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A MODIFIED SPINDLE STAGE PERMITTING THE DIRECT MEASUREMENT OF 2V

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Abstract

A modified spindle stage is described, with certain advantages over previous models. Conoscopic observations can be made using the short working distance of an ordinary polarizing microscope; the orientation of a mounted crystal can be adjusted by means of perpendicular slides and a ball-joint. Thus direct determinations of optic constants, including optic axial angle, are possible.

The construction, uses and operation of the stage are described and some results obtained during an investigation of the potassium-barium feldspar series are presented.

Introduction

Many different designs of a single-axis rotation apparatus for orienting a crystal in an oil-cell under a polarizing microscope have been proposed (Fedorov, 1891; Bernal and Carlisle, 1947; Hartshorne and Swift, 1955; Steinbach and Gibb, Jr., 1957; Wilcox, 1959, 1960; Fisher, 1960, 1962; Oppenheim, 1962; Hartshorne, 1963); Fisher (1962) used such a device with a temperature controlled stage. All spindle stages of this type were designed for orienting a grain in a suitable direction for determining the principal indices of refraction. Wilcox (1959) described an indirect method of 2V determination by using Fresnel's construction, but his spindle stage does not permit an angular displacement of a mounted crystal. In this respect Fisher's stage is advantageous. He used the "Umirg" as an axial-angle apparatus for direct determination of 2V conoscopically, for which the converger was mounted in a dovetail. A goniometer head was used to achieve the necessary movement of the crystal for proper orientation. He also described Fresnel's construction for indirect determination of 2V. In Steinbach and Gibb's (1957) device the crystal is mounted directly on the tip of the spindle, which restricts independent adjustments to orientate the crystal. Although the spindle-axis can be tilted relative to the microscope stage to set one of the bisectrices vertical, the optic axial plane will not then remain vertical when the spindle-axis is rotated, since it is not perpendicular to the optic axial plane. Thus the direct determination of 2V is not possible. In the present spindle stage, the modifications to the existing designs are such as to allow very short working distances; this permits the use of a standard polarizing microscope for conoscopic observation and the direct measurement of the optic axial angle.

Construction

Figure 1 shows a diagrammatic representation of the stage. It is clamped to the microscope stage by the fixing screw, after removal of the inner circular plate of the microscope stage. The main spindle is a steel rod which passes through a brass tube; rotation of the crystal through 360° can be achieved by rotating this spindle with the main knob, the amount of rotation being read from a drum graduated at 2° intervals. At the end of the brass sleeve through which the main spindle passes, there is, in addition, a coupling device to allow the rotation to be read on a steel protractor graduated at 1° intervals through 180°; this scale can be read to 3′ by means of the vernier attached to the brass tube. The main spindle can be used for the preliminary adjustment of the orientation of the crystal, and the 180° scale and the vernier coupled after the crystal has been set approximately in the most suitable orientation.

At the other end of the main spindle, there is a detachable threaded head for mounting the crystal. This head has two perpendicular slides on which is mounted a ball-joint with a pointed tip (Fig. 2a); a glass fibre to carry the crystal is cemented to this tip with Durofix. This head allows for independent lateral and tilting adjustments to permit accurate

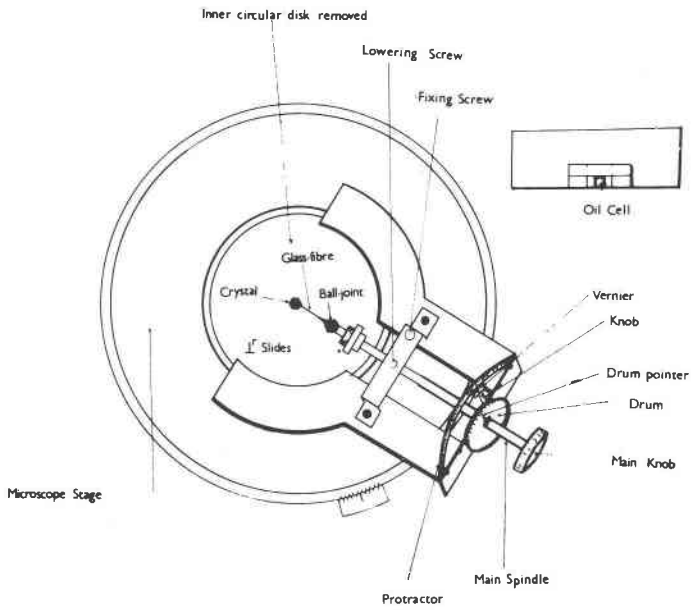

Fig. 1. Diagrammatic representation of the spindle stage.

