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Oscillatory zoning of plagioclase: Nomarski interference contrast microscopy of etched polished sections

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Abstract

Nomarski interference contrast microscopy is useful for the study of zoning in calcic plagioclases. Crystals are sectioned, polished, etched in fluoboric acid and coated with a layer of reflecting substance. Images of the surface formed from reflected light using Nomarski interference contrast reveal its relief. Comparison with images formed with crossed polarized transmitted light and plane polarized transmitted and reflected light and with microprobe analyses indicates that surface relief corresponds to Ab/An compositional structure. Zones as thin as 0.5 μm and with compositional contrast of about 1 percent An can be resolved using the method. Optimum etching occurs on polished surfaces which are inclined more than about 40° to the growth zones and the *c* and/or *a* crystallographic axes.

Introduction

Zoned crystals of plagioclase in igneous rocks may preserve a record of their history of growth. The purpose of this article is to describe how Nomarski interference contrast microscopy of etched, polished sections can reveal the zoning textures of plagioclases which are so calcic that ordinary petrographic observations are relatively insensitive. Although familiar to metallurgists and ceramists, the Nomarski method is relatively unfamiliar to geologists and petrographers.

Method

Nomarski interference contrast is a reflected light, beam-splitting technique useful for imaging surface relief (Nomarski, 1954,1955). Plane polarized light is split and resolved by a double-crystal prism such that the phase differences of superposed, laterally shifted wave fronts are resolved. Because the overlapping edges of the superposed wave fronts arising from relief on the specimen have opposite signs, a bright-dark paired shading results which resembles shadow relief. The intensity of monochromatic illumination (J) in the image plane is given by $J = J_0 \sin \Delta\lambda/2$ where J_0 is the maximum brightness achievable and $\Delta\lambda$ is the path difference in fractions of a wavelength. Relief as little as about 500Å is detectable (Nomarski, 1954). Pamphlets further explaining the technique are available from most manufacturers of petrographic microscopes.

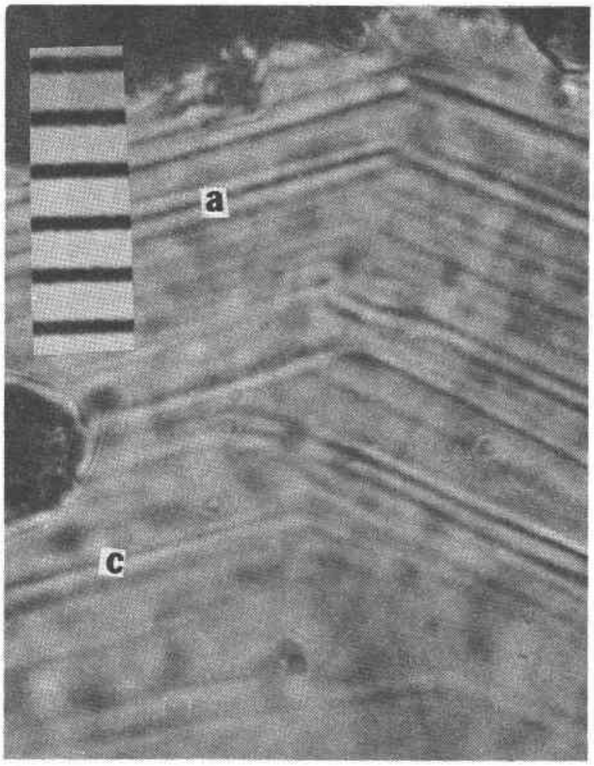

Fig. 1. Transmitted plane polarized light view of the outer portion of a phenocryst of plagioclase erupted from Fuego volcano, Guatemala on October 14, 1974. The scale divisions are 10 microns apart. The irregular outer rim of the phenocryst is visible at the top of the photomicrograph. Individual zones are marked by Becke lines and are 2 to 10 μm thick. The crystal growth zones are ($\bar{2}01$) and ($1\bar{1}0$) (respectively right and left) and they dip inward at angles of 85° to the polished surface.

