Cathodoluminescence of coexisting plagioclases, Boehls Butte anorthosite: CL activators and fluid flow paths

CLAUDIA I. MORA
Department of Geological Sciences, University of Tennessee, Knoxville, Tennessee 37996, U.S.A.

KARL RAMSEYER
Geologisches Institut, Universität Bern, Baltzerstrasse 1, 3012 Bern, Switzerland

ABSTRACT

Cathodoluminescence (CL) petrography and luminescence spectra of coexisting plagioclases from the regionally metamorphosed Boehls Butte anorthosite permit rapid visual distinction of complex plagioclase intergrowths and fine-scale, retrograde fluid flow features that are nearly invisible in transmitted light. Calcic plagioclase (An$_{97-99}$) exhibits bright yellow luminescence, whereas intermediate plagioclase (An$_{39-45}$) is green. Luminescence spectra indicate that red emissions are sensitive to anorthite content, shifting from 687 to 744 nm over the range An$_{97-99}$–An$_{39-45}$. Green peak positions are less sensitive, with shorter wavelength emissions in anorthite (540–548 nm), compared with coexisting andesine (549–561 nm). The Mn$^{2+}$ contents correlate well with the intensities of the peak at 540–560 nm, suggesting that Mn$^{2+}$ is an effective CL activator in plagioclase at levels as low as 19 ppm. Fe$^{2+}$ acts as neither a sensitive activator nor important quencher. Very weak Fe$^{3+}$-activated peaks (680–745 nm) but variable Fe$^{3+}$ abundance (23–2504 ppm) suggest that most Fe$^{3+}$ is present in a minute, coexisting phase such as hematite. Anorthite inclusions in andesine megacrysts have significantly different CL peak positions and intensities than granoblastic, polygonal anorthite forming thin laminae in the anorthosite. Plagioclase occurring in association with the laminae shows CL evidence of dissolution and replacement and of grain fragmentation accommodating dilation. The laminae are inferred to have been foci of retrograde fluid circulation through the anorthosite that resulted from rapid uplift of the Boehls Butte region after the peak of regional metamorphism.

INTRODUCTION

Cathodoluminescence (CL; electron-excited luminescence) has been used for more than 20 yr to examine compositional variations, replacement, and growth phenomena in sedimentary rocks (cf. Marshall, 1988; Barker and Kopp, 1991). The majority of these studies have employed qualitative evaluation of CL colors and petrographic analysis of compositional zonation or mineral intergrowths that may be exclusively visible in CL. Much additional information can be gained by spectral analysis of the CL. Spectral analysis permits identification of the activator elements, resolution of complex spectra (those containing more than one activator element), and quantitative comparison of CL in different samples.

In this study, we examine the CL petrography and luminescence spectra of plagioclases from the regionally metamorphosed Boehls Butte anorthosite, northern Idaho. This unusual anorthosite contains two or three compositionally distinct, coexisting plagioclases. The origin of the coexisting plagioclases is controversial, but may be due to shear differentiation of a single, igneous plagioclase (Juras, 1974) or a complex history of intrusion, assimilation, and metasomatic alteration of the anorthosite (Nord, 1973; Goldberg, 1983; Hietanen, 1963, 1984).

Plagioclases formed under different conditions may show different CL emission characteristics, depending on trace element uptake during growth or recrystallization. Careful textural description of the anorthosite, combined with CL spectral analysis, may therefore help to constrain the origin or postcrystallization history of the unusual feldspar assemblage. To help interpret the CL emissions, we have identified luminescence activators by combining trace-element analysis and measurement of the luminescence spectra.

The CL activators in many rock-forming minerals are sensitive to recrystallization and fluid-rock interaction, thus CL petrography has largely unexplored potential to identify fluid flow through metamorphic rocks. Recent studies have used CL in calcite to illuminate prograde metamorphic processes (Yardley and Lloyd, 1989) and retrograde metamorphic processes (Morrison and Valley, 1988; Mora and Valley, 1991) and to distinguish finescale structures, such as healed microcracks, that are not typically visible with transmitted light (Smith and Stenstrom, 1965; Sprunt and Nur, 1979; Padovani et al., 1982). CL petrography may be particularly useful to identify fluid pathways through relatively nonreactive or monomineralic rock types, such as quartzites and anorthosite. We have examined the anorthosite for CL zo-
nation, alteration, and microstructures that may indicate flow paths of prograde or retrograde metamorphic fluids through the anorthosite body.

