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MICROSTRUCTURE AND PORE STRUCTURE OF IMPACT-COMPACTED CLAYS

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Abstract—The microstructures of impact-compacted kaolinite and illite clays, after drying, were investigated by pore size-distribution measurements, X-ray orientation determinations, and scanning electron microscopy. Clays compacted on the dry side of the optimum moisture content exhibited a domain structure with adjacent domains largely separated by micrometer-size interdomain voids; clays compacted at or above the optimum moisture content showed a more nearly massive structure, large interdomain voids being absent. Parallel orientation was observed within domains, but neighboring domains were generally tilted with respect to each other. In kaolinite compacted on the wet side of optimum, regions of local parallel orientation could be identified at high magnification as domain units. A significant volume of 200 Å–800 Å dia. pores in this clay was identified with spaces observed between the kaolinite plates within domains, for samples compacted both on the wet and dry sides of optimum. The subdomain structure was tentatively classified as "intergrown" in character. (001)/(020), (002)/(020), (001)/(060), and (002)/(060) orientation indices were calculated for the compacted kaolinite and compared with analogous measurements for fully-random and fully-oriented specimens of the same clay. The results indicated only a small degree of preferential orientation normal to the axis of compaction, with little difference between samples compacted either wet or dry of optimum. These results were consistent with scanning electron microscope interpretations, which suggested that the domains did not appear to orient themselves significantly under the influence of the compaction employed.

INTRODUCTION

THE PRESENT paper represents an attempt at synthesis of the results of several different modes of investigation of the microstructure of a clay compacted in a manner similar to, but not quite identical with the standard procedures in use in soil engineering tests. The characterization tools used were X-ray diffraction measurements of overall orientation, pore-sized distribution analysis by mercury porosimetry, and observation of fracture surfaces by scanning electron microscopy. It was hoped that a coherent picture of the microfabric would emerge from such a coordinated study on the same samples.

Since the microstructure appeared to vary strongly depending on moisture content at compaction relative to the "optimum"* value for the method used, discussion of the results is keyed to moisture content as the primary independent variable.

Hypotheses by Lambe (1958) and by Seed and Chan (1959) suggested that clays compacted at moisture contents less than optimum would tend to have a "flocculated" (i.e. edge-face) structure, while samples compacted at moisture contents greater than the optimum would tend to have a "dispersed" microstructure featuring more nearly parallel orientation of primary particles in a direction normal to the axis of compaction. Sloane and Kell (1966) investigated the microstructure of compacted kaolinite by transmission electron microscopic examination of replica preparations of fracture surfaces. They found no evidence for edge-to-face structure for the kaolinite samples compacted on the dry side of optimum, but rather found an essentially random arrangement of "packets", i.e. domains. These workers did find some orientation of the domains normal to the compaction axis for samples compacted at moisture contents above optimum, but of course no quantitative measurements could be made. The nomenclature of supraparticle units in clay microstructures was recently clarified by Smart (1969) who defined a domain as an aggregate of platy particles, laths, or tubes, the aggregate being large with respect to the size of the primary particle, and being so constituted that almost all of the primary particles are approximately parallel to a smooth plane or a curved surface of the aggregate. Smart provided a useful classification of domain and subdomain structures.

Scanning electron microscopy has been recently applied to the study of clay microstructure by a number of workers, including McKyes and Yong

*Moisture content of a particular clay at which the highest density is attained with a given compaction procedure.

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(1970), O'Brien (1970), and Gillott (1969). This technique permits rapid visualization of the arrangement of particles on fracture or other surfaces of clay masses, and while admittedly qualitative, provides much insight into the three dimensional arrangements of particles, aggregations of particles, and void spaces that constitute the clay microstructure.

Instrumental methods of determining the degree of orientation of particles of clay parallel to a given surface were pioneered by Mitchell (1956) who studied a number of natural and remolded clays by optical microscopy. Mitchell developed a procedure for preparing thin sections of wet clay by slow replacement of water with Carbowax, which was subsequently adapted by Martin (1966) to prepare clay surfaces for measurement of orientation by X-ray diffractometry. Unfortunately neither of these workers studied compacted clays. Martin, employing a pole-figure diffractometer, was able to measure preferred orientation in consolidated clay slurries occurring in any direction in the sample; conventional diffractometer measurements measure only orientation parallel to the sample face. Further interesting applications of these techniques to consolidated clays have recently been reported by Martin and Ladd (1970).

A comparatively neglected aspect of clay fabric studies is the quantitative description of the pore or void system, particularly in terms of the pore size distribution. The application of mercury intrusion techniques to this problem was recently described by the writer (Diamond, 1970).

PREPARATION OF COMPACTED CLAYS

Two commercial clays were used in this study, a relatively fine-grained kaolinite ("Edgar Plastic Kaolin", furnished by the Edgar Plastic Kaolin Co.) and a poorly-crystallized illite ("Grundite", furnished by the Illinois Clay Products Co.). Both are largely but not completely monomineralic.

The air-dry clay, as commercially supplied, was mixed with appropriate amounts of water in a Patterson-Kelley twin shell solid–liquid blender. This mixer produces an almost perfectly homogeneous moisture content throughout a mass of material, without causing the smearing and shear-disaggregation commonly produced in rotating drum mixers. Approximately 3000g quantities of clay were mixed at one time, the water being added in 100 ml increments while mixing. The mixing process was continued for 1 min after each addition of water.

After final mixing, the clay was compacted in three layers in a cylindrical mold 3·3 cm in dia. and 7·1 cm high. The drop hammer weighed 1640g and was allowed to fall a distance of 25·4 cm at each blow. The level of compactive effort employed was usually 30 such blows for each of the three layers, although additional samples were prepared using lesser efforts of 20 and 10 blows per layer. The diameter of the drop hammer being only slightly less than that of the mold, essentially all of the surface of the sample was subjected to each impact; this avoids the disruptive effect of hammer penetration into the soil mass.

OPTIMUM MOISTURE CONTENT AND SHRINKAGE ON DRYING

The gross result of a compaction experiment is usually displayed in the form of a "compaction curve", a plot of dry unit weight vs. moisture content. Compaction curves vary considerably but they generally show a reasonably well-defined maximum in dry unit weight at a moisture content that is optimum for the particular soil, process, and level of effort.

