GENERAL

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QUANTITATIVE FABRIC OF WET KAOLINITE

by

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ABSTRACT

A METHOD is described for quantitatively expressing orientation of kaolinite particles in a wet clay mass. Wet clay is impregnated with a polyalcohol which permits grinding of a very flat surface (without disrupting the original fabric) for quantitative X-ray measurement. In the specific procedure discussed, a Norelco diffractometer equipped with a pole figure device and a Geiger detector was employed to measure by reflection the diffracted intensity of the 002 and 020 kaolinite reflections. The amplitude of the 002 peak to the amplitude of the 020 peak is called the peak ratio, PR, which quantitatively expresses particle orientation at any chosen angle to the specimen surface. Limits of orientation are random and maximum orientation. For the kaolinite tested, truly random orientation was achieved which gave a PR = 2.0. Maximum orientation found experimentally was PR = 200 for thoroughly dispersed slurry slowly dried on a glass slide. Vertical and lateral variations can be measured either continuously or in steps of any chosen size.

INTRODUCTION

BEHAVIOUR of clay–water systems, over a wide water content range, has been explained in terms of the structure of the clay phase. Structure is the combination of the geometrical arrangement of particles and the forces operating between them. The geometrical arrangement of particles and associated voids are called fabric and is the component of structure that is most amenable to measurement. Since the structure concept has been invoked to explain so many features of clay behavior, a reliable quantitative method for determining the fabric of a moist clay is extremely important.

The problem of measuring fabric is actually two interrelated problems: (1) to prepare for examination a specimen that preserves the fabric of the original sample, and (2) to examine some property of the clay crystallites that quantitatively expresses their geometrical arrangement. Procedures which have been employed to measure particle arrangement include light and electron microscopy, and X-ray diffraction. Brindley (1953) demonstrated that the X-ray powder camera could be used to measure fabric. A slight modification of Brindley's technique was employed by Raitburd (1958 and 1960) on dried clay specimens. While Raitburd's X-ray data are quantitative, the specimens were dried prior to preparation for X-ray, which seriously limited interpretation of the X-ray data relative to the fabric of the original

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wet clay. Mitchell (1956) made an innovation toward minimizing disruption of the original fabric by gradually replacing the water with a polyalcohol which is easily solidified to give the clay rigidity.

A description of the experimental procedure, along with illustrative data on the fabric of kaolinite clay, are the subject of the present discussion. The technique is: (1) to impregnate the wet clay with polyalcohol, (2) to grind a flat surface, and (3) to examine the surface by X-ray diffraction using a diffractometer equipped with a Schulz pole figure device for measuring the amount and the direction of particle orientation.

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PROCEDURE

General

The principle behind the study of fabric by X-ray diffraction is very simple. Clay particles, being very anisotropic, have a decided tendency to orient with their large faces approximately parallel. For a given clay, the intensity of X-ray diffraction from a particular crystal plane is controlled primarily by two variables, the clay concentration in the irradiated volume and the orientation of clay particles in that volume.

The extreme variation in porosity of the kaolinite clay phase in the present experiments was between 0.40 and 0.85. Since the mass absorption coefficient of kaolinite is about 5 times that of the carbowax, reducing the clay concentration from 1.0 (arbitrarily assigned to the lowest porosity) to 0.47 (equivalent to the highest porosity) will reduce the diffracted X-ray intensity by only 20%, if the fabric remains constant. The observed diffracted intensity variation on a particular peak varied by as much as 30 times. Because particle orientation is so important it becomes necessary to locate exactly the direction of preferred orientation relative to the specimen surface and finally to the original clay block.

The standard diffractometer permits only one motion of the specimen, θ rotation (see Fig. 1). The center of the specimen face is on the axis YY'. With the standard diffractometer, reflections can be recorded only from those crystallites which: (1) satisfy the Bragg equation and (2) are situated in a plane parallel to the axis of rotation, YY'.

The pole figure device permits three additional motions and at the same time maintains focusing geometry to satisfy the Bragg equation. Constant reference to Fig. 1 will help to clarify the different specimen movements which are otherwise difficult to present clearly. These motions are:

1. Tip, ϕ, the specimen rotates about the XX' axis with the detector set at the 2θ position of a diffraction maxima, generally the 002 kaolinite peak. The ϕ curve generated shows the angular distribution of diffracted intensity

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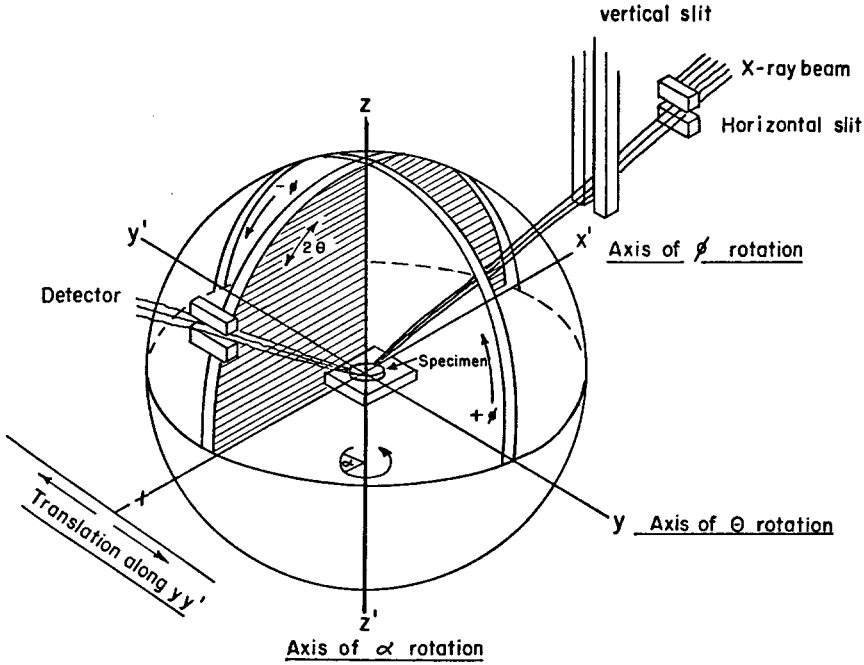


FIG. 1. Pole figure device movements.

about the zenith for a selected azimuth angle (α). $\phi = 0$ coincides with the Z axis. Plus ϕ is to the right.