THE BOEHLS BUTTE ANORTHOSITE

The samples described here were collected in the regionally metamorphosed, Proterozoic Boehls Butte anorthosite. The anorthosite is exposed in an uplifted block, surrounded by regionally metamorphosed metasediments of the Belt Supergroup and Cretaceous-Tertiary plutons of the Idaho batholith (Fig. 1). At least two episodes of regional metamorphism have affected the anorthosite, with late Cretaceous M2 metamorphism of the upper amphibolite facies (700–720 °C, 8–10 kbar) followed by nearly isothermal decompression accompanying rapid, early Tertiary uplift of the batholith (cf. Grover and Rice, 1988; Ziegler and Rice, 1991). Stable isotope compositions in the anorthosite and surrounding metasediments indicate that the study area was locally affected by retrograde hydrothermal or meteoric alteration (Mora and Valley, 1988, 1991).

The Boehls Butte anorthosite contains various combinations of two or three plagioclases that are compositionally distinct: andesine (An₃₅₋₄₈), labradorite (An₀₋₃₉), and bytownite-anorthite (An₉₂₋₉₉). Representative plagioclase compositions are given in Table 1. For this study, we examined two common examples of the textural varieties of anorthosite that are distinguished in the Boehls Butte area. The first is massive anorthosite characterized by andesine megacrysts (up to 20 mm) containing inclusions (≤0.2 mm in length) of bytownite-anorthite (Fig. 2A). The host crystal and calcic inclusions are sometimes in the same crystallographic orientation. Aggregates of 0.1–0.2 mm, granoblastic, polygonal anorthite crystals occur in thin layers (laminae, up to 3 mm wide) throughout the rock (Figs. 2A, 2C). A third plagioclase, An₉₀₋₆₉, is found in a small number of massive anorthosite samples occurring as twinned or untwinned crystals of 2–5 mm. The second example is sheared anorthosite, characterized by grain-size reduction of massive anorthosite assemblages; few megacrysts remain in these samples (Fig. 2G). More complete petrographic and compositional details of the anorthosite are given elsewhere (Hietanen, 1963; Nord, 1973; Walker and Mora, 1990).

Plagioclase (+ quartz) veins (10–30 cm in diameter) locally intrude anorthosite in the study area. These veins contain a single coarse-grained plagioclase (An₉₀) and are probably associated with Cretaceous-Tertiary plutonism in the general area. We have examined the CL of one plagioclase-rich vein to compare it with anorthosite samples, which are inferred to have a significantly different origin and geologic history.

METHODS

Plagioclase was examined on an improved CL microscope (Ramseyer et al., 1989), with an energy of 30 keV and a beam-current density of 0.3 μA/mm². Luminescence photomicrographs were recorded on Ektachrome 400 color transparency film, developed at 800 ASA. Luminescence colors are described according to their appearance under visual observation. The CL spectra between 370 and 860 nm (10 nm resolution) were recorded from a 200-μm spot using a PTI model 01-001 monochromator equipped with a Hamamatsu R928 photomultiplier. The monochromator was linked to the CL microscope by a flexible optic fiber bundle. The spectra were corrected for the spectral response of the instrument, which has been previously determined by means of a calibrated tungsten lamp. Peak intensity is given as a function of photons emitted at a given wavelength.

Quantitative analyses of major elements in plagioclase were made on a Cameca SX-50 electron microprobe at the University of Tennessee with an accelerating potential of 15 kV and a beam current of 15 nA. The mole fraction of anorthite is reported in Table 1. The low concentrations of CL activators in plagioclase, as well as the Fe³⁺/Fe²⁺ ratios, cannot be accurately measured on the electron microprobe. Therefore, separates of plagioclases having compositions of An ≥ 90 or An ≤ 50 were made...
TABLE 1. Plagioclase compositions and peak positions and intensities of cathodoluminescence emissions, Boehls Butte anorthosite

