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# A STUDY ON THE REMAINS OF ORGANIC COMPONENTS IN FOSSIL MOTHER-OF-PEARL

by Charles GRÉGOIRE (Liège).

(With 8 plates.)

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# A STUDY ON THE REMAINS OF ORGANIC COMPONENTS IN FOSSIL MOTHER-OF-PEARL (*),

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Disappearance of the original organic matter (conchiolin) in fossil mother-of-pearl has been generally reported (11, 10, 4). However, VON GÜMBEL (14) observed in decalcified nacreous layers of several tertiary shells (ammonites, *Nucula, Pinna, Mytilus, Trochus*) « zerrissene trübe Flocken und körnige Teile » (slashed opaque flocks and granular material). No information was given about the micro-architecture of these remains.

As shown previously (7, 5) conchiolin of mother-of-pearl from various living shells, decalcified and dissociated by the ultrasonic vibrations, resolves itself into perforated membranes and reticulated sheets with a lace-like appearance. Three main patterns of structures have been described in that material (7).

The present study is an attempt to detect by electron microscopy the traces of the organic structures in nacreous layers of 500 to 160,000,000 years old shells.

## MATERIAL AND METHODS.

Fragments of mother-of-pearl from the following fossils have been used :

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(*) An abstract of preliminary results appeared in *Arch. internat. Physiol. Bioch.*, 1958, **66**, 674.

*CEPHALOPODA*. — AMMONOIDEA : *Dactylioceras*, undet. sp., 6 specimens (Jurassic, slaty cardboard of lower Toarcian, Marne, France). NAUTILOIDEA : *Nautilus* sp. ≠ 1, *Nautilus* sp. ≠ 2 (Eocene); *Aturia* sp. (Oligocene, upper Rupelian, Belgium).

*GASTROPODA* : *Bolma rugosa, Turbinidae* (Pliocene, Italy).

*PELECYPODA* : *Mytilus edulis* L. (Pliocene); *Mytilus* sp. (Pliocene); *Pinna pectinata* L. (Pliocene); *Inoceramus dubius* SOWERBY (Jurassic, slaty cardboard, Toarcian); *Unio* sp. ≠ 1 (Holocene); *Unio* sp. ≠ 2 (Holocene, Antwerp); *Iridina spekii* WOODWARD (Holocene, Belgian Congo).

The nacreous layers of the ammonites, exposed on fragments of the drilling cores, were not detached from the piece of slate in which they were still partly embedded. The fragments of the other shells, gently brushed from dust and mud, were dipped mostly in saturated aqueous solutions of the disodium salt of ethylene-diamine tetra-acetic acid (sequestrene NA 2, Alrose Chemical Corporation, Providence, Rhode Island; titriplex III, Merck, Darmstadt, Germany). This organic chelating agent alters the textures of the organic materials embedded in hard mineralized tissues less than the acids (also used) (12, 5). In order to avoid contamination of the preparations by substances released by the other structures of the shells (periostracum, prisms), all the surfaces of the fragments but the nacreous ones, were protected from contact with the decalcifying agent by careful coatings with paraffine. The residues of mother-of-pearl, in suspension in the decalcifying fluid, were washed in several changes of distilled water and recovered by centrifugation (3,000 revolutions per minute during 20-45 minutes). Drops of the successive supernates and of the final sediment resuspended in a small amount of distilled water were deposited on copper mesh screens coated with films of formvar (0.1 per cent solution in ethylene dichloride). The loaded grids were shadowcast with palladium at angles varying from 11° to 30°, and examined with a R. C. A., E. M. U. electron microscope with, and mostly without, objective apertures. In several preparations, especially from holocene material, the sediments were dissociated by the ultrasonic vibrations (7), before being deposited on to the coated grids.

From the same fossils, positive replicas (double stage method) were prepared by the carbon replica technique of Bradley (3) before and after etching, from the innermost surfaces of the tests (especially in ammonites), from splinters of fracture, from polished surfaces of fragments variously orientated to the nacreous stratification. The successive steps of the procedure have been described elsewhere (5).

The ninhydrin test for proteins was carried out by Mrs. Dr. S. Bricteux-Grégoire on aqueous suspensions of nacreous residues from decalcified

*Nautilus, Aturia* and *Pinna*. Owing to the insufficient amounts of material available in the ammonites, chemical tests could not be performed on these specimens.

X-ray diffraction patterns were recorded on nacreous fragments of *Nautilus* and of *Aturia* by Mr. A. Lutts, using a camera type Seeman-Bohlin (diameter : 60 mm), X-radiation of cobalt K alpha ($\lambda = 1.79$ Å), and a reference spectrum of aluminium. The spectra of aragonite, calcite and of quartz, reported in table II, are those published by the American Society for Testing Materials (A. S. T. M.).

## OBSERVATIONS.

Macroscopy. — In the specimens used in the present study, the iridescence with hues varying with the species, characterizing mother-of-pearl, was perfectly preserved in all the ammonites, in *Bolma rugosa* (bright variegated, as vivid as in living *Turbinidae*), in *Nautilus* ≠ 1, *Unio* and *Iridina* (shining opalescent or pink creamy), in *Nautilus* 2 (bright red), in *Aturia* (silvery). The lustre was slightly faint in *Mytilus* and in *Pinna* (whitish opalescent). Iridescence was poorly preserved in *Inoceramus* (dull grey).

In the holocene material *(Unio, Iridina)*, the residues of decalcification of mother-of-pearl were soft and translucid iridescent membranes, as in living specimens. However, in contrast with the latter, the fossil membranes were dissociated at once by the ultrasonic vibrations. In the older fossil material, the remains, obviously in scarcer amount and brittle, consisted of tiny, translucid, incidentally iridescent *(Nautilus)* shreds of membranes, and sandy, frequently reddish-brown particles, floating in suspension in the chelating solution. A vigorous shaking was sufficient for crumbling these remains into iridescent suspensions of invisible particles, a result which, for recent material, requires such an energetic treatment as the ultrasonic vibrations.

The glistening aspect characterizing the aqueous suspensions of completely decalcified recent nacreous substance developed intensely in *Bolma, Iridina, Unio*. It was less apparent in the preparations of the other specimens.

In *Aturia*, the process of decalcification was accompanied by emission of a strong bituminous smell.

Microscopy. — In mother-of-pearl of living shells of molluscs, three patterns of structure have been provisionally recognized at the class level in the reticulated membranes or sheets of conchiolin disposed between the stratified lamellae and between the crystals of aragonite arranged side by side in each lamella (7, 5).

In the *nautiloid pattern*, a reticulum consisting of sturdy trabeculae, broad rounded or elongated openings of irregular outlines, has been recorded in the only still existing genus *Nautilus*. The *gastropod pattern* is characterized by a regular disposition of rounded or of polygonal holes of smaller diameter than those in *Nautilus*. The sieve- or lace-like appearance of the reticulum in that pattern depends upon the shape and the size of the trabeculae. In the *pelecypod pattern*, the reticulum is more compact than in the two other patterns. The trabeculae are more slender and twisted. The openings, of irregular outlines and size, generally much smaller than the holes in the two other patterns, are unequally dispersed in the sheets.

The observations reported below are based on the study of 1,160 micrographs.

*Ammonoidea*. — In the six specimens of jurassic, about 150,000,000 years old *Dactylioceras* sp. used, the preparations were replicas of the original innermost nacreous surface of the shell, exposed by fracture of the drilling core. In these replicas, as shown in fig. 3 and 7, the nacreous architecture appears unaltered : the tabular crystals of aragonite are disposed side by side in single layers within each of the superimposed lamellae.

In several regions of the surfaces, the plane of fracture of the drilling core coincided with areas of nacreous growth and the successive stages of formation of new nacreous layers could be observed. Rounded crystal seeds of various diameters, encircled by membraneous rings, are scattered on the surface of the large crystals belonging to the former nacreous layer, completely developed (fig. 4, 5, 6). Intense, possibly abnormal, proliferation of crystal seeds, aggregated into a mulberry-like elevation, was detected in a limited zone of the growing layer (fig. 9).

Reticulated sheets, variously preserved, cover the tabular surface of several crystals (fig. 1, 2, 3). The thick and short trabeculae of the fabrics, the broad and elongated openings in the reticulum (fig. 3) are distinctive features of the nautiloid pattern. The diameter of the trabeculae varies greatly : spherical tuberosities alternate with constrictions (fig. 1 : « iris rhizome » aspect, see 5). As shown in fig. 6 and 10, fragments of reticulum were found, in the areas of nacreous growth, in the cavities left in the surface of mother-of-pearl by accidentally detached crystal seeds, which had developed on this reticulum.