2. Spin, α, the specimen rotates about the ZZ' axis with the detector set at the 2θ position of a diffraction maxima, generally the 002 kaolinite peak. The α curve generated shows the angular distribution of diffracted intensity for all azimuth angles at a selected ϕ angle, generally $\phi = 0$. $\alpha = 0$ coincides with Y' which is at 90° to the X-ray beam XX', and rotation is counterclockwise as shown.

3. Translation, the specimen surface is translated in the XY plane along the YY' axis. Therefore, the pole figure device provides a means of examining the reflections from *all* surface crystallites. The ϕ and α angles for maximum intensity define the direction of orientation on a particular specimen surface which is then related back to reference coordinates in the original clay block.

The amount of orientation is specified by the peak ratio, PR, which is the amplitude of the 002 kaolinite reflection divided by the amplitude of the 020 kaolinite reflection where both are measured at the same specified ϕ and α. PR has the advantage of being independent of particle concentration and automatically minimizes mechanical and instrumentation variables in the experimental procedure. The numerical value of $(PR)_{\phi\alpha}$ is referred to reference values for random and/or maximum orientation on a particular clay, in this

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case kaolinite. Once the amount and direction of orientation in a batch are known, vertical and lateral variations are readily obtained by comparing PR on successive slices at constant ϕ and α conditions.

An alternative means for expressing the amount of orientation is the width of the ϕ curve, W measured at one half the maximum amplitude. This numerical measure of the angular spread in diffracted intensity about the maximum ϕ is proportional to the amount of particle orientation. However, W can be measured only at one spot at a time so that an adequate average on a particular surface requires considerable more effort than for PR measurements, which can be made either at one spot or as an average for a surface. The latter is obtained by the simple expedient of using a fast α rotation (120°/sec) during the recording of the 2θ diffraction data for PR determination.

A finished *specimen* of impregnated clay for fabric analysis is 22 mm square and 6 to 7 mm thick. The two large faces are very carefully ground to be flat and parallel to each other. The unimpregnated wet clay block must be at least 4 mm larger all around than the finished specimen. When the wet clay block is sufficiently large that more than one specimen can be cut from it, the block is called a *sample*. A sample represents a specific treatment and provides more than one specimen for fabric analysis. Samples may be obtained either by starting from raw clay or by cutting from a large block of wet clay. A block of wet clay large enough to make more than one sample is called a *batch*.

The reference plane is taken as the horizontal plane *ab* of the original block of wet clay whether the block of wet clay gave one specimen or fifteen samples. A $\beta = 0$ specimen is cut so that the surface to be X-rayed is parallel to the reference plane *ab* and is marked such that a ϕ rotation when $\alpha = 0$ is about the *b* axis. A $\beta > 0$ specimen is obtained by counterclockwise rotation of an *ab* surface about the *a* axis which for $\beta = 90$ yields an *ac* surface for X-ray examination. When $\alpha = 0$, ϕ rotation on a $\beta = 90$ specimen is about the *a* axis. The location of the *a* and *b* axes is somewhat arbitrary because the original clay block is generally cylindrical; however, once chosen, the *a* and *b* axes as well as $\alpha = 0$ position for all samples and specimens from this particular batch are fixed.

Specimen Preparation

In order to prepare a very flat surface for X-ray examination, the moist clay specimens are impregnated with Carbowax No. 6000 (Union Carbide Corp.). Carbowax is completely miscible with water when liquid at 65°C, and at room temperature is solid with a hardness on Moh's scale of one. The top surface of the specimen is left exposed to vapor the first day of immersion in liquid carbowax to allow entrapped gases to escape and to prevent specimen rupture. The exact time required to give sufficient exchange, wax for water, so that the specimen can be ground, will naturally depend upon the specimen: (1) size, (2) water content, and (3) bulk density. For a sample diameter of

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$1\frac{1}{2}$ in., which means a maximum diffusion path of $\frac{3}{4}$ in., 21 days in wax without changing the wax has proved entirely adequate. Ninety cm^3 of clay slurry at 190 g H_2O/100 g clay poured into 55 cm^3 of molten carbowax such that the maximum diffusion path was about $\frac{3}{4}$ in. had a final water content of about 1% at the end of 21 days, calculated from a material balance of initial and final weights. Molten wax in an open beaker at 60°C loses less than 0.1% weight in 21 days.

At the completion of impregnation, the specimen should be removed from the liquid carbowax and allowed to cool slowly to room temperature. It is very important that the surface to be X-rayed be perfectly flat; further, it is highly desirable that the opposite faces be exactly parallel because this greatly facilitates correct alignment in the pole figure device. To accomplish these requirements, a special jig was made from a triangular metal block 86 mm on a side and 13 mm thick. By proper setting of the three adjustable feet, one near each corner of the block it is possible to grind the surface of a specimen flat and parallel to its opposite face. It is also possible to grind off either a known thickness and thus examine successive slices, or a known tip to the original reference plane. The specimen to be ground is held to the flat machined surface of the block by vacuum. The actual grinding is done with No. 600 carborundum powder in kerosene on a plate glass surface.

