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OKLAHOMA GEOLOGY NOTES

CHARLES WELDON TOMLINSON

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Cover Picture

Charles Weldon Tomlinson

No other man is better known for his geological work in southern Oklahoma than Charles W. (Tommy) Tomlinson of Ardmore, who worked tirelessly for 40 years to understand better the complex surface and subsurface geology of this oil-rich province. In addition to being an outstanding student and pure scientist, he was a highly successful independent petroleum geologist and producer, a philanthropist, a willing worker in civic and religious activities, and an ardent, long-time member of the American Association of Petroleum Geologists.

Tomlinson was born in Detroit in 1892. He earned Bachelor's and Master's degrees at the University of Wisconsin and, in 1916, a Ph. D. from the University of Chicago. He lived in Ardmore from 1920 to his death on April 3, 1960, easily becoming Ardmore's favorite son and one of its most distinguished citizens, and at the same time devoting most of his productive life to a study of the structure, stratigraphy, and petroleum geology of southern Oklahoma. He was a charter member and later president of the Ardmore Geological Society, but his greatest public recognition was in being elected president of the American Association of Petroleum Geologists (1949-50), the largest organization of geologists in the world.

Dr. Tomlinson's best-known reports, most of which were published by the A. A. P. G., were those on southern Oklahoma, including descriptions of the Graham field, natural-gas pools, geologic structures, and Pennsylvanian sediments and orogenies. He contributed one of the best chapters (Carter County) to Oklahoma Geological Survey Bulletin 40, on the petroleum geology of Oklahoma, and he was the author of Oklahoma Geological Survey Bulletin 46, entitled *The Pennsylvanian System in the Ardmore Basin,* which established that region as a classic field for study of Pennsylvanian strata.

Professional memberships of Dr. Tomlinson attest to his varied interests. The American Association of Petroleum Geologists attracted much of his attention and time, but he also was active in the Geological Society of America, American Institute of Mining and Metallurgical Engineers, Society of Economic Paleontologists and Mineralogists, American Association for the Advancement of Science, American Petroleum Institute, and Independent Petroleum Association of America.

All tourists traveling through the Arbuckle Mountains on U. S. Highway 77 will come in touch with Tomlinson without ever having known him, for he provided the guiding hand in the erection of the geologic signs and cross section for which this highway is famous.

Mrs. C. W. (Maude R.) Tomlinson of Ardmore furnished the cover picture, which was used on a passport in the 1950's.

—W. E. H.

Conchiolin Membranes in Shell and Cameral Deposits of Pennsylvanian Cephalopods, Oklahoma

Charles Grégoire* and Curt Teichert†

Introduction
(by Curt Teichert)

Asphalt-impregnated limestones in Oklahoma contain fossils that in recent years have yielded much new and important information on organic components in the shell and camerae of cephalopods. These results have been communicated in widely scattered papers, published in three countries, and, because it is the purpose of this article to describe and illustrate the electron-microscopic structures of organic tissues found in these cephalopods, a general description of the occurrence and a brief review of the results obtained by other investigators is presented first.

The cephalopods discussed here come from an asphalt quarry which was first described by Eldridge (1901, p. 283-284, 295-297), who noticed the presence of "nacreous shells" in some asphaltic limestone beds. However, he did not indicate the affinities of the fossils and gave no names to them. He referred to the quarry as "No. 2 quarry of the Gilsonite Paving and Roofing Company." It is situated about 3 miles south of Sulphur in southern Oklahoma. From a stratigraphic section (fig. 1; reproduced from Eldridge, 1901, p. 282) it would appear that the shells occur throughout a thickness of about 20 feet of strata. According to Eldridge, they are mostly concentrated in two layers: the topmost limestone bed (no. 6 of Eldridge, p. 283) and in the upper 2 feet of the lowest limestone bed (no. 9) which is 10 feet thick. In this bed shells decrease in number downward and are almost absent in the lower half.

The fauna from this locality was presented monographically by Smith (1935) in a doctoral dissertation which, unfortunately, remains unpublished.

Smith was able to add little to Eldridge's description of the section because the outcrop had deteriorated since 1901. He observed, however, that "some of the limestone is so heavily impregnated that the rock seems entirely composed of asphalt and nacreous shell material. Such rock is quite brittle, breaks with a conchoidal fracture, and is a deep brown, almost black, color." According to Eldridge (p. 295), the bitumen content of the rock averages between 14 and 15 percent.

Smith noted that the most numerous fossils are straight nautiloids, almost equalled in number by pelecypods. Next in order of abundance are gastropods, ammonoids, and coiled nautiloids. Least abundant

* Department of Biochemistry, Institut Léon Fredericq, Université de Liège, and Centre National des Recherches Métallurgiques, Section de Liège, Belgium.

† Department of Geology, University of Kansas.

are brachiopods, which are sparsely represented, and vertebrates, which are represented by fish teeth and bone fragments.

According to Smith the fauna consists of 42 species and varieties, of which 17 are nautiloid cephalopods. Among the latter Smith distinguished seven species of straight-shelled forms which he assigned to the genera *Orthoceras*, *Dolorthoceras*, and *Pseudorthoceras*. In this group, *Pseudorthoceras knoxense* was the only previously named species described, the other six being judged to be new. In addition, Smith differentiated 10 species and varieties of coiled nautiloids, 13 species of ammonoids, and 12 species belonging to other groups mentioned above, with the exception of vertebrates, which he did not study.

Smith referred to the deposit as "Buckhorn asphalt," a term that was later widely used (see Wilson, Sando, and Kopf, 1957, p. 54), but has no standing in formal stratigraphic literature.

Ham (1955, p. 46) referred briefly to the "Buckhorn asphalt quarry" and showed its location on an aerial photograph. He indicated that these beds belong to the Deese Formation of Middle Pennsylvanian age. Moore and others (1944, column 37) regarded the Deese as a group and assigned to it a Desmoinesian age.

Detailed work on the mineralogy of the Buckhorn asphalt cephalopods began when Stehli (1956) reported that he had found shells of *Pseudorthoceras knoxense*, as well as unidentified shell fragments, to consist of aragonite. He believed that the cameral deposits found in this species consisted of calcite, but Lowenstam found later (Teichert and others, 1964, p. 34) that they also consist of primary aragonite.

Grégoire (1959a, 1959b) reported on electron-microscopic investigations on shells of *Pseudorthoceras knoxense* and described conchiolin membranes having the same type of reticulate pattern that had been found in shells of living *Nautilus* (Grégoire, Duchâteau, and Florkin, 1950, 1955; Grégoire, 1957, 1962). This work was followed by electron-microscopic study of the cameral deposits of the same species (Grégoire, 1962, p. 44-45), in which the author found structures similar to those occurring in the aragonitic infillings present in *Nautilus* at the angle formed by the posterior surface of the septa and the shell wall. This work was summarized and somewhat elaborated by Grégoire in the *Treatise on Invertebrate Paleontology* (Teichert and others, 1964, p. 17, 37), where several of Grégoire's electron micrographs were reproduced.

The aragonitic nature of the shell of *Pseudorthoceras knoxense* was confirmed by Grandjean, Grégoire, and Lutts (1964). In the same publication Grégoire presented additional electron micrographs of conchiolin tissue in the shell walls of an unidentified orthoconic nautiloid and of an unidentified ammonoid from the Buckhorn asphalt.

Finally, it is of interest that Lowenstam (1963, p. 189) reported the discovery by E. Hare of the following eight ammino acids in the shell and in the cameral deposits of *Pseudorthoceras knoxense*: lysine, histridine, arginine, aspartic acid, tyrocene, serine, glutamic acid, glycine, and alanine.

Almost all the cephalopods from the Buckhorn asphalt upon which tests and observations have been made in recent years have been identified as *Pseudorthoceras knoxense.* However, as already recognized by Smith, several different species and genera of orthocerids are present in the deposits. This observation has been confirmed by more recent studies (Fischer, Lowenstam, and Teichert, in press). Because some fragmentary orthoconic shells are difficult to identify with certainty, it is possible that not all specimens identified as *Pseudorthoceras knoxense* do indeed belong to that species.

Electron-Microscope Studies

(by Charles Grégoire)

MATERIAL AND METHODS

The ultrastructure of the organic and of the mineral components of the cephalopod shells embedded in the Buckhorn asphalt have been investigated with the electron microscope on 230 groups of different preparations. This study includes examination of remnants from decalcified shell layers and from cameral deposits, and of metallic positive replicas of all the shell surfaces available in the samples.

The present paper summarizes some results obtained on the organic structures. A more detailed description, based upon analysis of more than 1,800 electron micrographs, will be published elsewhere.

