

## ULTRASTRUCTURES IN RECENT RADIAL AND GRANULAR CALCAREOUS FORAMINIFERA

by  
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Bellemo, Sandro: Ultrastructures in recent radial and granular calcareous Foraminifera. *Bull. geol. Instn Univ. Upsala*, Vol. N. S. 4, pp. 117—122.

The ultrastructure of the test wall is studied in six species of recent foraminifers: three species of the radial structure type, and three species of the granular type. A new method for studying orientation of the microcrystals in crystal units is introduced. This method is based on etching of the test wall with glutaraldehyde solution. The stacks of microcrystals in the crystal units are dissolved so as to leave needle-shaped crystallites. The orientation of these crystallites is parallel to the *c*-axes. The use of this method permits a detailed mapping of crystal orientation which has hitherto been difficult in the granular type of the test wall.

The organic membranes separating the crystal units are partly demineralized with a chromium sulphate solution and their morphology is briefly described and illustrated.

The stacks of microcrystals constituting the crystal units strongly resemble, both in habit and optical characters, limbs of inorganic dendrites.

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### INTRODUCTION

A comprehensive, systematic study of numerous agglutinated and calcareous foraminiferal tests was published by Wood (1949); previous literature was also reviewed. His paper included both the radial and the granular wall structure of hyaline perforate foraminifera. Between crossed nicols, the radial wall was said to show “a black cross with concentric rings of colour closely mimicking a typical (negative) uniaxial interference figure”, confirming that “the test is built of crystals of calcite with their *c*-axes normal to the spherical surface” (Wood, 1949, p. 240). The granular wall was described by the same author to show that between crossed nicols “a multitude of tiny flecks of colour” is seen. In section, the wall appears “minutely granular”. The granules were said to be “equidimensionally sutured together ... diversely orientated and therefore showing differences in refractive index”. (Wood, 1949, p. 241—242).

Great taxonomic importance was given to these

structures. Although many authors after Wood expressed caution in using optical properties of the test wall for systematic purposes, the recent classification of foraminifera of Loeblich and Tappan (1964) has the radial and the granular wall structures characterizing different superfamilies. This classification was criticized by Towe and Cifelli (1967) who, on the basis of their Transmission Electron Microscope studies (TEM), came to the conclusion that the radial and the granular structures are “basically similar ... despite the dramatic differences seen in polarized light”, and that the terms radial and granular “have been indiscriminately emphasized”. Hansen (1971) found that two foraminiferal species of the genus *Turrilina*, which are externally indistinguishable, have different wall structure. Thus Hansen, as Towe and Cifelli (1967), recommended the use of radial and granular wall structures as criteria at the species level only.

Previous concepts on the radial and granular wall structures have been largely based on TEM

studies. In the present paper, the Scanning Electron Microscope (SEM) is used and new methods of investigation are introduced. The morphology of the mineral components of the test is mainly described. In a forthcoming paper, both the organic matrix of the test and the dendritic organisation of the mineral components will be treated in more detail.

## MATERIAL AND METHODS

The material described in this paper comprises of numerous tests from the following recent hyaline perforate foraminiferal species:

Radial: *Globobulimina turgida* (Bailey)  
*Globorotalia menardii* (d'Orbigny)  
*Ammonia beccarii* (Linné)

Granular: *Cassidulina laevigata* (d'Orbigny)  
*Melonis zaandamii* (van Vorthuysen)  
*Nonion labradoricum* (Dawson)

Except for *Globorotalia menardii*, which was obtained through Dr. Arnold, Berkeley, California, from the Caribbean, the material was collected during the summer of 1970 at the Kristineberg Zoologiska Station. Samples were taken in the Gullmar Fjord at a depth of about 50 m., from the upper 4—6 cm of the bottom sediment. As indicated by Rose Bengal staining (Walton, 1952), most specimens used in this study were alive when collected.

In the previous investigations, both single- and double-stage replica techniques for TEM have been used by Towe and Cifelli (1967), Pessagno and Miyano (1968), Hansen *et. al.* (1969) and Stapleton (1973). Before replication, the preparations were etched with EDTA or hydrochloric acid. For previous SEM investigations the test walls were studied either, after etching with EDTA (Hansen *et. al.*, 1969; Bé and Hemleben, 1970; Stapleton 1973) or unetched (Hemleben, 1969; Bé and Hemleben, 1970). The methods used in the present paper are now described.