In several preparations, the reticulum appeared variously disintegrated. Its remains were flexuous shreds, flattened patches (fig. 6), groups of spherical papillae protruding above the surfaces (fig. 8 and 11). Similarities of aspect (fig. 4, 5, 8, 11) between papillae and crystal seeds described above are only apparent : as shown in fig. 8 (upper right angle of the polygonal depression), papillae correspond to the rounded tuberosities set free by dislocation of the trabeculae.

*Nautiloidea*. — Compared with the reticulated sheets described in recent specimens (*Nautilus macromphalus* SOWERBY, 7, plates 3 and 4; 5, plates 251 and 252), the structures resisting decalcification in the eocene, about 65,000,000 years old *Nautilus* sp. ≠ 1 and ≠ 2, were variously altered, swollen, collapsed into rugged agglomerates of particles, or were dissociated into coarse rounded grains, 15 milli-microns and more in diameter, or into bundles of fibrils. However, the characteristic features of the nautiloid pattern were recognized in some fragments (fig. 13). As shown in fig. 12, the systems of interlamellar and of intercrystallinic membranes have largely subsisted.

The residues of decalcification of the oligocene, about 45,000,000 years old *Aturia* sp., contained well preserved reticulated sheets with a typical nautiloid pattern. In the structure shown in fig. 14, short dumpy cylindrical trabeculae surround irregular openings partly obliterated by shrinkage during desiccation on to the film of formvar. Larger openings, encircled by bulging trabeculae, are scattered at regular distances in the otherwise homogeneous reticulum. This double system of openings and of trabeculae suggests a greater complexity of structure in the nacreous conchiolin texture of *Aturia* than in the living *Nautilus macromphalus*.

Alterations in the reticulum, induced in part by artifacts of preparation, occur in the shape of flattenings of the trabeculae (fig. 17), condensation of the reticulum into rugged masses, dissociation, as in *Nautilus*, into rounded particles. Another kind of alteration is illustrated in fig. 15 and 16 : the trabeculae resemble pebbles or fragments of corals, perforated by tiny sieve-pits of various diameters (3-5 millimicrons), sometimes gathered in small groups or in rosettes around a central larger opening. Other trabeculae appear studded with small tuberosities, a particularity of structure formerly observed on the trabeculae of the reticulum in *Nautilus macromphalus* (7, 5). Dissociation of the reticulum into bundles of fibrils was detected in some preparations.

As shown in the replicas (fig. 17, 18, 19), the reticulum described above corresponds to the sheets of conchiolin disposed between the mineral structures and preserved in many, if not all (fig. 18) regions of the nacreous layers.

*Gastropoda.* — In the pliocene, about 14,000,000 years old *Bolma rugosa,* the sieve-like perforated membranes shown in the replicas of etched polished surfaces, variously orientated, of the nacreous layers (fig. 20 to 24), resemble closely those described in several living *Turbinidae,* especially in the near genus *Astraea* (compare with 7, plate 12, fig. 3). The topographical relations between organic remains (in substantial amounts, in the present specimen) and mineral components of mother-of-pearl appear with a special clarity in fig. 24, in agreement with the schematic drawing reported previously (5, text fig. 1, p. 800).

*Pelecypoda.* — The dense texture of the pelecypod pattern, variously altered, was detected in the fragments of reticulated sheets exposed by corrosion or isolated from most specimens, including the jurassic *Inoceramus dubius* Sowerby (fig. 26, 27, 28).

Taxonomic differences, at a level lower than the class, were recorded in the pliocene *Pinna pectinata* L. (fig. 25). As reported recently (6), the texture of the nacreous conchiolin pattern in the living *Pinnidae* studied until now is characterized by an extremely dense fabric and by a great thinness of the reticulated sheets. These characters contrast with the relatively coarser consistency of the sheets in several other groups of Pelecypods (see fig. 30 to 33).

In the holocene, 500 to 15,000 years old material *(Unio, Iridina)*, the reticulum, though obviously altered in its resistance to mechanical dissociation (ultrasonic vibrations), does not differ greatly in its appearance from the lace-like structures observed in related genera of living material (compare fig. 29-33 to 7, plates 16 and 17).

## DISCUSSION.

1. — The three structural patterns characterizing the organic reticulated sheets of mother-of-pearl in living Cephalopoda, Gastropoda and Pelecypoda, have been found in the residues of decalcification of fossil nacreous layers from 500 to 160,000,000 years old shells, belonging to the corresponding classes. A nautiloid pattern was detected in ammonites, in which the early stages of nacreous growth could also be observed.

In the fossil material, as in living mother-of-pearl, the sheets alternate in the lamellar stratification with the mineral lamellae. They are interconnected with bridges, which cross the lamellae between the crystals of aragonite grown side by side in each lamella.

Similarity in texture and coincidence in topography give evidence that these structures in fossils may be identified with the nacreous conchiolin, variously altered by burial and also by unavoidable artifacts of preparation (mechanical tearings, flattenings, shrinkages, washing out and swellings).

2. — A matter of discussion is the present state of chemical composition of the mineral and organic components in this fossil material. Integral or partial preservation of the original substances, molecular replacement of these substances by new elements (pseudomorphose, 9), penetration of new elements, such as colloidal solutions of silicic acid (silicification) into the original structures, without destruction of the latter (impregnation, 9), are among the alternatives to be considered.

A) Mineral components. — The electron micrographs and the X-ray diffraction records show that secondary alterations of the original aragonite, such as recrystallization into calcite (calcitic epigeny) and silicification, with subsequent possible destruction of all organic matter (4), did not occur in the material used.

### TABLE I.

Comparison between the X-ray Spectra of Mother-of-Pearl (recorded in 1 Fragment of Nautilus ≠ 1, in 2 Fragments of Nautilus ≠ 2 and in 1 Fragment of Aturia), of Aragonite, of Calcite and of Quartz. The Figures represent the Interplana Distances, d (Å), determined by Mr. A. Lutts.

| Nautilus ≠ 1 | Nautilus ≠ 2 |  | Aturia | Aragonite | Calcite | Quartz. |
|---|---|---|---|---|---|---|
|  | A | B |  |  |  |  |
| — | — | — | — | — | 3.86 | — |
| 3.38 | 3.38 | 3.38 | 3.38 | 3.39 | — | — |
| — | — | — | — | — | — | 3.35 |
| — | — | — | — | — | 3.04 | — |
| 2.70 | 2.69 | 2.69 | 2.70 | 2.72 | — | — |
| — | — | — | — | 2.50 | 2.49 | — |
| 2.47 | — | 2.46 | 2.46 | — | — | 2,46 |
| — | — | 2.38 | 2.39 | — | — | — |
| 2.36 | 2,35 | 2.34 | 2.35 | 2.36 | — | — |
| — | — | — | — | — | 2.28 | — |
| 2.18 | 2.17 | 2.17 | 2.17 | 2.19 | — | — |
| 2.09 | 2.09 | 2.08 | 2.09 | 2.09 | 2.09 | — |
| 1.97 (1) | 1.96 (1) | 1.96 (1) | 1.98 | 1.99 | — | — |
| — | — | — | 1.96 (2) | — | — | — |
| — | — | — | — | — | 1.92 | — |
| 1.87 | 1.86 | 1.86 | — | 1.88 | 1.87 | — |
| 1.81 | 1.80 | 1.80 | 1.80 | 1.83 | — | 1.81 |
| 1.75 | 1.73 | 1.72 | 1.74 | 1.74 | — | — |
| — | — | — | 1.62 (3) | — | 1.60 | — |
| 1.55 | 1.54 | 1.54 | 1.55 | 1.56 | — | 1.54 |
| — | — | — | — | — | 1.51 | — |
| — | — | — | 1.46 | — | 1.48 | — |
| — | — | — | — | — | 1.44 | — |
| — | — | — | — | — | 1.43 | — |
| 1.40 | — | 1.39 | 1.40 | 1.39 | — | — |
| — | — | — | 1.35 (3) | — | 1.35 | 1.37 |
| — | — | — | — | — | — | 1.32 |

(1) intense ray; (2) very faint ray; (3) random coincidence between two spectra.

As reported in table I, the rays of maximal intensity recorded in the fragments of *Nautilus* ≠ 1 and ≠ 2 (A and B) and of *Aturia* (3.38 Å, 1.96 Å, 1.98 Å) coincide with those of aragonite (3.39 Å, 1.99 Å) and not with those of calcite (3.04 Å).

