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THE MICROSCOPIC DETERMINATION OF THE THICKNESS AND PLANENESS OF PLATELETS IN FINE MATERIALS

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ABSTRACT

A procedure has been developed for the direct microscopic determination of the thickness and planeness of both opaque and nonopaque platelets which constitute fine powders ranging from a limiting size of 100 mesh to a nominal size of 5 microns. The procedure consists of (1) orienting the platelets by dusting the sample over the meniscus of an epoxy resin, (2) sectioning the resultant catalyzed resin mount normal to the meniscus and, correspondingly, the broad faces of the platelets, (3) remounting the sectioned block in additional resin to protect the thin layer of oriented platelets, and (4) polishing the sectioned face for microscopic examination in incident light.

The thickness of the platelets exposed on the polished surface of the mount may be measured with a micrometer ocular. The error in the measured or apparent thickness due to deviation of the platelets from the preferred orientation should typically be less than 15 per cent of the true thickness. The planeness or unevenness of the broad faces of the platelets may be assessed by visual inspection of their outlines on the polished mount. These outlines are the profiles of the platelets observed in a direction parallel to their principal plane.

The possibility of extending this method to include the direct measurement of the thickness of platy clay-mineral particles by means of the electron microscope and replication methods is under study.

INTRODUCTION

Many properties of finely divided materials are dependent on the shape, dimensions, and surficial roughness of the constituent particles. Accordingly, the industrial mineralogist is frequently requested to evaluate these characteristics. If the particles are roughly equidimensional, their diameters may be measured and the shape and roughness of the grains ascertained with either the light or the electron microscope. When the particles are either platy or lath-shaped, however, the problem of measuring their short dimension and of assessing the roughness or relief of their broad faces becomes formidable.

This paper is a description of a procedure by means of which the short dimension and the planeness of both opaque and nonopaque platelets may be directly measured and assessed with the light microscope.

CLASSICAL MICROSCOPIC METHODS FOR MEASUREMENT OF PLATELET THICKNESS AND ASSESSMENT OF PLATELET PLANENESS

General Discussion

In order to measure the short dimension of platy particles the platelets must be oriented so that their short dimension lies in the plane of the stage of the microscope. This type of preferred orientation is virtually

impossible to obtain by conventional techniques for petrographic examination and the usual procedure by which opaque particles are mounted in Lucite, Bakelite, or other suitable media so that they may be polished for microscopic examination in incident light.

A high degree of orientation is obtained with standard immersion methods, but the platelets are then oriented with their short dimension parallel to the optical axis of the microscope. A discussion of the applicability of classical methods for determining the thickness and planeness of nonopaque anisotropic platelets in this orientation follows.

Chaulnes Method

In the Chaulnes method (Winchell, 1937, pp. 75–76, 84–85) the true thickness of the platelet is the product of the apparent thickness, as measured on the micrometer screw of the microscope, multiplied by the index of refraction of the platelet. The platy particles of most micaceous minerals oriented with their short dimension parallel to the optical axis of the microscope have a birefringence of section which is less than 0.01 (Taylor, 1948). As a result, the intermediate index of refraction (β) may be used in the Chaulnes formula without significantly affecting the calculated thickness. It appears, therefore, as though the Chaulnes method should be suitable for the determination of platelet thickness. In practice, however, the usefulness of the method is limited because the surfaces of the platelets in sized fractions obtained by wet elutriation methods are essentially free from microscopically visible surface dust, and it is almost mandatory that subsieve powders (those passing a 325-mesh Tyler screen which has an aperture of 44 microns) be closely sized if reliable quantitative data are to be obtained. The absence of fixed reference points (dust particles) on which to focus leads to confusion in regard to the critical selection of the upper and lower surfaces of the platelets. Furthermore, the diameter-to-thickness ratio of minus 325-mesh platelets is almost invariably greater than 3 to 1 so that the true thickness of subsieve platelets is characteristically less than 15 microns. Inasmuch as one interval on the micrometer screw of most polarizing microscopes is 1 micron or greater, the error in the measurement of platelet thickness by the Chaulnes method could be excessive.

Multiple thickness determinations are required to detect thickness variations in a platelet by the Chaulnes method. Such a procedure, however, yields inadequate results even if dust particles for critical focusing occur on both the upper and lower surfaces of the platelet.