The material used consisted of large pieces of asphaltic rock, in which dark-brown fragments of crushed shells, mostly of unidentified nautiloids and ammonoids, together with tiny, better preserved conical shells of *Pseudorthoceras knoxense,* were dispersed at random. The blocks were broken and the freshly exposed samples were selected under a binocular microscope for separation of the outer from the inner layers of the shell wall.

The shell fragments were immediately decalcified by chelation (saturated aqueous solutions of the disodium salt of ethylenediaminetetraacetic acid (EDTA) at pH values ranging from 4.0 to 8.0. In order to cleave the agglutinated remnants into thin sheets permeable

(text continues on page 180)

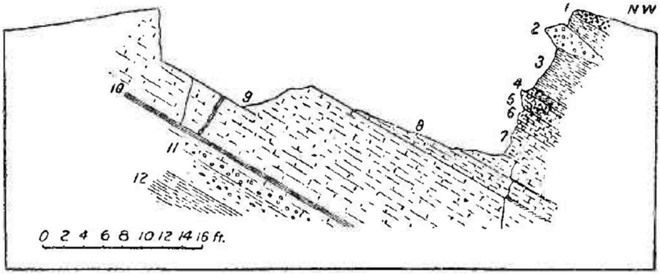


Figure 1. Section through Buckhorn asphalt quarry near Sulphur, Oklahoma. The aragonitic shells occur mostly in bed 6 and in the upper part of bed 9 (reproduced from Eldridge, 1901, fig. 21).

Explanation of Electron Micrographs

Except for plate I, figure 1, and plate V, figure 2, the illustrations show the appearance in the electron microscope of residues from decalcification of the shell layers in various nautiloids and ammonoids contained in the Buckhorn asphaltic limestone.

The suspensions of the decalcified and dissociated material were deposited on supporting films, drained, dried, and the residues were examined either directly or after shadow-casting with palladium.

The illustrations are of two types (as indicated in the table below):

(a) Direct prints made from the original negatives (white shadows directed upward or to the left).

(b) Reversed prints obtained through intermediary direct prints on plates (black shadows directed downward or to the right).

PLATE	FIGURE	
	DIRECT PRINT	REVERSED PRINT
I	1	2
II		1, 2
III		1, 2
IV	1-3	
V	1	2
VI	2	1
VII	1, 2	
VIII	1-3	
IX	2	1, 3
X	1	
XI	1	

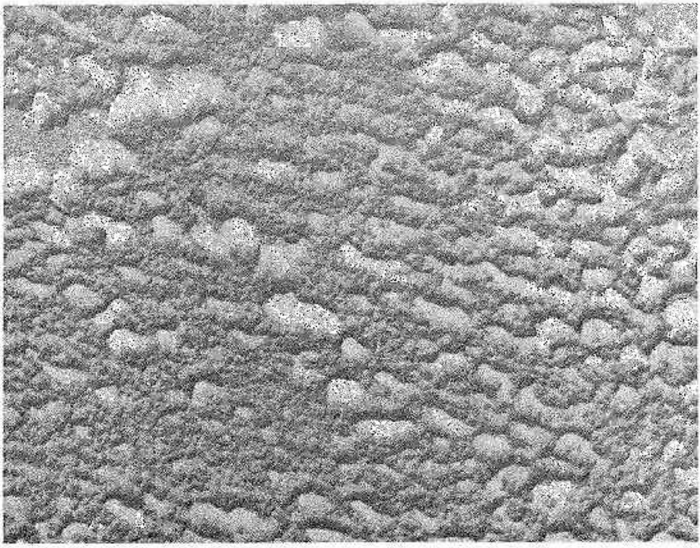
Explanation of Plate I

Figure 1. **Iridescent membrane of conchiolin from decalcified innermost nacreous layer of the shell wall in the living chamber of** Nautilus pompilius Lamarck **(Recent). In the lacelike reticulate sheet of nacreous conchiolin here shown, the nautiloid structure pattern consists of sturdy, irregularly cylindrical trabeculae or cords, studded with hemispherical protuberances of various sizes, and separating elongate openings of irregular outlines. Direct print: x42,000.**

Figure 2. **Iridescent blue-violet, loose flakes of the shell wall of an unidentified orthoconic nautiloid, supplied by C. C. Branson. Decalcification left abundant, biuret-positive, brown organic particles. Nautiloid pattern still recognizable.**

A comparison with figure 1 shows moderate alteration of the structures, consisting of flattening and coalescence of the trabeculae in parts of the preparation, with preservation of a few protuberances. Reversed print: x42,000.

Plate I



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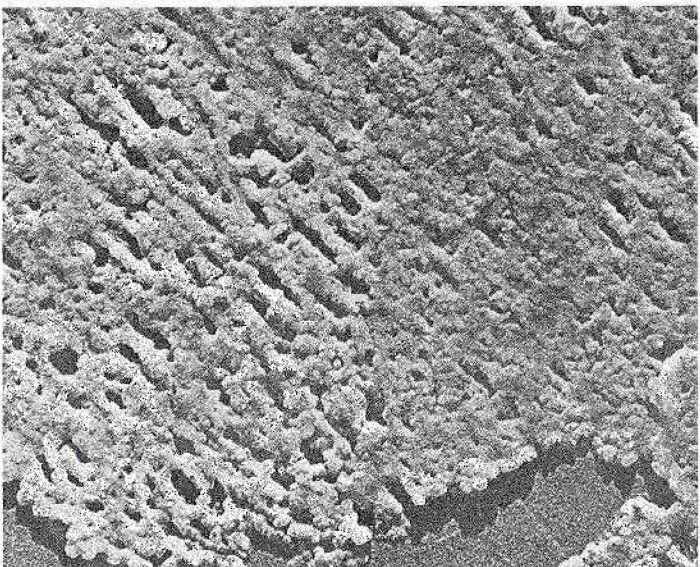

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(continued from page 177)
to the electrons, some suspensions of the organic material were exposed to ultrasonic waves. Drops of these suspensions were deposited on standard 3-mm copper screens coated with films of Formvar or of carbon and examined in the electron microscope (R. C. A. EMU-2), either directly or after shadow-casting with palladium.

OBSERVATIONS

Organic Tissues in Shells of Nautiloids

Inner nacreous layer.—The nautiloid pattern is one of the three structure patterns provisionally recognized in the reticulate sheets composing the nacreous conchiolin of Recent mollusk shells. In *Nautilus macromphalus* and *Nautilus pompilius* it is characterized by sturdy trabeculae, studded with hemispherical protuberances, delimiting elongate or irregularly rounded fenestrations (pl. I, fig. 1). As reported previously (Grégoire, 1957), these reticulate structures belong to the system of organic membranes or sheets which lie as continuous formations between adjacent mineral lamellae of mother-of-pearl.

As shown in plate I, figure 2, and in plates II and III, the chief characteristics of the nautiloid pattern are distinctly recognizable in the conchiolin debris collected from *Pseudorthoceras knoxense* and from several weakly curved fragments of mother-of-pearl belonging to unidentified larger nautiloids. In some samples these reticulate sheets are remarkably well preserved (pl. I, fig. 2; pl. II, fig. 1). In other samples (pl. II, fig. 2; pl. III) the structure of the trabeculae is variously altered, the changes consisting mostly of flattening, coalescence, and disappearance of the tuberosities. In still other samples the trabeculae have been fragmented into irregularly rounded bodies of var-

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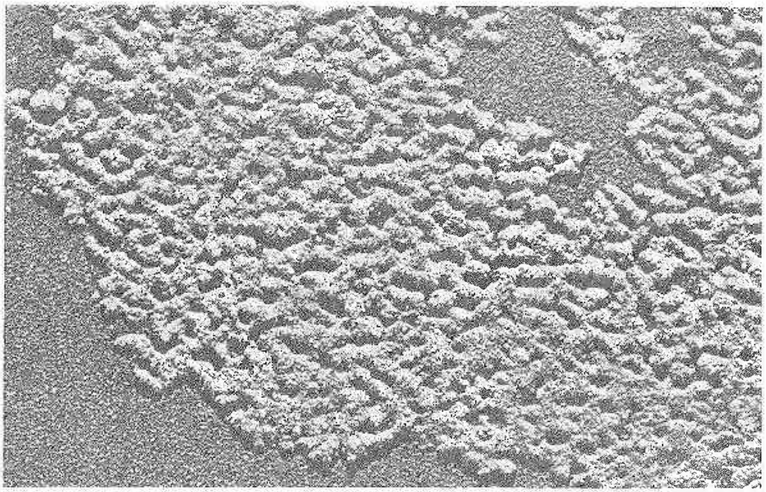
Explanation of Plate II

Figure 1. Decalcified splinter of curved dark-brown iridescent flake from nacreous portion of shell wall from an unidentified specimen, probably a nautiloid, supplied by G. Arthur Cooper, U. S. National Museum.

