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Oscillatory zoning and other microstructures in magmatic olivine and augite: Nomarski interference contrast observations on etched polished surfaces

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Abstract

The Nomarski interference contrast technique is used to enhance the visibility of microtopographic relief on the surfaces of etched, polished sections and etched, polished thin sections. As an outgrowth of A. T. Anderson's application of this technique to plagioclase, we have introduced modifications that permit the study of microstructures in olivine and clinopyroxene.

In thin section, titaniferous augite from an Arizona alkali basalt only faintly displays sector- and oscillatory-zoning, which is probably due to variations in (Al + Ti) content. Nomarski images of HF-etched surfaces of this mineral reveal the zoning with extraordinary clarity, far surpassing in detail the normal thin-section image. In many aspects the oscillatory zonation in the augite appears as fine as that of plagioclase, which it superficially resembles.

Nomarski imaging of HCl-etched surfaces of olivines in basalts from three widely separated localities unexpectedly reveals delicate oscillatory zoning. Whereas some areas of the olivines show fine zoning apparently similar to that in plagioclase and augite, others show distinctive, relatively broad bands alternating with narrow incised troughs. Etch pits probably related to dislocations or inclusions are also developed in several olivines.

Textural features—such as tapering of zones, re-entrants at zone interfaces, and angular unconformities—leave little doubt that, in both clinopyroxene and olivine, the etched oscillatory zones are a reflection of primary growth features. We believe this to be the first documented report of oscillatory zoning in olivine. The nature and origin of the microstructures in this mineral are, as yet, problematic. Differential etching in the olivines could be due to either slight compositional variations or structural differences between zones. In the light of the known high rate of internal diffusion for the major elements Fe and Mg in this mineral, we find the preservation of such delicate growth-related structures remarkable.

Introduction

Etching of mineral surfaces to reveal features that are not otherwise visible is a time-honored but seldom-used technique. Daly (1899) seems to have been the earliest practitioner in the field (see Wegner and Christie (1985) for an historical review). Etching techniques have been used mainly to display secondary features such as dislocation etch pits, exsolution lamellae, and fission tracks, although growth zones have been reported in plagioclase (Grossman et al., 1971), and a possible growth feature in clinopyroxene was reported by Wegner and Christie (1985). The technique we use to etch minerals appears to be simpler than those of Wegner and Christie, and, like Anderson (1983, 1984), we use a Nomarski interference contrast device to enhance subtle features of microtopography that might otherwise be undetectable.

The growth history of magmatic minerals may be preserved in the compositional zoning patterns of individual crystals. Because the distribution coefficients for Fe and Mg are particularly well defined, by experiment or observation, for the ferromagnesian minerals olivine, orthopyroxene, and clinopyroxene (Roeder and Emslie, 1970; Ford et al., 1983), the zoning profiles of these minerals constitute an important record of the crystallization history of a magma (Van Kooten and Buseck, 1978) and, in the case of olivine, of diffusional events during subsequent cooling (Onorato et al., 1978).

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Fig. 1. Nomarski interference contrast images of etched clinopyroxenes. The samples are polished sections of basalt (field no. AZ-81-11), from Oak Creek Canyon, southwest of Flagstaff, Arizona. All photographs have the same scale. Wavy surfaces probably due to solution are indicated by S. Wide outermost rims or mantles representing the last stage of growth are indicated by M. In Figure 1c, c indicates a change in the apparent width of the zones at a sector boundary.