Differential Retardation

Variations in the interference color from point to point on the broad face of a single-crystal platelet between crossed nicols represent variations

in thickness. Most micaceous minerals, however, have a very low birefringence of section in this orientation, and the combination of a low birefringence with a very thin platelet (<15 microns) results in interference colors that are, at most, shades of first-order gray. Insertion of the gypsum plate or the quartz wedge will enhance small differences in retardation from point to point within a low-retardation platelet and, when the stage of the microscope is rotated, the platelets with a variable thickness have a variegated appearance. A qualitative impression of the degree of roughness of the broad surface of a low-retardation platelet can thereby be obtained, but this is hardly adequate for critical comparative studies of the surfaces of platelets in different powders.

*Berek Compensator**

The thickness of an anisotropic platelet is the quotient of its retardation, as measured with the Berek Compensator, divided by its birefringence of section. Platelets mounted on a slide in an immersion liquid will orient themselves with their broad faces parallel to the stage of the microscope. Consequently, the birefringence of section of the platelets of the any micaceous substance so mounted will be a constant, which for a known substance can be obtained directly from handbooks and for an unknown substance can be determined by measuring its refractive indices in this orientation. In theory, therefore, the Berek Compensator should provide a means of determining the thickness of thin anisotropic platelets, but in actual practice its applicability is limited. This is due to the combined effect of (1) the thinness of the platelets, which are typically less than 15 microns thick in powders finer than 325 mesh, and (2) the characteristically low effective birefringence (<0.006) of micaceous minerals oriented with their broad faces normal to the optical axis of the microscope (Taylor, 1948). The net result is a low retardation manifested by either quasi-extinction or grays of the first order which cannot be measured accurately with the Berek Compensator. Furthermore, the calculated thickness of the platelets is subject to serious error when the birefringence of section is less than 0.006 because such values approach the usual limits of accuracy for refractive index measurements. For example, if the birefringence of section is 0.004, deviations of ± 0.002 change the calculated thickness by -33 per cent and $+100$ per cent of the true thickness, respectively.

Method Proposed in This Paper

The method is a procedure whereby the platelets are oriented in a resin matrix, sectioned normal to the broad faces of the platelets, and

* Descriptions of this instrument are given by Winchell (1937, pp. 132–135), and Rogers and Kerr (1942, pp. 19, 75–78).

polished for microscopic examination in incident light. The method has been successfully employed on a variety of opaque and nonopaque minerals, which, when finely ground, tend to have platy and lath-shaped particles. These include molybdenite, graphite, energite, muscovite, talc, pyrophyllite, and brucite.

Procedural Details

Mounting the Sample

An aluminum foil mold in the form of a rectangular parallelepiped is filled with Epon No. 828,* a cold-setting epoxy resin that is liquid at room temperature. A liquid catalyst, triethylene tetramine,† is added to the resin in the ratio of 10 parts resin to 1 part catalyst to make the resin consolidate at room temperature in about 1 hour. The curing time for the catalyzed resin can be shortened markedly by heating it in a laboratory oven at 275° F. for about 15 minutes and then quenching it in tap water.

The powder to be mounted is dusted over the surface of the catalyzed liquid resin in the mold until the meniscus is thinly coated with the powder. The minute platelets in the powder orient themselves with their broad faces parallel to the meniscus of the resin and slowly settle downward. Particles of the minerals listed above (specific gravity 2.3 to 4.7) that are finer than 150 mesh settle between 0.15 mm. and 0.70 mm. in the 45 to 60 minutes required for the catalyzed epoxy resin block to solidify at room temperature.

The sample to be mounted should preferably be closely sized for the purposes of quantitative microscopic analysis, and in order to obtain (1) good dispersion of the platelets in the mount and (2) platelets with surfaces free of fine particles which would otherwise obscure their surficial relief. Suitable size fractions may be obtained from material coarser than 325 mesh by wet screening on standard testing sieves. Equally acceptable size fractions may be obtained from material finer than 325 mesh by either wet elutriation, through the use of standard sedimentation methods or laboratory mechanical classifying devices, or dry elutriation by means of the Haultain Infrasizer. Mounts with a satisfactory dispersion and with dust-free platelets have been prepared from minus 200-mesh talc and muscovite which were deslimed at nominal limiting sizes of between 5 and 15 microns. For less rigorous quantitative studies, therefore, desliming of a minus 200-mesh sample by sedimentation prior to mounting is probably adequate preparation in order to insure a satisfactory finished mount.

