# High voltage electron microscopy of quartz particles from post-glacial clay soils

C. HAMMOND Department of Metallurgy, The University of Leeds
C. F. MOON Department of Civil Engineering, Sheffield Polytechnic
I. J. SMALLEY Department of Civil Engineering, The University of Leeds

Quartz particles from the Norwegian Lodalen clay and the Canadian St Jean Vianney clay have been examined by electron microscopy. The high-voltage electron microscope in particular allows the internal structure and the surface nature to be examined. Dislocation networks have been observed in the quartz particles and also Moiré patterns suggesting regions of structural deformation. The particles have a plate-like shape with a habit plane parallel to the basal plane which suggests that at small particle sizes ( $\sim$ 3 µm) a cleavage mechanism may operate in quartz. The edges of the particles appear to be heavily deformed and cracking fragmentation is apparent which may correspond to the deformed lattice region of Lidstrom. The observations cast some light on the origin of the sensitivity of the strength of such clays to disturbance.

## 1. Introduction

In a recent study of the mineralogy of the Leda clay of eastern Canada, Gillott [1] stated that "The clay-size fractions contain a significant proportion of primary minerals. This affects the engineering properties of clay soils because primary minerals have small specific surface area and weaker colloid chemical properties than clay minerals". It has been suggested [2] that the primary minerals (in particular quartz) which are present in the post-glacial clays of Canada and Scandinavia provide the major reason for the strange properties which many of these clays possess. The purpose of this paper is to report investigations carried out on two particular clay soils with a view to discovering the nature of the quartz particles which comprise such a large proportion of the material of such soils.

The soil samples used both come from landslide sites, one in Norway, at Lodalen near Oslo and one in Canada, at St Jean Vianney, Quebec. The Lodalen clay soil is a firm, comparatively homogeneous marine clay with some thin silt layers [3]. The sensitivity of the clay (the ratio of undisturbed to disturbed strength) varies between 3 and 15; this is a fairly low sensitivity for such a material; some post-glacial clays (the socalled "quickclays") have sensitivities greater than 200. Mineralogical investigations showed that the clay-size fractions ( $\sim 2 \mu m$ ) which, amounted to 30 to 50% of the clay, consisted largely of illite.

The Canadian soil sample is a Leda clay from St Jean Vianney in Quebec, [4]. Gillott's study of Leda clay mineralogy showed that Quebec clays had a high proportion of quartz and plagioclase [1] and Penner [5, 6] has demonstrated the very high sensitivities which occur. The problem to which observations of the nature of this type of clay soil material are directed is that of the cause of the sensitivity. The factor which investigators seek to isolate is that which allows a strong, brittle, load-bearing material to turn very rapidly into a viscous liquid with no real strength. This causes the catastrophic landslips which present a continuing geotechnical hazard in northern North America and in Scandinavia. Problems and theories have been reviewed by Kerr [7], Crawford [8], Soderblom [9], Rosenqvist [10], Osterman [11] and Mitchell and Houston [12].

# 2. The role of quartz particles in sensitive clay soils

The only substantial current theory to explain the sensitivity of quickclays and similar soils is that of Rosenqvist [10, 13] who proposed that the typical quickclay properties were produced by post-depositional leaching. The basic theory has been considerably developed since it was first proposed but it has been criticized (cf. Pusch and Arnold [14]) and does not explain certain observations on Canadian quickclays [5, 6].

Rosenqvist, and most other investigators, have considered quickclays to be real clay soils and have expected the clay mineral content to determine the failure mechanism. Analyses of landslide soils [1, 15, 16] show that clay minerals need not be present in significant amounts and suggest that other soil particles play the key role in determining quickclay properties. If a supply of fine non-clay mineral soil material is a requirement for quickclay formation then the geographical distribution is explicable. The abundant fine material formed by glacial action is incorporated into the quickclays.

In very simple terms there are essentially two types of interparticle bond in soils, long-range active bonds between clay-mineral particles and short-range inactive bonds between non-clay mineral particles, e.g. quartz. Thus if this approach to a quickclay system is valid, the quartz (and similar) particles have a critical role to play in determining properties. The behaviour of the quartz particles in the soil environment will depend largely on their nature.

The quartz particles in quickclays and other sensitive soils fall largely within the size range 1 to 5  $\mu$ m. Thus they are similar to clay mineral particles in at least one respect but little is known of their shape, their internal state and the nature of the critical surface regions. Their small size makes them particularly suitable for examination by high-voltage electron microscopy where the increased penetration and resolving power in thick sections compared with conventional 100 kV microscopy makes possible the investigations of internal structures.

# 3. Results

# 3.1. X-ray powder analysis

Dried samples were investigated using a diffractometer and Mo  $K\alpha$  radiation. The results for the Lodalen clay agree with those of Sevaldson [3] and show a predominance for quartz with some illite present; there also appears to be a small amount of plagioclase. In the Leda clay there was a clear indication of the presence of plagioclase feldspar particles. Gillott [1] found a considerable proportion of plagioclase in a Quebec clay from St Joachim de Tourell and the St Jean Vianney clay appears to be similar. Quartz predominates and some illite appears to be present.

