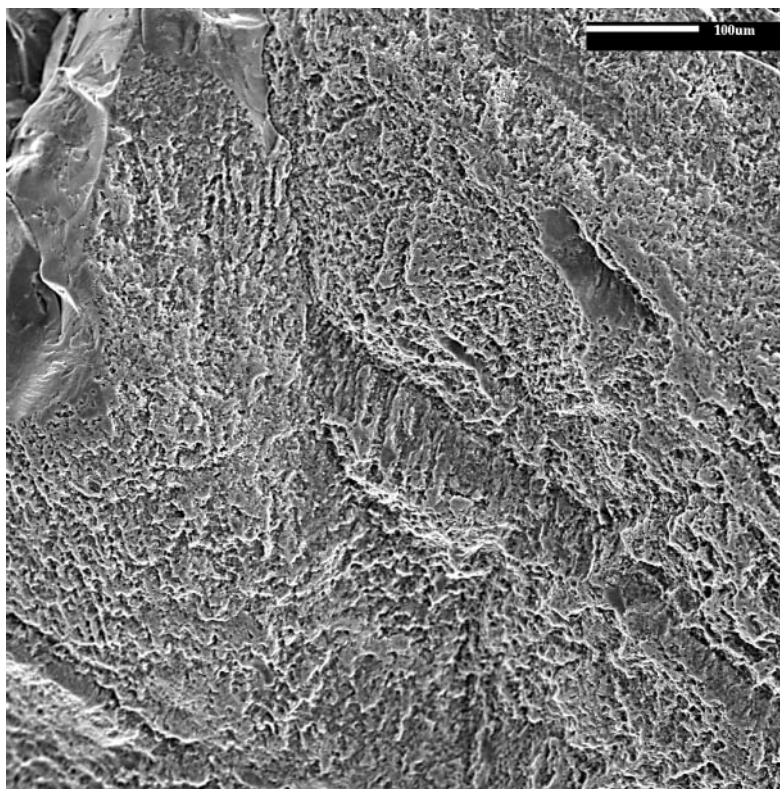


Fig. 2. SEM photograph of exterior (weathered) surface of Crowsnest analcime, showing etched and partially dissolved surface. Scale bar: 100 μm .

& Veblen 1990, and references therein). The low-temperature structure has three symmetrically distinct tetrahedral sites, which raises the possibility that long-range Si–Al ordering occurs during the transition, or may be the reason for the transition (Hatch *et al.* 1990). The rapid kinetics of the transition (Peacor 1968, Palmer & Salje 1990), the results of lattice-energy calculations (Dove *et al.* 1993), and those of neutron-diffraction studies (Palmer *et al.* 1997) are all consistent with the transition being a purely displacive one (Dove 1997), with Si–Al ordering playing no role.

Accepting this conclusion, igneous leucite would crystallize with a cubic structure, with long-range Si–Al disorder, and transform to tetragonal leucite upon cooling, maintaining its Si–Al disorder. The transformation to analcime will not change the state of order, producing analcime with long-range Si–Al disorder, but not necessarily of cubic symmetry. The symmetry of the analcime would depend on whether the ion exchange and hydration distorts the tetrahedral framework to produce a reversion to the cubic structure or not. This argument assumes that the transition between cubic and

lower-symmetry forms of analcime is displacive, as it is in leucite. In support of this assumption, Teertstra *et al.* (1994) argued that decreased symmetry in analcime resulted from “a slight, systematic rotation of the tetrahedra during growth, which does not involve ordering or even change during cooling from a higher-temperature, more symmetrical phase.” Further support comes from Hovis *et al.* (2002), who suggested that at room temperature, analcime is near the transition temperature for a displacive transformation from a cubic to a tetragonal structure.

Must analcime with space group *Ia3d* necessarily be produced by ion exchange from a leucite precursor? It seems reasonable that analcime crystallizing from a melt would have a disordered Si–Al arrangement, and hence would be cubic. Not all samples of cubic analcime are igneous or derived from ion-exchanged leucite; analcime of hydrothermal and sedimentary origin commonly are cubic and lack long-range Si–Al order (*e.g.*, Neuhoff *et al.* 2003, 2004). Neuhoff *et al.* (2003) did find significant short-range order, resulting from Al–Al avoidance (*cf.* Teertstra *et al.* 1994).