Compaction curves for the impact-compacted kaolinite and illite are given in Figs. 1 and 2, each for 30 blows per layer and 10 blows per layer. For the 30 blow per layer compactive effort, the kaolinite has a maximum dry unit weight of 95 lbs per cu. ft at a moisture content of approximately 26 per cent; reduction of the compactive effort decreases the dry unit weight and increases the optimum moisture content. The illite shows a maximum dry unit weight of 112 lbs per ft^3 at an optimum moisture content of 15 per cent, and reduction of effort similarly reduces the dry unit weight and increases the moisture content.

One of the ways in which the microstructure of a compacted clay influences its behavior is expressed in the degree of shrinkage observed when a sample of the compacted material is permitted to dry. In the present study shrinkage was measured by determining the volumes of small portions of the dried compacted clay of known weight in a dilatometer (prior to determination of the pore-size distribution by mercury intrusion), and comparing the bulk density of the dried clay with that of the original compacted clay. The latter was measured from the known weight of solids compacted and the dimensions of the mold. While no great accuracy is claimed for the measurements, they disclosed that shrinkage of samples compacted at less than optimum moisture content was relatively small, and that for samples compacted at optimum or above was quite significant, and greater with increased moisture content above optimum. The values obtained (in percentages of the original volume) were as follows: for the kaolinites of Fig. 3, the samples compacted dry of optimum shrank between 4 and 7 per cent, the sample compacted at optimum 13 per cent, and the sample compacted

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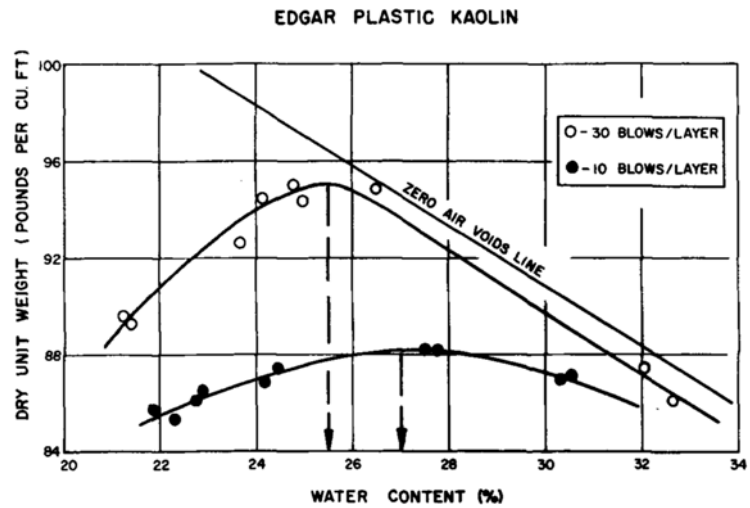


Fig. 1. Compaction curves for impact-compacted kaolinite at 30 blows per layer and at 10 blows per layer.

at the highest moisture content, 14 per cent; for the illites of Fig. 4, the "dry side" samples shrank 7 and 9 per cent respectively, the "wet side" samples 14 and 19 per cent, respectively.

The measurement of shrinkage on removal of water is of particular importance in the present work because two of the three methods of characterization used (mercury porosimetry and scanning electron microscopy) require complete removal of water from the specimen prior to examination. While it is possible to remove water from saturated clays by rapid freeze drying or by critical region drying (Gillott, 1969; Diamond, 1970), removal of the water without volume change is exceedingly difficult to accomplish for compacted clays. In consequence all of the examined were oven dried prior to examination, and appropriate consideration needs to be given to the extent of shrinkage and to the possibility of alteration of microstructure in the drying process in each case.

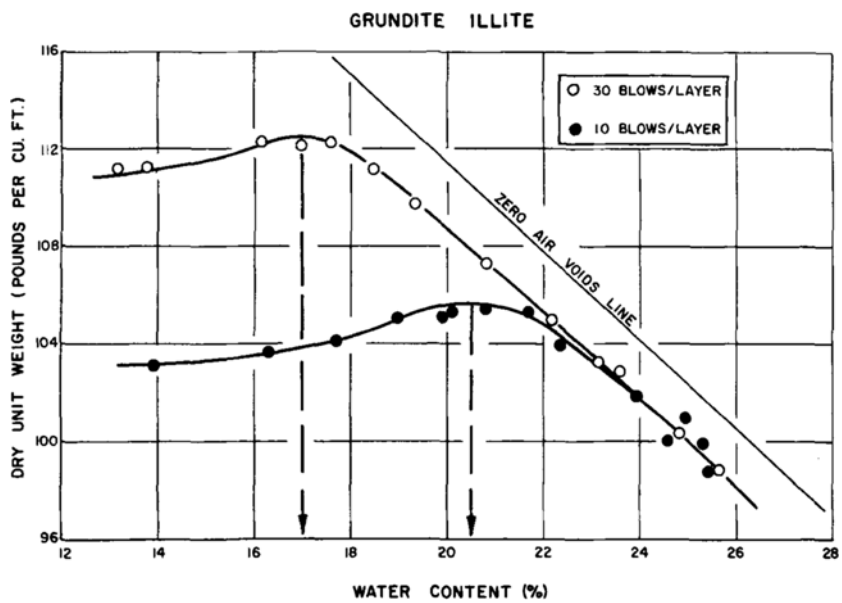


Fig. 2. Compaction curves for impact-compacted illite at 30 blows per layer and at 10 blows per layer.

