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SCANNING ELECTRON MICROSCOPICAL OBSERVATIONS ON WEAKLY CEMENTED MIOCENE SANDS

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ABSTRACT

The cementation that has led to the local formation of slightly indurated layers in the white Miocene sand deposits, is due to authigenic quartz growth. Scanning electron microscopical information reveals that the secondary quartz is not only found at the grain contacts. A rather unexpected finding is its presence all around very small as well as large grains. The former ones show the most advanced idiomorphic habit and seem to play an actual part in the cementation. Presolved surfaces could not be found, so that the used silica does not appear to be produced by pressure solution. It is concluded that the grain surfaces must have been clean and highly disturbed before cementation started. This is, because only such surfaces seem to provide numerous suitable sites for nucleation, which may result in a comparatively rapid and full-scale overgrowing. However, since silica under certain conditions also dissolves very readily from disturbed surfaces, it is still possible that a part of the crystallized silica has come from the deposit itself. This particular type of early stage of cementation suggests strongly that the nature of the detrital quartz grain surfaces may be a very important factor in the kind of silica cement being formed. It is suggested that the origin of these weakly indurated layers must have been associated with seasonally rising and falling silica-bearing groundwater.

INTRODUCTION

Silicified layers are frequently found in the Miocene sand deposits of South Limburg and adjacent areas (van den Broek and van der Waals, 1969). Field evidence demonstrates that their occurence is restricted to the well-known white sands. The source of the cementing silica, the purity of the glass-sand, and the time of cementation are still a matter of dispute.

To account for the purity i.e. for the high degree of textural and mineralogical maturity and the lack of staining by organic material and iron compounds, an hypothesis was formerly actuated by the combined occurence of lignite and these white sands. Percolating waters containing organic acids from the plant material of the lignite would have produced these properties. Recently de Jong and van der Waals (1971), and Kuyl (1973) have proposed that the impermeability of a covering layer including lignite would have contributed to maintain the purity of the white sands. The infiltration of iron-bearing solutions from overlying Pleistocene terrace deposits would have been prevented by this layer.

From field evidence it seems unlikely that pressure solution was the mechanism providing the silica for cementation. The indurated layers occur in loose sands and the thickness of the overlying Pliocene and Pleistocene sediments has probably never been more than a few tens of meters (oral communication Dr. L. van der Waals).

Recently, presolved surfaces of sandstone grains have been described and illustrated by Pittman (1972) who used the scanning electron microscope (SEM). The sandstones studied by him ranged in age from Ordovician to Eocene. As the present sands date from the Tertiary, and more data for the problems mentioned above is desirable, particles of these cemented layers have been examined with the SEM.

PREVIOUS OBSERVATIONS WITH THE PETROLOGICAL MICROSCOPE

Before the scanning microscopical observations were made, thin sections of samples from the indurated layers exposed in the sand-pits of Beaujean (The Netherlands) and Sibelco (Belgium) were examined. From these it appeared:

1) Porosity as determined with the use of the Blaschke ocular (Blaschke, 1967) is high. Measurements in this sections of the Beaujean sample gave values of about 33%, and in those of the Sibelco sample of about 29%. Hence the common occurrence of "floating" grains is quite apprehensible (fig. 1).
2) Tangential, straight and concavo-convex contacts are present. The first type seems to predominate. Both of the other types were occasionally observed to have originated from quartz overgrowing (fig. 2). Suturing of quartz grains has not been perceived, though sometimes the incipient coalescence of the overgrowths of nearby grains seems to result in a wavy or zigzag boundary line (Sippel, 1968).
3) Obvious euhedral overgrowths as illustrated in figure 3, however, were not found frequently, though dustlines are not uncommon.

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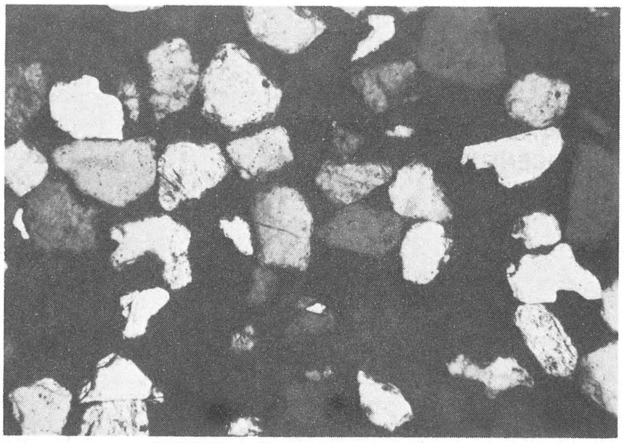


Fig. 1
Weakly cemented Miocene sand impregnated with artifical resin (Synolite). Objective p 3.5/0.10; ocular Periplan 10 x.

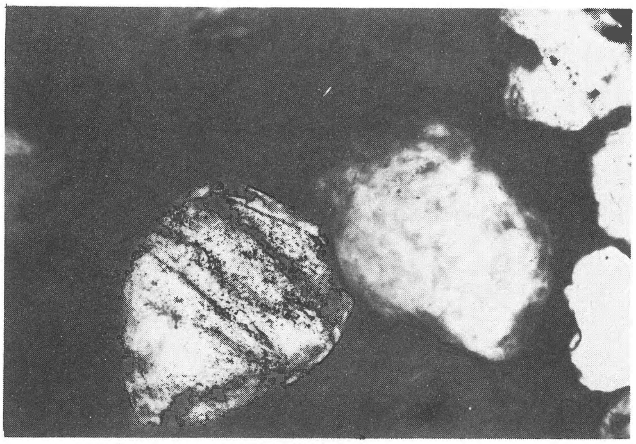


Fig. 2
Initial development of concavo-convex contact due to quartz overgrowing. Objective P 10/0.25; ocular Periplan 10 x.

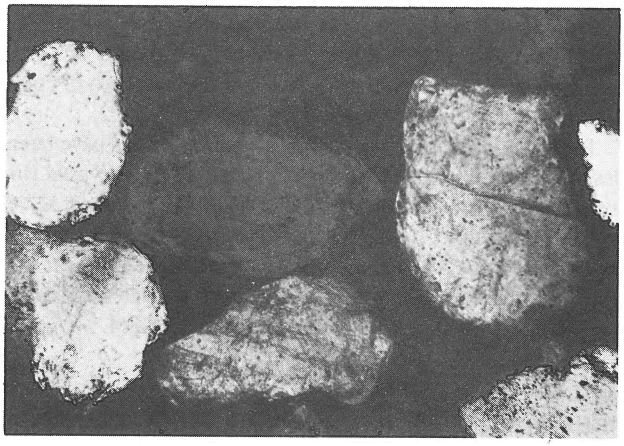


Fig. 3
Quartz overgrowths recognizable because of dustline and obvious crystal faces. Objective P 10/0.25; ocular Periplan 10 x.

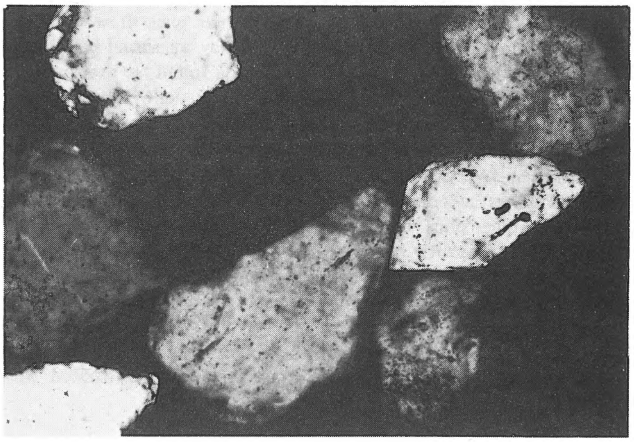


Fig. 4
Quartz grains showing straight boundaries. Objective P 10/0.25; ocular Periplan 10 x.

4) Although frayed outlines are present, smooth grain boundaries are more often found. Especially in the vicinity of grain contacts, very straight boundaries may be present (fig. 4). This suggests that the authigenic quartz has been mainly, if not exclusively, precipitated at the grain contacts, these being the sites where interstitial water remains longest.

