

(4) H in Equation (11) is nothing but total thickness. The same nomenclature has been used by mistake. This H in Equation (11) has nothing to do with length of drainage path. In the example solved H has been correctly taken as total thickness (8.0 m).

(5) Another correction in page 72 for the last line should be made. It is

$$C_{vv} = 0.2 \times C_{VR} = 0.001 \text{ sq cm/sec.}$$

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* * *

The Bakelite Resin—Impregnation and the Microstructures of Kaolinite*

by

P.K. De

(1) R. BHASKARAN**

The author has, in this paper, established the versatility of Bakelite resin as an excellent impregnating material, from his experience. The electron microscopes have lately been extensively used for study of soil fabric. One of the main drawbacks of electron microscopy in general is that, while it gives a great insight into the arrangements of particles, the sample area is too small for evaluation of total fabric. Therefore, to evaluate the total fabric or particle orientation patterns, the electron microscopic study has to be supplemented by optical and X-ray diffraction studies of fabric as well. No doubt, individually the latter two methods have also their own limitations, but the optical methods are best suited for identification of overall patterns and the X-ray is a good method for quantitative estimation of fabric least subject to human interpretation (Quigley and Thompson, 1966). Even between the two types of electron microscopes widely used at present, the scanning electron microscope appears to be preferred of late because of its relative simplicity, ease of preparation of samples, large depth of focus which gives a better insight into the three-dimensional arrangements of particles and aggregates of particles and the wide range of magnifications possible (Gilliot, 1969; Barden and Sides, 1970, 1971). Lately attempts have been made to quantitatively measure the particle orientation using the scanning electron microscope (Tovey, 1971). On the other hand the transmission electron microscopy requires, as stated by the authors, the specimen to be thin enough for the electron beam to penetrate. Thin sectioning and ultra thin sectioning may damage the specimen and the section may not be truly representative of the material in the bulk (Barden and Sides, 1970). Surface replication may be employed, but these do not give complete information if reentrant angles exist or if

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the surface is rough or irregular. Moreover both these techniques require considerable skill, and are also time consuming and tedious (Gilliot, 1969).

The resolving power of the scanning electron microscope is less than that of transmission microscope but is high enough for clay studies (Barden and Sides, 1971). However, according to Yong (1971) the finer detailed points are better presented using transmission electron microscope. The various techniques for specimen preparation for fabric studies using the electron microscope has been treated by Smart (1969) and Barden and Sides (1971). The latter has treated specially the sources of sample disturbance. The aim of this discussion so far has been to briefly cite the limitations of the technique, but nevertheless the writer fully realises that considerable inroads are being made in developing techniques for micro-structure observation of clay fabric and recognises the work of the author as a positive contribution. The following factors have also to be considered before a general analysis of the results are undertaken:

- (1) Normal stress applied during shear on the plane of shearing is very low when compared to the original consolidation stress, therefore the test is being carried out on an O.C. (over-consolidated) soil. Moreover depending upon direction of the sample the O.C. Ratio will differ as the normal stresses which acted on the different planes during the initial one-dimensional consolidation were different. In so called horizontal sample $OCR = \frac{45}{2.4}$ and in the vertical sample $= \frac{45}{2.4} \times K_0$ and in the I sample the value would be in between (in addition a shear stress would have been acting on the plane). The shearing rate is sufficiently slow and therefore the test would have been a drained one which means that volume changes would have occurred doing shear. The volume change will naturally be different for specimens at different orientations not only because of the particle orientation influence but also due to the different stress history effects on the plane of shear.
- (2) The particle reorientation in shear plane during a drained test wherein volume changes take place will be different from that which takes place during an undrained shear where volume changes do not take place. The study is applicable only to *drained condition and the pattern of fabric changes may be different if drainage is prevented and for shear at faster rates (strain rate may have other influences besides preventing drainage).*
- (3) As can be understood, these tests have been conducted on horizontal specimens wherein there is an axis symmetry due to the fact that direction of shear is on a plane parallel to the plane of orientation of particles and vertical and inclined specimens wherein the orientation in a simplified elevation is as shown in Figures 1 and 2. (The infilled lines in the figures indicate orientation of particles). Markedly different behaviour is possible if the vertical and the inclined specimens are sheared with a different orientation as shown in Figures 3, 4 and 5. (The particle orientation has been shown in the side view in Figures 3 and 5).

