Clays and Clay Minerals, 1971, Vol. 19, pp. 31–47. Pergamon Press. Printed in Great Britain

OPTICAL AND ELECTRON MICROSCOPIC INVESTIGATION OF SHEAR INDUCED STRUCTURES IN LIGHTLY CONSOLIDATED (SOFT) AND HEAVILY CONSOLIDATED (HARD) KAOLINITE

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(*Received* 17 *March* 1970)

Abstract—A review of fabric studies of clays suggests the need for relating those fabric characteristics which are revealed at the two levels of magnification provided by optical and electron microscopy, and a technique to achieve this has been developed and is described within the context of the initial stages of a long term study of the interrelation between fabric and engineering behaviour. Two kaolinitic clays with contrived fabrics were prepared by controlling particle size, moisture content and pH of suspension, and consolidation load and were subjected to shear loading to failure. Resin impregnation techniques which permit the kaolinite to be cut into thin sections for transmission electron microscopy have been optimized with the object of minimizing fabric strain and damage during ultratomy.

The fabrics of the hard and soft ambient material are qualitatively compared by means of electron micrographs and are explained in terms of the preparatory procedures adopted for fabric control. The fabrics of the two types of shear induced structures are also qualitatively compared and explained in terms of the original fabrics and the subsequent shear loading.

INTRODUCTION

THE INFLUENCE of soil micro-structure on the response of soil to an applied stress regime has been the subject of investigation by a number of workers of whom T. W. Lambe was one of the first (Lambe, 1953). The topic has been reviewed by Morgenstern (1969). Micro-structure is to be distinguished from macro-structure which describes discontinuities, including fissure and crack patterns within the soil mass, which are visible to the naked eye. Microstructure, which cannot be discerned with the naked eye, is a characteristic of assemblies of soil particles which range in size down to smaller than 1 μ, and has two elements; geometrical arrangement which is referred to as fabric, and interparticle forces comprising firstly quasi-reversible physico-chemical forces between particles including attractive and repulsive forces and secondly quasi-irreversible cementation. The terminology used in describing fabric is given by Morgenstern and Tchalenko (1967b). Fabric may be observed by microscopy at two levels of magnification: optical microscopy permits observation of the overall pattern of shear induced structures including discontinuities and intervening domains, and also the average preferred orientation of particles within such structures; electron microscopy permits observation of individual particles and particle assemblies within shear induced structures and ambient material. Interparticle attractive forces may be weakened or strengthened in the course of time by leaching (Moum *et al.*, 1968; Bjerrum *et al.*, 1969; Bjerrum, 1969) or enhanced by deposition of cementing agents which produce quasi-irreversible rigid and semi-rigid bonding at points of contact (Kenney *et al.*, 1967).

The project to be described is concerned principally with the fabric of an artificial kaolin soil and the following appraisal of earlier work therefore refers primarily to fabric studies. Reviews of the fabric of clay soils have been given by Sloane and Kell (1966) and Meade (1964). Clay fabric has been quantified, particularly in terms of the degree of preferred orientation of the assembled plate-like particles, and also in respect of variation of this orientation within a soil sample and its variation consequent upon loading. Martin (1965) has successfully used X-ray diffraction techniques to investigate fabric orientation and variation of orientation of single solid phase kaolin.

An assembly of kaolin particles exhibits "form birefringence" (Grim, 1953) when observed under polarized light and this characteristic has been made use of for the determination of preferred fabric orientation of aggregated particles (Mitchell,

1956; Morgenstern and Tchalenko, 1967a and 1967b). The value of this technique lies in establishing the overall pattern of shear induced fabric and the preferred orientation of particles within the domains and discontinuities, but it does not permit detailed study of particle assemblies. Morgenstern and Tchalenko (1967b) remarked that a shear discontinuity which appeared as a distinctive feature at a particular level of magnification may itself be reducible to a system of shear discontinuities at a higher level of magnification, thus the pattern of shear structures observed is a function of the method of microscopic observation. Rosenqvist developed an ingenious carbon replica technique for obtaining electron micrographs which illustrate the fabric of sensitive marine clay (Rosenqvist, 1959). Pusch (1967) successfully applied transmission electron-microscopy for investigation of scandinavian marine quick clay fabric. Both transmission (Smart, 1967) and scanning (Tovey, 1970) electron microscopy have been used for investigation of clay fabric and in particular kaolin fabric.

The resolving power of the transmission electron microscope permits observation of individual particles of kaolin within a maximum field of view of 40 μ dia. at a magnification of 2000 and can thus be used for detailed examination of fabric. However, it will be appreciated that to obtain a thin section of soil suitable for electron-microscopy precisely within or to include a shear induced structure of 10–300 μ, which were the dimensions of such structures observed by Morgenstern and Tchalenko (1967b), would be a matter of some considerable chance without a suitable technique for selection. Conversely unless the observer is previously aware of the overall pattern of the shear induced structures from which the thin section being observed has been taken, he is unlikely to appreciate fully the significance of the geometrical arrangement of particles revealed to him in his field of view. A technique which facilitates these requirements will be described.

The fabric of soils is dependant on a large number of intrinsic and environmental factors, many of which have been discussed by Lambe (1958a) relative to compacted soils and more recently by De (1970). One important factor is the mineralogy of the constituent particles (Roscoe, 1967). The concentration of suspended particles at the time of deposition is another factor which affects the extent of flocculation in the suspension and therefore the microstructure of the resulting sediment (Rosenqvist, 1955). A third factor is the nature of the electrolyte, which has been investigated by Rosenqvist (1962) and Lambe (1958a) who has demonstrated the influence of electrolyte concentration, ion valence, dielectric constant, temperature, size of hydrated ion, and pH on the void ratio of a sediment. Rosenqvist (1955) comments on the difference in distribution and orientation of the finest flaky (clay) minerals which while exhibiting horizontal parallelism in fresh water clays are randomly distributed in marine clays. He furthermore demonstrates that flocculation depends on the electrolytic concentration and pH, and that the pressure to which the sediment is subjected has an effect on the degree of parallelism perpendicular to the direction of consolidating pressure; Lambe (1958b) also describes this phenomena. The fabric of Leda clay, which is attributed with having a "card-house" type of structure, and its response to consolidation loads, has been successfully measured using X-ray diffraction techniques by Quigley and Thompson (1966) who speculate that for Leda Clay, the void ratio produced under a given load in an oedometer consolidation test is a function of the degree of induced particle parallelism. Morgenstern and Tchalenko (1967b) conclude that sedimented flocculated kaolin develops intense orientation under low effective stresses. The variability of permeability has been related to fabric differences in the horizontal and vertical directions and as a consequence of one dimensional consolidation (Mitchell, 1956). Massachusetts Institute of Technology research in Earth Physics has been concerned with the development of a fundamental understanding of the behaviour of particle systems, especially cohesive soils, under varying conditions of stress and environment (Martin, 1965), and includes specific studies of "true cohesion" and fabric of kaolinite. Orientation in kaolin due to shear has been observed by Martin (1966). Those factors on which fabric is dependant have been controlled in the work to be described.

OBJECTIVE

Long term. When a homogenous intact soil, i.e. having no macro-structure, is subjected to stress its resistance to deformation or its engineering behaviour is ultimately derived from the manner in which its constituent particles react one with the other as they are constrained to move relative to one another (Bjerrum, 1967; Skempton, 1960; Rosenqvist, 1962). The response of an intact soil body to external loading is thus in part dependant on its micro-structure, and it is the determination of the nature of this dependance which is the long term objective of this work. The micro-structural response or behaviour is a function of fabric, physico chemical bonding, cementing, the nature of the pore fluid, and the degree of saturation of the pore spaces. The strength derived from fabric is a

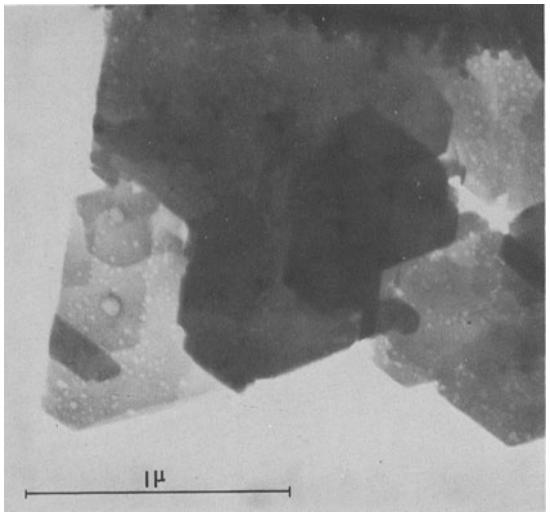

Fig. 1. Electron-micrograph of dispersed fractionated kaolin.

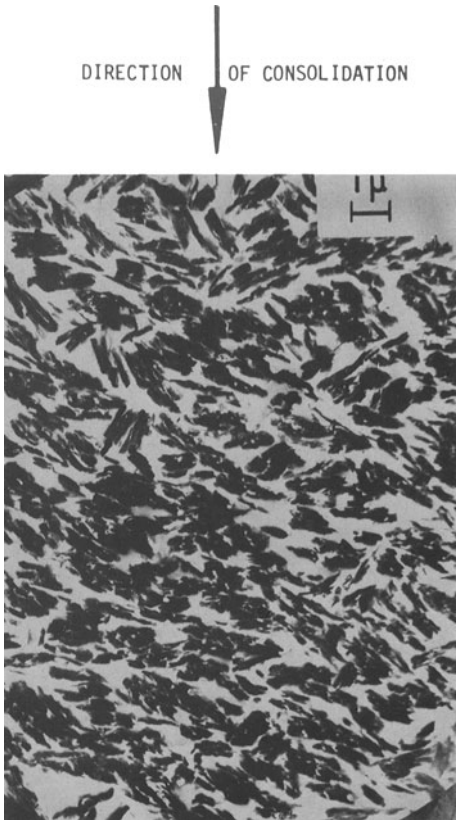

Fig. 3. Electron-micrograph of thin section of 900 Å through kaolinite impregnated with Araldite AZ15, consolidated from 150 per cent moisture content, pH8, under 4·75 Kg/cm². (Soft material).