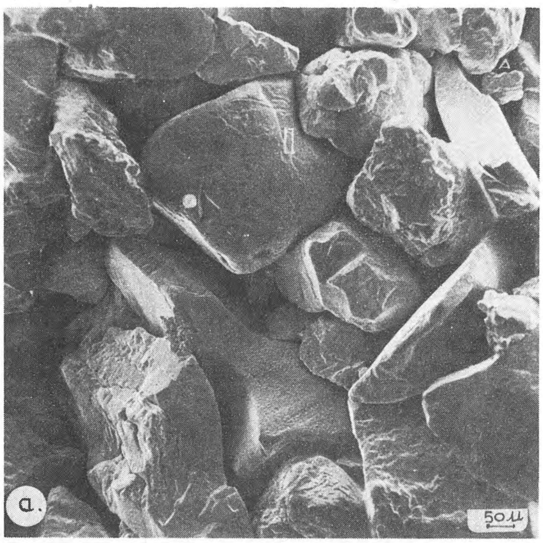
From the above it is concluded that the weak cementation of these layers was very probably due to the formation of secondary quartz, grown as optically continuous overgrowths at the grain contacts. This in spite of the fact that clear instances were rather scare in the examined thin sections.

SCANNING ELECTRON MICROSCOPICAL OBSERVATIONS

A stereoscan Mark II (Cambridge Instruments) was used. The sandstone fragments were carefully brushed off and mounted on a sample holder with aluminium paint. Coating with gold was done in a vacuum evaporator. During evaporation the sample holder was rotated continuously.

According to Pittman (1973) textures characteristic of presolved surfaces are "circular to ellipsoidal spots being relatively smooth or consisting of ridges and knobs with corresponding furrows and depressions". The ridges and knobs

Fig. 5
Scanning electron micrographs; a shows the mutual position of the grains in a cemented sand fragment; b illustrates a rather unusual surface ▶ covering with irregularly curved steps (see also d) and c the overgrowth development in a narrow fissure; d, e, and f show face development of nearby grains. The signs drawn in these and the following micrographs indicate the corresponding places in the different magnifications.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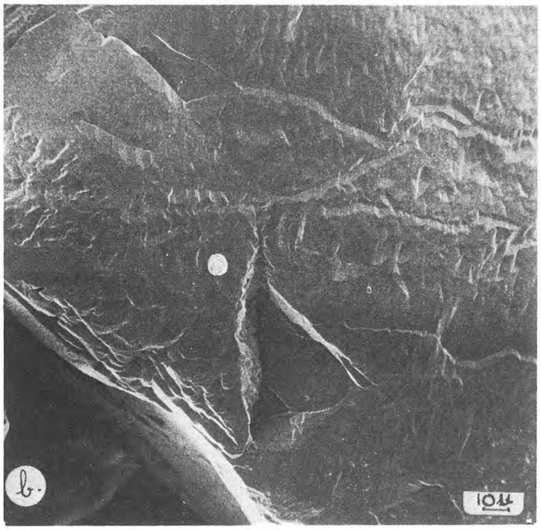
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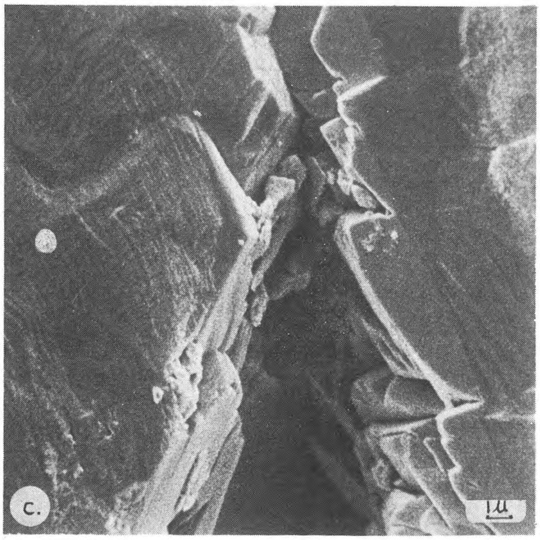
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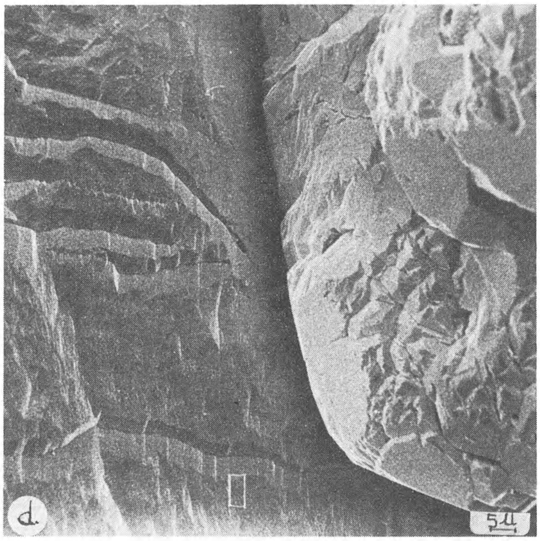
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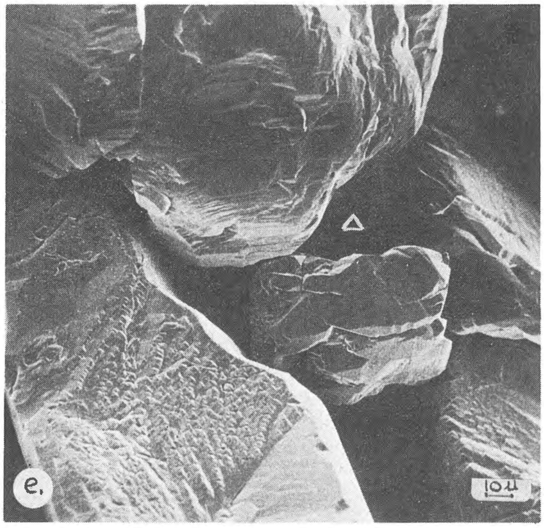
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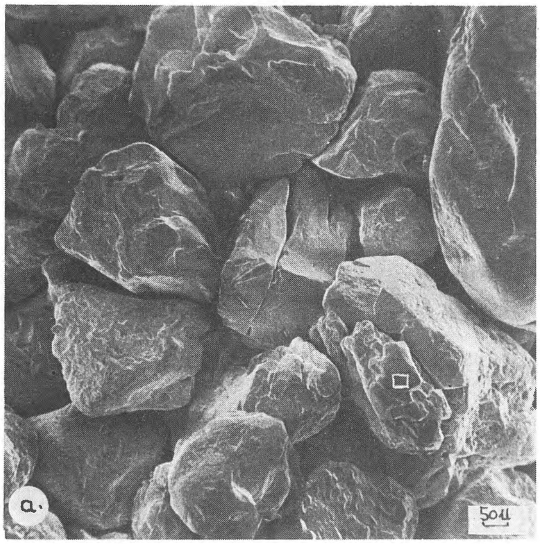
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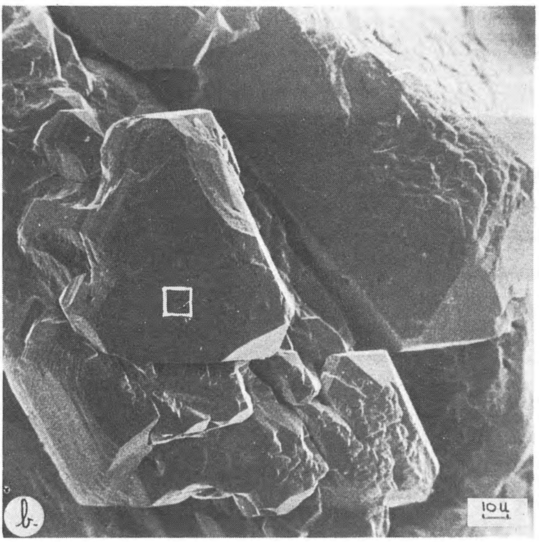
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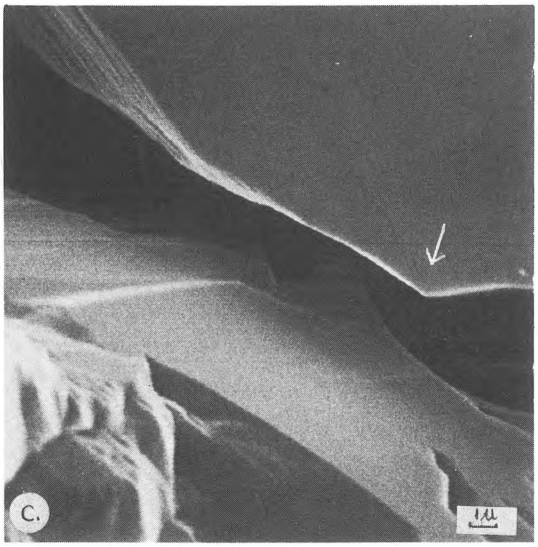
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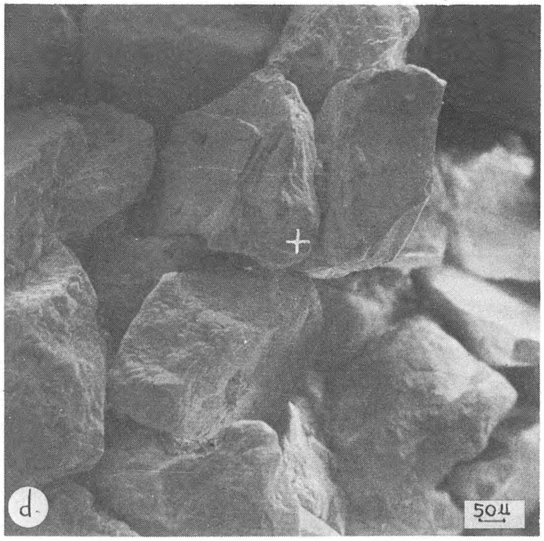
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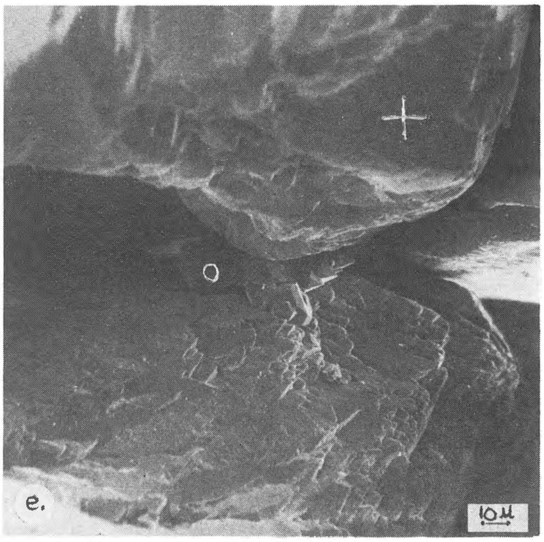
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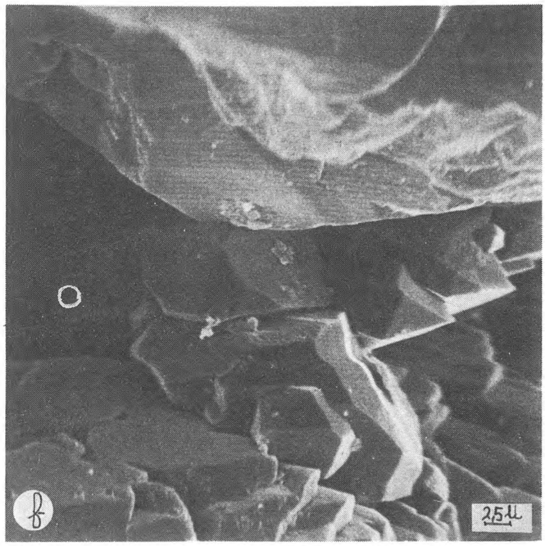
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occurring on some grains are considered to fit into furrows and depressions of a formerly adjacent grain.

