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QUANTITATIVE METHODS FOR MEASUREMENT OF SCANNING

ELECTRON MICROGRAPHS OF SOIL FABRIC

by

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ABSTRACT

The geometric arrangements of the constituents of soil fabric can be measured by using photogrammetric methods on micrographs from the scanning electron microscope. However, the observed specimen is only a small part of the parent material, and it is necessary to consider not only the perspective geometry in the microscope itself, but also any rotations of the specimens which may take place during preparation. Soil fabric is vary variable, and the detailed information may be unrepresentative of the sample as a whole, and rapid optical diffraction methods of a semi-quantitive nature are needed to select areas for the more detailed photogrammetric treatment.

INTRODUCTION

Soil fabric is defined as the spatial arrangement of the constituent particles and voids within the matrix of a soil. The particles and voids are frequently elongate or planar, and the geometric arrangement of these individual components into groups or aggregates, and the subsequent higher level arrangements of these aggregates, affects the stress-strain behaviour of soil as a whole. These inter-relationships between the stress-strain behaviour of soils and microfabric have been studied by several workers in recent years, but hitherto most have been of a qualitative nature using optical microscopic or transmission or scanning electron microscopic techniques. However, only by using detailed quantitative, or sometimes semi-quantitative, methods will it be possible to gain a complete understanding of the relationships.

The quantitative methods which may be employed depend on the type of instrument used. In electron microscopy, there are automatic methods

using image analyses on transmission electron micrographs (e.g. Foster and Evans (1971), Bennett et al (1978)), and an ingenious technique for scanning electron micrographs devised by Sokolov et al (1979-1980). However, all these studies have been related primarily to overall porosity measurements, and for the orientation of the constituent features it is normally necessary to resort to photogrammetric techniques either directly or indirectly. This paper examines two different techniques: a conventional photogrammetric method complements a method using optical diffraction. A third method using the digital intensity gradients at each point on a micrograph is described in detail in Tovey (1980), and will not be discussed further in this paper.

Before proceeding, it is necessary to briefly discuss the reasons for tackling the investigations by several different methods. The soil fabric can be very variable and even within an apparently homogeneous level. Several different types of microfabric have been recognised (e.g. Sergeev et al. 1978), and these range from an open honeycomb fabric in which the particles are in edge-face contact forming an open fabric with no preferred orientation to highly orientated laminar microfabric in which the particles are closely packed in a face to face manner. A third important type (see Fig.3) is a domain microfabric in which the individual particles are aligned in a nearly perfect arrangement into domains which themselves are randomly orientated with respect to each other. Thus it is necessary to place any conventional photogrammetric methods into perspective by resorting to rapid approximate two dimensional methods.

SELECTION OF AREAS OF STUDY

It is also necessary to use these techniques to select representative areas of specimens to assess the size of the area required for detailed examination, and to determine the distribution of particle arrangements of the different types throughout the sample as a whole. Further, since soil is a three phase material containing solid, liquid and gas it is important that the full effects of any damage to the fabric during preparation are known. All the pore water must be removed before the sample is placed in the vacuum of the microscope of the electron microscope specimen chamber. The best methods for sample preparation depend on the sample, and range from air-drying for materials below the shrinkage limit to careful freeze-drying (Tovey and Wong (1973, 1978), Sergeev et al (1978) for partially saturated soils or swelling soils and critical point drying methods for recently sedimented deposits (Tovey and Wong (1978)).

Fresh undisturbed surfaces of the soil must be revealed and although many workers have used fracturing methods, this is considered unsatisfactory as it can generate surfaces the orientations of which are not know with any precision. The present authors prefer to select a surface with known orientation by grinding or trimming in the desired direction, and by removing the disturbed layer of the specimen by either of the peeling techniques suggested by Barden and Sides (1971), or Tovey (1970) and Wong and Tovey(1975). The surface so produced can be treated in the non-photogrammetric methods as two dimensional orthoscopic photographs if the magnification is kept to about x 500 and the true working distance (or principal object distance W_p see Lane 1969, Tovey

19/3b) is kept as large as possible (typically 30-40mm as opposed to the normal setting of about 10mm).

Conventional photogrammetric methods are inappropriate in trying to assess any effects of preparation on the disturbance of the microfabric. The reason for this is that high magnifications ($\sim$x 5000) are needed to adequately resolve the particles, and the resulting area covered is so small that it is necessary to examine many areas on each of a large number of specimens and compare these with the results from other preparation techniques.