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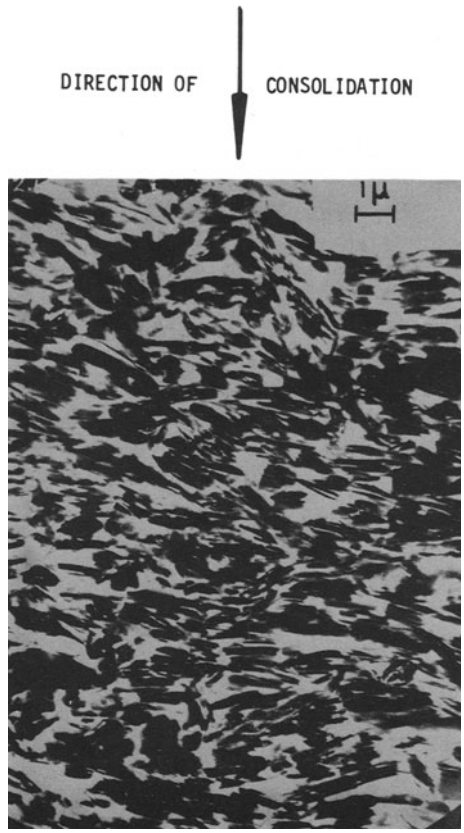

Fig. 4. Electron-micrograph of thin section of 900 Å through kaolinite impregnated with Araldite AZ15, consolidated from 500 per cent moisture content, pH8, under 45 Kg/cm². (Hard material).

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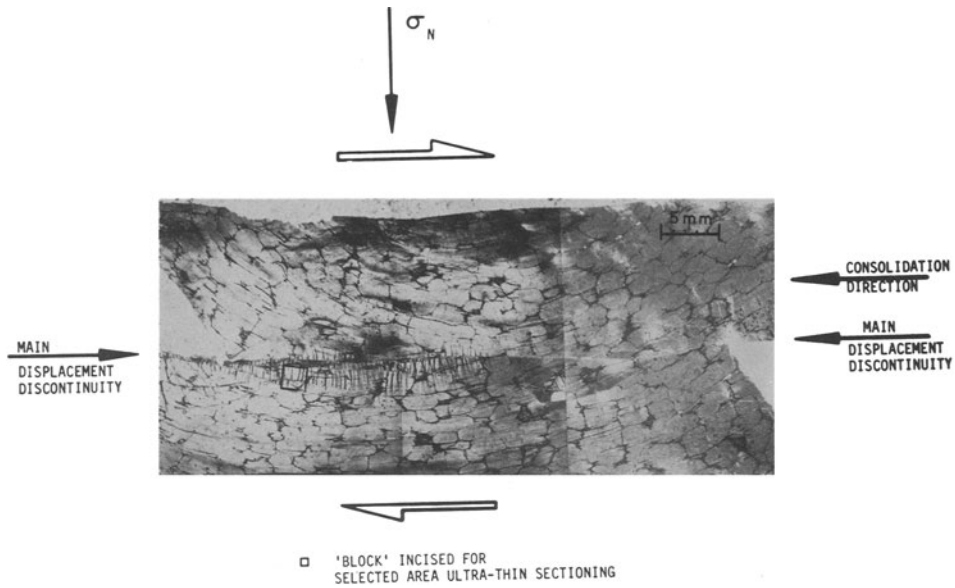

Fig. 11. Optical-micrograph of soft ("vertical") material (Fig. 3) after shearing, showing shear induced structure pattern after peak stress, including main displacement discontinuity and kink-bands.

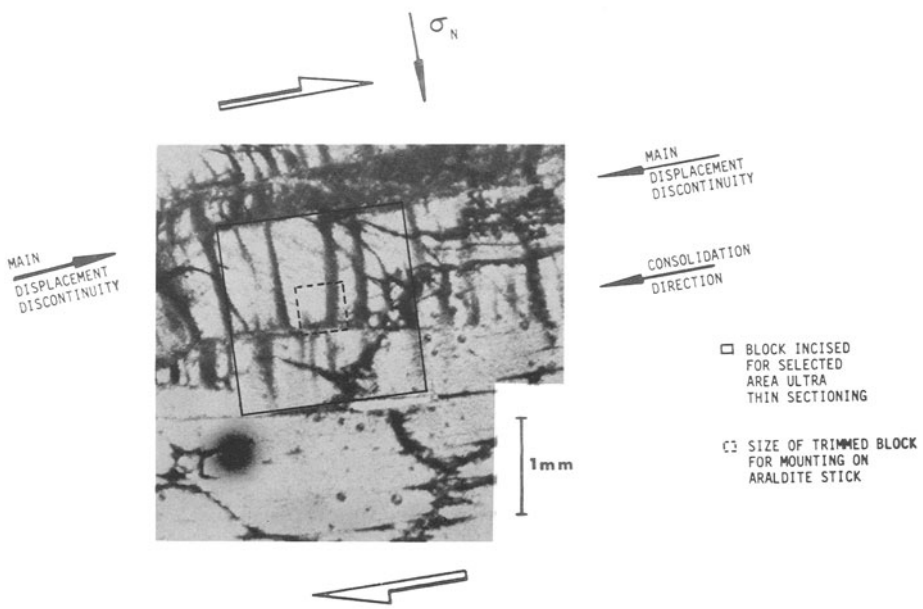

Fig. 12. Optical-micrograph of soft ("vertical") material (Fig. 11), showing appearance of field of view for selection of material for thin section.

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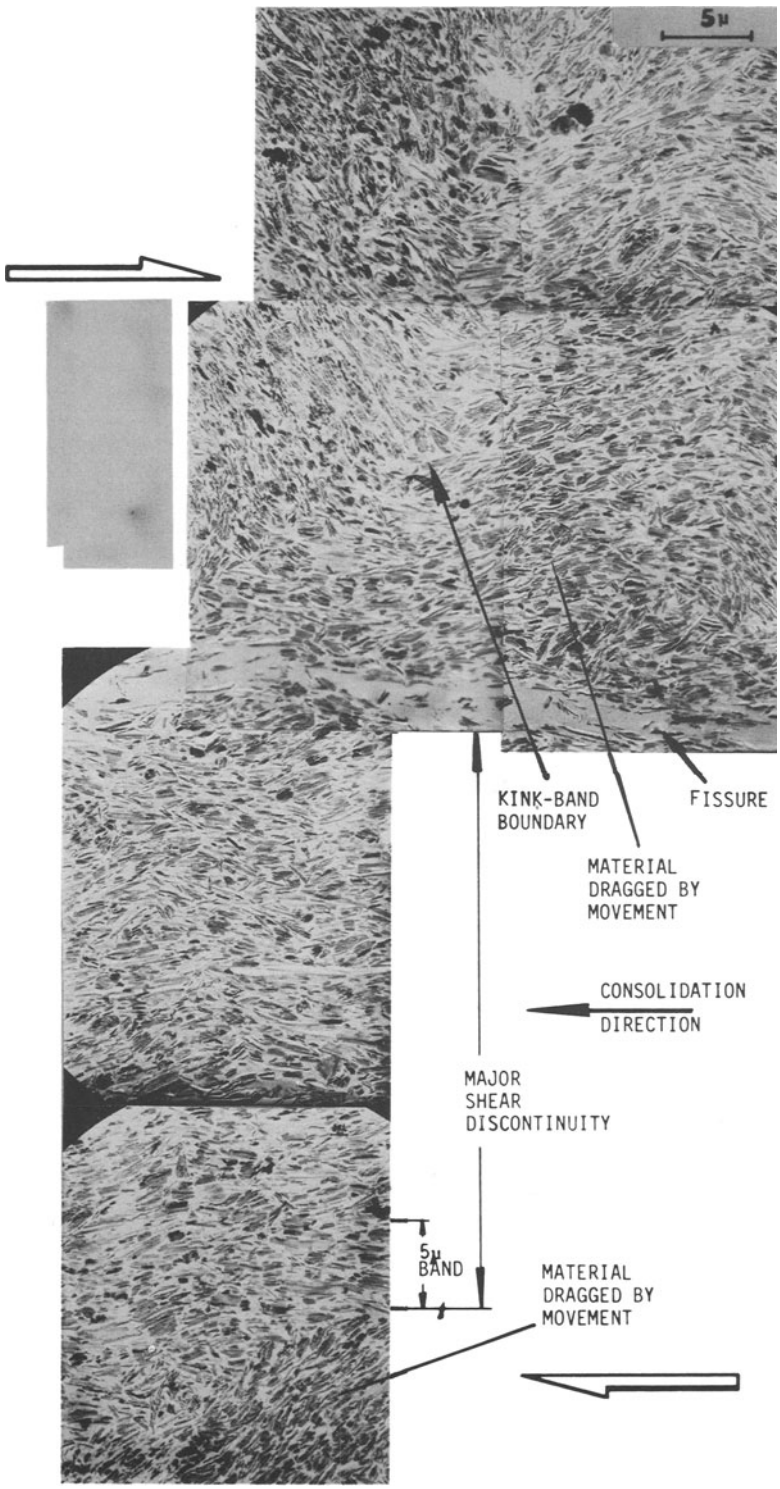

Fig. 13. Electron-micrograph showing micro-structure of a major shear discontinuity and associated kink bands (Fig. 12) in soft ("vertical") material after peak stress. Thin section of Araldite AY 18 impregnated material 700 Å thick.

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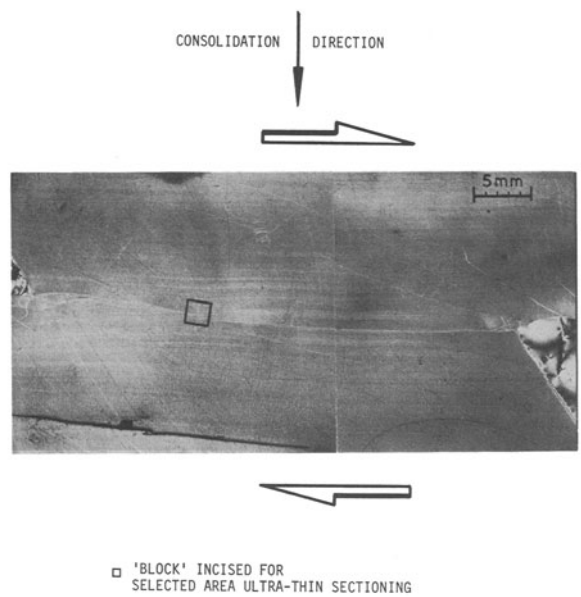

Fig. 15. Optical-micrograph of hard ("horizontal") material (Fig. 4) after shearing showing location of material selected for thin sectioning.