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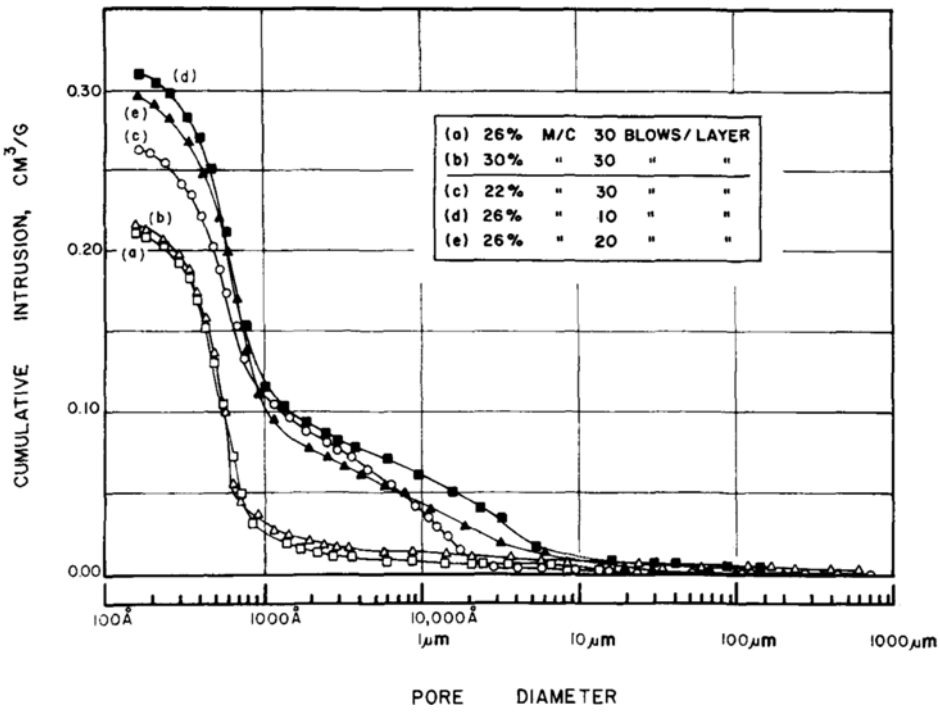


Fig. 3. Pore-size distribution curves for impact-compacted kaolinites.

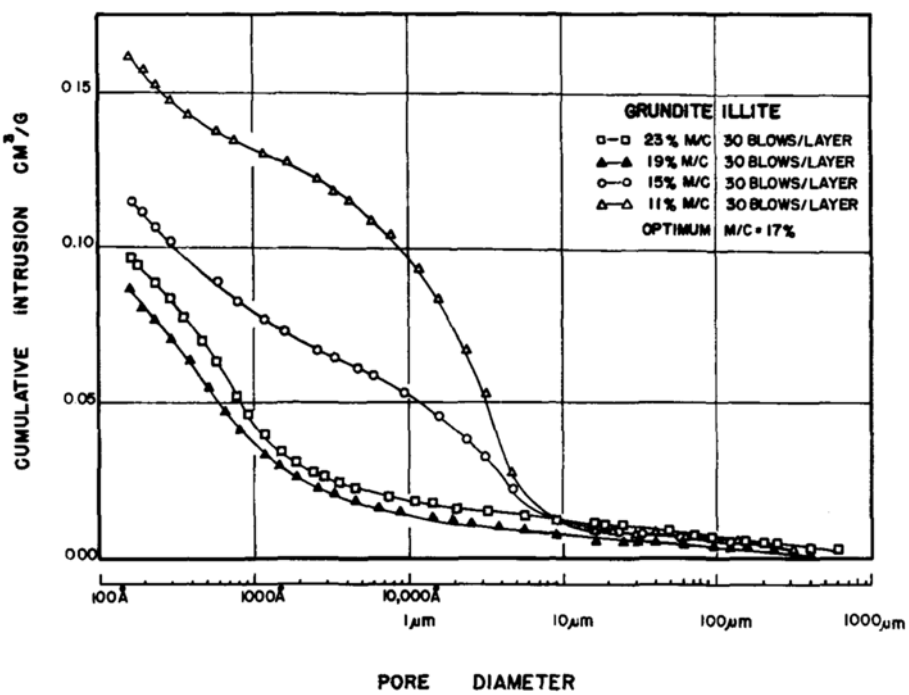


Fig. 4. Pore-size distribution curves for impact-compacted illites.

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PORE SIZE DISTRIBUTION CURVES

The interested reader is referred to an earlier paper (Diamond, 1970) for an exposition of the method of mercury porosimetry as applied to clays. The pore size distributions were determined with a modified Aminco-Winslow porosimeter of 15,000 psi capacity, using a horizontal filling device capable of reading pores of up to about 800 microns in diameter. In each case the data of intrusion of mercury vs. pressure was converted to a curve of pore diameter vs. pressure using the equation appropriate for cylindrical pores (Washburn, 1921):

$$P = -4\gamma \cos\theta/d \qquad (1)$$

where P is the pressure, γ is the surface tension of the mercury (484 dynes/cm^2), θ is the contact angle, and d is the diameter of the smallest pore intruded at a given pressure. A contact angle of 147° (Diamond, 1970) was used for both clays. The data are presented as cumulative distributions cumulating from right (large diameters) to left (small diameters), the latter feature serving to preserve the normal logarithmic sense of the abscissa. The pore volume is expressed in cm^3 of pore space per oven-dry g of clay.

Figure 3 presents previously published pore-size distribution curves for compacted kaolinite (Diamond, 1970). The curves fall into two distinct groups, those compacted at or above the optimum moisture content for the compactive effort used (Figs. 3a and b), and those compacted at less than optimum (Figs. 3c–e). The former show negligible pores larger than 0·1μm in dia.; the latter show a considerable volume of such pores, which range in diameter upward to about 5μm. All of the curves show a considerable and approximately constant volume of pores of diameters between about 200 and 800 Å.

Similar data are presented in Fig. 4 for illite clays compacted at various moisture contents using a 30 blow per layer compactive effort, for which this clay's optimum moisture content is 17 per cent. For this clay as well, samples compacted at or above the optimum moisture content lack the micrometer-sized pores that are present for samples compacted at less than the optimum moisture content.

X-RAY ORIENTATION MEASUREMENTS

A series of measurements of the overall orientation of the particles on surfaces normal to and parallel to the compaction axis were carried out for the dried impact-compacted kaolinite specimens. The X-ray diffraction pattern of the illite was found insufficiently intense to make the observations practical for this clay.

As previously mentioned, all the specimens in this study were examined after oven drying, and some shrinkage accompanied the drying process. The extent to which this shrinkage altered the pre-existing overall orientation of the primary particles is conjectural. The writer is of the firm opinion that in the present case of a highly compacted stiff kaolinitic clay with well developed domain structure such changes should be relatively small. One could use the Carbowax procedure with undried samples, and avoid the drying step. However, although Mitchell (1956) felt that "the effects of the water replacement on the clay are believed to be negligible, since no volume changes were observed on treatment", Quigley and Thompson (1966) found volumetric shrinkage of about 21·5 per cent on using the same method with Leda clay.