The differences in fabric may often be small and it is only by studying a large number of areas by the quantitative approximate methods can this information be gained. Such a method is the optical diffraction technique described later in this paper. Thereafter, selected representative areas can be measured by more conventional means. Apart from the methods of preparation, the techniques described in this paper are equally applicable to transmission electron microscope studies.

THE PHOTOGRAMMETRIC METHOD

In the scanning electron microscope a condition of central projection exists but unlike aerial photogrammetry, the semi-field angle is small and varies with magnification from about 28° at x 20 to 17' at x 1000 and 1.7' at x 10,000. (all values assume a constant object distance of 10mm). The effective perspective centre is situated at the final crossover of the scan. Some specimen stages have up to three separate stations the associated rotation matrices of which must be specified in the correct sequence in the normal photogrammetric way. In addition to these mechanical rotations, there may be two electronic rotations one of which can be selected at will, the other arises as a direct consequence of the rotation of the scan raster which occurs as the current in the final lens is altered to focus the electron beam on the specimen. These last two rotations are about the effective optic axis of the system. The relationships involved in electron microscopes in the symmetric parallel projection case have been given by Helmcke (1954), while Lane (1969) considered the symmetric central projection system. Tovey examined the more general situation in both the parallel (1973) and the central projection (1973b) system. Ghosh in a series of papers (e.g. 1971, 1975) has used a similar approach.

The object axes are a right handed set and located at a suitable point on the specimen surface. The Z direction is defined as being orthogonal to the mean specimen surface, while the X and Y axes lie in a plane parallel to the mean specimen surface. In the fully general case, the mean specimen surface though representing a known orientation with respect to the parent sample may not be parallel to the plane of the specimen stub. In such a case photogrammetric equations will have up to two additional rotation matrices $[L]$ and $[N]$. If the image coordinates x,y, z have an origin at the perspective centre.

$$\text{then} \begin{bmatrix} x_j \\ y_j \\ -c \end{bmatrix} = s \; [\phi_z][M][N][L] \begin{bmatrix} X_j \\ Y_j \\ Z_j \end{bmatrix} - \begin{bmatrix} U_o \\ V_o \\ W_o + {}^oW_p \end{bmatrix} \qquad - (1)$$

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where x_j, y_j, are the image coordinates of the jth point

X_j, Y_j, Z_j are the object coordinates of the jth point

M is the normal rotation matrix which may consist of up to three separate rotations.

ϕ_z is the net effect of scan raster rotation and equals the algebraic sum of the two electronic rotation

S is the scale factor

U_0, V_0 and W_0 are the distances in the microscope coordinates between the object principal point and the origin of the object axes

In the subsequent section for convenience, it is assumed that $\left[N \right]$ and $\left[L \right]$ are both unit matrices and that

$$\left[A \right] = \left[\phi_z \right]\left[M \right] = \begin{bmatrix} \alpha_{11} & \alpha_{12} & \alpha_{13} \\ \alpha_{21} & \alpha_{22} & \alpha_{23} \\ \alpha_{31} & \alpha_{32} & \alpha_{33} \end{bmatrix} \qquad - (2)$$

If the scale factor S_0 is defined as that at the principal point and if x_0 and y_0 refer to the image coordinates of the object then :-

$$\begin{bmatrix} X_j \\ Y_j \\ Z_j \end{bmatrix} = \left[B \right]^{-1} \begin{bmatrix} (x'_j - x'_0) /S'_0 \\ (y'_j - y'_0) /S'_0 \\ (y''_0 - x''_0 \left\{ \dfrac{y''_j}{x''_j} \right\}) /S''_0 \end{bmatrix} \qquad - (3)$$

where the (') and (") notations refer to distances on the first and second micrographs respectively, and the components of B are given by :-

$$\beta_{1k} = \alpha'_{1k} + \frac{x'_j}{c'} \cdot \alpha'_{3k} \qquad \left.\begin{array}{c}) \\ ) \\ ) \\ ) \end{array}\right.$$

$$\beta_{2k} = \alpha'_{2k} + \frac{y'_j}{c'} \cdot \alpha'_{3k} \qquad \left.\begin{array}{c}) \\ ) \\ ) \end{array}\right\} - (4)$$