Biuret-positive residues. Except for general shrinkage of the preparation, possibly induced in part during desiccation on the supporting film, the characters of the nautiloid pattern of mother-of-pearl, including presence of tuberosities on the trabeculae, appear distinctly in this well-preserved fragment of a reticulate sheet of conchiolin. Reversed print: x42,000.

Figure 2. Decalcified inner nacreous layer of the shell wall of Pseudorthoceras knoxense, supplied by G. Arthur Cooper, U. S. National Museum. In this preparation, alteration of the nautiloid pattern consists of flattening and coalescence of the trabeculae, which exhibit a smooth surface from which the tuberosities have disappeared. Reversed print: x42,000.

Plate II



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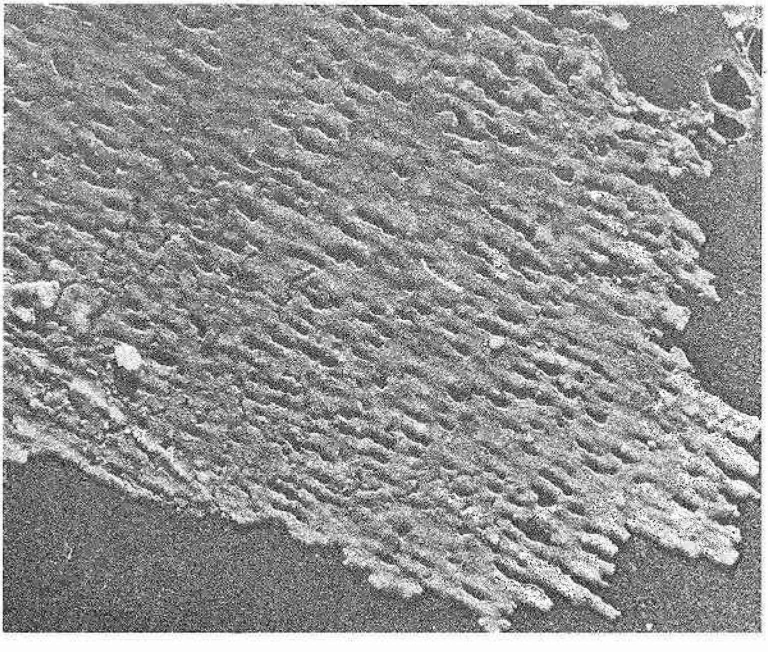

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ious sizes, resembling pebbles (pl. VI, fig. 2). The last kind of alteration is common in the reticulate sheets of nacreous conchiolin of Paleozoic and Mesozoic shells (Grégoire, 1964; Grandjean, Grégoire, and Lutts, 1964).

Outer shell layers.—Decalcification of the generally strongly weathered outer layers of the shells of Buckhorn asphalt nautiloids in which, however, the growth lines were still more or less recognizable, left various structures, including debris of coarse networks (pl. V, fig. 1), granular veils condensed into folded membranes (pl. IV, fig. 2), abundant entangled, contorted, vermicular filaments (pl. IV, fig. 3), and altered lacelike nacreous fragments (pl. IV, fig. 1), the latter proceeding obviously from the outermost portions of the nacreous layer immediately adjacent to the outer layers of the shell.

Cameral deposits.—The material of the cameral deposits used in this study consisted of symmetrically stratified lamellae or concentric rows of palisades, and of soft, crumbly, hardly calcified substance filling the central portion of the camerae in *Pseudorthoceras knoxense*. Decalcification of this material left abundant biuret-positive shreds of substance. The illustrations on plates VI, VII, and VIII show the appearance of some of the more representative structures found in preparations of these cameral substances:

i. Fragments of lacelike reticulate sheets of nacreous conchiolin, sometimes predominant in the samples. Some sheets show the mural nautiloid pattern of mother-of-pearl (pl. IX, fig. 1). Many of them present the characters of the septal nautiloid pattern (pl. VI, fig. 1, fragment on the right). Other sheets exhibit various alterations, such as flattenings, shrinkage, coalescence of their trabeculae (pl. VI, fig. 1, fragment on the left), or fragmentation of these trabeculae into clusters of pebble-shaped bodies (pl. VI, fig. 2; pl. VII, fig. 2).

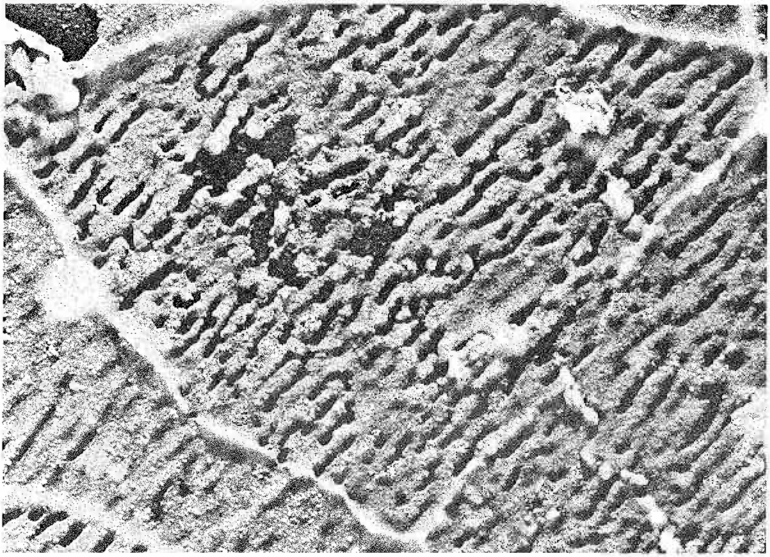
Explanation of Plate III

Figure 1. Splinter of curved, dark-brown, iridescent nacreous flake from shell wall of an unidentified specimen, supplied by W. M. Furnish. This splinter was part of a fragment of a coiled shell, either an unidentified nautiloid, or possibly an ammonoid, identified by W. M. Furnish as Eoasianites? sp. The brown organic residues of the splinter were biuret-positive.

Inside the triangular area, alteration in the nautiloid pattern of conchiolin consists, as in plate II, figure 2, of flattening and scattered coalescence of the trabeculae. The white ridges rising slightly above the sheet are intercrystalline conchiolin bridges, originally sandwiched between the edges of polygonal aragonite crystals involved in the lamella resting on this reticulate sheet and dissolved by the decalcifier. Reversed print: x42,000.

Figure 2. Dark-brown, curved, loose flake from an unidentified specimen supplied by N. D. Newell. Substantial amount of biuret-positive organic residues. In other fragments of this sample, reticulate sheets closely resembled those represented in plate I, figure 2; plate II, figures 1, 2; and plate III, figure 1. Reversed print: x42,000.

Plate III



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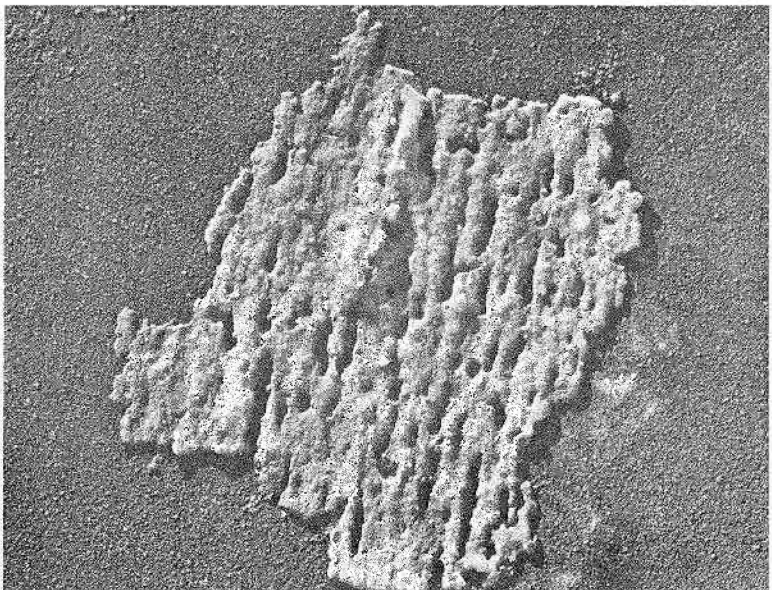

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ii. Loose, coarse networks, composed of sturdy strings or elongate ribbons, appearing as rigid and stretched formations, some perforated by openings (pl. VII, fig. 1; pl. VIII).

iii. Heterogeneous, unidentified opaque structures of large size (not illustrated), appearing mostly as fragments of thick amorphous membranes.

iv. Large bundles of fibrils, few found in the samples so far investigated.

