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PREFACE

Anisotropic crystals become amazingly tractable in optical crystallography if they can be rotated about an axis that is parallel to the stage of a polarizing microscope. Although single-axis stages that permitted this were available in the 1860s, they seem to have fallen into disuse until their recent revival by Joel, Tocher, Hartshorne, Saylor, and in particular by Ray E. Wilcox of the U.S. Geological Survey. Wilcox vigorously espoused single-axis techniques and introduced a very inexpensive version, which he named the *spindle stage*. The term was so apt that it has practically become a synonym for *single-axis stage* and is so used in the title of this book.

The spindle stage itself has proved equally apt. With it, undergraduate students who have mastered indicatrix theory can quickly orient a small crystal or crystal fragment so that all of its principal indices can be accurately measured by the immersion method. Wilcox (1959, p. 1272) enumerates the following advantages of the spindle stage for study of individual crystal fragments by the immersion method:

1. *Any* direction in the crystal can be rotated into the plane of the microscope stage. Consequently, all three principal indices may be determined from the same crystal fragment.
2. No corrections are required for angular rotations.
3. Either orthoscopic or conoscopic illumination may be used (and readily alternated).
4. It is simple and inexpensive (costing roughly one-thirtieth the price of a universal stage).

After the writer introduced his classes to spindle-stage techniques, the response was enthusiastic from all sides. There is little doubt that students in geology, mineralogy, ceramics, and chemistry should be instructed in these techniques early in their careers. To aid in this instruction, the writer has herein attempted to provide a unified treat-

ment of spindle-stage techniques. These techniques should permit a student to determine the optic axial angle $2V$ (and locate X, Y, and Z) with an accuracy that even an expert, if using a universal stage, would be pressed to achieve. Moreover, such errors in the measurement of the refractive indices α, β, and γ as result from crystal misorientation will become inconsequential. Nor is there need to extrapolate, as is frequently necessary with a universal stage, from a measured nonprincipal index, α' or γ', to obtain a principal index α, β, γ. The principal index can always be measured directly if within the range of the oils.

In preview, Chapter 1 introduces a spherical coordinate system to simplify and standardize the stereographic plotting of spindle-stage data. Equations are developed so that, if the coordinates for two different optic directions are known, one can calculate the angle between them or the coordinates of the line perpendicular to these two directions. Spindle-stage methods for orienting anisotropic grains are described and discussed for uniaxial crystals (Chapter 2) and biaxial crystals (Chapter 3). Chapter 3 also illustrates graphic methods whereby, from the stereographic plot of the crystal's extinction curves, one can determine $2V$ to within a degree or two. Several amateur mineralogists currently used this technique with great success to identify unknown minerals in their home laboratories. The $2V$ of unknown inclusions in volcanic glasses or in diamond crystals could thus be determined, subject to error due to refraction, without the need to crush the host.

Chapter 4 discusses the dispersion of refractive indices (n) with wavelength (λ) and describes the methods of measuring a material's refractive indices at different wavelengths by the dispersion method (λ variation) and by the double variation method (λ, T variation). Accuracy of these techniques is improved through a simple linear regression analysis of the data, a linearized Sellmeier equation (Eq. 4-6) serving very successfully as a model. The two constants, a_0 and a_1, thereby obtained permit the refractive index to be calculated, often to within 0.0002, for any desired visible wavelength. Eventually, it is hoped, compendia of optical data will report a_0 and a_1 values for each principal refractive index and state the wavelength range within which these values pertain. This will be more concise and informative than giving the values of each index for the C, D, and F Fraunhofer lines.

Focal masking (dispersion staining) techniques are discussed (Chapter 5) because, employed in conjunction with a monochromator and with a photometer in place of the eye, they may eventually permit highly objective determinations of wavelengths of match between grain and oil (even beyond the visible range). Moreover, through focal masking, a normally colorless solid may be caused to display vivid color in an

appropriate immersion oil (the Christiansen effect). Walter C. McCrone Associates, among others, immerse powders or dust samples in appropriate oils so that impurities – for example, asbestos particles – display vivid colors and can thus be detected even though present in such small amounts as to be indetectable by X-ray techniques. Trace phases in experimental-run products similarly become detectable.