*Studies of the crystalline elements.* Numerous tests of each species were crushed. Some of the frag-

ments were treated with a concentrated solution of sodium hypochlorite, containing 8—12 % active chlorine, for periods between 10 minutes and 24 hours. By this treatment the organic substance was completely dissolved both on the inner and outer test surfaces, as well as on the fracture planes. After the removal of the organic substance, the specimens were rinsed several times in distilled water and glued to the SEM specimen-holders. Test fragments treated with sodium hypochlorite were studied under the polarizing microscope for their optical characters (Pl. 1, Figs. 1, 2).

*Studies of the organic substance.* Another part of the crushed tests was etched with glutaraldehyde solution (Mutvei, 1974). Glutaraldehyde fixes the shell proteins very efficiently, and simultaneously dissolves the crystalline elements. The specimens were glued to the SEM specimen-holders before immersion in glutaraldehyde solution; they were then stirred continuously during the treatment. The etching solution was used at different concentrations and pH values. At a concentration of 25 % and at pH 3.5, satisfactory results were obtained after between 5 and 20 minutes etching. Using longer etching times the fragments became too brittle to manipulate after drying. They would thus be more easily damaged by the electron beam. During immersion in etching solution, the degree of demineralisation of the test wall was controlled using light microscope. As the crystalline elements became dissolved, the transparency of the test increases; finally only a three-dimensional organic network remained (Pl. 1, Figs. 3a, 3b, respectively).

A 5 to 20 minute etch with 2.5 % glutaraldehyde solution using a phosphate buffer to about pH 5 also gave good results. At a still higher pH, solution of the test proceeded very slowly or ceased altogether.

Etching with glutaraldehyde solution, as just described, helped to interpret the crystalline structure. However, for obtaining organic residue of the test wall, the demineralisation with chromium sulphate solution (Sundström, 1968; Mutvei, 1970, 1972) was preferred. The specimens were then immersed in chromium sulphate for between 8 and 30 minutes. After etching, the exposed orga-

nic residue shrank and tended to collapse because the crystalline support had been removed.

The preparations on the specimen-holders were coated with evaporated gold and studied with a Jeolco SEM instrument, ISM-U3, at the Wallenberg Laboratory, Uppsala University. A double coating was often necessary to avoid charging artefacts.

## OBSERVATIONS

*The crystalline wall.* Both radial and granular structural types of the test wall are built up of calcite elements which in the previous literature has usually been called "calcite crystals" (see e.g. Wood, 1949, Hansen, 1970). These elements have also been called "crystal units", "calcite units" or "calcite grains" by Towe and Cifelli (1967), and "calcite platelets" by Hansen and Reiss (1971). In the present paper the term microcrystals will be used for these elements, *i.e.* the smallest morphologically distinguishable crystalline elements.

*Microcrystals and crystal columns.* In the species investigated, the diameter of the microcrystals varies from between 0.3 and 1.5 micron. The smallest microcrystals are thus at the lower limit of the practical resolution power of the SEM.

In the radial test wall the microcrystals are clearly discernible in *Ammonia beccarii* (Pl. 1, Fig. 4), *Globobulimina turgida* (Pl. 2, Fig. 1, 3, 5) and *Globorotalia menardii* (Pl. 3, Fig. 3). These microcrystals are euhedral, rhomboidal and are exposed on the test surface as three-sided pyramids representing the three rhombic faces.

In the granular test wall the microcrystals are clearly seen in *Cassidulina laevigata* (Pl. 6, Fig. 1, 2) and *Melonis zaandamii* (Pl. 5, Fig. 1, 4). In a third species, *Nonion labradoricum*, the microcrystals are usually too small for satisfactory photographic reproduction (Pl. 4, Fig. 2), although exceptions do occur (Pl. 4, Fig. 1). These microcrystals are also euhedral rhomboids, but not tabular as in the radial wall structure. Their orientation is different to that of the radial type having a rhombohedron face parallel to the test surface.