Organic Tissues in Shells of Ammonoids

The material used consisted of small fragments of nacreous portions of the shell wall of crushed unidentified specimens, of *Eoasianites* sp., and of *Eoasianites hyattianus* (Girty). Decalcification of these slabs left debris of reticulate sheets of conchiolin shown in plate IX, figures 2 and 3, and plates X and XI.

The chief characters of the nautiloid pattern, sturdy trabeculae and elongate or irregular openings, are recognizable in plate IX, figures 2 and 3, and in some areas of plate X. However, the type of fabric of the nacreous conchiolin recorded in these specimens of ammonoids and the nature of its alterations differ from those of the nautiloids illustrated in plate I, figure 2, and plates II and III.

Explanation of Plate IV

Figure 1. Outer portion of the shell wall with well-preserved growth lines of Pseudorthoceras knoxense, supplied by G. Arthur Cooper, U. S. National Museum.

This sample, detached from the inner portion of the shell wall, contained remnants of the outer porcellaneous layer and portions of the adjacent outermost nacreous layer adhering to the former.

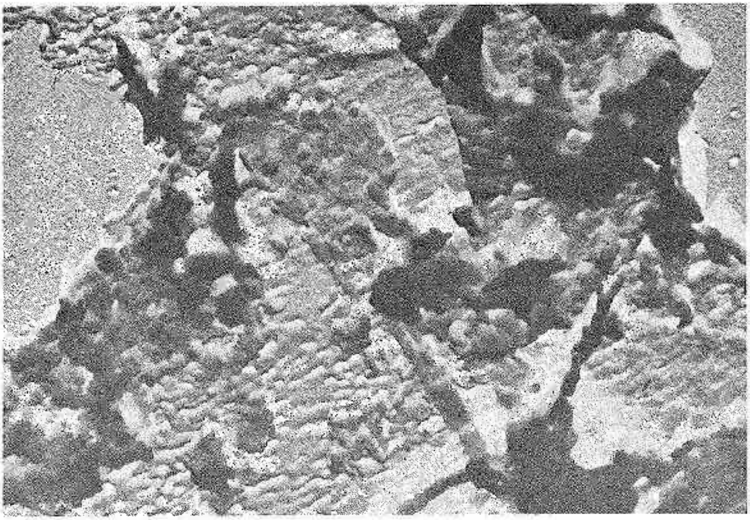
Decalcification of this and other identical samples left various structures, some of which are shown in this plate and in plate V, figure 1.

Thick, softly contorted cords and ribbons of irregular outlines lie on a background consisting of a fragment of a reticulate sheet having a typical nautiloid pattern. These cords are, at least in part, induced by folding and rolling up of the edges of the sheet fragment. Other cords might belong to the structures of the immediately adjacent outer shell layer anchored on this fragment of nacreous conchiolin (see discussion and pls. VII, VIII). Direct print: x20,500.

Figure 2. A granular membrane, in part shrunk and folded into cords, from the outermost portion of the shell (region of the growth lines); specimen of Pseudorthoceras knoxense, supplied by W. M. Furnish. Direct print: x27,500.

Figure 3. Decalcification of the outermost portion of the shell wall of specimen of Pseudorthoceras knoxense, supplied by G. Arthur Cooper, left this agglomeration of contorted, coarse, densely entangled, vermicular ribbons or cords (see discussion). Direct print: x6,000.

Plate IV



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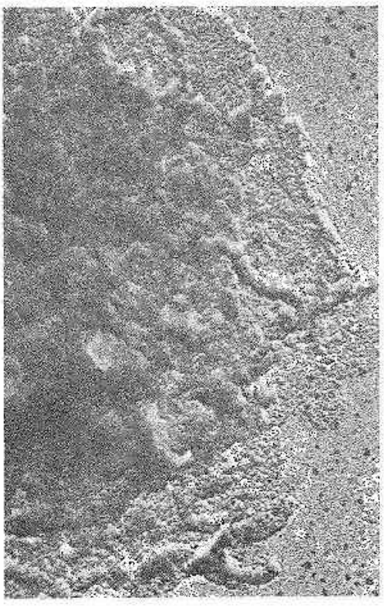

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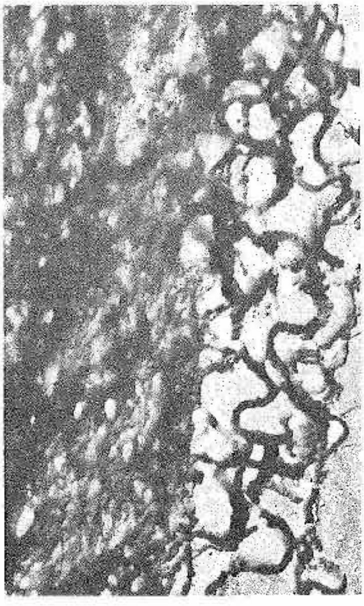

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As seen in plate IX, figures 2 and 3, the trabeculae are broader and shorter, and some are barrel-shaped (pl. IX, fig. 3). In the preparations selected here, relatively large hemispherical protuberances such as those studding the nautiloid trabeculae do not appear or are exceptional. In these reticulate conchiolin sheets of ammonoids the surface of the trabeculae appears to be smooth or finely granular. The general aspect of the fabrics is more "dumpy" than in the lacelike reticulate sheets of nautiloids.

As shown in plate IX, figure 2, and plates X and XI, the alterations in the ammonoid trabeculae consist of flattenings (pl. IX, fig. 3; pl. X), coalescence (pl. IX, fig. 2; pl. X), dislocation and fragmentation (pl. XI), bloating of the fragments into vesicles (pl. IX, figs. 2, 3; pl. X), some of considerable size (pl. X), or into pebble-shaped or hemispherical corpuscles (pl. IX, figs. 2, 3), scattered at random on the supporting film (pl. IX, fig. 2), or clustered (pl. XI).

DISCUSSION

Pattern of the Nacreous Reticulate Conchiolin Sheets in Nautiloids and Ammonoids

Preservation of structural features of the reticulate conchiolin sheets in the Buckhorn asphalt shells contrasts with the important alterations generally recorded in many other Paleozoic and Mesozoic shells.

Results so far have not permitted detection in nautiloids of variations of texture which could safely be considered indicating taxonomic differences at a level lower than that of order. Such differences have been reported in shells of Recent pelecypods in which statistically significant variations in the pelecypod pattern have been detected between families (Grégoire, 1960). On the other hand, within the genus

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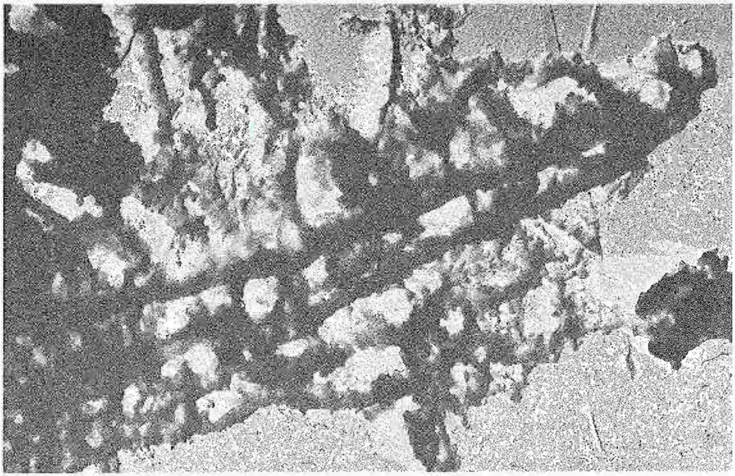
Explanation of Plate V

Figure 1. Structure consisting of a system of stout, straight ribbons crossing at right angles and mixed with debris of a thin granular material, found in the decalcified remnants of the outer portion of the shell wall of the specimen shown in plate IV, figure 3. Direct print: x11,500.

Figure 2. Polished surface of a longitudinal, sagittal section of the shell of Pseudorthoceras knoxense **(supplied by A. G. Fischer), embedded in a hard resine (Palatal). Positive metallic replica of the region of the concentric palisades in the cameral deposits (see Grégoire, 1962, p. 44-46, and text-fig. 8, p. 46).**

Four successive palisades are shown. Each palisade is composed of parallel, imbricated, elongate tablets, blades, or bars, appearing in form of needles when seen on their edges. Palisades with abundant organic material (white shreds on the figure, corresponding to the white strands of Grégoire, 1962, text-fig. 8) alternate with palisades in which the mineral elements are predominant. Reversed print: x10,000.