$$\beta_{3k} = \frac{y''_j}{x''_j} \cdot \alpha''_{1k} - \alpha''_{2k} \qquad \left.\begin{array}{c}) \\ ) \\ ) \\ ) \end{array}\right.$$

where k = 1, 2 or 3

In some cases, it is possible to arrange so that the origin of the object axes is taken to coincide with the principal point. In such a case:-

$$
\begin{Bmatrix} X_j \\ Y_j \\ Z_j \end{Bmatrix} = \dfrac{\left[B \right]^{-1}}{S'_o} \begin{Bmatrix} x'_j \\ y'_j \\ o \end{Bmatrix} \qquad - (5)
$$

which, it should be noted, does not require a knowledge of the magnification of the second micrograph.

The extent of the parallel projection approximation can be judged from the effects on the coefficients of the matrix B. For the parallel projection case,

$$
\begin{aligned}
\beta_{1k} &= \alpha'_{1k} \\
\beta_{2k} &= \alpha'_{1k}
\end{aligned} \right\} \quad \text{for } k = 1,2,3.
$$

Fig. 1 illustrates the magnitudes of the errors which occur when this approximation is used. They represent the worst situation near one of the corners of the micrograph.

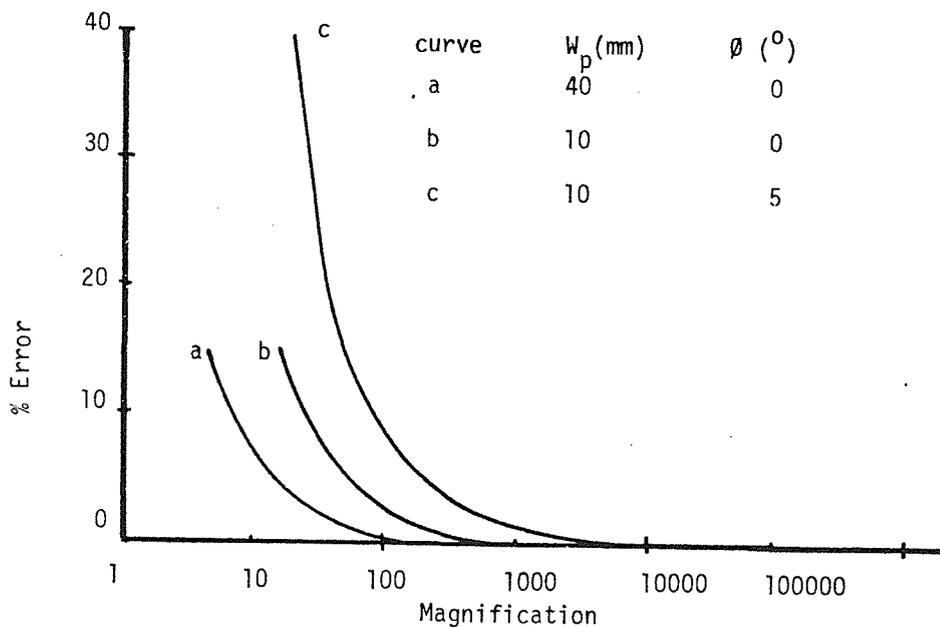| curve	W_p (mm)	$\emptyset$ (°)
a	40	0
b	10	0
c	10	5

Fig.1 Errors arising from use of parallel projection approximation

In soil fabric analysis it is often necessary to relate orientation directions to those in the field, or testing apparatus and this is achieved by defining an additional right handed set of axes Q, R, and S. These are labelled the original axes and can be set parallel to principal stress directions σ_1, σ_2, σ_3, or the terrestial coordinates such as Easting, Northing, and Heighting. Up to three further matrices may be needed to specify the relationships between Q, R and S, and X,Y and Z. If the overall resultant matrix is $\begin{bmatrix} T \end{bmatrix}$ then

$$
\begin{bmatrix} Q_j \\ R_j \\ S_j \end{bmatrix} = \begin{bmatrix} T \end{bmatrix}^{-1} \begin{bmatrix} B \\ S_o \end{bmatrix}^{-1} \begin{Bmatrix} x'_j \\ y'_j \\ 0 \end{Bmatrix} \qquad - \quad (6)
$$

In many instances, the matrix $\begin{bmatrix} T \end{bmatrix}$ will be of simple form e.g. for examples from a oedometer test, a vertical surface would be represented by $\begin{bmatrix} T \end{bmatrix}$ being a unit matrix, while for a horizontal surface,

$$
T = \begin{bmatrix} 0 & 0 & -1 \\ 0 & 1 & 0 \\ 1 & 0 & 0 \end{bmatrix} \qquad - \quad (7)
$$

Note for orientation analyses it is unnessary to specify any translation in Q, R, S space between the object and original origins.