After oven drying, plane faces were cut normal to and parallel to the axis of compaction with minimal disturbance of the orientation of the particles then existing, by the following procedure: block samples of the clay were broken so as to expose a rough face with the required orientation. A new sharp heavy duty single-edged razor blade was clamped rigidly a fraction of an inch above a working surface, and the clay block was passed horizontally under the blade in such a way that a thin layer of approximately 0·5 mm was removed and a flat surface generated in a single pass. Successive surfaces could be cut and X-rayed sequentially from a single block, thus providing convenient preparation of replicate surfaces. Data are subsequently presented indicating that this procedure yields orientation measurements entirely equivalent to those obtained by the method of Carbowax intrusion and subsequent grinding of plane surfaces with no change in orientation.

X-ray measurements were confined to a single centered position on the sample holder for each replicate surface prepared. Since no provision was available for either tilt or rotation of the specimen, the measurements reflect only the degree of orientation in the plane of the cut face. Nickel-filtered copper radiation and conventional slit systems (1° beam slit, 0·2° detector slit, and medium resolution Soller slits) were used with a G. E. XRD-5 diffractometer for all of the measurements.

The parameters measured were the net peak heights above background of the (001), (002), (020), and (060) reflections of kaolinite. The order of magnitude of the individual net peak heights was approximately 2000, 1000, 250, and 150 cps, respectively, with background levels being approximately 200 cps for the first three peaks named and 75 cps for the last.

The extent of overall, as distinguished from local,

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orientation parallel to the surface presented to the X-ray beam is indicated by an "orientation index", such as the (001)/(060) or (002)/(060) indices of Brindley and Kurtossy (1961) or the (002)/(020) index used by Martin (1966). The present data permit four such separate indices to be constructed namely the (001)/(020), (002)/(020), the (001)/(060), and the (002)/(060). Table 1 gives the results of such measurements for replicate surfaces of kaolinite compacted using 30 blows per layer at approximately 30 per cent (above optimum), 26 per cent (optimum) and 22 per cent (below optimum) moisture contents.

The data of Table 1 suggest first that reproducibility is satisfactory, the standard error of the mean being generally above 3 per cent of the magnitude of the orientation index. Thus small differences in orientation index may be determined with a considerable degree of confidence.

reference show some such tendency, the comparisons using the (020) peak as a reference do not. It appears that at least for measurements on specimens that have been allowed to dry, the overall orientation normal to the axis is not significantly better for "wet-compacted" samples than for "dry-compacted" samples.

The orientation indices used in Table 1 do not indicate where the samples lie on the continuum between fully-random and perfectly plane-parallel arrays of particles. To formulate a scale for this purpose one must know the values of the indices to be expected for the particular clay at both extremes. Such data were secured as follows. For the random condition specimens were prepared using a slightly modified McCreery procedure (McCreery, 1945) with a mount that exposed a rectangular face of approximately the same dimensions as the compacted clay samples. For the maximum orientation

Table 1. X-ray orientation indices for kaolinite specimens

Orientation of surface with respect to axis of compaction	Moisture content (%)	Peak ratio* (001)/(020)	S.E.†	Peak ratio* (002)/(020)	S.E.†	Peak ratio‡ (001)/(060)	S.E.§	Peak ratio‡ (002)/(060)	S.E.§
Normal	30	5·91	0·11	2·99	0·09	10·73	0·22	5·59	0·14
	26	6·64	0·17	3·30	0·04	10·94	0·35	5·59	0·11
	22	6·40	0·37	3·05	0·14	10·35	0·35	5·14	0·12
Parallel	30	4·60	0·17	2·22	0·07	7·31	0·24	3·60	0·15
	26	4·39	0·22	2·04	0·09	7·97	0·30	3·95	0·17
	22	4·05	0·11	2·19	0·09	6·84	0·15	3·50	0·08

*Mean of eight replicate determinations, each on a separate surface.
†Standard error of the mean of eight determinations.
‡Mean of sixteen replicate determinations, each on a separate surface.
§Standard error of the mean of sixteen determinations.

On the basis of previous results by other workers, one would expect some overall preferred orientation or alignment of particles in the plane normal to the axis of compaction, i.e. parallel to the original surface of the compacted specimen. The results of Table 1 are in accord with this expectation; in each case the orientation indices of samples cut normal to the compaction axis are significantly higher than those cut parallel to the axis, the increase in index being of the order of 50 per cent.

One would also expect, on the basis of results previously cited, that particle orientation on the plane normal to the compaction axis would be greater for samples compacted at or above the optimum moisture content as compared to samples compacted at a lesser water content. The results of Table 1 are not in accord with this expectation. While the comparisons using the (060) peak as a

condition, after a number of trials of various procedures it was found that the best orientation was attained by slowly drying relatively concentrated suspensions of the kaolinite (3 to 4 ml water per g of clay) on glass microscope slides. This is essentially the procedure of Martin (1966).

After preliminary trials, eight replicate samples of each kind were prepared and X-rayed, with the results shown in Table 2. The wide range in orientation index in going from the random to the oriented condition is immediately apparent. For example, the (001)/(060) index goes from 4·4 to 170.

It is now proposed to define a parameter, to be called the "degree of orientation" which will express the extent to which a given sample surface is oriented relative to the best experimental estimate of "perfect" orientation for that particular clay. The degree of orientation is defined as follows:

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Table 2. X-ray orientation indices for "random" and for "fully-oriented" kaolinite*

Specimen type	Peak ratio (001)/(020)	S.E.	Peak ratio (002)/(020)	S.E.	Peak ratio (001)/(060)	S.E.	Peak ratio (002)/(060)	S.E.
Random powder mount	2·28	0·04	1·27	0·03	4·41	0·07	2·45	0·03
"Fully-oriented" layer	118·3	5·2	76·9	3·9	170·0	6·9	110·0	4·8

*Data are means and standard errors of the means of eight replicate determinations, each on a separate specimen.

$$\text{Degree of orientation} = \frac{\left(\begin{matrix}\text{Orientation index}\\ \text{of sample surface}\end{matrix}\right) - \left(\begin{matrix}\text{Orientation index}\\ \text{of random mount}\end{matrix}\right)}{\left(\begin{matrix}\text{Orientation index of}\\ \text{"fully"-oriented mount}\end{matrix}\right) - \left(\begin{matrix}\text{Orientation index}\\ \text{of random mount}\end{matrix}\right)}$$

In this expression the denominator is the numerical range in orientation index value possible with the particular clay, and the numerator is the observed excess of the orientation index for the sample surface from that of the least oriented (random) condition. The degree of orientation is thus expressed as a number between 0 for the random state and 1 for the perfectly oriented state. A separate estimate of the degree of orientation can be made for each of the four orientation indices. Use of four parallel indices might be expected to yield four different numerical values for a given surface, but one would expect that the different estimates be reasonably concordant, and certainly they should all describe the same trends when applied to a suite of samples. Values for the "degree of orientation" for each of the kaolinite samples for which data was given in Table 1 are given in Table 3.