Several fragments of the cemented sand (dimensions about 6 X 6 X 3 mm) and grains obtained by gently pulverizing some fragments, have been examined and several hundreds of micrographs carefully studied. Presolved surfaces have not been found. On the contrary, this search revealed that nearly all grain surfaces have been greatly influenced by quartz overgrowing (fig. 5-11). From the thin-section investigation it has been already inferred that authigenic quartz was probably the binding material and that the attachment must act mainly at the contact-points. But from the micrographs it is evident that non-contacting surfaces are also abundantly covered with secondary quartz (see for instance the figures 5e, 5f, 6b, 6c, 8b, 8c, 8e, 8f, 9c, 9d and 9f) and to such a degree that previous detrital surfaces are seldom to be seen. In theory, indications of pressure solution could be present under the overgrown quartz skins. The few detrital surfaces observed, however, did not show any of these characteristics.

As a consequence of this full-scale covering of the original grains with authigenic quartz, it could scarcely be ascertained how the overgrowths are attached to the detrital grains; nor could it be established whether the boundaries between the detrital grains and secondary quartz were open or closed. That is whether or not the boundaries are continuous or interrupted by voids which result from the isolated attachment of the overgrowths (Pittman, 1973). Open boundaries were found by Pittman in many sandstones and in the opinion of this author the well-known dustlines in thin sections originate by the subsequent filling up of the voids of such open boundaries. Early development stages, marked by the presence of numerous, more or less isolated, projections were found on some grain surfaces (figs. 7d, 7e, 7f, 9d and 9e). It is suggested by Waugh (1970) that by the mergence and overlap of these projections further growth is accomplished. Such a pattern of overgrowing might indeed allow the origin of voids. Other surfaces, also showing early stages of overgrowing (figs. 5b, 5d, 9c, 11f, 11j and 11l), exhibit a different pattern that seems to point to a very close attachment.

The surfaces of small as well as large detrital particles must have acted as a favourable substrate. There is, however, a conspicuous difference in their idiomorphic development. Polycristalline grains are easily discerned, even at low magnifications, because their single grain units generally show the most advanced development of idiomorphic outlines (figs. 7a, 7b, 7c and 8a). This greater progress in idiomorphic development is likewise exhibited by separate small grains occurring individually (figs. 8f, 9b and 10f) or in clusters attached to each other or to the larger grains (figs. 10a, 10b, 10e, 11a, 11b, 11c, 11d and 11g). The large grains do clearly show quartz overgrowing (fig. 9f), but more fully developed faces (figs. 6b and 6c) are rarely fond, though, as already stated, almost all of their surfaces are affected by quartz growth. Due to local concentrations of highly oriented rhombohedral projections, however, some large grains also create the impression of having gained a more advanced idiomorphic development (figs. 7b, 10d, 11h and 11i). Even in narrow and deep surface depressions, overgrowing has evidently taken place (figs. 5a, 5b and 5c).

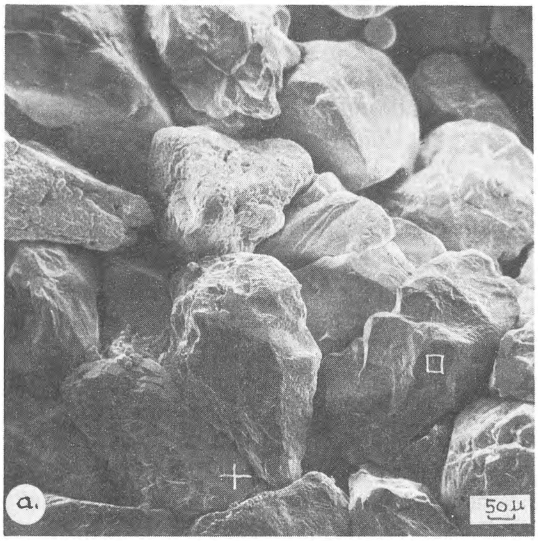
A further striking phenomenon is offered by the idiomorphic, small particles (figs. 6d, 6e, 6f, 8a, 8b and 8c) found, firmly locked between the large ones, as well as by the small idiomorphic hollows (figs. 10b, 10c, 11e and 11f) in the overgrown rims or faces of the large-sized grains. The hollows have very probably been produced by the loss of the small particles. This phenomenon suggests two things. Firstly, at least some of the small particles must have gained their idiomorphic habit before the overgrown rims on the adjacent large grains had reached their present thickness. Secondly, they must have played an important role in the cementation procress.

Especially on the large grain surfaces, irregular and fairly regular steps and striations frequently occur (figs. 9c, 11f, 11j and 11l). They have been observed on the flat surface parts as well as on the rough, irregular ones, e.g. those resulting from conchoidal breaking. Occasionally these steps bear resemblance to the semiparallel steps described by Krinsley and Donahue (1968), and attributed by these authors to the influence of glacial environments. Here, however, these features are probably mostly due to the rapid growth rate of the basal plane (Balman and Laudise, 1963), combined with a development of rhombohedral and prism faces.

◀ Fig. 6
Scanning electron micrographs of silicified sand fragments (a and b); b shows comparatively well-developed crystal faces on large grains; note the crystal formation when pore space is sufficiently available (c); e and f illustrate the occurrence and idiomorphic development of small particles at the contacts of large ones.