Instrumentation

A Norelco X-ray diffraction unit equipped with a Geiger detector and a pole figure device was used. Unfiltered copper radiation was generated at 50 kV and 20 mA. The most satisfactory speeds for 2θ and chart during diffraction were found to be $\frac{1}{4}°$ 2θ min and $\frac{1}{2}$ in. min respectively. Slits employed were: (1) 1° divergence and scatter, (2) 0.006 in. receiving, and (3) 0.02 in. vertical. For both α and ϕ rotations, the 20°/min drive motor was used which gave an α rotation of 20°/min and a ϕ rotation of $3\frac{1}{3}$°/min.

With the pole figure device it is necessary to limit the width of the primary X-ray beam with a vertical slit in order to maintain correct diffraction geometry, especially for ϕ rotation. The vertical slit greatly reduces the intensity of the diffracted beam reaching the detector and the area of the specimen diffracting. By proper choice of power settings on theX -ray target and the width of vertical slits, one may examine a fairly narrow width of specimen surface while maintaining acceptable intensity of diffracted radiation at the detector. For example, with kaolinite clay, the 0.02 in. vertical slit irradiates a specimen width of about 1.4 mm. The length of specimen irradiated varies with θ. Again, for the 002 kaolinite peak, which has been chosen for fabric study, the length of specimen irradiated is about 12 mm.

VERIFICATION OF PROCEDURE

The experiments described in this section were specifically designed to investigate qualitative and quantitative aspects of X-ray as a method for

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evaluating clay fabric. The clay used in all experiments is kaolinite Peerless No. 2 from R. T. Vanderbilt Co. Unless otherwise specified, the clay was used as received.

Choice of Diffraction Peaks

The three main reasons for choosing the 020 ($2\theta = 19.9$) and the 002 ($2\theta = 24.9$) kaolinite reflections to express the peak ratio, PR, are that these X-ray reflections are: (1) strong reflections and thus provide a sensitive measure of fabric, (2) distinct from other reflections, and (3) close enough together that approximately the same volume will be irradiated in the determination of the peak amplitudes for both peaks. The volume irradiated in cm^3 is essentially constant irrespective of 2θ; however, it is not the same volume. As 2θ increases the area decreases and the depth increases.

The intensity of the k index of hkl when k is not a multiple of three may be markedly reduced as a result of shear displacements in the individual crystals (Hayes, 1963). One way to overcome this would be to use the 060 reflection where k is a multiple of three. However, for many of the kaolinite samples investigated, the depth of specimen irradiated at the 2θ of the 060 reflection would be excessive.

The PR is really a comparison of peak amplitudes, not peak intensities. The product of peak amplitude times the peak width at half amplitude was used as a measure of intensity for the 002 and 060 peaks (actually the 060, $33\bar{1}$, $3\bar{3}\bar{1}$ triplet). The intensity ratio, IR, equals the intensity of the 002 peak divided by the intensity of the 060 peak and is independent of any change in degree of crystallinity of the kaolinite resulting from shear distortions of the individual crystals.

In order to ascertain whether or not PR is sensitive to shear disorder from manipulation of the clay, both PR and IR were measured on 165 separate surfaces from clay samples with consolidation pressures ranging from 1 to 70 kg/cm^2. A plot of PR versus IR gave a straight line with a coefficient of correlation r^2 for the least square line of 0.97, clearly demonstrating that consolidation to 70 kg/cm^2 produced no measurable change in crystallinity of the kaolinite used in these tests. Further verification that the 020 peak on this kaolinite is not apt to be affected by ordinary engineering manipulation comes from the following experiment. Dry grinding of the kaolinite for 7 hr in a mechanical mortar and pestal did not significantly change PR or IR.

A typical trace for the 2θ ranges of primary interest is shown in Fig. 2 where the methods of measuring the amplitude and intensity are illustrated. The method of defining the base line for amplitude measurement is somewhat arbitrary but since the same procedure is always used, any relative error should be minimized. Comparison of the traces for kaolinite powder with that for the kaolinite–wax mixture shows that the wax does not raise the background level for base line measurement a great deal. In spite of the large wax percentage the kaolinite diffraction peaks are not greatly diminished,