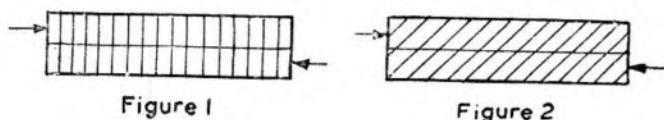


Figure 1

Figure 2

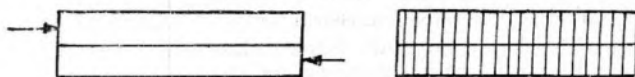


Figure 3



Figure 4

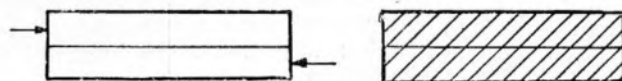


Figure 5

FIGURES 1-5

FIGURE 1 : Vertical specimen as tested by the author.

FIGURE 2 : Inclined specimen as tested by the author.

FIGURE 3 : Vertical specimen—A second possible orientation.

FIGURES 4 & 5 : Inclined specimens—Other possible orientations.

In addition to the authors, Morgestern and Tchalenko (1967) and Slone and Kell (1967) have tried to study the microstructural changes accompanying shear in a direct shear apparatus, using optical methods and electron microscopy respectively. Morgestern and Tchalenko (1967) have used consolidated kaolinite whereas Slone and Kell (1967) have used compacted kaolinite. The writer feels that to get complete picture of the fabric changes, the same has to be studied by the use of two or three techniques, as mentioned earlier. Study of fabric changes accompanying shear in simpler stress configuration like such as compression tests with lubricated end p'atens and plane strains and simple shear conditions are of interest and it is hoped it would be for forthcoming.

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(2) D. VENKATESHWAR RAO*

The author has to be commended for the interesting study made on the bakelite resin impregnation technique on consolidated kaolinite. The writer has following few specific remarks to seek clarification on impregnation techniques and on ultramicrotomy, as some of the statements made by the author in the paper are found to be contradictory with the reported experiences in literature.

Impregnation of clays by bakelite resin admixtures does not appear to be a new method as reported by the author on page 322, but this has been used successfully for impregnating the montmorillonitic clays (Ross and Hendricks, 1945) and for various other ceramic products and sedimentary rocks (Franklin, 1968) to study their two-dimensional microfabric features by transmission electron microscopy employing the technique of ultramicrotomy.

Among the important organic compounds used to impregnate the clays for microstructural studies are epoxide resins, unsaturated polyester resins and monomer admixtures (Bowels, 1969, Franklin, 1968 ; Pusch, 1967 ; Smart, 1966 ; etc.)

The important types of *epoxides* are araldite admixtures (CIBA product) and bakelite resin admixtures (R 18774/1 + Hardner Q 19127) (Franklin, 1968 and Pusch, 1967). The viscosity of the epoxide admixture is about 1.2 to 1.130 poise (It has to be borne in mind that the viscosity of water is one centipoise). The admixtures generally yield hard impregnated samples upon proper curing. The epoxide admixtures make vacuum impregnation difficult due to frothing (Franklin, 1968 ; Smart, 1966 and Singh, 1967).

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Depth of impregnation may not exceed 0.1 in. even in the case of porous sandstones (Franklin, 1968). These admixtures are employed by using hot and/or cold hardness. The important *polyester resins* are Trylon compounds and Filabond compounds (Franklin, 1968). These admixtures have viscosity of the order of 3 poise. They are generally cold curing compounds and offer good dimension stability to the samples upon impregnation. These admixtures yield hard specimens. The impregnation of the admixtures into clay mass even under vacuum is very difficult. *Monomers* have excellent fluidity and can impregnate into small pores of clays by diffusion process. Hardness of the impregnated clay samples depends upon the choice of monomer (Franklin, 1968 ; Pusch, 1967). The impregnated clays can be easily cut with care. The monomers generally used are Styrene or Methyl Methacrylates mixed with catalysts. The viscosity of these admixtures are generally about 0.54—0.62 centipoise.

