

A TECHNIQUE FOR REVEALING THE STEREO- STRUCTURE OF FOSSIL CRINOIDS

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ABSTRACT. The stereom of fossil crinoid ossicles preserved in an argillaceous matrix can be revealed by treating them with hydrofluoric acid. The clay filling the stereom pores is dissolved and the skeletal calcite is faithfully replaced by fluorite. Features discovered in selected Lower Carboniferous crinoid ossicles prepared by this method include the following: large canals penetrating the areola in the columnals of a particular inadunate crinoid; triple aboral nerve canals, and labyrinthic stereom in the muscle fossae of distinctive inadunate brachials; and a regular arrangement of trabeculae forming a cubic structure in the stereom of flexible crinoid brachials.

OVER the last decade, there have been a number of studies of the detailed morphology of recent echinoderm ossicles, using scanning electron microscopy. Of these, we pick out the surveys of crinoid microstructure by Macurda and Meyer (1975, 1976) and the extensive investigations of the crinoid stem by Roux (1970, 1971, 1974a, 1974b, 1975) as having particular significance for fossil crinoid studies. In all of these studies the architecture of the stereom has been shown to have a functional significance. Unfortunately the details of the stereom are difficult to discern in most fossil material, because carbonate cements precipitated epitaxially on the skeletal calcite occlude the stereom pore spaces. In some examples, however, the stereom is clearly visible in thin section. The most common cases of this are when the stereom pores are filled by an iron-rich carbonate cement (which can be differentiated by staining), or by micritic sediment or cement, or by iron sulphides or oxides, or by clay minerals. When such mineralogical or textural differences are exploited by natural weathering or by controlled acid etching, the three-dimensional stereom architecture may be revealed: Lane and Macurda (1975) established the presence of muscular articulation in naturally weathered brachials of the Pennsylvanian cladid crinoid, *Aesiocrinus*; and Lapham, Ausich, and Lane (1976) have illustrated the structure of the stereom of Mississippian crinoid ossicles which had been etched in weak formic acid.

Whilst trying to recover micropores from a Carboniferous marine shale, we accidentally discovered that some crinoid ossicles treated with hydrofluoric acid (HF) showed surprisingly detailed microstructure: clay filling the stereom pores was dissolved and the calcite of the ossicles was faithfully replaced by fluorite, a process which has been named fluoridization by Upshaw, Todd, and Allen (1957, p. 793). The use of hydrofluoric acid in the preparation of calcareous fossils has been independently (and often accidentally) discovered several times (Cookson and Singleton 1954; Grayson 1956; Wetzel 1921). Most stress has been laid on the translucent nature of fluoridized fossils when they are immersed in liquid. Sohn (1956) was able to make visible ostracode muscle scars by treating the valves with HF, and Upshaw *et al.* (1957) illustrated the internal structures of fluoridized foraminifers. Sprinkle and Gutschick (1967) used HF to prepare blastoids preserved in a fine-grained sandstone.

We have applied the fluoridization technique to Carboniferous crinoid material preserved in a variety of rock types, ranging from plastic clays of the mid-western United States to indurated silty mudstones from Ireland. Examples of the results obtained are shown in Plates 94 and 95.

METHODS

The material for which the fluoridization technique is most effective is that preserved in clay mudstones or shales where the clay has penetrated deeply into the stereom pores. We have generally used bulk samples rather than attempting to fluoridize particular individual specimens, because of the risk of damage. However, most of the microcrinoids described by Lane and Sevastopulo (in press) were first picked from washed clays and then fluoridized.

It is worth while to remove as much matrix from the sample as possible. Soft clays may be disaggregated by being air-dried, soaked in paint thinner or paraffin, and then vigorously boiled in water with soda ash. More indurated mudstones and shales may require simmering in Quaternary 'O' (Zingula 1968), but since that detergent is weakly acid, prolonged treatment results in some etching of skeletal calcite; we prefer to treat particularly intractable samples directly with HF.

The partially cleaned fossil material is reacted with HF; the optimum strength of the acid and length of the reaction time vary from sample to sample. We have used 48% HF and reaction times of between 5 minutes and 1 hour for small specimens; for larger specimens weaker acid (approximately 6%) and longer reaction times (up to 24 hours) as advocated by Grayson (1956, p. 78) lead to better results. The fluoridization can be judged to have proceeded far enough when the surfaces of the ossicles appear bleached; it is not necessary to convert whole ossicles to fluorite.