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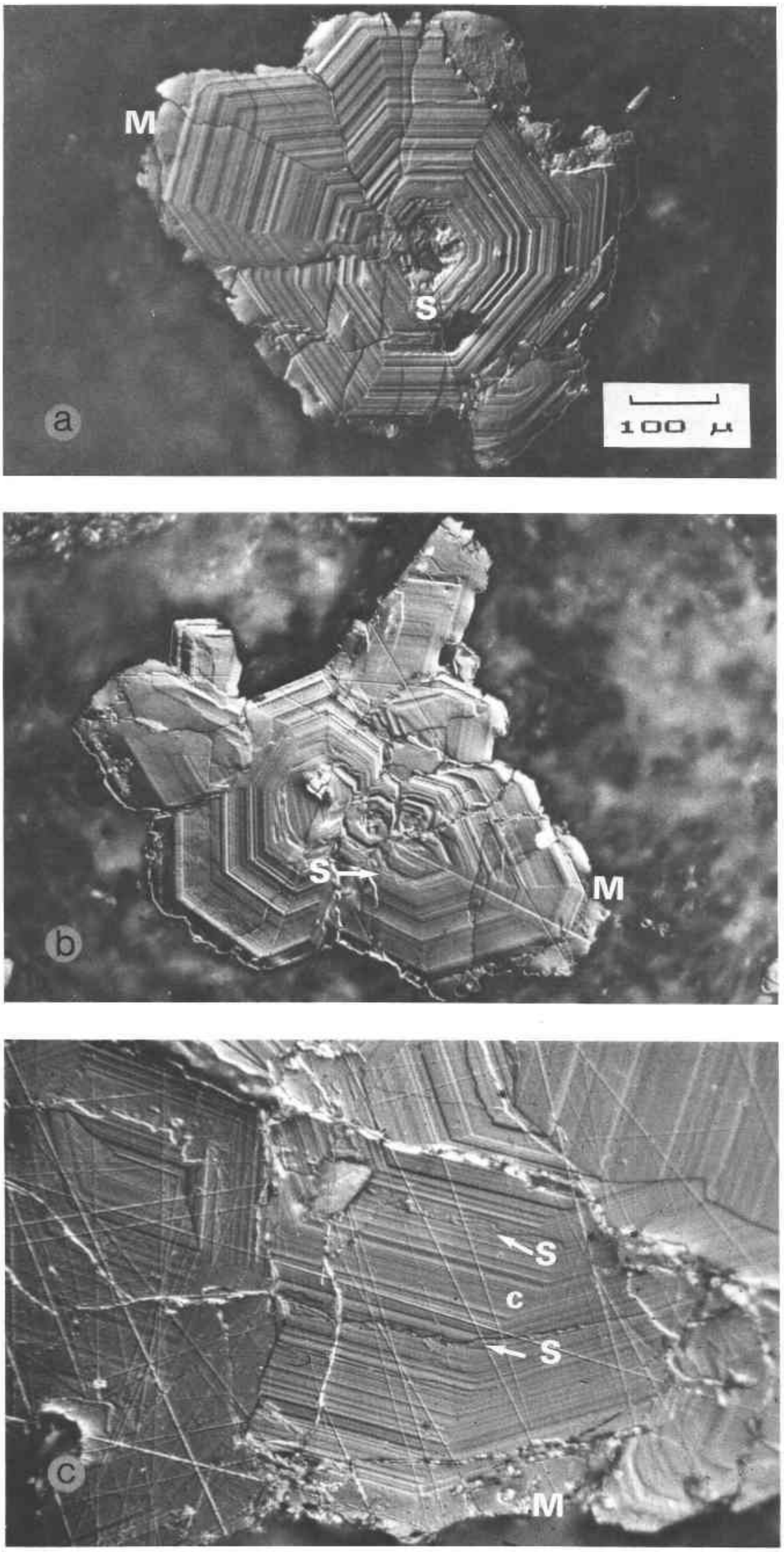
M
S
100 μ
a
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In the present study, several volcanic rocks containing plagioclase, clinopyroxene, and olivine were investigated. We specifically discuss the application of the technique to (1) clinopyroxene in a basalt flow from Oak Creek Canyon, southwest of Flagstaff, Arizona, and (2) olivines in olivine basalt from the Kana-a flow (Sunset Crater), San Francisco volcanic field, Arizona, from the 1868 Mauna Loa flow, Hawaii, and from Olfusvatnsa, southwestern Iceland, and in a shoshonite from the Cordillera Oriental of southeastern Peru. Combined laser-interference microscopy and electron-microprobe analysis of zoning in olivine and orthopyroxene in the latter rock have recently been documented by Kontak et al. (1984).

Our aim herein is to demonstrate the efficacy of the Nomarski-based techniques in the definition of zoning patterns in etched ferromagnesian minerals.

Methods

Nomarski interference contrast microscopy (Nomarski and Weill, 1954; D'Orey, 1970) was used in this study to examine microtopographic details of etched ferromagnesian minerals. Polarized light is split by a Wollaston prism into two beams, which are reflected by the specimen, recombined by the prism, and then passed through a crossed analyzer. Surface irregularities cause phase differences in the reflected wave fronts, producing variations in brightness or color. Different light intensities on either side of an imaged microtopographic feature produce a three-dimensional appearance, as though the specimen were under oblique illumination.

