

MICRO-STRUCTURE OF COMPACTED KAOLIN CLAY

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ABSTRACT

The present paper deals with the micro-structure of a kaolin clay compacted in accordance with JIS*** A 1210. The methods used for investigation are the scanning electron microscopy using samples impregnated with polyethylene glycol, the X-ray diffraction, and the permeability test method. From this series of experiments, it is recognized that the micro-structure of the compacted kaolin clay mainly consists of block structures which are almost independent of the inner particle arrangement and are of the three-dimensional structure. The two-dimensional feature, therefore, varies depending upon the direction and position to be observed. It is also recognized that the parallelism as a soil mass increases more or less according to the molding moisture content as indicated by the results of X-ray analysis and $k_0 S^2$ from the Kozeny-Carman equation, and the change of this parallelism may be caused not by the change of each particle arrangement but by flattening and/or deforming of block structures.

Key words: soil structure, density, clay compaction, microscopy, permeability

IGC: D3

INTRODUCTION

In the study of the engineering properties of clayey soils, many investigators have been concerned with the fundamental problem of how these properties depend on the micro-structure and/or the particle (crystal) composition. It is generally believed that the arrangement of particles in fine-grained soils has a profound effect upon the properties of such soils.

In these investigations, the contribution by Lambe (1958) has led us to recognize the importance of micro-structure problem. According to his concept, as shown in Fig. 1, the structure on the dry side of the optimum moisture content is of flocculation of particles, and when

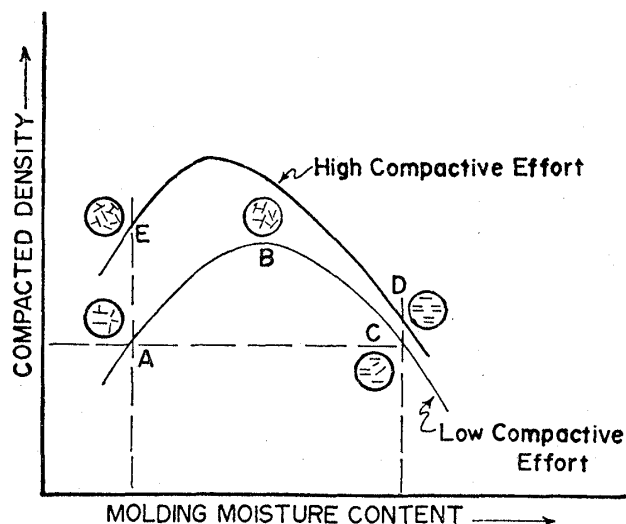


Fig. 1. Effect of compaction on soil structure (after T. W. Lambe)

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remolding moisture increases a more oriented or parallel particle structure (dispersed) is formed by development of double layer water film. His concept was obtained from the colloid chemical consideration in addition to the experimental data obtained by Mitchell's method (Mitchell 1956). Seed and Chan (1959) supported the Lambe's concept, and suggested that particle orientation was caused by shear strain in soil during compaction. However, the development of electron-microscopes (EM), especially a scanning EM, brought an epoch-making progress to observation technique for micro-structure. Sloane and Kell (1966), using the replica method of EM to a compacted soil, supported the Lambe's concept in general, but in detail, showed the block-like structure in soil, and defined the terms of "book-house" and "parallel packet." Barden and Sides (1971) using a scanning EM concluded that under high magnification there was no marked difference between structures on the dry side and the wet side of the optimum, the turbostratic structure (Aylmore and Quirk, 1960) was noted in all cases and only a small number of individual particle structures were observed. They also concluded that under low magnification (by naked eye) there was a marked difference, in which the "pellet-like macro-peds" about 3-6 mm in diameter were observed characteristically on the dry side of the optimum, but they disappeared with increase in moisture content.

From the previous works mentioned above, it is considered that the development of EM would clarify the proposition that the structure of clayey soils is made up of several structural units (dimensions) from micro- to macro-order, and would reveal the truth of apparent features (e.g., packet, stack, turbostratic, book-house, ped, domain, and so on) by the high degree of magnification. During the early stages of development, only the individual particle relationships (dispersed, flocculated, etc.) were discussed.

X-ray diffraction from a surface of crystal aggregate is controlled by the crystal concentration and the crystallographic orientation in the irradiated volume. The index for a degree of orientation of clay particles has been proposed in several ways; the absolute value of peak height (Quigley and Thompson 1966, and many others), the peak ratio of the amplitude and/or the intensity ratio of the product of the peak amplitude and the peak width (Martine 1966), or the ratio of area of the (00 l) to the ($hk0$) peaks (Quigley and Ogunbadejo 1972), the fabric index (Odom 1967, Gillott 1970), and others.

Though generally the method of analyzing preferred orientation is two-dimensional, Martine (1966) used the special technique for a three-dimensional analysis (Higgs, Friedman and Gebhard studied in 1960 using the device with three rotating axes for a rock sample).

MATERIAL AND TESTING METHODS

A kaolin, "Takahata Kaolin," sold by the Zeeklite Chemical and Mining Co. Ltd., was used in this investigation and its index properties are shown in Table 1. The kaolin was chosen because of its good uniformity and crystallinity of fine-grained aggregation at least finer than 20 μ . From the X-ray diffraction pattern, the following principal clay minerals were identified: kaolinite»sericite»quartz.

Table 1. Index properties of kaolin

G_s	LL (%)	PL (%)	Clay (%)	Silt (%)
2.69	70.5	33.6	90	10

G_s : specific gravity, LL: liquid limit, PL: plastic limit

The kaolin was ground to powder finer than 420μ , and mixed with a required amount of distilled water. Each sample was then stored in a pan for at least 24 hours to allow the moisture content to equalize. Cylindrical samples of 10 cm in diameter and 12.7 cm high were compacted in accordance with JIS A 1210 (compactive effort 5.6 cm kg/cm³) which was nearly equivalent to Standard Proctor. Furthermore, permeability tests and direct shear tests were performed on the compacted clays to determine the engineering properties.