Sectioning the Mounted Sample and Remounting the Sectioned Block

The aluminum foil is removed by hand from the resin block that contains the sample, and the mount is sectioned normal to the original upper liquid surface (meniscus) of the resin with a Carborundum cut-off wheel. The sectioned face is then placed face down in another rectangular mold of aluminum foil whose dimensions are about twice those of the original mold. The mold is then filled with additional catalyzed Epon No. 828 epoxy resin. The sectioned surface of the original cast resin block which contains the sample is now exposed on the lower face of the newly cast resin block (see Fig. 1). The exposed cross section of the sample is surrounded by resin and is now sufficiently protected so that it can withstand the stresses of grinding and polishing.

* Manufactured by the Shell Chemical Corporation, Union Commerce Building, Cleveland 14, Ohio.

† Obtained from the Carbide and Carbon Chemicals Company, 30 East 42nd Street, New York 17, New York.

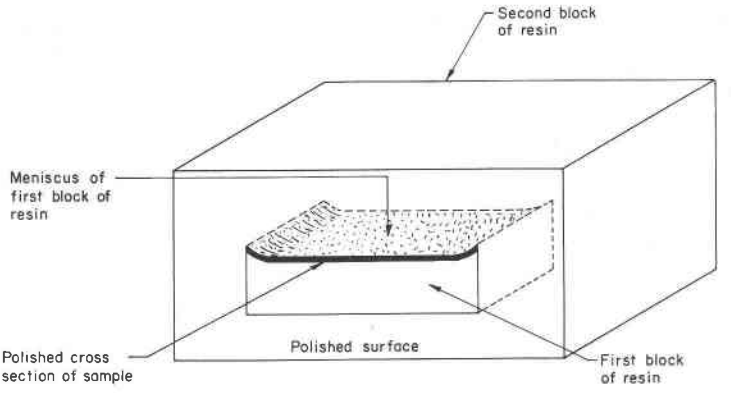

FIG. 1. Diagram of a finished mount.

Grinding and Polishing

The choice of a grinding and polishing procedure will depend principally on the nature of the mounted material. The method described here is a simple and rapid procedure which has given consistently satisfactory results for a number of materials with diverse physical properties.

The face of the mount on which the sectioned sample is exposed is ground on a 600-grit silicon carbide paper-backed disk which is mounted on a wheel rotating at 1250 rpm. The disk is lubricated with Johnson's Stik Wax.* The ground surface is then polished with rouge on a water-lubricated wheel covered with Buehler Miracloth† which rotates at 250 rpm. The mount is now ready for microscopic examination in incident light.

The maximum elapsed time for the preparation of a finished mount by the method described above is about 2 hours and 20 minutes. Two hours represent the curing time at room temperature of the two successive blocks of resin required for each mount; this interval can be shortened by the application of heat. Grinding and polishing takes less than 5 minutes.

The Finished Mount

The sectioned sample on the polished surface of the finished mount is confined to a zone less than 1 mm. in thickness (Fig. 1). At magnifications greater than 250 diameters, the sectioned platelets are readily discernible and appear as long thin rods on the polished surface (Fig. 2). Many platelets of nonopaque micaceous minerals show the traces of their cleavage planes oriented parallel to the long axes of the rods. Although the reflectivity of many nonopaque micaceous minerals in polished section is close to that of the resin matrix, the platelet boundaries are still easily recognized. Their distinctiveness is enhanced by the positive relief of even the softest platelets, such as those of talc, on the polished surface when the mount is first prepared. If the mount is repolished by the same procedure after 3 or 4 weeks, however, the differential relief between the particles and the matrix changes. Talc particles, for example, no longer stand up in positive relief but are essentially

* Manufactured by S. C. Johnson and Son, Inc., Racine, Wisconsin.

† Obtained from Buehler Limited, 2120 Greenwood Street, Evanston, Illinois. Miracloth is a blend of 85 per cent cashmere and 15 per cent silk; it has a medium nap.

flush with the polished surface of the resin. This change in the relative polishing hardness of the resin and the platelet cross sections is probably due to post-solidification curing of the resin which increases its intrinsic polishing hardness.