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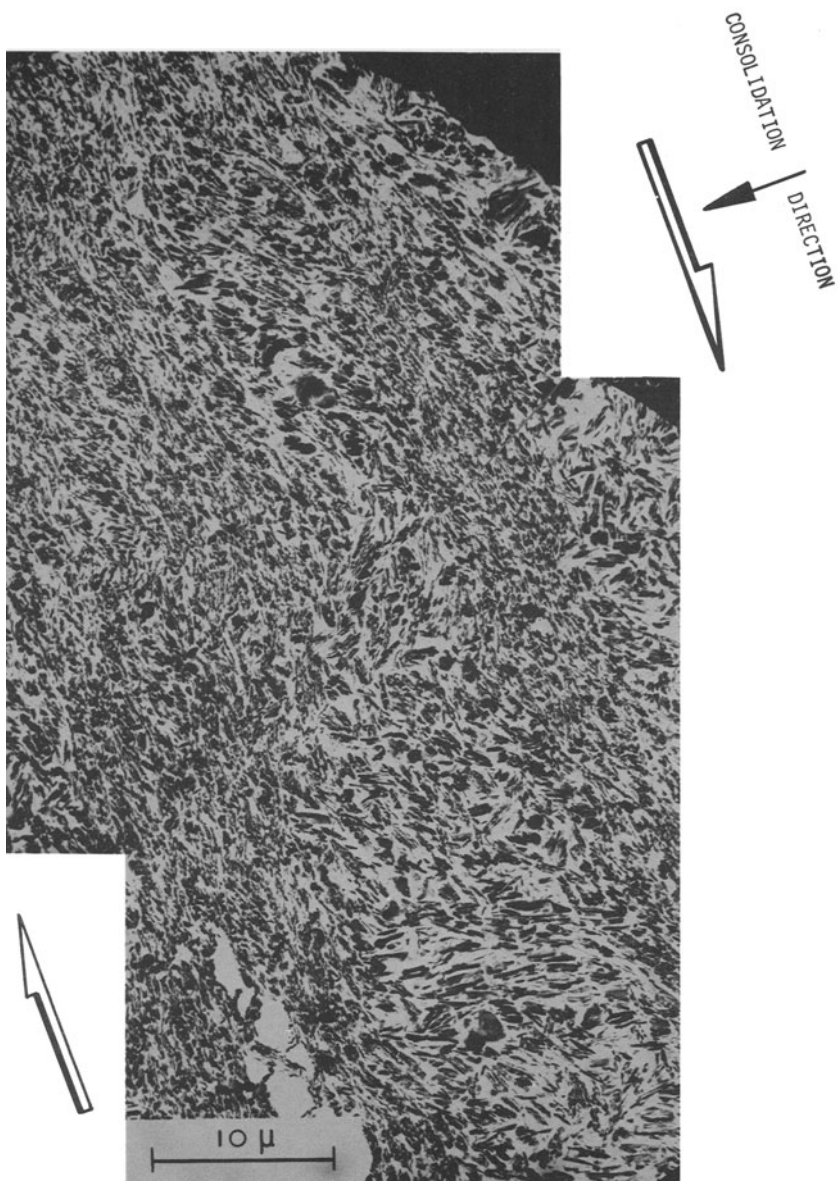

Fig. 16. Electron-micrograph showing micro-structure of the junction of two main shear discontinuities in hard ("horizontal") material (Fig. 15) after peak stress. Thin section of Araldite AY 18 impregnated material 700 Å thick.

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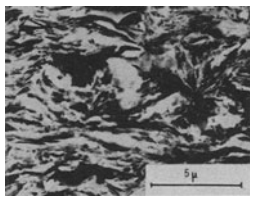

Fig. 18. Electron Micrograph of vertical, 900Å thin section through Oxford Clay from depth of 9 ft 6 in. impregnated with Araldite AZ15.

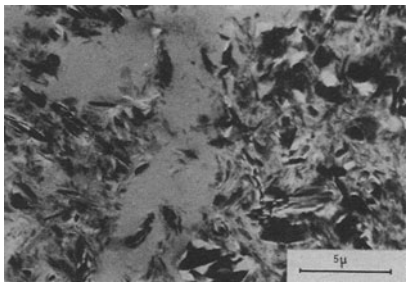

Fig. 19. Electron Micrograph of vertical .800Å thin section through Norwegian Marine Clay from depth 6 m in undisturbed deposit. Impregnated with Araldite AY18.

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function of density of packing, mode of packing, grading, shape and degree of preferred orientation of particles. Physico-chemical bonding is a function of the mineralogy of particles, their proximity to one another, the type of adsorbed cations, and the nature and concentration of the electrolyte in the pores. Cementing occurs at points of contact and is a function of long term weathering processes.

It should of course be recognized that the response of a soil body to loading is also dependant on the mode of testing (Ward, 1967; Bishop, 1966).

The present project has been of an exploratory nature prior to a more comprehensive investigation of the relationship between soil micro-structure and engineering behaviour. Its primary limited objective was to develop satisfactory techniques for preparing soil for microscopy and for observing by means of transmission electron microscopy fabric existing before and produced by stressing; however, the long term purpose of relating fabric characteristics and fabric changes to engineering behaviour has been promoted by using two materials which were prepared by using different consolidation loadings and which were subjected to shear stressing in a modified 6 cm × 6 cm shear box.

A secondary objective was to produce fabrics which would be susceptible to quantitative appraisal. The result would not necessarily provide models of natural soil, but by restricting the number of factors influencing the contrived fabrics the resulting particulate assemblies should be more amenable to micro-structural investigation than natural soils. To achieve this objective those factors influencing fabric have been controlled. The mineral chosen was "Supreme" Kaolin the particles of which have a characteristic plate-like, irregular hexagonal shape (Fig. 1), its properties are listed in Table 1. With a view to eliminating large particles which might produce macro-structural affects, and infinitesimally small particles which might influence interparticle bonding in an indeterminate manner, the kaolin was fractionated by sedimentation to produce a material having a restricted particle size range (Fig. 2). Thus mineralogy, shape and grading were predetermined within

Table 1. "Supreme" kaolin properties

Chemical analysis element	Per cent
SiO_2	46·6
Al_2O_3	38·3
Fe_2O_3	0·49
TiO_2	0·05
CuO	0·2
MgO	0·2
K_2O	0·68
Na_2O	0·07
Classification after fractionation	
Liquid limit	69·3
Plastic limit	53·6
Plasticity index	15·7
Activity	0·23
Uniformity coefficient	1·9
Particle form factors	
Length/width (Fig. 1)	1·1
Length/thickness	12

"Supreme" Kaolin is supplied as a paper clay by English Clays Lovering Pochin, St. Austell.

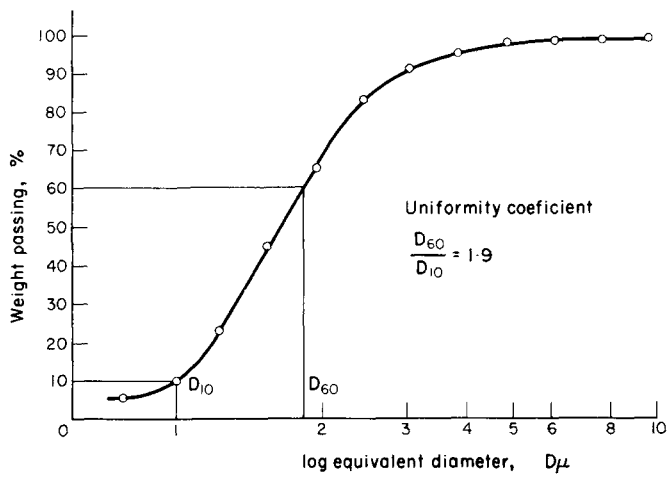

Fig. 2. Particle size distribution of fractionated kaolin.

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narrow limits, further control was attained by preparing the two consolidated materials from dilute suspensions with high pH to induce dispersed fabrics.

A further objective was to relate the broad pattern of shear discontinuities observable under the optical microscope (Figs. 11 and 12) with the much more intricate micro-structural pattern (Fig. 13), which is contained within the broader pattern. The problem of isolating one feature of the broad pattern for microstructural observation and conversely relating an observed microstructure to its parent macro-structure has been overcome by the junior author by the method of selected area ultra thin sectioning (De, 1970).

EXPERIMENTAL PROCEDURE

Fabric control

Before fractionation a 50 per cent concentration of kaolin in distilled water was allowed to age for 48 hr, prior to dispersion in distilled water to give a 6 per cent suspension. Sodium hexametaphosphate (0·05 per cent by weight of the dry clay) was used to deflocculate together with sufficient 0·1N sodium hydroxide solution to adjust the pH to a value between 7 and 8. High speed (2000 rev/min) stirring in a 60 liter polythene bottle for 30 mins ensured an adequate degree of dispersion, immediately after which the suspension was sedimented in a 2 ft × 2 ft × 10 in. covered tank; it was intended to produce a uniform material having a 1–2 μ particle size range. The depth of suspension was varied between 6 in. and 8 in. to conform with a convenient and appropriate settling time for the critical size of either 1 μ or 2 μ. Settling velocities were assumed to be constant and from Stoke's law were found to be: 1 μ Stokes diameter, 1 ft in 93 hr; 2 μ Stoke's dia., 1 ft in 23·4 hr. For each dispersed batch the initial sedimentation time adopted was sufficient to allow all 1 μ particles to settle out, and the remaining supernatant suspension was discarded. The residue was subjected to 2 further successive similar sedimentations so that the final resultant sediment was substantially free of sizes smaller than 1 μ. This residue was subjected to a similar sedimentation sequence for which the sedimentation time adopted was sufficient to allow all particles larger than 2 μ to settle out. The successive supernatant suspensions which contained the required range of sizes were accumulated and the residue in each case was used for resedimentation so that ultimately it contained substantially those sizes larger than 2 μ, and was discarded. While the fraction obtained (Fig. 2) had an acceptable uniformity coefficient, the bulk (80 per cent by weight) of the material had a particle size range of 1–3 μ. The presence of the unwanted coarse particles is attributed to disturbance of the sediment during siphoning off the supernatant suspension in the second sequence.