In our technique, polished sections or polished thin sections containing olivine are etched by immersion in hot (45°C), concentrated hydrochloric acid for 10 to 20 min. Sections containing clinopyroxene are etched with concentrated hydrofluoric acid for 2 to 4 min. HF attacks plagioclase and olivine with vigor, corroding and bleaching the polished section, and in polished thin sections, degradation of the mounting medium may occur. Extensive leaching of the rock matrix also results from HF etching. If olivine coexists with pyroxene in a section, it is recommended that HF etching be restricted to selected areas, thereby avoiding destruction of all of the olivine. After etching, the acids are neutralized by immersion in a saturated solution of Na_2CO_3; the polished surfaces are then rinsed with water and dried. The section is finally carbon-coated to increase its reflectivity and to eliminate internal reflections in the crystals.

Although significant relief may be developed between the crystals and matrix in volcanic rocks, the overall relief of individual etched crystals is less than 1 μm, and typical local relief between microscopic zones is less than 0.3 μm. These vertical distances were measured with the Nomarski fringe interferometer and confirmed with a Michelson interferometer attached to a microscope using a HeNe laser as a light source. The agreement between these techniques is within 8% of the combined average (in one case, 0.14 ± 0.1 μm). Because of the double image of the Nomarski technique, we find it difficult to make comparisons over a substantial distance, say from the core to the rim of a crystal. The Michelson has a flat reference wave-form and is thus easier to interpret. For this reason, we confirm all "long distance" comparisons with the Michelson interferometer.

In our studies to date, we have employed the Nomarski interference contrast device manufactured by C. Reichert AG. The electron-microprobe analyses were conducted with an ARL-SEMQ at 15 kV. The Mg and Fe analyses of the olivine were carried out using wavelength-dispersive spectrometers and a sample current of 0.02 to 0.05 μA. The counting time for each analysis was 1 min, and a standard olivine from Hawaii (Queen's University reference number, S-68) was used as a standard. The pyroxene analyses were conducted using an energy-dispersive spectrometer and standards close in composition to the mineral. All intensities were corrected using the procedure of Bence and Albee (1968).

Observations

Clinopyroxene

For investigation of the zonation of a clinopyroxene, we selected augites from an alkaline basalt from Arizona. In polarized light, the subhedral phenocrysts display weak sector zoning and, faintly, fine oscillatory zoning in some orientations. Electron-microprobe analysis of ten zones in one crystal revealed only minor variations in MgO (11.61–14.66 wt%) and FeO (5.84–7.42 wt%). There is an antipathetic relationship between the atomic proportions of (Al + Ti) and (Si + Mg + Fe) except in the marginal zone of the grain. This titaniferous augite (TiO_2 = 1.10–2.72 wt%) is unusually rich in alumina (4.62–9.36 wt%). We tentatively infer that the observed optical zoning is a reflection of variations in (Al + Ti), as documented by numerous previous investigators (e.g., Barton et al., 1982; Grapes, 1975; Downes, 1974). Wegner and Christie (1985) have also cited differences in (Al + Ti) in neighboring zones.

As shown in Figure 1, the Nomarski images reveal with extraordinary detail and clarity both the sector and oscillatory zonation of the augite, far surpassing in resolution the normal thin-section images. The disposition of sector-zone boundaries and the true form of the fine oscillatory zones (most of which are undetectable in thin section) are clarified. In numerous respects, the oscillatory zones, averaging ca. 10 μm in thickness, resemble those figured by Anderson (1984) for plagioclase from Fuego, Guatemala, and those we have found in that mineral from other localities (Nixon et al., 1985). The boundary traces of individual zones are predominantly linear (Fig. 1a), but may be "wavy" or "convolute" (Fig. 1b, area S; Fig. 1c, areas S). Individual zones or zone-clusters also show marked changes in width at sector-zone interfaces (e.g., Fig. 1c, area c). Groups of fine oscillatory zones on one sector surface are continuous with thinner, apparently unzoned, and/or very thin growths on the contiguous surface. As noted by Anderson (1983), the orientations of the lines defining the ends of zones in such intersections reveal the contemporaneity of such distinct growth formats on adjacent crystal faces. Again, as with the Fuego plagioclases, the wide (20–60 μm) outermost zones (M in Figs. 1a, 1b, 1c) on the augites have the appearance of anhedral mantles, which probably developed during final extrusion and quenching of the lava. These overgrowths appear to be gradationally zoned, in the Nomarski images, from the gradation in the degree of etching.