From the above discussion, it can be concluded that from the point of view of effective impregnation by the process of diffusion into clay mass and with low viscous admixtures (as stated by the author on page 321), the monomers are the best suited impregnating admixtures than epoxides and unsaturated polyester resins. It has also been reported that impregnation with admixture of methacrylate monomer and dichlorobenzoylperoxide (EWM) catalyst on illitic clays and natural soft marine sediments has yielded satisfactory ultra thin sections of the order of 300Å with no shrinkage effect (Pusch, 1967).

Replacement of pore water by alcohols has been reported to cause swelling of clays (Pusch, 1967 ; Silva et al 1965) and not shrinkage as reported by the author on page 317. The author has stated on page 316 that the dissected slices from the consolidated (45 kg/cm² pressure) kaolinite samples have been immersed in distilled water immediately after slicing. This may also cause slaking effect in clay microstructure due to absorption of water with concomitant swelling. Thus, the in situ clay microstructure may get affected. This may be further aggravated by subsequent alcohol and epoxide impregnations and curing at high temperatures up to 105°C.

The writer agrees with the statement of the author (p. 324) that thicker ultra thin sections yield good reliable and meaningful microfabric details than thin thin-sections, because the artifact effects are eliminated in the case of thicker thin section microtomy. A well known disadvantage of the ultramicrotomy is the deformation of the thin sections caused by cutting operation. It is assumed that a hard material deforms elastically, whereas a soft material deforms plastically (Pusch, 1967 ; Smart, 1966). Distortions of the thin sections may be caused when the diamond edge meets a coarse particle. In the case of coarse particles or crystals having layers of easy cleavages, the layers are generally parted without distortions of the surrounding material if the cutting and cleavage planes coincide. This is regardless of the size of particle. If the particles are not oriented, this may be pushed up ahead of the edge and thin-sections may be distorted. Generally, such distortions have no effect on clay particles less than 0.2 thick (Pusch, 1967). Thicker thin sections of the order of 1-10 and high voltage transmission electron microscope of the order of 1.50 MV are preferred to overcome the above defects due to ultramicrotomy. High voltage microscope provides with high penetrability of electron radiation than in the case of ordinary transmission electron microscopes. Thus, the high voltage transmission electron microscope facilitates to study

the microfabric of clays through thicker thin sections as well as specimens under desired relative humidity and pressure as reported by Pusch 1967.

The writer also would like to add that the two-dimensional microfabric studies by transmission electron microscopy are gradually replaced by scanning electron microscopy, because the later technique involves simpler sampling procedures and affords to study satisfactorily the three-dimensional microfabric details with greater depth of focus.

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(3) R.B. SINGH*

The writer has discussed the use of Araldite CY 212 vis-a-vis Araldite AZ 15 for use in impregnation of clay soils in the context of author's companion paper (De, July 1972); those comments are generally valid in the context of the present paper also.

The author has reported the use of a three-phase diffusion resin impregnation technique, which can be briefly called the 'methanol-acetone-resin' technique, for the replacement of pore water of clay slices followed by resin impregnation and curing. The writer (Singh, 1969) used a two-phase 'acetone-resin' diffusion technique for similar purpose for use in thin section polarising microscope studies, and had suggested its use in electron microscopy work (Singh, 1971). The two-phase diffusion resin impregnation technique could perhaps be used equally satisfactorily for electron microscopy work, and would possess the advantage of saving in time and effort as compared to the three-phase diffusion resin impregnation technique.