The accumulated material was allowed to mature for one month after which the suspensions used for consolidation were prepared. The lightly consolidated (referred to as "soft") material (Fig. 3) was prepared from a 150 per cent moisture content suspension with pH adjusted to 8 by using sodium hydroxide; the consolidometer was 4 in. in diameter, the consolidation pressure 4·75 Kg/cm^2, and the thickness of material produced 4 in. The heavily consolidated (referred to as "hard") material (Fig. 4) was prepared from a 500 per cent moisture content suspension with pH also adjusted to 8; the consolidometer was 6 in. in dia., the consolidation pressure 45 Kg/cm^2, and the thickness of material produced 4 in. In both cases the material was subjected to an unloading and reloading cycle before being finally unloaded prior to removal from the consolidometer, it being considered that the preferred orientation would thereby be enhanced, although no observations have been made to confirm this.

Sample preparation

The cylindrical "cakes" of consolidated material were carefully dissected (Fig. 5) and trimmed to produce 60 mm × 60 mm × 25 mm direct shear box samples, some of which were cut with the direction of consolidation loading perpendicular to the direction of shear loading (referred to as "horizontal") and others with the direction of consolidation loading parallel to the direction of shear loading (referred to as "vertical"). The terms "vertical" and "horizontal" thus refer to the direction of anticipated preferred orientation in the ambient material in the shear box.

From the remaining consolidated material 25 mm × 12 mm × 2–3 mm slices for ultratomy were cut (Fig. 5) in which the direction of consolidation loading was "perpendicular" to the 25 mm side and parallel to the 12 mm side. The reason for adopting these dimensions was that such slices could be cut easily from the end of a 25 mm × 25 mm × 12 mm block of material without risk of the slice distorting and thus producing fabric strain; a fine wire was used to cut the "soft" material, and a razor blade for the "hard". The thickness of 2–3 mm was principally governed by the requirements of impregnation; 2 mm was the minimum which could be tolerated if mechanical damage to the fabric in the middle of the slice during slicing was to be avoided. Observations were made on a transverse section through an impregnated slice using optical microscopy and

the disturbed surface zone extended for an average depth of 0·5 mm. 3 mm was the maximum permissable if full penetration of the impregnating resin was to be achieved with absolute certainty.

After removal from the consolidometer any material not under test in the shear box or being impregnated was wrapped in double polythene sheets or bags and stored over water in a dessicator at room temperature; after 3 months the decrease in moisture content of a control sample of soft material was 1·5 per cent to 56·5 per cent. No doubt this can be improved upon by use of further wrapping and a constant temperature control, but it is regarded as acceptable for the immediate purpose.

Shear testing

An attempt was made to simplify the complicated pattern of shear induced structures associated with the direct shear box (Tchalenko, 1967; De, 1970) which induces a succession of displacement discontinuities (Fig. 6). It had been observed that the edge discontinuities which propagate at low strains were on average inclined at 125° from the

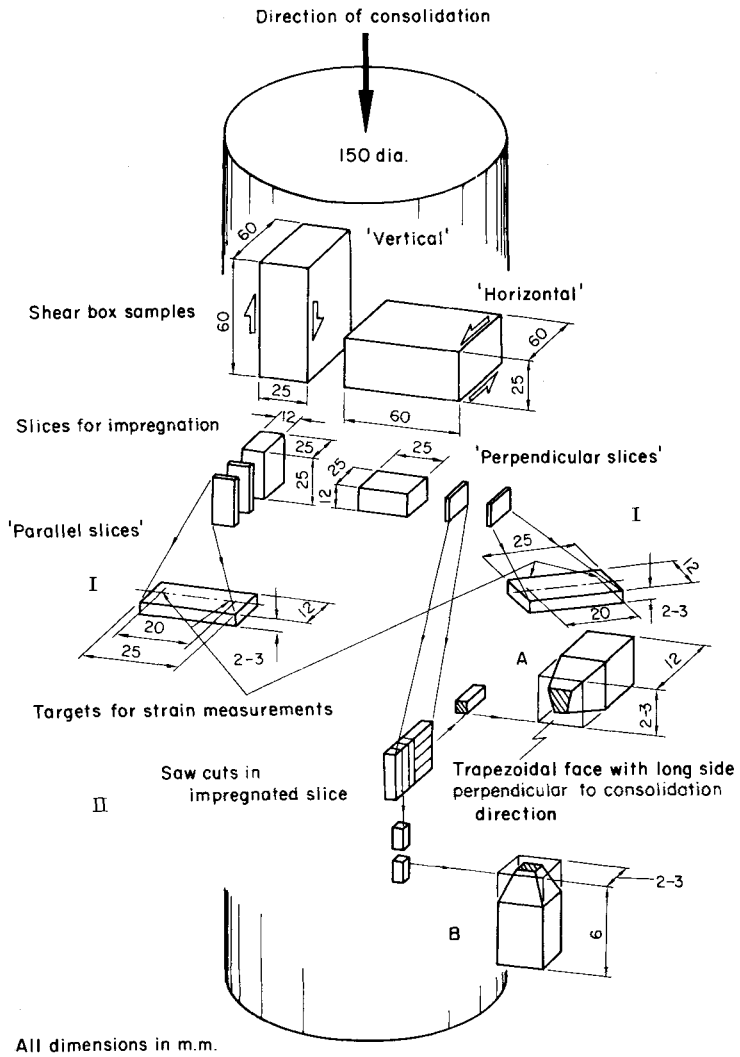

Fig. 5. Method of cutting consolidated material for direct shear box testing, and for slices for impregnation. I. Slices for strain measurement; II. Cutting and trimming impregnated sticks. A. Stick for thin sectioning in planes parallel to direction of consolidation; B. Stick for thin sectioning in planes perpendicular to direction of consolidation.

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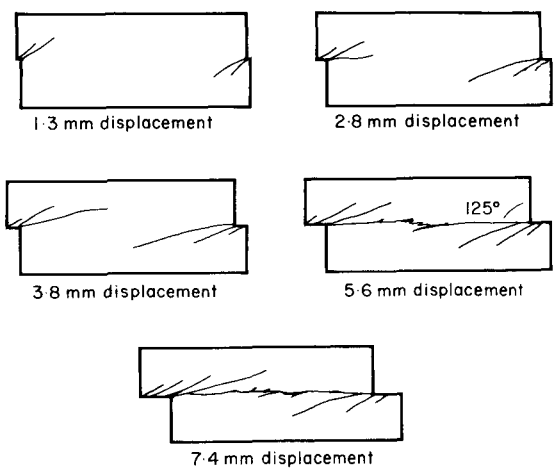

Fig. 6. Sequence of shear induced displacement discontinuities in "vertical" specimens sheared in 6 cm × 6 cm conventional shear box normal to direction of preferred orientation of original fabric (Tchalenko, 1967).

moving vertical face and for this reason these moving faces were set an an angle of 125° from the horizontal; it being anticipated that. by rotating the advancing face of the box through 35° in this manner, the edge discontinuities would be rotated through a similar amount and thus would become parallel to the direction of shear. The modification (Fig. 7) was achieved by introducing brass wedges into the ends of the standard 60 mm × 60 mm × 25 mm direct shear box. Samples for use in the modified shear box were trimmed by means of a template to accept the brass wedges before introduction into the apparatus. In the case of the two tests referred to in this paper the samples were "vertical" in the case of the "soft" material and "horizontal" in the case of the "hard" material. Shear loading was carried out under normal effective stresses (σ_N) of 0·55 kg/cm for the "soft" material and 2·4 kg/cm^2 for the "hard" material which thus had an overconsolidation ratio of 18·75. The K_0 ratios were not found and the overconsolidation ratio for the soft material is therefore indeterminate. The rate of shear deformation was 6×10^{-5} mm/sec, so that stressing occurred virtually under drained conditions in both cases.

On completion of a shear test the sample was sliced in the longitudinal vertical plane to yield 2–3 mm thick slices which thus contained the shear induced structures in a two dimensional array. These slices were to be impregnated prior to production of thin sections for microscopy, and since they were 60 mm × 25 mm × 2–3 mm it was impossible to cut them with a fine wire, so that a process of careful dissection with repetitive cuts of a razor blade was adopted and proved satisfactory, while however involving an excessive wastage due to rejection of damaged slices. It may be remarked that the difficulty of taking large slices in heavily overconsolidated material with low moisture content has been subsequently overcome by immersing the block of material to be sliced in molten carbowax 6000 for 24 hr. This procedure is not however efficacious with material having high moisture content and further work is required to establish its reliability; ideally it results in an advantageous partial impregnation which hardens the material sufficiently for it to be sliced easily and without damage, using a scalpel. The carbowax in the slice is removed by the subsequent impregnation sequence which precedes resin impregnation.

Electron microscopy

For the purpose of investigating soil fabric by transmission electron microscopy it is necessary either to produce a replica of a fractured surface or to impregnate the voids with a material which will permit ultra thin sections to be taken (Kay, 1965), both techniques involve tedious processes. Replication permits stereoscopic appraisal of fabric (Rosenqvist, 1962) but reveals nothing of the size and distribution of pores, on the other hand ultra

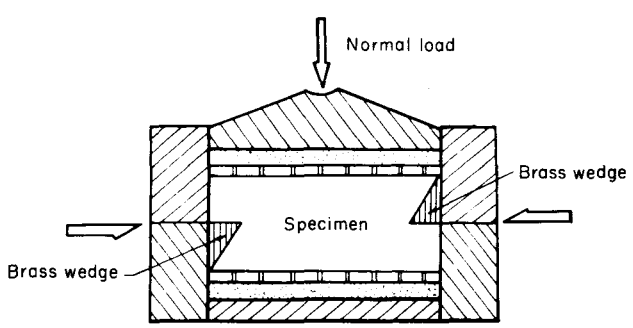

Fig. 7. Modification to ends of 6 cm × 6 cm direct shear box.

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thin sectioning does reveal the pattern of pore spaces and for this reason is preferred.