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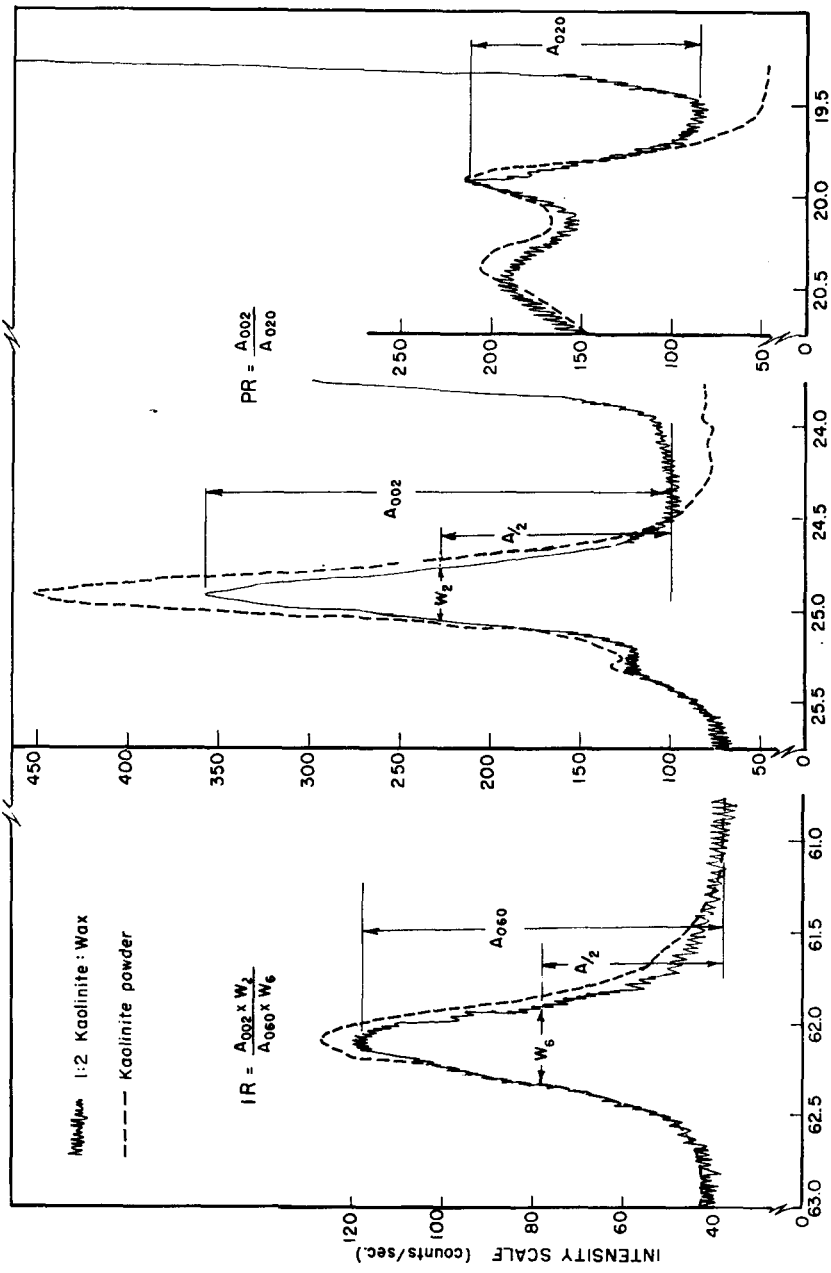


FIG. 2. *Diffraction trace for the 2θ ranges used in fabric measurements.*

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partly because of the low mass absorption coefficient for the wax and partly from the increased clay concentration per unit volume in the kaolinite–wax mixture.

Limits of Preferred Orientation

Limits of orientation are random and maximum orientation. For the kaolinite clay tested, random orientation experimentally measured was PR $= 2.0 \pm 0.2$ and $W = 100 \pm 4° \phi$ both of which are independent of β. Maximum orientation found experimentally was $(PR)_{\phi max} \geq 200$ and $W = 22 \pm 3° \phi$ on thoroughly dispersed clay slurry slowly dried on a glass slide and was $(PR)_{\phi max} = 25 \pm 2$ and $W = 36 \pm 2° \phi$ on flocculated clay slurry slowly dried on a glass slide.

Random.—Absolute random particle arrangement precludes a flat surface at the microscopic level. The problem of obtaining near random particle arrangement while simultaneously maintaining a reasonable flat specimen surface for use in the pole figure device requires some care. It is possible by very careful sieving of air dry kaolinite powder to consistently obtain about one out of four specimens that will be random. Preparing a mixture of clay and wax, then grinding the mixture to a minus 74 μ powder does reduce the variability in the results and produces a near random fabric as shown by Brindley and Kurtossy (1961); however, even this later method is very time consuming.

The simplest procedure for obtaining random fabric is to dump air dry kaolinite into molten wax to give a 1:2 clay–wax mixture. The mixture is allowed to stand *without* disturbance until the wax covers the clay. The specimen when cooled is ground to give a flat surface for X-ray diffraction. The results in Table 1 indicate random fabric because: (1) the IR of 2.07 agrees remarkably well with the IR of 2.12 calculated from the data on ideal intensities for the peaks involved (Brindley and Kurtossy, 1961), (2) the PR and IR are the same for $\beta = 0$ and for $\beta = 90$ specimens, and (3) W is large and is the same for both $\beta = 0$ and $\beta = 90$ specimens. The variation with depth in a given specimen is 10 to 15%, which is as great as the variation between different specimens prepared at different times and by different operators.

For ideal random fabric the α and ϕ rotations on a 2θ diffraction peak would be horizontal lines. The ϕ curves of Fig. 3 are far from horizontal lines.

TABLE 1.—RANDOM KAOLINITE AVERAGE VALUES

	$(PR)_{\phi=0}$	IR	W
$\beta = 0$ surface	2.03 (0.2)*	2.08 (0.1)	99 (5)
$\beta = 90$ surface	1.94 (0.19)	2.06 (0.1)	103 (5)

* Standard deviation for 25 determinations over a 2 month period, except for $(PR)_{\phi=0}$ on $\beta = 0$ surface which is for 75 determinations over a 3 year period.