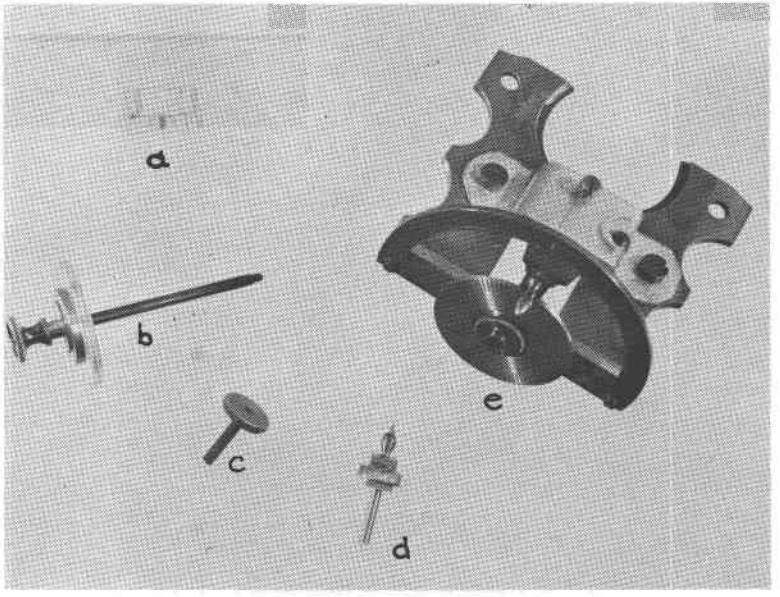

FIG. 2a. Photograph of the different parts of the spindle stage: (a) oil-cell, (b) main spindle with the drum-pointer, (c) fixing screw, (d) head (showing perpendicular slides and the ball-joint), (e) drum, protractor, vernier etc.

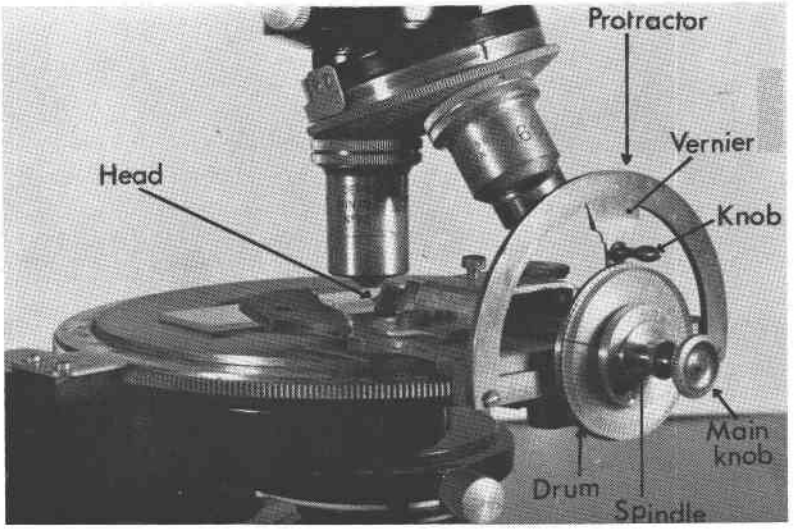

FIG. 2b. Photograph of the spindle stage screwed in position on the microscope.

setting and centering of the crystal. Figure 2b shows the stage screwed in position on the microscope.

Mounting the Crystal

Although the stage permits moderate adjustments to be made, the crystal must be mounted approximately in the correct orientation. Some idea of the optic orientation of a crystal to be mounted can be obtained from a preliminary optical investigation. During the preliminary optical studies great emphasis must be given to the morphological features of the