Plate V



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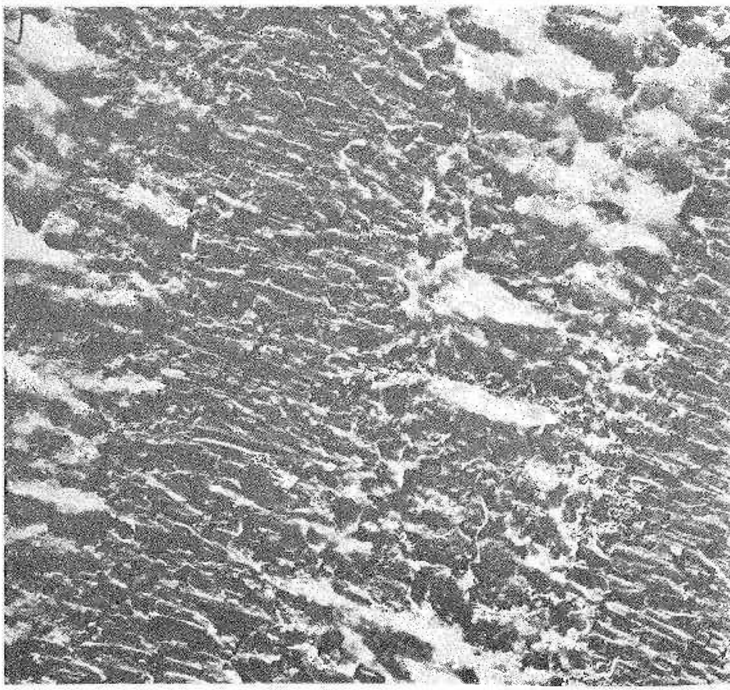

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Nautilus differences between the pattern of nacreous conchiolin in *Nautilus macromphalus* and in *Nautilus pompilius* are not appreciable (Grégoire, 1962).

The slight differences in appearance of the nautiloid pattern illustrated in plate I, figure 2, and plates II and III, namely, flattening of the irregularly cylindrical trabeculae into ribbons, are probably due to secondary diagenetic processes or to different conditions of preservation.

Comparison between the structure of the sheets in nautiloids (pls. I-III) and in ammonoids (pl. IX, figs. 2, 3; pls. X, XI) suggests that in the latter the texture differs by several peculiarities from that of nautiloids. The ammonoid material available for the present study was, however, too limited to permit conclusion that an ammonoid pattern should be distinguished from a nautiloid pattern.

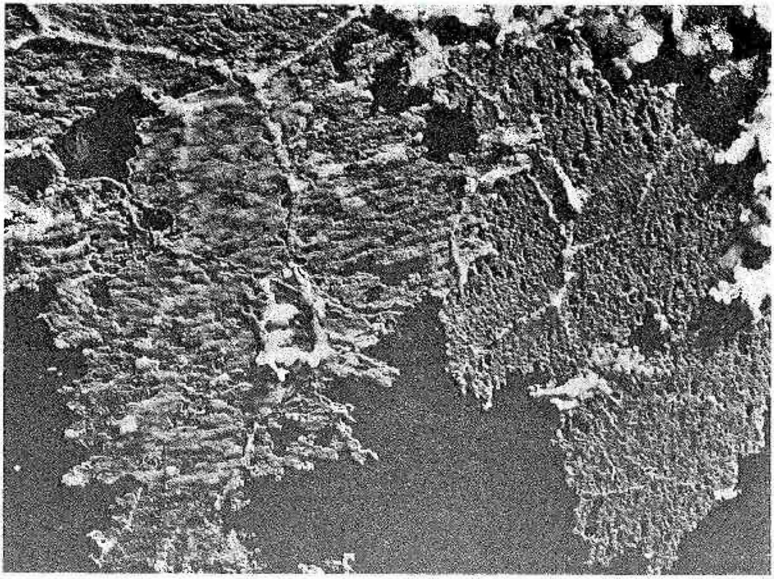
The nature of the vesicles, some of considerable size, associated with the debris of sheets of nacreous conchiolin in the specimens of ammonoids examined in the present study, is unknown. Such vesicles have not yet been detected in the Buckhorn nautiloids. However, they have been seen occasionally in specimens of Carboniferous age, especially in goniatites (Grégoire, unpublished results). Some of these vesicles were suspected to be parts of foreign organisms contaminating the specimens. Merely superimposed during desiccation on the supporting film, these structures might give deceptive pictures and appear as if they were parts of the sheets. On the other hand, other preparations suggest that some vesicles were formed by diagenetic alterations of the original trabeculae, consisting of dislocations and bloating of their fragments (pl. IX, figs. 2, 3).

Explanation of Plate VI

Figure 1. Two fragments (Pseudorthoceras knoxense, **supplied by A. G. Fischer) of reticulate sheets of nacreous conchiolin from decalcified, crumbly matter of the cameral deposits exhibit various alterations when compared with those shown in plate IX, figure 1, though their nautiloid pattern is still distinctly recognizable. In the fragment on the left, the trabeculae are flattened with varying degrees of coalescence. The fragment on the right might belong to nacreous material from a septum. As shown previously in Recent** Nautilus **(Grégoire, 1962, fig. 59), the nacreous organic components of the septa are characterized by a more delicate fabric, slender trabeculae, and smaller openings than those of the wall. Note the imprints of the aragonite crystals and the intralamellar intercrystalline bridges freed by decalcification and appearing as white ridges. Reversed print: x16,000.**

Figure 2. Illustrates another type of alteration in nacreous conchiolin found in fragments collected from the soft central matter of the cameral deposits (Pseudorthoceras knoxense, **supplied by A. G. Fischer). The trabeculae have been transformed into agglutinated pebble-shaped bodies. The ridges between the original aragonite crystals dissolved by the decalcifier appear as soft, irregular opaque cords. Direct print: x16,000.**

Plate VI



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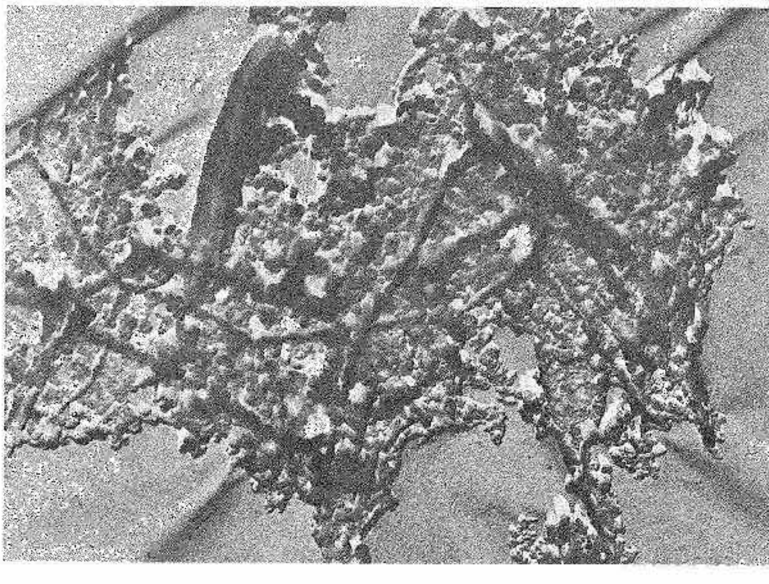

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The Outer Layers of the Shell Wall in Nautiloids and Ammonoids

In the shell wall of the Recent *Nautilus,* at least in the dried, variously weathered specimens available, the organic material of the outer layer, including mostly porcellaneous substance and possibly remnants of the periostracum, appears as a dull-gray, transparent membrane which contrasts with the iridescence of the immediately underlying soft, nacreous membranes of conchiolin.

In the electron microscope, this outer membrane is seen to consist of networks of thin fibrils embedded in veils, or within an amorphous substance (Grégoire, 1962, figs. 33, 34, 36). In further observations (unpublished) other structures, including agglomerations of unidentified corpuscles and various debris of epibionts, were found in the decalcified, outer layer of the *Nautilus* shell.