Carbowax 6000 (Polyethylene Glycol) is commonly used for impregnation (Mitchell, 1956) of material required for optical microscopy. Quigley and Thompson (1966) have also used it for X-ray diffraction studies and have investigated the strain consequent upon impregnation of marine clay. Carbowax however, is not sufficiently strong for ultratomy which is necessary for obtaining the ultra-thin sections required for electron microscopy, and resins including Araldite (epoxy), Vestopal (polyester) (Smart 1967, De 1970) and Bakelite (epoxide) (De 1970) have been used for this purpose.

The essential requirements of impregnation for ultratomy are that the pore spaces should be saturated with the resin without distortion of the fabric, and that when cured the resin should form an extremely strong but flexible matrix which can be cut with a diamond knife to thicknesses of not more than 1100Å without loss of or damage to particles. The resulting thin sections should be clear, free from holes due to lost particles and tears, should not have crinkled during cutting and be capable of withstanding electron bombardment. Observations for optimisation of impregnating procedures have been made.

Impregnation procedures

Prior to the main experimental work with the direct shear box an investigation of impregnating techniques was conducted in which four resins, Araldites AY18 and AZ15, Vestopal W, and Bakelite R18774/1, were used (De, 1970). The apparatus and experimental procedures were similar to those described by Smart (1967), with modifications (De 1970); the successive solutions in which the slice was immersed followed the sequence; water–methanol, methanol, methanol–acetone, acetone, resin or acetone–resin depending on the type of resin, and resin–hardner. Four differing initial proportions of water–methanol were used, also the subsequent proportions of water to methanol and methanol to acetone were reduced at different rates: the programmes for these sequences are given in Table 2. The material used for this preliminary investigation had been consolidated from a moisture conent of 150 per cent under a pressure of 4·75 kg/cm^2 and is assumed to have a similar micro-structure to that of the "soft" material (Fig. 3).

From the consolidated material two types of 25 mm × 12 mm × 2–3 mm slices were cut, in which the direction of consolidation loading was either "parallel" to or "perpendicular" to the 25 mm side and in the latter case the 12 mm side was parallel to the direction of consolidation loading (Fig. 5). Each slice was fitted with 2 targets (Fig. 5) formed from short wire needles mounted at each end of a central gauge length of approximately 20 mm, parallel to the long side. Observations were taken with a travelling microscope on each gauge length and continued to the end of the curing process; typical plots of progressive strain are shown in Fig. 8. It is seen that the major part of any strain occurred in the first bath of methanol–water, the change from methanol to methanol–acetone produced insignificant deformations while the change from acetone to resin produced negative strain.

An attempt has been made to correlate fabric strain due to impregnation with the change in surface tension from that of free water (75 dynes/cm) to that of the first bath of methanol–water. It is postulated that the diffusing methanol which becomes a third phase within the existing two phase kaolinite–water system changes the effective thickness of the adsorption phase surrounding each particle of kaolinite as well as affecting interparticle repulsion and as a consequence the effective specific surface of the fabric is changed.

Surface tension, which may be strictly regarded as a measure of the change in free energy or of the work done in increasing an area of free liquid surface, is thus by implication used as a measure of the interaction energy involved in changing the effective specific surface of the fabric. Available data includes surface tensions for methanol in water, and acetone (Weast, 1968). It is noteworthy that the surface tensions in air of 100% methanol and 100% acetone at 20°C are respectively 22·65 and 23·70, dynes/cm, and this small difference may be related to the negligible strain experienced during the sequence methanol, methanol–acetone, acetone (Fig. 8). Figure 9 gives a correlation between the change of surface tension and strain after immersion in the first bath; it illustrates the significant difference in strain behaviour between directions "parallel" and "perpendicular" to the consolidation direction. The reason for these differences is obscure, although they are conceivably associated with differences between integrated "parallel" and "perpendicular" interparticle forces and changes in those forces consequent upon changes in the adsorption phase. The strains observed appear to be tolerable although clearly the deviatoric strain implies a shear distortion of the fabric due to impregnation.

Fabric strain may be regarded as having two components, one being the volumetric strain d which is the sum of three lineal strains in mutually perpendicular directions which may be the principal strain directions. The second component is the

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Table 2. Impregnation sequences

Period (days)	Very rapid	Rapid	Medium	Slow
1	W:M::96:4	W:M::75:25	W:m::80:20	W:M::90:10
2	W:M::80:20	W:M::50:50	W:M::60:40	W:M::80:20
3	M:100	W:M::25:75	W:M::40:60	W:M::70:30
4	M:100	M:100	W:M::20:80	W:M::60:40
5	M:A::96:4	M:100	M:100	W:M::50:50
6	M:A::80:20	M:A::75:25	M:100	W:M::40:60
7	A:100	M:A::50:50	M:A::80:20	W:M::30:70
8	A:100	M:A::25:75	M:A::60:40	W:M::20:80
9	Resin	A:100	M:A::40:60	W:M::10:90
10		A:100	M:A::20:80	M:100
11		Resin	A:100	M:100
12			A:100	M:A::90:10
13			Resin	M:A::80:20
14				M:A::70:30
15				M:A::60:40
16				M:A::50:50
17				M:A::40:60
18				M:A::30:70
19				M:A::20:80
20				M:A::10:90
21				A:100
22				A:100
23				Resin

shear strain which may be conveniently expressed as octahedral shear strain ϕ_{oct} (Scott, 1963):

$$\phi_{oct}^2 = \tfrac{4}{9}\{(\epsilon_1-\epsilon_2)^2+(\epsilon_2-\epsilon_3)^2+(\epsilon_3-\epsilon_1)^2\} \quad \text{(i)}$$

where ϵ_1, ϵ_2 and ϵ_3 are the principal strains. The octahedral normal strain is given by:

$$\epsilon_{oct} = \frac{d}{3} = \frac{1}{3}(\epsilon_1+\epsilon_2+\epsilon_3). \quad \text{(ii)}$$

In the case of the material under investigation the major principal stress and strain directions are assumed to be parallel to the direction of consolidation and therefore vertical; the minor and intermediate principal strains are thus horizontal and presumed to be equal. Equations (i) and (ii) may then be reduced to:

$$\phi_{oct} = \tfrac{2}{3}\sqrt{2}(\epsilon_1-\epsilon_2)$$

$$\epsilon_{oct} = \tfrac{1}{3}(\epsilon_1+2\epsilon_2).$$

These two strain functions are plotted (Fig. 9) against the change in surface tension in the initial methanol–water bath for two instants in the impregnation sequence; at the end of immersion in the initial methanol–water bath (suffix *i*) and at the end of curing (suffix ult.). It is seen that if account is taken of the equivalence of the averages of ϵ_1 and ϵ_2 at an initial change of surface tension of 27·5 dynes/cm, ϕ_{octi} becomes zero at this surface tension whilst ϵ_{octi} is not greatly in excess of its minimum value. This suggests that an optimum methanol concentration for the first bath is 22%, which has a surface tension of 47·5 dynes/cm at 20°C.

No data has been found for surface tensions of resin mixtures and the appearance of Fig. 8 suggests that an investigation of the relationship between strain and change of surface tension at the resin bath stage would be useful. For the purpose of this preliminary study average values of ϕ_{octult} and ϵ_{octult} for all the cured material, irrespective of resin type have been plotted on Fig. 9 and although clearly a more detailed investigation of each resin is required the inference may be drawn that there is some correlation between ultimate strain and the surface tension in the first bath. This data implies that the optimum initial methanol concentration is 25%.

The relevant conclusion in respect to the observations now presented is that both the volumetric and shear strains in the fabric under examination are tolerable so far as the "soft" material is concerned. Clearly impregnation strains in the "hard" material should be similarly investigated, however it is assumed that these strains were of the same order.

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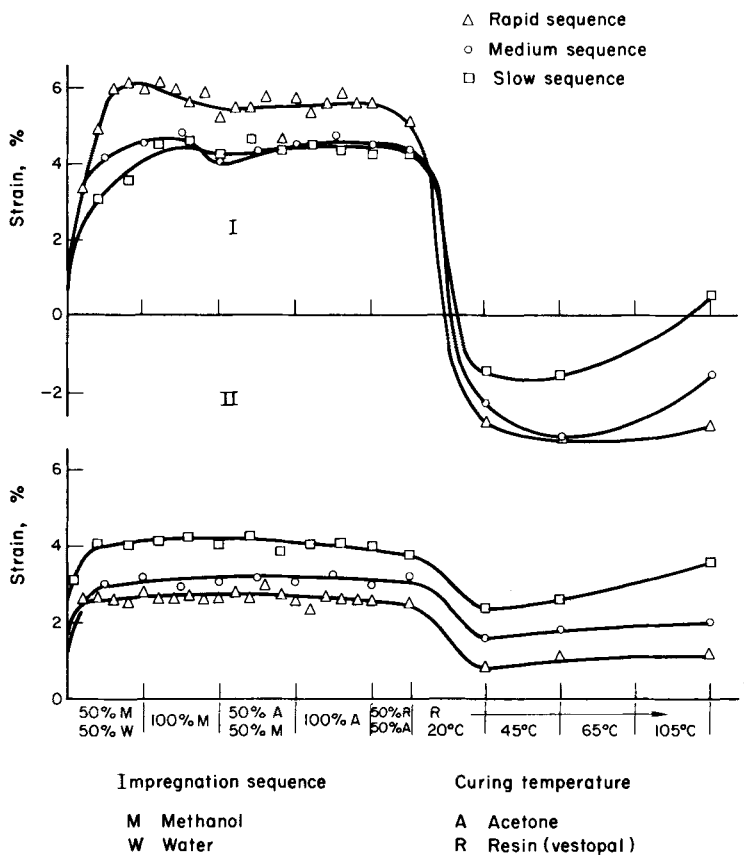

Fig. 8. Strain in soft kaolinite due to impregnation; typical sequence (Table 2). I. Parallel slice (Fig. 5); II. Perpendicular slice (Fig. 5).

The "rapid" impregnation sequence was adopted for the main work, since it appears to achieve the best compromise (Fig. 9).

Araldite AY18 with hardner HZ18 was found to be an excellent resin for impregnation. It was used in proportion AY18:HZ18::100:75 by weight. It has a low viscosity; 0·2 poise at 21°C and therefore impregnates easily, also it polymerizes slowly taking four days to solidify at room temperature, thus allowing time for full impregnation; it sets to give a hard clear resin.