A number of points emerge from consideration of Table 3:

(1) Most important, it is clear that for all of the samples, the overall degree of parallel orientation in the plane of the specimen is small, no estimate being as high as 4 per cent of the range between random and fully oriented.

(2) The individual estimates of degree of orientation are systematically higher for estimates involving the (001) peak height than for corresponding estimates involving the (002) peak. The latter are consistently only about two-thirds of the former.

(3) The choice of (0*k*0) peak is immaterial, the estimates using (020) peak being essentially consistent with corresponding estimates using the (060) peak.

(4) Within the small range of degree of orientations observed (roughly 1–4 per cent of the possible range), the orientations normal to the axis of compaction are clearly better by a factor of two than the almost random condition of the faces cut parallel to the axis.

(5) No consistent difference is observed between samples compacted at or above optimum moisture content and samples compacted dry of optimum.

COMPARISON OF DRIED AND CUT SPECIMENS WITH CARBOWAX IMPREGNATED AND GROUND SPECIMENS

Criticism might be expressed that the orientations measured may not have reflected those pre-existing in the compacted clay before specimen preparation. Unfortunately, no data are available which provide a direct comparison of the present results with those of other procedures. However, a

Table 3. Degree of orientation of kaolinite specimens

Orientation of surface with respect to axis of compaction	Moisture content (%)	Degree of orientation: From (001)/(020) orientation index	From (002)/(020) orientation index	From (001)/(060) orientation index	From (002)/(060) orientation index	Av.
Normal	30	$0{\cdot}03_1$	$0{\cdot}02_3$	$0{\cdot}03_8$	$0{\cdot}02_9$	$0{\cdot}03_0$
	26	$0{\cdot}03_8$	$0{\cdot}02_7$	$0{\cdot}03_9$	$0{\cdot}02_9$	$0{\cdot}03_1$
	22	$0{\cdot}03_6$	$0{\cdot}02_4$	$0{\cdot}03_6$	$0{\cdot}02_5$	$0{\cdot}03_1$
Parallel	30	$0{\cdot}02_0$	$0{\cdot}01_3$	$0{\cdot}01_8$	$0{\cdot}01_1$	$0{\cdot}01_5$
	26	$0{\cdot}01_8$	$0{\cdot}01_0$	$0{\cdot}02_2$	$0{\cdot}01_4$	$0{\cdot}01_6$
	22	$0{\cdot}01_5$	$0{\cdot}01_2$	$0{\cdot}01_5$	$0{\cdot}01_0$	$0{\cdot}01_3$

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series of comparisons is available for the same kaolinite compacted in a different manner (static compaction alternately from two ends). The clay used was mixed with 21 per cent water in the same manner as previously described, and statically compacted to void ratios of 0·8 and 0·6, a somewhat higher degree of compaction than that used with the impact-compacted samples. Blocks were cut from samples in their original moist-compacted state, impregnated with Carbowax at 65°C, cooled, and ground with 600-mesh silicon carbide, as described in detail by Martin and Ladd (1970). Companion samples were oven dried and cut in accordance with the procedure previously described. Both sets of specimens were X-rayed in the standard manner and the results obtained are given in terms of orientation indices in Table 4.

It is apparent that the present method gives results quite consistent with those obtained by the Carbowax intrusion and grinding procedure, except that the individual numerical values are slightly higher in each case in terms of orientation index. The "degree of orientation" that one would calculate for individual specimens described in Table 4 would be for all practical purposes identical, for the two methods.

It should be noted, however, that the shrinkage on oven drying of these statically-compacted samples is negligible in comparison with that undergone by the less highly compacted impact-compacted clays that form the subject of this report; thus the evidence of Table 4 does not preclude the possibility that the orientations observed on the dried samples might have been influenced by the drying process.

OBSERVATION OF MICROSTRUCTURE BY SCANNING ELECTRON MICROSCOPY

Specimens of the dried compacted kaolinite and illite were prepared by fracturing the compacted samples so as to generate small blocks of the order of 1 cm on a side, with surfaces that were essentially either normal or parallel to the compaction axis. The blocks were glued to metal stubs with Duron plastic cement, and coated to provide surface electrical conductivity. The coating consisted of a layer of carbon about 50 Å thick topped with a layer of gold-palladium alloy about 200 Å thick. Conductance to ground was insured by painting a small stripe of silver paint between the upper portion of the specimen and the edge of the metal stub. The instrument used was a Cambridge Stereoscan scanning electron microscope, operated usually at 25 kV. A tilt angle of the order of 30°–60° was usually employed, but this varied to suit the convenience of the operator. The photomicrographs were exposed on Ilford HP-3 roll film and specially processed for high resolution.

Insofar as appearance was concerned, it was not possible to differentiate between normal and parallel surfaces of the same sample; no consistent differences being established despite efforts to do so. This is taken as confirmation that orientation normal to the compaction axis, while large enough to be detected by the sensitive X-ray method, is of too minor a degree to be apparent in the qualitative visual observations in the microscope.