Samples for the scanning electron microscope were prepared at the molding moisture contents of 4.27% (dry side), 25.9% (approximately the optimum) and 45.0% (wet side). After molding, two blocks ($2 \times 2 \times 1$ cm) normal and parallel to the axis of compaction were cut off from the central part of the second layer of each compacted sample. The blocks were immersed in liquid polyethylene glycol #6000 (produced by Lion Fat and Oil Co. Ltd.) at 63°C for four days by the procedure similar to that described by Mitchell (1956). After removing from polyethylene glycol, they were allowed to cool down to room temperature. Once impregnated with polyethylene glycol, the blocks were cut into smaller blocks by a cutter. Because of the large depth of focus in the image produced by the scanning electron microscope, the surfaces of specimen need not be flat. The surfaces for the scanning electron microscope were exposed by fracture. Two sections, the horizontal section oriented normal to the compaction axis and the vertical section oriented parallel to the compaction axis, were examined.

The microscope used in this investigation was a JSM-S1 manufactured by the Japan Electron Optics Laboratory. The instrument was operated at 10 KV. A tilt angle of the order of 15°-35° was usually employed, but it varied to suit the convenience of the operator.

For X-ray diffraction, two blocks normal and parallel to the compaction axis were cut off from the central part of each layer of compacted samples. The horizontal and vertical specimens of approximately 1.0 mm in thickness were cut off from blocks with a sharp razor blade to minimize disturbance of the orientation of the particles. X-ray diffraction patterns were obtained with Cu-K α radiation (30 KV, 15 mA) using Model ADG-101, Tokyo Sibaura Electric Co. The size of the rectangular surfaces exposed to X-ray was about 1.0×1.5 cm.

TEST RESULTS

Soil Testing

Fig. 2 shows the compaction curve of kaolin which indicates the maximum dry density of 1.350 g/cm³ at the optimum moisture content of 28.0%. The shear strength (normal stress maintained constant at $\sigma_c = 1.0$ kg/cm²) and the permeability versus the molding moisture content of kaolin are shown in Fig. 3 and Fig. 4, respectively. The relations between these engineering properties and the moisture content of compacted kaolin are similar to those obtained by other investigators. The difference in the dry density and in the shear strength of each layer caused

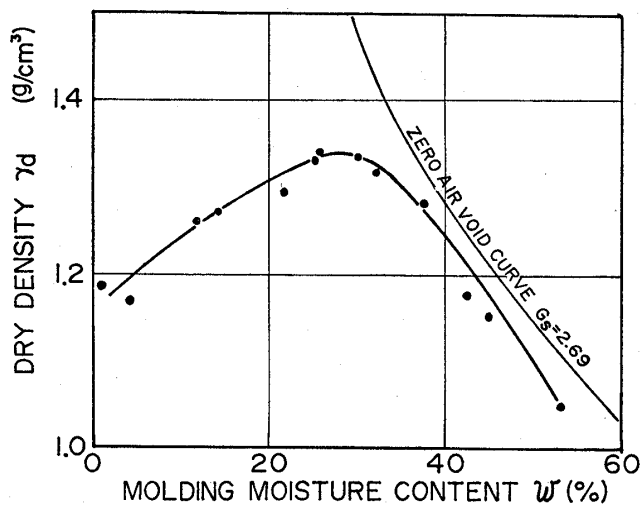


Fig. 2. Compaction curve for kaolin

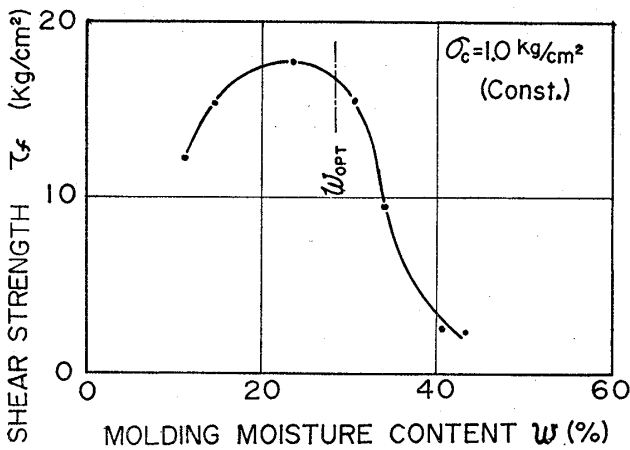


Fig. 3. Relationship between shear strength and molding moisture content for kaolin

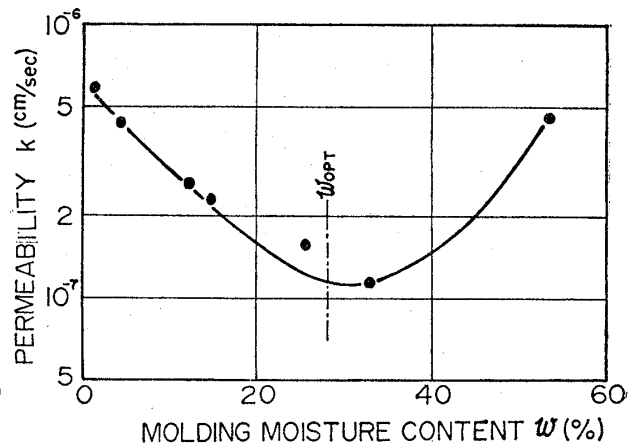


Fig. 4. Relationship between permeability and molding moisture content for kaolin

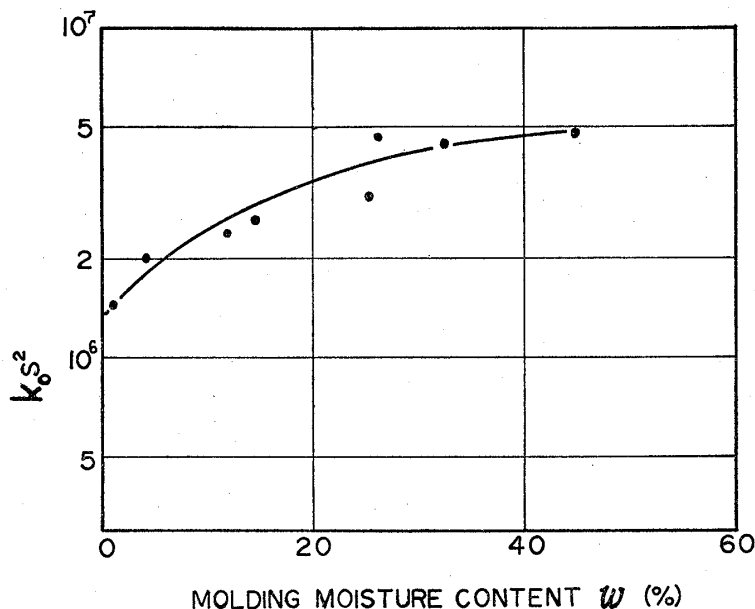


Fig. 5. Relationship between $k_0 S^2$ and molding moisture content for kaolin

by inequality of compaction energy is 10-20% and 10-40%, respectively. As the moisture content increases, however, the difference decreases and it is not recognized above 35%.