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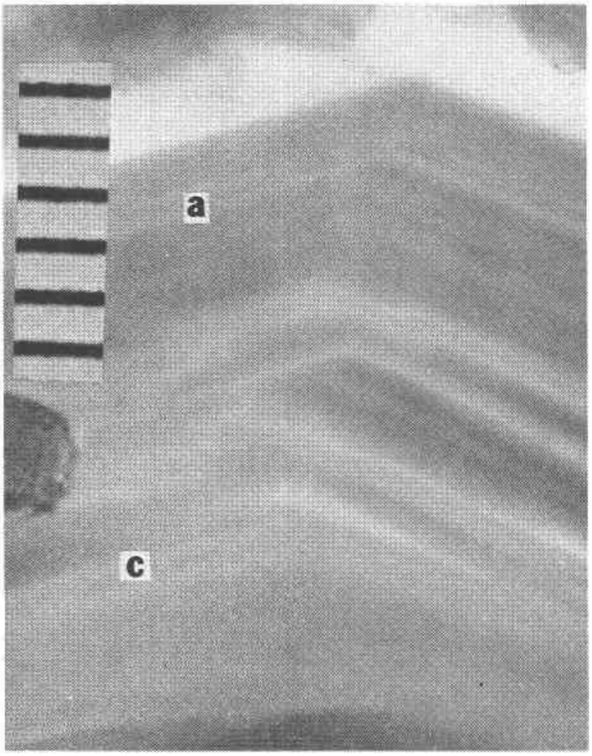

Fig. 2. View in crossed polarized light of the same field as shown in Fig. 1. The sodic rim and other sodic horizons appear relatively bright.

The method relies on etched polished surfaces. Polished sections can be prepared following Moreland (1968). Polished sections etched in concentrated fluoboric (HBF_4) acid for 30 seconds to 2 minutes are suitable. The acid may be quenched by transferring the section directly from the acid into a solution of Na_2CO_3, and subsequently rinsing with water. A vapor-deposited film of highly reflecting carbon, Au–Pd or other substance will increase the reflectivity of the polished surface. A vapor-deposited coat of Au–Pd about 300Å thick is suitable. The coated section is ready for detailed study by reflected light microscopy, Nomarski interference contrast microscopy or by scanning electron microscopy.

Observations and discussion

Universal stage and Nomarski study of 133 haphazardly oriented crystals of An_{85} to An_{93} plagioclase revealed that etched relief on the polished surface is optimized if: (1) the growth zones intersect the plane of polish by an angle of about 40° or greater, and if (2) the *a* or *c* crystallographic axes make an angle of 40° or greater with the plane of polish.

Observations of zoning in plagioclase by various methods are compared in Figures 1 through 6. Figure 1 reveals that oscillatory zones a few microns thick can be resolved by their contrasting refractive indices, if the crystal face is nearly perpendicular to the plane of the section. Zones with the most pronounced Becke lines evidently are those with the greatest contrast in refractive index. Figure 2 shows that variations in extinction angle reveal a zonal structure similar to that evidenced by variations in refractive index shown in Figure 1. The reflected light view of the etched polished surface (Fig. 3) yields a comparable pattern of zones. The resolution is better for light reflected from the surface and reveals numerous zones 1 to 2 μm thick, but the optical contrast is low. Ridges and valleys on the etched polished surface can be identified in reflected light by focusing slightly above the plane of the surface. Because ridges scatter light

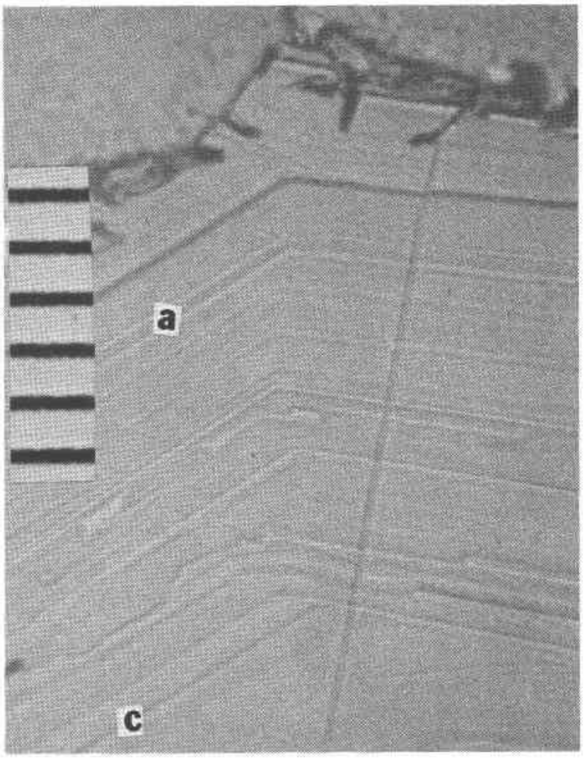

Fig. 3. Reflected light view of the same field as shown in Figs. 1 and 2. The plane of focus of the objective is slightly below the level of the etched polished surface so that light reflected from sodic ridges is converged into bright lines. Note that the same prominent zones appear in Figs. 1, 2, and 3.

