The application of quantitative interference microscopy to mineralogic and petrologic investigations[1]

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Abstract

This paper demonstrates the applicability of quantitative interference microscopy to the study of minerals and rocks in transmitted light. For readers who are not familiar with interference microscopy, the principle, the detectability and measurement of interference fringe shifts, and the precision of its use are discussed. The following are some of its applications: (1) the far superior ease and precision of determination of the refractive indices of minerals over the traditional Becke line method (there is no need to estimate the match point, but instead it measures the difference of the index between the mineral and immersion medium, hence minimizes the operator's error; a precision of ±0.0002 can be readily obtained); (2) the possible identification of micron-size inclusions; (3) identification of minerals in thin sections by the zero-fringe method; (4) display of chemical variations in minerals; and (5) the study and possible identification of microstructures, such as twinning, zoned crystals, rimmed crystals, exsolution, and various types of lamellar microstructures produced by static or shock deformation. Procedures for measuring interference fringe shifts in various applications are included in an appendix. Complementary use with X-ray diffraction, electron-microprobe analysis, and transmission electron microscopy and electron diffraction gives the proper perspective of the role of interference microscopy in mineralogic and petrologic investigations.

Introduction

Interferometry has a wide range of application. It is used in the inspection of optical lenses and mirrors, in examination of microtopography of metal spheres, in biology, in the synthetic fiber industry, in measurement of indices of refraction of gases, in aerodynamics, and recently in laser holography, etc. (Tolansky, 1973). Within the last decade, interference microscopes have been developed for use mainly by metallurgists and biologists. Except for a few cases, such as the limited study of industrial uses of diamonds and mica, interference microscopy has not received its proper share of attention and emphasis from mineralogists and petrologists. In fact, interference microscopy is not included in a recent laboratory handbook of petrographic techniques (Hutchinson, 1974).

Since 1968 this author has used a Leitz transmitted-light interference microscope to study tektites, impactites, shock-produced lamellar microstructures, lunar glass, volcanic rocks, and breccias. This paper reports some of the experience gained in applying interference microscopy in mineralogic and petrologic investigations. Wherever possible the validity of the methods developed was checked with known techniques. The basic procedures suggested are included. As experience in various applications increases, interference microscopy, when used intelligently in combination with other analytical techniques, emerges as a very powerful and desirable research tool that deserves the attention of modern mineralogists and petrologists.

The interference microscope

There are three kinds of microscopic techniques that employ the principle of optical interferometry: 1. the Nomarski contrast; 2. the Jamin-Lebedeef single objective set-up; and 3. the Horn separated objective interference microscope (Richardson, 1971). The Nomarksi contrast is a qualitative method that shows exaggerated relief difference between adjacent mineral grains, and is useful particularly for detecting textures of intricate intergrowths of mineral phases. The single objective Jamin-Lebedeef method gives quantitative results, but it is not very versatile. It is

suitable for measuring the refractive index of isolated individual grains in oil immersion or other embedding media. The cost of the attachment for the Jamin-Lebedeef method is much less than the interference microscope. It is, however, impractical for studying thin sections. The separation between the reference or control beam and the measuring beam is less than adequate (300μm at 150X magnification). It can only be used along edges of the thin sections where blank areas for the control beam can be located. This paper concentrates on the use of the versatile separated objective interference microscope.

Since the author's experience with quantitative interference microscopy has been largely with the Leitz[2] transmitted light interference microscope, only the principle, major design aspects and the use of this particular instrument are briefly described here. The emphasis is on its most direct application to mineralogic and petrographic studies, which represents an advance in methods of mineralogic and petrologic research. The presently available model of the instrument was developed nearly 10 years ago, primarily for biological research and industrial applications.

The Leitz instrument is a combination of a Mach-Zender interferometer and a transmitted light microscope. It consists of a pair of parallel microscopes having precisely matched small aperture condensers and precisely matched objectives; separation of the measuring beam and control beam is 6 cm (Fig. 1). The instrument is equipped with tungsten white and mercury vapor lamp illumination. With the white light source, a slit is provided for narrow-band filters for monochromatic illumination, such as the Na D line. Hence the dispersion of any transparent material may be measured directly.

The incident light beam goes through a polarizer and then a beam splitter. The beam splitter sends one half of the beam (the measuring beam) into an essentially petrographic microscope on the left, and the other half (the control or reference beam) into a matching microscope on the right (Fig. 2). The two optical path lengths are identical. A second beam splitter recombines the two beams in interference to the viewer according to the principle of interference fringes from a double light source (Jenkins and White, 1950). All bright lines in the interference pattern occur where the distances from the two sources differ by an integral number of wave lengths ($n\lambda$), and dark bands occur when the dis-

tances differ by $(n + 1/2)\lambda$. The P_1 plate compensators (Fig. 2) are used in the alignment of two beams. The lower plates, P_3, which rotate on an EW-axis, tilt the interference fringes. The upper plates, P_4, which rotate on a NS-axis, adjust the width of the interference fringes produced by the microscope from any selected width to infinity (known as fringe contrast). An analyzer is located above the upper beam splitter for viewing with crossed-polarizers in mineralogic and petrographic studies. A metal plate located at B slides laterally to block the control beam when desired. The wedge and plate compensators, P_2, are used to measure the interference fringe shift. The measuring beam objective is fixed and is not centerable by the operator. In order to obtain maximum contrast of interference fringes, the images of the field-of-view diaphragms formed by the two beams must be perfectly coincident. This is accomplished by adjusting the focusing lever and centering screws of the control beam objective only. Matched objectives of 6X, 20X, 50X, and 100X (oil immersion) are available.

The calibration and operation of the instrument are quite simple. Anyone familiar with a petrographic microscope can learn to operate it. The maximum amount of information and the quality of contrast of the interference fringes depend, however, on the quality of the prepared sample to be studied and the blank prepared for the control beam. For the best fringe contrast and quick operation, the control beam blank should be prepared nearly the same way as the sample prepared for study. For example, one should use an oil immersion cell as the blank for immersion study, polished thin section blanks of identical slide thicknesses for polished thin sections, and covered thin section blanks for covered thin sections.

As noted above, with the matched objectives in place but without either the sample or the blank for the control beam, interference fringes are produced. The introduction of a sample in the measuring beam increases the optical path for this beam and causes the fringes to shift (Fig. 3). The phase shift, Γ, or the amount of shift of the interference fringes, is proportional to the product of the thickness of the material (t) and the difference in refractive index (R.I.) between the subject of interest and the adjacent material. This is expressed by the equation:

$$\Gamma = (n_a - n_b)t \qquad (1)$$

where n_a is the R.I. of material a; n_b the R.I. of the immersion oil, embedding medium, or adjacent material; and t the thickness of the material (Krug *et al.*,

[2]The use of a trade or brand name is for descriptive purposes only and does not constitute an endorsement of the product by the U.S. Geological Survey.
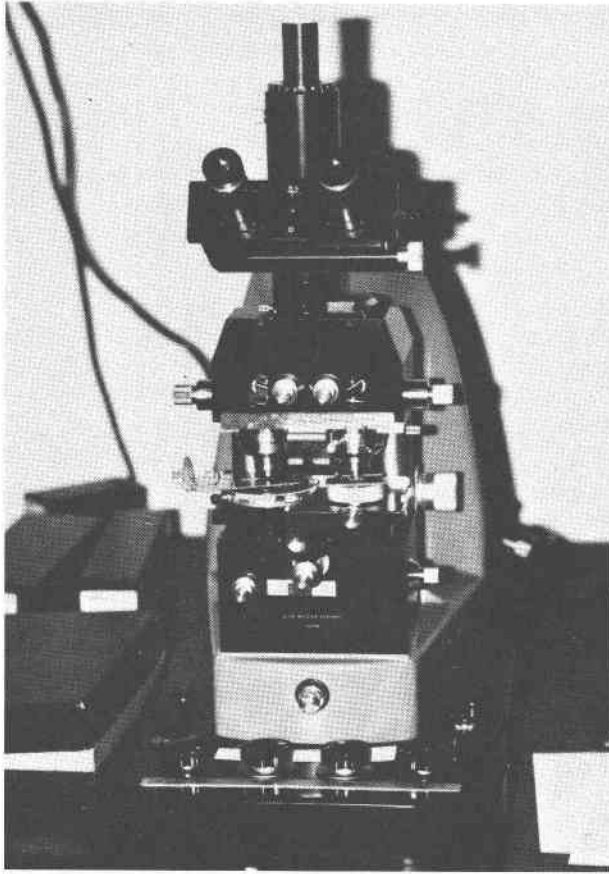
FIG. 1. Photograph of the Leitz transmitted light interference microscope with a spindle stage showing the arrangement of two parallel microscopes, the center axes of which are separated by 6 cm.

1964). Hence n_a or n_b or t can be determined if the other two are known. (Alternatively, $n_a - n_b$ may be determined if only t is known.)