B) Organic components (conchiolin). — Preservation of delicate protidic structures, such as muscle striation and cell nuclei of eocene fishes and frogs, has been reported (13). Biochemical compounds, stable throughout geological times, have also been identified (see 8). Among the factors of preservation, burial and thermal history are more important than age (1, 2). Paper chromatograms (1, 2) of amino acids found in 1,000 (Pleistocene) to 25,000,000 years (Miocene) old shells of *Mercenaria mercenaria* and *Glycymeris parilis,* two species characterized by absence of mother-of-pearl, have shown a high thermal stability in various amino acids, while others had disappeared.

In the present material (table II), organic substance does not seem to be absent from the residues of decalcification, containing the reticulated sheets, of *Nautilus, Aturia, Bolma* and *Pinna*. Appreciable variations, a frequent finding in fossils (4), were observed in the organic content in different samples of a same shell (*Nautilus* ≠ 1 and ≠ 2). However, bacterial contamination of the specimens contributed likely in part for the amounts recorded. Germs, scarce or absent in the preparations of *Nautilus* ≠ 1, *Aturia* and *Bolma*, were detected by electtron microscopy in *Nautilus* ≠ 2 (second sample only) and in *Pinna*, explaining the relatively high figures recorded in these samples.

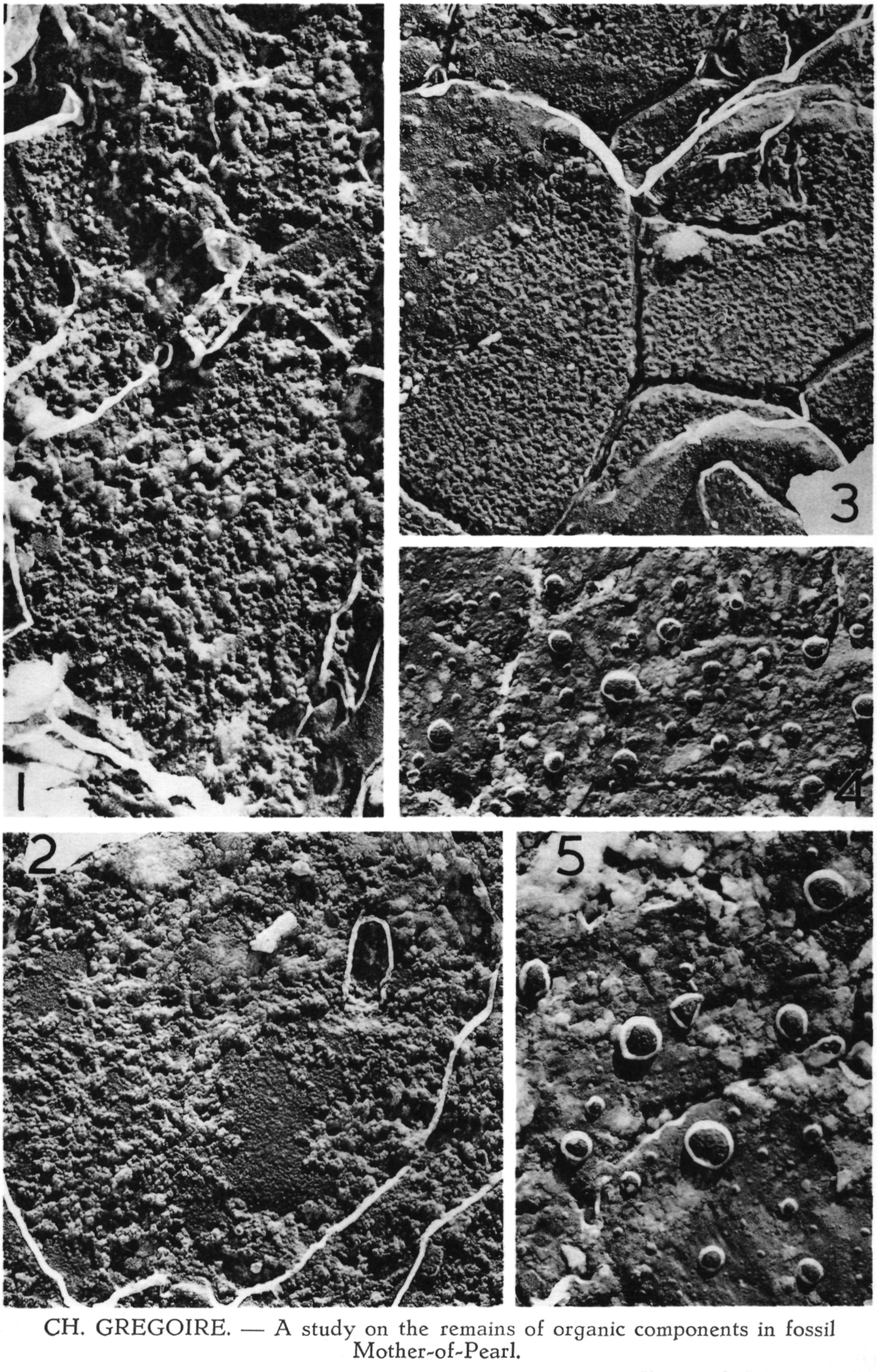
TABLE II.

Amino Acid (or NH 3) and α Amino Nitrogen Contents in Fragments of fossil Mother-of-Pearl, measured by the Ninhydrin Method by Mrs. Dr. S. Bricteux-Grégoire.

| Material | Amino Acid (or NH 3) content | α Amino Nitrogen content |
|---|---|---|
| | *micromols per c.c.* | *micrograms per c.c.* |
| *Nautilus* sp. ≠ 1 (two samples) | 0.040/0.023 | 0.56/0.32 |
| *Nautilus* sp. ≠ 2 (two samples) | 0.400/0.630 | 5.54/8.80 |
| *Aturia* sp. ... ... ... ... ... ... ... | 0.040 | 0.56 |
| *Bolma rugosa* ... ... ... ... ... ... | 0.230 | 3.22 |
| *Pinna pectinata* L. ... ... ... | 0.890 | 12.40 |

In the present study, the degree of preservation of the conchiolin cannot be appreciated, but only in morphological terms. The brittleness observed in the residues of decalcification from the older material (Ammonites, *Nautilus, Aturia, Bolma*) and the easier dislocation by ultrasonic vibrations of the membranes from the holocene nacreous layers *(Iridina, Unio)*, compared to the living material from the same genera, suggest that the nacreous conchiolin, even intact in its texture, has not been integrally preserved in its composition.

In that respect, trabeculae, spontaneously dissociated into bundles of flexuous fibrils *(Nautilus, Aturia)* have been incidentally observed in the present material. These structures resemble closely the fibrillar residues of living nacreous conchiolin after successive extractions by a borate buffer and by sodium hydroxide of watersoluble constituents of the protidic material. In that fibrillar material, relatively high concentrations in alanine and in glycine have been recorded (7). These compounds belong precisely to the group of the most stable amino acids in fossil shells (1, 2). Alterations in the original organic structures of fossil mother-of-pearl,



