STATENS GEOTEKNISKA INSTITUT

SWEDISH GEOTECHNICAL INSTITUTE

No. 15

SÄRTRYCK OCH PRELIMINÄRA RAPPORTER

REPRINTS AND PRELIMINARY REPORTS

Supplement to the ''Proceedings'' and ''Meddelanden'' of the Institute

Investigation of Clay Microstructure by Using Ultra-Thin Sections

by Roland Pusch

STOCKHOLM 1966

INVESTIGATION OF CLAY MICROSTRUCTURE BY USING ULTRA-THIN SECTIONS

by

Roland Pusch

Swedish Geotechnical Institute, Stockholm

Stockholm 1966

INTRODUCTION

Clay microstructure, orientation and aggregation of silt size particles and large clay particles are generally studied by using thin section techniques and petrographic microscopy. A large number of methods for saturating the clay with a suitable embedding substance have been used for preparation of thin sections by grinding. The most well-known procedures have been suggested by KUBIËNA (1938), MITCHELL (1956), BREWER and HALDANE (1957) and ALTEMÜLLER (1962). MITCHELL used a melted polyethylene glycol compound (Carbowax) which can be used not only for saturating dried clay specimens but also for replacement of pore water without any large change of the microstructure. Detailed microscopical studies of thin sections (40 μ) by using polarized light have indicated characteristic microstructural features such as particle alignment. The thin sections used by MITCHELL were prepared by grinding, which causes considerable variation in the thickness of the specimen. An investigation by PUSCH (1964) based on MITCHELL's technique has shown that especially useful information about the microstructure can be obtained from thin sections (10 μ) which have been cut by a precision microtome (LKB). Such sections have an almost uniform thickness. MITCHELL's preparation method has also been used in a series of X-ray diffraction measurements for identification of particle parallelism.

It should be mentioned that ROSENQVIST was one of the first to study thin microtome-cut sections of clay (ROSENQVIST, 1955). He suggested several procedures for clay specimen preparation and he was able to draw certain conclusions concerning clay microstructure from his micrographs.

A detailed study of the arrangement of clay size particles cannot be made without the aid of an electron microscope. Such investigations of clay microstructure, using replicas of fractured surfaces of freeze--dried clay specimens, have been made frequently in the last decade. One of the most well-known studies of this kind was published by ROSENQVIST in 1958. Electron micrographs of such replicas, although taken stereoscopically, give only an impression of the "topographic" variation of the fractured surface.

In 1963 when research work was initiated at the Swedish Geotechnical Institute on the relationship between the microstructure and the mechanical properties of clays, a suitable method for microstructural investigation was needed. It was found necessary to obtain information on the detailed particle arrangement, which presupposed the application of electron microscopy. None of the ordinary embedding substances could be used because the mechanical strength of the treated clay was inadequate to make possible a preparation of sufficiently thin specimens. In cooperation with Professor G. Glimstedt, Department of Histologi, University of Lund, a method used on organic tissues was tried and found applicable. By this method the pore water of small samples of undisturbed clay is replaced by acrylate plastic through a diffusion process. The technique has been refined and it is now possible to cut sections as thin as about 300 Å for investigation in the electron microscope. This technique is described in the following.

CLAY PREPARATION

1) A prismatic specimen with a base area of approximately 1/4 - 1/2 cm^2 and a length of approximately 1 cm is cut from a clay sample with a thin steel wire or, in the case of hard clay, with a sharp knife. The clay sample should be cut in such a way that the orientation of the individual clay layers in situ can be defined.

2) The sample is stored in 50 % ethyl alcohol for 30 min and in 70 % alcohol for 5 min und thereafter in 90 % alcohol and finally in 99.5 % alcohol for 5 min each.

3) The sample is stored in a monomer consisting of 85 % butyl methacrylate and 15 % methyl methacrylate for 45 min. This process is repeated once.

4) The sample is stored for 90 min in a solution consisting of 98 % monomer and 2 % 2.4-dichlorbenzoylperoxide (EWM) catalyst. Thereupon the sample is placed in a gelatine capsule (Parke, Davis & Co. No. 00) which is filled with monomer and catalyst.

5) After polymerization by storing the sample for 15 hours in an oven at 60° C the gelatine capsule is washed off with water. The sample can then be trimmed and cut. The trimming is done in such a way that the original orientation of the thin sections obtained in the microtome can be defined.

After trimming the plastic treated specimens, they are cut by using a precision microtome, for instance the LKB Ultrotome 4801 A/4802 A, equipped with a diamond knife. The liquid in its collecting trough should be a 10 % acetone solution. The sections are picked up and placed on carbon coated 150 - 400 mesh grids.

