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OPTICAL EXTINCTION OF ANORTHITE AT HIGH TEMPERATURES

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INTRODUCTION

Investigations by Brown *et al.* (1963) recently provided new insight on the sub-solidus phase relationships for crystals of anorthite compositions. Their *x*-ray photographs, taken of anorthite crystals at temperatures between 25° and 350° C., showed the *c*-reflections—that is, those for which $h+k$ is even whereas l is odd—to become increasingly diffuse and eventually disappear as 350° C. was approached. Thus a continuous and reversible structural transformation between 25° and 350° was demonstrated. Only in specimens quenched from temperatures in the range 1100°–1540° were structural stages comparable to those observed between 25° and 350° C. retained at room temperature, being closer to the 350° C. stage to the extent that the quench temperature approached 1540° C.

Using anorthite as an example, this present study reveals that transformations from one phase to another, as temperature is raised, may be determined relatively quickly by measuring the rate of change in the privileged directions of crystal sections as temperature is raised. Thus, for example, the change in value of the extinction angle, $[100]:\alpha'$, for (001) cleavage flakes of anorthite, as temperature is increased, offers further confirmation of a continuous structural transformation at about 350° and, moreover, indicates that a displacive transformation may also occur at about 800° C. The results from this present study indicate that a polarizing microscope used in combination with a heating stage may provide a ready means for detecting phase transformations at elevated temperatures. Details of the study follow.

METHOD AND APPARATUS

A high pressure mercury lamp (Zeiss HBO 200) was used as the light source for an ordinary polarizing microscope equipped with a Leitz

(1350°) heating stage. A light filter (Zeiss Hg 546-63) was located between the hot stage and the objective lens of the microscope so as to filter out the light resulting from incandescence of the crystal specimen at high temperatures yet transmit the strong 5461 Å mercury line of the source light which had passed through the crystal. To provide more precise measurements of extinction positions, the half-shadow-wedge (*Halbschattenkeils*) of Mace de Lepinay (Burri, 1950, p. 156) was used in conjunction with a Wright ocular equipped with a cap analyzer. Consequently extinction positions could generally be reproduced to a few tenths of a degree. However, cleavage traces could not be measured with equal precision and hence the extinction angles here reported could all be subject to a systematic error of a degree or two.

Prior to making extinction measurements on crystals, the temperature-indicating scale for the Leitz heating stage was calibrated by observing materials with accurately known melting points and plotting a calibration curve. This was particularly important because the temperature at the upper surface of the quartz glass disc on which the crystals lay differed from the temperature indicated on the scale by as much as 120° C. at high temperatures. Thus when the scale read 1200° C. the temperature atop the glass disc was only 1080° C. The difference decreased for lower temperatures so that at about 400° C. the scale read true. The increasing difference at higher temperatures was probably the result of increasing heat loss at the upper disc surface caused by convectional air currents.

Extinction measurements, as temperature was increased, were made on cleavage flakes and randomly oriented sections of anorthite crystals. These were ground to an appropriate thickness, then polished on both sides to increase their transparency. The optimum thickness varied with the birefringence of the plate. In general those plate thicknesses were best if they resulted in retardations approximating $(n+1/2)\lambda$ for the 5461 Å line of the source.

RESULTS

The anorthite used was from the Vesuvius locality as described by Kratzert (1921). Accepting Kratzert's analysis to apply to this specimen, its composition is about 97.1% An. Moreover, anorthite from this locality appears to possess low-temperature optics, a fact which seemed desirable from the point of view of this study.

Many of the anorthite sections studied showed changes in their privileged directions of only 2 to 5° as their temperatures were increased from 20° to 1080° C. Others, however, proved particularly interesting because, in response to the same temperature change, their privileged

directions changed up to 22° . The degree to which the privileged directions of a section change with temperature probably depends upon (1) the angular attitude of the section to the optic axes at room temperature and (2) the directions (in relation to the section) along which the optic axes migrate as temperature is increased. As may be inferred from Burri's (1950, p. 293 and Fig. 166-8) discussion, sections more nearly perpendicular to an optic axis are rather likely to show large changes