Accurate methods of measuring crystal extinction, by eye and photometrically, are discussed in Chapter 6. This chapter also discusses EXCALIBR, the latest version of the Bloss-Riess (1973) computer program for analyzing spindle-stage extinction data. EXCALIBR calculates extremely accurate values of $2V$ and of the positions of the five significant optic vectors, namely, the two optic axes plus the indicatrix axes X, Y, and Z. If extinction measurements made at several different wavelengths are submitted to EXCALIBR, subroutine DISPER calculates the angular changes of the five optic vectors with wavelength and whether the change is significant. Where such directional dispersion is not pronounced, experimental error may obscure the results. Such error can be reduced by grinding the crystal approximately into a sphere and immersing it in an oil of an index equal to β for the crystal (for that wavelength). Photometric measurements of crystal extinction will provide input data for EXCALIBR for wavelengths beyond the visible. The optical properties of crystals can thus be determined beyond the visible range, and the actual wavelength limits will largely depend on the wavelength range of sensitivity for the detector(s) used, of output from the source, and of transparency for the grain, lenses, and slides interposed in the light train of the microscope. It should soon be possible to measure $2V$ for a crystal at 100-nm intervals over, say, a 200- to 2000-nm range, by using more than one type of detector. Changes in $2V$ as infrared absorption bands are approached should be of interest.

Further research capabilities of the spindle stage, particularly one that accommodates an X-ray goniometer head, are cited in Chapters 7, 8, and 9. For example, if crystals can be fashioned into polished cylinders or spheres, certain of their optical properties [$2V$; birefringences $(\gamma - \alpha)$, $(\gamma - \beta)$, $(\beta - \alpha)$; and locations of X, Y, and Z] can be determined at elevated temperatures (to melting in many cases) or at temperatures below 25°C by employing a heating or cooling stage. Consequently, phase transitions that occur as a crystal is heated or cooled can be detected optically. Bloss (1978) thus demonstrated a minimum (at 350°C) in the $2V$-versus-temperature curve for the Miyakejima anorthite. This minimum apparently marks completion of the transition from "primitive" to "body centered" that anorthite undergoes when heated from 25°C to above 350°C.

Equations are also provided whereby, after a spindle-stage study, one can calculate the goniometer arc setting required to orient, parallel to the X-ray beam or dial axis, the c axis of uniaxial crystals and X, Y, or Z for biaxial crystals. A prior spindle-stage examination may thus reduce the number of X-ray photographs required to orient a crystal, particularly if it is anhedral. At the same time, one can determine the angles between X, Y, and Z and the crystallographic axes of monoclinic or triclinic crystals. Chapter 7, which describes these techniques, necessarily presupposes a familiarity with single crystal X-ray methods. However, those lacking such may bypass Chapter 7 without creating undue difficulties in reading Chapters 8 and 9.

The techniques described in this book are so useful and powerful as to be most exciting to the writer. He hopes that the brief book that follows this rather lengthy preface will do them justice.

The writer is grateful to Tom Quigley, who drew the major number of illustrations on which this text depends so heavily, and to Sharon Chiang and Martin Eiss, who also drafted or modified illustrations. All suggestions for improving this book or the techniques involved will be welcome. Readers are invited to submit computer programs for optical data, particularly if adopted to hand-held calculators, for publication in a possible future edition of this book.

As in my previous textbooks, the figure captions are lengthy, to say the least. Some readers may decry the resultant double coverage, but most students (judging from past comments) truly appreciate the practice. These captions sometimes provide an alternative approach and, in any case, ensure that each illustration will retain its desired impact even when, despite expert editing, it necessarily appears overleaf from its discussion in the text.

Last, but by no means least, the writer wishes to express his debt to Max Carman and Ray Wilcox, enthusiastic colleagues in the practice of optical crystallography, who very kindly read the manuscript and offered many suggestions for its improvement. The addition of the "360° option" to EXCALIBR is due to the work of Max Carman, who developed it when modifying the original Bloss-Riess (1973) program.

My deep thanks are due Mr. Michael Rohrer for reorganizing the Bloss-Riess program into EXCALIBR. For help in reading proof I am grateful to Brian Cooper, James Downs, Russell Guy, and especially to Dr. Alex Speer and my assistant, Mickey Gunter.

F. Donald Bloss

Blacksburg, Virginia
October 1980