The relationship between $k_0 S^2$ from the Kozeny-Carman equation and the moisture content is shown in Fig. 5. Since the value of $k_0 S^2$ indicates the tortuosity of flow or actually the orientation of soil particles, it may be found from this figure that clay particles become oriented to the horizontal plane as the moisture content increases.

Scanning Electron Microscopy

The results of the scanning electron microscopy may be summarized as follows:

(a) Micro-structure on the dry side of the optimum moisture content

The results of the observation confirm the tendency of the parallelism of platelet particles to the horizontal direction, that is, the parallel arrangement of particles is more conspicuous in the V-section (viewed from the direction parallel to the fall of

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Photo. 1. ×1,000

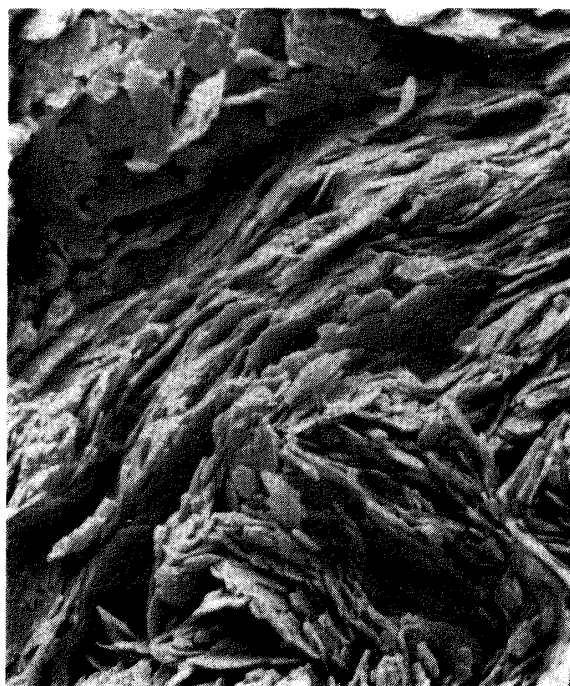


Photo. 2. ×3,000



Photo. 3. ×3,000

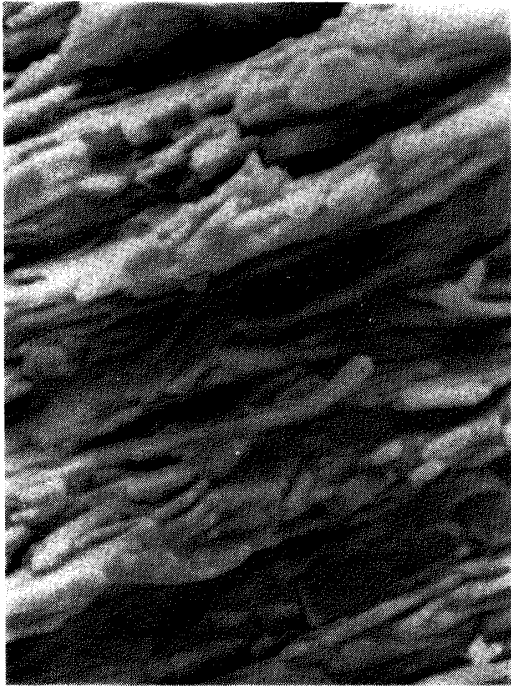


Photo. 4. $\times 10,000$

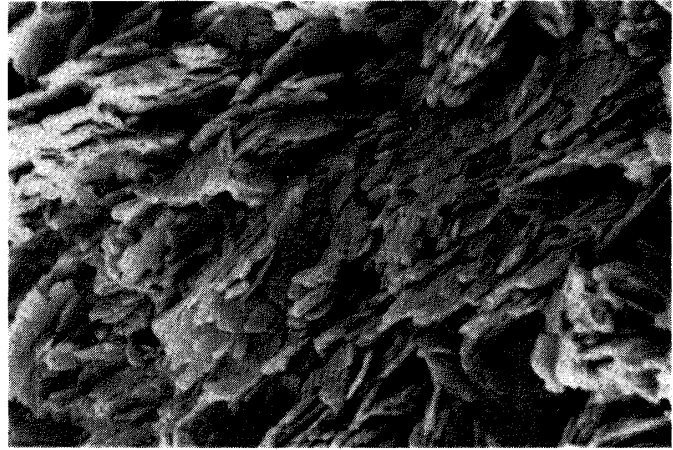


Photo. 5. $\times 3,000$

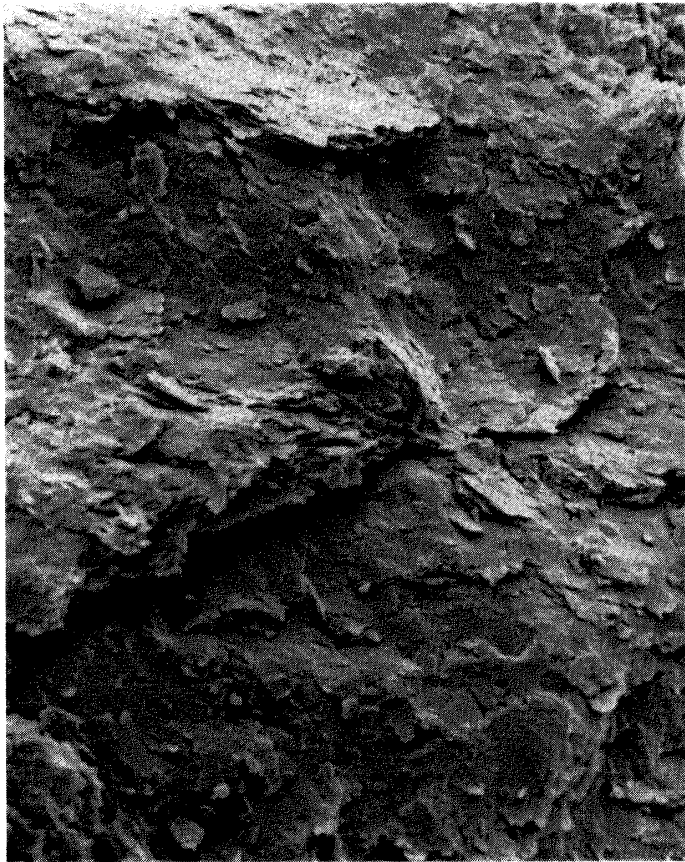


Photo. 6. $\times 1,000$

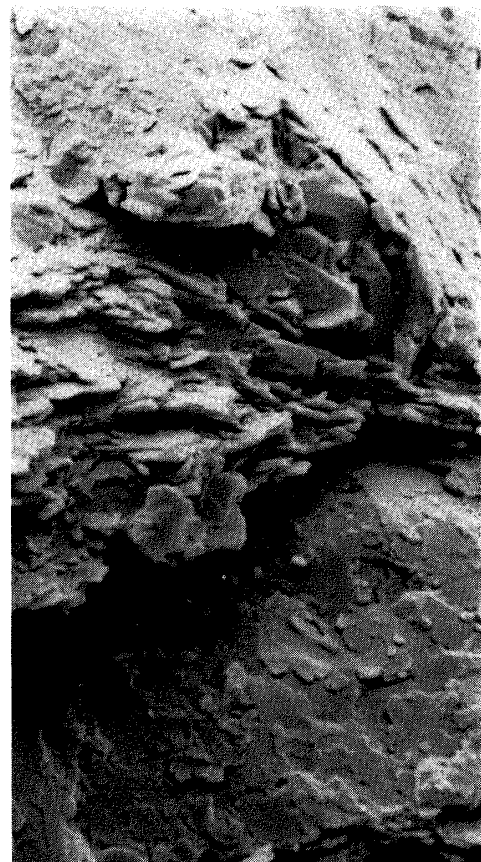


Photo. 7. $\times 3,000$

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Photo. 8. $\times 1,000$

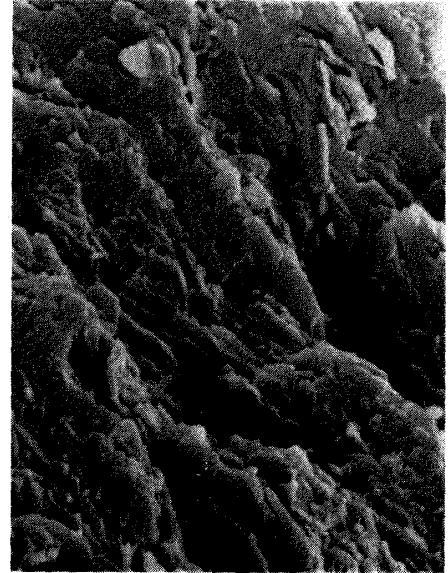


Photo. 9. $\times 3,000$

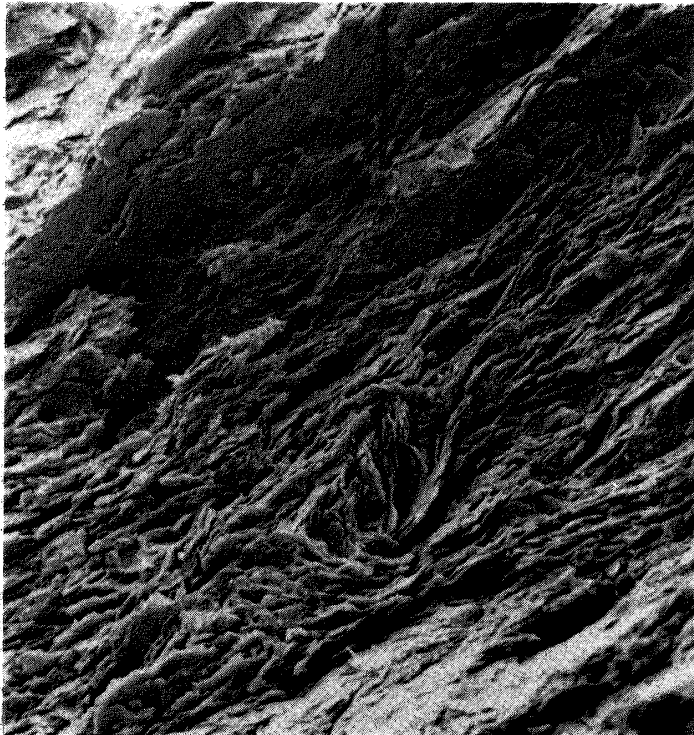


Photo. 10. $\times 1,000$

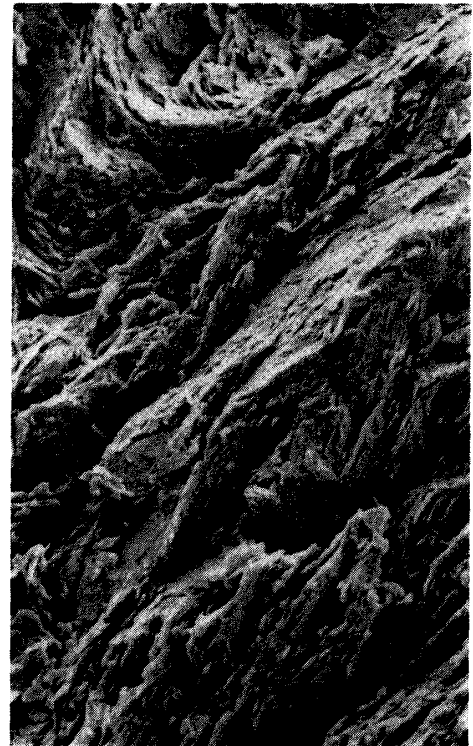


Photo. 11. 1,000



Photo. 12. $\times 1,000$

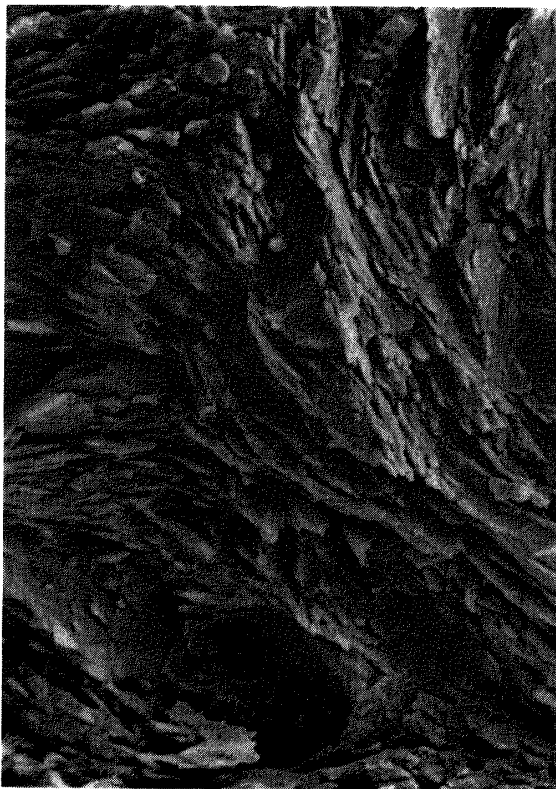


Photo. 13. $\times 3,000$

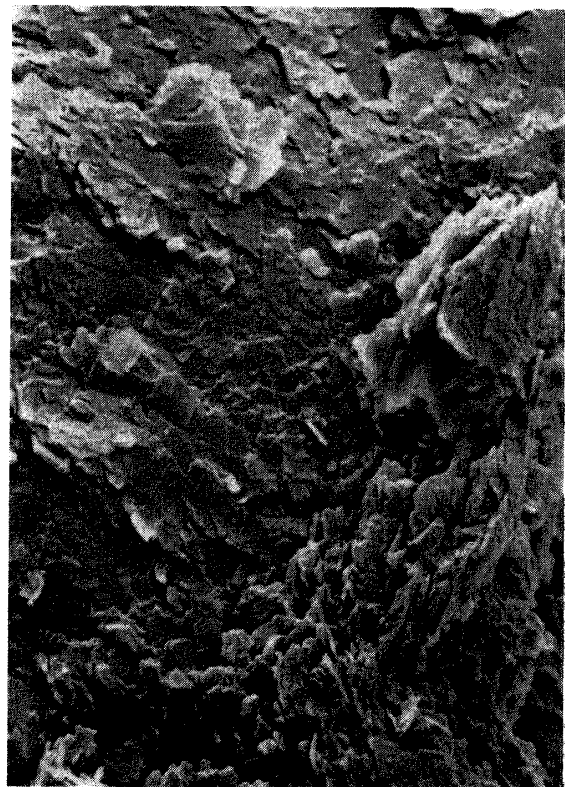


Photo. 14. $\times 3,000$

rammer) as shown in Photo. 1, while in the H-section (perpendicular to the V-section) the base planes of platelets are noted as shown in Photo. 3. When observed in detail, however, the following characteristics of micro-structure should be pointed out:

(1) Particles consisting of two or three platelets in face-to-face contact form a base unit of micro-structure. This base unit appears to be the same as what was referred to as "parallel packet" by Sloane and Kell (1966).