Almost all the sections from pure clay can be used by this procedure. When the diamond edge meets coarser particles they may be pushed up in front of the edge and cause a disturbance. The sections which appear to be intact can be selected by observing the cutting process in the eye-piece of the microtome.

ELECTRON MICROGRAPHS, PREPARATION AND INTERPRETATION

At present a series of different clays has been investigated. These clays have a negligible content of expanding clay minerals. The possible influence of volume change on the microstructure by the plastic preparation has thus been very small.

Three different major types of soft clays have been used, viz., fresh- and brackish-water clays from Skå-Edeby, organic brackish-water clays from Morjärv and salt (marine) quick and normally sensitive clays ($S_t \approx 10$) from Lilla Edet. Samples from the stiff Silurian clay in the Burgsvik sandstone (Gotland) have likewise been studied, besides a series of clayish moraines from Skåne. At present the research work is concerned with remoulded clays under thixotropic strength regain and with structural changes in clay samples in laboratory tests.

A Siemens Elmiskop I was used for the preparation of micrographs. This microscope is equipped with a platinum-iridium aperture of 50 μ in the objective lens. The voltage was 80 kV and the electronic magnification 5500 x or 11000 x, depending on the microstructural character.

Some photographic examples are shown in Figs. 1, 2 and 3. The dark details in the micrographs represent the solid phase while the bright fields represent the pore system. Mechanical disturbances can be identified adjacent to certain bright spots.

Fig. 1 indicates the typical tendency of particles in fresh-water clays to be dispersed. Figs. 2 and 3 show characteristic flocculated structures which are formed under more saline conditions. A full report on these clays will be available within a year.

In connection with the development of the technique for direct illustration of clay microstructure, methods for statistical and mathematical description of structural features have been worked out. The following three procedures have been used here:

1. Calculation and description by statistical means of preferred particle orientation. This procedure is based on a method suggested by DAPPLES and ROMINGER (1945).

2. Calculation of median values, skewness numbers and sorting coefficients of the maximum diameters of the sectioned micropores.

3. Calculation of the two-dimensional "porosity" expressed by the ratio between pore area and total sectioned area.

In several instances the calculated parameters have been found to be characteristic of the clay in question. Also, in some cases, a relationship has been found between parameter values and mechanical properties of the natural clay in bulk.

ACCURACY

The most important question respecting the practical applicability of the preparation technique is concerned with its influence on the original microstructure.

For testing of the acrylate technique, two other plastic substances were used, Epon epoxy resin (Shell Co.) and Durcupan (Fluka AG). Only very small differences could be found when comparing the statistical parameters of the interpreted micrographs.

Precision measurement of the dimensions of the clay specimens during the different steps in the acrylate plastic preparation should indicate an influence on the microstructure. Since the linear change in dimensions did not exceed 3 %, whereas the untreated clays showed a linear shrinkage of up to 23 % when dried, large microstructural change had probably not taken place.

Thanks to Professor Glimstedt the author was also able to study sections of clay in a high-voltage electron microscope at the Laboratoire d' Optique Electronique du Centre National de la Recherche Scientifique,

Toulouse, France. In this microscope, which operates at a potential of up to 1.5 MV, the penetrability of the electron radiation is much greater than in ordinary electron microscopes. This makes it possible to study comparatively thick specimens as well as specimens enclosed in a cell. Theoretically, 5-20 μ thick slices of clay with the natural water content can be investigated in this microscope. Unfortunately, specimens with a thickness of this magnitude cannot generally be cut without severe disturbance. Soft clay, for instance, cannot be investigated for this reason but thin slices peeled from a laminated stiff clay may be studied. Specimens of moist stiff Silurian clay were investigated and the preliminary results from the interpretation of the micrographs indicate a close similarity between the natural and the plastic treated clay.

Although there is no definite proof that the acrylate preparation does not influence the original microstructure, the tests indicate that it is preserved.

A full report on the tests including a discussion of the microtome operation will be made separately.

REFERENCES

ALTEMÜLLER, H., 1962. Verbesserung der Einbettungs- und Schleiftechnik bei der Herstellung von Bodendünnschliffen mit Vestopal. Zeitschr. Planzenernährung, Düngung, Bodenkunde. Verl. Chemie, Weinheim.

BREWER, R. and HALDANE, A.D., 1957. Preliminary experiments in the development of clay orientation in soils. Soil Sci. 84, p. 301-309.