<table>
<thead>
<tr>
<th>SPL no.</th>
<th>Type*</th>
<th>(X_n)**</th>
<th>Mn (ppm)</th>
<th>Fe(^{2+}) (ppm)</th>
<th>Fe(^{3+}) (ppm)</th>
<th>Position†</th>
<th>Position</th>
<th>Position</th>
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<tr>
<td>M96A</td>
<td>M</td>
<td>40.8</td>
<td>25</td>
<td>942</td>
<td>78</td>
<td>558(4.4)</td>
<td>743(2.8)</td>
<td></td>
</tr>
<tr>
<td>MB31</td>
<td>M</td>
<td>42.6</td>
<td>43</td>
<td>572</td>
<td>589</td>
<td>561(5.3)</td>
<td>735(6.2)</td>
<td></td>
</tr>
<tr>
<td>LGM39</td>
<td>M</td>
<td>39.4</td>
<td>34</td>
<td>688</td>
<td>39</td>
<td>558(5.0)</td>
<td>738(3.4)</td>
<td></td>
</tr>
<tr>
<td>MB45</td>
<td>M</td>
<td>39.1</td>
<td>19</td>
<td>512</td>
<td>40</td>
<td>451(0.8)</td>
<td>730(2.4)</td>
<td></td>
</tr>
<tr>
<td>LGM18C</td>
<td>M</td>
<td>43.8</td>
<td>27</td>
<td>690</td>
<td>23</td>
<td>555(8.3)</td>
<td>743(3.6)</td>
<td></td>
</tr>
<tr>
<td>LGM70</td>
<td>M</td>
<td>94.3</td>
<td>60</td>
<td>1422</td>
<td>1140</td>
<td>550(27.0)</td>
<td>740(3.1)</td>
<td></td>
</tr>
<tr>
<td>MB61</td>
<td>C</td>
<td>28.0</td>
<td>12</td>
<td>290</td>
<td>117</td>
<td>387(7.2)</td>
<td>545(10.5)</td>
<td></td>
</tr>
</tbody>
</table>

Note: Major element composition determined by electron microprobe, trace elements determined by wet chemistry on separates containing all plagioclase types having either \(X_n < 50\) or \(X_n > 90\).

* Plagioclase occurrence where M = megacryst, F-M = fine-medium grained plagioclase without inclusions, I = inclusions in megacryst, L = granoblastic-polygonal grains in laminae, C = single plagioclase phase.

** Values of \(X_n\) are 0.3–0.8 in megacrysts, 0.0–0.1 in calcic plagioclases.

† Positions of CL peaks are given in nanometers and intensities (given in parentheses) are peak height values of the corrected spectra.

Using heavy liquids, and trace amounts of Mn\(^{2+}\), Fe\(^{2+}\), and Fe\(^{3+}\) were analyzed by wet chemistry (J. Husler, analyst). Fe\(^{3+}\) was determined by dissolution and titration, Mn\(^{2+}\) and Fe\(^{2+}\) were determined by atomic absorption. The abundance of Fe\(^{3+}\) was determined by difference.

CATHODOLUMINESCENCE PETROGRAPHY

All plagioclases in the Boehls Butte anorthosite exhibit cathodoluminescence, with the color largely a function of plagioclase composition. Representative CL photomicrographs are shown in Figure 2. Calcic plagioclase (An\(_{97-99}\)) is readily distinguished by its yellow CL color; intermediate plagioclase (An\(_{90-92}\)) has a green to green-yellow color (Fig. 2B). By comparison, a more sodic plagioclase sample (An\(_{93}\)) that occurs in veins intruding the anorthosite (Table 1) has a dull green-blue color. The different colors permit rapid visual distinction of the plagioclase phases, complex plagioclase intergrowths, and compositional zonation.