(2) The finger-like base units overlap one another and it form larger structures as shown in Photo. 4. The arrangements of these larger structures formed by packets are not uniform in a plane view as shown in Photos. 2 and 3.

(3) In the cross-section perpendicular to the sheet plane of crystals (00 l), the arrangement described above has continuous flow-like patterns typically shown in Photo. 1.

(4) It seems that this pattern forms in three dimensions a block structure, about 50 μ in diameter, consisting of concentric arrangements of particles. A cross-section of the blocks characteristically shows a flow pattern in two dimensions as shown in Fig. 6. For instance, three block structures at least may be recognized in Photo. 1. This block may be the same structure as the "ped" defined by Barden and Sides (1971) and others. Thus, in the case of a two-dimensional cross section, various micro-structures are observed depending on the relative position to blocks. It appears that the structures included in Photo. 1, are turbostratic (defined by Aylmore and Quirk, 1962) and curved trajectories (Sloane and Kell, 1966). The form of blocks may be deformed and elongated, to various degrees, by compaction with a dropping rammer.

(5) It seems that the amount of voids in the strongly curved (folded) parts of a flow-like arrangement is more than that in the form of more straight flow-lines, judging from the comparison of those two parts shown in Photo. 2. Therefore, it is concluded that void rich structures in the compacted kaoline clay on the dry side of the optimum are formed in the folded part, because the packets as basic units are obliquely in contact with one another, forming a structure similar to the "book-house" two-dimensionally.

(b) *Micro-structure at the optimum moisture content*