Fig. 7
Scanning electron micrographs of cemented sand (a); b and c show remarkably well-progressed development stages of a monocrystalline and a polycrystalline grain; d,e and f illustrate at different magnifications a very early overgrowth stage characterized by isolated overgrowths. ▶

Fig. 8
Scanning electron micrographs; a, b and c illustrate the contact between two overgrown grain surfaces; in d,e and f, grain surfaces are shown which exhibit in addition to quartz overgrowing, features pointing to contacts with formerly adjacent grains; note in f the very small euhedral grains still attached to the large one. ▶▶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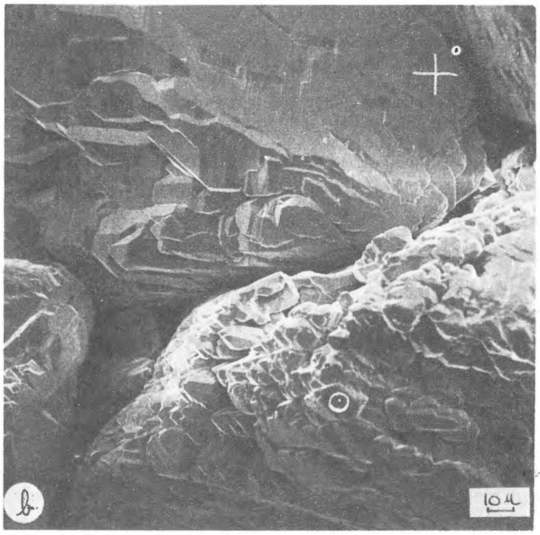
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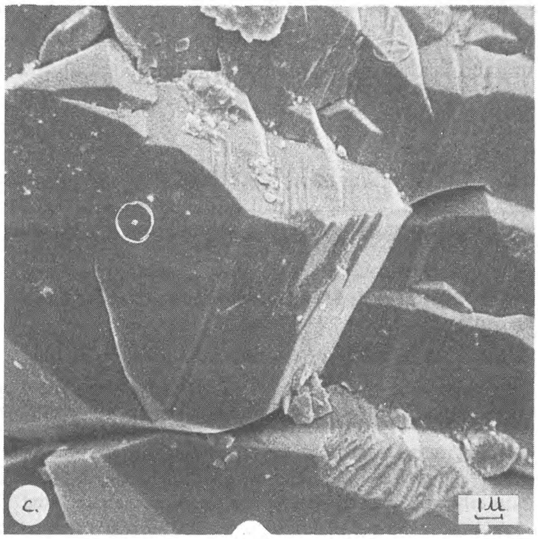
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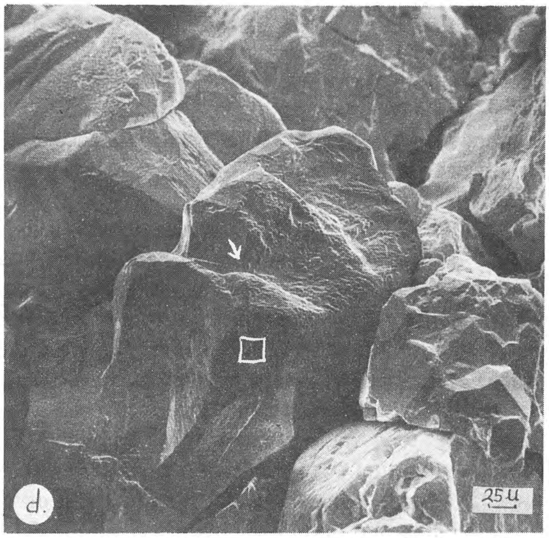
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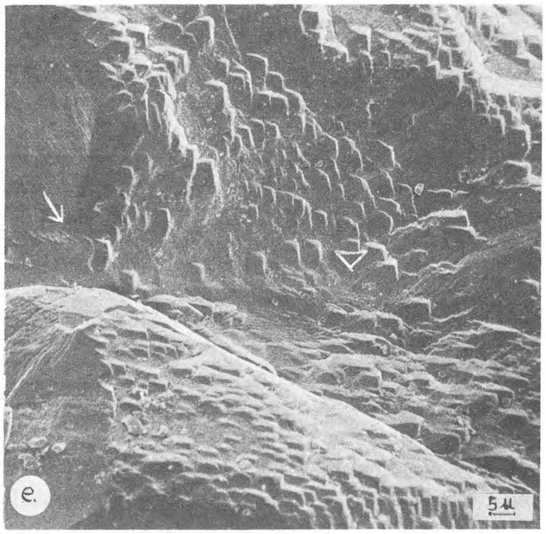
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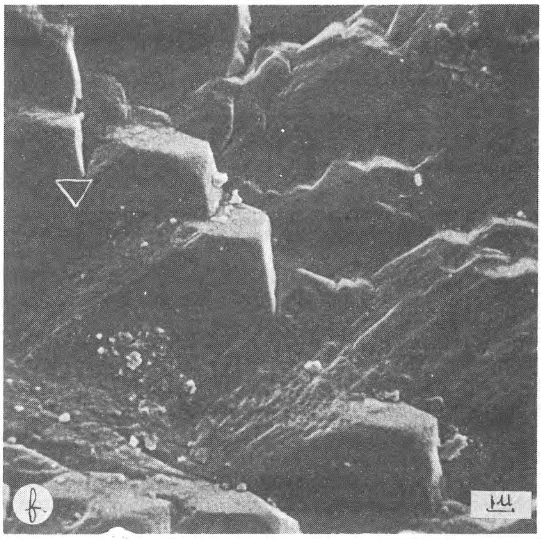
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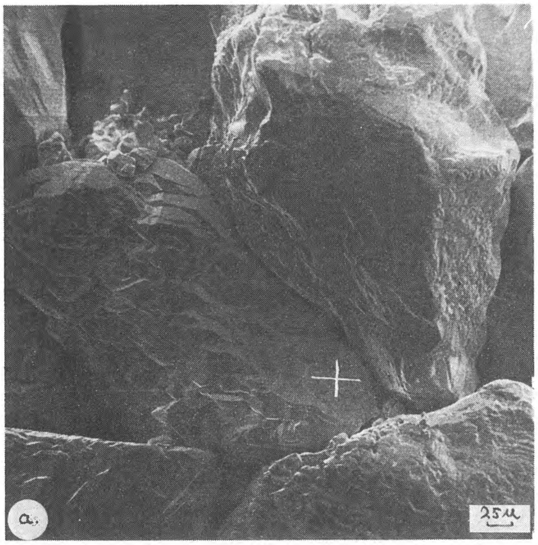
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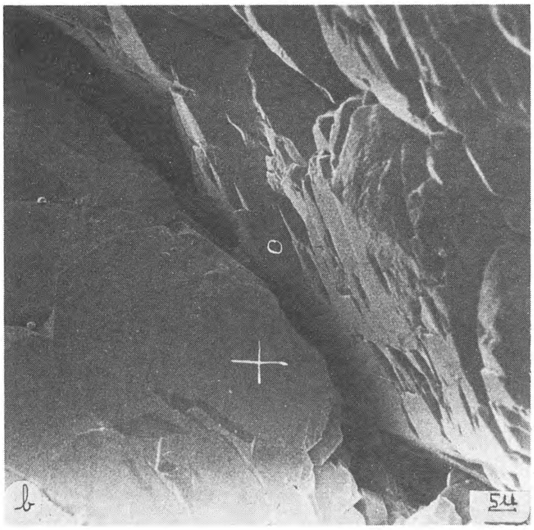
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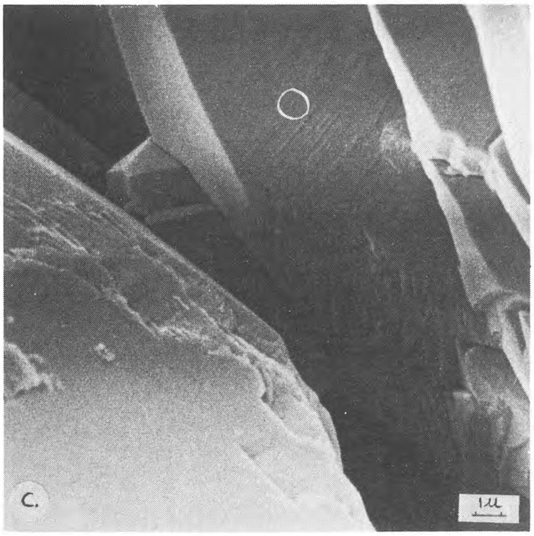
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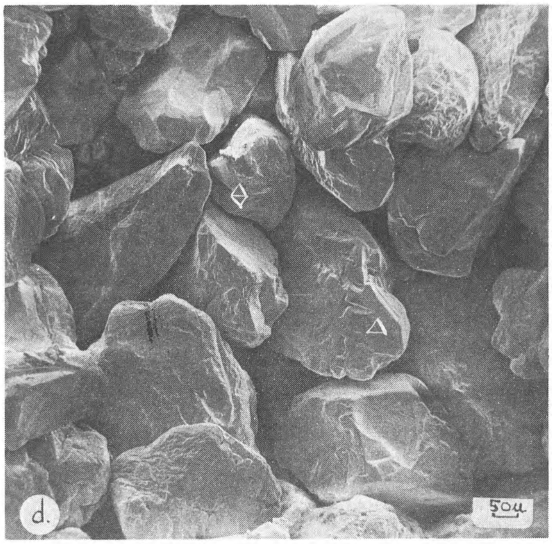
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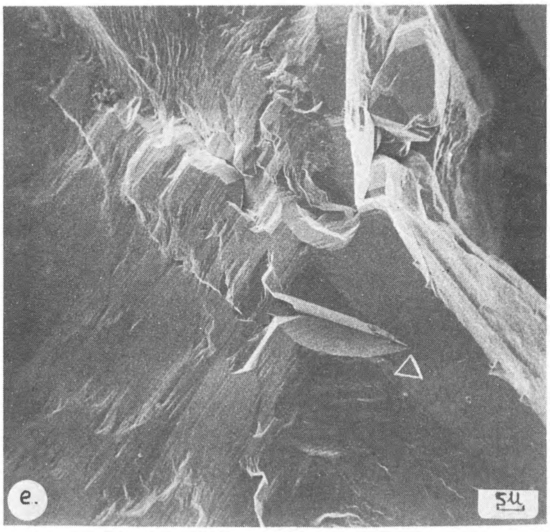
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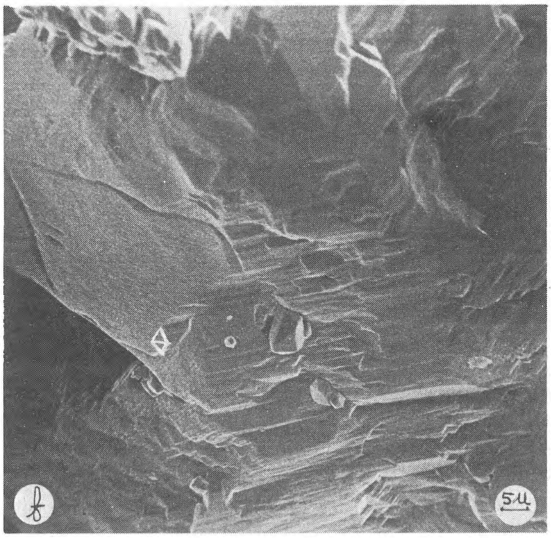
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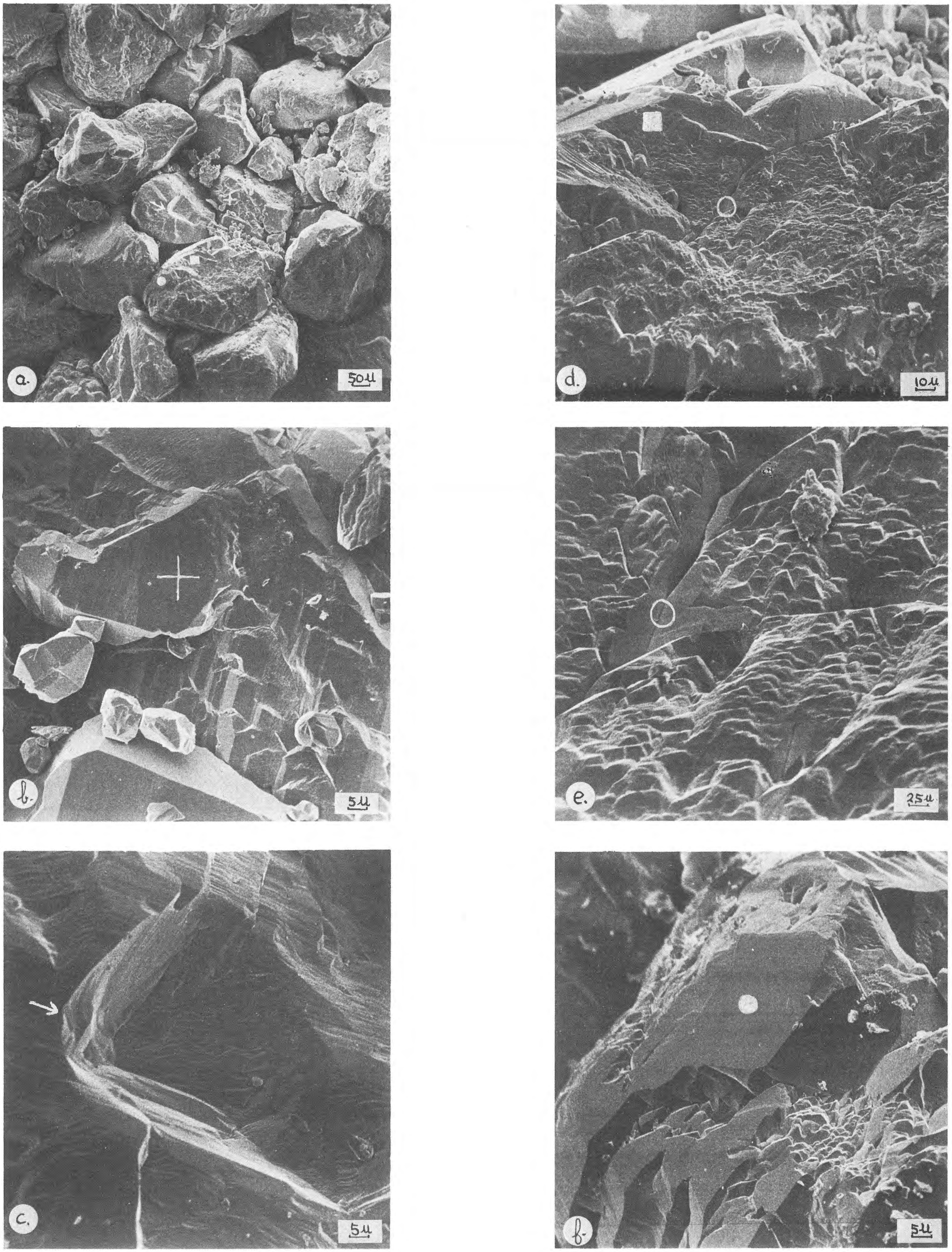
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DISCUSSION AND EVALUATION OF THE OBSERVATIONS