Significant differences with respect to the plagioclases studied both by Anderson (1983) and ourselves include the "patchy" zonation of the apparent core areas of some

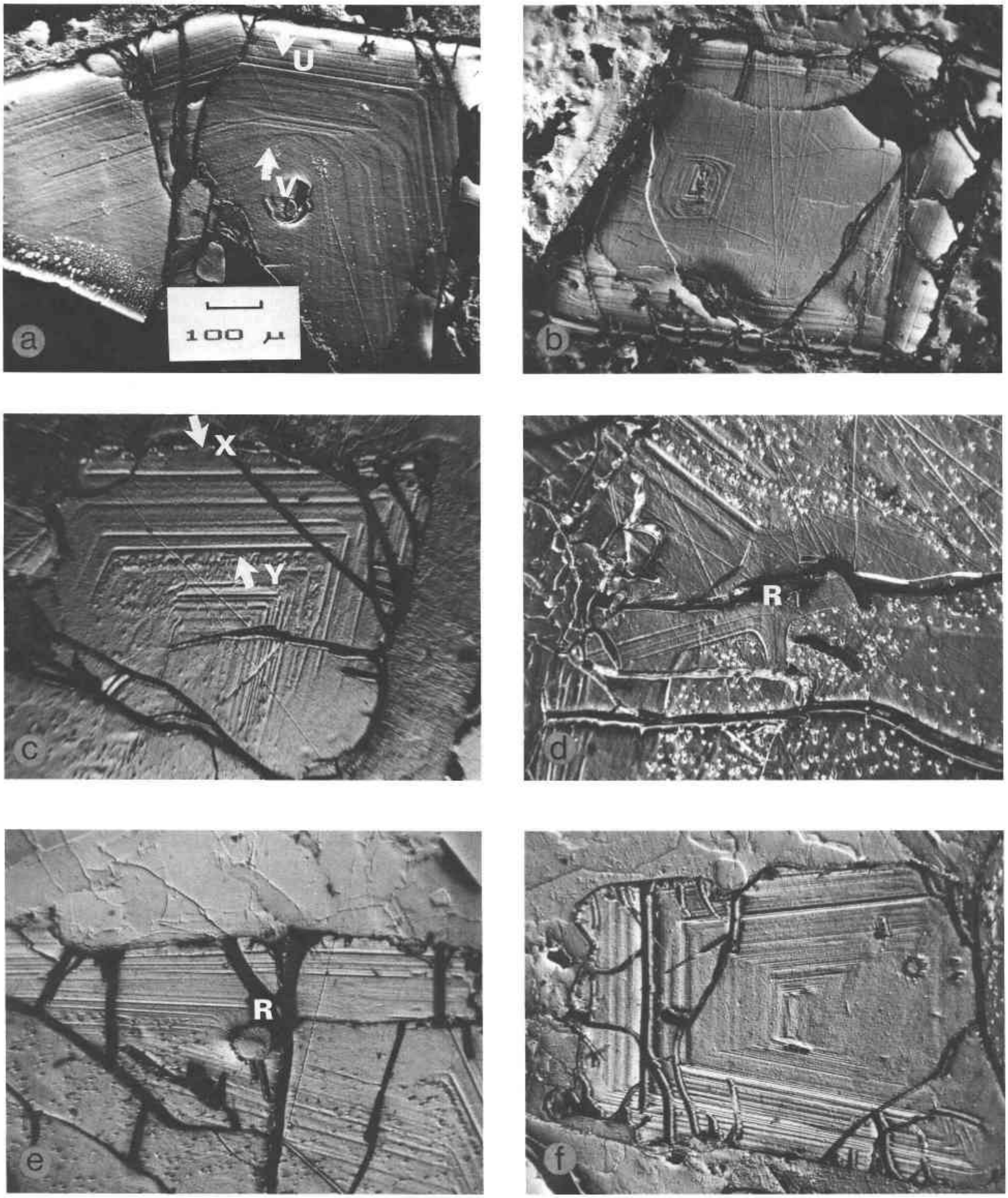

Fig. 2. Nomarski interference contrast images of etched olivines (all photographs have same scale). (a and b) Olivine from Kana-a flow, Arizona. (a) A probe traverse between the arrows (U, V) is presented in Figure 5. (b) Note the apparent oscillatory zoning in the core surrounded by a broad area exhibiting little or no microtophography. (c–f) Olivine from 1868 Mauna Loa flow, Hawaii. (c) A probe traverse between the arrows (X, Y) is presented in Figure 5. Note that the zone near Y has inclusions that are shown as etch pits. (d) Olivine with fine zoning and a re-entrant, R appears to have been deposited on earlier olivine that now exhibits prominent etch pits probably due to dislocations. (e) An inclusion of chromite and glass is centered in a prominent re-entrant, R. (f) Fine-scale oscillatory zoning; the apparently poor zonation on the right-hand side of the crystal is an artifact of specimen orientation.