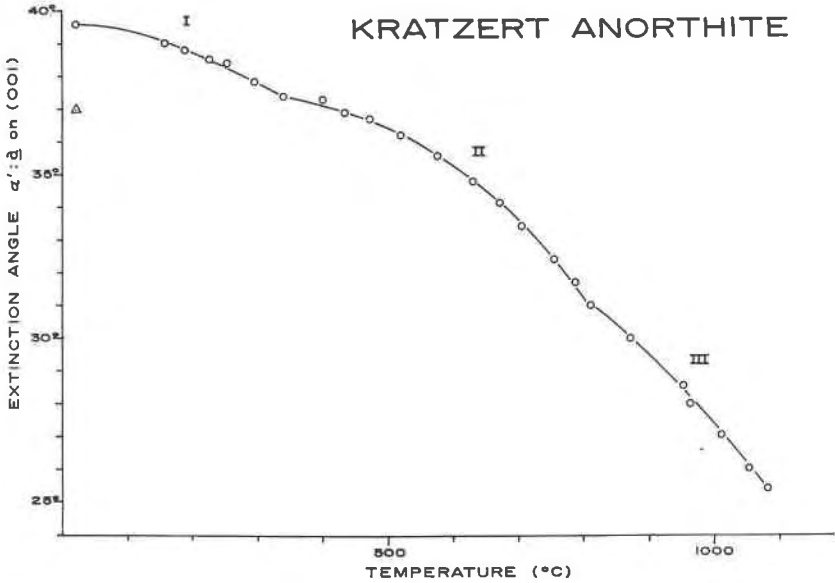


FIG. 1.

in privileged directions in response to small, thermally produced migrations of the optic axes.

Results for a (001) cleavage flake of anorthite (Fig. 1) show a continuous decrease in value of the angle between α' and the trace of the (010) cleavage—that is, of the angle $\alpha' : a$ —as temperature increases. Each point plotted in Fig. 1 is actually the average of six or seven closely spaced observations. The data seem to fit along the three curves labelled I, II and III in Fig. 1. Using an IBM 1620 computer, the equations for these curves were computed to be:

$$\begin{aligned} \alpha' : a &= 39.632675 - 0.00166886 T - 0.00001437 T^2 & \text{I.} \\ \alpha' : a &= 35.267848 + 0.01453075 T - 0.00002430 T^2 & \text{II.} \\ \alpha' : a &= 36.851779 + 0.00381852 T - 0.00001335 T^2 & \text{III.} \end{aligned}$$

where T is temperature in degrees centigrade.

The value of angle $\alpha':a$ actually observed at room temperature was 39.6° . Inserting a value for T of 20° C. in equation I yields the same value. The value agrees with the value of 40° cited by Winchell and

TABLE 1. OBSERVED AND CALCULATED EXTINCTION ANGLES FOR (001)
FLAKES OF KRATZERT ANORTHITE

Temperature °C	Extinction Angle ($\alpha':a$) on (001)				
	Observed ¹	Predicted from			
		Eq. I	Eq. II	Eq. III	Eq. IV
20	39.6	39.594			39.587
157	39.0	39.016			38.952
187	38.8	38.818			38.766
225	38.5	38.530			38.506
252	38.4	38.299			38.305
295	37.85	37.890			37.956
340	37.40	37.404			37.554
400	37.30		37.191		
433	36.90		37.002		
472	36.70		36.711		
520	36.20		36.251		
578	35.55		35.546		
630	34.80		34.775		
670	34.15		34.092		
705	33.40		33.431		
755	32.40		32.383		
788	31.70		31.625		
810	31.00		31.090		
872	30.00			30.029	29.927
953	28.60			28.365	28.300
962	28.00			28.169	28.112
1011	27.05			27.066	27.060
1052	26.00			26.093	26.145
1081	25.45			25.378	25.479

¹ The values in this column are the averages of 6 or 7 readings, rounded off to 0.05. Accuracy to 0.05 is not claimed.

Winchell (1951, p. 286) for pure anorthite but is slightly less than that indicated by Deer *et al.* (Vol. 4, 1963, Fig. 54).

The data fit the curves extremely well. The differences between the observed and predicted values of angle $\alpha':a$ were surprisingly small (Table 1). Of the seven points from which curve I was derived, only once did the observed and the predicted angle differ by as much as 0.1° . Of the eleven points from which curve II was derived, in only two did ob-

served-minus-predicted values differ by as much as 0.1° , being 0.109° and -0.102° respectively. For curve III agreement between observed and theoretical values were not quite so good; the maximum differences observed were 0.23 and -0.17° , respectively. The greater difficulty of making the observations at higher temperatures probably accounts for this.