The author used electron microscopy approach to the study of microstructures of kaolin during shear, pointing out that the resolution and magnification of the optical microscopes is limited for such investigations. The writer feels that electron microscope studies such as those reported by the author must be supplemented by thin section polarising microscope studies, since only too small an area can be sampled in the electron microscope. It has been clearly brought out by Barden and Sides (1971) Yong

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(1971), and Parry (1972), that misleading or different interpretations of microstructures may result in electron microscope work (especially so in scanning electron microscope work), particularly for highly oriented structures viewed at large magnifications. Sample preparation techniques are more likely to vitiate the real microstructure picture in electron microscope work than in polarising microscope work. Whereas electron microscope studies of microstructures may not be considered complete in themselves (Yong, 1971), thin section polarising microscope studies can be considered so to a larger extent, as for example shown by studies of Morgestern and Tchalenko (1967).

Certain interpretations of microfabric features given by the author from the electron micrographs presented in the paper do not seem to be fully borne out. This is attributable in some measure to the poor quality of the micrographs as printed. However, the author's main conclusions regarding formation of shear discontinuities and their features are well borne out by the micrographs presented.

The writer would like to congratulate the author for the interesting research presented. Study of microstructures of soils is important to understand the fundamental mechanisms pertaining to their engineering behaviour; unfortunately related research reported in the literature is still meagre.

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AUTHOR'S REPLY

(1) The author would like to thank Sarvashri Bhaskaran and Rao for their discussions and interest in his paper.

It is agreed that the optical methods which use polarised light are probably the best suited for a quantitative evaluation of the total fabric of a clay matrix exhibiting birefringence in terms of β values. However, the birefringence ratio β varies according to the magnification under which observations are made. Although β is a measure of the degree of orientation, with β approaching zero for 'perfect' orientation and unity for 'random' arrangements, the degree of orientation is not a linear function even in a two-dimensional fabric model. So any quantitative analysis made by an optical method is not an easy one and always needs to be compared

on the basis of equal magnifications. This restricts the application of this powerful tool and although the author did use this important tool for various research programme, nevertheless it means that it is definitely not suitable for all types of research work.

The advantages of the scanning electron microscope which were stated in particular by Shri Bhaskaran, are to a certain extent misleading. The author is fully aware of certain well known advantages of the scanning electron microscope over the transmission electron microscope. However, apart from the restricted and often the complex sample preparation involved, the three-dimensional arrangements of clay particles result in a very complex picture of soil microstructure, which in the author's opinion can be described as a source of too much information in three-dimensional nature which cannot be evaluated in the light of present day knowledge. Nevertheless the Author agrees that often sample preparation for a transmission electron microscope can also be tedious and definitely requires considerable skill and practice.

The second possible orientation of the vertical specimens was studied at the same time but this did not indicate any major difference in the response to shear strength. The electron microscopic investigation of the shear induced microstructures of the shear zone did not show any marked differences in behaviour when the results were compared to the original microstructural data presented by the author, except the boundary of the shear plane was in general less defined. However, a significant difference in terms of strain at failure, shear strength at failure and at extended strain approaching residual condition for samples inclined at 45° and 135° was observed (Figure 1). Figure 2 shows a relationship between the peak and residual shear stresses and the direction of orientation of the initial fabrics. The shear strength behaviour of the samples at different orientations of