Application to mineralogic investigations

The application to mineralogic studies is direct, without any need for modification of the instrument. One obvious application is the determination of the precise R.I. of any loose mineral grain as a means for identifying the mineral as well as the correlation of its optical properties with compositional variations. In a similar manner, the study of R.I. variations in glasses of different shapes can be readily determined.

Interference microscopy as a part of the routine mineralogic study of single mineral grains

The quickest way to orient a mineral grain in order to measure its principal R.I. is, in my opinion, to use
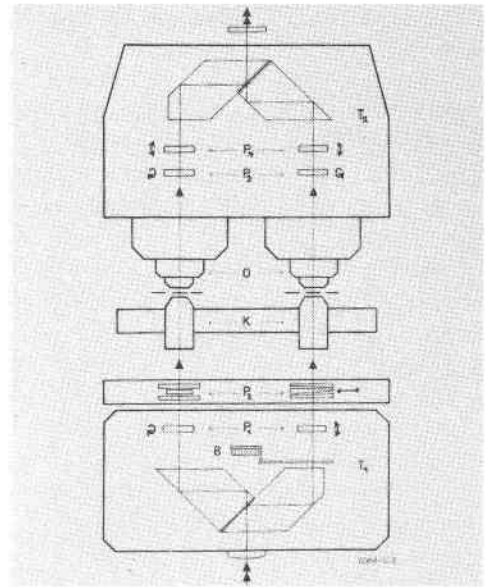

FIG. 2. Ray diagram of the transmitted light interference microscope shown in Figure 1, taken from the microscope manual. See explanation in text.

a spindle stage (Wilcox, 1959) with the fragment in calibrated immersion oil of known R.I. This can be done quickly with a petrographic microscope. Then transfer the spindle stage to the measuring beam side of the interference microscope. With the directions of vibration of the α, β, and γ indices of the fragment
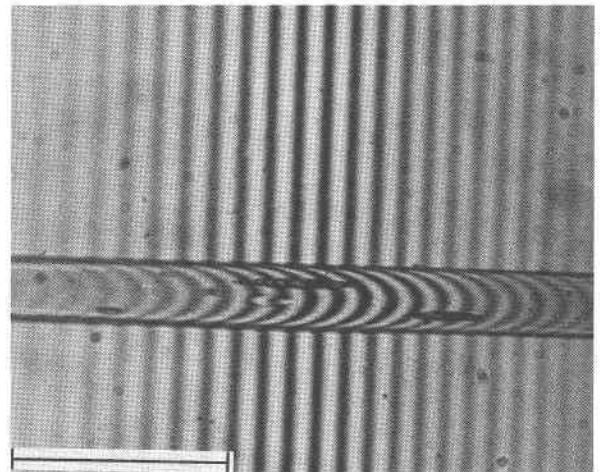

FIG. 3. Photomicrograph of a Pele's hair with inclusions in an immersion oil of R.I. 1.550. The fringe shift along any fringe corresponds to the precise difference of R. I. between the glass fiber and the oil. The R.I. so determined of the Pele's hair with a diameter of 30.7 μm is 1.6000 $\pm$ 0.0002 at 24 °C. The two elongate inclusions with R.I. 1.02 are probably trapped air bubbles. The R.I. of the lenticular inclusion is 1.546, probably that of a siliceous glass particle. Transmitted light; bar scale 100 μm.

known, it is a simple matter to measure the fringe shift for a principal vibration direction in the mineral fragment compared to the immersion oil. An easily recognizable point should be selected for the measurement because the thickness of a mineral fragment is variable. By changing oil once, and measuring the fringe shift at the same point, two simultaneous equations of (1) are obtained and the effect of the unknown thickness is eliminated. Thus interference microscopy enables one to determine the difference in R.I. in terms of fringe shifts that can be measured to within a fraction of a wave length. If the mineral is of low birefringence, as are the feldspars, all three indices can be measured in one oil and the next. The precision of such measurement between different operators, for example between my colleagues and myself using quartz as a test mineral, is ±0.0002, which is the precision of the calibration of the immersion oil (with the necessary temperature correction applied in an air-conditioned laboratory). For minerals with R.I.'s higher than 1.70, the stability of the immersion liquid introduces more of a problem, reducing the precision of measurement to about ±0.0005 to 0.0010, still more than adequate for most if not all mineralogic studies.

The extensive use of the electron microprobe for mineral analysis may have reduced somewhat the need for accurate determination of the optical properties of minerals. Precise optical properties are necessary, however, for the characterization of a new mineral species, or for better correlation of the optical properties of known minerals with either chemical composition or unit cell parameters.

Investigation of glass particles and the homogeneity of natural glasses

Lunar glass particles, variously shaped as spheres, oblate spheroids, tear drops, dumbbells, etc., cover a wide range of color and chemical composition. Their R.I.'s range from about 1.49 for siliceous glass (clear) to 1.75 for high iron and high titanium glasses (deep red) (Chao *et al.,* 1970a). Using the spindle stage, the fringe shift for a glass sphere in any chosen immersion oil close to the index of the glass is readily measured with the interference microscope. The diameter of the sphere at the point measured can be precisely determined with the Vickers A.E.I. Image Splitting Device (to ±0.1µm). It is then a simple matter to calculate the R.I. of the glass. This method drastically reduces the number of immersion oil changes required for matching and is an order of

magnitude more precise than the traditional Becke line method. In addition, it is not affected by the color of the glass. The advantage of this method is not fully appreciated unless thousands of glass particles of a wide range of color and composition are to be studied. Being able to select those that cover the wide range of chemical variation can avoid hundreds of unnecessary duplicate electron microprobe analyses.

The diameter of a glass fiber of Pele's hair immersed in 1.550 oil (Fig. 3) was accurately measured optically, and its R.I. was then readily determined from the fringe shift Γ to be 1.6000 ± 0.0002. Within the fiber, two elongate inclusions have R.I. of about 1.02 (uncertainty due to difficulty of counting the narrow fringes), suggesting that they are air bubbles. A moderately low-index lenticular inclusion having R.I. 1.546 is also present. This is probably a more siliceous glass particle. All three R.I. determinations were made without changing oil.

The interference microscopic method can likewise be extended to the study of large samples of heterogeneous glasses, such as impactites, tektites, or mixed or interlayered volcanic glasses. The details of heterogeneity and flow bands (schlieren) in tektites are shown clearly by the fringe shifts, so that meaningful points can be chosen for electron-microprobe analysis (Chao and Desborough, unpublished data). Volcanic glasses are usually formed from melts produced slowly, and hence are essentially uniform in chemical composition. Interference fringes in homogeneous glass are straight (Fig. 4a). Lunar or terrestrial impactites and tektites were formed by rapid fusion associated with meteorite impact. As the melts formed rapidly from heterogeneous starting materials, the glasses show well-developed schlieren (Fig. 4b) and strong chemical variations.

From personal experience, I am convinced that the combined use of spindle stage and interference microscopy supersedes and is faster and much more precise than the traditional method of determination of the R.I. of minerals and glasses by the Becke line immersion method using either single variation (variation of wave length) or the double variation method (variation of wave length and temperature).

Application to the study of petrographic thin sections

The combined variations of microrelief and R.I. of minerals provide an advantage for quick recognition of minerals and mineral intergrowths using the interference microscope. In many cases mineral identi-
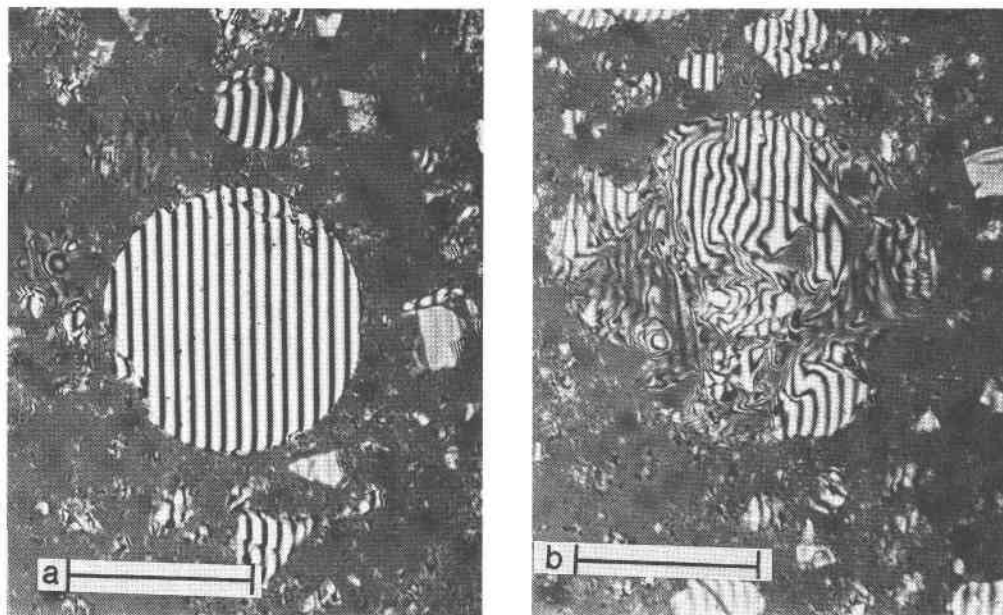
FIG. 4. Photomicrograph of a doubly polished thin section of an Apollo 11 lunar regolith microbreccia (see Chao *et al.*, 1970b). Interference fringes in the homogeneous glass sphere (a) are straight; those in the larger heterogeneous glass sphere (b) are highly irregular (in places the fringes are interrupted by holes). Transmitted light; bar scales 100μm.

fication is possible. In most cases, detailed chemical variations within a single mineral species or between different grains are revealed.