Zones in CL color are observed in both plagioclase

Fig. 2. Transmitted light (left) and cathodoluminescence (CL; right) photomicrographs of the Boehls Butte anorthosite; all CL photomicrographs taken under identical operating conditions. Scale bar is 500 \(\mu m\); (A), (B) Massive anorthosite (sample MB31) showing green andesine (An\(_{99}\)) megacryst (M) and yellow anorthite (An\(_{99-99}\)) occurring both as inclusions (I) in megacryst and small granoblastic, polygonal grains in lamellar structures (L). Patchy CL colors in the megacryst (arrow) may indicate zoned distribution of CL activators. The grain boundaries between host megacryst and inclusions are sharp, arguing against exsolution and coarsening as a mechanism to produce the coexisting plagioclases. The megacryst appears embayed by anorthite in the lamina, suggesting dissolution along its grain boundary. The CL is slightly zoned within the lamina, having brighter yellow CL toward the center. (C), (D) Megacryst (M) and lamina (L) in massive anorthosite (sample MB31). Note the variability of CL intensity within the lamina; this is not the result of differences in major-element content (Ca, Na) of plagioclase. Note also the CL zonation of plagioclases near the lamina and the very fine, diffuse fractures emanating from the lamina (arrow). (E), (F) Lamina within massive anorthosite (MB45). Note the apparent fragmentation of grain (arrows) within lamina; fragmentation appears to have occurred by dilution, with little rotation of the grain fragments. (G), (H) Complex of CL color distribution within sheared anorthosite (LGM70). The CL of medium-grained, intermediate plagioclase is strongly zoned. Aggregates of granoblastic, polygonal anorthite engulf several large plagioclase grains at the bottom center and appear almost anastomosing in the left portion of the photomicrograph.
Fig. 3. Spectra of cathodoluminescence colors from plagioclase. All spectra are corrected for the instrumental response.

An objective measurement of luminescence color can be made by examining the positions and intensities of peaks in the emission spectra. In general, plagioclase spectra are characterized by three bands: 420-460, 550-570, and 680-750 nm (cf. Marshall, 1988). The CL spectra were measured in the range 370-860 nm (10-nm resolution) for three samples of Boehls Butte anorthosite (Figs. 3a-3c); these spectra were recorded under the same operating conditions and are all directly comparable. The samples show a pronounced peak in the range of 540-570 nm, with minor peaks in the range 387-446 and 690-750 nm (Table 1). The lack of a prominent peak in the range 690-750 nm is notable, as this peak is often strong, or dominant, in terrestrial plagioclases (Sippel and Spencer, 1970; Geake et al., 1971). Weak red emissions in the Boehls Butte plagioclases may reflect relatively low \( f_{\text{o}_2} \) during anorthosite crystallization; weak or absent emissions in the near IR are characteristic of lunar plagioclases and have been ascribed to low \( f_{\text{o}_2} \) during plagioclase crystallization (Geake et al., 1973, 1977).

The main differences observed in the spectra are the intensity of the green peak (540-570 nm) and the position of the red peak (690-750 nm). The relative intensities of the green peaks vary by up to a factor of 20. Mega-
crysts and inclusion-free crystals of intermediate...
composition ($\text{An}_{39-54}$) have the lowest intensity green peaks (M, I in Fig. 3). The highest intensities are measured for fine-grained, calcic plagioclase ($\text{An}_{40-50}$) occurring in the thin laminae (L, Fig. 3). The CL spectrum for vein plagioclase ($\text{An}_{45}$) is clearly different than the anorthosite plagioclase spectra, with almost no green peak and a relatively intense red peak (Fig. 3d). In this sample, a peak at approximately 380 nm was unstable during electron bombardment.

Differences in the color or intensity of CL in plagioclase may be controlled, in part, by plagioclase composition ($X_{an}$, as it is likely that a divalent CL activator such as Mn$^{2+}$ substitutes for Ca$^{2+}$, and Fe$^{3+}$ for tetrahedrally coordinated Al$^{3+}$, in plagioclase (Geake et al., 1971, 1973, 1977; Telfer and Walker, 1975, 1978). The dependence of the red peak position on $X_{an}$ has been previously reported (30-nm range, Sippel and Spencer, 1970; 60-nm, Geake et al., 1973; 100-nm, Telfer and Walker, 1975), although the green peak has been reported to be relatively insensitive to An content ($\pm 10$-nm; Geake et al., 1973). Our study differs from these previous studies in that naturally occurring plagioclases from a single occurrence are examined, rather than a suite of unrelated terrestrial and synthetic samples. Figure 4 shows the relationship of CL wavelength and plagioclase composition in the Boehls Butte plagioclases. Spectral response-corrected data reported by Telfer and Walker (1975) are shown for comparison; peak positions reported by Geake et al. (1973) are uncorrected and therefore cannot be included in this diagram. The data confirm the sensitivity of the red peak to anorthite content, with the peak position ranging from 687 nm for plagioclase of $\text{An} \geq 90$ to 744 nm for $\text{An}_{54}$ (Fig. 4). There is a significant difference between the red peak position in anorthite inclusions (average $\text{An}_{90}$, 701-705 nm) and anorthite in laminae (average $\text{An}_{85}$, 687 nm) that may not only be due to the slight compositional difference. Other factors that can affect the red peak position include the structural state of the plagioclase and the tetrahedral site occupancy of the activator (Telfer and Walker, 1978; Boroznovskaya et al., 1982). The position of the green peak appears to be slightly sensitive to plagioclase composition, with calcic plagioclase associated with shorter wavelength luminescence (540-548 nm) compared with more sodic compositions (549-561 nm, Fig. 4). Thus, increasing anorthite content in plagioclase results in red and green peak shifts toward shorter wavelengths.