Araldite AZ15 with hardner HZ15 and acetone as a diluent mixed in the respective proportions 100:30:10 was used successfully. Without acetone its viscosity is 4·5 poise; the acetone ensures full penetration and the resin when cured is harder than AY 18. It takes over 4 days to solidify at room temperature, which enhances the likelihood of full impregnation. However curing, which involved heating to a temperature of 105°C, unless allowed to proceed gradually is accompanied by a possible sudden evaporation of acetone with attendant disruption of the fabric.

Vestopal W with styrene to lower its viscosity, 0·5% cobalt octoate as an accelerator, methyl ethyl ketone peroxide (M.E.K.P.)—SD as a catalyst, and acetone as a diluent were used in the respective proportions 95:5:2:2:100. Extremely thorough mixing is essential if non uniform shrinkage is to be avoided during curing. While Vestopal will penetrate easily when mixed properly, its flow characteristics are susceptible to temperature changes. Also while it is, when cured, strong and clear it is unfortunately brittle and prone to fracture in the ultratome.

Bakelite R18774/1 mixed with hardner DQ 19262 and acetone as a diluent in the proportions 100:30:20 has all the advantages of AY18 except that to cure without cracking involves heating with comparatively small temperature increments up to

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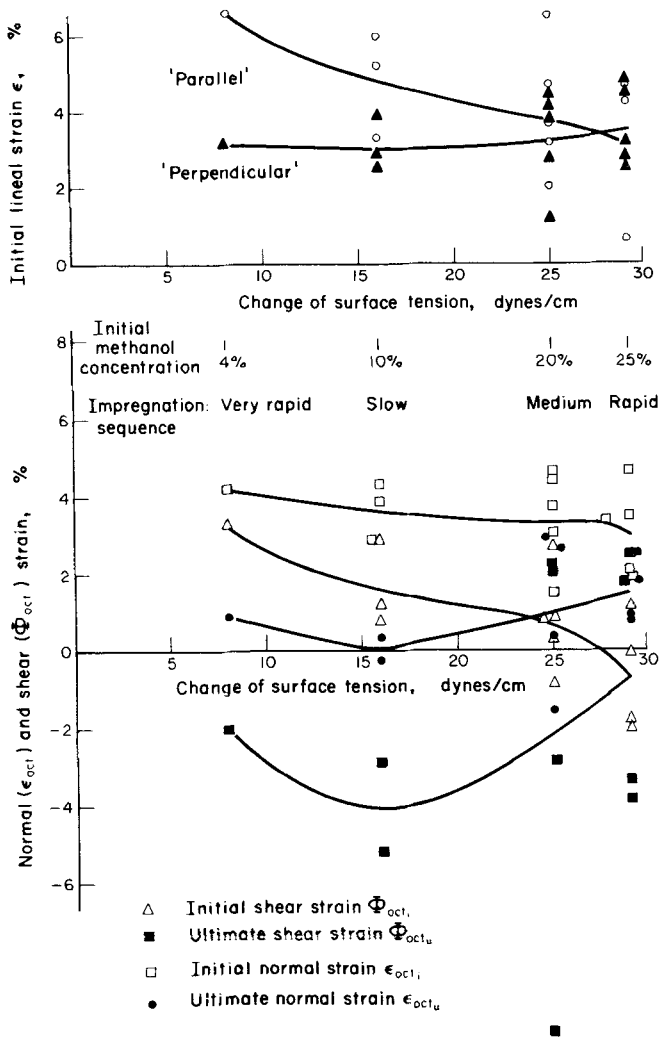

Fig. 9. Initial lineal strain, shear strain, and normal strain, also ultimate shear and normal strains in soft kaolinite related to change in surface tension on immersion in first bath of impregnation sequence.

105°C. Bakelite has the advantage of being flexible and is therefore less liable to damage than AY18 in the ultratome.

Of these four resins equally consistently good results were obtained with AY18 and Bakelite.

Ultratomy

After curing, a "perpendicular" slice intended for ultratomy would normally be cut in half (Fig. 5) by hacksaw across the 12 mm width; one half would then be cut with further parallel cuts into sticks of 12 mm × 2–3 mm × 2–3 mm which would be cut in two thus exposing two 2–3 mm square faces of "undisturbed" material. The square sectioned stick was carefully positioned in the ultratome and trimmed with a glass knife so that the 2–3 mm square face was reduced to a square face of approximate dimensions 0·5 × 0·5 mm; consequently any fabric which may have been damaged either by slicing of the wet soil or by sawing was removed. The resulting thin sections cut from the trimmed stick were thus from planes perpendicular to the direction of consolidation. The remaining half slice was cut into similar sticks parallel to the original 25 mm side, however the trimmed ends were given trapezoidal (Kay, 1965)

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end faces of approximate dimensions 0·5 × 0·5 mm. The trapezoids had their longest sides parallel to the hacksaw cuts and therefore perpendicular to the direction of consolidation. The resulting trapezoidal thin sections which were in planes parallel to the direction of consolidation were recognised under the electron microscope and the fabric observed could therefore be related to the direction of consolidation.

An L.K.B. Ultratome III was used for thin sectioning with a 1·4 mm diamond Ge–Fe–Ri knife having a knife angle of 45° and set to a clearance angle of 5°–6°; it is important to follow the makers' instructions regarding cleaning of the knife between uses if damage to sections was to be minimised. The optimum thickness of thin sections was found to be 700–900 Å; sections of this thickness were easily cut without the damage referred to above, they are self supporting under electron bombardment up to 80 kV except on 100 mesh (100 μ aperture) and long slit type grids (117 μ wide × 24 mm), and resolution under the electron microscope was satisfactory. Thinner sections down to 500 Å gave better resolution even at 60 kV but were difficult to obtain without damage in the ultratome and always needed a carbon film for adequate support on the grid which could not be larger than 200 mesh (50 μ aperture). A section of 1000 Å may easily be obtained without loss of particles or other damage, it will withstand electron bombardment up to 100 kV but with some loss of resolution and is self supporting on any type of grid. The grid preferred for scanning across a shear structure was the slit type of 117 μ width, and for the preferred 700–900 Å thin section this necessitated limiting the voltage to 60 kV if the use of a carbon support film was to be avoided.

The two types of "stick" referred to previously may be taken at random from randomly taken "perpendicular" slices obtained from a bulk sample, and still yield acceptable data regarding the fabric of the consolidated material. This procedure assumes that the fabric is homogenous throughout and whilst this may be justifiable for the contrived fabric it is not viable when seeking information regarding shear structures. Such structures, are not amenable to random sampling and a different technique is required: for this purpose the method of selected area thin sectioning was developed.

Selected area thin sectioning

It has been observed above that a longitudinal vertical slice through the direct shear box, which may be assumed to impose a two dimensional stress system, will contain the shear induced structures in a two dimensional array. These shear induced structures were samples for electron microscopy in the following manner. After curing the resin impregnated slices, approximately 1 mm was carefully ground off one face using successively carborundum powders 320 mesh and 600 mesh to give a plane surface which was then mounted on a geological slide with Canada Balsam. The reverse face was then ground in a similar manner until the sample thickness was 40 μ. Optical microscopic examination (×24) under transmitted polarised light then permitted the pattern of shear induced structures to be established (Morgenstern and Tchalenko, 1967b). Having located a feature (Figs. 15, 16) of specific interest within a shear induced structure, it was isolated within a 2 mm square block of material by incision with a razor blade whilst under observation under the polarising microscope, care being taken to ensure firstly that the point of interest was approximately in the middle of the incised area and secondly that the original orientation of the 2 mm × 2 mm block relative to the slice was precisely maintained throughout all the subsequent operations. By melting the Canada Balsam the 2 mm square block was easily removed and after setting on a moistened micro-scopic slide was further trimmed to approximately 0·5 mm square and at the same time one corner was chamfered so that subsequent orientation was facilitated. The next stage involved preparation of an Araldite stick having a 2 mm × 2 mm section with a precisely squared end at right angles to its axis. This end was painted with Araldite and lowered vertically over the 0·5 mm × 0·5 mm block as it rested on the horizontal slide so that the block became integral with the stick. After curing, the stick was ready for the previously described preparatory trimming prior to ultratomy.

Optical microscopy was conducted in a similar manner to that described by Mitchell (1956) and Tchalenko (1967).

Electron microscopy was performed with a Siemens Elmiskop I.

OBSERVATIONS

The dry densities of the "hard" and "soft" materials were 1·24 and 1·04 g/cm^3 respectively, and these reflect the relative hardness of the two materials.

Ambient fabric

The fabrics of the "soft" (Fig. 3) and "hard" (Fig. 4) materials illustrate a greater degree of flocculation in the former which, since this phenomena is associated with the state of the original suspensions, is attributable to the much lower

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initial moisture content and the tendancy therefore for edge to face contacts to be established between particles which would be in much closer proximity than was the case with the "hard" material suspension. The "hard" material appears to contain packets, although within many of these the individual particles are separated suggesting a comparatively dispersed material. The birefringence ratios (Morgenstern and Tchalenko, 1967) β of the soft and hard materials are respectively $0{\cdot}63 \pm 0{\cdot}06$ and $0{\cdot}24 \pm 0{\cdot}07$; a value of 1 represents perfect randomness and 0 represents perfect orientation. The significantly higher degree of preferred orientation perpendicular to the consolidation direction in the "hard" material which is confirmed by the appearance of the micrographs is attributable both to the greater dispersion and to the much higher consolidation load, and this greater degree of order in the "hard" fabric is reflected in its lower void ratio which was 1·14 compared with 1·54 for the "soft" material.

Shear loading

Shear loading was carried out with the "hard" material in a state of heavy overconsolidation and as might have been expected it dilated (Fig. 10). The "soft" lightly overconsolidated material on the other hand collapsed before shear failure. The "hard" material, characteristically has a considerably higher peak strength (1·36 kg/cm^2) compared with the soft material (0·42 kg/cm^2), and the stress strain curves are typical of heavily overconsolidated and lightly overconsolidated material (Wu, 1966). Straining continued until well past peak stress and the material within the main displacement discontinuities would then be in a state intermediate between peak and residual (Skempton, 1964).