# 3.2. Electron microscopy

Dried samples were dusted on to a carbon film mounted on a grid. These were examined using both a 100 kV instrument and a 1000 kV microscope (the AEI EM7 instrument at Swinden Laboratories, Rotherham). The quartz particles may be distinguished from the clay minerals (a) by their shape (b) their diffraction patterns and (c) their stability under the electron beam, particularly at 100 kV. The (illite) clay particles occur as hexagonal shaped flakes  $0.5 \sim 4 \ \mu m$ across and in stacks ~0.5  $\mu m$  thick. Essentially single crystal electron diffraction patterns were obtained, indicating epitaxiality of the individual crystals.

Tilting experiments show that the quartz particles also occur as plate-shaped particles of the same order of size and thickness as the illite aggregates. However, the surfaces and edges are very irregular. In general the diffraction patterns indicate a habit plane approximately parallel to the basal plane. The internal structure of the quartz is best observed in the 1000 kV instrument where the improved resolution at greater thicknesses allows the internal features to be examined. In addition, dark field micrographs give a better contrast over bright field owing to a reduction in the number of inelastically scattered electrons entering the objective aperture.

The internal substructure of the quartz consists of a random network of dislocations, there being a greater number of dislocations at the irregular edges (Fig. 1); dislocations forming a low angle subgrain boundary network and regions showing Moiré fringe contrast (Fig. 2). These features suggest that the quartz particles occurred as a result of severe mechanical deformation. The fine mottled background and dislocation decoration arises from beam damage, an effect particularly marked in (hydrated) quartz from a wet environment. The Moiré fringe contrast may arise as a result of Brazil twinning or from the collapse of dislocations in the sub-boundaries (Fig. 2). The plate-like morphology, approximately parallel to the



*Figure 1* Quartz particle (Lodalen clay) showing random network of dislocations. (Dark field from  $(10\overline{1}0)$  reflection.)



*Figure 2* Quartz particle (Lodalen clay) showing subgrain boundary network. (Dark field from  $(10\overline{1}0)$  reflection.)

(0001) plane of the quartz suggests that a cleavage mechanism may be operative at these very fine particle sizes. Similar features have also been observed in the quartz particles of an artificially ground pegmatite (Fig. 3) where Moiré fringes and apparent cleavage facets may be observed.

The quartz particles, like the clay minerals, sometimes occur in stacks, the crystals in this case not being epitaxially related. Quartz/clay mineral aggregates also occur which complicates the analysis. Furthermore, a large proportion of the quartz particles (particularly those in the St Jean Vianney clay) appear to have an amorphous surface layer which obscures the microstructures. (Fig. 4 a to d). These features may correspond to silica deposition from groundwater (Fig. 5).



*Figure 3* Quartz particle (crushed pegmatite) showing Moiré fringes. (Bright field.)

# 4. Discussion

#### 4.1. Observations

The most interesting observation is that the fine quartz exists as plate-shaped particles. This suggests that some cleavage mechanism operates which is not observed on a macroscopic scale. Although quartz has no pronounced cleavage, observation with a scanning microscope on crushed quartz and loess particles led Krinsley (private communication) to propose that a cleavage mechanism may operate below a certain critical size and this does indeed appear to be the case. The dislocation networks suggest that plastic deformation has occurred, particularly at the edges, where apparent cracking of the particles is discernible. The depth of this distortion is of the order  $\sim 1000$  to 2000 Å and could correspond to the distorted lattice layer described by Lidström [17]. The occurrence of dislocation subgrain-boundaries indicates a recovery process which has occurred in the 10000 vear interval between formation and examination and supports Lidström's suggestion that in soil particles many of the defects introduced during the formation process have been eliminated. In addition, it is possible that a process of silica solution and re-deposition has occurred.

#### 4.2. Formation of quartz particles

Quartz tends to arrive in the sedimentary systems as particles broken out of granitic rocks by various weathering agencies, aided and abetted by certain internal factors [18-20]. The shape and size of the particles is largely determined by the formation of the quartz crystals in the molten rock system. The bulk of smaller sedimentary quartz particles tend to be formed



Figure 4 Quartz particle (St Jean Vianney clay) (a) bright field, (b) diffraction pattern, (0001) zone, (c) dark field from  $(11\overline{2}0)$  reflection, (d) dark field from  $(10\overline{1}0)$  reflection.

from the sand particles by breakage. Since quartz is a hard resistant mineral, considerable energies are required to produce small quartz particles. Glacial grinding is the most effective and produces the significant amounts of siltsized quartz required for the major loess deposits [12, 24].