Covered versus uncovered doubly-polished thin sections

The interference fringe shift observed at any point is a measure of the total retardation at that point in a thin section. Hence it is affected not only by the mineral and its orientation but also by the embedding medium above, below, and sometimes impregnated along cracks of the mineral. A doubly-polished thin section, such as is employed in the study of lunar samples, has appreciable relief between the mineral grains and the embedding medium. Determination of the absolute R.I. values of α' or γ' of a mineral by comparison with the embedding medium may be a difficult problem if such relief is not determined (see below). In covered thin sections, the cementing medium below the cover glass compensates for the relief difference so that the determination of the absolute R.I. values of α' or γ' of a mineral can be readily made.

However, despite the relief problem in interference microscope studies, doubly-polished thin sections have great advantages over covered thin sections for petrographic work. Because the thin section is doubly polished, it is far superior in quality and in revealing details of substructures; above all, it is readily available for study with incident light by reflected light microscopy, and for electron microprobe analysis. Moreover, if the user is proficient in identifying transparent minerals by their reflectivity, the reflected light study reveals details of textures as if it were an ultra thin section. In addition, a more reliable modal analysis can be made with reflected light if opaque minerals make up some percent of the mineral assemblage. There is no reason whatsoever why terrestrial samples should not be prepared in this manner. In order to take advantage of it, however, a modern research petrographic microscope having a lever that can switch from transmitted light to reflected light illumination is most desirable.

Thickness variations, R.I. of the embedding medium, and the identification of minerals by the zero fringe method

The thickness of a thin section and the R.I. of the embedding medium are of prime importance in the optical identification of minerals in thin section using the interference microscopic technique. Even the best thin sections are not uniform in thickness. Since fringe shifts are sensitive to minute differences in both

relief and index, it is important to check the thickness variation of a thin section prior to measuring the mean R.I. $(\alpha' + \gamma')/2$ of any biaxial mineral, or ω of any uniaxial mineral.

Fortunately, from my experience, the thickness variation in local areas between adjacent minerals in most thin sections is negligible and produces no measurable shift in the position of the zero fringe.

The R.I. of the embedding medium should be known in any thin section to better than ±0.001, be it epoxy of various types or Canada balsam, etc. If the rock contains quartz, the thickness of the thin section at many points where quartz is adjacent to the embedding medium can then be determined using equation (1). As quartz is uniaxial, ω is always available as a standard for comparison with any adjacent mineral. With adequate thickness control, the mean index of any mineral can then be measured.

If the rock contains plagioclase but no quartz, as in most lunar rocks, one should analyze a few of the plagioclase grains with the electron microprobe and establish a correlation with the mean R.I. of the plagioclase. Other minerals are then studied using plagioclase as a reference or control mineral. Hence, knowing the R.I. of the embedding medium of a thin section and one reference mineral, the entire thin section including micron-size inclusions can be explored.

In contrast to the measurement of fringe shifts of loose grains in immersion oil, the R.I. of a mineral in thin sections is measured by the phase shift with respect to the position of the zero fringe either in the embedding medium or in a known mineral nearby. The position of the zero fringe of ω or α' of any anisotropic mineral is dependent on the thickness and R.I. of the mineral. Before using the zero fringe method for mineral identification in thin sections, it is necessary to identify the fringes along which fringe shifts are measured. The method will not be valid if the zero fringe in one mineral becomes the first- or second-order fringe in another mineral, and thus misidentified. Such cases are rare in thin sections 30 to 35μm thick and can be avoided or corrected if certain precautions are taken (see below). With the help of an ocular micrometer, the relative position of the zero fringe is always quickly visible in the field of view of the microscope with white light illumination. The position of the zero fringe also readily identifies the ω and ϵ' or α' and γ' of a birefringent nonopaque mineral and measures the birefringence or the mean index at any point desired.

In a thin section of granite from the Black Hills,

South Dakota (Fig. 5), the major minerals, including the microcline microperthite, are readily recognized by the position of the zero fringe. The precise zero fringe shift between minerals, however, should be measured only with each mineral in extinction position (see procedures in the appendix). Rapid identification may even extend to micron-size inclusions. If the R.I.'s of the minerals overlap, an alternative method is available based on the property of birefringence. The observed apparent birefringence of each grain can be measured quantitatively by the same zero fringe method. My limited experience has shown this, under favorable circumstances, to be a promising method for differentiating between grains of or-
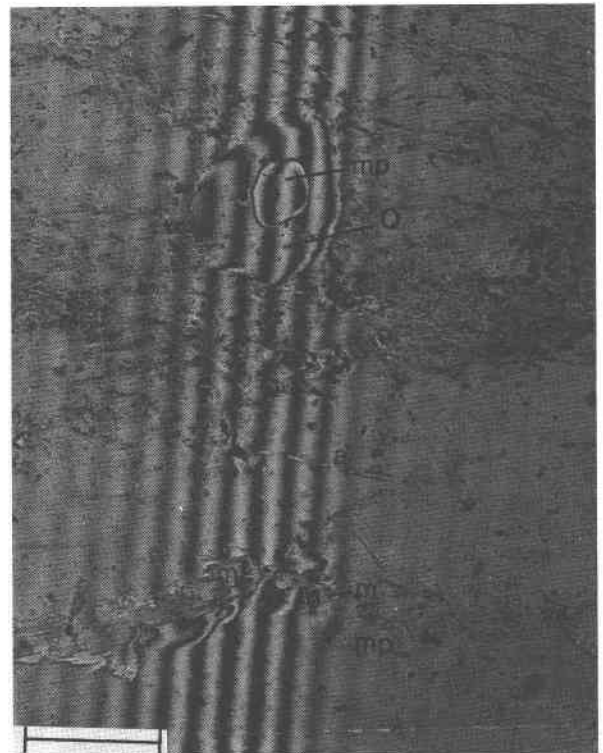

FIG. 5. Photomicrograph of a covered thin section from the Black Hills, South Dakota, showing the method of mineral identification by the zero fringe method. Note the shift or relative position of the zero fringe (0) in each mineral: Q, quartz; a, albite; mp, microperthite (plagioclase lamellae of higher index at top of grain shift to the right with respect to the microcline host); m, muscovite. The zero fringe position in the inclusion surrounded by quartz is easily identified as microperthite, since it lies approximately in the same position as that of the large microperthite below. The amount of the shift of the zero fringe should be measured only at the extinction position of each mineral, which is not the case in this figure (see procedure in appendix). ω of quartz or the mean index of the minerals $(\alpha' + \gamma')/2$ can be determined. Transmitted light; bar scale 100μm.

thopyroxene, pigeonite, and augite and between pyroxenes and olivine in fine-grained metamorphic lunar rocks.

Microrelief characteristics of minerals

Many minerals have different inherent polishing characteristics due to hardness, cleavage, alteration products, etc. These effects produce minute differences in relief and surface textures that are immediately recognizable in doubly-polished thin sections under interference. Many of the secondary alteration products such as clays versus mica can be readily distinguished. Covered thin sections can also be used. The straight and smooth fringes in quartz and the zig-zag fringes in plagioclase and mica are characteristic for each (Fig. 5). They are easily recognizable even without noting the relative position of the zero fringes in them.

Correlation of intra- and intergrain variation in R.I. and chemical composition of a single mineral species

It is well known that the variation of chemical composition of a single mineral species is reflected by the variation of its R.I. Such variation sometimes results from differentiation (for example plagioclase phenocryst versus groundmass plagioclase) or reaction (zoning or reaction rim). Any index variations are reflected and portrayed in detail by the interference fringe shifts. It is evident that photomicrographs of minerals showing the distribution of fringe shifts can be used to select areas of compositional variation for electron microprobe analysis. Interference photomicrographs not only define the objective and pinpoint the areas to be analyzed, but also frequently predict trends of chemical variations.

Furthermore, chemical variations between grains can be detected easily by determining Δn of two adjacent grains directly or by comparison with the embedding medium. In this case the absolute mean index need not be of concern. One needs to be aware, however, of thickness and relief variations which contribute to the measurable fringe shifts. This type of study has extensive application in lunar breccias in which a wide variety of plagioclase-bearing and pyroxene-bearing lithic clasts occur together.

Detectability and precision of fringe shift measurements

It is clear from equation (1) and Table 1 that the amount of measurable fringe shift or detectability

increases with increasing Δn and Δt. If Δn is constant, detectability or fringe shift increases with thickness. If Δt is constant, fringe shift increases with increasing Δn.