DAPPLES, E.C. and ROMINGER, J.F., 1945. Orientation analysis of fine-grained elastic sediments. Journal of Geology. Vol. LIII, No. 1.

KUBIËNA, W.L., 1938. Micropedology. Collegiate Press. Inc. Ames, Iowa.

MITCHELL, J.K., 1956. The fabric of natural clays and its relation to engineering properties. Highw. Res. Board Proc. Vol. 35, p. 693-713.

PUSCH, R., 1964. On the structure of clay sediments. Stat. Råd för Byggn.forskn. Handl. Nr 48.

ROSENQVIST, I.Th., 1955. Investigations in the clay-electrolyte-water system. Norg. Geot. Inst. Publ. Nr 9.

ROSENQVIST, I.Th., 1958. Remarks to the mechanical properties of soil water systems. Geol. Fören. Stockholm Förh. Bd. 80. Nr 4, p. 435-457.

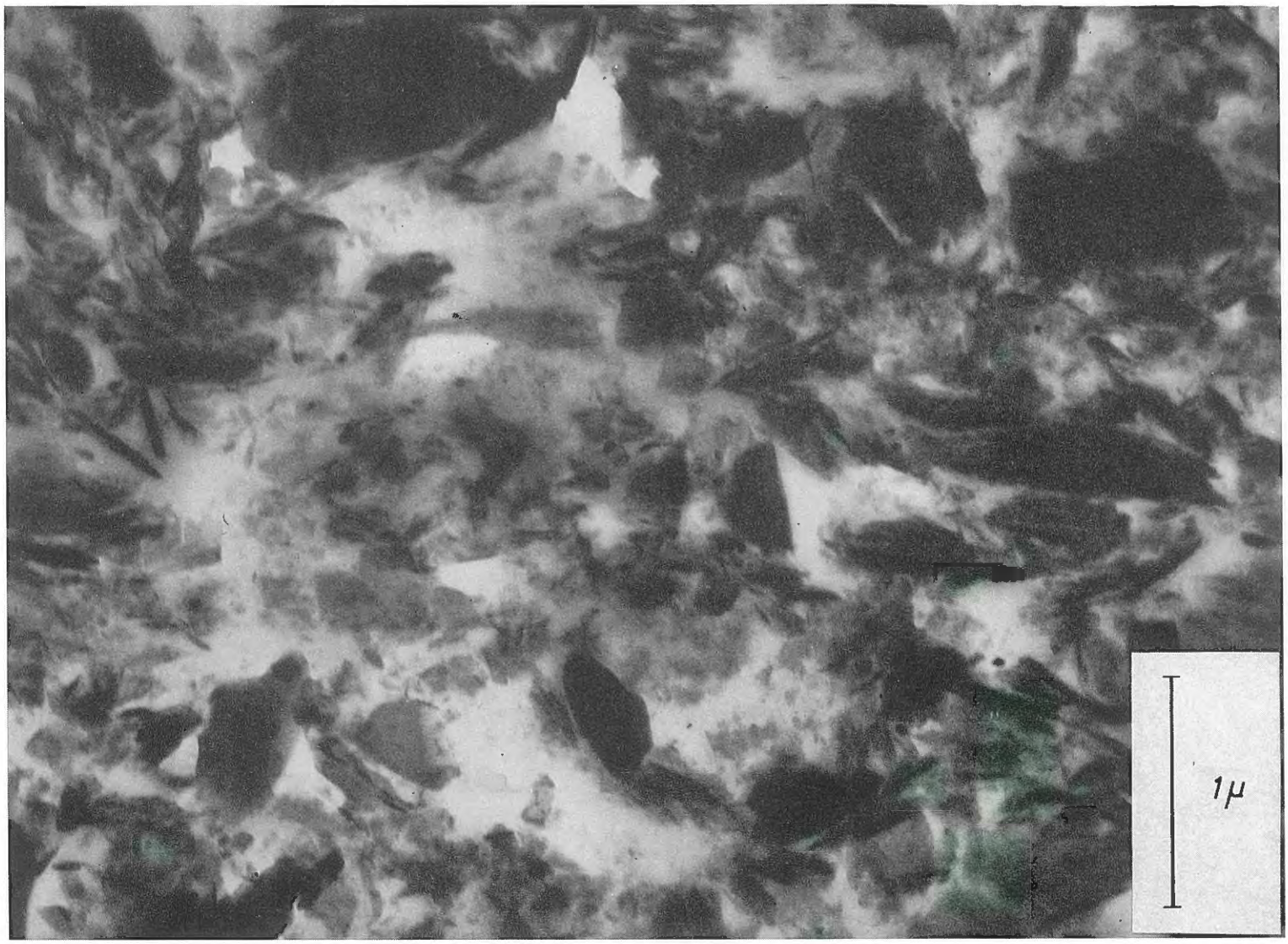

Fig. 1. Fresh-water clay from Skå-Edeby. Sampling depth 10 m.
Electronic magnification 10500x.