Two adverse effects can occur during fluoridization. Firstly, the ossicles may crack and pieces may spall off. This can be largely avoided by reducing the reaction time to a minimum and by diluting the acid. Secondly, a glaze-like precipitate of fluorite may form on the surface of the ossicles. This can be prevented by using a large enough quantity of acid (we have found five times the volume of material being fluoridized a suitable amount). When the specimens have been fluoridized, they should be thoroughly washed and dried. Specimens for study under the scanning electron microscope should be transferred to stubs immediately, because their delicate surfaces can be easily damaged by abrasion.

Although we have been interested principally in the preparation of crinoid material, our bulk samples have contained many other fossils, most of which appear perfectly preserved after fluoridization. We believe that the technique may have general application in cleaning small fossils for study under the scanning electron microscope.

Because hydrofluoric acid is extremely dangerous, the fluoridization process should always be carried out in a properly designed fume cupboard with an efficient extraction system, by an operator wearing protective clothing, rubber gloves, and a face-mask. The reaction between the sample and the acid may be very vigorous, and large amounts of carbon dioxide may be generated rapidly. It is important, therefore, to treat the sample in an adequately large polythene vessel to prevent froth from forming and spilling out. We fluoridize approximately 10 g of bulk sample in an 80 mm-diameter 400 ml polythene beaker.

COMMENTS ON THE SPECIMENS ILLUSTRATED

The four ossicles illustrated in Plates 94 and 95 were obtained from a bulk sample of the soft clay shale above the Charlestown Main Limestone, collected near the bathing pool, St. Monance, Fife, Scotland (National Grid Reference NO 536 020). The shale is of Lower Carboniferous (Brigantian) age and has been correlated with the Neilson Shell Band (George *et al.* 1976, fig. 14, p. 53). The sample was partly disaggregated by being soaked in paraffin, and then boiled in water with soda ash. Small amounts of the disaggregated material were reacted with 48% HF for 1 hour. The fluoridized ossicles were mounted on stubs and coated with carbon and a gold palladium mixture, and were examined using an ETEC Autoscan, Model H-1, scanning electron microscope. The diameters of stereom pores were measured on enlarged scanning electron micrographs and the surface porosity by point counting along two mutually perpendicular axes as suggested by Macurda and Meyer (1975, p. 2). The terminology used is from Ubaghs (1978, T. 58 *et seq.*). The illustrated specimens and other representative material are deposited in the palaeontological collections of Trinity College, Dublin (catalogue numbers prefixed TCD).

Pentagonal columnals (TCD 19861-3) (Pl. 94, figs. 1, 3)

Columnals of this kind are moderately abundant in the sample. The longest pluricolumnal found consists of a nodal between two pairs of internodals. The nodal is cirrus-bearing and approximately

0.8 times as long as wide; the internodals are of two orders with length to width ratios of 0.4 and 0.6. The sides of the columnals are straight, or have a ridge or swelling around the equator, a feature particularly well developed on the nodals. Each nodal has one or two cirral sockets positioned between the equator and the joint surface. The sockets are comparable in some respects to cirral facets of Mesozoic crinoids illustrated by Ubaghs (1978, T. 85, fig. 61). They are gently concave and slope towards the joint face. The lumen of the axial canal is a vertical slit. The half of the socket closest to the equator of the columnal is furnished with short culmina; the half closest to the joint face is smooth. The sides of the columnal are formed of dense stereom with a surface ornament of slightly raised granules approximately 15 μm in diameter.

In facet view (Pl. 94, fig. 1) the following regions of the articulum can be differentiated:

1. The lumen, approximately 20–25% of the width of the articulum, which appears faintly five- or ten-lobed in well-preserved specimens.
2. An adaxially sloping concave area surrounding the lumen (the floor of the spatium), approximately 10–12% of the width of the articulum. The degree to which this region is depressed is variable; it is very shallow in the specimen illustrated. It is floored by open stereom (round to ovoid pores, with diameters from 6 to 14 μm , mostly about 12 μm) which in broken specimens can be seen to form a thin layer overlying denser paraxial galleried stereom like that flooring the areola.
3. In some specimens (but not the figured example) the outer margin of the floor of the spatium is raised to form a narrow perilumen constructed of denser stereom.
4. The areola (approximately 10–15% of the width of the articulum) which is flat and floored by paraxial galleried stereom (pore diameter 6–9 μm ; surface porosity approximately 44%). Most pores are subrounded and bounded by four trabeculae and many are arranged in long slightly arcuate rows.
5. The crenularium (approximately 10–15% of the width of the articulum) consisting of steep-sided culmina and crenellae (Pl. 94, fig. 3). The top of the culmina and base of the crenellae are approximately equidistant from the level of the areola. The surfaces of the culmina are dense with conspicuously thickened trabecular intersections (pore diameters are 2.5–5.0 μm ; surface porosity 30% or less), but are underlain by paraxial galleried stereom. The crenellae are mostly floored by galleried stereom similar to that of the areola (pore diameters 7–10 μm), but in some the stereom is much more open and labyrinthic.