A minimum of three points on each particle are needed to enable its spatial orientation to be determined. These can be used to define a plane in Q,R, S space:-

$$
S = aQ + bS \qquad - \quad (8)
$$

The spatial orientation can then be specified by two angles: η is equivalent to 'dip' and X is equivalent to 'strike' in the geological sense. The orientation of up to 500 particles on a pair of micrographs has been measured (e.g. Tovey 1970, 1971, Tovey 1978). In this analysis each particle is represented by its normal vector, and the concentrations of these in 3-D space can be projected onto a Schmidt equal area net to give a pole diagram. Alternatively the data can be used in a least squares analysis to obtain a solid of anisotropy which in most cases approximates to an ellipsoid, the direction of the major axis of which can be related to the direction of the Q-axis. In addition, an index of anisotropy (I_A) can be obtained from the ratios of the lengths of the principal axes. In many analyses, the ellipsoids have two axes of almost identical magnitude. In this case I_A will be the ratio of the major axis the mean of the other two.

Tovey (1970) gave examples of two such analyses finding that I_A was 4.29 and 1.61 in a consolidated and unconsolidated sample respectively.

In the above discussion no mention has been made of distortions inherent in the instrument. Tovey (1973) illustrated the extent of the distortions while Murray and Windle (1973), Ghosh (1975) and Ghosh and Nagaraja (1976) developed methods for calibration using sets of polynomial equations.

THE OPTICAL DIFFRACTION METHOD

In a typical optical diffractometer (Fig.2), a transparency of the micrograph is illuminated by a parallel coherent source of light. A lens placed behind the transparency enables the optical (fourier) transform of the object to be recorded in its back focal plane. In some instances it is desirable to use an additional lens to magnify
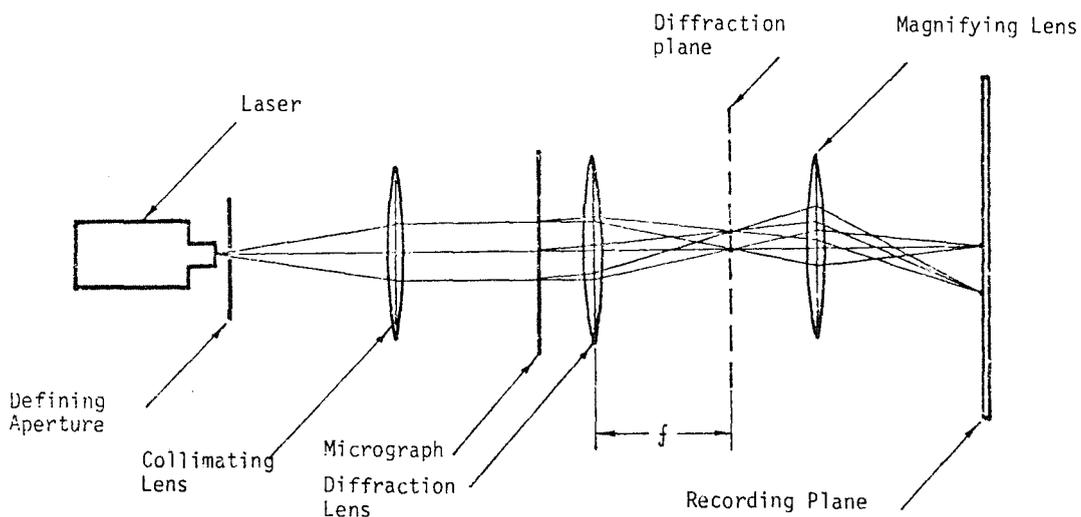
Fig. 2 Optical Diffractometer

the pattern, however, this can increase the allignment problems of the instrument. Periodicities or orientation patterns in the micrograph will be clearly evident in the diffraction pattern. For a series of regularly spaced particles of similar shape, there will be a series of diffraction spots, the spacing (r) of which will be related to the mean distance (D) between features on the specimen.

$$r = \frac{m \lambda f}{2SD}$$

where λ is the wavelength of the light used, f is the focal length of the diffracting lens.