Measurement of Thickness

If, as the platelets settled in the resin, they increasingly deviated from the preferred orientation, a systematic trend of increasing apparent thickness from the original meniscus to successively lower levels of the

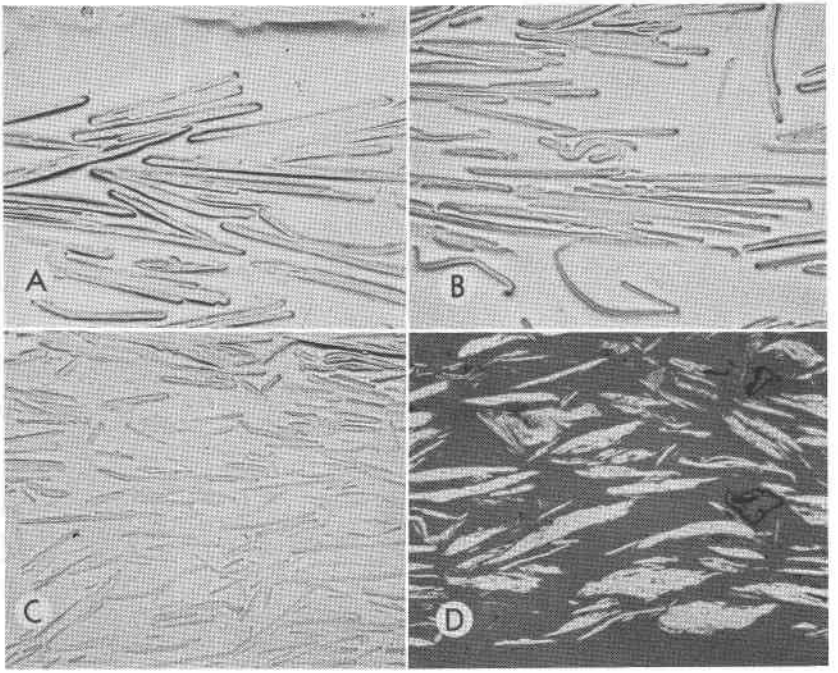

FIG. 2. Photomicrographs of ultrasonically disintegrated muscovite and of commercial flake graphite mounted in accord with methods described in this paper. Incident light. ×220.

A. Muscovite: −150 +325-mesh fraction. Note trace of meniscus at top.

B. Muscovite: −150 +325-mesh fraction. Note deformed particles (center and lower left) and "peeled back" cleavage flake (center).

C. Muscovite: −325-mesh fraction (unsized).

D. Graphite: −270-mesh (unsized).

mount should be evident, inasmuch as the true thickness of a platelet is its minimum thickness. The absence of such a trend in mounts of closely sized samples suggests that deviations from the preferred orientation are probably of little importance with respect to the average or median

thickness which is calculated from the measured thicknesses of a statistically representative number of platelets.

The relationship of the true thickness of a platelet to its apparent thickness as measured on the polished surface of a mount prepared by the method described in this paper is

$$\text{Apparent Thickness} = \frac{\text{True Thickness}}{\text{Cosine } (90 - \alpha)}$$

where α is the angle between the normal to the polished surface of the mount and the pole of the platelet. The ratio of the interplatelet distance to the diameter of the platelets is typically less than 0.5 in mounts prepared by this method. This means that, for any platelet, the maximum deviation of α from 90° will be 30° because of the physical resistance offered by adjoining platelets. Although the magnitude of the error introduced into the apparent thickness by the deviation of α from 90° increases exponentially as the deviation of α increases linearly, a limiting deviation of 30° corresponds to a maximum error in the apparent thickness of only 15 per cent of the true thickness. The permissible error will, of course, be greater if (1) the platelets in adjoining levels are arranged in echelon so that there is little or no opposition to the deviation of an intervening platelet from the preferred orientation by those in adjoining levels, (2) two or more platelets rotate in unison which would result in an increase in the effective interplatelet distance and a corresponding increase in the permissible deviation of α, and (3) the ratio of interplatelet distance to diameter of the particles in a particular mount is greater than 0.5. With respect to the first two circumstances, however, a statistically significant number of thickness measurements should suppress any exceptionally large individual errors in apparent thickness due to exceptionally large deviations of α.