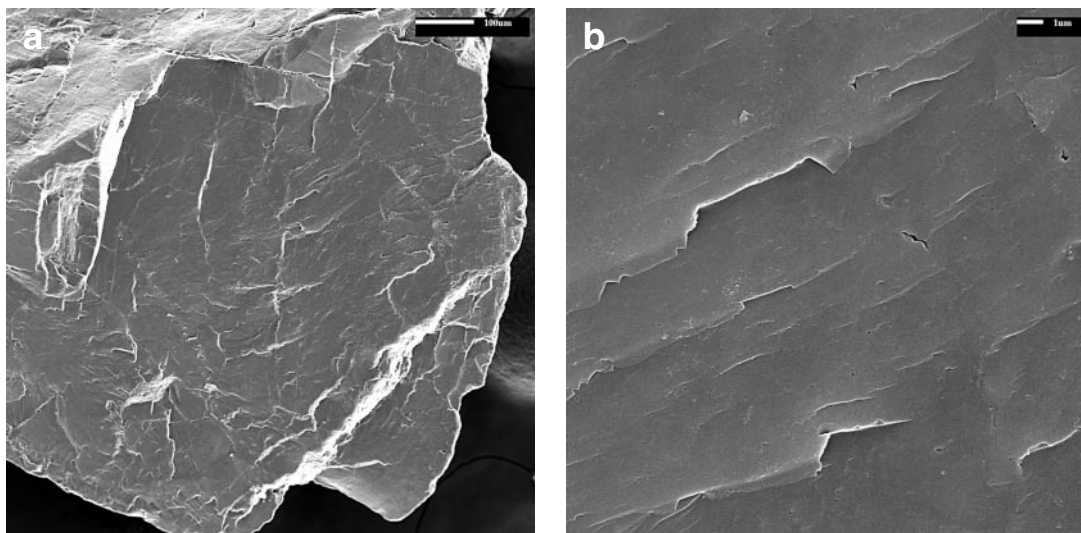


FIG. 3. SEM photographs of freshly fractured surface at low (a) and high (b) magnification, showing a smooth, planar surface. Scale bars: 100 μm in (a) and 1 μm in (b).

If we consider the parageneses of non-cubic analcime reported to date in the literature, none appear to be igneous. Mazzi & Galli (1978) refined the crystal structures of seven samples of non-cubic analcime from single-crystal X-ray-diffraction data. All of these samples are from localities where the analcime forms a crust on or fills vugs in altered volcanic rock (Mazzi & Galli 1978, King 2001). The non-cubic sample of Kohn *et al.* (1995) (*cf.* discussion in Neuhoff *et al.* 2004) is from Monteccio, Maggiore, Italy, where the analcime is found in vugs in basaltic blocks (Brigatti *et al.* 1997, <http://www.mindat.org/loc-29986.html>, <http://www.a-m.de/englisch/lexikon/mineral/geruestsilicate/analcimbild1.html>). The tetragonal ($I4_1/acd$) analcime from Newfoundland studied by Papezik & Elias (1980) was found as "...locally forming encrustations on the walls of joints and fractures..." in Late Precambrian felsic pyroclastic rocks. The authors interpreted the analcime to have formed by precipitation from solution at low temperature. Akizuki (1981) showed that isotropic and anisotropic sectors coexist in hydrothermal analcime.

We therefore contend that igneous analcime need not be non-cubic, in disagreement with Gottardi & Galli (1985), and that by analogy with leucite, analcime from higher-temperature parageneses should be cubic. Any non-cubic nature in igneous analcime would result from small amounts of solid solution that increase the temperature of a displacive cubic-tetragonal transition (Hovis *et al.* 2002).

To provide insight into the origin of the analcime, we focus on the microstructures revealed with the SEM, which are consistent with the analcime precipitating

from a solution. The next issue is whether the solution from which the analcime crystallized was magmatic, or hydrothermal. Several lines of evidence argue against a hydrothermal origin. First, Peterson *et al.* (1997) interpreted the values of $\delta^{18}\text{O}$ (9.4‰) for pristine ("gem-quality") Crowsnest analcime to be reasonable for a magmatic origin for lower-crust melts. They also found that the values of $\delta^{18}\text{O}$ increase systematically with degree of alteration. These authors discussed the higher values (13.5–14.2‰) reported by Karlsson & Clayton (1991), and suggested that their samples were altered or even secondary. Second, the analcime crystals are found in a variety of volcanic rocks (trachyte, analcimite), some arguably lavas, others clearly pyroclastic in origin (Adair & Burwash 1996). They appear to be phenocrysts in hand sample and thin section; if they were hydrothermal in origin, it is puzzling that they do not form in veins. We cannot rule out, however, the possibility that the microporous texture produced from ion-exchange formation of analcime from leucite could be eradicated by recrystallization or annealing at low-grade metamorphic conditions, which the Crowsnest Formation has experienced (Bégin *et al.* 1995). These authors estimated temperatures of metamorphism of 180–280°C and pressures of 1.5–3 kbar. One of the analcime crystals converted from leucite studied by Putnis *et al.* (1994) was experimentally altered in hydrothermal solution at 150°C. This sample showed the microporous texture discussed above.

We conclude that the microstructural evidence adds to the weight of evidence favoring the hypothesis that analcime in the Crowsnest volcanic suite is primary and

truly phenocrystic. This leaves us with the puzzle of determining the conditions at which analcime, esseneite-rich clinopyroxene, K-feldspar, and titanian andradite could crystallize from the parental magmas. In this context, it is worth noting that the high ferric iron contents of the garnet and clinopyroxene may be essential to understand how analcime, a hydrous zeolite, could precipitate from a melt accompanying anhydrous ferromagnesian minerals, rather than biotite or amphibole. With increasing $f(\text{O}_2)$, biotite decomposes to sanidine + hematite or magnetite + fluid (Wones & Eugster 1965). Amphibole stability is likewise dependent on $f(\text{O}_2)$: ferropargasite $[\text{NaCa}_2\text{Fe}^{2+}_4\text{AlSi}_6\text{Al}_2\text{O}_{22}(\text{OH})_2]$ decomposes with increasing $f(\text{O}_2)$ to assemblages containing andradite-rich garnet (Gilbert 1966). The absence of hydrous ferromagnesian minerals in the Crowsnest Volcanic Suite may well be a natural consequence of crystallization under oxidizing conditions.

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