The grain size or the effective thickness for single grain-immersion oil study is generally greater than $100\mu m$. In this case the detectability of fringe shift by the visual compensator method is about 10nm.

For thin sections of standard thickness of about 30 μm (about 1/3 the thickness of a single grain), the visual measurable shift of the zero fringe is about 1/20 of a wave length or about 30nm. This corresponds to a detectable limit Δn of about 0.001, adequate for most petrographic studies. If the Δn between adjacent minerals is 0.01, a thickness difference Δt of $1\mu m$ (Table 1) is barely detectable. Such a relief difference (1/30 of the thickness of a thin section) may arise due to the effect of hardness differences on polishing. If Δn between adjacent minerals is greater than 0.05, a Δt of $0.05\mu m$ can be detected at the zero fringe shift.

The precision of visual fringe shift measurement using the wedge compensator ranges from 1/10 to 1/50 of a wave length. If the total fringe shift is 1 wave length or greater the precision is about 1/30 of a wave length or about 5 percent of total fringe shift. If the total fringe shift is only 1/10 of a wave length, the precision is about 20–35 percent of total shift. Better precision can be obtained if films or photographs and densitometers are used. For most mineralogical problems, a precision of 1/30 of a wave length is more than adequate.

Examination of a typical thin section, cut, polished, and physically measured on edge, shows that

TABLE 1. Variation of Δn and Δt with the amount of fringe shift Γ.

Δn	Δt (nm)	Γ (nm)
0.001	100	0.1
	500	0.5
	1000	1.0
0.01	100	1.0
	500	5
	1000	10
0.05	100	5
	500	25
	1000	50
0.1	100	10
	500	50
	1000	100

gradual thickness variations of 5–7μm within a thin section are common. Keeping this and the data shown in Table 1 in mind, actual measurement of thin sections of either ω of quartz or the mean index of a biaxial mineral shows that an accuracy of ±0.002 is perhaps the best that can be expected. Further evaluation of accuracy is needed.

The study of micron-size inclusions

It is difficult if not impossible to identify with the petrographic microscope inclusions in minerals or in thin sections that are only a few microns to micron-size. Preliminary trials have shown that gaseous, liquid, glass or mineral inclusions from 1 μm to tens of microns in size may be identified with the interference microscope.

Quartz grains in a granite from the Black Hills of South Dakota contain abundant irregular-shaped inclusions of low index (Fig. 6). The index of such inclusions between 2 and 10μm in size and of equidimensional inclusions (carefully chosen on the basis of the characteristics of the fringes) have been measured. The R.I.'s of these inclusions are 1.34 and 1.35, suggesting that they are aqueous solutions with variable amounts of dissolved salt. Verification of such an identification is not available. As shown in the same grain (Fig. 6), a high index inclusion 19.1μm across is also present. The approximate α' and γ' determined as compared to the ω of the enclosing quartz are 1.563 and 1.605, respectively. Large grains of mica are also present with approximate γ' R.I. of 1.600, suggesting that this high-index inclusion is also muscovite. The inclusions in Pele's hair (Fig. 3) mentioned above are another example of this application. As a further exercise, I attempted to check the R.I. of a high pressure phase, suspected to be jadeite, in plagioclase glass of oligoclase composition. This sample occurs in a shocked amphibolite fragment from suevite from the Ries crater of Bavaria, Germany. Using the same method, the R.I. close to the mean index was determined to be about 1.59, consistent for jadeite (James, 1969).

Potentially, as illustrated by the above examples, small gaseous, liquid, or solid inclusions in minerals can be investigated with the interference microscope. How accurate and reliable these results are must await further tests.

The study of microstructures

The study of microstructures in minerals is an important part of mineralogic and petrologic investiga-
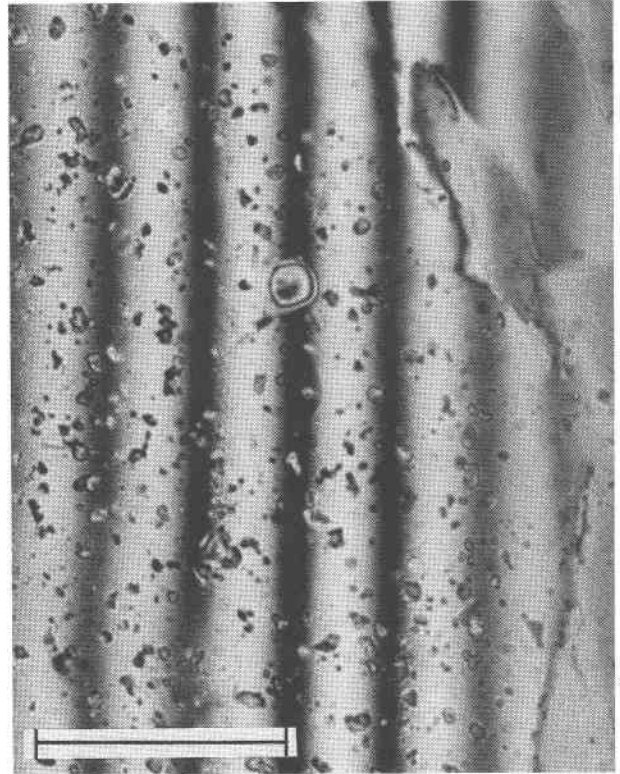

FIG. 6. Photomicrograph of a quartz grain in the same section of granite shown in Figure 5. By the method of zero fringe (in this case in ring fringes), the large 19.1 μm grain lying on the zero fringe is tentatively identified as muscovite, and the abundant irregular inclusions as inclusions of saline or mineralized water. Transmitted light; bar scale 100μm.

tions which are directly process-related. Optically resolvable microstructures such as parallel fracture planes, twinning, zoning, exsolution, rimmed crystals, and deformation-produced lamellar structures can be studied with the interference microscope both in single mineral fragments using the spindle stage and in thin sections. The additional information obtainable with interference microscopy is described in this section.

Open and filled fractures

Fractures may, in some instances, be confused with other fine lamellar structures. Investigation with interference microscopy can eliminate this uncertainty. Open fractures filled with air and not with mounting media or other minerals, for example, those in quartz or feldspar, will always cause the interference fringe to shift to the left with respect to that fringe in the

quartz or feldspar grain. (For consistent or standardized operation, the Jamin plates, P_4 in Figure 2, are always set up to incline down toward each other; in such case a lower-index mineral or material (air) causes the interference fringe to shift to the left. The reverse will be true if the Jamin plates are inclining down away from each other.) If the fracture is filled with glass of lower R.I. than the host, the fringe shift is also to the left but not as great as for air. If the fracture is filled with secondary mica, the shift will be to the right. Table 2 shows the calculated fringe shift for fractures filled with materials of different index.

In most cases, it should be possible to differentiate or identify the filling material based on the direction and magnitude of the fringe shift. A positive identification is possible if the width (or thickness) of the fracture can be measured. Experience shows that air-filled or open fractures can be quickly differentiated from fractures filled with Canada balsam or epoxy. Veinlets filled with quartz or mica can also be identified. The best and most rapid test of open fractures is to use a grain mount in immersion liquid. Quartz with micron-size open fractures (Fig. 7) will quickly and unambiguously show these to be open fractures in oil because the oil fills the fractures and causes a fringe shift to the left against the ω of quartz for 1.540 oil (Fig. 7a) and to the right for 1.560 oil (Fig. 7b). This will be further discussed in the section on shock-produced lamellar structures below.

Twinning

Normal polysynthetic twinning in plagioclase is readily identified by any experienced petrographer unless such twinning becomes so fine as to resemble some exsolution features, fine zoning, or deformation lamellae. Some of these cases are difficult if not impossible to resolve even with the interference microscope and should be studied by transmission electron microscopy and diffraction. Frequently, however, optically resolvable twin features can be quickly identified. Depending on the twin law, some obviously twinned grains show no measurable fringe shifts across the composition planes. These are easily identified as twins rather than compositional variations or deformational lamellae with strain birefringence. Other twinned grains show distinct fringe shifts. As the position of α' and γ' in each twinned member can be quickly identified by the shift of the zero fringes at the same point of observation, they can be easily compared with the α' and γ' of the adjacent twinned member (Fig. 8). This is particularly evident on twinned grains with twinned members in equal illumination, so that the direction of fringe shift at extinction positions 90° apart is reversed. A criterion for differentiating twin lamellae from exsolved lamellae is that the fringe shift for composition difference is consistent, whereas this is not the case for common polysynthetic twinning.

Zoned phenocrysts

The compositional variation of zoned plagioclase or pyroxene phenocrysts can be analyzed with the electron microprobe. The advantage of interference microscopy in preparation for probe analysis is that it provides a clear display of such variations. It provides the choice of individual zoned crystal and specifies the most informative traverse to be analyzed. Without this help, a much larger number of analyses will be required, and there is no guarantee that the most critical parts of the crystal will be analyzed. Variations in analyses may present a very confusing picture. Figure 9 shows a zoned plagioclase phenocryst in a quartz latite. The zoning is clearly seen both in bright field and in interference. The specific nature of the oscillatory zoning is however only revealed in interference. High-index zones are

TABLE 2. Calculated interference fringe shift (Γ) in nm in white light for various thicknesses of air, silica glass, Canada balsam, epoxy, muscovite, and biotite in quartz ($\omega = 1.544$) 30μm thick. Those with index less than ω of quartz shift to the left ($-$). Those with index greater than ω of quartz shift to the right ($+$).