Micrographs representing the appearance at low magnification of fracture surfaces of kaolinite compacted at 30 per cent, 26 per cent and 22 per cent moisture contents, respectively, are shown as Figs. 5a–c. It is clear even at this level of magnification that the first two are very much alike, and that the sample compacted at the moisture content less than optimum has a different microstructure. In the latter case, the material is seen to be made up of individual aggregations or domains several μm in size, with void spaces of the same order of size forming an inter-connected system between the domains. In contrast, in Figs. 5a and b individ-

Table 4. Comparison of orientation indexes for dried, cut specimens with those for corresponding carbowax-impregnated specimens (statically-compacted kaolinite)*

Void ratio	Specimen type	Surfaces normal to compaction axis				Surfaces parallel to compaction axis			
		(001)/(020)	(002)/(020)	(001)/(060)	(002)/(060)	(001)/(020)	(002)/(020)	(001)/(060)	(002)/(060)
0·6	Dried and cut	5·2	3·3	9·0	5·8	3·0	1·8	5·4	3·3
	Carbowax intruded	4·7	2·9	8·1	5·0	2·5	1·5	4·3	2·5
0·8	Dried and cut	4·8	2·8	8·5	5·0	3·5	2·0	6·2	3·5
	Carbowax intruded	4·4	2·5	7·6	4·2	3·0	1·9	5·1	3·3

*All data are means of eight replicate determinations.

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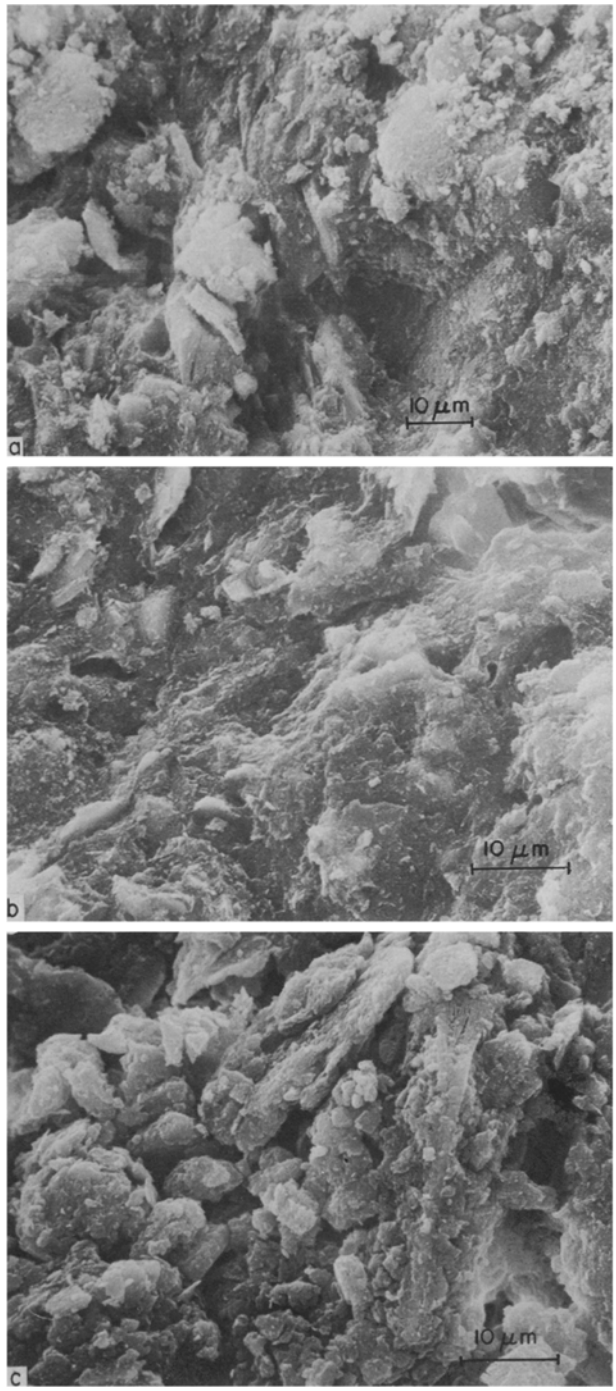


Fig. 5. Low-magnification scanning electron micrographs of impact-compacted kaolinites: (a) Compaction moisture content 30 per cent; (b) Compaction moisture content 26 per cent; (c) Compaction moisture content 22 per cent.

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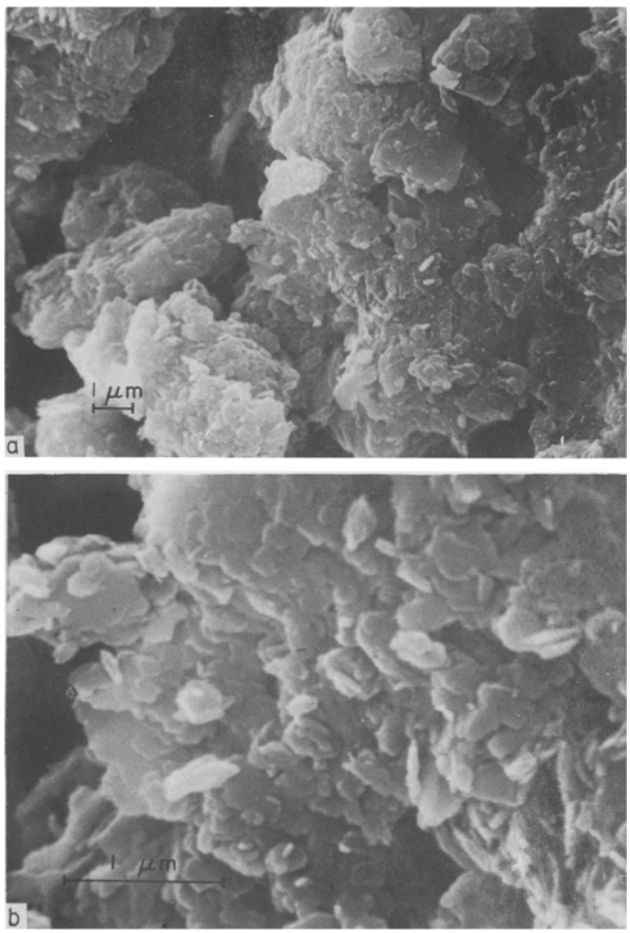


Fig. 6. Scanning electron micrographs of kaolinite compacted at 22 per cent moisture content, showing detailed structure of individual domains.