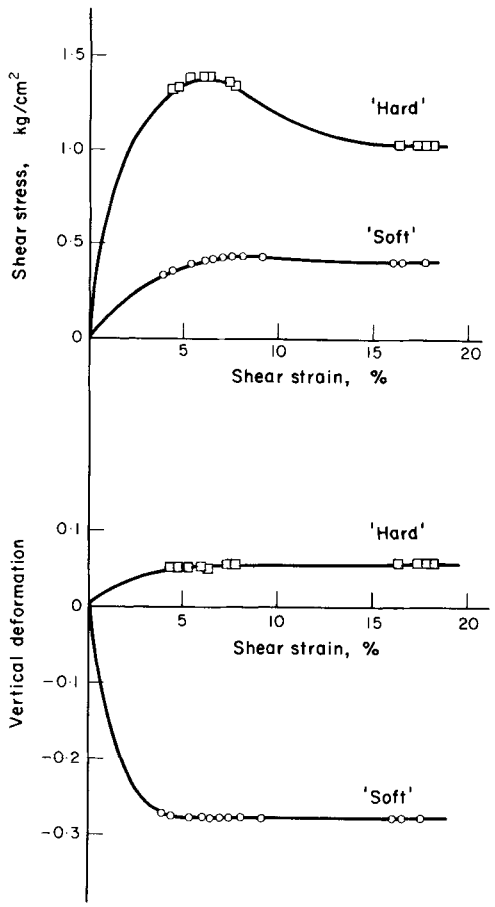

Fig. 10. Stress-strain curves for kaolinite in direct shear box; σ_N for hard ("horizontal") and soft ("vertical") material is 2·4 and 0·55 Kg/cm^2 respectively. Rate of shear-strain is 6×10^{-5} mm/sec.

Shear induced structures

It is seen (Figs. 11, 15) that the modified shear box was not entirely sucessful in simplifying the pattern of shear induced structures, although a reasonable degree of success was generally achieved with "soft" material. It should be noted that Figs. 11, 12 are of a slice impregnated with Carbowax and not resin. This slice was taken immediately adjacent to the resin impregnated slice from which the thin section for Fig. 13 was obtained, and which was regretably destroyed before an optical micrograph had been taken; the pattern of shear induced structures was identical in both slices. The mottled appearance of the micrograph is a characteristic of carbowax impregnation.

The shear induced structures illustrated in Figs. 11–17 may be compared qualitatively and related to the characteristics of the two consolidated materials, it being observed that the soft and hard materials behave in different and distinctive modes. The high degree of preferred orientation in all shear induced structures compared with the original material is noteworthy.

When comparing optical and electron-micrographs it may be observed that whereas in the former (Figs. 11, 12 and 15) the shear induced structures appear to have almost straight or slightly curved boundaries and to be of uniform width, in the latter the boundaries are irregular and structures vary in width. It is inferred that the regular and simple overall pattern observable by optical microscopy is an average of the pattern observed by electron microscopy and this suggests that while local variations in ambient fabric occur and cause irregularities in the shear structures, the

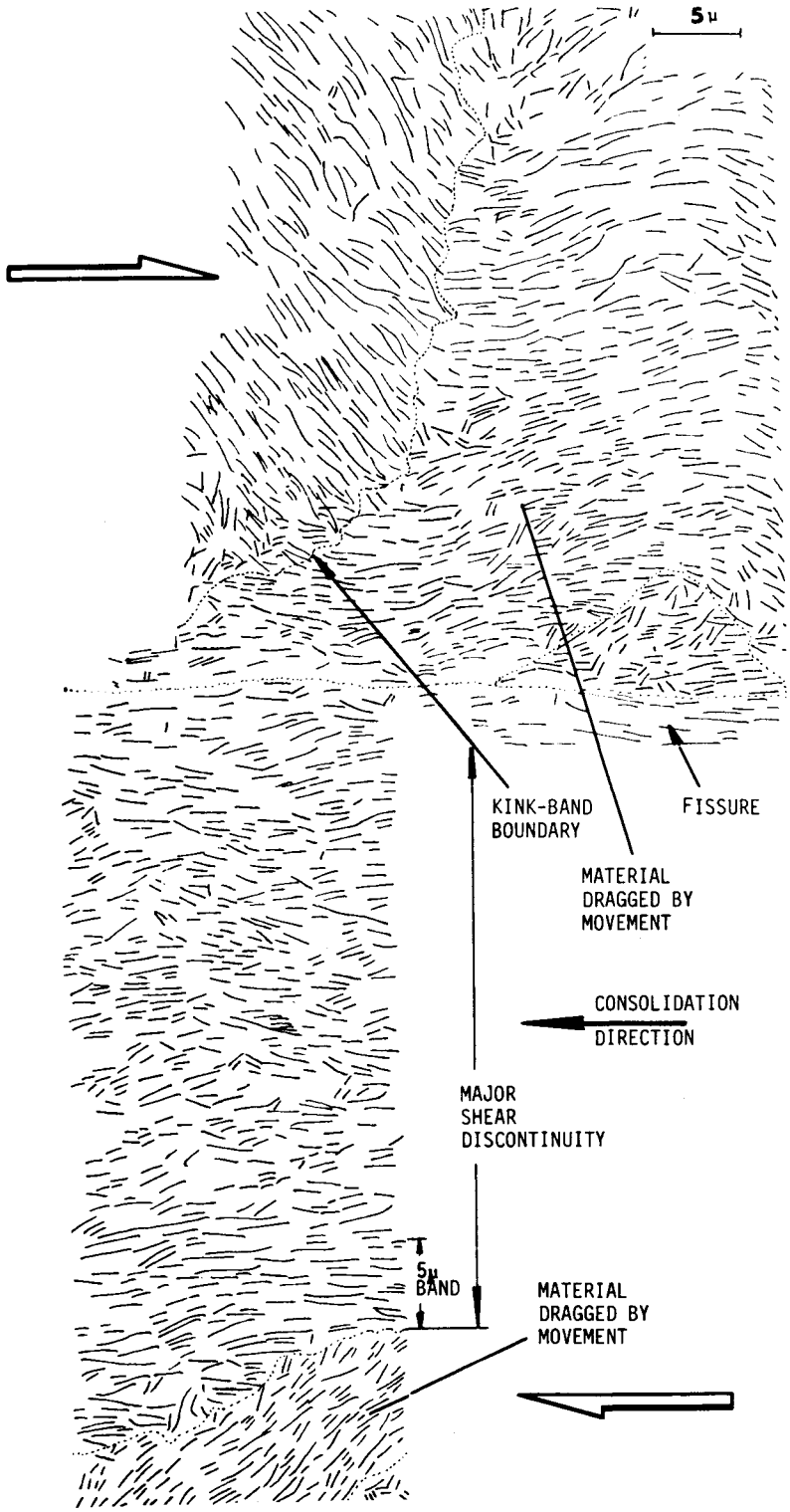

Fig. 14. Graphical representation of Fig. 13.

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average behavioural response is ultimately uniform. Nevertheless the presence of local fabric irregularities would explain the phenomena of local failure which has been adduced to explain the non-elastic behaviour of cohesive soil (Bjerrum and Kenney, 1967) under low stress.

Kink band structures in the soft material (Fig. 12) are similar to those found by Morgenstern and Tchalenko (1967b), and it would appear from the description of the preparation of their material that it would probably have a fabric similar to that illustrated in Fig. 3. It is remarkable that the clay platelets (Fig. 13) are highly oriented not only within the major displacement discontinuity but also in the kink bands. It has been observed by geologists that kink bands are usually developed in materials which possess well developed thin layering (Ramsay, 1967). It has also been found (De, 1970) that kink bands are not found in kaolinite subjected to shear loading until after failure. It is seen from a comparison of Figs. 3 and 13 that the sheared material has a significantly lower void ratio, which may be associated with the compression observed before peak stress (Fig. 10). It is therefore inferred that this "vertical" material, albeit with an almost random fabric, collapsed under shear loading before peak shear stress, its particles sliding and turning through angles between 0° and 90° and on average through an angle in excess of 45°, and consequently became highly oriented parallel to the direction of shear loading. Since there is little sign of particle degradation in Fig. 13 it is also inferred that this large rotational deformation took place without mutual fracturing. Having had this well oriented fabric imposed upon it the material would then be susceptible to kink band formation, the mechanism for which appears to be a collapse of the highly oriented fabric in the direction of shear loading, characterised by folding about narrow hinge zones (Fig. 13). It is inferred from the appearance of the extremely irregular hinge zones, which while normally forming a distinct kink band boundary are in places almost indefinite and across which particle clusters from each side are interlocked, that while there has been displacement perpendicular to the shear direction there has nevertheless been no significant differential strain along the hinge zones. However the magnitude of strain perpendicular to the shear direction is significant and its change of direction at each succeeding hinge suggests that each kink band has been subjected to simple shear strain.

It is noteworthy that kink band structures are absent from the heavily overconsolidated material even though it had an initial preferred orientation parallel to the shear direction; it is inferred that the degree of parallelism was insufficient to allow folding to initiate. Furthermore it may be observed that this material dilated prior to peak stress with the implication that the fabric became slightly more random so that initiation of folding was further inhibited.

The main displacement discontinuity in the hard material and its branch contain between them a large inclusion of ambient material (Figs. 16, 17). The appearance of this inclusion implies that prior to peak stress the original material was not greatly affected by the imposed loading; it is inferred that it has merely suffered bodily rotation through almost 90° since its fabric is very similar to that illustrated in Fig. 4 in respect of degree of preferred orientation, particle size, and appearance of packets. It is also inferred that the rotation of this inclusion would occur at the time of formation of the main displacement discontinuity and therefore that, if such a movement is typical of many others, failure was accompanied by a considerable energy input and significant dilation. Two further features suggest a large energy requirement; firstly the considerable degradation of particles within the main displacement discontinuity and its branch (see Figs. 4, 16) and secondly the extremely irregular jagged boundaries to the displacement discontinuity which implies a high degree of interlocking which had to be overcome. The strength of this interlocking may be attributed to the strong bonding at interparticle contacts; it has been suggested above that the high initial moisture content of the suspension of the hard material would limit the number of interparticle contacts due to flocculation, however the intensity of the subsequent consolidation pressure would directly govern the proximity of particles at those contacts which had been previously made and a high consolidation pressure would thus produce very strong interparticle attraction since Van-der-Waals bonding is inversely proportional to (interparticle distance)^5 (Rosenqvist, 1968) and becomes effective when the interparticle distance is not greater than 10–20Å. These observations conform with the characteristic shape of the stress strain curve (Fig. 10) which indicates that a comparatively high peak stress was required to overcome the shear resistance of the hard material.