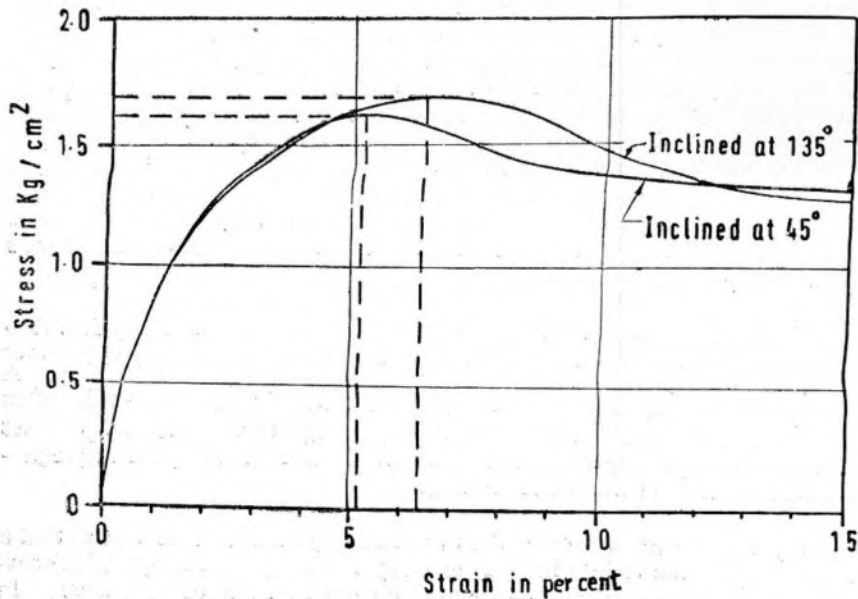


FIGURE 1 : Comparative stress/strain curves for two inclined specimens.

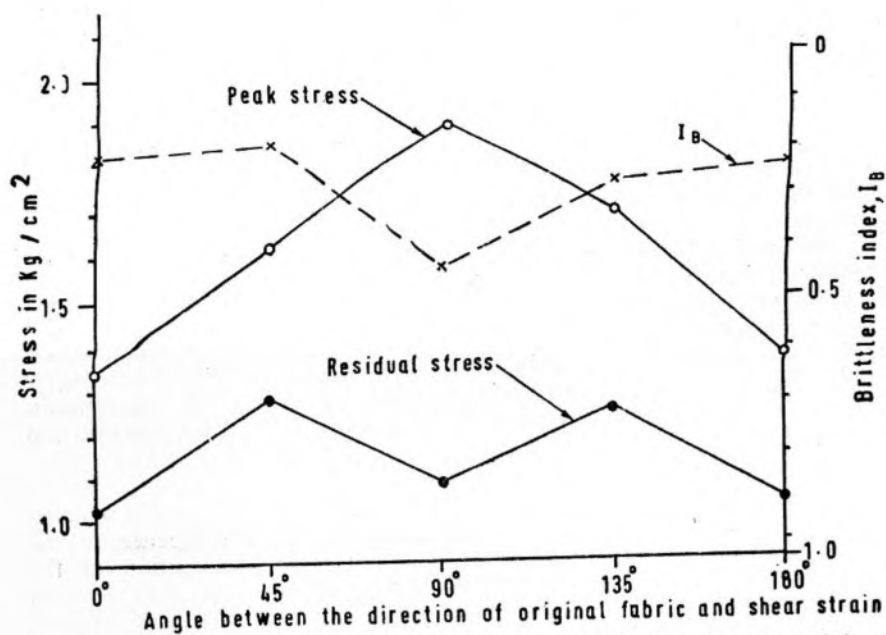


FIGURE 2 : Relation between the shear stresses and the direction of original fabric.

initial fabric and the brittleness index of each sample can be explained in a similar way to that described in the Author's original paper.

(2) In reply to Rao's comment, the Author feels that he never claimed to be the first researcher to use bakelite resin as an impregnating material. It is the detailed technique that matters. It is stressed again that the shrinkage of the thin clay slices was observed only in the resin impregnation stage and not at the methanol and/or the acetone stages of impregnation. Rao apparently assumed that it took place in the latter stages without going into the details given in the paper. Also it is desired to point out and assure him that the deformation, distortion and disruption of ultra thin sections at the diamond knife edge was taken into account in the reported investigation. The ultra thin sections that deformed, distorted and/or became disrupted were disregarded and eliminated. Any section that was even suspected to have undergone such distortion and deformation was automatically discarded.

The general discussions on various impregnating materials by Shri Rao is repetitive and hence need no comment. However, the author's experience with Methacrylates shows that it is a good impregnating material only. A dense non-homogeneous material impregnated with Methacrylates is often soft and in most cases definitely not strong enough to hold the clay particles at the knife edge, i.e., ultra thin sections get distorted due to poor impregnation in relation to ultra thin sectioning.

(3) The author welcomes the discussion by Singh. It is agreed that a two-phase 'acetone-resin' diffusion technique is adequate for optical microscopic investigation and can be extended for electron microscopic work. In fact the author modified the three-phase technique into two-phase at the