Although the formation of silt-sized quartz particles (as found in loess) has been fairly thoroughly studied, the production of sub-silt sizes has been neglected. Until recently, with the development of high-voltage electron microscopes, it has not been possible to obtain a really satisfactory view of the particles of clay size, and thereabouts, and their importance has not been realized. Much of the very fine quartz detritus formed by glacial grinding has been incorporated into post-glacial clay sediments and appears to play a significant role in determining the material properties. It has been assumed that the shape of the very fine particles was much the same as that of silt-sized particles but it appears that when the particle size is very small some cleavage mechanism operates in quartz which allows plate-shaped particles to form.

Using simple probability theory it is possible to calculate the percentage of various shapes which might be expected when a relatively isotropic rock material like quartz is broken by some crushing action [25]. A large departure from the calculated distribution suggests that a cleavage mechanism operates.

Other factors are probably involved since artificially ground quartz does not appear to deform in quite such an extremely non-random fashion. Krinsley and Margolis [26] observed an "upturned plate" structure on the surface of sand grains and it is possible that this is a sign of incipient cleavage which could produce platelet particles (see also Krinsley and Smalley [20]). It is possible that basal plane cleavage may be related to Dauphiné-twinning (rotation of 180° about the *c*-axis) but there is no direct evidence for this.



*Figure 5* Quartz particle (St Jean Vianney) showing small  $(0.1 \ \mu m)$  regions of possible silica deposition. (Dark field from  $(30\overline{3}0)$  reflection.)

## 5. Conclusions

The most significant result of these investigations appears to be that small quartz particles in sensitive clay soils are distinctly plate-shaped. As such they could pack together in an open "house of cards" structure as originally suggested by Rosenqvist [13] in connection with clay mineral particles. Basic structural rigidity is provided by the short-range bonding forces and this may be supplemented by silica deposition and hence cementation at the particle contacts.

This model is more in accordance with the observed irregular packing of the quartz platelets than that of clay-mineral "face-to-face" aggregates.

Although it is probable that at least some of the dislocations arose as a result of the deformation associated with glacial grinding, it is also possible that dislocations were present in the quartz from the primary igneous rocks [27]. However, a comparative study would be necessary to distinguish these possibilities.

#### Acknowledgement

We thank Professor A. N. Schofield of the University of Manchester Institute of Science and Technology and Mr L. W. Gold of the National Research Council of Canada for assistance in obtaining specimens.

#### References

- 1. J. E. GILLOTT, Canad. Mineralogist 10 (1971) 797.
- 2. I. J. SMALLEY, Nature 231 (1971) 310.
- 3. R. A. SEVALDSON, Norwegian Geotech. Inst. Publ. 24 (1957) 1.
- 4. F.TAVENAS, J-Y. CHAGNON, and P.LA. ROCHELLE, Canad. Geotechnical J. 8 (1971) 464.
- 5. E. PENNER, Nature 197 (1963) 347.
- 6. Idem, Canad. J. Earth Sci. 2 (1965) 425.
- 7. P. F. KERR, Sci. Amer. 209 No. 11 (1963) 132.
- 8. C. B. CRAWFORD, Eng. Geol. 2 (1968) 239.
- 9. R. SODERBLOM, Proc. Swedish Geotech. Inst. no. 22 (1969).
- 10. I. ROSENQVIST, Eng. Geol. 1 (1966) 445.
- 11. J. OSTERMAN, Proc. 12th. Nat. Conf. Clays and Clay Min. (1964) 87.
- 12. J. K. MITCHELL and W. N. HOUSTON, *J. Soil Mech.* Found Div. Amer. Soc. Civ. Eng. **95** (1969) 845.
- 13. I. ROSENQVIST, Geotechnique 3 (1953) 195.
- 14. R. PUSCH and M. ARNOLD, Eng. Geol. 3 (1969) 135.
- 15. J. BELAND, Proc. Geol. Assoc. Canada 8 (1956) 143.
- 16. R. B. PECK, H. O. IRELAND, and T. S. FRY, Soil Mech. Studies Univ. Illinois no. 1 (1951).
- 17. L. LIDSTRÖM, Acta Polytech. Scand. Chem. & Met. series no. 75 (1968).
- 18. I. J. SMALLEY, Nature 211 (1966) 476.
- 19. A. J. MOSS, J. Geol. Soc. Australia 13 (1966) 97.
- 20. D. H. KRINSLEY and I. J. SMALLEY, Amer. Scientist 60 (1972) 286.
- 21. I. J. SMALLEY, J. Sediment. Petrol. 36 (1966) 669.
- 22. I.J. SMALLEY and C. VITA-FINZI, ibid 38 (1968) 766.
- 23. PH.H. KEUNEN, ibid 39 (1969) 1361.
- 24. C. VITA-FINZI and I. J. SMALLEY, *ibid* 40 (1970) 1367.
- 25. I. J. SMALLEY, *ibid* 36 (1966) 626.
- 26. D. H. KRINSLEY and S. MARGOLIS, Trans. New York Acad. Sci. 31 (1969) 457.
- 27. S. WHITE, J. Mater. Sci. 8 (1973) 490.

Received 12 September and accepted 20 November 1972.