The shape of the stress–strain curve for the soft material may similarly be related to the appearance of the major displacement discontinuity illustrated (see Figs. 3, 13) which is principally comprised of intact particles and has comparatively straight or slightly curved boundaries. These features imply a lower requirement of energy input at failure; this is consistant with the microstructure of the soft material which although flocculated, implying a significant number of interparticle contacts, was

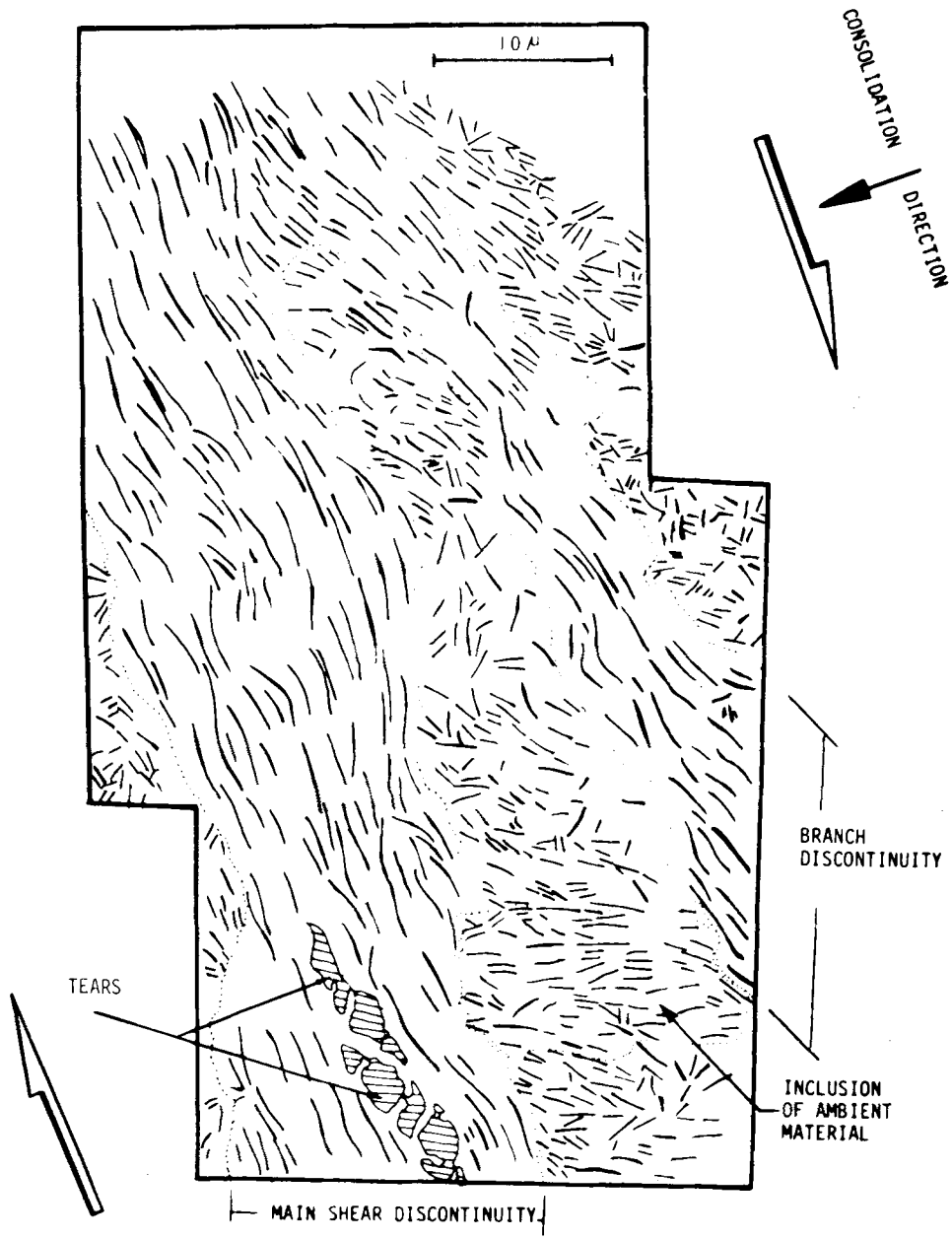

Fig. 17. Graphical representation of Fig. 16.

lightly consolidated so that bonding would be less effective.

It is interesting that there is a high degree of preferred orientation and dispersion in the displacement discontinuities in both materials and this condition is associated with the large shear strains to which both samples had been subjected subsequent to shear failure. It is also apparent, particularly in the case of the soft material that there is an even higher degree of preferred orientation immediately adjacent to the moving boundaries; a 5 μ band of very well oriented particles extends from the lower boundary and a pronounced fissure has been opened up by the impregnating resin at the upper boundary (Fig. 13). The fact that resin has entered so freely is indicative of a weakness in

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bonding across the boundary and this would be explained by an excess of face to face, i.e. parallel contacts between particles. This observation supports the suggestion (Foster, 1967) that any moving body will adsorb the plate-like clay particles in a veneer which gradually thickens and becomes smoother as the surface moves over the dispersed material in the disturbed zone. Further evidence that increased parallelism adjacent to moving boundaries is caused by their movement is the alignment of particles within the kink bands where the latter abut the shear discontinuity (Fig. 13); their attitude relative to particles within the main body of the band suggests dragging of the boundary over the zone of disturbance within the discontinuity.

CONCLUSIONS

The success of the primary objective may be assessed from the appearance of the electron-micrographs illustrated in Figs. 3, 4, 13 and 16, in which there are few artefacts and clear representations of fabric both in consolidated material and shear induced discontinuities. Impregnating techniques have been developed and optimized within limits, but further work is required on this aspect of microscopy. Whether the fabric is susceptible to quantitative analysis or not has yet to be established although the appearance as compared with natural clay (Figs. 18, 19) or unfractionated remolded kaolinite (Smart, 1967) suggests that this possibility may ultimately be achieved.

The objective of relating macro- and micro-structure has met with a large measure of success (Figs. 11–17) and the technique of selected area ultra thin sectioning offers a useful means of investigating fabric and fabric changes and their relationship to engineering behaviour.

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Résumé – Une revue des études de structure des argiles suggère la nécessité de rapporter ces caractéristiques qui sont révélées à deux niveaux de grossissement fournis par la microscopie optique et électronique, et une technique, spécialement élaborée pour cette étude, est décrite dans le contexte des premiers stades d'une étude à long terme sur la relation entre la structure et son comportement du point de vue engineering. Deux argiles kaolinitiques, avec structures aménagées, ont été préparées en contrôlant la grosseur des particules, la teneur d'humidité et la pourcentage de la suspension, ainsi que la charge de consolidation, et ces argiles ont été ensuite soumises jusqu'au bout à une force de cisaillement. Les techniques d'imprégnation de résine qui permettent de découper la kaolinite en sections minces, pour la microscopie électronique, ont été optimisées en vue de réduire au minimum la contrainte et l'endommagement de la texture pendant l'ultratomie.

Les structures des matériaux ambiants dures et souples sont comparées qualitativement au moyen de micrographes électroniques et sont expliquées en terme de procédés préparatoires adoptés pour le contrôle de la structure. Les textures des deux types de structure à cisaillement induit sont également comparées, du point de vue qualitatif, et une explication est donnée en terme des textures originelles et de la force de cisaillement ultérieure.

Kurzreferat – Eine Übersicht über Untersuchungen der Textur von Tonmineralen lässt es geboten erscheinen diejenigen Texturmerkmale, die auf zwei Vergrösserungsebenen durch optische und durch Elektronenmikroskopie sichtbar gemacht werden, in eine Beziehung zu einander zu bringen, und es ist zu diesem Zwecke eine Methode entwickelt worden, die im Zusammenhang mit den Anfangsphasen einer langzeitigen Untersuchung der Beziehungen zwischen Textur und technischem Verhalten, beschrieben wird. Zwei kaolinitische Tone mit geplanter Textur wurden bereitet durch Kontrolle der Teilchengrösse, des Feuchtigkeitsgehaltes und des pH der Suspension sowie der Verdichtungslast, und wurden einer Scherbeanspruchung bis zum Bruch unterzogen. Verfahren der Kunstharzimprägnierung, die es gestatten den Kaolinit in dünne Schnitte für die Transmissionselektronenmikroskopie zu schneiden, wurden optimisiert um die Beanspruchung der Textur und die Beschädigung während der Ultratomie auf ein Minimum zu beschränken.

Die Texturen des harten und weichen Umgebungsmaterials werden qualitativ mittels Elektronenmikrographien verglichen und werden mit den für die Kontrolle der Textur angewendeten Verfahren in Beziehung gebracht. Die Texturen der zwei Arten durch Scherung hervorgerufener Gefüge werden ebenfalls qualitativ verglichen und im Zusammenhang mit der ursprünglichen Texturen und der nachfolgenden Scherbelastung erörtert.

Резюме — Обзор текстурных исследований глин выявил необходимость сопоставления тех их характеристик, которые получаются на двух уровнях увеличения, т. е. с помощью оптической и электронной микроскопии. Для решения этой задачи разработана методика, которая изложена ранее вместе с результатами длительного предварительного исследования соотношений между структурно-морфологическими и техническими характеристиками глин. Две каолинитовые глины с заданными текстурами приготавливались с контролем размеров частиц, влажности, pH суспензии и степени спрессованности, и подвергались сдвиговым напряжениям с целью образования нарушений. Методика пропитывания пластическими наполнителями, позволяющая получить тонкие срезы каолинита для исследования в электронном микроскопе, была несколько усовершенствована для уменьшения структурных напряжений и повреждений при ультрамикротомировании.

Строение твердых и мягких материалов качественно сравнивалось по электронномикроскопическим снимкам и трактовалось с учетом методик препарирования, применяемых при структурно-морфологических исследованиях. Особенности сдвиговых структур двух типов также количественно сравнивались и объяснялись на основе данных о строении исходных материалов и степени последующей сдвиговой обработки.