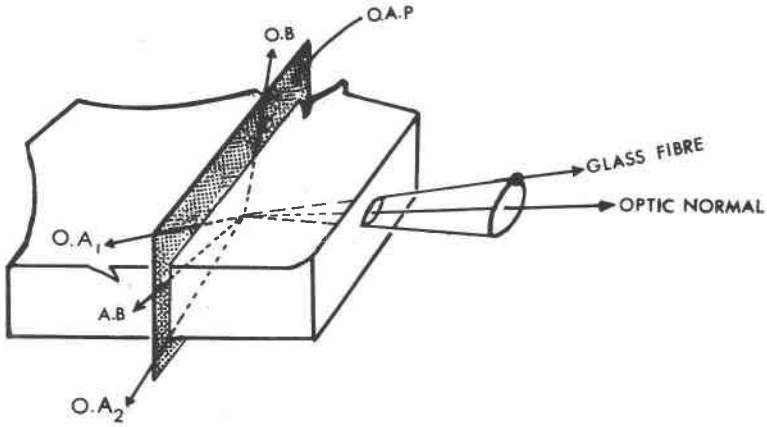

Fig. 3. Diagrammatic representation of a crystal mounted for study.

crystal under investigation relative to its optic orientation. A diagram of the crystal, relating the morphological features and the approximate orientation of the optic axial plane, should be drawn on a sheet of paper to facilitate the mounting of the crystal. For a biaxial crystal, the required orientation for mounting is that in which the normal to the optic axial plane is along the spindle-axis. A uniaxial crystal should be mounted with the unique direction parallel or normal to the spindle-axis. The crystal is therefore attached to the glass fibre, which has been previously cemented to the ball-joint, as close to this orientation as possible. Figure 3 is a diagram of a biaxial crystal showing the approximate orientation of the optic axial plane relative to its morphological features. It indicates the required direction along which the crystal must be mounted.

With a view to determining the extinction angle accurately, prior to optical study on the spindle stage, x-ray investigation should be made on the crystal to identify the cleavage directions. It is very helpful to

keep the crystallographic orientation diagrams and any data relating cleavage directions and crystallographic axes (measured on the goniometer head), as these are useful in extinction angle determinations.

Various mounting materials have been proposed for attaching a crystal to a glass fibre; in this work, a crystalline form of carpenter's glue (known as Cascamite One-Shot), soluble in water, has proved very suitable. In addition to setting hard and quickly, it is unaffected by immersion in refractive index liquids.

Adjustment of the Crystal

When the crystal has been mounted, the head carrying the crystal is screwed into the main spindle. When the alignment of the axis of rotation and the crystal has been roughly checked by eye, it is accurately centered under the microscope. With the crystal centered, necessary height adjustments are made to ensure that it rotates within the oil-cell during a complete revolution of the main spindle. The oil-cell, a cavity 3 mm$\times$3 mm$\times$1.25 mm (these dimensions are suitable for the 40$\times$ normal objectives, N.A.$=$0.72), is made by cementing three pieces of ground glass, 1.25 mm thick on to an ordinary microscope slide (Fig. 1); it may then be filled up with oil, covered with a square cover slip and held in position with a spring clip, so that the crystal remains immersed. Conoscopic observation allows any final adjustments to be made by means of the ball-joint, which allows deviation of 10–15°, to set the optic axial plane normal to the spindle-axis. This part of the operation requires patience, since after each adjustment of the ball-joint the crystal must be recentered.

Measurement of Optic Constants

Principal indices of refraction. With the crystal accurately set, rotation of the main spindle permits the recognition of the bisectrices; the accurate setting of one of these directions parallel to the axis of the microscope is made by using the vernier adjustment. It is sometimes helpful to check the bisectrix either with a sensitive tint plate or by a rough measurement of 2V as described below, since confusion may arise when the 2V value is very high and the birefringence is low. The principal indices of refraction may then be determined by the standard immersion method: for a biaxial (−) crystal with its obtuse bisectrix vertical, the plane containing the α- and β-vibration directions is parallel to the microscope stage, and so the measurements of α and β indices can be made. Upon rotating the crystal through 90° in the N-S plane by means of the knob, the acute bisectrix is set vertical while the β- and γ-vibration directions lie in a

plane parallel to the microscope stage permitting the measurements of β and γ. A refractometer is used to determine the refractive index of the matching liquid.