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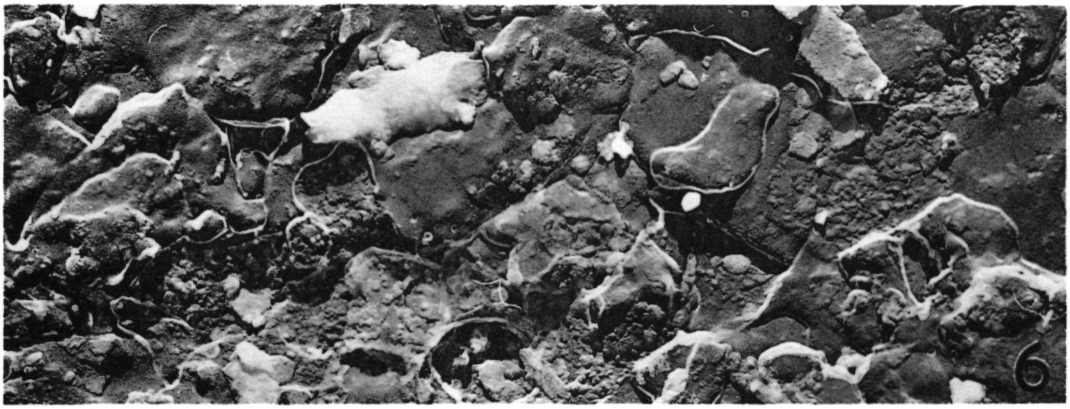
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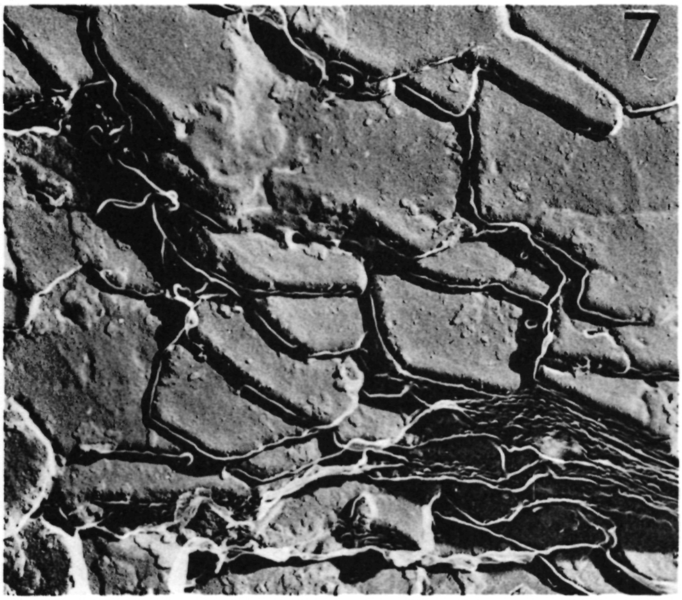
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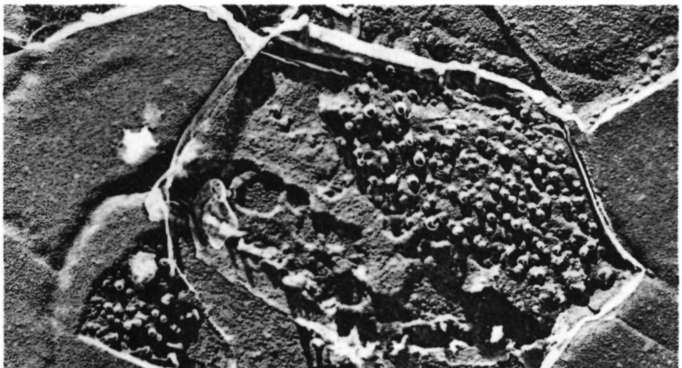
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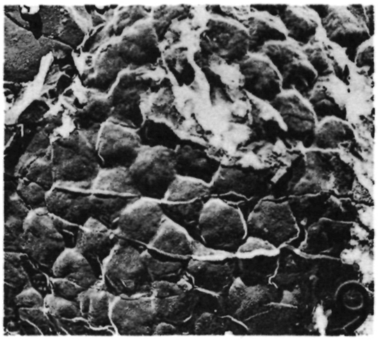
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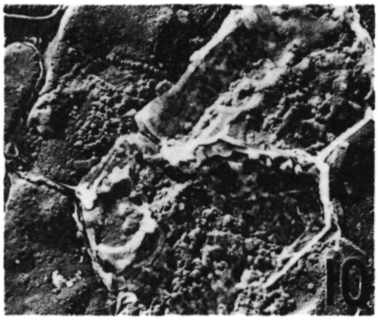
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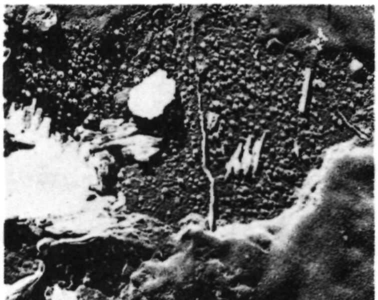
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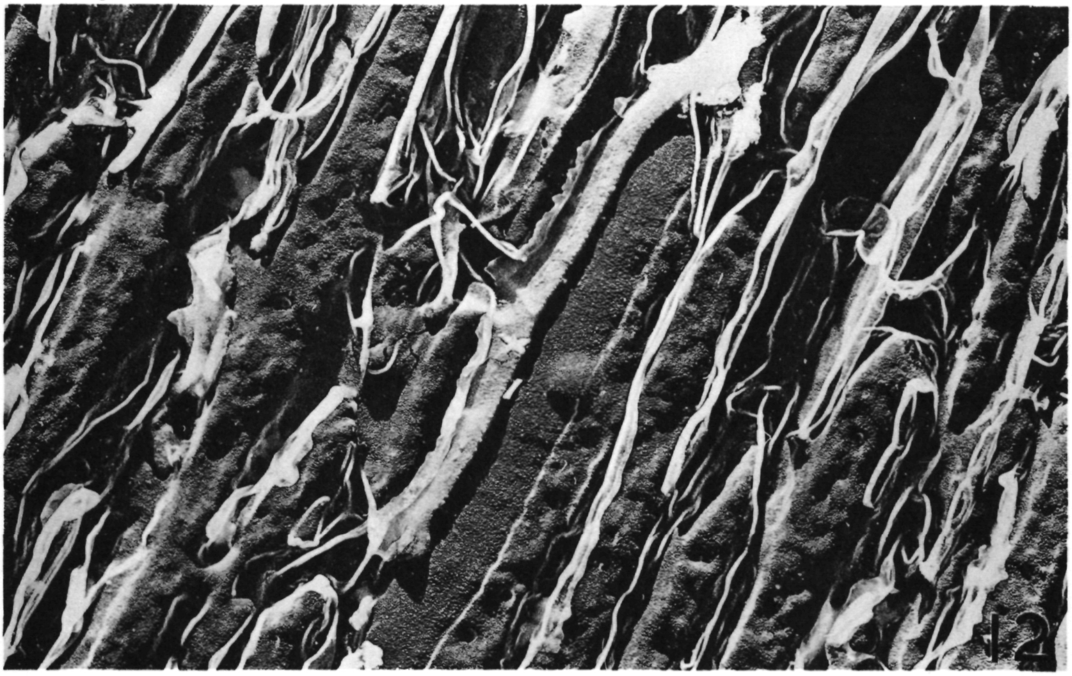

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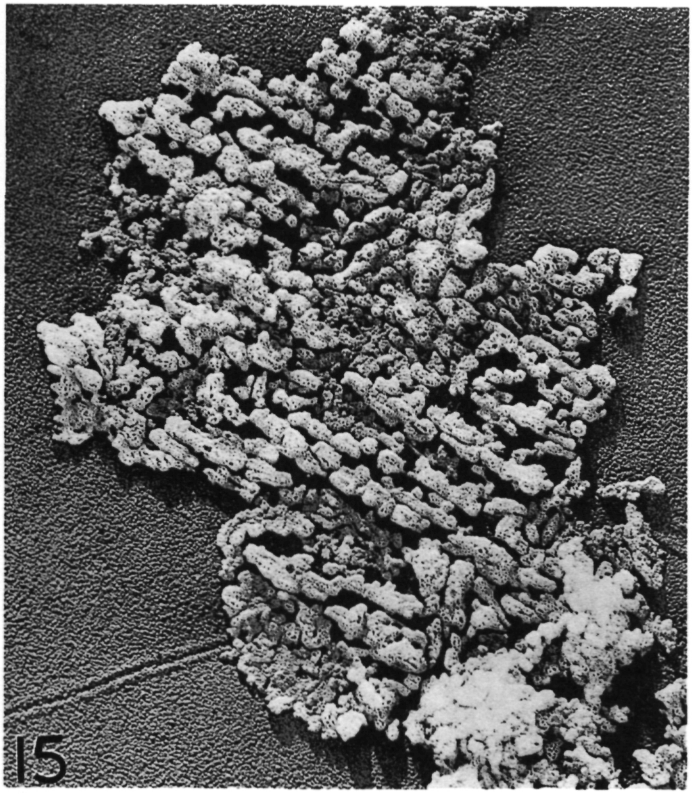