The intensity of CL peaks is principally governed by the concentration of the activator element in the plagioclase. Peak intensity is related to the concentration of the activator, up to a maximum concentration of approximately 1 mol% for Mn$^{2+}$ and 1.5 mol% for Fe$^{3+}$ (Telfer and Walker, 1975); above this level of concentration, the activator may become self-quenching. Subtle variations in the activator content, resulting from differences in the environment of formation or recrystallization of the grain, may thus affect the CL color and spectrum. To this extent, it is interesting to note that the intensity of the green peak in granoblastic, polygonal anorthite occurring in laminae is significantly greater than the peak intensity in anorthite inclusions in megacrysts. These samples have Mn$^{2+}$ and Fe$^{3+}$ contents significantly below levels at which self-quenching is predicted (Table 1). Together with differences in the red peak position noted above, the intensity data suggest that the two types of anorthite formed or recrystallized in environments containing significantly different concentrations of activators.

**Cathodoluminescence activators**

Examination of CL spectra in natural and synthetic plagioclases have identified elements that can cause cathodoluminescence in plagioclase; lattice defects resulting from shock damage may also cause CL (Sippel and Spencer, 1970). The following elements behave as CL activators: Ce$^{3+}$ (wavelength = 355 and 490 nm), Eu$^{2+}$ (420, 470 nm), Cu$^{2+}$ (420 nm), Ti$^{4+}$ (450-460 nm; Ti$^{4+}$ may only act to enhance intrinsic CL), Mn$^{2+}$ (550-570 nm), and Fe$^{3+}$ (680-750 nm) (Geake et al., 1971, 1972, 1973; Laud et al., 1971; Mariano et al., 1973; Mariano and Ring, 1975; Telfer and Walker, 1975). It is uncertain whether Fe$^{2+}$ behaves as a CL activator (550-570 nm, Mariano et al., 1973) or quencher element (Geake et al., 1972, 1973). The Mn$^{2+}$, Fe$^{3+}$, and Fe$^{2+}$ contents of Boehls Butte plagioclases are given in Table 1. Intermediate plagioclases ($\text{An} \leq 50$) contain low abundances of Mn$^{2+}$ and moderate amounts of Fe$^{2+}$ ($\leq 60$ ppm and 512-979 ppm, respectively; Table 1). Coexisting calcic plagioclases ($\text{An} \geq 90$; granoblastic, polygonal anorthite and anorthite inclusions in megacrysts were not mechanically separable) contain similar amounts of Fe$^{2+}$ and approximately two times as much Mn$^{2+}$. The Fe$^{3+}$ abundances (23-2504 ppm) and Fe$^{3+}$/Fe$^{2+}$ ratios (0.03-5.3) are much less predictable. Anorthite separates contain four to six times as much
Fe$^{3+}$ as coexisting intermediate plagioclase in massive anorthosite; this factor is 50 times in sheared anorthosite (Table 1). By comparison with the anorthosite samples, vein plagioclase contains less Mn$^{2+}$ and Fe$^{2+}$ (12 and 290 ppm, respectively; Table 1) and has an Fe$^{3+}$/Fe$^{2+}$ ratio of 0.40. The trace element contents are all within the range reported for high-grade metamorphic plagioclases (Smith and Brown, 1988).