Fig. 3. SEM image of etched olivine from Arizona (specimen tilt 80°). The area shown is near V in Figure 2a. The prominent east-west crack is visible in Figure 2a approximately 100 μm left of V. The angle of viewing is from the bottom-left and above (compared to Fig. 2a). Note that the zones that are clearly visible in Nomarski imaging (Fig. 2a) are barely visible using the SEM (the boundaries are indicated by the arrows).

of the augites (Fig. 1a), and the less extensive development of marked discontinuities and truncation fractures between zones. Whereas a typical 1-mm plagioclase phenocryst may exhibit as many as 20 wavy or scalloped interzone contacts, the majority of the clinopyroxenes we have observed display only one or two (e.g., S in Figs. 1a, 1b, 1c), and some show none. Such features are reasonably ascribed to resorption (magmatic corrosion) alternating with growth, and it is implied that plagioclase may more readily suffer solution than does augite. The more regular nature of zoning in augite and the lower incidence of resorption, relative to those of apparently cogenetic plagioclase, have been recorded by Boesen (1964).

Many of the augite phenocrysts examined appear to have multiple cores. Because of statistical problems associated with random sections through crystals (see Pearce, 1984, for example), we cannot determine if this is due to irregular growth about a single core or to intergrowth around multiple cores.

We have not yet definitely correlated specific zones on the Nomarski images with composition, because the microprobe analyses were conducted on a flat surface before etching, but it is reasonable to infer that variations in Al [or (Al + Ti)] content are responsible for the differential leaching in the present case, in that there is less variation in Mg/Fe ratio. Etching by HF is likely to be more sensitive to Al/Si variations than to Mg/Fe (A. T. Anderson, written comm., 1985). We have not assessed the applicability of our etch technique to those alkali pyroxenes in which optical zonation is a function of variations in Na + Fe^{3+} (Ericksson, 1985).

Olivine

Whereas fine oscillatory-zoning patterns were anticipated in the augite, finding delicate zonation in etched olivine was unexpected. In Figure 2, we illustrate such zoning in olivine phenocrysts from Quaternary basalts from three widely separated localities.

Oscillatory zoning is well displayed by predominantly fresh olivine phenocrysts from the Kana-a basalt, Arizona (Figs. 2a, 2b), from Mauna Loa, Hawaii (Figs. 2c–2e), and from Iceland. These grains appear wholly homogeneous in thin section. We know of no other published records of comparable features in this mineral, although Wegner and Christie (1985) have reported etch pits and tracks in etched sections. While superficially similar to the oscillatory zones in etched augite and plagioclase, the majority of the olivine zones lack the clear, bipartite, "ridge-and-valley" microtopography shown by those minerals (Anderson, 1984; this study). Instead, the olivine zones generally comprise relatively broad (average 30 μm), flat "mesas" separated by "troughs" (see Figs. 2a, 2c, and, especially, Fig. 4d). This distinctive microtopography is also evident, albeit less clearly, in scanning-electron microscope (SEM) images (Fig. 3). Some crystals (Fig. 2b) display markedly zoned cores and marginal zones, separated by broad areas with reduced microtopography. In detail, some aspects of the augite zonation are here repeated, including tapering of individual zones (Figs. 2a, 2e), abrupt changes in band thickness between contiguous and coeval internal crystal faces (Fig. 2f), and "unconformities" between zones (Fig. 2a).

In many crystals (Fig. 2f), it is evident that the clarity of the zones in etched olivine is markedly affected by the orientation of the crystal relative to the Nomarski prisms (a feature less significant in clinopyroxene owing to its reduced symmetry), and by the inclination of the zones relative to the etched surface. It is also known from the work of Wegner and Christie (1974) that there is a significant variation in etch rate between the different crystal faces of olivine. Although these factors influence the visibility of specific zonal features in this mineral, they clearly do not disguise the overall form of the zoning.