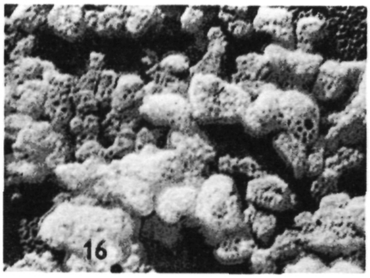

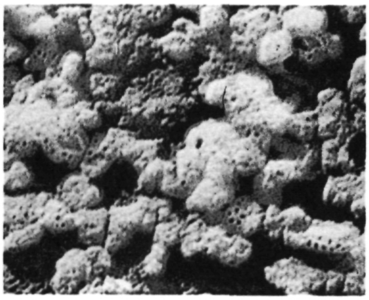

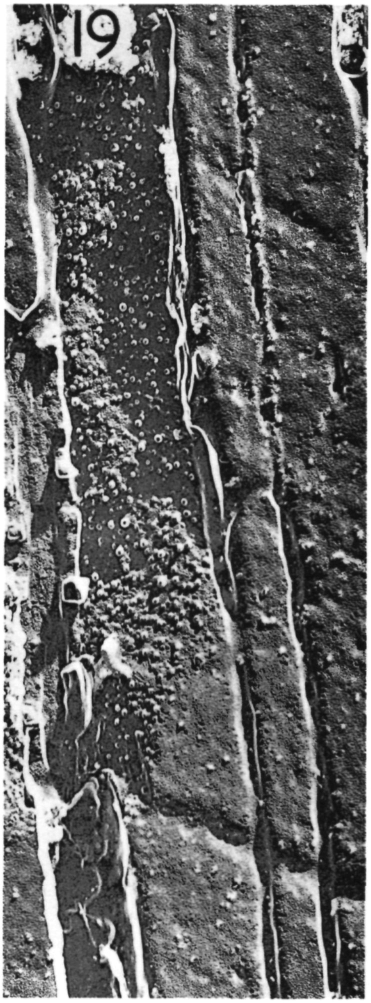

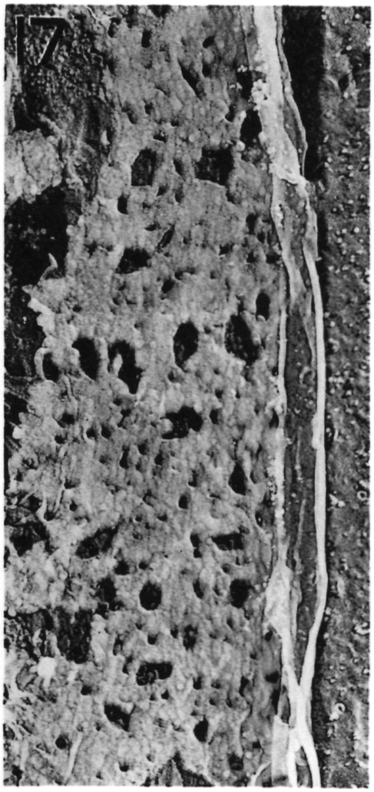

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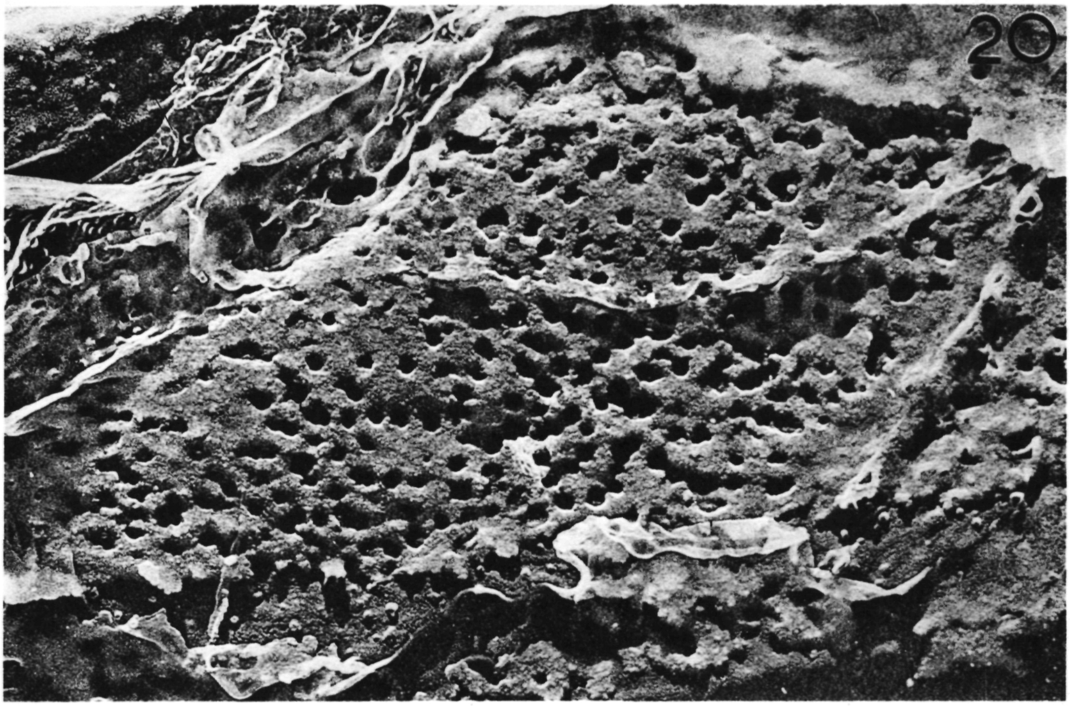