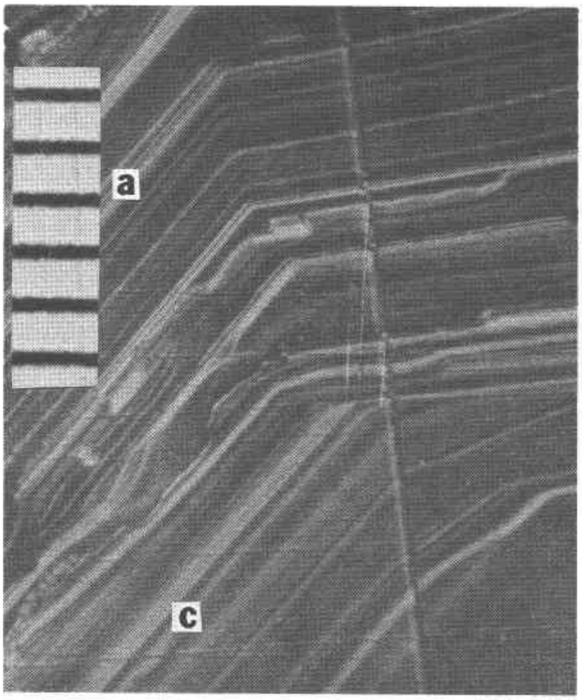

Fig. 4. Nomarski interference contrast view of the same area as shown in Figs. 1–3. The zones evident in Fig. 3 have greater contrast. Some additional zones can be seen, but some are artifacts which arise because the interference technique can yield multiple interference contours on a single inclined surface.

away from their crests, a ridge darkens as the focal plane is moved above the reflecting surface.

A Nomarski interference contrast view is given in Figure 4. It reveals the same features as Figures 1–3. The image in Figure 4 is sharper than those in Figures 1 and 2, and more contrasting than Figure 3. Some features not resolvable on the printed reproductions of Figures 1–3 are resolvable on the printed reproduction of Figure 4. Figure 4 reveals artificial doublings of some zonal boundaries in Figure 3. The crystal illustrated in Figures 1–4 is in a compromise orientation: it is neither optimum for etching nor for extinction angle—optimum orientations for extinction angle variation are different from those for etching. The orientation is useful primarily for demonstrating equivalence of methods.

Scanning electron microscopy (SEM) allows greater magnification than is possible with Nomarski interference microscopy. Figures 5 and 6 compare a high magnification Nomarski interference contrast view with an SEM view. The SEM view, although distorted by the projection angle,

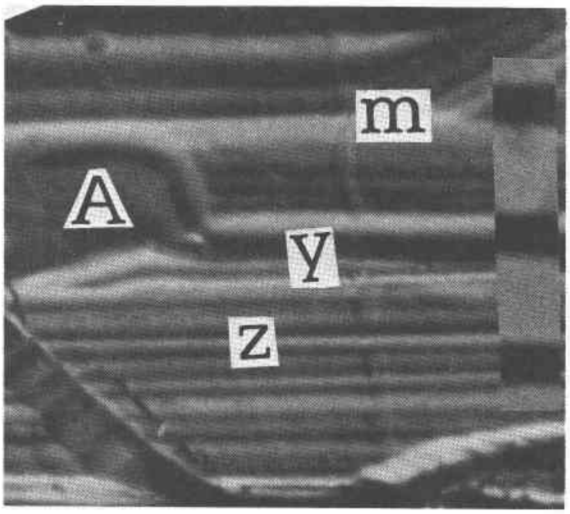

Fig. 5. Nomarski interference contrast view of another etched, polished crystal of plagioclase from Fuego volcano, Guatemala. Individual bright bands m, y, and z and complex reentrant feature A are labeled for reference and comparison with Fig. 6. The text explains how the sinuous scratch passing between m and y reveals the variable relief and related composition of the plagioclase. The scale bars are 10 μm apart and the thinnest zones are about 1 μm thick. The rim of the crystal is directed toward z and y and m.