Additional features of the etched olivines, well shown by the Mauna Loa phenocrysts, are marked re-entrants (R) within the crystals (Figs. 2d, 2e). Thus, in Figure 2d, an early-formed olivine with a scabrous etched surface appears to be penetrated by a more smoothly etched layer (generation) that shows "mesa and trough" zoning close

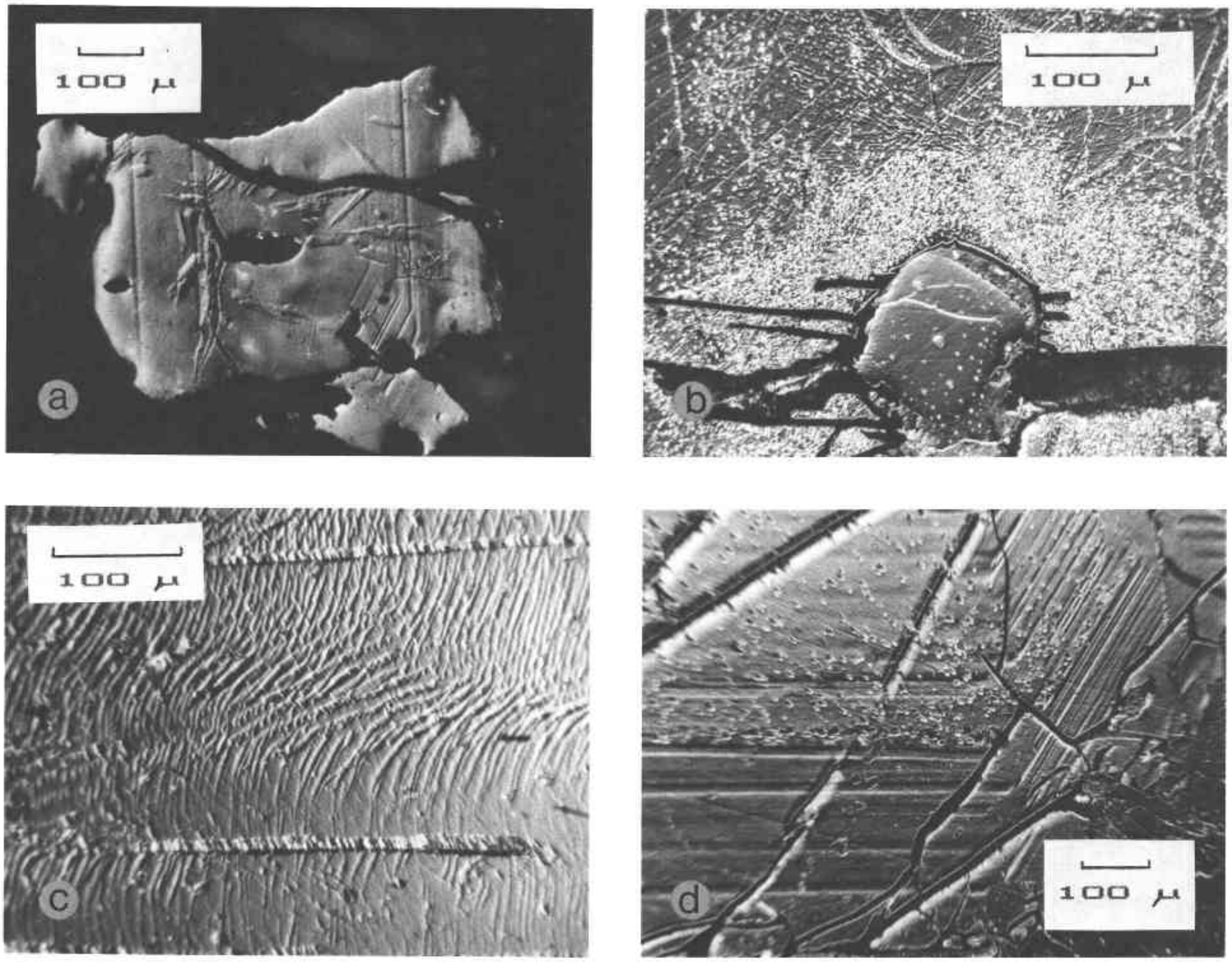

Fig. 4. Nomarski images of etched olivines with structures probably not related to crystal growth. (a) Olivine microphenocryst from a lower Miocene shoshonite, Cerro Moromoroni, southeastern Peru. Note the bladed structures. (b) Olivine phenocryst from Olfusvatnsa, southwestern Iceland, showing a chromite grain surrounded by a dense field of minute etch pits, probably due to dislocations. The concentration of pits in the strained area around the inclusion is consistent with this interpretation. (c) Icelandic olivine showing curvilinear features thought to be dislocations intersected at a shallow angle by the surface of the section. (d) Olivine from 1868 Mauna Lao flow. The concentration of etch pits is toward the core of the crystal. The etch pits, some of which are clearly triangular, are probably due to dislocations rather than to inclusions. In the dislocation-free area, note the troughs incised in an otherwise flat microtopographic region.

to and conformable with the contact with the earlier olivine. This may represent extensive magmatic resorption and infilling by later olivine or a period of growth inhibition caused by an adhered mineral grain (not intersected by the surface of the sample). The latter process is probably exemplified by Figure 2e, in which etched growth laminations are deflected adjacent to a composite chromite-glass inclusion. Figure 2e shows marked discordance between the orientation of the earlier (lower part of photograph) and later laminations.