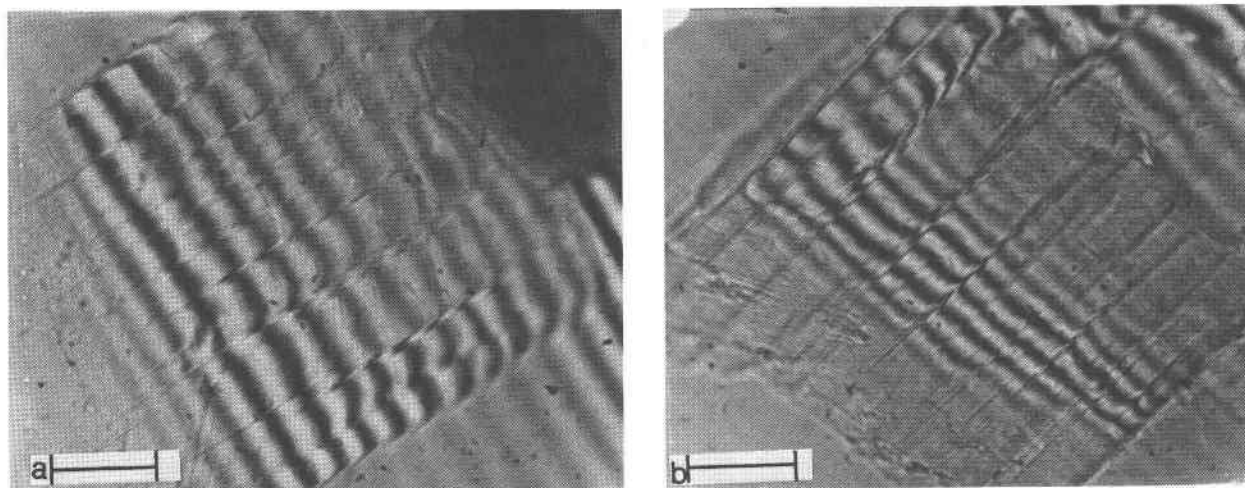| Thickness of substance in quartz	Γ air $n = 1.000$	Γ silica glass $n = 1.460$	Γ Canada balsam $n = 1.543$	Γ araldite epoxy $n = 1.552$	Γ muscovite $\alpha' = 1.560$	Γ biotite $\alpha' = 1.600$
1/10 of 30μm (3μm)	−1632	−252	−30	+24	+48	+168
1/20 of 30μm (1.5μm)	− 816	−126	−15	+12	+24	+ 84
1/30 of 30μm (1μm)	− 544	− 84	−10	+ 8	+16	+ 56
1/60 of 30μm (0.5μm)	− 272	− 42	− 5	+ 4	+ 8	+ 28

FIG. 7. Experimentally shocked quartz with parallel open fractures ranging from 1μm to several μm in width. (a) In 1.540 oil, the fringe shift across the oil-filled fracture is to the left; (b) in 1.560 oil, the fringe shift across the fracture filled with oil is to the right. Transmitted light; bar scales 100μm.

clearly shown by their magnitude and direction of shift to the right (Fig. 9b). In interference contrast, the variation in composition of the zoned crystal is revealed as a map, so that the choice of the location of traverse for electron microprobe analysis can be selected to reveal the entire history of the compositional changes during crystallization of this phenocryst. The specific advantage is that, with the interference microscopic technique, the investigator can plan the electron microprobe analysis in a much more efficient, specific, and economical way. The saving of electron microprobe analysis time can be considerable.

Exsolution

This is similar to the problem of studying zoning with interference microscopy. Exsolution features that are familiar to the petrographer, such as perthites and microperthites of feldspars, are no problem. In lunar samples, however, most of the pyroxenes are exsolved, and the widths of the exsolved lamellae and blebs range from microscopic to submicroscopic dimensions. Such fine exsolution lamellae can be confused with other fine lamellar structures such as twinning. Although the fine submicroscopic exsolution textures must be studied by
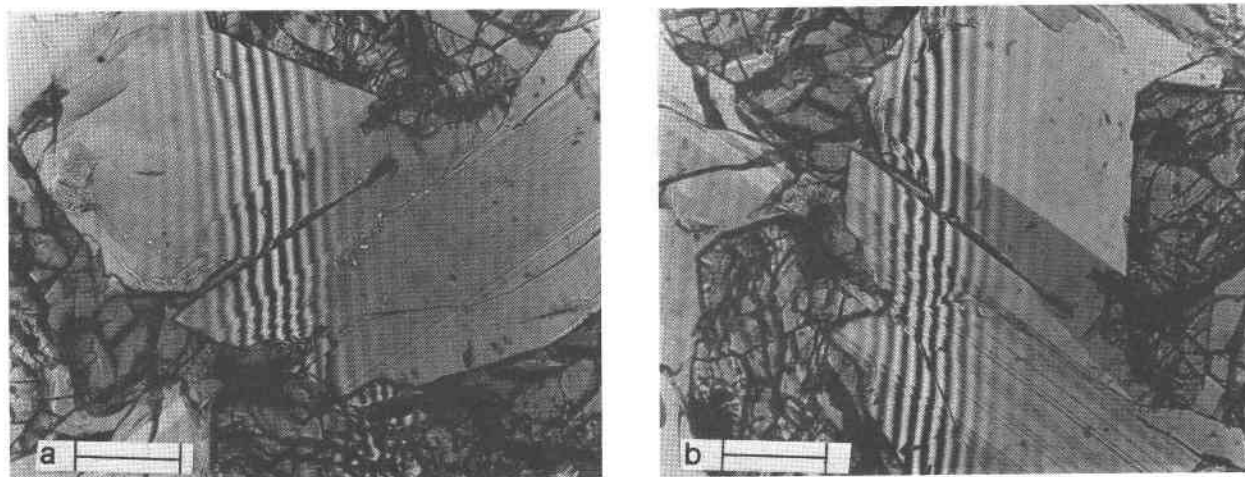

FIG. 8. Photomicrographs of a doubly polished thin section MP-146 from a basalt from the Makaopuhi lava (Hawaii), showing polysynthetically twinned (albite law) plagioclase. The shift of the twinned lamellae in (a), where $\gamma'_1 > \alpha'_2$ is reversed in (b), where $\gamma'_2 > \alpha'_1$. This clearly identifies the twin characteristics. Transmitted light; bar scales 100μm.
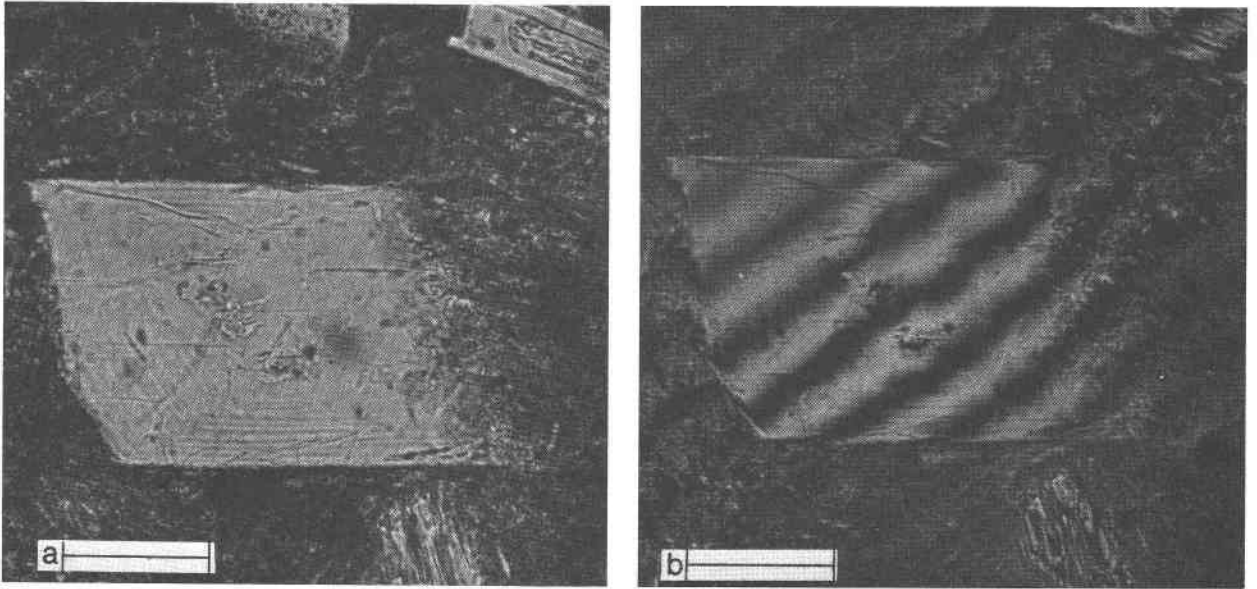
FIG. 9. Photomicrograph of a zoned plagioclase phenocryst in a quartz latite. (a) The zoned phenocryst in normal bright field illumination; (b) with interference fringes. Note in (b) the specific shifts in the various zones: those to the right are of higher index than those to the left, and the amount of shift is directly correlated with the chemical composition. Note, further, the shape of the high-index core which is elongated toward the lower right. Transmitted light; bar scales 40μm.

transmission electron microscopy and X-ray diffraction, those that are wide enough to be analyzed by electron microprobe are also wide enough to be studied by interference microscopy. The latter can detect shifts that suggest near-submicroscopic exsolution that cannot be analyzed by the electron microprobe. Closely coordinated study between interference microscopy and electron microprobe analysis is clearly indicated.