S is the scale of the transparency
and M is the enlargement provided by the auxiliary lens
Thus r ≃ 0.1m/SD for the instrument used by the present authors. It is important that the value of S be carefully selected. A typical scanning electron micrograph has 1000 line resolution, or 0.025 mm on a

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25mm x 25mm transparency. Thus S must be greater than X125 if the thickness of a typical clay particle ($\sim$ 0.2μm) is to be resolved. If the transparency contains a series of edge on particles of this size equispaced by 0.2μm, the first order reflection would appear at 4mm (if m = 1) from the centre and could thus be clearly distinguished from the undiffracted beam. On the other hand, if the particles are face-on, their associated size ($\sim$ 1μm) would yield a pattern with the first order peak at 0.8μm (m = 1) and would probably be lost in area of the photographic emulsion which becomes over exposed as a result of the undiffracted beam. Increasing the value of m, or decreasing the value of S will overcome this problem. For studies of this kind it has been convenient to adopt a standard magnification of about X500 (x125 on the transparency) and set m in the range 2-4. For each setting of m, the instrument must be carefully calibrated, and this is achieved by obtaining the diffraction pattern of a 3mm diameter grid which has lines spaced at 0.125 mm intervals.

For soil fabrics which have a high degree of regularity, the diffraction maxima appear as a series of discrete regularly spaced spots, from which it is possible to estimate the mean spacing of features more reliably, and more rapidly than can be done by conventional techniques. With fabrics of less regularity, the patterns become diffuse with the individual spots merging with one another. Since the spacings of the spots have an inverse relationship with the features in the micrograph, the patterns in general will be elliptical in shape, the major axis of which will lie perpendicular to the mean alignment direction of features in the micrograph. Further the shape of this pattern can be used as an index of anistropy. Some typical examples of soils from the Soviet Union are shown in Figs. 3 and 4.

Fig. 3a is a typical domain micro fabric in kaolin showing randomly orientated domains of particles. A selection of the diffraction patterns from certain small areas of Fig.3 are illustrated in Figs. 3b-3e together with a pattern from a large area of the micrograph. The first pattern clearly shows that the overall effect of the fabric as a whole is nearly isotropic. Compared to this the selected area patterns are distinctly anisotropic. Much information can be obtained by a detailed examination of these patterns. For instance, the spacing between maxima in Fig. 3e. correspond to mean spacings of 0.36μm, 0.18μm, 0.11μm and 0.08μm in the original specimen. Some of the reflections appear as arcs of a circle and this indicates that though the associated features are equi-spaced, their orientation varies. The magnitude of this change in orientation can be judged from the length of the arc. Since the diffraction patterns can be recorded rapidly it is possible to study the effects on anistropy of increasing the area under observation. Thus the shape of the pattern becomes more circular as the size increases although even in Fig.3b the distribution is still slightly elongate.