The flow-like or gently waved structures revealed in dry and wet samples are also observed in the optimum moisture sample as shown in Photo. 6. The strongly folded structure that is typically shown in wet and dry samples, however, is hardly observed in this sample. The block structures, instead of concentric or "micro-onion" structures observed in other moisture conditions, are formed of parallel oriented packets at the optimum moisture condition. These structures are seen two-dimensionally "turbostratic" in any sections as shown in Photos. 6 and 8. Photos. 8 and 9 show the view of H-sections and Photos. 6 and 7 the view of V-sections. The size of blocks are about

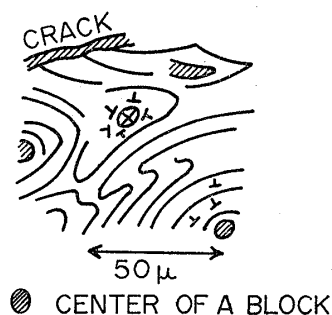


Fig. 6. Block structure on the dry side of the optimum

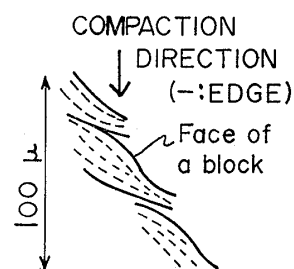


Fig. 7. Block structure at the optimum moisture content

20–30 μ in thickness and 80 μ in width. As shown in Photo. 8, voids within a block are very small, about 1–2 μ in diameter, while voids between the blocks are large.

Fig. 7 illustrates the inter-relation between the shape of blocks and individual particles. While flattened blocks interwrap or interwedge, the packets to form each block arrange themselves nearly parallel to the surface of a flattened block. Since the orientation of blocks is not always the same with the interfaces of blocks inclined and waved with respect to the compacted plane, the view in the H- and the V-sections may show similar appearance. It seems that the micro-structural properties mentioned above originate in "lubrication" (Lambe 1958) due to the development of double layers of water film between packets or individual plates which are in face-to-face contact at the optimum moisture condition. In other words the strongly folded micro-structure can not or hardly been formed at this condition due to the poor fluidity as compared with the conditions on the wet side of the optimum moisture (refer to (c)). The shearing strains induced by the fall of a rammer causes the sliding action between contacted faces of packets or platelets and rearrangement of particles, while in the case of the wet side of the optimum, the whole mass including blocks is deformed more easily due to rich fluidity.