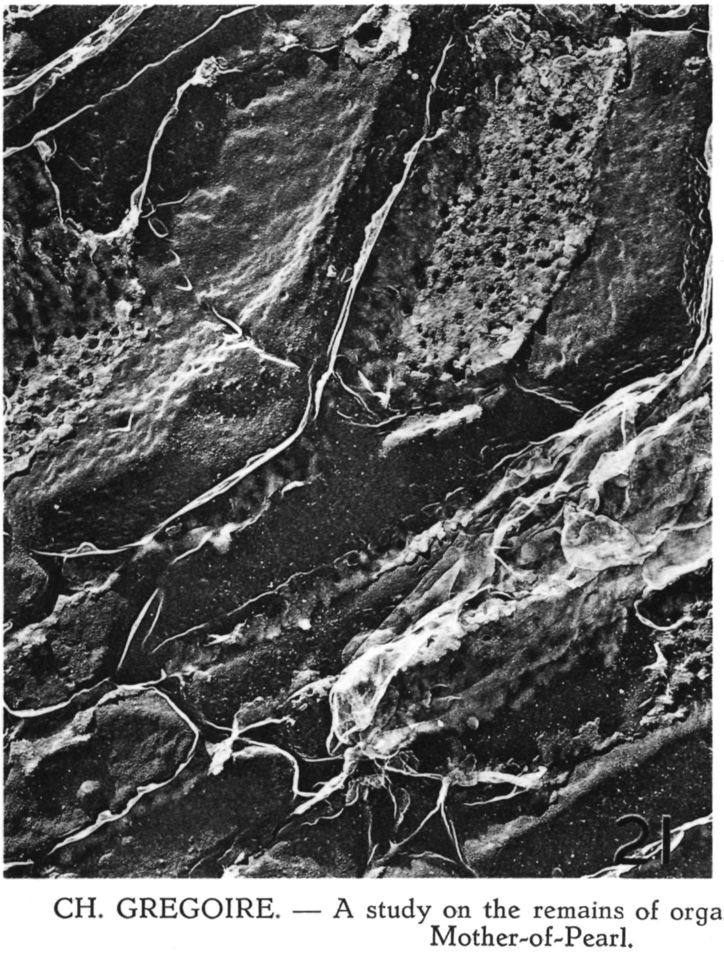

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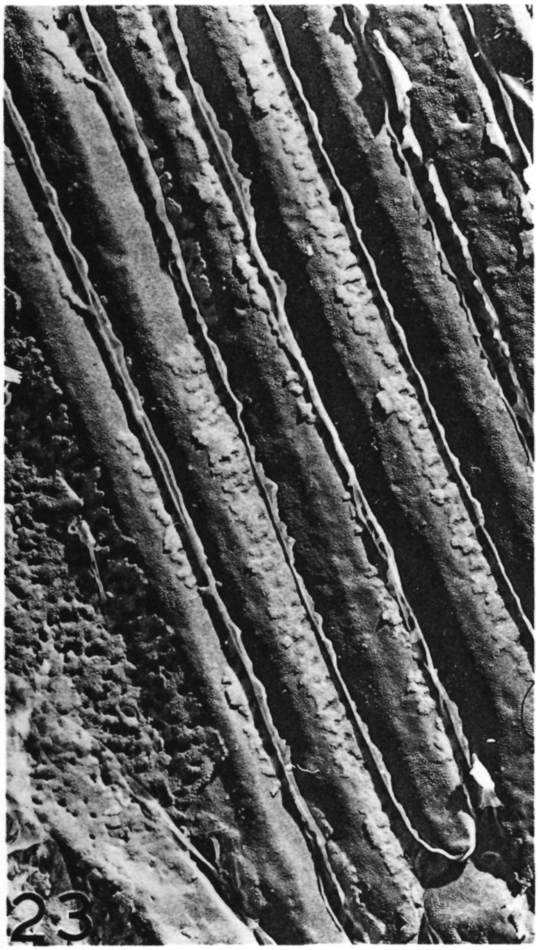
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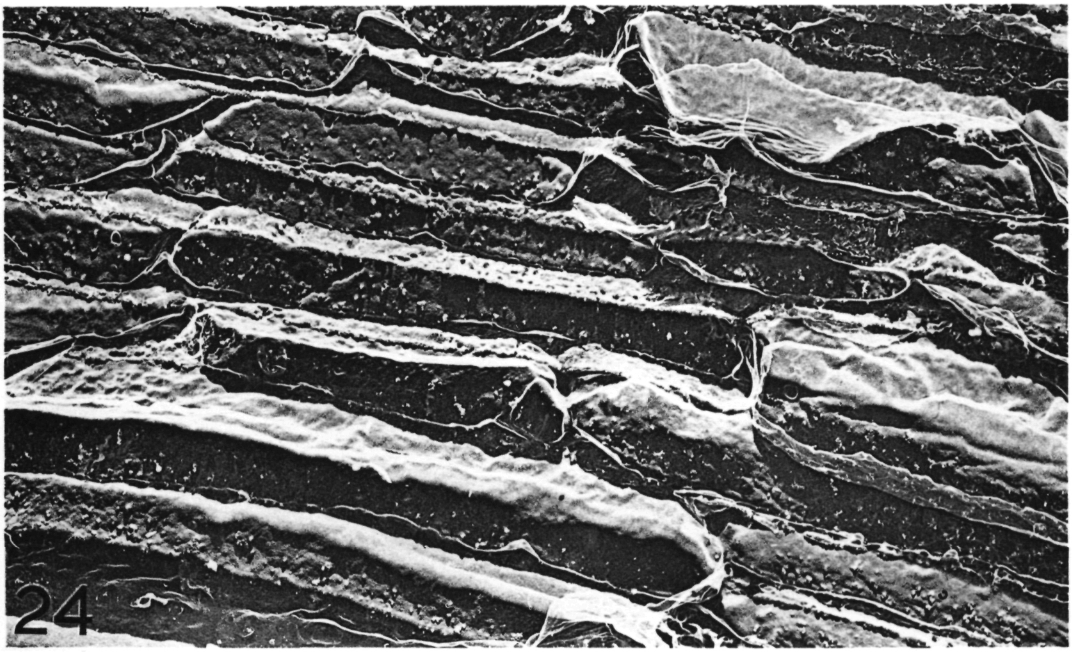
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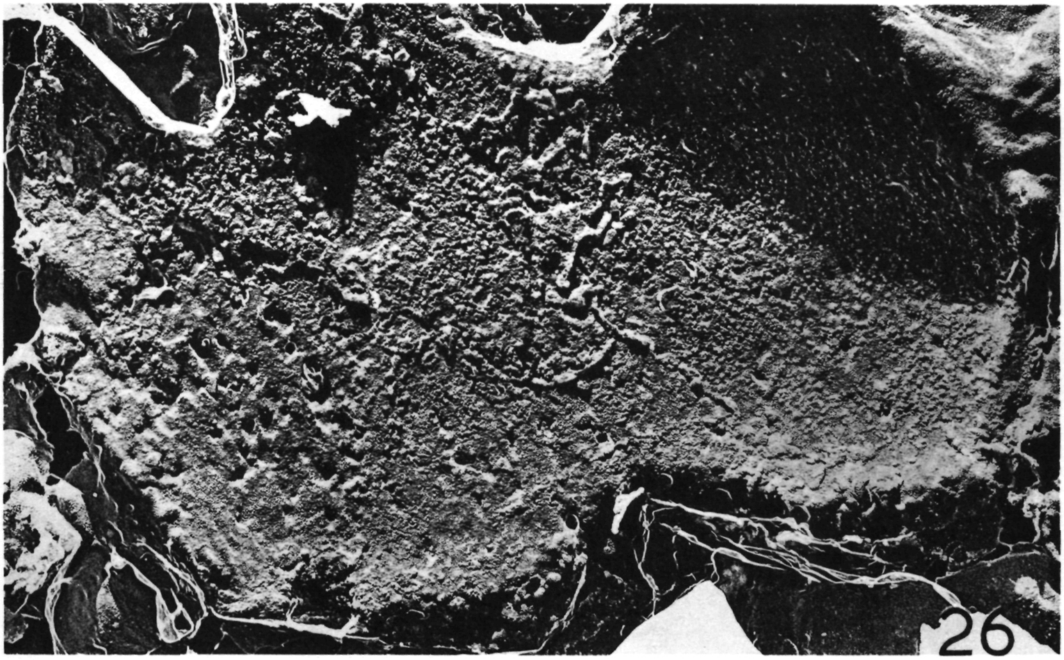
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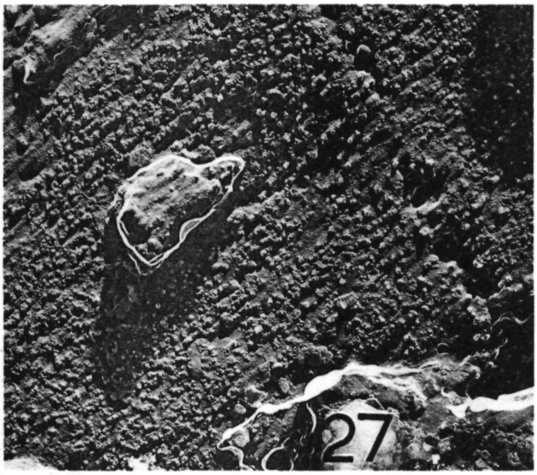
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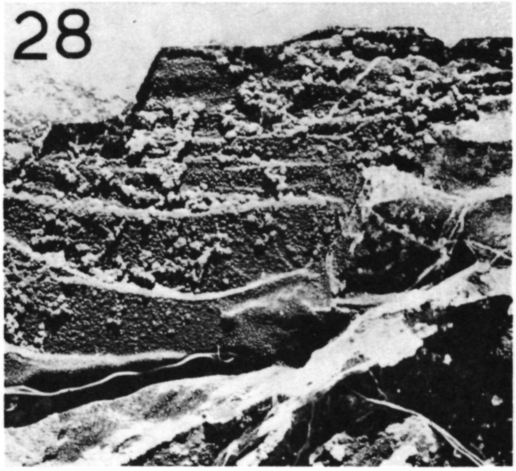
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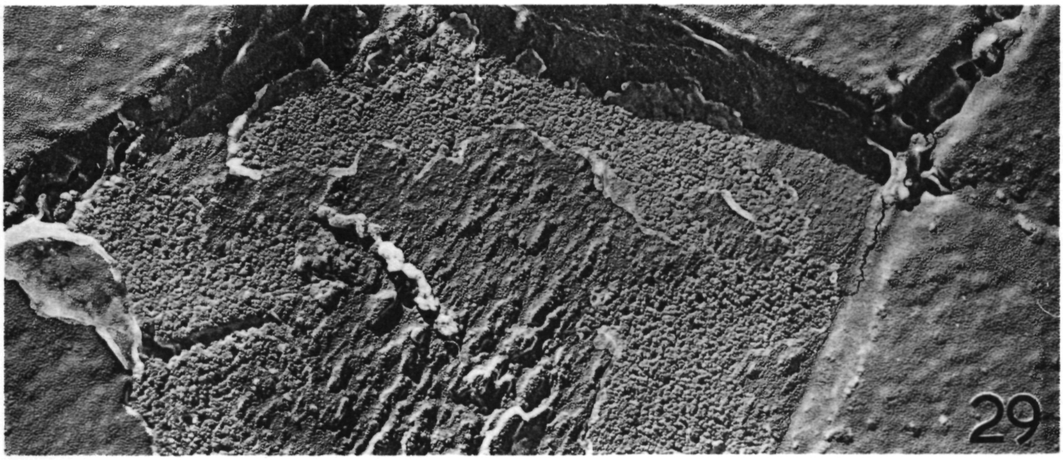
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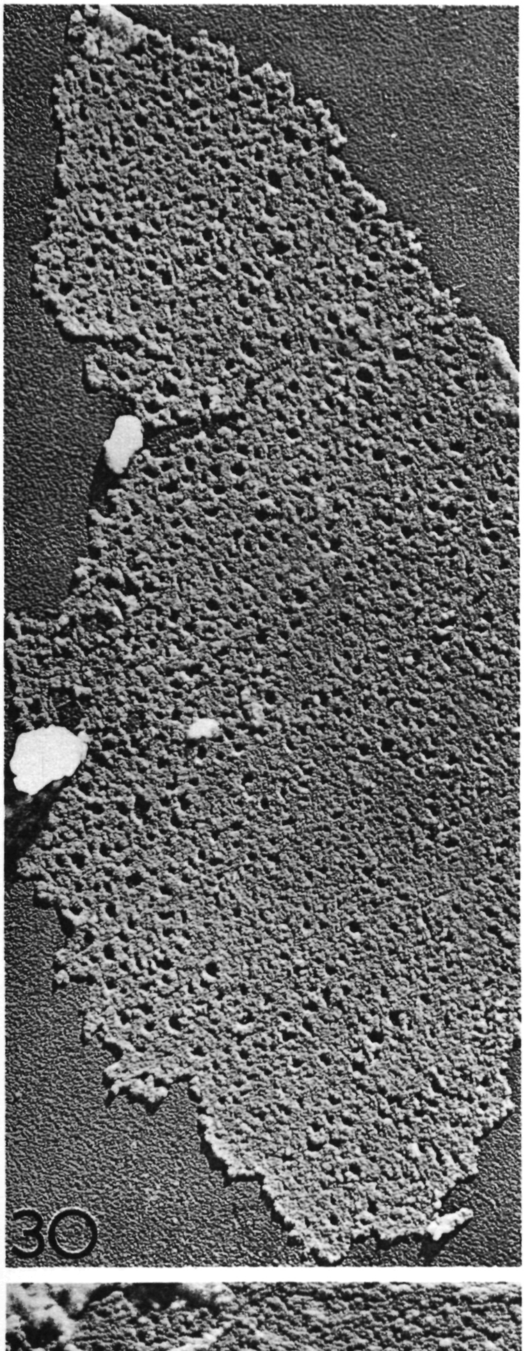