Diffraction patterns of large areas (e.g. Fig. 5a) of a lower magnification micrograph of the same soil (Fig. 4a) are similar even though those from small areas show some variability. The shape of the patterns such as Fig. 5a vary only slightly as the area under observation is changed, and this size of area is thus the smallest in which the fabric is representative of the whole sample. This particular sample has a similar pattern when an orthogonal surface is observed (e.g. Figs. 4b and 5b). This contrast strongly with the fabric of clay slate shown in Figs. 4c and d where a highly anistropic fabric is seen in the
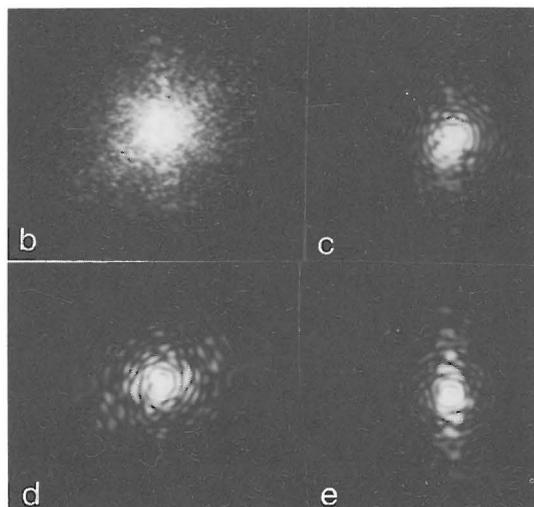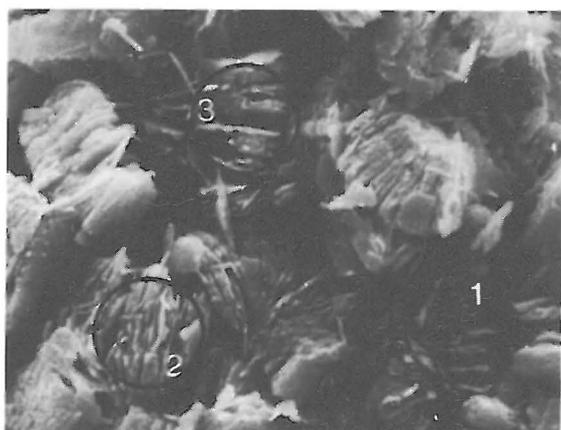
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Fig.3 (a) domain microfabric: picture width = 17μm; (b) optical diffraction pattern of whole of (a); (c)-(e) selected area patterns.
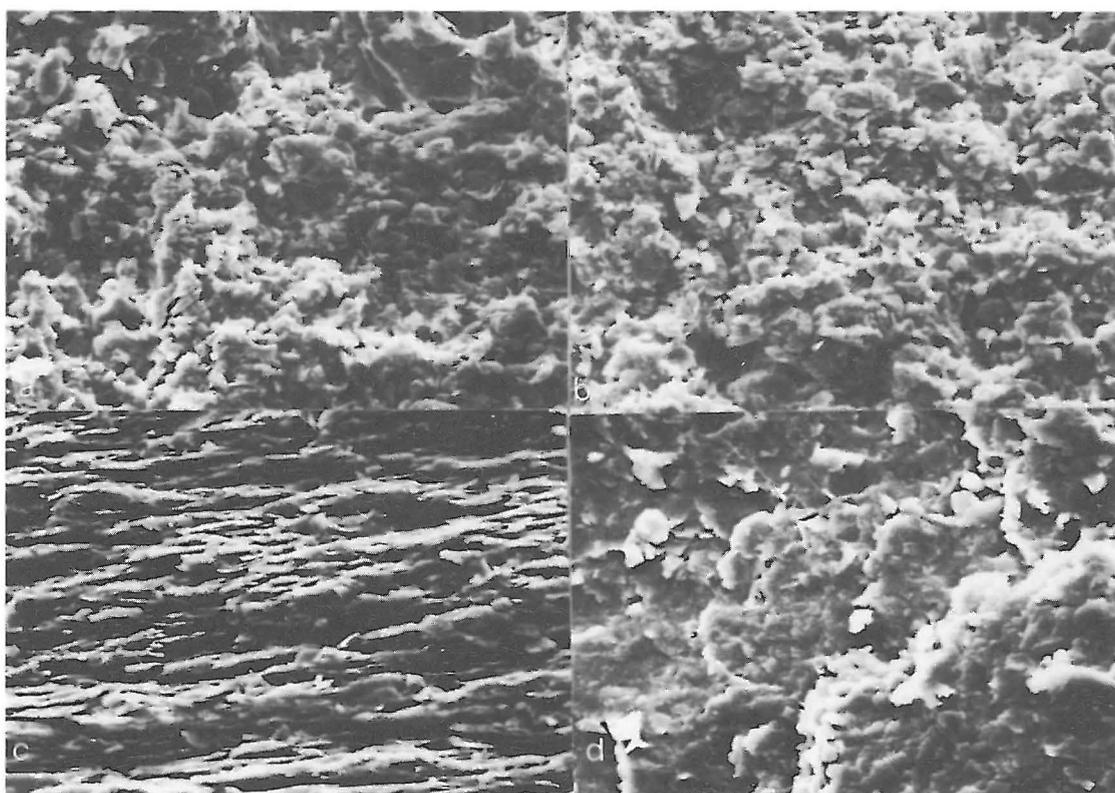