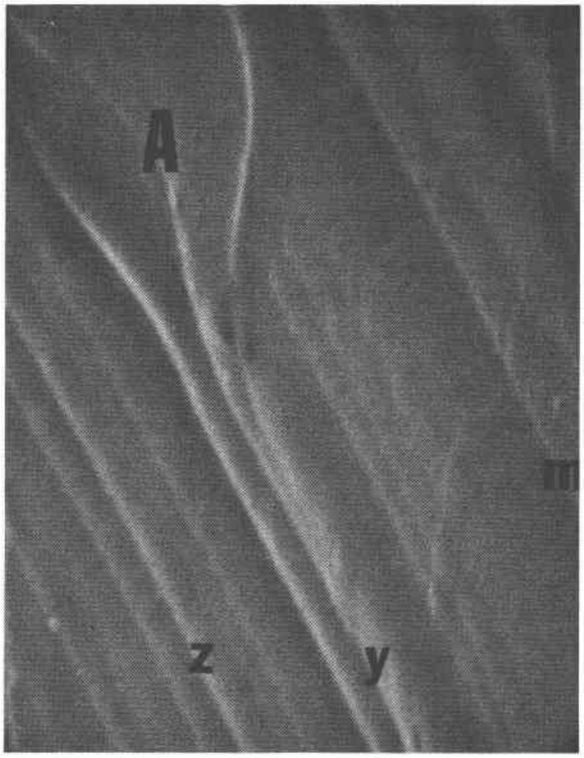

Fig. 6. Scanning electron microscope image of the same area as Fig. 5. The features here are geometrically distorted by projection. The etched, sinuous scratch between y and m is evident. The same zones are visible here as in Fig. 5 although the SEM enlargement is about twice that of Fig. 5.