The absence of presolved surfaces on grains in these indurated layers shows that the material of the overgrowths had not been provided by pressure solution acting within these layers. This conclusion agrees with evidence obtained from the field. The origin of the overgrowths seems to be quite obvious. The fact that they occur all around the grains and that they are abundantly developed in the proximity of grain contacts and at those sites where the grains are very closely situated, suggests that they must have originated by precipitation from water. The marked higher degree of faces development, shown by the very small grains is a further indication of such an origin. The euhedral small particles seem to play an actual role in the binding of the materials since after an initial period of growth, they interlock with subsequent overgrowth of the large grains.

These observations and their interpretations, combined with field evidence allow certain conclusions in relation to the problems raised in the introduction. At the time when the development of the overgrown quartz started, the deposit must already have gained its present textural and mineralogical maturity. This means that in that phase of its post-depositional history quartz was the predominant mineral, silt and clay particles were very scarce, while the grain surfaces were largely devoid of adhering material (Cech and Heald, 1971). Further, pore space must have been in the same order of magnitude.

It is difficult to determine when the sands gained their maturity. This could have been during the period between deposition and cementation, but it is also possible that the material deposited was already of such a mature nature. The position of the small silt-sized quartz particles suggests that their amount beforme cementation may have been greater. Only those particles being trapped between the large grains have apparently escaped from the action of a removing agent. However, again, this removal may have been active during the deposition process as well as later.

The fact that the overgrown material is composed of crystalline quartz must also be considered. According to experiments under hydrothermal conditions, amorphous silica is converted to quartz through cristoballite and this transformation is mainly governed by temperature and time (White and Corwin,, 1961). These results led investigators involved with the diagenesis of silica in sediments, to relate the authigenic silica modifications to their age, other possible effective factors having been considered as being of minor importance (Mizutani, 1966). The thin sections examined here, however, do not indicate that such a transformation has taken place. Moreover, recent experiments indicate that the formation of quartz overgrowths on crushed quartz particles by precipitation from water is possible in a comparatively short time (Mackenzie and Gees, 1971); Paraguassu, 1972). The origin of quartz at room temperature and 1 atmosphere seems thus to be possible without other silica modifications preceding it. Therefore it seems likely that factors, generally considered unimportant in the genesis of silica modifications, may under certain conditions be very effective.

Whether or not this can be accepted must depend also on field data. It has already been mentioned that the occurrence of cemented layers is confined to the white pure sands. In Miocene sands rich in glauconite, clay, silt or ferruginous and micaceous material, they are lacking entirely.