Figure 10 shows a lunar orthopyroxene xenocryst in a xenocrystic feldspathic pigeonite basalt, 77115, with complex patterns of blebs and lamellae of exsolved augite. The coarse blebs are easily recognized with or without interference microscopy. The nature of the fine lamellae however is less clear. The consistent shift of the fringes suggests exsolution. This was confirmed subsequently by electron microprobe analysis.
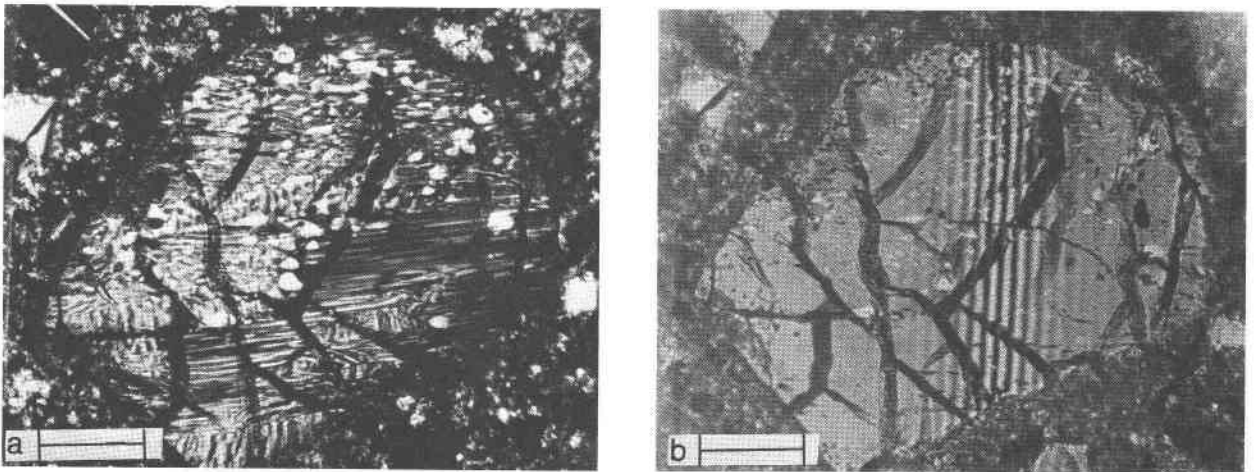

FIG. 10. Photomicrograph of a lunar orthopyroxene xenocryst in 77115,53, showing the exsolved augite both in the shape of irregular blebs (upper left) and in twin-like lamellae (lower right). The nature of the consistent shift suggests exsolution rather than twinning. 10a in crossed-polarizers; 10b in interference fringes. Transmitted light; bar scales 100μm.

Reaction rims and rimmed crystals

Rimmed olivine, plagioclase, and pyroxene occur in typical Apollo 17 xenocrystic feldspathic pigeonite basalts such as sample 77115. They provide a splendid opportunity for joint study by interference microscopy and electron microprobe analysis. In this paper only examples of a rimmed olivine and a pyroxene xenocryst are given. The rimmed olivines present a simple and straightforward compositional variation whereas the pyroxenes have a complex compositional variation and history.

There is no indication in bright field that most of the olivine xenocrysts are rimmed except for a slight change in birefringence in one or two xenocrysts (Fig. 11a). Figure 11b shows the olivine xenocryst of Figure 11a with interference fringes. The fringes are straight in the core but distinctly inclined to the right in the rim. The amount of shift varies. The straightness of the fringes in the core means the composition of the core olivine (Fo_{91}) is uniform. The sharpest bend along the rim reveals the steepest gradient in increase of the index of refraction of either α' or γ', whichever one chooses as a reference. This gradient represents a change from Fo_{91} to Fo_{73} which is close to the olivine composition in the matrix.

Electron microprobe analyses show excellent curvilinear correlation between the amount of fringe shift and the amount of FeO or the FeO:MgO ratio (positive correlation) and MgO (negative correlation). There is also a strong positive correlation with TiO_2 content (Chao *et al.*, unpublished data). It is clear from Figure 11b that the area of steep chemical gradient can be pinpointed for traverse by the electron microprobe. We have used the interference method to select such olivine xenocrysts for electron microprobe analysis.

Figure 12 shows a rimmed clinopyroxene xenocryst. The core is an exsolved augite. The host of the core is augite $Wo_{42}En_{34}Fs_{24}$ containing exsolved intermediate pigeonite $Wo_{11}En_{44}Fs_{45}$. Here the mean index of the pigeonite (near β) is greater than the mean index of augite, hence the fringes distinctly shift to the right (Fig. 12b). The mean index value cannot be determined because the thickness of the thin section is unknown. The difference of the α' of augite compared to the α' of pigeonite is clearly evident. The rim consists of augite $Wo_{35}En_{46}Fs_{19}$ containing exsolved magnesian pigeonite $Wo_{12}En_{67}Fs_{21}$. The zero fringe in the rim is shifted distinctly to the left. This is a result of the low iron content in the rim. It is also clear that the composition of the rim approaches the composition of the augite and magnesian pigeonite in the matrix, whereas the core is clearly not in equilibrium.

The advantages gained from such a study are as follows: (1) because of the texture revealed, electron
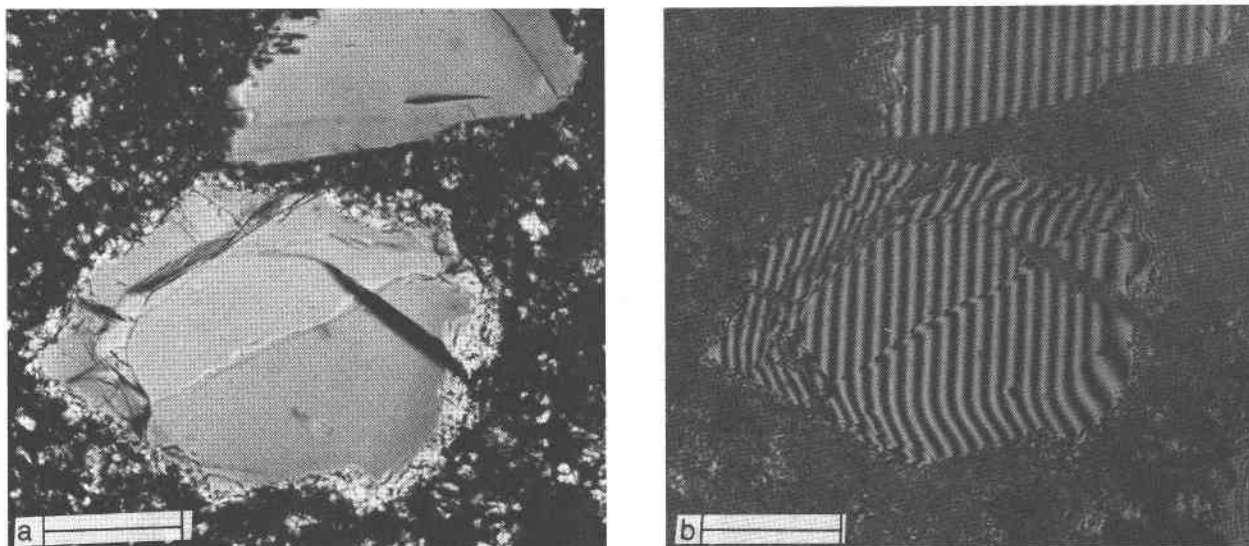