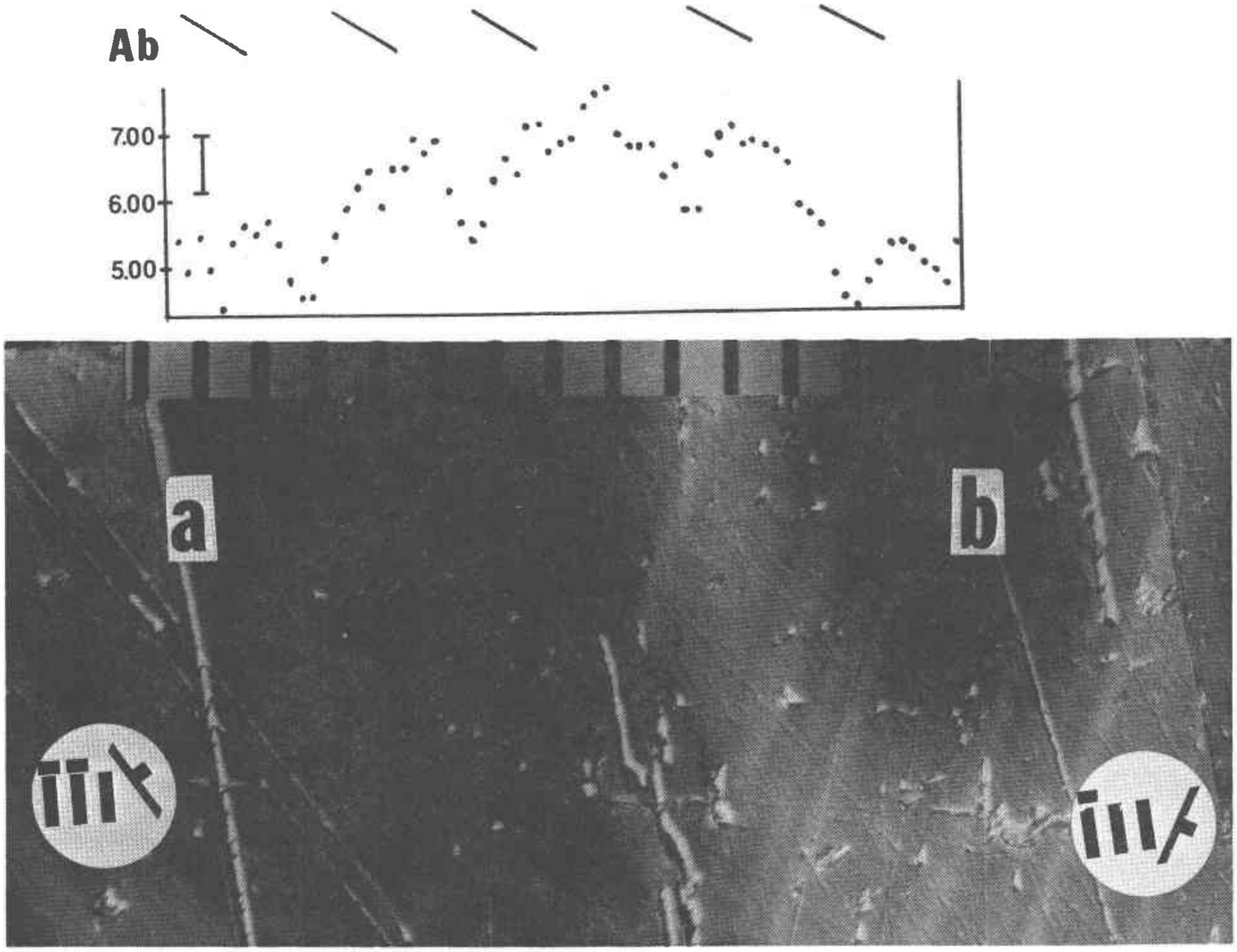

Fig. 7. Compositional profile of a plagioclase crystal from allivalite, Hachijo-Jima. The photograph is positioned so that the microprobe profile (a–b) is almost directly below the point at which the composition is plotted. Compositions were determined on the etched, polished surface by microprobe analysis for Na. The compositions are plotted in terms of mole percent Ab. The length of the bar represents the 2σ uncertainty for a single analysis. The scale divisions are 10 μm apart. The apparent dips of crystal growth zones are indicated above the graph.

reveals the same zones as the Nomarski image. The SEM views have greater magnification, but reveal little or no additional texture, and are more tedious and expensive to obtain. In some cases SEM would allow compositional analysis, but this is difficult with zones as thin as 1 μm. Evidently zones as thin as about 0.5 μm can be resolved by the Nomarski method, if the etched polished surface has optimum relief.

A compositional profile across part of a zoned crystal is compared with a Nomarski image in Figure 7. Most zones differ in composition by less than about 2 percent An. Some zones represented on the Nomarski interference contrast view have compositional contrast of about 1 percent An.

The relation between composition and microscopic appearance is further demonstrated in Figures 6 and 8. Figure 6 illustrates the sinuous trace of an etched scratch as it cuts across zones. The sinuous trace results from the inclined plane of deep structural damage related to the scratch. Where the plane of scratch damage intersects a ridge of plagioclase resistant to the etchant it curves one way, where it intersects a valley it curves the other. The relation between scratch curvature and composition can be calibrated by referring to the displacement of the trace of the scratch at the outer margin of the crystal. Here the composition is known to be about $An_{70} \pm 3$ from microprobe and extinction angle measurements. The interior has an average composition

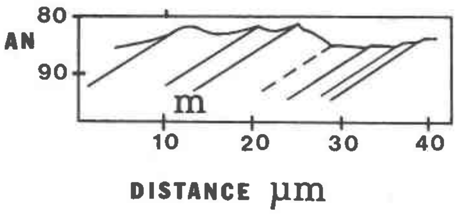

Fig. 8. Tracing of etched scratch across oscillatory zones shown on Fig. 6. The up direction of Fig. 8 is to the right (toward m) in Fig. 6 and represents increasing elevation of the etched surface (total relief about 1 μm). Based on compositional data on other grains, extinction angle measurements on this grain and the displacement of scratches on the sodic rim of this grain, the total compositional variation is less than 6 percent An and lies between An_{82} and An_{88}. The vertical coordinate is an estimate of the compositional significance of the relief on the etched grain. The zones dip inward toward the interior of the grain at 44° which is 32° apparent dip in the section along the scratch. The distance along the scratch is plotted on the abcissa. The aggregate thickness of the 6 complete zones depicted is 9 μm, corrected for geometrical distortion.

of about An_{85}. Assuming that the resistance to etch varies linearly with An content, the trace of the scratch can be rendered as a curve of composition with distance. There may be some rounding of the relief on the etched grain. Consequently, the observed trace probably is not as rough as the actual variation in composition. The calibrated curve of the trace of the scratch reveals that the zones consist of oscillations of etch-resistant, sodic ridges. The surface relief indicated by the sinuous scratch appears to correspond to compositional variations between 0.5 and 4 percent An.

Because the zones evident in the Nomarski views correspond to those defined by variations in Becke line (refractive index), extinction angle and An content, it is concluded that the Nomarski interference contrast method can reveal compositionally based textures of calcic plagioclases.

Acknowledgments

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