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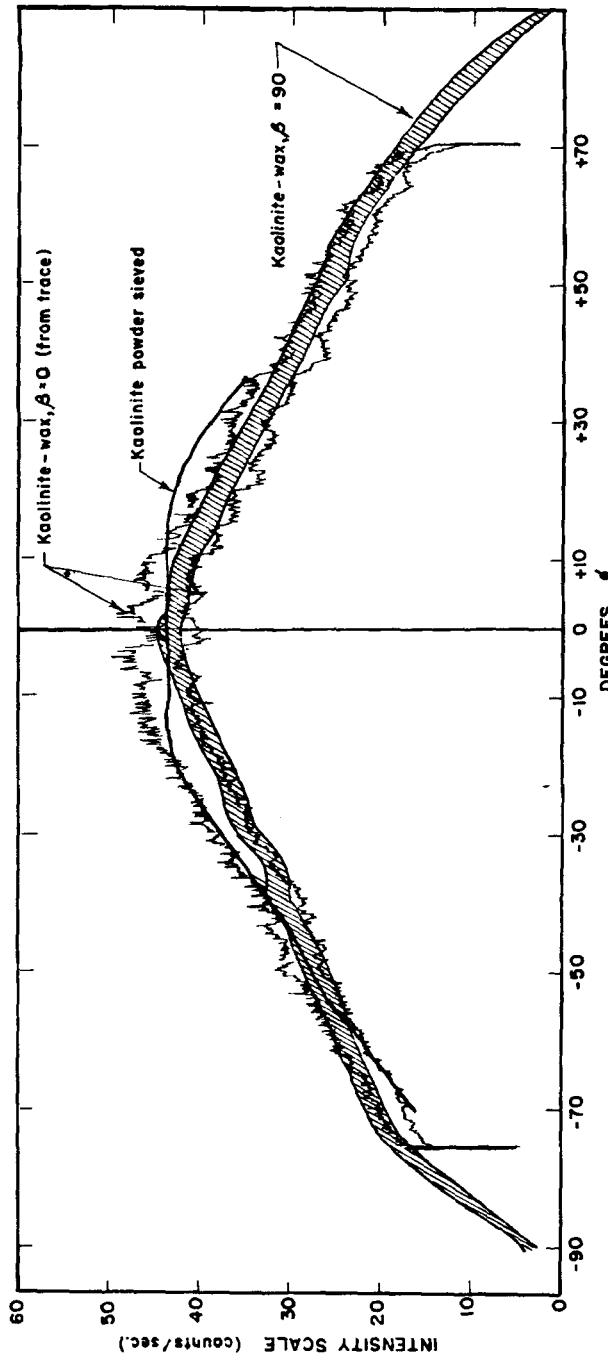


FIG. 3. ϕ Rotation on the (002) peak of randomly oriented kaolinite.

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Nevertheless, these ϕ curves are considered to represent random particle orientation for the following reasons: (1) the ϕ curve for sieved kaolinite powder is flat for 15 to 20 degrees each side of $\phi = 0$ and gives an IR within 5% of the theoretical (2) the IR for the kaolinite–wax specimen is within 5% of the theoretical and, although the ϕ curve is not flat, W is the same as for the sieved powder and (3) the IR, PR, and W are the same for $\beta = 0$ and $\beta = 90$ on kaolinite–wax specimens prepared in the suggested manner to give random particle orientation.

The pole figure represents the stereographic projection of amplitude contours obtained from 002 kaolinite diffraction peak data on a series of ϕ curves at many α angles. In Fig. 5a and 5d the zenith, $\phi = 0$, is at the center and the concentric fine lines give ϕ in 10° increments out to 90°. The heavy numbered lines are the amplitude contours obtained from diffraction data. Figure 5a and 5d is a pole figure for random kaolinite, where the contours in Fig. 5a and 5d were taken on specimen surfaces at 90° to each other. The fact that both quadrants give nearly circular contours, symmetrical about the zenith, $\phi = 0$ and no large discontinuities is excellent verification of the particle orientation in this kaolinite sample.

Maximum orientation.—For X-ray diffraction from the (001) face of a completely idealized single crystal the $(PR)_{\phi\ max}$ would approach infinity and W would approach zero. The amplitude of the 020 reflection, as viewed from the (001) face, also approaches zero; however, the error in the 020 amplitude will give an increasingly large error to $(PR)_{\phi max}$ as $(PR)_{\phi max} \rightarrow \infty$. For any real crystal the limit of W is something greater than zero.

Experimental measurement of W for the 003 reflection on single flakes of muscovite was 4° ϕ, and for the 004 reflection on single flakes of chlorite was 8°ϕ. These peaks were chosen for proximity to the 002 kaolinite reflection. The width of the diffraction maxima at half peak height w was 0.06° 2θ for muscovite and was 0.125° 2θ for chlorite. From the single flakes W/w is approximately 65. One might reasonably expect that as a given 2θ diffraction peak became broader, whether from less perfect crystallinity or from a reduction in particle size, that the minimum W would also broaden. For maximumly oriented minus 20 μ muscovite particles, the calculated W is 14° ϕ while the experimentally measured W was 17° ϕ. The width of the (002) 2θ diffraction peak on the particular kaolinite used was 0.31° θ; therefore, the lower limit of W likely to be encountered with this kaolinite is approximately 20° ϕ.

The maximum amount of particle orientation that can be obtained experimentally on a clay depends a great deal upon the procedure employed to produce the orientation. One of the most effective and simplest ways to obtain pronounced preferred orientation is to allow a clay slurry to dry on a slide. From a systematic investigation of the variables influencing particle orientation of kaolinite, it was found that slow drying at 50% R.H. of dilute slurry (5.65 g H_2O/g clay) on a glass slide gave the most pronounced orientarion. The practical experimental limit of preferred orientation is very strongly

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influenced by the state of aggregation of the clay as shown by the fact that the maximum orientation obtained from drying a dispersed slurry gave $(PR)_{\phi max} \geqq 200$ and $W = 23$, while drying a flocculated slurry gave $(PR)_{\phi max} = 25$ and $W = 36$. The large uncertainty in $(PR)_{\phi max}$ from the dispersed slurry is primarily due to the very large error associated with the