Fig. 11. Photomicrograph of an olivine xenocryst in 77115,50, an Apollo 17 xenocrystic pigeonite basalt, (a) shows the olivine xenocryst in crossed-polarizers with no evidence of chemical variation; (b) the same specimen showing interference fringes. Note the straight fringes in the homogeneous core of the olivine Fo_{91} and the varying higher index of the rim. The strongest shifts in the lower part and upper right pinpoint areas that are highest in FeO. Position for electron-microprobe traverse can be easily chosen on the basis of this map-like interference photomicrograph. Transmitted light; bar scales $100\mu m$.
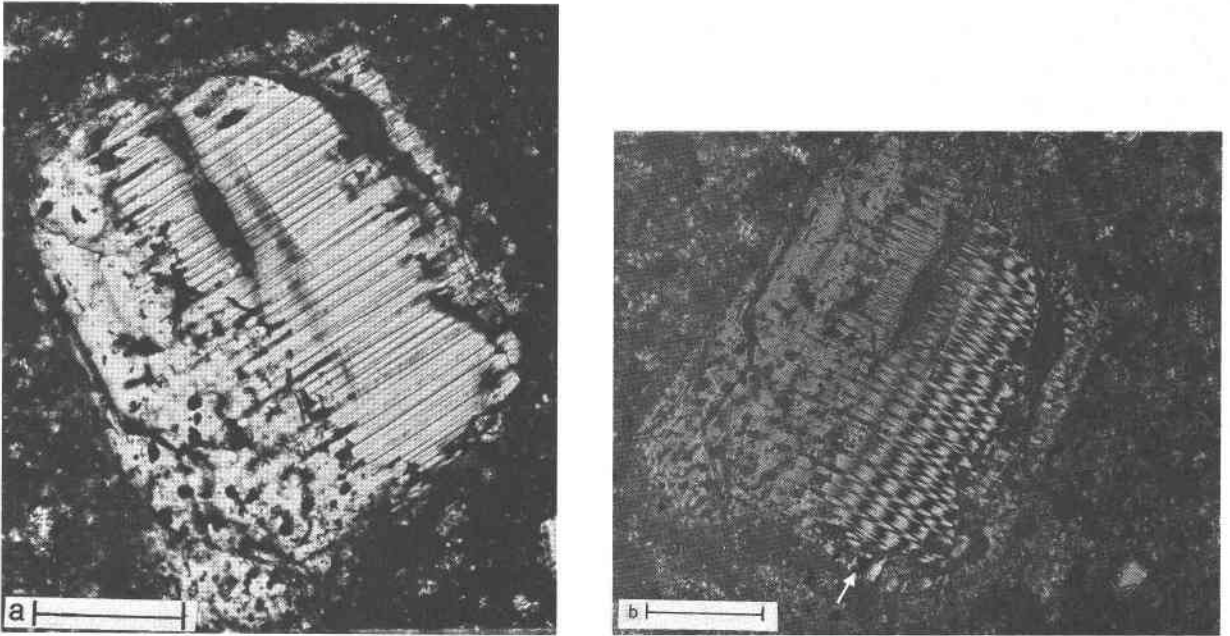
FIG. 12. Photomicrograph of a rimmed clinopyroxene xenocryst in 77115,50, (a) in bright field, showing the "ribbed" core and the narrow rim on the right; (b) showing interference fringes. Note in (b) that in the core, the exsolved intermediate pigeonite $Wo_{11}En_{44}Fs_{45}$ of high R.I. shows the strong shift to the right, as compared to the augite $Wo_{42}En_{34}Fs_{24}$. The rim augite $Wo_{35}En_{46}Fs_{19}$ is more magnesian (less iron) than the core, and the exsolved rim phase is a magnesian pigeonite $Wo_{12}En_{67}Fs_{21}$ similar to that in the surrounding matrix. The zero fringe in the rim is shifted about 1 order to the left (arrow). The complex area on the left will not be discussed in this paper. Transmitted light; bar scales 100μm.

microprobe analyses are carefully monitored to make sure that analyses were not obtained from mixtures; (2) areas in the rim having greatest fringe shift are analyzed for both the host and the exsolved lamellae. Prior to the interference microscopic study we were not aware that the rim pyroxene was also exsolved, and that analysis of the rim represented mixtures. Examination of the interference photomicrograph revealed the necessity for repeating the analysis.

Other pyroxene xenocrysts in 77115 were found to be much more complicated. We now realize that it would not be possible to make much sense out of a large number of analyses of such complex crystals if interference microscopic data were not available.

Lamellar structures produced by static and shock deformation

Lamellar structures produced by static deformation (slow strain rate) consist of fractures, mechanical twinning, and deformation lamellae. Lamellar structures produced by shock deformation (high strain rates) are more complex. As a group, not specifically identified, they are referred to as planar features, a general but not very informative term. They consist of closely spaced decorated or undecorated subparallel microfractures, deformation lamellae similar to those produced by static deformation, and low-index shocked glassy lamellae (lamellae with partial or complete vitrification; Chao, 1967). Each type of lamellar structure characterizes not only the mechanism of yielding but also the magnitude of pressure required to produce it. Proper identification would not only eliminate confusion but also specify the significance of its presence.

In the study of microstructures produced by shock deformation, unless extensive diffusion, flow-melting, and a high-pressure phase were produced, electron microprobe studies are not involved. The primary techniques fall on interference microscopy, single crystal X-ray diffraction, and transmission electron microscopy and diffraction.

Figure 7 shows a fragment of a single crystal of quartz produced experimentally by shock loading with an estimated peak pressure of about 130 kbar. (R. Graham, verbal communication, 1969). It shows a widely spaced subparallel set of "lamellae". As mentioned above in the section on open and filled fractures, these have been identified by interference microscopy as open fractures, as the fringe shifts across them are to the left against ω of quartz in 1.540 oil which penetrated and filled these micron-width

fractures (Fig. 7a) and to the right in 1.560 oil (Fig. 7b).

Deformation lamellae are characterized by reduced and strain birefringence, with compression on one side and tension on the opposite side of the lamella (Carter *et al.*, 1964). The total reduced birefringence is generally less than 0.004, therefore accurate measurement of birefringence is highly desirable. In my experience such lamellae occur in weakly shocked rocks, which are comparatively rare among the natural shocked samples I have studied. Illustration of deformation lamellae is not given here.

Figure 13 shows a fragment of a large crystal of quartz collected from a shocked pegmatite from the impact structure of Lake Lappajärvi in Finland. It contains several sets of closely spaced subparallel planar structures. The dominant sets are parallel to $\{01\bar{1}3\}$. It is desirable to determine the nature of these planar structures. Under the interference microscope, regardless of the index of the immersion oil (R.I. as high as 1.560), the interference fringes across these lamellae shift to the left, demonstrating that they are not open fractures. They may be shock lamellae having an R.I. of about 1.473 and consisting of about 90 percent silica glass and about 10 percent quartz, or they may be sealed partly-healed open fractures. Further study in transmission microscopy is necessary. Similar application combined with X-ray diffraction for a suite of shocked quartz grains representing progressive stages of vitrification in the solid state has been reported previously (Chao, 1968). Vitreous shock lamellae are common in shocked plagioclase from granodiorite fragments in suevite, an impact ejecta from the Ries Crater (Chao, 1967), and from shocked lunar rocks (Chao *et al.,* 1970b; Christie *et al.*, 1973).

Disadvantages and minor problems encountered in applications

Any new application of a technique is likely to have disadvantages and pitfalls. The single major disadvantage of the interference microscope may be its cost, which is nearly twice that of a modern research petrographic microscope. One must then balance this cost against the additional information one can obtain and the amount saved by the reduced number of electron microprobe analyses required.

Problems encountered in the application of interference microscopy to the study of doubly polished thin sections are as follows: (1) Matched objectives of the interference microscope for the measuring beam are not centerable by the operator; hence the center
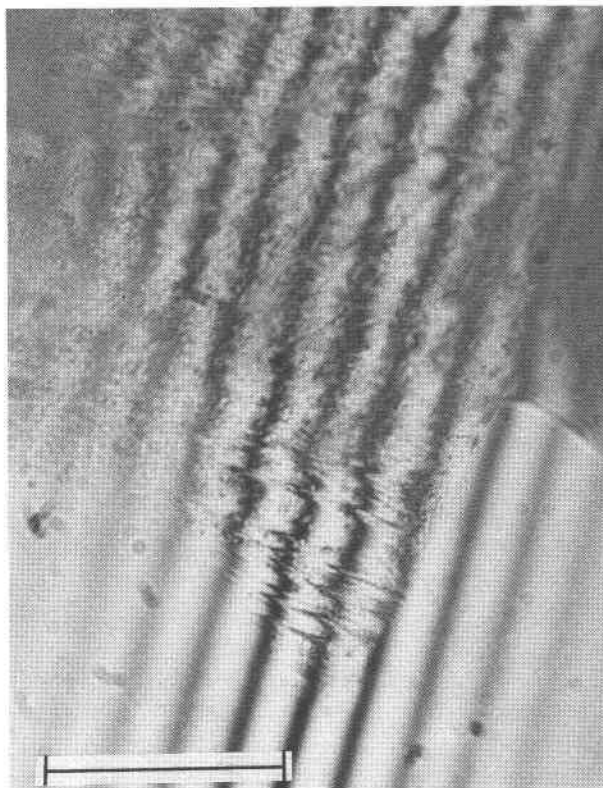

FIG. 13. Interference photomicrograph of a fragment of shocked quartz from a large single crystal, Lake Lappajärvi, Finland. Note the shift to the left of the low-index lamellae, which is consistent regardless of the change in R. 1. when the immersion liquid is changed (as high as 1.560). See text for interpretation. Transmitted light; bar scale 100μm.

of rotation of the stage may not coincide with the center of the measuring beam objective. (2) In doubly-polished thin sections, the minerals stand significantly higher in relief than the softer epoxy. (3) Proper identification must be made of fringes for the zero fringe method. The first situation renders precise discrimination of α' and γ' of a mineral of low birefringence difficult. The second makes the determination of the R.I. of the mineral with respect to the epoxy impossible. The third will render the measurement of fringe shift in error if the fringes are misidentified. Each of these problems is minor, as precise R.I. measurement may not be of prime importance in the application. The first problem can be remedied if the center of rotation of the stage is noted and a grid-type ocular micrometer is used. The second problem can be remedied if covered thin sections with only one epoxy as embedding medium are used. The use of the latter is, however, often unnecessary, because in most petrologic problems regarding variations in structure or composition, only Δn is of importance.