Other, quite distinctive, microtextures are evident in olivines from several localities. Thus, Figure 4a illustrates an olivine phenocryst from a Peruvian shoshonite. In addition to zoning similar to that described previously, this crystal displays bladed or wedge-shaped features. In some optically homogeneous and unaltered grains (not illustrated), the entire surface is observed to be affected by this feature, which may represent twinning in response to rapid cooling, or simply a different type of etch pit.

A feature shown by most etched olivine grains is the development of minute (ca. 1 μm wide) subequant or elongate features, which are clearly visible in Figures 2c–2e and 4b and in the SEM image of Figure 3. Of these, those with a crudely triangular surface expression are closely comparable to the etch pits of Wegner and Christie (1974, 1985). In addition, linear or curving arrays of minute etch pits are displayed in etched olivine grains (Fig. 4c). In many cases these features are concentrated adjacent to chromite inclusions (Fig. 4b) or fractures (some with serpentine: Fig. 4c), or in annular interval zones broadly concordant to the zonation defined by etching (Fig. 4d). The elongated features, in particular, resemble the dis-

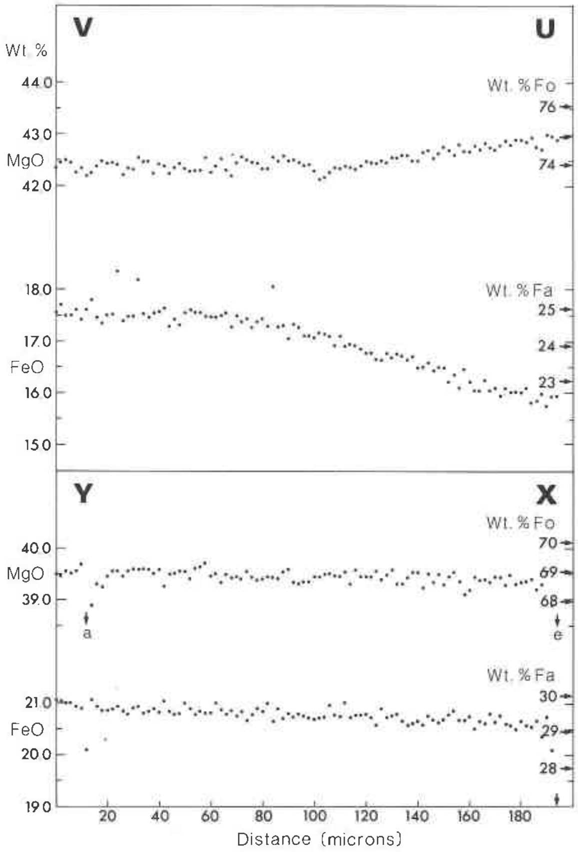

Fig. 5. Electron-probe traverses across two olivines. The scales are in weight percent; the numbers with arrows indicate corresponding weight percent of Fo and Fa. Upper plot: The V-U traverse is from an Arizona olivine (see Fig. 2a). The three anomalously high points on the Fe traverse are probably due to inclusions of magnetite. Whereas there is a suggestion of oscillatory zoning in this graph, the observed variation is within two standard deviations of the mean. Note that the crystal is in part reversely zoned. See text for explanation. Lower plot: The Y-X traverse is from a Hawaiian olivine. Note the essentially flat composition profile with a slight suggestion of oscillatory zoning, which again falls within the error of the analysis. Point *a* is a crack and point *e* is the edge of the crystal.

locations described by several authors (e.g., Green, 1976) in transmission-electron microscope studies of olivine.