A considerable knowledge of the formation of quartz crystals has been gained during attempts to produce perfect crystals for industrial purposes. Apart from impurities and inclusions, natural crystals contain a number of deviations from the perfect structure. Some of these structural imperfections are called dislocations or line defects. It has been recognized that dislocations may also be introduced by the act of cleavage, breakage or damage of a solid. These lines of defects being thermodynamically unstable, are characterized by an enhanced reactivity. In recent years it has been increasingly realized that dislocations are of primary importance with respect to such phenomena as crystal growth and dissolution. They form sites on a solid surface where dissolution is enhanced in low undersaturation and where preferential nucleation occurs in supersaturation. Solid surfaces with a large number of dislocations show a rate of growth and dissolution greater than the maximum possible values computed on the assumption that dissolution or nucleation takes place on essentially flat surfaces, (Thomas,, 1970).

Also in the field of earth sciences experimental results have been obtained which point to such an enlarged surface reactivity (Fournier, 1960, Morey et al., 1962).

Generally it may now be stated that quartz grains with fresh, rough surfaces due to breakage and crushing and uncovered by adhering materials, can form an abundant source of silica when the unsaturated interstitial waters are continuously and rapidly drained and replenished. At the same time, such grains provide a very suitable substrate for crystal growth in supersaturated stagnant water.

ENVIRONMENTAL RELATIONSHIPS DURING THE GENESIS OF THE SILICIFIED LAYERS

According to investigations of de Jong and van der Waals (1971) the white Miocene sands of Southern Limburg have been deposited in a shallow-marine tidal and littoral environment. In places, however, sedimentary structures seem to indicate such changes in environmental

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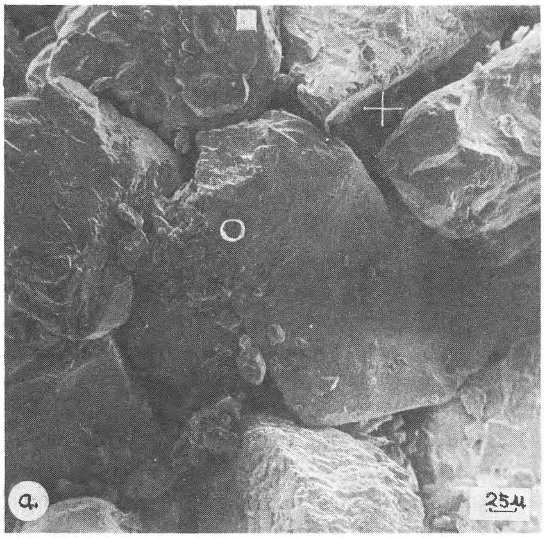
◀ Fig. 9
Scanning electron micrograph of a cemented sand fragment with a somewhat higher content of silt-sized particles (a); b, c and d illustrate the surface conditions on different grains; note the sheet-like growth in c; e and f exhibit different growth stages on a single grain.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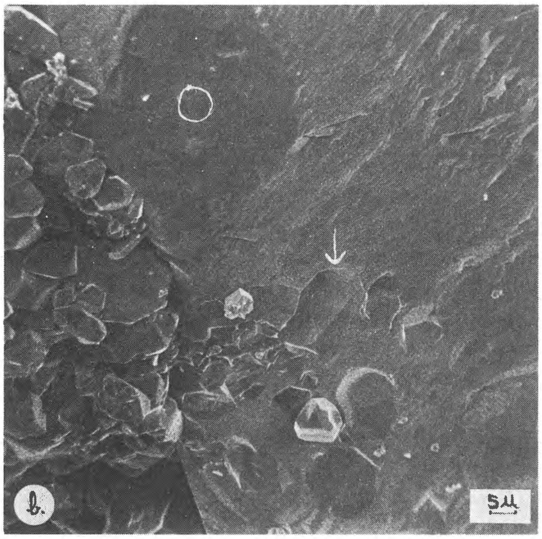
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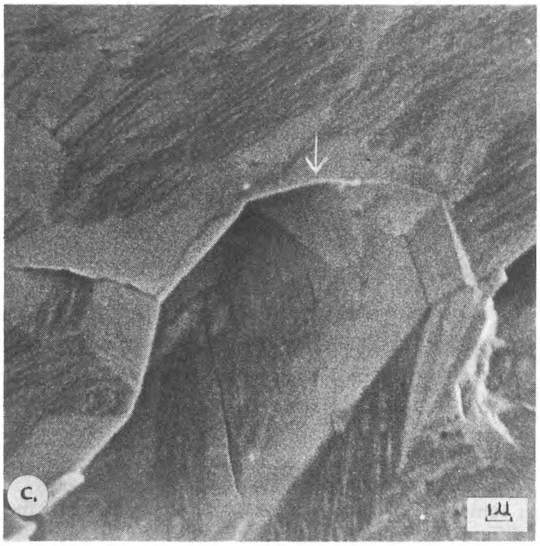
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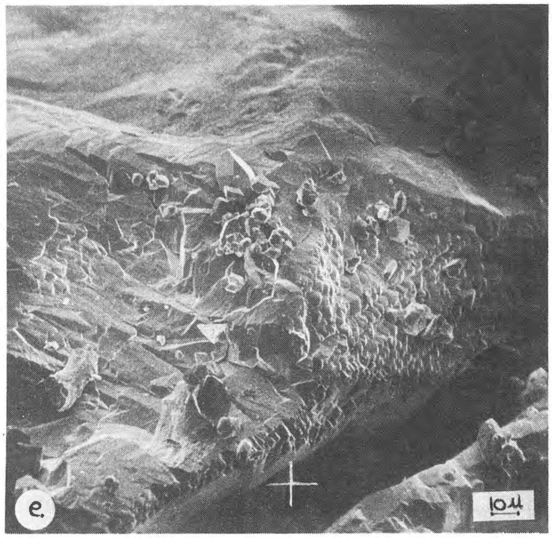
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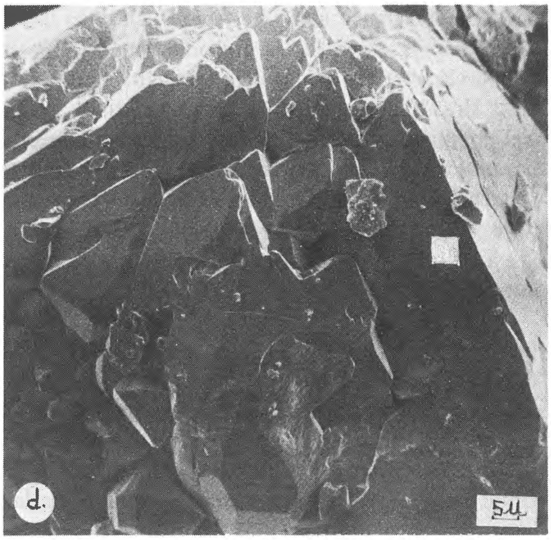
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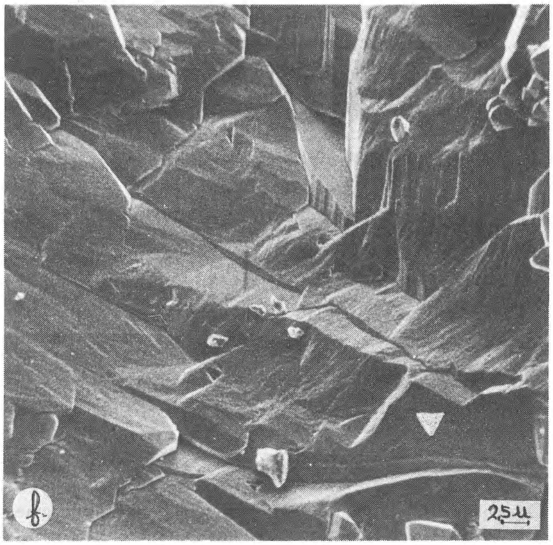
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conditions, that the authors tend to consider these sands locally as aeolian dune deposits. Sedimentary structures in the cemented layers have not been reported. The authors, however, do mention root tubes as a common feature of the sillicified layers, though their occurrence is not limited to these layers. The known data, including the presence of lignite layers, seem to point to an occasional emergence of the littoral zone during minor and major regressions.

Summarizing the data from field and laboratory, it seems rather unlikely that the cementation was due to the precipitation of quartz from supersaturated stagnant waters while the deposits were still submerged at greater depths. It would then have to be assumed that the conditions permitting this precipitation prevailed by accident in certain parts of the deposits, because firstly the indurated layers show very restricted horizontal and vertical dimensions, secondly they are underlain as well as overlain by loose sands and thirdly they may even occur twice in one section, one on top of the other, separated by unconsolidated sands. Moreover, these loose sands and the sand from the silicified layers generally exhibit a marked similarity in textural and mineralogical properties. Finally it would also have to be assumed that such features as root tubes have originated after the cementation process. Therefore the precipitation of quartz has undoubtedly occurred at the earth's surface during an emergence of the littoral zone.