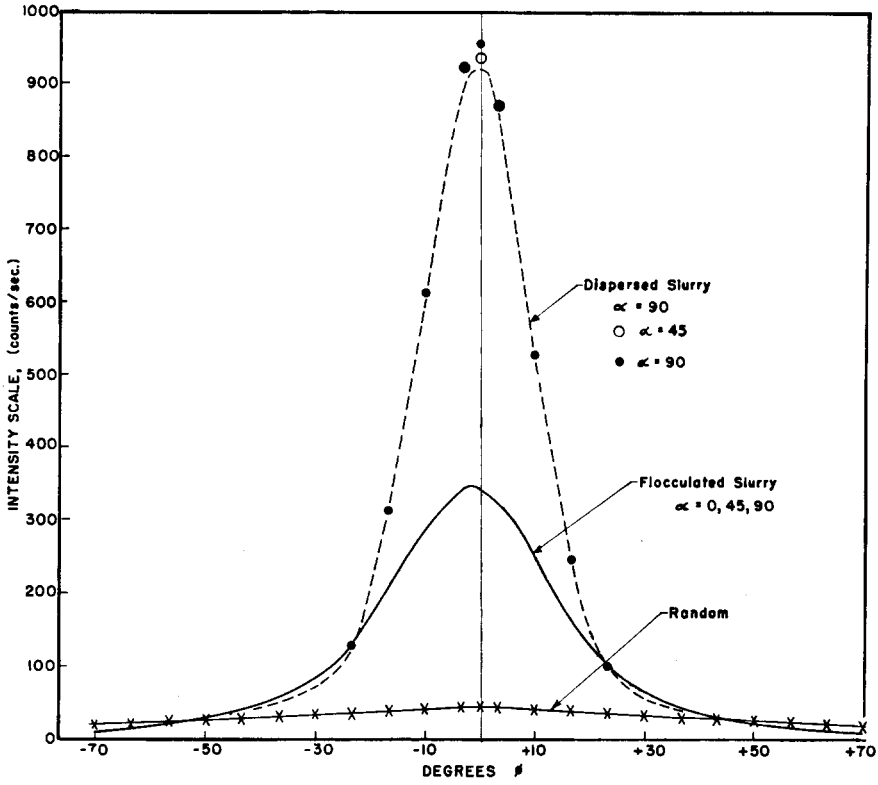


FIG. 4. Comparison of ϕ rotation on the (002) kaolinite peak at maximum oriented and at random.

tiny amplitude of the 020 peak.

At maximum orientation the ϕ curve is independent of α position because the clay particle anisotropy is essentially in the c direction. Figure 4 gives representative ϕ curves on the 002 kaolinite peak for maximum orientation of flocculated and dispersed kaolinite. One may visualize readily the change in orientation by comparing random and maximum orientation in Fig. 4. For uniform lateral distribution and the ϕ curve maxima at $\phi = 0$ one expects the pole figure to be nearly symmetrical about the normal to the specimen surface, the zenith or $\phi = 0$; therefore, for comparative purposes one

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need only plot one quadrant of the pole figure to obtain a clear picture of the orientation. Such a procedure facilitates comparison of pole figures for different surfaces, as shown in Fig. 5 where random and oriented kaolinite are compared to each other. For clarity the ϕ grid was omitted from Fig. 5b and 5c.