In nautiloids from the Buckhorn asphalt, in which growth lines were still visible, collection of outer shell layers uncontaminated by other materials, either by substance from the external mold or from adjacent mother-of-pearl, is always difficult because of the blurred limits between the layers. Among the heterogeneous structures left by decalcification of these outer layers, fragments of lacelike nacreous, reticulate sheets are not absent from the preparations (pl. IV, fig. 1), and granular veils (pl. IV, fig. 2), some containing fibrils, are probably the remnants of structures similar to some found in the outer layer of the Recent *Nautilus.*

Several preparations of organic materials from the outer layers of fossil nautiloids and ammonoids are characterized by the presence of abundant residues, consisting of sturdy, densely entangled, in many cases worm-shaped, ribbons or cords (pl. IV, fig. 3; pl. V, fig. 1). The nature of these structures is unknown. They might be parts of foreign organisms that contaminated the shells prior to their burial. Associa-

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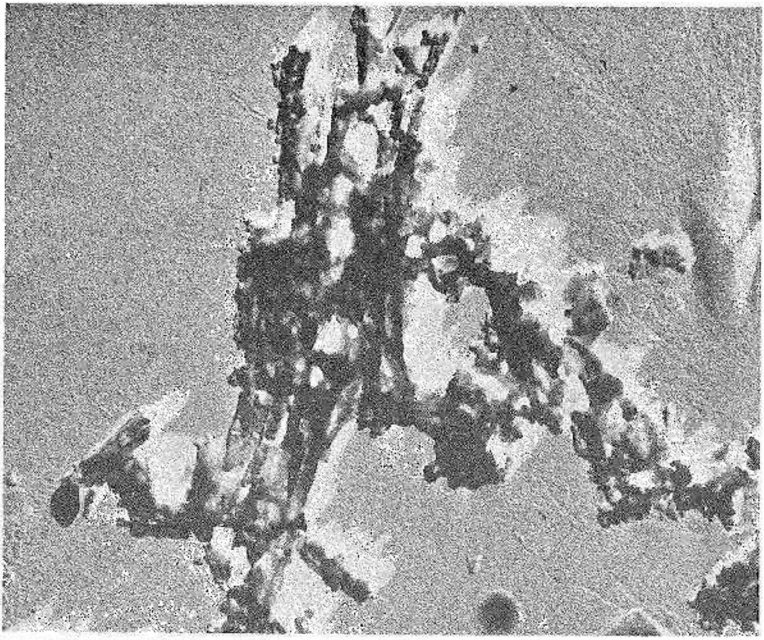
Explanation of Plate VII

Figure 1. Another type of structure found in the concentric palisades and in the soft matter of the cameral deposits of Pseudorthoceras knoxense **(supplied by A. G. Fischer) is shown in plates VII and VIII. It consists of assemblages, or networks, of rigid, elongate, ribbon- or girder-shaped elements, a few perforated by tiny holes.**

These elements seem to differ from the soft cords or intercrystalline bridges of conchiolin shown in plate VI, which result from compression of the nacreous organic material by the expanding crystals of aragonite during growth of the shell. The rigid structures might be the organic matter appearing as white shreds on the replica shown in plate V, figure 2, associated with the elongate mineral elements of the palisades (see discussion). Direct print: x20,500.

Figure 2. A bundle of stretched rigid elements, identical to those shown in figure 1 and in plate VIII, appears superimposed on altered fragments of reticulate sheets of conchiolin. Direct print: x16,000.

Plate VII



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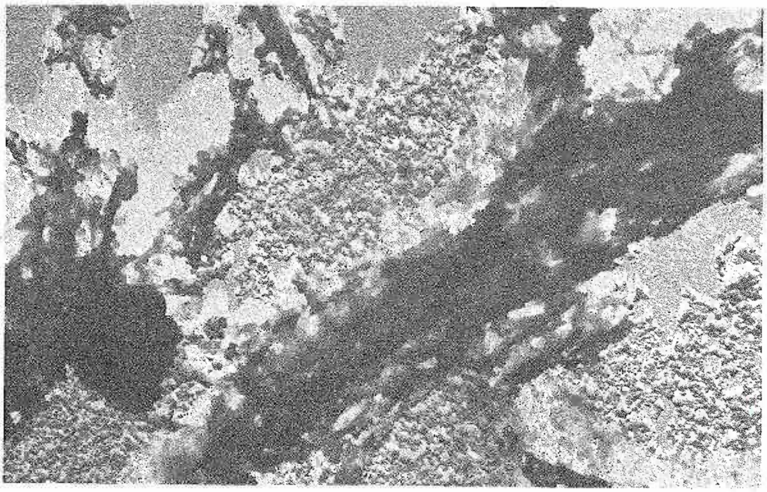

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tion with mollusk shells of foreign organisms has been reported in the literature (Schindewolf, 1962, 1963).

Cameral Deposits

Origin and mode of development of primary cameral deposits is still controversial. The presence of organic deposits in the camerae has long been reported (Barrande, 1857). Teichert (1933, 1934) first showed the essential differences between primary deposits of organic origin and secondary deposits of inorganic origin, and suggested that the former had been formed continuously during life by some kind of organic tissue in the camerae. Flower (1939, 1955) developed this concept and suggested that the cameral deposits were secreted upon the surface of cameral tissues functioning as mantles (cameral mantle), and maintaining metabolic contact with the rest of the animal through the siphuncle. Both Teichert and Flower suggested that the material for the cameral deposits had been transported into the camerae in solution by body fluids that permeated the wall of the siphuncle. On the other hand, according to Mutvei (1956) all cameral deposits are no more than crystallized precipitates of lime-saturated water, percolated through the distal division of the siphonal funnel into the shell chambers, when, after death of the animal, the shell sank down onto a calcareous bottom sediment and was gradually embedded in it. In a recent paper Mutvei (1964) seems to have somewhat modified his former interpretation when he wrote (p. 89):

> It is not impossible that the complicated and comparatively regular structure of this precipitation in certain forms, e. g., in the families Pseudorthoceratidae and 'Lamellorthoceratidae' of the order Michelinoceratida, is due to a special chemical reaction between the liquid secreted by the epithelium of the siphonal cord into the shell chamber and sea water penetrating post-mortally into the shell chambers.

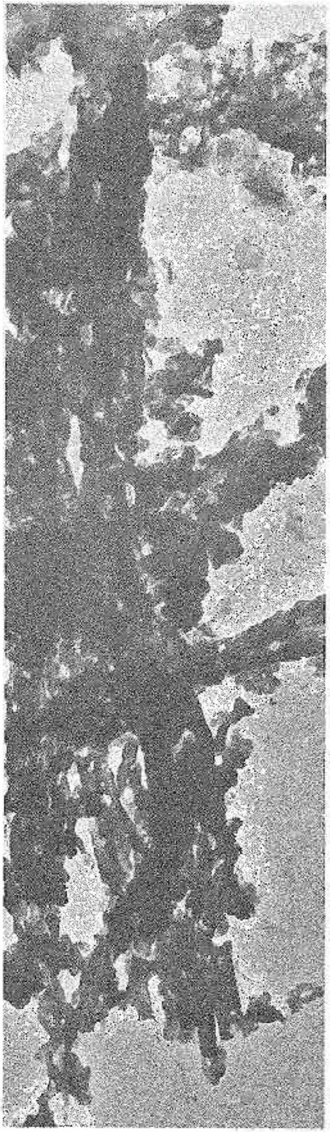
In a recent communication, Joysey (1961) again suggested, upon the basis of new observations on living specimens of *Nautilus*, that these precipitations might have occurred during life from solutions transported through the walls of the siphuncular tube.

As described above, the abundant organic shreds left by decalcification of the cameral deposits consisted of variously altered reticulate sheets of nacreous conchiolin, of systems of stretched strings or ribbons, and of other unidentified opaque structures and bundles of fibrils.

Explanation of Plate VIII

Figures 1-3. Fragments of shell of Pseudorthoceras knoxense **(supplied by A. G. Fischer) show systems of networks consisting of elongate, rigid, frequently straight, ribbon-shaped elements, with perforations irregularly scattered. The fragments deposited by sedimentation of suspensions are lying at random on the supporting film. In figure 3 note the fanlike arrangement of the components of the network. Direct prints: x42,000.**

Plate VIII



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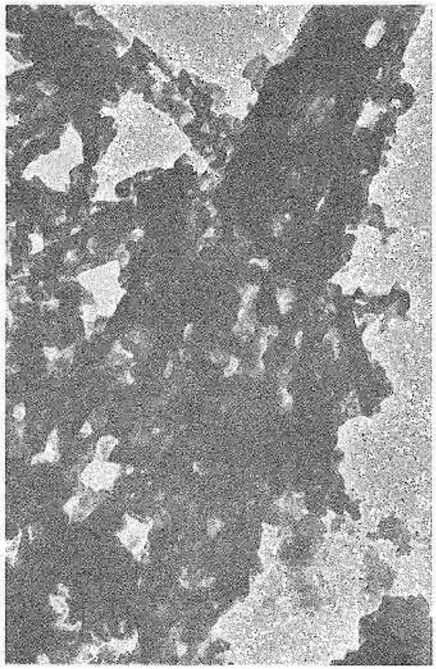

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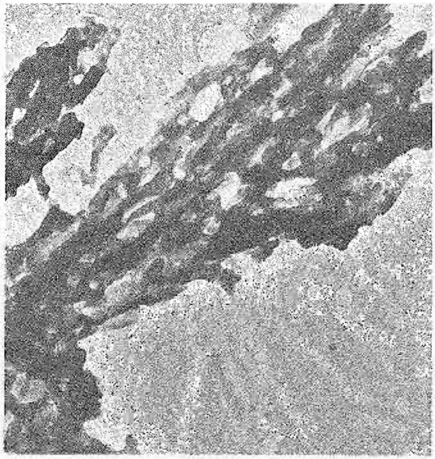

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Imprints, left on the nacreous, reticulate sheets, of the original aragonite crystals forming the lamellae between which these sheets were sandwiched (pl. VI, figs. 1, 2) indicate unequivocally that these sheets were once parts of mural and septal mother-of-pearl.