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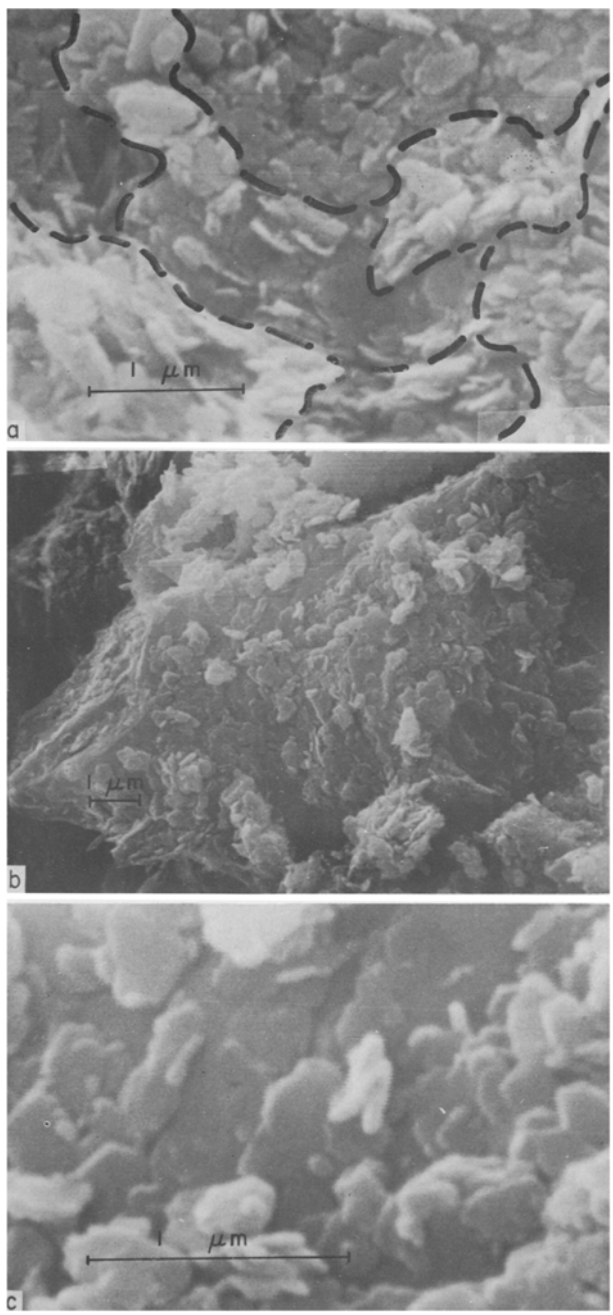


Fig. 7. Scanning electron micrographs of kaolinite compacted at 30 per cent moisture content: (a) Local domain structure and small pores between kaolinite platelets; (b) A very large domain; (c) High-magnification view of top center of above, showing local extent of parallel orientation and possible intergrown domain structure.

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ual aggregations cannot be distinguished, and there are few μm-sized voids visible. These observations with respect to voids corroborate the pore-size distribution data plotted in Fig. 3, which indicate significant void contents in diameters between about 0·2 and 5μm for samples compacted dry of optimum, but not for samples compacted wet of optimum.

Details of the microstructure of the specimen shown in Fig. 5c are shown in Figs. 6a and b; Fig. 6b is high magnification view of the center of Fig. 6a. It is clear that the domains are composed of individual kaolinite plates in approximately parallel orientation. Adjacent domains are mostly not oriented in the same direction, but rather the prevailing orientation planes are tilted at angles to each other. Thus while internal orientation within a domain is considerable, large-scale preferential orientation in any given plane does not occur.

Figure 5c and Fig. 6a indicate that neighboring domains touch at contact points but not generally along surfaces for specimens compacted dry of optimum. In contrast, in specimens of clays compacted at or above the optimum moisture content, the domains are in sufficiently close contact that it is hard to distinguish their boundaries. They remain as regions of more-or-less parallel particle orientation within the seemingly massive structure depicted in Figs. 5a and b. An illustration of a local region of the field shown by Fig. 5a at high magnification is given in Fig. 7a. Adjacent domains may be approximately delineated by drawing boundaries around local regions of parallel orientation, in the manner done by Smart (1967) for thin-section transmission electron micrographs.

The pore-size distributions of all the kaolinite samples indicate a more-or-less uniform and considerable content of pores in the diameter range of about 200 Å to about 800 Å. The nature of these fine pores is revealed in Fig. 7a, which suggests that they are largely gaps between individual kaolinite plates within the domains.

No effort was made to systematically explore the distribution of domain sizes in this study, but the comparatively few observations made suggest that they might range from perhaps 2 to more than 10 μm in size; and that they do not have any characteristic shape. The appearance of a relatively large domain is illustrated in Fig. 7b. The extent of local parallel orientation within this structure is illustrated in Fig. 7c, which is a high-magnification view just above the center of Fig. 7b.

Figure 7c also provides some tentative information on the nature of the contact between individual particles within a given domain. While it is difficult to be certain since we are operating at the limit of resolution of the scanning electron microscope, it appears that individual platelets are partially intergrown with their neighbors at lines of contact within a layer of platelets. Such a structure would correspond to the "intergrown domain" classification of Smart (1969). If true, such an intergrown structure would by itself tend to account for the relative constancy of the size distribution of fine pores discussed earlier.

In addition to the observations on the compacted kaolinite samples, a limited amount of investigation was conducted on the compacted illites. Similar conclusions were drawn for this clay; i.e. that samples compacted at moisture contents less than optimum showed a microstructure of aggregates separated by interconnected voids, while samples compacted at or above the optimum moisture content showed a more nearly massive structure presumably comprised of domains largely in mutual contact. The identification of individual domains was considerably more uncertain with illite since the individual particles seem to be thinner and twist and bend at the fracture surface.

CONCLUSIONS

(1) The microstructure of impact-compacted clays (after drying) is different for samples compacted on the dry side of optimum than it is for samples compacted at or above the optimum moisture content.

(2) These differences are shown to relate primarily to mutual packing of "domains" or aggregates of individual plates, the domains being of the order of 5μm in size in the kaolinite examined. In dried clay that was compacted below optimum, they touch each other only at peripheral points, and leave large interdomain void spaces; in dried clay that was compacted at or above optimum they are in close contact and leave few interdomain spaces.

(3) There is only a small degree of overall preferential orientation normal to the compaction axis as compared with planes parallel to the compaction axis. For dried specimens, this preferential orientation is not more pronounced for wet-compacted clay as compared to clay compacted on the dry side of optimum.