(c) *Micro-structure on the wet side of the optimum moisture content*

Micro-structures observed in the condition of the wet side of the optimum moisture content are similar to those on the dry side of the optimum moisture condition. However, it is recognized that there is a difference in the degree of deformation of a block structure between two conditions, that is, blocks observed on the wet side of the optimum are flatter than those on the dry side. This phenomenon is well shown in the microphotograph of the section parallel to the direction of compaction (H-section). Photos. 10 and 11 show the arrangement of long-axes of elongated blocks. Fig. 8 illustrates the cross-section of the structure derived from Photos. 10 and 11.

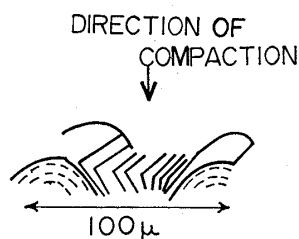


Fig. 8. Elongated block structure on the wet side of the optimum

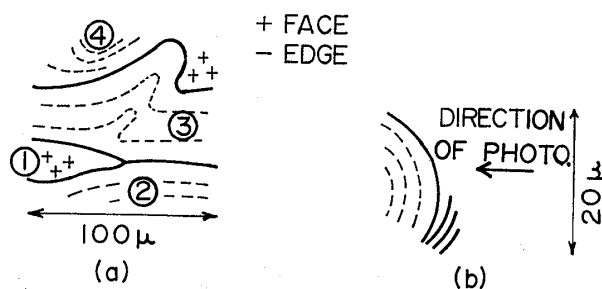


Fig. 9. Micro-structure normal to compaction direction on the wet side of the optimum

Photo. 12 is a view of the V-section and Fig. 9(a) illustrates the internal structure which can be derived from this photograph. From these results, the following micro-structural characteristics should be recognized; the flow-like structures ②, ③ and ④ shown in Fig. 9(a), the folded structure ③ shown at the central part in Photo. 12, and the structures formed by four elongated blocks ①, ②, ③ and ④, which show the varied view of the cross-section.

Photo. 14 shows a feature of the V-section and its large area is occupied by the basic planes of clay mineral. It is considered that the feature shown in Photo. 4, taken to be that of the H-section, is the side view of the elongated block or the folded structure

illustrated in Fig. 9(b).

As a result it is concluded as follows:

(1) Block structures are flatter than those on the dry side of the optimum, the parallelism between the flattened blocks is increased, and thus the flow-like arrangement in two dimensions is more marked.

(2) Generally, crystal planes (00 l) of each particle are arranged perpendicularly to the direction of compaction, but because of the formation of blocks and folded structures, the features in the V-section and H-section appear analogous.

(3) The tendency of distribution of voids is similar to that on the dry side of the optimum as described in (a). Dimensions of voids is approximately 20–30 μ in thickness and 50–100 μ in width as shown in Photos. 12 and 13.

Fabric Index Measurement by X-ray Diffraction

The specimen of parallel orientation was prepared by the procedure described by Meade (1961), and that of random orientation was prepared by adding acetone. X-ray diffraction patterns of these specimens are shown in Fig. 10. The (001) reflection and the (002) reflection of kaolinite are distinct and strong, but the (020) reflection is very weak. In order to determine quantitatively the degree of orientation of clay particles in compacted samples using (001) and (002) reflections, "fabric index" (FI) was defined in the form of

$$FI = \frac{H_v W_v}{H_h W_h + H_v W_v}$$

where H_h and H_v are the intensities of horizontal and vertical sections, respectively, and W_h and W_v are the width at half intensity of horizontal and vertical sections, re-

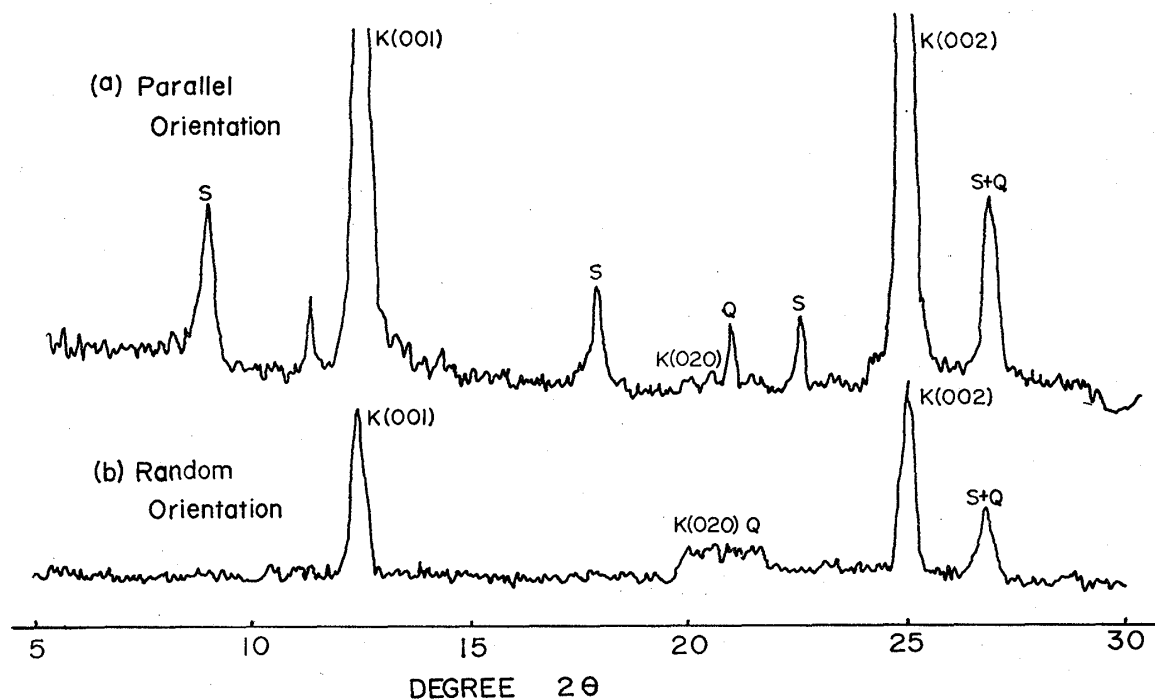


Fig. 10. Effect of particle orientation in X-ray diffraction patterns (scale factor 2)