Since the Becke Line method is not unduly sensitive, precise adjustment of the crystal is not necessary. Provided that the birefringence of the mineral is less than 0.010, and the optic axial plane is set to within 2–5° of the vertical, the refractive indices may be determined with an accuracy of ± 0.001. The reader is referred to the work of Gillberg (1960) on the error caused by inexact orientation of a crystal in the determination of its refractive indices by the immersion method. Higher accuracy may be obtained with the Becke Line method using a variable monochromator, in the following way. The crystal must be perfectly set so that the plane containing any two of three vibration directions is exactly parallel to the stage of the microscope. Using successively at least four refractive index liquids whose values lie within ± 0.005 of the true value of the mineral, corresponding wave-lengths of light are determined repeatedly by the Becke Line method. Then from the r.i. *vs.* λ plot, the refractive index value of the mineral for Na-D light ($\lambda = 5896$ Å) can be obtained by inspection.

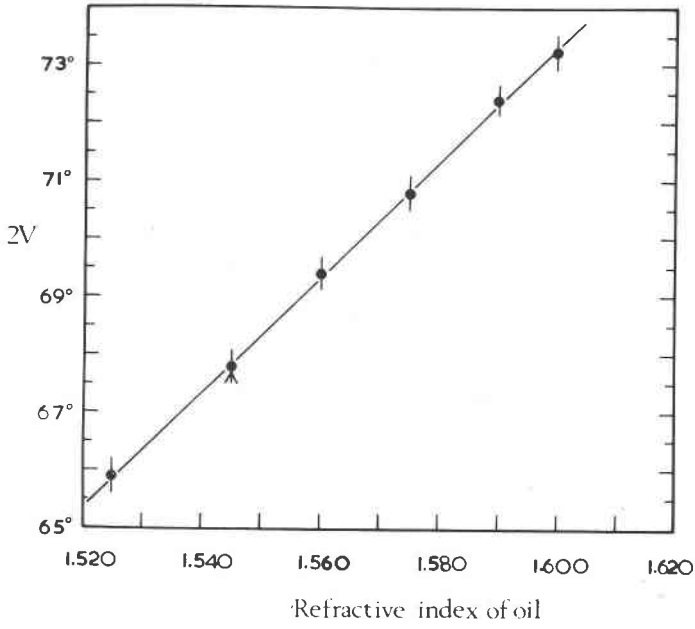

FIG. 4. Variation of optic axial angle measured in various refractive index liquids in Na-D light. ↑ indicates exact value of 2V.

Determination of 2V. The determination of 2V is carried out directly, by rotation, with the interference figure in the 45°-position and the crystal immersed in a liquid whose refractive index matches β of the mineral; this liquid minimises the error due to refraction. Figure 4 shows the variation of the optic axial angle of a crystal measured in various refractive index liquids.

To check the reliability of measurements in the present investigation of the potassium-barium feldspar series, the optic axial angles were also determined conoscopically for the same crystals on a standard universal stage (fitted to Leitz Wetzlar microscope with changeable condenser). The transfer to the universal stage is easily accomplished using the lowering screw (Fig. 1). After the acute bisectrix has been set vertical, the crystal is cleaned with carbon tetrachloride and a microscope slide with a spot of Durofix is placed underneath it. The crystal is depressed by means of the lowering screw until it is in contact with the Durofix. It is then allowed to set for half an hour. Amyl acetate is used to dissolve the Durofix which holds the glass fibre to the tip of the ball-joint, and a cover glass with Canada balsam placed over the crystal. With such an orientated crystal, measurement of 2V on a universal stage is readily made. Measurements made with the universal stage are adversely affected by refraction at oblique internal interfaces, particularly at high angles of tilt; the errors may be reduced by using larger segments (Munro, 1963). Refraction errors are particularly serious when the crystal has high birefringence. The great advantage of the spindle stage lies in the complete elimination of these effects since no oblique interfacial refraction is involved when the grain whose optic axes have been set vertical, is immersed in an oil of refractive index equal to β of the mineral. Table 1 shows the values of 2V measured on members of the orthoclase-celsian series both with the spindle stage and on the universal stage (after necessary corrections). Data in the Table are in good agreement but the accuracy obtained in the measurements on the universal stage is at the very best $\pm 0.5°$, while it is possible to mount and measure a crystal with prominent cleavages on the present spindle stage in about half an hour to the same degree of accuracy, and with greater care, even higher accuracy may be obtained.