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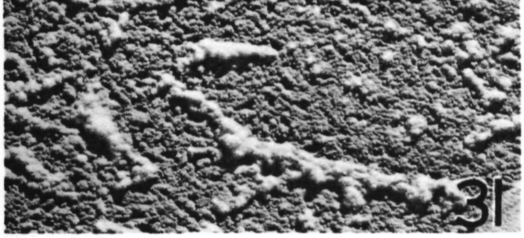

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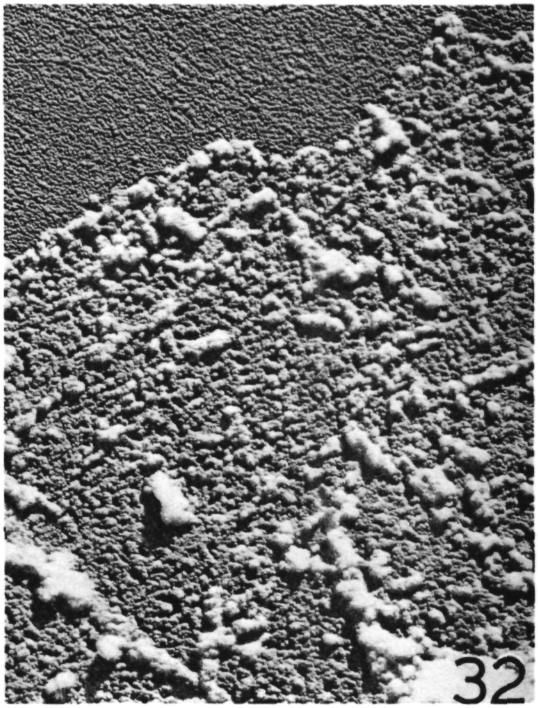

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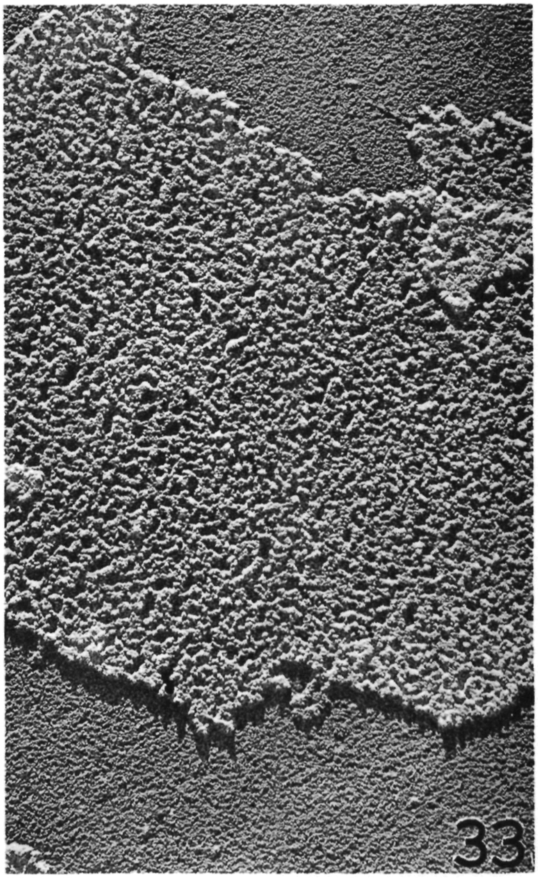

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namely decrease in mechanical cohesiveness and spontaneous dissociation into fibrils, might result from losses of unstable amino acids.

The nature of the alterations detected in the reticulated sheets of *Aturia* (fig. 15 and 16) is provisionally unsolved. Unshadowed preparations of this material were opaque under the beam, which suggests the presence in the altered trabeculae of heavy electron scattering material. The pebble-like structures characterizing these alterations are obviously different from fragments of incompletely decalcified crystals of aragonite. Such fragments, resembling closely those illustrated in fig. 11, appear in the shape of rods or sticks frequently arranged in parallel rows, and are fixed together by bridges of mineral substance still undissolved.

Pseudomorphose or impregnation, especially silicification, of the original organic substance might also be involved in these alterations. However, as shown in table I, the most intense ray characterizing the spectrum of silicium in quartz (3.35 Å) was absent from the spectra of mother-of-pearl in *Aturia*.

## SUMMARY.

1. — Mother-of-pearl of fossil shells (Ammonoidea, Nautiloidea, Gastropoda, Pelecypoda) has been studied by electron microscopy.

2. — The preparations included residues of decalcification of nacreous fragments, positive metallic replicas of the original innermost nacreous surfaces of the shells, of planes of fracture, of surfaces polished in various orientations, before and after etching with a chelating agent.

3. — The residues consisted of fragments of reticulated sheets or of perforated leaflets, disposed, as shown in the replicas, between the parallel mineral lamellae, and in each lamella, between the single crystals of aragonite.

4. — The three patterns of structure, characterizing, at the class level, the organic components of mother-of-pearl, and described previously in living Cephalopoda, Gastropoda and Pelecypoda, have been recognized in their distinctive features in the residues of decalcification of mother-of-pearl in fossils from the corresponding groups.

5. — In the ammonites, the early stages of formation of new nacreous layers could be observed on replicas of the original zones of growth, set free by fracture of the drilling core.

6. — The present results show that conchiolin of mother-of-pearl, with its morphological patterns preserved through considerable periods of time, may be ranged in the category of structures, reported in the paleontological literature, characterized by a high degree of stability.

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## EXPLANATION OF PLATES.

The fig. 13-16, 25, 30-33, are micrographs of residues of decalcified fossil mother-of-pearl, dissociated mechanically into fragments of perforated leaflets and of reticulated sheets. Drops from aqueous suspensions of these fragments were deposited on to copper mesh screens, previously coated with films of formvar.

The fig. 1-5, 6-11, 17-19, 20-24, 26-29, are micrographs of positive carbon replicas of the innermost nacreous surfaces of shells, of regions of fracture inside mother-of-pearl, of nacreous surfaces polished in various orientations, before and after etching with a chelating agent (sequestrene NA2 and titriplex III) during various lengths of time (indicated in brackets).

All the preparations, sediments and replicas, were shadow-cast with palladium, at angles varying from 11 to 30°. The prints are negative throughout and the shadows appear black.

### PLATE 1:

*Dactylioceras* sp. (Cephalopoda, Ammonoidea). Jurassic, lower Toarcian, about 150,000,000 years old.

*Fig. 1 and 2.* — Specimen ≠ 3. Original surface of the innermost nacreous layer, exposed by fracture of the drilling core. A reticulated sheet, better preserved in *fig. 1* than in *fig. 2,* covers the tabular surfaces of the crystals of aragonite constituting the mineral leaf or lamella underneath. The pattern of the reticulum is nautiloid. The diameter of the trabeculae varies greatly: globular tuberosities alternate with narrowings (« iris rhizome » aspect). The white ribbon (*fig. 2*) corresponds to membranes which separate the crystals of aragonite and cross the mineral lamellae (× 24,000).

*Fig. 3.* — Specimen ≠ 4. Innermost nacreous layer, after corrosion with sequestrene (90 seconds).

The texture of the reticulum, freshly set free from the protecting mineral by etching, appears less disorganized than in *fig. 1 and 2* (× 17.000).