K: kaolinite, S: sericite, Q: quartz

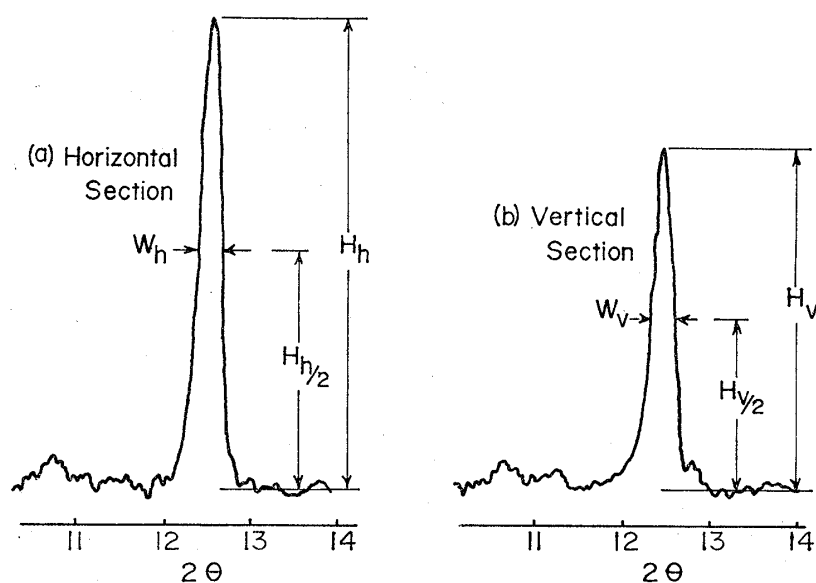


Fig. 11. Illustration of fabric measurement

spectively, as shown in Fig. 11.

As mentioned previously a method similar to Gillot's method (Gillot 1970) was used to simplify the calculation. The fabric index calculated by the above equation may range from 0 to 1.0. The fabric index of 0.5 indicates a random orientation, while the fabric index of 0 indicates a perfectly parallel orientation to the horizontal plane (normal to the compaction axis) and the fabric index of 1.0 a perfectly parallel orientation to the vertical plane (parallel to the compaction axis). It seems that the fabric index ranging from 0 to 0.5, as seen in a usual state of clay particles, may express parallelism to the horizontal and bedding plane. Table 2 shows the relation of fabric index to the degree of orientation of clay particles proposed by Odom (1967). The descriptive terms applied to various ranges of the fabric index are used as indicated for simplicity.

Table 2. Fabric index as related to the degree of orientation of clay particles

Fabric index	Degree of orientation
0.0	
0.1	Very Good
0.2	Good
0.3	Fair
0.4	Poor
0.5	Very Poor

X-ray diffraction patterns of a compacted kaolin, as an example, are shown in Fig. 12. Fabric indices of (001) and (002) reflections of kaolinite were calculated from these experimental results. The relationships between FI and molding moisture content are shown in Fig. 13. Though the fabric indices in each layer are scattered, the increase of molding moisture content results in a slight decrease in fabric index, and moreover its value of the first layer is smaller than that of the third layer. This result shows that clay particles are slightly more oriented to the horizontal plane as the moisture

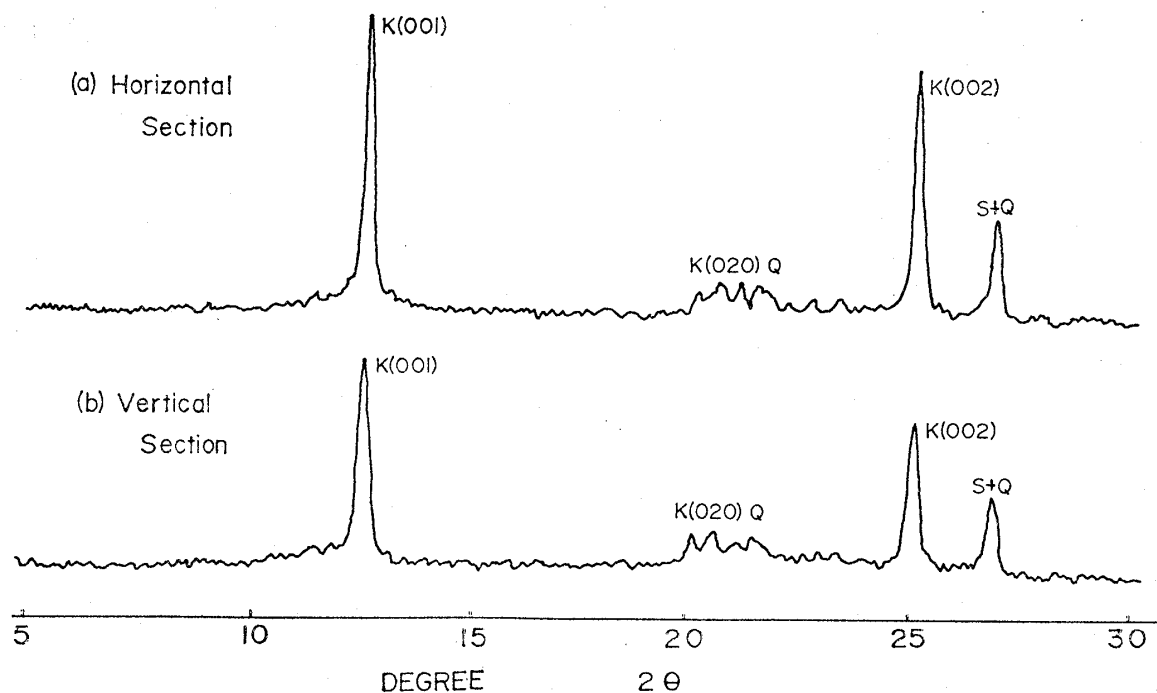


Fig. 12. X-ray diffraction pattern of compacted kaolin at moisture content of 30.5% (scale factor 4, the third layer)