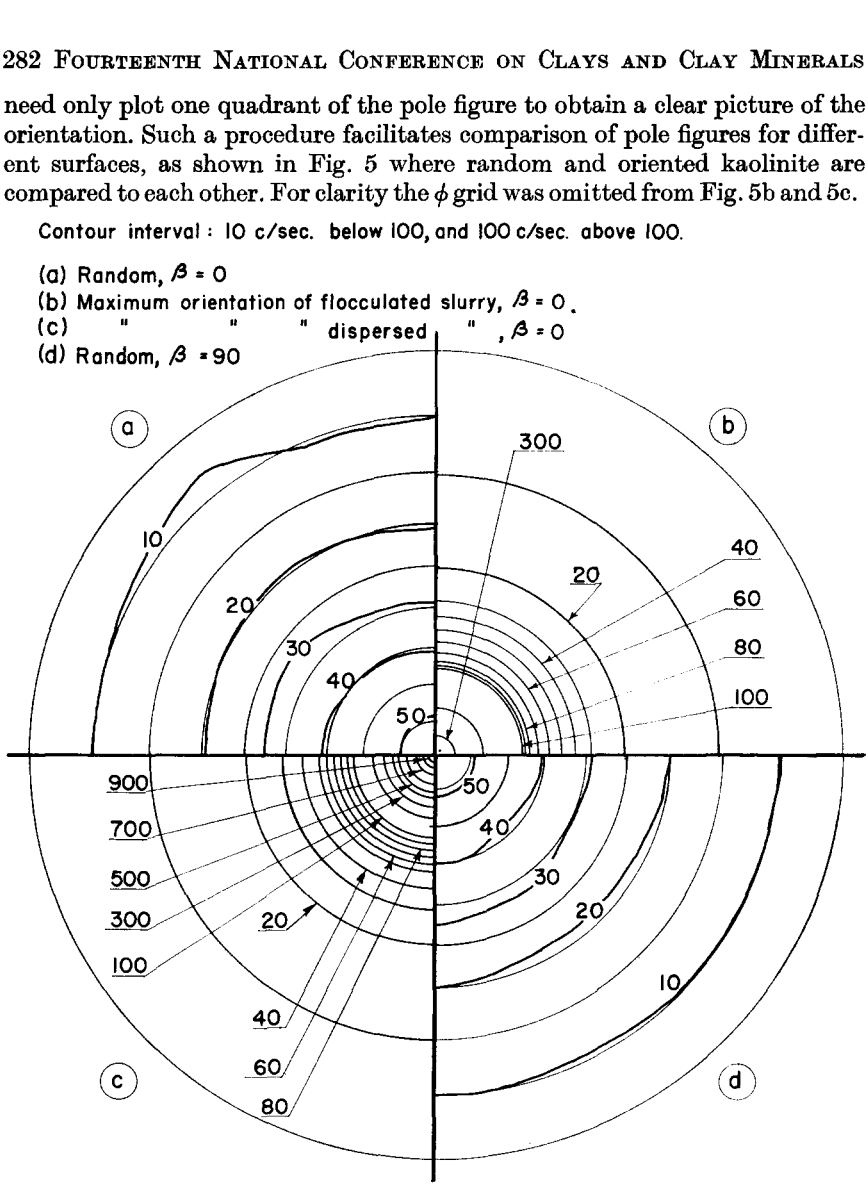


FIG. 5. Pole figure for (002) kaolinite peak: (a) random at $\beta = 0$; (b) maximum orientation on dried flocculated slurry at $\beta = 0$; (c) maximum orientation on dried dispersed slurry at $\beta = 0$; (d) random at $\beta = 90$.

Specimen Preparation

Impregnation with carbowax has been shown to have no effect on the fabric of a wet clay mass (Mitchell, 1956). However, in view of the fact that interpretation of results is so completely dependent upon this fact, additional

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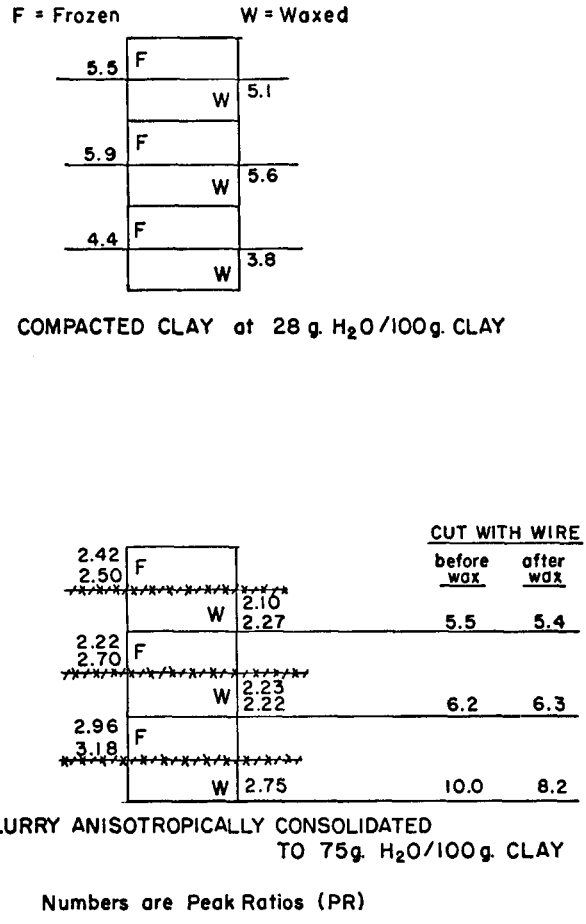


FIG. 6. Effect of impregnation.

tests were performed. Figure 6 gives a comparison of PR for surfaces prepared on specimens impregnated with carbowax and for surfaces prepared on specimens quick frozen in liquid nitrogen (— 196°C). The compacted kaolinite was cut into slices with a wire and alternate slices either frozen *F* or waxed *W**; then opposing faces were X-rayed to determine PR. The consolidated slurry was also cut into slices with a wire and alternate slices either frozen or waxed. One surface on each slice was X-rayed prior to impregnation and again after impregnation. These data are given under "cut with a wire" before and after, respectively in Fig. 6. After fixing the fabric by either freezing or

* *W* in Fig. 6 refers to impregnation with carbowax, not the width of ϕ curve as previously defined.

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waxing, the surface of the slices were ground flat and X-rayed. Removal of an additional 0.01 in. from each surface is indicated in the figure by the PR furthest from the hatched line. Orientation produced by cutting wet clay with a wire is clearly evident in Fig. 6. It is equally apparent that grinding a waxed specimen or filling a frozen specimen effectively removes the oriented zone produced by the wire cut.

In view of the fact that fixing procedures as different as freezing in liquid nitrogen — 196°C and impregnation with carbowax at + 65°C give the same PR, within a reasonable experimental error, is considered good evidence that the standardized impregnation procedure has no detrimental effect upon the fabric of the wet clay.

The following experiment showed that the usual grinding of the flat surface has negligible effect upon the fabric. A specimen where the kaolinite particles had a pronounced tendency to be at 45° to the specimen surface gave a PR at $\phi = 0$ of 3.4 and a PR at $\phi = 45$ of 23, as determined by normal grinding procedure. Now, a deliberate effort was made to smear the clay by rubbing the dry specimen on the ground glass grinding plate for the same time normally required to grind off 0.02 in. After this severe treatment, the PR at $\phi = 0$ was increased to 4.6. Removal of 0.01 in. from this specimen surface by normal grinding gave a PR at $\phi = 0$ of 3.3 upon X-ray diffraction. The very modest change that occurred even when a very deliberate effort was made to smear out the clay particles demonstrates that the normal grinding procedure has no adverse effect upon clay fabric.