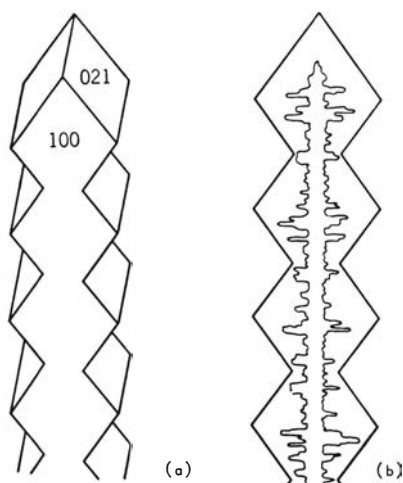
Towe and Cifelli (1967) found that the micro-

crystals in the radial test wall are arranged in vertical "elongate columns and wedges" ("columnar structure" by Hansen 1970; "stacks of platelets" by Hansen and Reiss, 1971). In the material described here, such crystal columns are clearly seen in the radial test wall of *Globobulimina turgida* (Pl. 2, Fig. 1, 3). It seems probable that the microcrystals in the granular test wall also have a corresponding columnar arrangement; however, the crystal columns are not arranged vertically, as in the radial wall, but inclined at about 45° to the test surface (see above).

*Orientation of c-axes.* By etching molluscan shells with glutaraldehyde solution, Murvei (1974) noticed that the aragonitic nacreous crystals reveal large numbers of needle-shaped crystallites orientated parallel to the *c*-axes. Similar crystallites were also found in inorganic calcite and aragonite crystals.

In foraminifers, I have tested this etching method on *Globorotalia menardii*. Here the test wall is covered by a secondary calcite crust made up of large calcite crystals with their three rhombic faces clearly exposed (Pl. 3, Fig. 1). By etching these crystals in glutaraldehyde solution, numerous needle-shaped crystallites remain which are oriented parallel to the *c*-axes of the crystals.

In the primary developed lamellar test wall, the test wall proper, the etching of the microcrystalline columns (see above) with the glutaraldehyde solution gives the following structural information. (1) In the radial type of the test wall of *Ammonia beccarii* (Pl. 2, Fig. 2, 4) and *Globorotalia menardii* (Pl. 3, Figs. 2, 4), the glutaraldehyde etching reveals numerous needle-shaped crystallites which are orientated normal to the test surface. Each crystallite represents a remnant of a microcrystalline column, probably the axial part of this column. Thus, the crystallites are orientated parallel to the *c*-axes of the microcrystal columns. The growth of the microcrystals takes place on a basal (0001) face, as supposed by Towe and Cifelli (1967). (2) In the granular type of the test wall the orientation of the needle-shaped crystallites incline at an angle of about 45° to the test surface (Pl. 4, Fig. 4a, 4b; Pl. 5, Fig. 2; Pl. 6, Fig. 3, 4). This proves that measure-



*Text-Figs. 1a, b.* Filled-in dendrites of acid potassium oxalate (after Buckley, 1951; p. 449, Fig. 147 c, d). The dendritic crystal (a) is very similar to a microcrystal column in the test wall of the radial foraminifer described here (see Pl. 2, Figs. 1, 3). The axial limb of the dendritic crystal (b) seems to correspond as an acicular crystallite after etching with glutaraldehyde solution. Therefore, the orientation of the acicular crystallites coincides with that of the  $c$ -axis of the microcrystal column (see Pl. 2, Fig. 4).

ments of the optical axes of the microcrystals in polarized light, made by Stapleton (1973), are correct. He observed that "the extinction occurs only in a small portion of the wall ... and appears to include clumps of grains of similar crystallographic orientation" when the thin sections were positioned at  $45^\circ$  to the direction of polarization. As Towe and Cifelli (1967), and Stapleton (1973) have already stated, it can be confirmed that in the granular test wall, the microcrystals grow "on a (1011) rhombohedron face".

The glutaraldehyde etching of the foraminiferal test walls is a new practical method by which the crystal orientation can be studied directly, and in detail. In the granular type of the wall structure particularly, one can now easily map the crystal orientation in comparatively large areas.