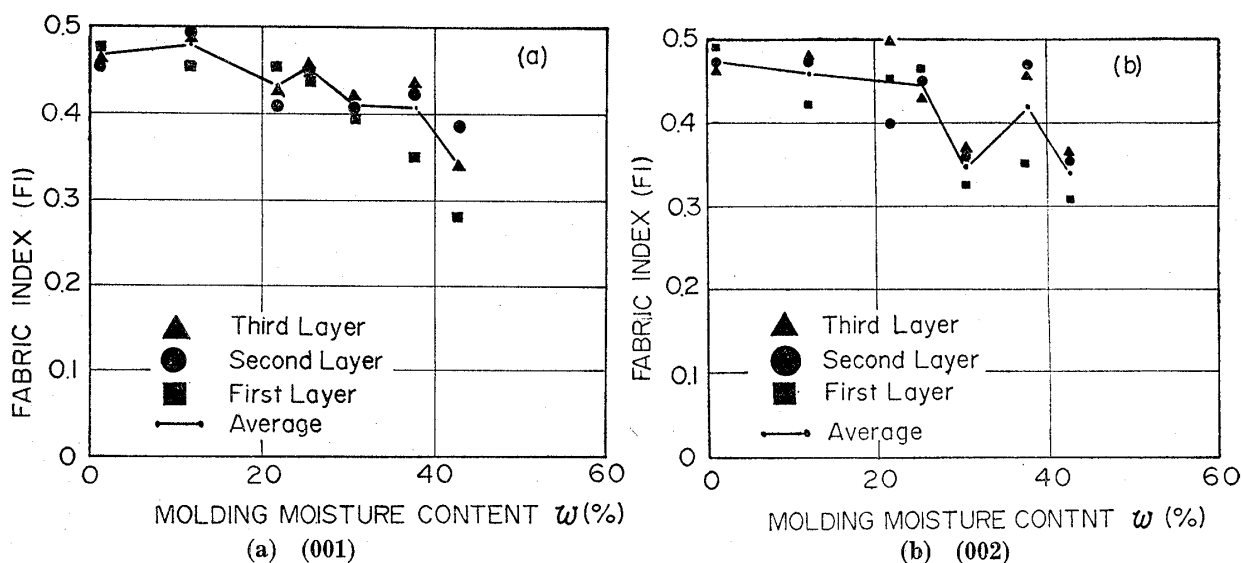


Fig. 13. Relationship between fabric index and molding moisture content for compacted kaolin

content increases, and this tendency is stronger in the first layer than the third layer on the wet side of the optimum. The fabric index shows the same tendency as $k_0 S^2$ concerning the orientations of clay particles in Fig. 5. This may suggest that the parallelism of a compacted soil by the Standard Proctor compaction is "poor" or "very poor," which is not different from Diamond's (1971) experimental results.

DISCUSSIONS

It can be said from our observation using a scanning electron microscope that the two-dimensional features upon micro-structure of compacted kaolin are very complicated

because compacted kaolin mainly consists of the structure with three-dimensional features of particles as described in the previous section. Under compaction at increasing molding moisture contents the flattening of blocks including "micro-onion" structures will be progressed, and the parallelism of particle arrangement will increase in the whole mass of soil. This is indicated by the experimental data of the micrograph (Photos. 10-12), the fabric index (FI) determined by X-ray measurements (in Fig. 13) and the tortuosity of flows represented by the values of k_0S^2 (Fig. 5). In other words, the slight difference in two factors, FI and k_0S^2 , is fundamentally due to the presence of three-dimensional micro-structures in each case, and the slight increase of FI depends on the increase in flatness of blocks which may be produced before compaction, as pointed out by Sloane and Kell (1966). According to these phenomena, it seems that Lambe's concept shown in Fig. 1 should be limited to the explanation for the properties of a soil mass, and should not be applied to that for the individual particle arrangement.

As seen evidently in Photo. 4, the arrangement on the dry side of the optimum is by no means random from the viewpoint of individual arrangement of particles. It is formed by flow-like parallel arrangement, though it is random as a mass. Therefore, it can be said that the dispersed structure consisting of parallel packets is a common structure in all moisture conditions. The block structure by combining dispersed structure and/or parallel packets depends on the molding moisture condition, that is, on the dry and wet sides of the optimum the curved and folded arrangement of particles are predominant in a block, while at the optimum moisture condition the preferred parallel arrangement is predominant without the presence of strongly folded structures.

These characteristics on the micro-structure may suggest the effect of compactive effort. The faces of a block which coincide with internal-particle arrangement at the optimum, incline to the direction of compaction, which is likely to be the relic of shearing strains accompanied by drops of a rammer, and actually incline in various directions in the soil mass. The parallelism in the whole mass of compacted soil, therefore, cannot be of strong tendency.

The differences in micro-structures from the dry to the wet of the optimum will be based on the physico-chemical interaction between particles and water, as pointed out already by many investigators, but the preferred parallelism at the optimum moisture described above should be noted specially.

The voids in the micro-structure are classified into micro-voids in dispersed structures which are in the size of a clay particle being about 0.2μ in thickness and $1-2\mu$ in length, and voids in folding or strongly curved structures which are in the size of a packet. It seems that the distribution of these types of voids determines the dry density of a compacted clay. Thus the relation between the dry density and the micro-structure, or the molding moisture content, could be understood.

CONCLUSIONS

From the results of the present study, it may be concluded that:

- 1) The micro-structures of compacted kaolin clay mainly consist of three-dimensional block structures, which have a strong tendency of parallel arrangement of particles even if the arrangement in macro-scale is random or isotropic.
- 2) Slight increase in the parallelism caused by compactive effort, shown by the intensity of X-ray diffraction and k_0S^2 from the Kozeny-Carman equation, is due to the existence of block structures in all cases of moisture conditions.
- 3) The change of parallelism is caused not by each particle arrangement but by

flattening and/or deforming of block structures in clay mass.

ACKNOWLEDGEMENTS

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NOTATION

- FI =fabric index by X-ray analysis
 H_v, H_h =intensity of X-ray diffraction from vertical and horizontal section
 $k_0 S^2$ =tortuosity index from Kozeny-Carman equation
 (lmn) =Miller's indices for representation of crystal planes
 W_v, W_h =width at half intensity of vertical and horizontal section
 σ_e =normal stress

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APPENDIX

List of scanning electron micrographs

No. of Photo.	Magnification	Section photographed	Molding moisture content (%)
Photo. 1	1,000	parallel to rammering direction	4.27
Photo. 2	3,000	parallel to	4.27
Photo. 3	3,000	normal to	4.27
Photo. 4	10,000	parallel to	4.27
Photo. 5	3,000	parallel to	25.9
Photo. 6	1,000	parallel to	25.9
Photo. 7	3,000	parallel to	25.9
Photo. 8	1,000	normal to	25.9
Photo. 9	3,000	normal to	25.9
Photo. 10	1,000	normal to	45.0
Photo. 11	1,000	normal to	45.0
Photo. 12	1,000	parallel to	45.0
Photo. 13	3,000	parallel to	45.0
Photo. 14	3,000	parallel to	45.0

} Optimum