We interpret the apparent oscillatory zoning in the olivines as a record, or at least a palimpsest, of multiple stages in crystal growth. However, electron-microprobe analysis (upper plot, Fig. 5) of the Arizona olivine crystal illustrated in Figure 2a reveals that it displays only very minor compositional Mg/Fe zoning (<2 wt% Fo). An inner zone with relatively constant composition is surrounded by a domain in which Mg shows a minor outward enrichment and then by a narrow, more ferroan rim (the latter not shown). There appears to be no relationship between the extent of microtopography in the Nomarski image and the composition of the olivine. It is possible that the etch zones may represent discrete compositional differences; however, if so, they must differ by less than 0.5 wt% Fo (upper plot, Fig. 5). The minor degree of reverse zoning in this crystal is matched by the inner parts of some of the olivine phenocrysts studied by Van Kooten and Buseck (1978) from Crater 160, also in the San Francisco volcanic field. However, the small-scale compositional perturbations and overall compositional ranges recognized by those authors are considerably greater (±2 wt% Fo) than those associated with the possible zoning in the olivine under discussion.

The Mauna Loa olivine grain illustrated in Figure 2c was also analyzed (lower plot, Fig. 5). The average composition for 91 analyses is MgO = 39.44 ± 0.13 (one standard deviation) and FeO = 20.79 ± 0.13 wt%. Thus, this grain shows no obvious variation in composition in an area with marked microtopographic etch features, although it is possible that the analytical data reflect small-scale fluctuations in composition (Fig. 5). The distributions of the minor elements Ca, Mn, and Ni in the Hawaiian olivine were determined along three traverses parallel to the Y-X traverse shown in Figures 2c and 5. These minor elements were chosen because they have quite different partition coefficients between olivine and silicate liquid: Ni strongly partitions into olivine, Mn has a partition coefficient of about one, and Ca strongly partitions into the liquid. Thus it was reasoned that the distribution of these elements might be sensitive to the growth conditions of the olivine. More than one hundred points were analyzed at 2-μm intervals in each traverse. No correlation was found between the three parallel traverses (except for a general Ca decrease toward the crystal edge), and no correlation was found between the distribution of these elements and the topographic features shown in Figure 2c. Approximately 2% of the analytical determinations were well outside three standard deviations of the average and were most likely due to small inclusions such as chromite and silicate glass. The remaining 98% of the determinations showed a small standard deviation (SD) and gave for this olivine CaO = 0.20 wt% (SD = 0.03), MnO = 0.32 wt% (SD = 0.05), and NiO = 0.34 wt% (SD = 0.03). Thus the distribution of minor elements, like that of the major elements, in olivine showed no correlation with the microtopographic etch features.

We conclude that the clear-cut patterns in the Nomarski images of the olivines reflect the successive accretion of zones during phenocryst crystallization. The etching has presumably been controlled by differences in composition and/or microstructure. At least in the case of the Arizona and Hawaiian samples, only very minor, if any, major-element compositional differences are evident between zones. This may reflect crystal growth under conditions of essentially constant temperature and magma composition. It is, however, quite likely that any original compositional differences between the zones would have been substantially reduced by postcrystallization diffusion (Maaloe and Hanson, 1982). The analytical data for the Hawaiian olivine suggest that variations in the abundance of minor elements, some of which might have diffusivities significantly different from Fe or Mg, are not responsible for the etch textures. Another possible explanation for the microzoning is a varying growth rate with attendant changes in, e.g., defect density.

Discussion

Nomarski interference contrast imaging constitutes a useful, and inexpensive, technique for the investigation of the growth history and subsequent development of ferromagnesian mineral phenocrysts in volcanic rocks. The approach outlined in this work is inferred to reveal the existence of unexpectedly fine growth stages in olivine, which has optical properties that normally prevent the detection of such features in thin section, and provides remarkably clear definition of fine oscillatory zoning in augite. Our sample coverage is as yet limited, but we suggest that zonal textures such as we illustrate in Figure 2 may be widespread in olivine phenocrysts in basic to intermediate volcanic rocks: the minimal observed compositional differences (Mg/Fe) between the zones in this mineral, if real, would render this feature difficult, or impossible, to detect by, e.g., electron-microprobe analysis. Conversely, the Nomarski approach should permit the parsimonious microanalysis of mineral grains, owing to its capacity to reveal the zonation modes of numerous, associated, phenocrysts in a rock. It should also be possible to make detailed comparisons between the zonal "stratigraphy" of several different mineral species in a rock, thereby increasing the definition of overall magmatic crystallization history. It is also evident that other aspects of ferromagnesian crystals, such as dislocations and, probably, twinning, are readily delimited, as has already been reported by Wegner and Christie (1985).

We consider that we have recorded only some of the features of zoned mafic crystals, and undoubtedly much remains to be done. However, we would emphasize the effectiveness of the sensibly two-dimensional image yielded by the reflected-light Nomarski technique in the clear definition of the true form of zone boundaries, a feature that is generally uncertain in transmitted light.

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