*Fig. 4 and 5.* — Specimen ≠ 1. The early steps of formation of a new nacreous layer are shown in a region of the original surface of the innermost nacreous layer of the shell. About 80 (*fig. 5*) and 100 (*fig. 4*) rounded seeds of crystals of aragonite appear scattered on the mineral background, partly covered with debris of sheets, of the formerly fullgrown lamella. The seeds, at different stages of their growth, are surrounded by membraneous material (white rings). The smallest seeds visible on the picture have a diameter of about 17 millimicrons. *Fig. 4* (× 12,000); *fig. 5* (× 17,000).

### PLATE 2:

*Dactylioceras* sp. (continued).

*Fig. 6, 10, 11.* — Specimen ≠ 3. Original innermost surface of the shell, coinciding with the plane of fracture of the drilling core. *Fig. 6 and 10:* in a new nacreous layer in the process of formation, crystal seeds, more developed than those represented in *fig. 4 and 5,* are shown. Flattened debris of reticulated sheets are scattered on the mineral background. Accidental tearing away of crystal seeds leaves pits, in which the reticulated sheets, on which the crystals were growing, are disclosed. See *Fig. 6,* below the white polygonal spot corresponding to a pseudoreplica of a mineral fragment, and especially *fig. 10,* in which the trabeculae of the reticulum, connected to pericrystallinic membranes encircling originally the detached seed, are clearly visible.

*Fig. 11* shows a breach of the surface in another region of the same specimen. Rounded papillae, remains of a reticulated sheet, and rod-like fragments of crystals of aragonite are shown together (see the text) (× 17,000).

*Fig. 7.* — Specimen ≠ 5. *In a region of fracture, corroded with sequestrene* (30 seconds), the terrace-like disposition of about ten superimposed consecutive mineral lamellae is shown. Membraneous material, set free by corrosion, is visible between the crystals of aragonite, and in the shape of parallel leaflets inside a breach (bottom right), where the mineral substance has completely disappeared (× 10,000).

*Fig. 8.* — Specimen ≠ 6. Following tearing away of a splinter corresponding to a *polygonal crystal of aragonite, from the original innermost surface of the shell,* remains of a reticulum appear in the shape of papillae and of fragments of trabeculae, still partly embedded inside the mineral substance. At the upper right angle of the polygonal cavity, debris of trabeculae are visible, in which papillae appear as tuberosities detached from the reticulum. Intercrystallinic membranes are shown at the boundaries of the detached crystal (× 24,000).

*Fig. 9.* — Specimen ≠ 5. Corrosion with sequestrene (90 seconds). A mulberry-like mound, consisting of a close agglomeration of crystal seeds, was detected on the innermost surface of this shell. The finding suggests unusual conditions of crystal growth in that limited region of a new nacreous layer (× 10.000).

## PLATE 3 :

*Fig. 12.* — *Nautilus* sp. ≠ 1 (Cephalopoda, Nautiloidea). Eocene (about 65,000,000 years old).

Polished shell. Plane of polishing tranverse to the surface of mother-of-pearl, cutting about 14 consecutive nacreous lamellae. Heavy etching with sequestrene (150 seconds).

A substantial system of interlamellar and intercrystallinic membranes, variously folded, torn or erected, is shown. A few large openings are visible in the membranes collapsed on to the section of crystals (center) (× 24,000).

*Fig. 13.* — *Nautilus* sp. ≠ 2. Debris of a reticulated sheet, in which the nautiloid pattern is still recognizable. Compare with ref. 7, plates 3 and 4 (× 36,000).

*Fig. 14.* — *Aturia* sp. (Cephalopoda, Nautiloidea). Oligocene (about 45,000,000 years old).

A well preserved reticulated sheet exhibits the typical nautiloid pattern. Large openings, encircled by thick trabeculae and scattered at regular distances in the otherwise homogeneous reticulum, suggest that the organic components of mother-of-pearl have a more complex architecture in *Aturia* than in the living *Nautilus* studied previously (× 36,000).

## PLATE 4 :

*Aturia* sp. (continued).

*Fig. 15 and 16.* — Pebble — or coral — like alteration in a reticulated sheet. The oval or rod-like trabeculae appear tapped with tiny openings, frequently arranged in rosettes around a larger hole *(fig. 16). Recordings of the intermediary stages of* transformation of the reticulum represented in *fig. 14* into the structures shown in *fig. 15-16* give evidence that the structures are not fragments of incompletely decalcified crystals of aragonite. *Fig. 15* (× 36,000); fig. 16 (× 71,000).

*Fig. 17.* — Polished shell. Plane of polishing tangential to the surface of the mineral nacreous lamellae. Etching with sequestrene (4 minutes).

A substantial reticulated sheet is exposed on the mineral background. In spite of alterations (flattening, shrinkage), the features shown in *fig. 14* are still recognizable (× 17,000).

*Fig. 18 and 19.* — Polished shell. Plane of polishing transverse to the surface of the nacreous lamellae. Etching with sequestrene (4 minutes).

Interlamellar membranes protrude in the grooves between the successive lamellae. In *fig. 19,* remains of a membrane in the shape of spherical papillae were disclosed by tearing away of a fragment of crystal. *Fig. 18* (× 17,000); *fig. 19* (× 24,000).

## PLATE 5 :

*Bolma rugosa* (Gastropoda, *Turbinidae)* Pliocene (about 14,000,000 years old).

*Fig. 20 and 22.* — Polished nacreous region of the shell. Plane of polishing tangential to the nacreous stratification. Etching with sequestrene (*fig. 22 :* 60 seconds; *fig. 20 :* 150 seconds).

A fragment of a reticulated sheet exposed on a crystal surface exhibits the gastropod pattern, closely similar to that recorded previously in the living *Astraea,* a genus near *Bolma* (compare with ref. 7, plate 12). *fig. 20* (× 35,000) *fig. 22* (× 24,000).

*Fig. 21.* — Polished nacreous region of the shell. Plane of polishing oblique to the nacreous stratification. Etching with sequestrene (150 seconds).

The topography of the systems of interlamellar (fragments of well preserved reticulated sheets covering the tabular surfaces of the crystals) and intercrystallinic membranes is shown in six consecutive lamellae (× 17,000).

## PLATE 6 :

*Fig. 23 and 24.* — *Bolma rugosa* (continued). Nacreous region polished in transverse orientation. Etching with sequestrene (*fig. 23 :* 150 seconds; *fig. 24 :* 4 minutes).

Brickwall appearance, with integral preservation of the conchiolin structure between the lamellae and between the crystals. The gastropod pattern is visible in the fragments of membranes collapsed on to the sections of the crystals (*fig. 23*) and especially in the membranes wrapping the crystal, at the center of *fig. 24. Fig. 23* (× 17,000); *fig. 24* (× 10,000).

*Fig. 25.* — *Pinna pectinata* L. (Pelecypoda, *Pinnidae*) Pliocene (about 14,000,000 years old).

The structure of the reticulated sheets described in the living *Pinnidae* (6), and which, at a subclass level, differs distinctly from that characterizing several other families of Pelecypoda (see plate 8), is shown in this fossil specimen. Fibrils are recognizable in the reticulum (× 36,000).

## PLATE 7 :

*Fig. 26, 27 and 28.* — *Inoceramus dubius* SOWERBY (Pelecypoda, *Pernidae*) Jurassic (about 160,000,000 years old).

Innermost nacreous surface of the shell, exposed by fracture of the drilling core. Etching with sequestrene (60 seconds).

Variously altered remains of a reticulum, exhibiting the characters of the pelecypod pattern (dense texture, slender trabeculae) cover the surface of crystals. *Fig. 26 and 27* (× 17,000); *fig. 28* (× 25,000).

*Fig. 29.* — *Iridina spekii* WOODWARD (Pelecypoda, *Mutelidae*) Holocene.

Nacreous layer of the shell polished tangentially to the lamellar stratification. Etching with sequestrene (75 seconds).

Fragments of interlamellar membranes (pelecypod pattern) cover the surface of an underlying crystal (× 24,000).

PLATE 8 :

*Fig. 30.* — *Unio* sp. ≠ 2 (Pelecypoda, *Unionidae)* Holocene (500 to 15,000 years old). Fragment of a reticulated sheet (pelecypod pattern) (× 35.000).

*Fig. 31 and 32.* — *Unio* sp. ≠ 1. In this specimen, less preserved than ≠ 2, swelling, shrinkage and folding were possibly involved in altering the still distinct pelecypod pattern of the sheets. In *fig. 31,* fibrils (also detectable in fig 30), are visible in the fabric (× 36,000).

*Fig. 33.*— *Iridina spekii* WOODWARD, Holocene.

Fragment of a reticulated sheet of conchiolin (pelecypod pattern) (× 36,000).

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