The present records of abundant fragments of nacreous conchiolin in the cameral deposits are in agreement with the results of biochemical investigations carried out by Hare (Lowenstam, 1963). Hare found that in *Pseudorthoceras* specimens from Buckhorn asphalt the organic components of cameral deposits have an amino acid composition identical with that of the decalcified organic matrix of the shell. According to Lowenstam (1963), these conditions indicate clearly that the carbonate of the cameral deposits is an original organic precipitate of these nautiloids.

In the present material, biuret tests were positive, with varying degrees of intensity, in samples of decalcified organic matrix of the shell walls and in several organic components of the cameral deposits. These findings are in agreement with records of preservation of material of the specific protidic constitution of conchiolin in Holocene, Oligocene, and Eocene shells (Florkin and others, 1961). Preserved reticulate sheets of conchiolin had been previously detected in these shells by electron microscopy (Grégoire, 1958, 1959a).

Exfoliation during life, either of mineralized mother-of-pearl or of soft nacreous conchiolin sheets from the mural and septal surfaces of the camerae, followed possibly by a mechanical drift of the nacreous splinters or shreds towards the central perisiphuncular region of the camerae, seems to have played an important part in the collection of abundant organic matter in this area.

Explanation of Plate IX

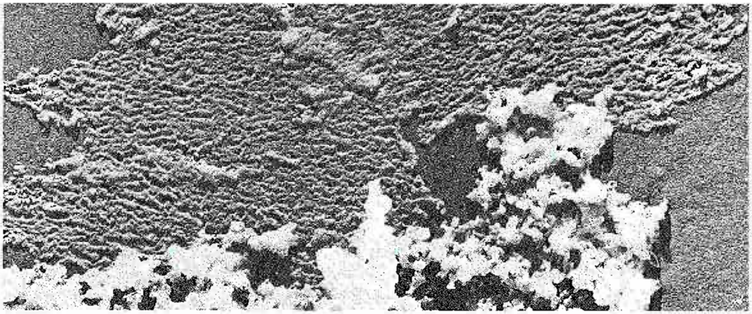
Figure 1. Two fragments of nacreous reticulate sheets of conchiolin of Pseudorthoceras knoxense **(supplied by A. G. Fischer), exhibiting the typical mural nautiloid pattern (compare with pl. II, fig. 2, at higher magnification) are mixed with a material of unknown nature appearing in form of agglutinated, rounded, opaque corpuscles.**

This picture has been selected for comparison, at the same magnification, with related structures collected from the cameral deposits, especially from the soft organic matter in the central portion (see pl. VI, figs. 1, 2). Reversed print: x16,000.

Figures 2-3. Decalcification of inner portion of the shell wall of an unidentified ammonite, supplied by F. G. Stehli, left biuret-positive fragments of reticulate sheets, in which the nautiloid pattern differs from that so far observed in nautiloids of the same deposit. The lacelike reticulate sheets appear to consist of short, more or less flattened, in some cases barrel-shaped, trabeculae (fig. 3). Vesicles of various sizes (with triangular white shadows behind them) scattered on the supporting film (fig. 2) or attached to the trabeculae (fig. 3, bottom left) are possibly fragmented and bloated portions of the same trabeculae.

Figure 2 is a direct print, x33,000; figure 3 is a reversed print, x42,00.

Plate IX



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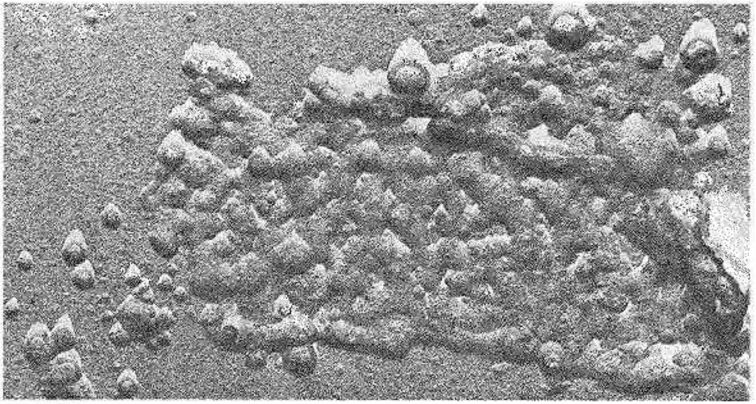

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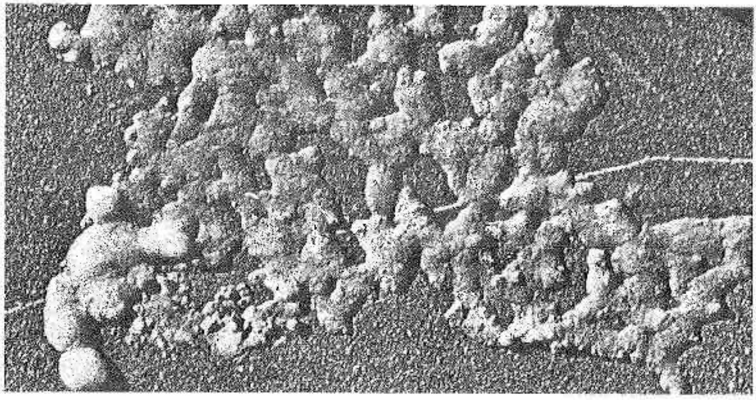

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The best preserved reticulate sheets belong probably to mineralized fragments in which they remained protected during diagenesis, whereas the most altered ones were exposed to diagenetic factors. Possibly also a portion of these nacreous sheets results from decalcification of tiny splinters of brittle septal material collected unavoidably together with the crumbly matter during preparation of the samples.

Identification of the second predominant type of structure found in the cameral deposits, the straight girder-shaped strings or ribbons, is more difficult. Some of these strings are obviously derived from foldings, curlings, agglutination, and coalescence of the nacreous reticulate sheets and also from freed intercrystalline bridges (pl. IV, fig. 1; pl. VI, figs. 1, 2).

The strings or ribbons illustrated in plates VII and VIII contrast in all their structural features with the nacreous remnants (see, for example, pl. VII, fig. 2, where both structures are superimposed). The two following tentative explanations about their origin are based upon peculiarities of their structure.

Several of these strings appear to be rigid, elongate structures, exhibiting perforations, and in many cases disposed in parallel, or diverging fan-shaped arrangements. These strings might be, at least in part, nacreous conchiolin that had been compressed and stretched, after exfoliation, when the animal was alive, during the growth of the elongate crystals (the aragonite crystals of Flower, 1939) arranged in rows of palisades and characterizing the concentric stratified regions of the cameral deposits.

Alternatively, these stretched strings might be the organic components forming some kind of sheaths to these elongate crystals. In this case, they could possibly be the product of secretion, differing structurally from the nacreous reticulate sheets, of a living tissue (? cameral mantle of Flower, 1939), left behind in some camerae after completion of the septa, and connected with the animal through the siphonal system.

On the other hand, alternance of rows of crystals associated with abundant and scarce organic structures respectively (pl. V, fig. 2), provides evidence about the existence of a rhythmic process of secretion, thus further supporting the concept that the cameral deposits were formed when the animal was alive.