(4) The content of fine pores (200–800 Å dia.) found in kaolinite samples irrespective of compaction moisture content is ascribed to intradomain spaces between individual clay plates. The subdomain structure is tentatively classified as "intergrown domain" in the classification of Smart.

(5) X-ray diffraction measurements of overall particle orientation should be interpreted with caution, and in particular, should include some indication of where the particular results obtained fit on the continuum between perfectly random and

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fully plane-parallel and member states. A measure of this characteristic, called the "degree of orientation" is suggested.

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Résumé—Les microstructures de kaolinite et d'illite compactées par choc, ont été étudiées, après séchage, par des mesures de distribution des pores, des déterminations d'orientation aux rayons X, et par microscopie électronique à balayage. Les argiles compactées à une humidité inférieure à l'humidité optimum montrent une structure par domaines, avec des domaines adjacents très nettement séparés par des vides "intermédiaires" de la taille du micron; les argiles compactées à une humidité égale ou supérieure à l'humidité optimum montrent une structure beaucoup plus proche de l'état massif, les grands vides séparant les domaines étant absents. Une orientation parallèle a été observée à l'intérieur des domaines, mais les domaines voisins sont généralement désorientés les uns par rapport aux autres. Dans la kaolinite compactée au dessus de l'humidité optimum, des régions localisées à orientation parallèle ont pu être identifiées, aux forts grossissements, à des domaines unitaires. Un volume significatif de pores compris entre 200 et 800 Å a été identifié, dans cette argile, avec les espaces observés entre les plaques de kaolinite présentes à l'intérieur des domaines, pour les échantillons compactés à la fois à des humidités inférieures et supérieures à l'optimum. On a tenté de classer selon un caractère "d'interdéveloppement" la structure des sous domaines. Les indices d'orientation (001)/(020), (002)/(020), (001)/(060) et (002)/(060) ont été calculés pour la kaolinite compactée, et comparés aux résultats de mesures analogues concernant des échantillons de la même argile totalement désorientés. Les résultats indiquent seulement un petit degré d'orientation préférentielle normale à l'axe de compaction, avec une faible différence entre les échantillons compactés soit au dessus, soit au dessous de l'optimum. Ces résultats sont en accord avec l'interprétation des observations au microscope électronique à balayage, qui suggèrent que les domaines ne semblent pas s'orienter d'une façon significative sous l'effet de la compaction utilisée.

Kurzreferat—Die Mikrogefüge von stoss-verdichteten Kaolinit-und Illittonen, nach dem Trocknen, wurde durch Messungen der Porengrösseverteilung, Bestimmungen der Röntgenorientierung und abtastende Elektronenmikroskopie untersucht. Tone, die an der trockenen Seite des optimalen Feuchtigkeitsbereichs verdichtet wurden wiesen ein Domänengefüge auf, wobei angrenzende Domänen weitgehend durch Leerstellen von Mikrometergrösse getrennt waren. Die Tone, die an oder oberhalb des maximalen Feuchtigkeitsgehaltes verdichtet wurden, zeigten eine mehr annähernd massive Struktur, in der grössere Leerstellen zwischen den Domänen fehlten. Innerhalb der Domänen wurde Parallelorientierung festgestellt, doch waren benachbarte Domäne im algemeinen gegeneinander geneigt. In Kaolinit, der an der nassen Seite des Optimalwertes verdichtet wurde, konnten Bereiche

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mit örtlicher Parallelorientierung bei starker Vergrösserung als Domäneneinheiten identifiziert werden. Ein bedeutendes Volumen von 200–800 Å Durchmesser Poren in diesem Ton wurde mit Zwischenräumen, die in den Kaolinitplatten zwischen Domänen beobachtet werden, identifiziert, und zwar in Proben die sowohl an der nassen als auch an der trockenen Seite des Opttimums verdichtet wurden. Das Subdomängefüge wurde vorläufig als "zwischengewachsen" klassifiziert. Es wurden (001)/(020), (002)/(020), (001)/(060) und (002)/(060) Orientierungsindexe berechnet für die verdichteten Kaolinite und mit analogen Messungen für komplett dem Zufall überlassene und komplett orientierte Proben des gleichen Tons verglichen. Die Ergebnisse deuteten an, dass nur ein geringes Mass an bevorzugter Orientierung senkrecht zur Verdichtungsachse bestand, mit wenig Unterschied zwischen Proben, die an der nassen oder an der trockenen Seite des Optimums verdichtet worden waren. Diese Ergebnisse stimmten überein mit Beobachtungen am abtastenden Elektronenmikroskop, die andeuteten, dass die Domänen sich unter dem Einfluss der zur Verwendung gelangenden Verdichtung nicht bedeutend orientieren.

Резюме — Методами измерения распределения пор по размерам, рентгенографии и сканирующей электронной микроскопии изучена микроструктура подвергнутых ударному сжатию каолинитовых и иллитовых глин после их высушивания. Глины, подвергнутые сжатию в сухом состоянии при оптимальной влажности, обнаруживают доменную структуру, в которой соседние домены разделены междоменными полостями. Глины, подвергнутые сжатию при оптимальном или более высоком содержании влажности, характеризуются более массивной структурой, в них отсутствуют большие междоменные полости. Внутри доменов устанавливается параллельная ориентация частиц, но соседние домены обычно наклонены по отношению друг к другу. В каолините, подвергнутом сжатию в мокром состоянии при оптимальной влажности, области локальной параллельной ориентировки могут быть идентифицированы при большом увеличении как доменные ячейки. Существенный объем пор с диаметром в 200–800 Å в этой глине обусловлен промежутками между каолинитовыми пластинками внутри доменов в образцах, подвергнутых сжатию как в сухом, так и в мокром состоянии. Субдоменная структура предварительно была классифицирована как «прорастание». Для компактного каолинита вычислены ориентационные индексы (001)/(020), (002)/(020), (001)/(060) и (002)/(060) и сопоставлены с аналогичными показателями для образцов этой же глины с полностью беспорядочным и полностью ориентированным расположением пластинок. Полученные результаты указывают на слабую степень преимущественной ориентировки перпендикулярно к оси сжатия с незначительными различиями для образцов, подвергнутых сжатию в сухом и мокром состоянии. Подобные результаты согласуются с данными сканирующей электронной микроскопии, указывающими на то, что домены сами по себе не приобретают существенной ориентировки под влиянием сжатия.

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