The relief or thickness variations do not affect the Δn determination as Δn between two minerals with respect to the epoxy (even of unknown R.I.) is constant.

The third problem can be corrected if some precautions are taken. Before using the zero fringe method for mineral identification in thin sections, it is necessary to identify the fringes along which fringe shifts are measured. As observed, the zero fringe position in a mineral in a thin section 30μm thick is different from that in the same mineral in a thicker section. Also the interference color changes along individual fringes as the thickness of the mineral increases. Hence the fringe shift from one mineral to the adjacent mineral along the same fringe must be carefully identified or traced and not be dependent on the color of the fringe. The greater the difference of R.I. between the minerals, the stronger is the effect of increasing thickness on fringe color changes. Fortunately for a standard thin section, the thickness of 30 to 35 μm is generally less than that which causes the fringe colors to change, resulting in misidentification of the interference fringes. Nevertheless, as a safeguard, and in order to avoid this possible pitfall, it is advisable to explore and trace the fringe color changes for each mineral of different R.I. along the edges or openings of the thin section where the mineral is in direct contact with the embedding medium or with a mineral of known R.I. As a general rule, if two adjacent minerals differ greatly in R.I., caution should be exercised for proper fringe identification in order to measure the correct fringe shift. Except for thick wafers for the study of inclusions in a particular sample, the problem of misidentification of fringes, in my experience, does not happen very often. The precaution discussed here is for the determination of the absolute values of R.I. of a suitably oriented mineral in a thin section. The illustrated applications given in this paper are representative examples. Some of the procedures, such as identification of grain shape by fringe characteristics involved in inclusion studies, need to be refined. Obviously, wider applications should come as more users adopt the interference microscope for mineralogic and petrologic investigations.

Summary

Interference microscopy has direct and valuable applications to mineralogic and petrologic investigations. These are:

1. In the study of the optical properties of mineral grains, I recommend the use of interference microscopy as part of the routine procedure. In the determination of principal indices of mineral grains, the interference method is much faster and an order of magnitude more precise than the traditional Becke line single or double-variation method. The interference method should be integrated with X-ray single-crystal study, electron microprobe analysis and electron microscopy.

2. In the study of petrographic thin sections, interference microscopy is a method for quick identification of minerals. If the R.I. of the embedding medium is known, and if quartz is present or a plagioclase of known index and composition can be used as a reference mineral, then all minerals, including micron-size inclusions in the thin sections, can be explored. It can portray in detail intramineral and intermineral index variations and pinpoint specific grains and traverses for electron microprobe analysis. If desired, it can be used to establish correlations between index variation and variation in composition of any mineral of petrogenetic significance.

3. Interference microscopy provides a means of identifying micron-size inclusions where they are not readily accessible for study by X-ray diffraction, electron microprobe, or transmission electron microscopy.

4. Interference microscopy is a powerful tool for the study of microstructures such as closely-spaced parallel fractures, twinning, zoned phenocrysts, exsolution, rimmed crystals, and lamellar structures produced by static and shock deformation.

5. The transmitted light interference microscope is an expensive, precisely engineered instrument. Justification for its extensive use must rest with the acquisition of additional information not obtainable with a normal research petrographic microscope and with the savings and specific knowledge gained for planning electron microprobe analysis.

Appendix

Procedures for measuring fringe shifts

In a loose grain of specific R.I. but of variable thickness, the position of the zero fringe within the grain is dependent on the thickness. In a thin section of uniform thickness, the position of the zero fringe is dependent solely on the R.I. Hence the zero fringe is used for measuring fringe shifts in thin sections but not in loose grain mounts. For inclusions a few microns in size, and particularly if the index of the

inclusion is drastically different from the enclosing mineral, a separate procedure must be used.

The following procedures were developed, and whenever possible the soundness of the procedure was checked with known mineral species or immersion liquids of known and carefully calibrated R.I. Thin sections were cut and polished on the edges so that the actual thickness of the rock slice and the mounting media could be measured with the Vickers-A.E.I. Image Splitting Device. As wider applications of interference microscopy to mineralogic and petrologic problems are developed, the procedures described below may well be modified and refined. In my opinion, however, these procedures are more than adequate for obtaining the precise optical data desired.

1. Procedure for measuring fringe shift in a loose grain mounted in oil after the principal planes of vibration have been determined with the spindle stage on a petrographic microscope.
    a. Place grain in extinction position for the index chosen for measurement, then uncross the analyzer.
    b. Select an easily recognizable point in the grain for measurement.
    c. Spread the interference fringes to the desired width in maximum interference contrast using Na D illumination. Rotate the fringes so that the fringes are normal to the contact between the edge of the grain and the immersion oil.
    d. Rotate the vertical cross hair in the eyepiece to be precisely parallel to the fringes in the immersion liquid.
    e. With the wedge compensator, measure and record the fringe shift Γ_1 with reference to the vertical cross hair between the point chosen for measurement and the same fringe in the oil.
    f. Change to another oil (about 0.004 higher or lower than the previous oil) and make another fringe shift measurement Γ_2 at the chosen point. The measured index of refraction of the grain is:

$$n_x = \frac{\Gamma_2 n_{\text{oil}_1} - \Gamma_1 n_{\text{oil}_2}}{\Gamma_2 - \Gamma_1}$$

2. Procedure for measuring α' of mineral A against α' of mineral B in thin section:
    a. Check the center of cross hair to see if it coincides with the center of rotation of the stage of the measuring beam. If not, note the displacement and use a grid-type ocular micrometer.
    b. Set mineral A to extinction so that α' is parallel to the vibration direction of the polarizer.
    c. Place point of measurement of mineral A at the center of rotation and set the zero fringe in white light illumination parallel to the vertical reference line. Record the wedge compensator reading.
    d. Place point of measurement in the adjacent mineral B at the center of rotation. Rotate so that α' of mineral B is in extinction and parallel to the vibration direction of the polarizer. The zero fringe should lie either to the left of the vertical reference line if α' of mineral B is less than α' of mineral A, or to the right if it is greater.
    e. Use the wedge compensator and move the zero fringe to coincide with the vertical reference line. The difference is the phase shift corresponding to Δn of α' between A and B. This measured phase shift is identical to the measured zero fringe shift across the grain boundary between minerals A and B if both α''s are parallel (i.e., the extinction positions of both minerals happen to coincide, a situation that may be found in a thin section). The same procedure can be used to measure the mean index of refraction of a mineral either against the ω of quartz if present or the mean index of a reference mineral (analyzed mineral).

3. Procedure for measuring the fringe shift of an inclusion a few μm in diameter having R.I. n_i quite different from that of the host mineral grain of known (or previously determined) index n_h:
    a. Using white light, identify and place the zero fringe of either α' or γ' of the host mineral parallel to the vertical cross hair. Move the center of the inclusion in one of its extinction positions to also coincide with the vertical cross hair. Record the reading on the compensator.
    b. With the wedge compensator, move the zero fringe either to the left ($n_i > n_h$) or to the right ($n_i < n_h$) of the vertical cross hair so that the different orders of interference fringes will move into or out of the grain respectively. The direction for moving the fringes should be determined before the compensator reading is recorded.
    c. Record the reading on the compensator at a point where the zero fringe in the inclusion appears under the vertical cross hair.
    d. The fringe shift Γ is the difference between the

second and the first reading in nm, which may cover several wave lengths or orders.

e. Measure the diameter d of the inclusion in nm with the Vickers–A.E.I. Image Splitting Device.

f. The index of the inclusion:

$$n_i = n_h - \Gamma/d \qquad (n_i < n_h)$$

$$\text{or } n_i = n_h + \Gamma/d \qquad (n_i > n_h)$$

If it is desirable to measure the fringe shift in monochromatic light, the number of wave lengths of fringe shifts must first be determined in white light, as the zero fringe cannot be identified in monochromatic light. Then proceed exactly as with white light. In the case of micron-size inclusions in loose grains, the diameter of the inclusion may be directly measured. The procedure used is identical to that of the thin section when the point being measured is stationary, as in all cases.

Acknowledgments

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