In general, it is difficult to establish in the exposed sections – apart from those in which lignite layers are present – whether the periods of emergence of these littoral areas were long or short. It may have been possible, especially during minor regressions, that the littoral areas were subjected to a continual shifting or to alternating periods of subsidiary emergence and submergence. However, root tubes in any case indicate that sometimes the emerged areas existed long enough to be invaded by a vegetation and long enough for the calcareous organic material to be removed from the deposits.

It is plain that in such coastal landscapes, only those micro-environments have to be considered where the conditions necessary for this type of crystalline quartz growth, and leading at the same time to the formation of clearly distinguished cemented layers, are fully complied with. In theory, all sites in the landscape where the appropriate material is permanently covered or surrounded by supersaturated waters would fullfill these requirements. Since many surface waters are saturated or slightly oversaturated with respect to crystalline quartz (Siever, 1957, 1962), the silica content does not seem to provide any serious limitations in defining the environmental conditions, especially not in those areas where the deposits are mainly made up of quartz particles showing disturbed surfaces (Fournier, 1960; Morey et al., 1962; Mackenzie and Gees, 1971). In this respect, a more important limiting factor may be the required period of stagnation of the pore waters, which restricts the necessary conditions to those materials that remained continuously under the groundwater level. However, because of the restricted dimensions of these layers, such a view raises the same objections as mentioned above, though it may be possible that immediately under the groundwater level some physico-chemical gradients may have been operating locally.

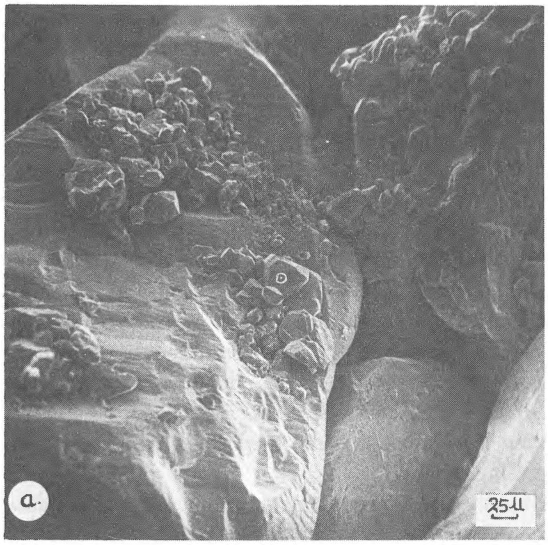
On relatively flat land surfaces this reduces the favourable environmental conditions to the capillary fringe immediately above the groundwater table and to the low-lying areas that are intermittently covered by a shallow water-body. The necessary sequence of inundation, draining and evaporation of the interstitial waters may be hypothesised as being induced by a climate with distinct wet and dry periods and/or to the tidal movements of a nearby sea. In these ways, continuous and discrete waterfilms could be generated around the quartz grains.

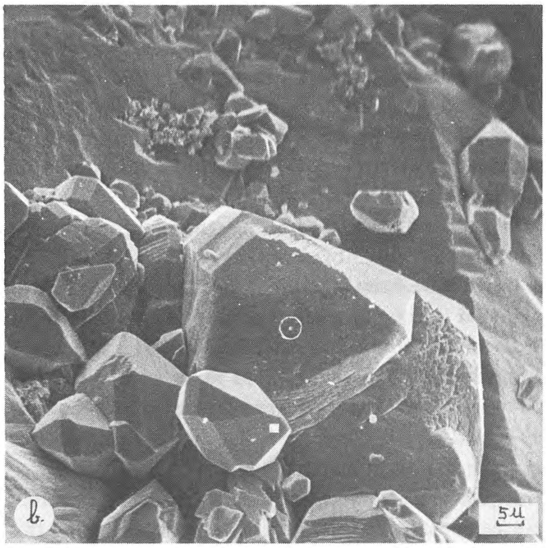
Unfortunately, no data concerning the elevation of the cemented layers in the different exposures are available. Such data might have been of value in deciding whether these layers originated in the depressions of the landscape or in the zone of intermittant groundwater saturation. The latter possibility is favoured since the surface layer in a low-lying, temporarily soaked or water-covered area generally collects substances which give it properties that probably inhibit this particular precipitation of quartz.

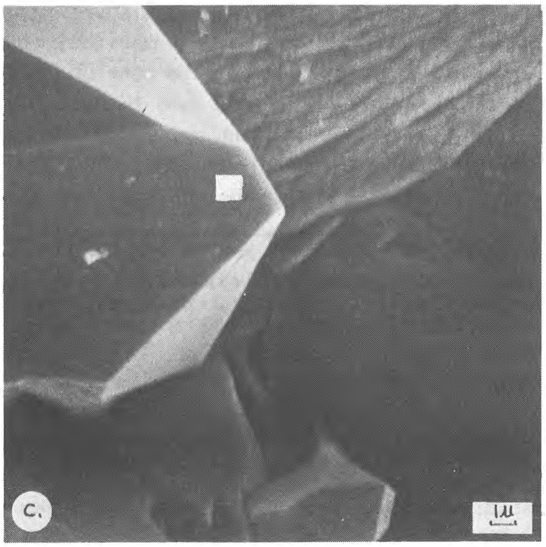
The precipitation of crystalline quartz has led to a rather incomplete cementation. Two obvious explanations for this are possible. The most plausible one is that the progress of the cementing process was stopped by a comparatively rapid submergence or emergence. In both of these cases the layers at issue would have disappeared from the zone of intermittent water saturation; in the first case to be partly protected by a resuming sedimentation, in the second to be largely destroyed by subsequent erosion. The other explanation may be found in changes of the

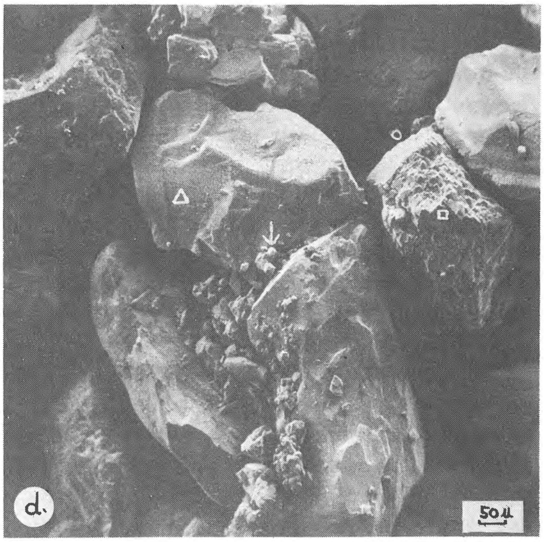
◀ Fig. 10 Scanning electron micrograph of a fragment showing clusters of silt-sized particles on some grains (a); note the faithful reproduction of their euhedral habit as shown by micrograph b and c; d illustrates the abundant development of rhombohedral faces at the distal end of a grain; e exhibits a small cluster of idiomorphic silt particles (note the abrupt change in surface characteristics of the large grain) and f some isolated ones still parly embedded in the overgrown material on the large grain.

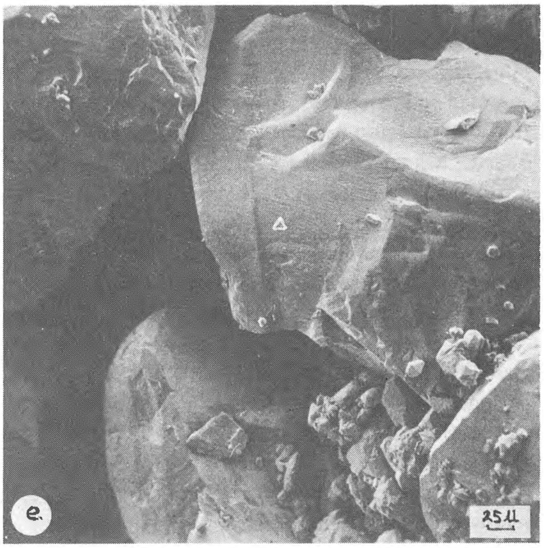
Fig. 11
Scanning electron micrographs. One of the most progressed stages of overgrowing found is demonstrated by the particles of silt size in the ▶ micrographs a (enlargements b and c) and d (enlargement g); d, e and f illustrate the growth features often found on large grains that were apparently formerly covered with conchoidal fracture planes (see also k and l); note the shown in l exhibits a feature (see arrow) that may possibly idiomorphic hollows in f; d, k and i exhibit oriented rhombohedral overgrowths which led locally to the formation of a large crystal face (see i, left side); the grain surface shown in 1 exhibits a feature (see arrow) that may passibly point to the presence of dislocations; these features are, though less distinct, presumably also present in j.