Curve I represents the optical expression of the continuous structural transformation which produced the disappearance of the *c*-reflections in the *x*-ray study by Brown *et al.* (1963); the intersection between curves I and II, calculated from their equations to be about 341° C., probably represents the temperature at which the aforementioned structural changes achieved completion—this value agreeing fairly well with the 350° C. temperature observed by Brown *et al.*

In the absence of supporting *x*-ray evidence, curves II and III cannot be interpreted unambiguously. Interestingly, the intersection between curves II and III can be calculated to be 797° C., a value which is in fair agreement with the location at 780° C. of a sharp, endothermal DTA peak reported for anorthite from Pesmeda, Italy by Köhler and Wieden (1954). Thus the inflection at about 797° C. in Fig. 1 might indicate inversion from a low to a high structural form of anorthite.

One possible interpretation of the results in Fig. 1 is that anorthite at atmospheric pressure undergoes a continuous structural transformation which comes to completion at about 341° C. Above 341° C. there is a structural form which remains stable up to about 797° C. and then undergoes a displacive transformation. Above 797° C. it is possible that a continuous structural transformation similar to that which occurred below 341° C. is resumed. This last statement is particularly conjectural and is merely based on the observation that curves I and III resemble segments of a single curve which is interrupted by curve II. Thus, if we fit a curve to the combined observed data of sets I and III we obtain

$$\alpha':a = 39.650262 - 0.00297704 T - 0.00000937 T^2 \quad \text{IV.}$$

Despite the fact that the six observations underlying curve III were added to the seven observations underlying curve I, equation IV differs little from I. The maximum difference between the observed and predicted values of $\alpha':a$ for the thirteen points on which equation IV was based is about 0.1° for all except the observation at 953° C. (for which the difference amounts to almost 0.3°). This latter observation, however, probably involves a relatively large experimental error. Curve IV, not drawn in Fig. 1 because it coincides too closely with curves I and III, intersects curve II at two temperatures, 362° C. and 810° C. These differ somewhat from the comparable values, 341° and 797° C.

For anorthite flakes which showed large changes in their privileged directions when heated, it was sometimes observed that upon cooling to room temperature the originally observed privileged directions at that temperature were not restored. Provisionally stated at this time, because further experimental verification is desired, a change in the room temperature extinction angle occurs if (001) flakes of anorthite are heated to above 800° C., the suspected inversion temperature. The degree to which the crystal's temperature exceeds 800° C., appears to control the amount of change produced. In two instances when a crystal plate was heated to 1080° C. and cooled to room temperature, the extinction angle (and optical orientation?) present at about 430° C. was "quenched in." An example is the extinction angle at room temperature for the (001) flake of anorthite after it was heated to 1080° C. (Fig. 1).

The migration of the optic axes as anorthite is heated to 1080° C. is currently being determined and will be described at a later date.

In conclusion, investigations of the change in privileged direction with temperature such as reported here may serve as excellent reconnaissance studies to precede temperature-controlled *x*-ray investigations of crystals. Such studies should permit *x*-ray photographs to be confined to the more strategic temperatures and thus reduce the number of temperature-controlled *x*-ray exposures necessary to determine the nature of thermally induced phase transformations.

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calculated the parameters in Eqs. I-IV as well as the predicted values cited in Table 1.

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COMBINED ROCK AND THIN SECTION MODAL ANALYSIS

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During recent years the theory of modal analysis has been extensively developed (Chayes, 1956), and has been shown to be a valuable tool in the precise determination of the proportions of minerals present in fine to medium grained rocks. It is also being used extensively in the estimation of areal variation of granitic complexes (Whitten, 1961).

Similarly, techniques and apparatus have been described to facilitate point count analysis on coarse grained rocks (more especially granites) which otherwise would have required a large number of counts over several thin sections, (Jackson and Ross 1956; Emerson 1958; Fitch 1959; Smithson 1963).

While studying "granitic" rocks from South Greenland (Nesbitt 1961 and in preparation) the writer encountered difficulty in obtaining reliable modal analysis data from rock types which were of medium to coarse grain and were characterized by irregularly spaced microcline and plagioclase phenocrysts amounting to about 20% of the rock. The results given in Table 1 indicate that the modal analyses of single thin sections of rocks of this texture are most unreliable.

The writer considers that the modal analysis of rock slabs is an unsatisfactory solution to the problem because fine-grained components,

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