The characteristic columns of microcrystals in the foraminiferal test wall strongly resemble arms

of three-dimensional dendrites precipitated inorganically. Such dendritic arms have been illustrated in several papers, notably by Buckley (1951, p. 499, Fig. 147). He states that a large number of crystals occurring naturally are three-dimensional dendrites (see Text-Fig. 1). Experimentally, dendritic precipitation is favoured when the original liquid is rich in impurities. In the foraminiferal tests the inter- and intracrystalline organic matrices play the role of impurities and favour precipitation of calcite as dendrites.

*Crystal units.* Columns of microcrystals (see above) form together, larger structural units which are often called the crystal units. These units occur in most radial and granular test walls. On the basis of previous studies by Towe and Cifelli (1967) and his own studies, Hansen (1970, p. 14) defined the crystal units as "one or more crystals with identical optical orientation enveloped by a membrane; the membrane is regarded as the delimiting factor".

*Crystal units in the radial wall structure.* In the radial test wall the  $c$ -axes of all crystal units are vertical to the test surface. Since, however, the adjacent crystal units are isolated from each other by the vertical (sutural) organic membranes, the remaining axes ( $a_1, a_2, a_3$ ), which are perpendicular to the  $c$ -axes, have a random orientation in the crystal units. This condition is apparent on the vertical fracture plane of *Globobulimina turgida* (Pl. 2, Fig. 5). Owing to the rotation of the  $a_1, a_2$ , and  $a_3$  axes, the direction of dip of the crystal faces (1011) changes between the crystal units (indicated by arrows in Pl. 2, Fig. 5).

Another structural pattern related to the crystal units in the radial test wall is the "herringbone pattern", which occurs *i.e.* in *Ammonia beccarii* (Towe and Cifelli, 1967, "chevron like structure"). This pattern is described by Hansen and Reiss (1971, p. 331) as follows: "The platelets of each stack are seen in transverse section to be orientated with respect to these of adjacent stacks with an angle of about  $110^\circ$ ". The individual "stacks of platelets" were supposed by these writers to correspond the crystal units. According to my un-

published observations, the "herringbone pattern" is probably formed by alternate rotation, about  $180^\circ$ , of the dip direction in adjacent columns of microcrystals.

Theoretically this "pattern" may be formed when the crystal twinning occurs between adjacent microcrystal columns. Such a twinning is often formed between dendritic arms of inorganic crystals, the arms being "strictly parallel to same crystallographic direction" (Buckley, 1951, p. 483). In the present "herringbone pattern", the dendritic arms are parallel to the *c*-axes.

In my specimen of *Ammonia beccarii* a pattern similar to the "herringbone pattern" could be seen, occasionally, in adjacent crystal units, but each crystal unit consists of several columns of microcrystals.

*Crystal units in the granular wall structure.* Between crossed nicols, Wood (1949) observed in the granular type of the test wall, numerous "tiny flecks". Such flecks are here shown in Pl. 1, Fig. 2, and they correspond to the crystal units.

As in the radial type of the test wall, the crystal units are surrounded by vertical (sutural) membranes. These membranes are particularly well developed in *Nonion labradoricum*. After treatment with sodium hypochlorite solution, the membranes are dissolved, and a jig-saw like pattern of sutures on the outer and inner test surface remains. These sutures separate the adjacent crystal units (Pl. 4, Fig. 2). The membranes which originally occupied the sutures become well exposed when the test wall is etched with a chromium sulphate solution (Pl. 4, Fig. 3). A corresponding pattern of organic membranes appears in the outer test surface of *Melonis zaandamii* (Pl. 5, Fig. 3) treated in the same way. The membrane around each crystal unit is, in the latter species, connected with the organic walls of the pore tubes. The test of *Melonis zaandamii* is covered by an outer and inner membrane (Pl. 5, Fig. , 5 resp.). As already pointed out, the *c*-axes in the crystal units of the granular type of the test wall are orientated at  $45^\circ$  to the test surface. The microcrystals in the crystal units are equidimensional, with rhombohedron face (see p. 119)

parallel to the test surface (Pl. 5, Fig. 1). The needle-shape crystallites which remain after the etching with the glutaraldehyde solution permit detailed observations of the dip-direction of the *c*-axes in the individual crystal units. On the etched outer surface of the test wall, and on the vertical fracture plane in *Melonis zaandamii* (Pl. 5, Fig. 2) and *Cassidulina laevigata* (Pl. 6, Figs. 3, 4), it is distinctly seen how the needle-shaped crystallites in all crystal units have a constant dip of  $45^\circ$ ; the dip-direction is highly variable. This means that each crystal unit has undergone a rotation in relation to the adjacent crystal units.