Reproducibility

The reproducibility of data is influenced by instrument factors and sample preparation procedures. A check on the instrument, including the alignment of an impregnated clay specimen, was established from data on the PR variation measured on the same identical surface of three specimens over a six month time period. The results were: PR = 1.75, standard deviation (SD) = 0.10; PR = 4.8, SD = 0.5; and PR = 11, SD = 1.1. The larger per cent variation for the larger PR arises primarily from the large error associated with the smaller 020 peak.

Reproducibility starting from a flocculated kaolinite slurry is indicated in Table 2. The standard deviation in Table 2 for each batch represents the combined effects of vertical and lateral variation within each batch. Essentially, zero consolidation was obtained by dumping 90 cm^3 of slurry at a water content of 1.9 g H_2O/g clay into 60 g of molten carbowax (treatment 2, Table 2). Isotropic consolidation was obtained by dumping flocculated slurry into a balloon lined with elastic strips, so that during consolidation, produced by slow evacuation over a two month period, drainage would be essentially radial. Anisotropic consolidation was obtained by dumping the flocculated slurry into either a 3 or 9.5 in. diameter oedometer cell. The final height of the clay batch was about half the cell diameter for both cells.

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The importance of using identical preparation procedures is well illustrated by the data obtained on a series of samples consolidated in the 3 in. diameter cell. The starting material was flocculated natural kaolinite slurry at 1.90 g H_2O/g clay for all samples. From the data in Table 2 treatment 4, it would appear that sample reproducibility is non-existent. Although the slurry was at 1.9 g H_2O/g clay in each instance, there was considerable variation in the mechanics of preparing the slurry and transferring the slurry to the consolidation cell. For example, with batch No. 6 an effort was made to evacuate the slurry to remove all air. Batches 7 and 8 were prepared separately but simultaneously by an identical procedure except that No. 7 was poured into the cell rapidly, requiring about 5 sec for the operation and No. 8 was poured into the cell somewhat more slowly, the transfer requiring about 3 min. The data show that there is not a large difference in the average PR, $\overline{PR}$, between batches 7 and 8: however, the top third of No. 8 had an $\overline{PR}$ of 15 while the bottom third had an $\overline{PR}$ of 9.

TABLE 2—REPRODUCIBILITY OF FABRIC BETWEEN BATCHES OF KAOLINITE PREPARED FROM SLURRY*

Treatment	Isotropic consolidation	Batch	Average peak ratio for maximum orientation Direction	SD†
1	In balloon (1 kg/cm²)	1962	2.77	0.2
		1964	2.65	0.2
2	In wax (0 kg/cm²)	A	2.65	0.4
		B	2.84	
	Anisotropic consolidation			
3	In 9.5 in. diam. cell (1.5 kg/cm²)	I	10.8	1.2
		II	11.2	1.7
4	In 3 in. diam. cell (1.0 kg/cm²)	1	8.6	1.8
		3	23	5.5
		6	13	11.0
		7	9.2	2.2
		8	11	4.0

* 1.90 g H_2O/g kaolinite.
† Standard deviation of PR.

The excellent reproducibility obtained for all cases except the 3 in. cell, where variations in preparation are known to exist, suggests that a major factor for the wide scatter of data from the 3 in. cell arises from small variations in sample preparation. However, one must also recognize the possibility that the 3 in. cell, at least to some degree, is responsible for the large data variations. Conceivably, the PR variation results from sample disturbance during extrusion from the cell. The standard deviations in Table 2 generally exceed those for repetition on the same identical surface and are indicative of the vertical heterogeneity within the clay mass.

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DISCUSSION

The definition of fabric involves the spatial arrangement and distribution of both solids and voids. The present measurements are on the average geometrical arrangement of clay particles. The pole figure clearly reveals the direction and relative amount of orientation on a particular surface. Comparison of pole figures from surfaces taken parallel to some reference plane but at different lateral and vertical positions can provide a detailed picture of fabric variability. Such a procedure is very tedious and time consuming; further, the results do not lend themselves to numerical expression. For comparative purposes on the amount of orientation, PR was chosen because of the ease with which a numerical value on a particular surface can be obtained.

The data given herein clearly show a dependent relationship between PR and W; however, it is equally evident that the relationship is not a linear one. The PR of 2.7 given for treatment 1, Table 2, is only 3% of the way toward the maximum PR obtained on flocculated slurry while the W of 80° ϕ for the same treatment is 30% of the way toward the minimum obtained on flocculated slurry. At the other end of the scale, the maximum PR of 25 obtained on flocculated slurry is 12% of the way toward the maximum PR obtained on dispersed slurry while the W of 35° ϕ for flocculated slurry is 84% of the way toward the minimum W obtained on dispersed slurry. That is, as random fabric is approached W increases much faster than PR decreases, while the reverse holds for strong orientation.

In all experiments it has been observed that the direction of maximum orientation is within a few degrees coincident with the principal stress direction. The necessity to verify the maximum orientation direction cannot be over emphasized. For example, kaolinite anisotropically consolidated to 197 kg/cm^2 gave an average PR normal to the principal stress direction (a $\beta = 0$ specimen) of 25 while a specimen cut at $\beta = 45°$ gave an average PR normal to the specimen surface of only 2.8. The maximum amplitude ϕ curve of the $\beta = 0$ specimen peaked at $\phi = 0 \pm 2°$ while the maximum amplitude ϕ curve of the $\beta = 45°$ specimen peaked at 45° ϕ. PR measured at the spot of the maximum ϕ curve and at $\phi = 45°$ was 23 on the $\beta = 45$ specimen; this is within experimental error the same as obtained on the $\beta = 0$ specimen at $\phi = 0$. Thus, the pole figure device allows one to obtain powerful confirming data that the PR measured corresponds to the direction of maximum orientation.

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