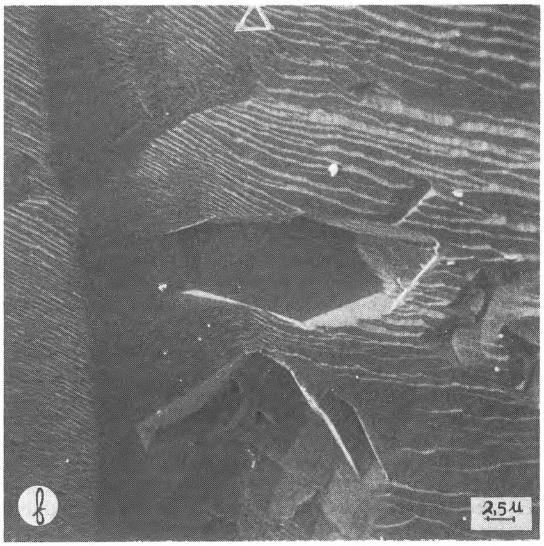

a.
25µ

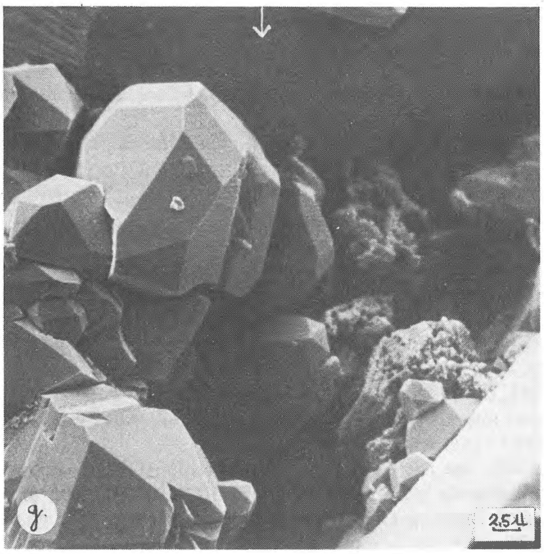

b.
5µ

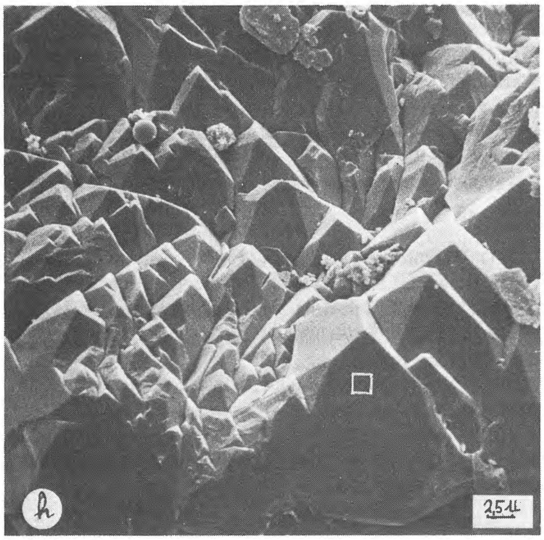

c.
1µ

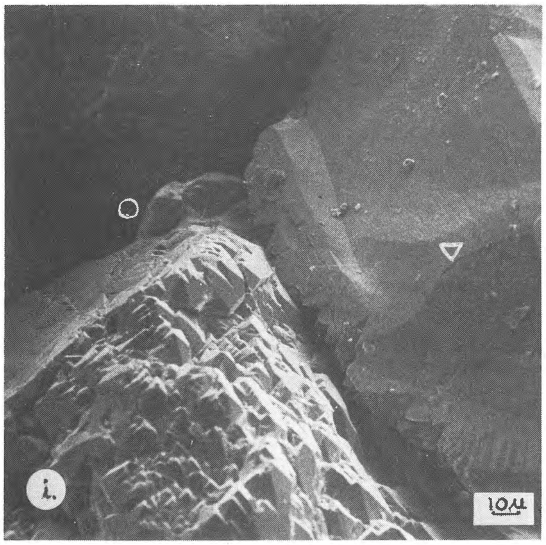

d.
50µ

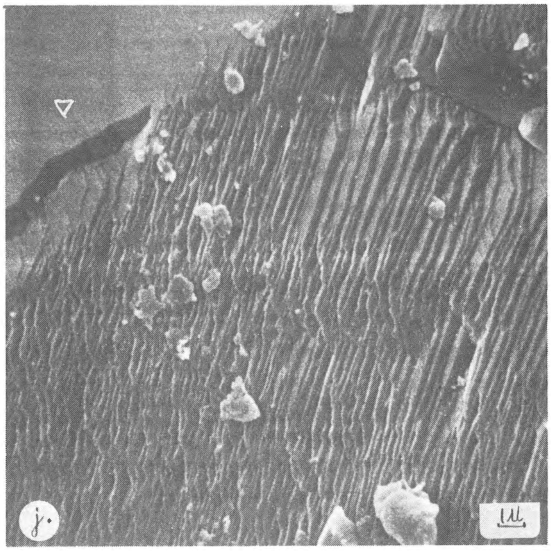

e.
25µ

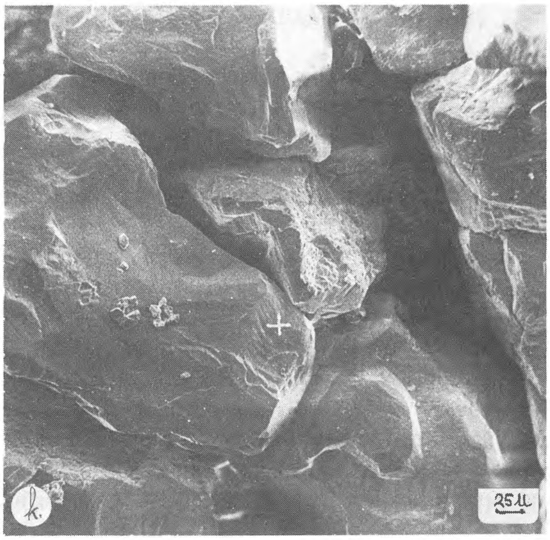

f
2,5µ

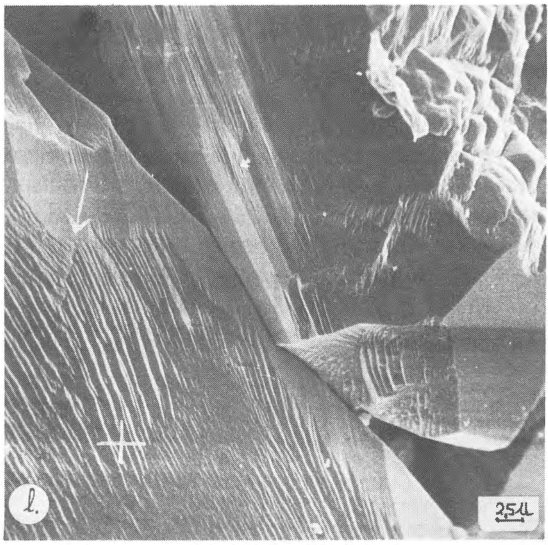

g.
25µ


h
25µ


i.
10µ


j.
1µ


k.
25µ


l.
25µ

physico-chemical conditions prevailing in the solid-solution system. Earlier it was stated that surface waters are generally saturated in relation to crystalline quartz. However, a supply of water bearing lower concentrations of silica might result in the overgrowing either being slowed down or stopped. A changing balance between evapotranspiration and precipitation might also have been an additional or even a primary factor, especially when the supplied water is unsaturated in respect to crystalline quartz. Finally, diffusion-controlled, as well as several types of surface-reaction-controlled growth mechanisms may have been acting in the grain-waterfilm system. Among these mechanisms, owing to the strongly disturbed grain surfaces, it seems likely that dislocation-controlled growth was initially very important. In a later stage of the overgrowing process this mechanism may have been less effective because the majority of the quartz grain surfaces to a great extent have been healed.

CONCLUDING REMARKS

Since it is realized that the formation of silica minerals is a common process under earth surface conditions, an increasing number of investigations are being devoted to this topic. Several factors have been recognized as being significant in this respect. Until recently, it was thought that the formation of crystalline quartz at earth surface conditions was only possible in a very long period of time. Recent experiments have indicated that quartz formation could take place in a relatively short time. The experiments also suggested that the conditions of the grain surfaces on which crystallization of quartz occurred, may be a decisive factor. This explains satisfactorily the observations described here, and in future the abundant presence of disturbed quartz surfaces has perhaps to be considered as possibly effective for the rapid formation of crystalline quartz at the earth surface.

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