Contribution of elements from the siphuncular system to other organic structures collected in the central portions of the cameral de-

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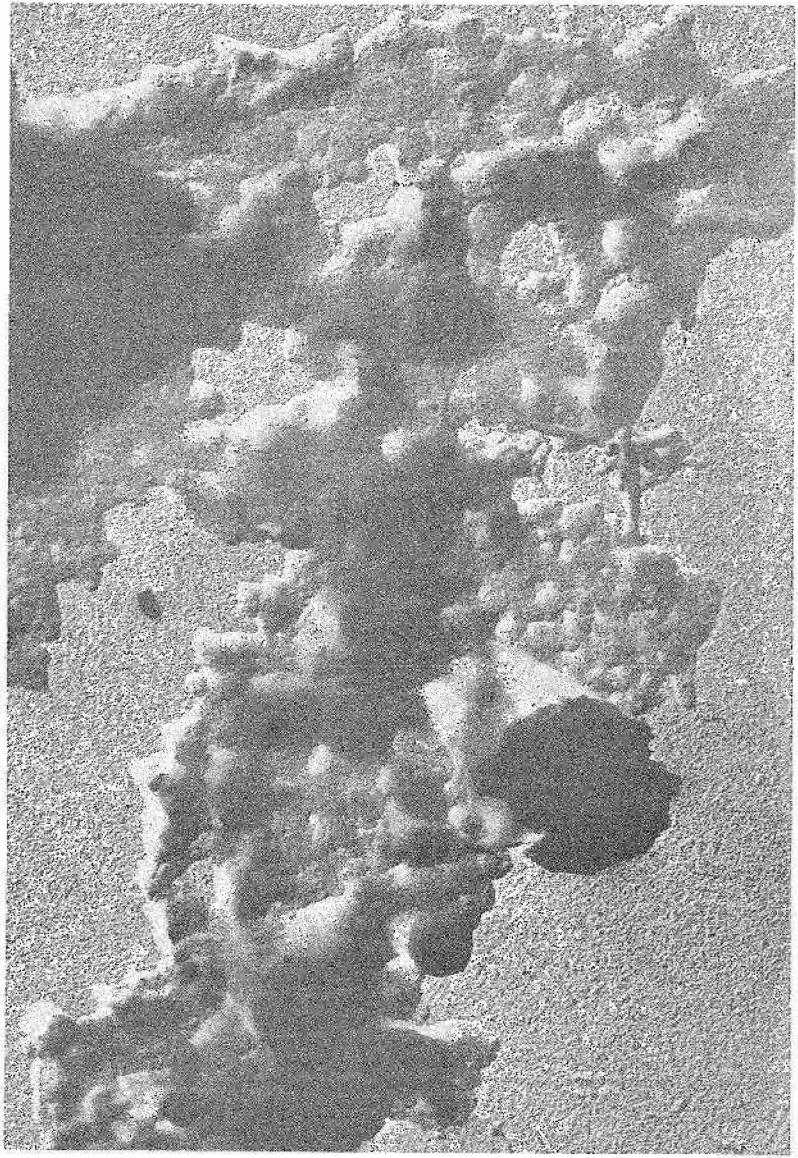
Explanation of Plate X

Figure 1. Decalcified inner layer of the shell wall of an unidentified ammonite, supplied by F. G. Stehli (see pl. IX, figs. 2, 3).

The picture shows, assembled in the same area, various alterations in the reticulate sheets of nacreous conchiolin, found in much of the ammonitic material from the Buckhorn asphalt so far studied: flattening, condensation through shrinkage (middle right), coalescence (top and middle extreme left), inflation of the trabeculae into irregular vesicles, sometimes of considerable size (bottom middle and center). Direct print: x42,000.

Plate X



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posits (thick membranes, bundles of fibrils: see description p. 182, 184) appears also to be possible. The connecting ring of the Recent *Nautilus* shell, selected as a control, and prepared in the same way as the cameral deposits, left various structures, composed in parts of membranes and fibrils, entangled with rounded bodies, and similar to the structures found scattered in the soft, crumbly, central matter of the camerae of fossil forms.

CONCLUSIONS AND SUMMARY

In cephalopods buried in the Buckhorn asphalt, remnants of organic structures from various layers of the shell (outer and inner layers of the wall, cameral deposits) have been studied with the electron microscope.

Well-preserved nacreous, reticulate sheets, characterized by the type of texture described as the "nautiloid pattern" in the shell of Recent *Nautilus*, have been detected in the shell and cameral deposits of crushed nautiloids (including *Pseudorthoceras knoxense*) and of ammonoids.

Variations in this pattern that have been observed among different samples of nautiloids and ammonoids might be due in part to diagenetic processes. Owing to the limited material available and to the uncertainties in the identity of the fragments, evidence of taxonomically significant differences at the subclass (nautiloids and ammonoids) or order level (various nautiloids) was not conclusive.

The significance of some of the extensive alterations, such as formation of vesicles, which seemingly affect especially the organic shell structures of the ammonoids, is unknown.

Observations on the weathered, outer shell layers suggest occurrence in several samples of substantial contaminations by epibionts.

The organic material left by decalcification of cameral deposits

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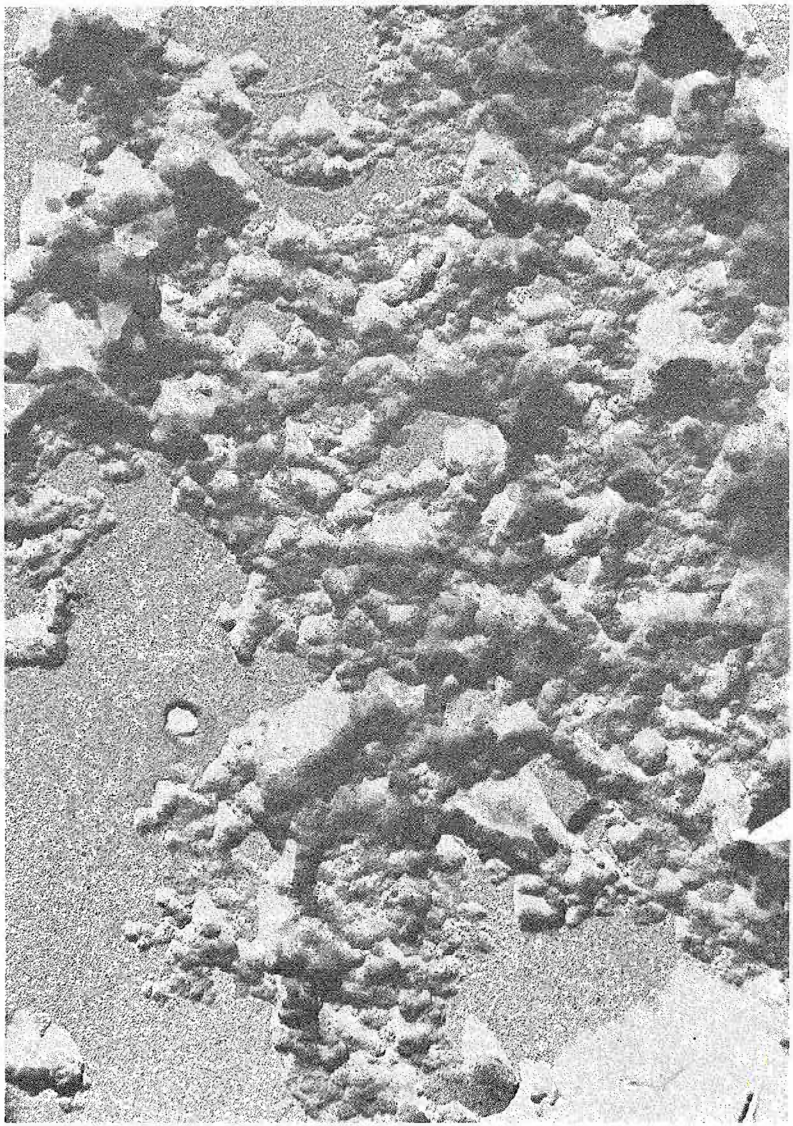
Explanation of Plate XI

Figure 1. Decalcified fragment of mother-of-pearl from the shell wall of Eoasianites hyattianus **(Girty), supplied by W. M. Furnish and B. F. Glenister (State University of Iowa, 8888).**

A reticulate sheet in which the nautiloid pattern appears to be considerably disorganized. The structural changes of fragmentation and transformation of the trabeculae into agglomerated pebbles and coalescence of the structures in various areas. In this sample, oil, impregnating most of the specimens entombed in Buckhorn asphalt, was removed in part by immersing the ground samples in pyridine (on the advice of Francis G. Stehli, personal communication). Similar alterations were, however, also observed in untreated specimens. On the other hand, transformation of the trabeculae into pebble-shaped corpuscles is observed consistently in many Paleozoic and Mesozoic specimens (Grégoire, unpublished observations, and in Grandjean, Grégoire, and Lutts, 1964). During investigations on the factors of experimental diagenesis (Grégoire, 1964), this alteration could be reproduced experimentally in mother-of-pearl of the Recent Nautilus **by exposing it to dry heat.**



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and forming also the soft, hardly calcified substance gathered in the central region of the shell of *Pseudorthoceras knoxense,* includes predominantly variously altered nacreous, reticulate sheets, resulting from exfoliation of mural and septal substances from the cameral surfaces, and systems of rigid strings, many stretched or arranged in coarse networks, all of which are clearly of organic origin.

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