Assessment of Platelet Planeness

The outlines of the platelets in the polished mounts represent the profiles of the platelets as viewed in a direction parallel to the principal plane (principal cleavage) of the flakes (Fig. 2). Significantly, the profiles reflect the degree of irregularity or unevenness of the broad surfaces of the platelets. The cross sections of the platelets may be classified into three overlapping but distinctive categories as follows:

(1) *Platelets with plane parallel sides*

In cross section these particles appear as long rod-shaped forms with rectilinear boundaries. Cleavage traces parallel to the length of the rods may be visible, particularly in nonopaque minerals.

(2) *Platelets with rough or uneven surfaces*

In cross section these particles appear as elongate forms with irregular boundaries. These irregularities reflect either convexities and concavities in the broad surfaces of the platelets or structurally controlled "treads and risers" wherein the "tread" represents the broad surface of a cleavage flake which was peeled off the platelet, and the "riser" represents the fracture along which the cleavage flake was torn off the platelet. The cleavage traces of the platelets run approximately parallel to the elongation of the irregular cross sections, but do not conform to the irregularities in their outlines.

(3) *Deformed platelets*

Platelets which have been warped, bent, crumpled, and otherwise deformed appear as elongate forms which have been bowed, singly or multiply folded, crimped on one or both ends, deformed into S-shapes, etc. The traces of the cleavage planes run parallel to the outlines of the deformed platelets. This is a useful criterion for differentiating deformed platelets from platelets with uneven surfaces.

By classifying a statistically significant number of platelets on this basis or some modification of it that is of greater significance for the particular material under study, it is possible to assess quantitatively the comparative planeness of the platelets in various samples which constitute a suite.

Within the confines of the two-dimensional polished surface of the resin mount, it is possible for a rough particle to appear as a plane particle if the irregularities on the broad surfaces of the platelet are nonuniformly distributed. The recorded percentage of plane particles in a sample will, as a result, be greater than the true value. On the other hand, this error should not seriously affect the usefulness of the acquired statistical data on planeness for comparative purposes.

Possible Extension of the Proposed Method to Platy Clay Minerals

The dimensions of clay particles are of considerable technological importance because in large part they control the colloidal properties of clay suspensions. The diameters of platy clay particles may be determined directly with the electron microscope, although some uncertainty as to the exact equivalence of their diameters as measured on the electron micrograph and that of the particle in suspension is introduced because the sample must be dried in order to prepare it for electron-optical study. Electron microscopy is evidently the ideal method for the determination of particle thickness, but the thickness of platy clay particles cannot be determined readily from ordinary electron micrographs in which the broad faces of the platelets lie in the plane of the micrograph. Shadowing techniques may be employed, but they yield results that are usually

difficult to evaluate quantitatively. Kahn (1959) has recently attempted to determine the thickness of platy clay particles by combining electro-optical birefringence data with ultracentrifuge data and viscosity data. The two sets of derived thicknesses show a fair correlation with respect to trends in their relative magnitude, but the absolute thicknesses obtained by these methods are widely divergent.

Studies are now in progress to extend the method described in this paper to the determination of the thickness of platelets within the particle-size range of clays. When techniques for obtaining a preferred orientation of clay platelets in a resin mount have been developed, it should be possible to replicate the polished surface for direct measurement of the thickness of the clay platelets with the electron microscope.

Acknowledgments

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Note Added in Press

A recent paper by Sahama (*Am. Mineral.*, **44**, 1959, 1303–1305) directed our attention to the applicability of the rotating elliptical mica compensator (the Sénarmont 1/4 λ plate and the Brace 1/10 to 1/30 λ plate) in determining the thickness and planeness of fine platelets. These compensators can be used to measure accurately small retardations in the 1 to 50 $m\mu$ range which is the approximate retardation range of fine platelets oriented parallel to the microscope slide in the particle-size range considered in this paper. Errors in the calculated thickness may be large, however, because it is difficult to determine the birefringence of section with a precision greater than ± 0.001. For example, if the birefringence of section is 0.004, deviations as small as ± 0.001 will change the calculated thickness by -20 per cent and $+33$ per cent of the true thickness, respectively. The elliptical compensators could be quite useful for differentiating platelets with plane-parallel sides from rough-sided platelets of variable thickness, but the profile method proposed in this paper should be superior for differentiating crumpled and deformed platelets from undeformed rough-sided platelets of variable thickness.