## SUMMARY

- (1) There is strong evidence in favour, that the granular type of the test wall is composed of similar stacks of microcrystals, as previously described, in the radial type. However, in the granular type, the microcrystal columns seem to be oblique and not vertical as in the radial type.
- (2) The columns of microcrystals strongly resemble, both in habit and in optical characters, limbs of inorganic dendrites. This is further indicated by the occurrence of twinning between adjacent microcrystal columns described as a "herringbone pattern". Such a phenomenon is a common peculiarity in optically parallel orientated limbs of inorganic dendrites.
- (3) Columns of microcrystals form crystal units which are separated from each other by organic membranes. These membranes were obtained well preserved, by etching the test with a chromium sulphate solution, and their morphology was briefly described and illustrated.
- (4) A new method for the determination of the optical orientation of the *c*-axes in foraminiferal test walls has been introduced here. This method is based on the etching of calcite crystals with a glutaraldehyde solution, and is particularly useful for the granular type of the test wall where the determination of the *c*-axes orientation has hitherto been difficult.

(5) The columns of microcrystals are etched by the glutaraldehyde solution preferentially along their *c*-axes. After a partial solution of the columns, acicular crystallites remain. The individual crystallites appear to represent the axial portion of the crystal columns. This condition clearly indicates that each microcrystal column is a vertical crystallographic continuum.

#### ACKNOWLEDGEMENTS

My sincere gratitude is expressed to Docent H. Mutvei of the Palaeozoological Department of the Swedish Museum of Natural History and the Palaeontological Institute, Uppsala University, Uppsala, Sweden, for supervising this study and his constant criticism of the manuscript. Further thanks is due Mr. N. Mateer of the Palaeontological Institute, Uppsala University for also critically reading the manuscript.

Technical assistance was given by Mr. Gustav Andersson of the Palaeontological Institute, Uppsala University, Uppsala.

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## PLATES

### Plate 1

Fig. 1. *Globobulimina turgida* (Bailey); fragment of the test wall showing radiate extinction. Crossed nicols,  $\times 60$ .

Fig. 2. *Nonian labradoricum* (Dawson); fragment of the test wall showing granulate extinction. Crossed nicols,  $\times 300$ .

Fig. 3, a—b. *Globobulimina turgida* (Bailey); a, entire test in transmitted light,  $\times 60$ ; b, the same test after 3 days in 25 % glutaraldehyde; transmitted light,  $\times 60$ .

Figs. 4—6. *Ammonia beccarii* (Linné).

4. Vertical fracture plane of the test wall showing large tabular rhombohedral microcrystals. Treated with sodium hypochlorite for 24 hours,  $\times 10\,000$ .

5. Etched inner surface of the test wall showing the typical pore-centered crystal units. Etched with 25 % glutaraldehyde at pH 4 for 10 minutes,  $\times 10\,000$ .

6. Fracture plane of the test wall showing needle-shaped crystallites vertical to the test surface. Etched as in Fig. 5,  $\times 10\,000$ .

### Plate 2

Figs. 1—5. *Globobulimina turgida* (Bailey).

1. Vertical fracture plane of the test wall. The tabular microcrystals are stacked in vertical columns. The edges of the microcrystals form three-sided pyramids on the inner test wall surface. Treated with sodium hypochlorite for 24 hours,  $\times 10\,000$ .

2. Outer surface of the wall viewed adjacent to the *c*-axes of the needle-shaped crystallites. Around the ellipsoidal pore openings, the structure is more compact. Etched with 25 % glutaraldehyde at pH. 4 for 20 minutes,  $\times 10\,000$ .

3. Vertical fracture plane of the test wall with tabular microcrystals stacked in vertical columns. Treated as in Fig. 1,  $\times 10\,000$ .

4. Needle-shaped crystallites on a etched vertical

fracture plane of the test wall. Note the orientation of the crystallites parallel to that of the *c*-axes. Treated as in Fig. 3,  $\times 10\,000$ .