There is a strong correlation between Mn$^{2+}$ content and peak intensities at 540–570 nm (Fig. 5); no correlation exists for Fe$^{2+}$. Thus, Mn$^{2+}$ appears to be the dominant CL activator for this peak at concentrations as low as 19 ppm (Table 1), far lower than activation levels previously reported (≥100 ppm, Mariano et al., 1973). The Fe$^{2+}$ content has no apparent influence on the CL color or intensity. The relative intensities of the Fe$^{3+}$-activated red peaks (680–740 nm) in the Boehls Butte samples are small ($I \approx 6.2$; Fig. 5; Table 1), despite Fe$^{3+}$ contents as great as 2504 ppm. In fact, the most intense red peak is observed in vein plagioclase ($I = 10.6$; Fig. 5) containing only 117 ppm Fe$^{3+}$. Because the anorthosite contains significantly less than a self-quenching level of Fe$^{3+}$ (self-quenching at 1–1.5 wt%, Geake et al., 1973; Telfer and Walker, 1975), it is likely that much of the measured Fe$^{3+}$ is not incorporated in the plagioclase structure but is instead present in a phase such as hematite. Large amounts of Fe$^{3+}$ in anorthite (Table 1) may reflect oxide precipitation or plagioclase alteration more than Fe$^{3+}$ substitution for Al$^{3+}$. Anorthite in laminae forms the largest proportion of calcic plagioclase separates, suggesting a direct correlation between Fe$^{3+}$ content and the laminae. For example, sample LGM39 (266 ppm of Fe$^{3+}$; Table 1) contains predominantly megacrystic plagioclase, with few well-defined laminae. By comparison, sample MB31 contains more abundant laminae and Fe$^{3+}$ (2504 ppm). Possible explanations for this result are examined in the following section.

**Retrograde Fluid-rock Interaction**

Any interpretation of the CL phenomena observed in this study must address several points. As a general observation, all of the plagioclases are luminescent, suggesting that all phases formed or were recrystallized at relatively high temperatures because authigenic and low grade metamorphic plagioclase rarely luminesces (Kastner, 1971). Other observations include (1) the occurrence of two distinct modes of anorthite (inclusions and in laminae), having different CL peak positions and intensities, (2) subtle CL zonation and embayment of andesine megacrysts along laminae, (3) variable Fe$^{3+}$ contents, especially in calcic plagioclase separates, and (4) numerous brittle features such as the apparent dilational fragmentation of anorthite grains within laminae, diffuse fractures emanating from laminae, and very fine-scale fractures cross-cutting all phases in the anorthosite. These observations are best explained by retrograde fluid-rock interaction focused along laminae in the anorthosite. Recrystallization of anorthite accompanied fluid flow along these layers, resulting in changes in activator concentrations (peak intensities increased) and possibly changes in structural state (peak positions shifted). Alteration or oxide precipitation along these layers increased the overall abundance of Fe$^{3+}$ in the laminae; possible changes in other trace elements mobilized by the hydrothermal fluid can also be inferred. The timing of fluid flow is inferred from the numerous brittle structures and the concentration of CL changes along the laminae. The rapid, nearly isothermal decompression of the Boehls Butte area from peak metamorphic pressures of 8–10 to approximately 3 kbar (Criss and Fleck, 1987; Grover and Rice, 1988; Ziegler and Rice, 1991) may have resulted in fluid circulation into the anorthosite during retrograde cooling. Stable isotope evidence for local, retrograde meteoric or hydrothermal alteration of Belt Supergroup metasediments and the Boehls Butte anorthosite has been previously reported (Taylor, 1969; Mora and Valley, 1988, 1991). In both cases, alteration was not widespread and pervasive but variably affects the rocks on a scale of millimeters to meters.

The results of this study highlight the usefulness of a detailed cathodoluminescence study to identify evidence...
for fluid-rock interaction that can be virtually invisible during routine petrographic or whole-rock geochemical analysis. Indeed, except for stable isotope studies, retrograde fluid-rock interaction has largely been unrecognized in previous studies of the anorthosite (Hietanen, 1963; Nord, 1973; Juras, 1974; Goldberg, 1983). CL petrography helps to identify fluid pathways and to constrain the scale of fluid-rock interaction; these observations are useful guides to further geochemical studies. Clearly, cathodoluminescence petrography can be a powerful tool in studies of high-grade metamorphic rocks.

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