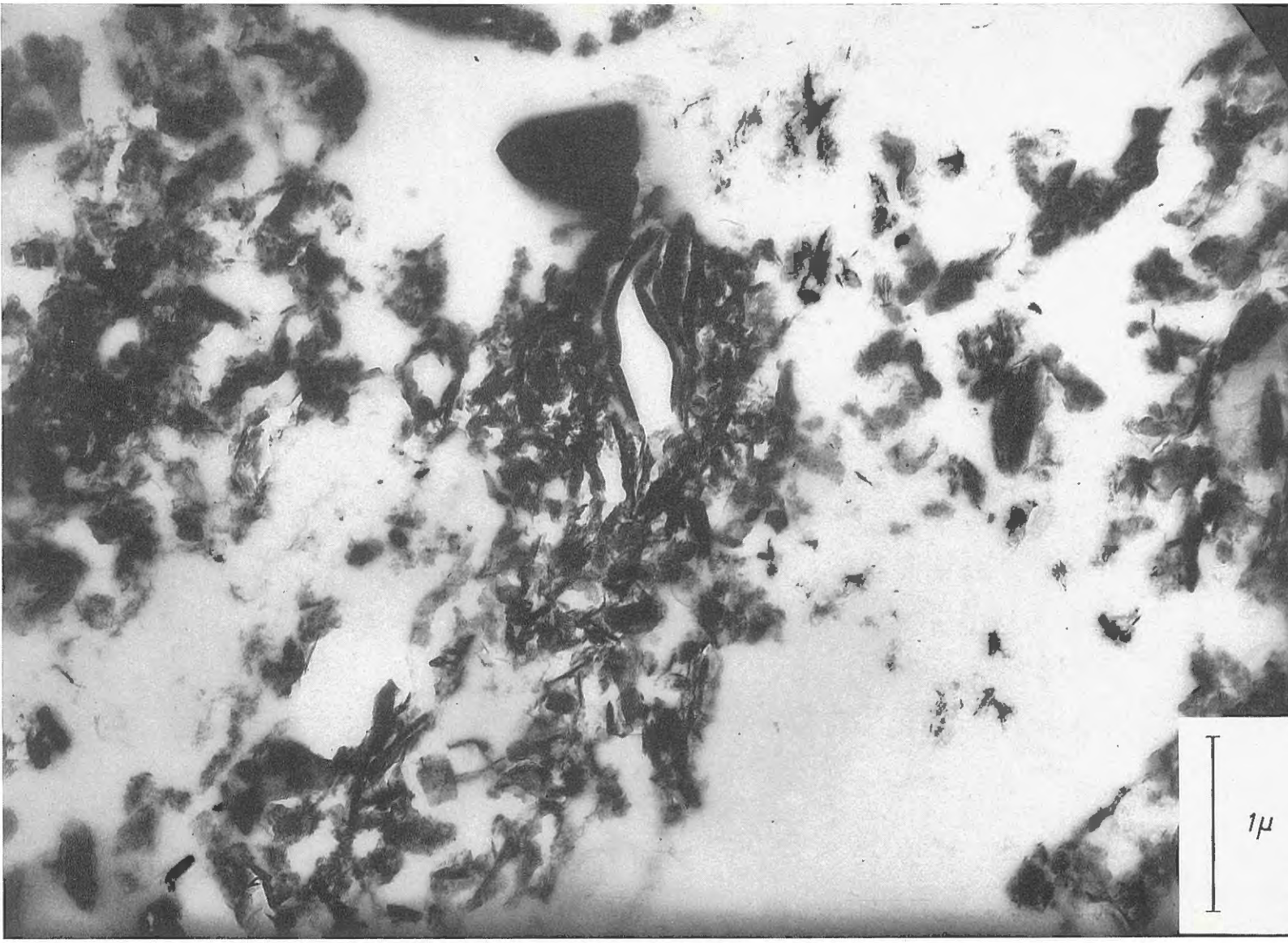

Fig. 2. Brackish-water, organic clay from Morjärv. Sampling depth 4 m.
Electronic magnification 11000x.

Fig. 3. Marine, quick clay from Lilla Edet. Sampling depth 3 m.
Electronic magnification 10000x.