5. Vertical fracture plane of the test wall. The arrows indicate changes in dip-direction of the microcrystals in adjacent crystal units. Treated as in Fig. 1,  $\times 10\,000$ .

### Plate 3

Figs. 1—4. *Globorotalia menardii* (d'Orbigny).

1. Three-sided calcite pyramids growing in the calcite crust of the outer test surface. The calcite crust is here at a less advanced growth stage than in Fig. 2. Treated with sodium hypochlorite for 24 hours,  $\times 11\,000$ .

2. Crystallites on the etched faces of a three-sided pyramid of the calcite crust. Note the orientation of the crystallites parallel to the pyramid axis and vertical to the test surface. Etched with 25 % glutaraldehyde at pH 4 for 5 minutes,  $\times 10\,000$ .

3. Vertical fracture plane of the test wall showing tabular wall orientated microcrystals. Treated with sodium hypochlorite for three hours,  $\times 10\,000$ .

4. Etched vertical fracture plane on the test wall showing needle-shaped crystallites orientated vertically to the test surface. Etched with 25 % glutaraldehyde at pH 4 for 20 minutes,  $\times 6\,000$ .

### Plate 4

Figs. 1—4, a—b. *Nonian labradoricum* (Dawson).

1. Fracture plane of the test wall showing microcrystals at high magnification. Treated with sodium hypochlorite for 3 hours,  $\times 10\,000$ .

2. Pattern of sutures on the outer test surface left after solution of organic membranes between the crystal units (compare with Fig. 3). Treated with sodium hypochlorite for 24 hours,  $\times 10\,000$ .

3. Outer surface of the test wall showing the organic membrane between crystal units (compare with Fig. 2). Etched with chromium sulphate for 20 minutes,  $\times 11\,500$ .
- 4, a—b. Etched vertical fracture plane of the test wall showing the needle-shaped crystallites (marked with arrows) inclined to the test surface. Etched with 25 % glutaraldehyde at pH 3.5 for 20 minutes,  $\times 10\,000$ .

### Plate 5

Figs. 1—6. *Melonis zaandamii* (van Voorthuysen).

1. Microcrystals on the outer surface of the test wall arranged with a rhombohedral face parallel to the test surface. Treated with sodium hypochlorite for 24 hours,  $\times 10\,000$ .
2. Etched outer surface of the test wall showing the different orientation of the acicular crystallites in adjacent crystal units. Note also the constant inclination of the crystallites toward the test surface. Etched with 2.5 % glutaraldehyde at pH 5.5 for 20 minutes,  $\times 3\,000$ .
3. Network of vertical organic membranes between the crystal units. The pore tubes often constitute part of the organic network (compare with precedent figure). Remnants of the collapsed horizontal membrane of the outer test surface are also visible. Etched with chromium sulphate for 20 minutes,  $\times 3\,000$ .

4. Vertical fracture plane of the test wall showing well orientated small sized microcrystals. Treated as in Fig. 1,  $\times 10\,000$ .
5. Etched inner surface of the test wall covered with an organic membrane. Etched as in Fig. 3,  $\times 3\,000$ .
6. Sutured pattern on the outer surface of the test wall, left by the dissolved organic membranes, separating the crystal units; treated as Fig. 1,  $\times 3\,000$ .

### Plate 6

Figs. 1—4. *Cassidulina laevigata* (d'Orbigny).

1. Equidimensional rhombohedral microcrystals through a vertical cross section of the test. Crystal units are not clearly distinguishable. Treated with sodium hypochlorite for 24 hours,  $\times 10\,000$ .
2. Microcrystals arranged with a rhombohedral face parallel to the outer test surface. Treated as in Fig. 1,  $\times 10\,000$ .
3. Etched outer surface of the test wall showing the different orientation of the acicular crystallites in adjacent crystal units. Etched with 2.5 % glutaraldehyde at pH 5.5 for 20 minutes,  $\times 10\,000$ .
4. Etched vertical fracture plane of the test wall showing the acicular crystallites (*c*-axes), marked with arrows, in the adjacent crystal units. Etched as in Fig. 3,  $\times 6\,000$ .