Extinction angles. The measurement of an extinction angle is straightforward with the help of orientation diagrams drawn during the x-ray investigation on the crystal. Such diagrams are unnecessary if the cleavages are known and parallel to the crystallographic axes. In the simple case of a monoclinic optically negative crystal whose optic orientation is such that $\gamma \| [010]$, the direction of the obtuse bisectrix, γ,

TABLE 1. COMPARISON OF OPTIC AXIAL ANGLES OF POTASSIUM-BARIUM FELDSPARS MEASURED ON THE (i) SPINDLE AND (ii) UNIVERSAL STAGE

Optic axial angle			
On the spindle stage	On the universal stage[1]	On the spindle stage	On the universal stage[1]
40.0°	40.5°	75.6°	76.5°
63.1°	62.0°	73.8°	74.4°
67.1°	66.6°	86.1°	84.2°
66.0°	65.9°	88.2°	87.0°
75.4°	75.8°	88.0°	88.5°
75.4°	75.5°	87.5°	86.2°
74.3°	74.0°	86.3°	87.2°
76.0°	76.6°	84.5°	83.6°
78.2°	79.5°	85.3°	84.1°

[1] After necessary corrections.

is set parallel to the axis of the microscope: rotation of the microscope stage then permits the measurement of α:[100] on (010). Upon turning the crystal through 180° in the N-S plane by means of the knob of the main spindle, α:[100] is remeasured. The mean of the two readings is taken. For a crystal with optic orientation, $\beta \| [010]$, the measurement of γ:[100] may be made by rotating the crystal in the N-S plane by means of the knob to bring successively the obtuse bisectrix and (100)-cleavage vertical; the difference in readings on the protractor is recorded.

ADVANTAGES AND LIMITATIONS OF THE STAGE

The present spindle stage has been designed to facilitate the direct measurement of the optic axial angle under conoscopic observation, while retaining simple construction. The optic axial angle may be determined within $\pm 0.5°$ or better, with negligible refraction effect. Other optic constants also can easily be determined. If necessary, an orientated crystal may easily be transferred to a universal stage by means of the lowering screw. The stage permits direct correlation between x-ray and optical orientations of small crystals (*e.g.* 50–100μ). In addition, the head permits the angular displacement and accurate centering of a crystal. The crystal can independently be rotated through 360° and can be coupled to the protractor at any stage during this movement. The readings on the stage can be transferred to a stereogram, if required.

Because the angular movement of the ball-joint is restricted, the crystal must be mounted with the glass fibre nearly normal to the optic axial plane. Experience is needed to carry out with ease the accurate

mounting and adjustment of the crystal; but once the technique has been mastered, all the optic constants may be determined on a crystal with good cleavages in about one hour. If only approximate values are required, a much shorter time is necessary, and the setting procedure is quicker than in the standard spindle stage because of the greater facility for adjustment.

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References

Bernal, J. D. and C. H. Carlisle (1947) A simple stage goniometer for use in connection with X-ray crystal analysis. *Jour. Sci. Instr. London*, **24,** 107.

Fisher, D. J. (1960) A new universal-type microscope. *Zeit. Krist.* **113,** 77

——— (1962) Temperature control spindle stage. *Am. Mineral.* **47,** 649.

Federov, E. S. (1891) Eine neue Methode der optischen Untersuchung von Krystallplatten in parallelem Lichte. *Tschermaks Mineral. Petro. Mitt.* **12,** 505.

Gillberg, M. (1960) The error caused by inexact orientation in the determination of refractive indices of minerals by the immersion method. *Ark. Mineral. Geol.* **2** (38), 509.

Hartshorne, N. H. (1963) A single axis rotation apparatus. *Mineral. Mag.* **33,** 693.

——— and P. M. Swift (1955) An improved single axis rotation apparatus for the study of crystals under the polarizing microscope. *Royal Microscope Soc. Jour.* **75,** 129.

Munro, M. (1963) Errors in the measurement of 2V with the universal stage. *Am. Mineral.* **48,** 308.

Oppenheim, M. J. (1962) The spindle stage: A modification utilizing a hypodermic syringe. *Am. Mineral.* **47,** 903.

Steinbach, B. J. and J. R. P. Gibb, Jr..(1957) Device for orienting the small crystals under the microscope. *Anal. Chem.*, **29,** 860.

Wilcox, R. E. (1959) Use of the spindle stage for determination of principal indices of refraction of crystal fragments. *Am. Mineral.* **44,** 1272.

——— (1960) Optical axial angle determination on the spindle stage (abstract only). *Bull. Geol. Soc. Am.* **71,** 2003.

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