Fig.4. (a) & (b) domain microfabric; (c) & (d) clay slate: (a) & (c) perpendicular, (b) & (d) parallel to bedding plane: picture widths 180μm.
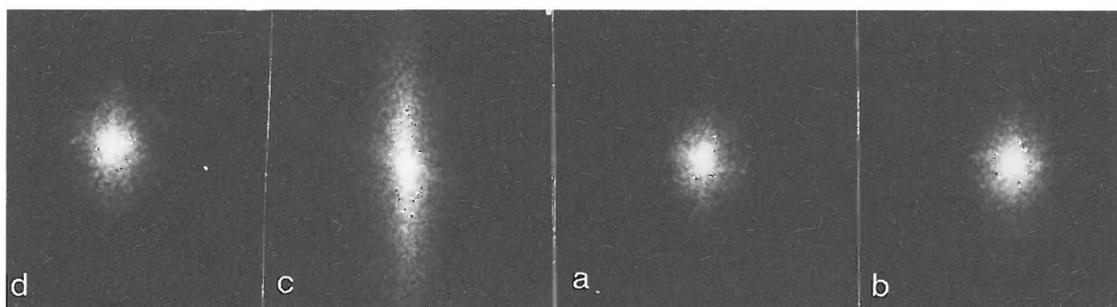

Fig.5 diffraction patterns of micrographs in Fig.4.

vertical section, but a nearly isotropic fabric is evident in the ortho-gonal section. The shapes of the diffraction patterns clearly indicate the extent of the anistropy.

CONCLUSIONS

Conventional photogrammetric techniques can be used to assess the detailed spatial orientation of soil particles. However, such a detailed study is of no value if the area selected for study is unrepresentative of the whole sample. To assist in this optical diffraction methods can be used to select representative areas, to check against micro fabric distortion during preparation, and used to assess mean spacings between particles.

ACKNOWLEDGEMENTS

REFERENCES

BARDEN L., and SIDES G.R., (1971) Geotechnique Vol 21, p211-222.
BENNETT R.H., BRYANT W.R., KELLER G.H., (1978) Nat. Oceanic and
    Atmospheric Admin. Professional Paper No 9.
FOSTER R.H., and EVANS J.S. (1971) The Microscope Vol 19pp377-401.
GHOSH S.K. (1971) Photogrammetric Engineering Vol. 37 p187-191.
GHOSH S.K. (1975) Photogrammetric Vol. 30 pp91-114.
GHOSH S.K., and NAGARAJA H.N. (1976) Photo. Eng. Rem. Sens., Vol 42
    No 5 pp649-657.
HELMCKE J.G. (1954) Optik Vol.2 pp201-225.
LANE G.S. (1969) Jour. Sci. Instr. (Jour, Phys E) Series 2 pp565-569.
MURRAY S., and WINDLE A.H. (1973) SEM Systems and Applications. Inst.
    Phys., London p88-93.
OSIPOV V.I., and SOKOLOV V.N. (1978) Bulletin. Int. Assoc. Engineering
    Geology No.18 pp73-81.
SERGEEV Y.M., GRABOWSKA-OLSZEWSKA B., OSIPOV V.I., and SOKOLOV V.N.
    (1978) Proc. III Int. Conf of Eng. Geology, Madrid.
SOKOLOV V.N., OSIPOV V.I., TOLCKACHEV M.D., RUMIAWZEVA N.A., and ROMM
    E.S. (1979). U.S. Patent No 4146788.
SOKOLOV V.N., OSIPOV V.I., and TOLCKACHEV M.D. (1980) to be published
    in Jour. Microscopy.
TOVEY N.K. (1970) PhD Thesis, Cambridge University.
TOVEY N.K. (1973a) Proc. Int.Symp. Soil Structure, Gothenburg. pp50-57.
TOVEY N.K. (1973b) SEM Systems and Applications. Inst. Phys   London
    pp82-87.
TOVEY N.K. (1978) SEM in the study of Sediments, Ed. Whalley, Geo-abstracts pp105-117.
TOVEY N.K. (1980) to be published in Jour. Microscopy.
TOVEY N.K. and WONG K.Y., (1973) Proc. Int. Symp. Soil Structure,
    Gothenburg. pp58-66.
TOVEY N.K., and WONG K.Y., (1968) SEM in the Study of Sediments, Ed.
    Whalley, Geoabstracts pp181-199.
WONG K.Y., and TOVEY N.K.(1975) Geotechnique Vol25 pp42-45.