A conspicuous feature of the articulum is the set of large tunnel-like pores (up to 35 μm in diameter) which in several specimens can be seen to completely penetrate the columnal. They are crudely arranged in ten lines and extend to the outer part of the areola.

In most respects the microstructure of these Carboniferous columnals is comparable with that of Recent and Mesozoic columnals described by Macurda and Meyer (1975, 1976) and Roux (1971). The galleried stereom of the areola probably housed ligament fibres. The denser stereom of the perilumen and of the crenularium served as bearing surfaces. The large pores penetrating the columnals may have contained nerves, as suggested by Macurda and Meyer (1975, p. 3) for similar pores in the columnals of the Recent species *Isocrinus blakei*. The pore diameters of the columnals described here are consistently smaller than those reported for most Recent and Mesozoic forms.

The taxonomic affinity of the specimens is not known. They almost certainly belonged to a cladid inadunate, possibly an ampelocrinid in view of the pentagonal stem and cirrus-bearing nodals.

Elliptical columnal of Platycrinites (TCD 19864–6) (Pl. 94, figs. 2, 4)

Columnals with elliptical articular surfaces are moderately common in the sample. They vary considerably in shape. The majority, mainly smaller specimens, are longer than wide and have a distinct equatorial waist. Most of them bear scattered nodes or blunt spines. A few specimens are wider than long, and some of these, possibly nodals, have conspicuous equatorial spine-bearing flanges. The articular surfaces are also variable although a basic pattern can be observed in all of them: a raised fulcral region along the major axis of the face separates two gently concave fields. The lumen is small and elliptical and is surrounded by open paraxial galleried stereom (pore diameters up to 13 μm ; porosity approximately 37%). The central parts of the bifascial fields are floored by paraxial

galleried stereom with pore diameters typically 6–10 μm and porosity approximately 32%. The peripheries of the faces are slightly raised above the bifascial fields and are formed of denser stereom (pore diameter 3–5 μm ; porosity less than 30%). The long axis of the articular surface is occupied by a fulcral region which in many specimens consists of a broad slightly raised ridge of dense stereom (pore diameter typically 4 μm ; surface porosity approximately 20%). In some specimens the surface of the fulcral region is crossed by low, dense, vermiform ridges. At each end of the major axis of the articular surface are raised culmina, generally three in number, which rise above the level of the fulcral region (Pl. 94, fig. 4). They interlock with crenellae of adjacent columnals. The culmina are formed of dense stereom (pore diameters typically less than 4 μm ; porosity less than 20%) and the crenellae are floored by galleried stereom (pore diameter typically 9 μm). The major axes of opposing faces of many of the columnals are set at 90° to each other.

The ossicles are easily identified as belonging to *Platycrinites* but their specific identity is not known. In many respects their structure is comparable with that of the columnals of the Recent millericrinid *Democrinus* (Macurda and Meyer 1975, pp. 4, 5) which also has synarthral articulation. In the Scottish *Platycrinites*, however, the fulcral ridge is much less dense than in *Democrinus*, and the elaborate keying mechanisms of that genus are not developed. Instead, limited symplectial articulation occurred at both ends of the fulcral ridge.

Inadunate brachial (TCD 19867–9) (Pl. 95, figs. 1, 3)

This kind of brachial is the most common in the sample. All examples that have been found are cuneate, pinnule-bearing, higher than long, and most have nodes or blunt spines on the aboral surface, particularly along the distal margins. All the brachials were joined by oblique muscular articulations; the fulcral ridges on the two faces of a brachial may diverge by as much as 60°. The following regions may be differentiated on the articular surfaces (Pl. 95, fig. 1):

1. The fulcral ridge, which is narrow at its mid-point and widens slightly at both ends to approximately 75 μm in typical specimens. The ridge is constructed of dense stereom (pore diameters typically 4 μm or less; porosity approximately 20%).

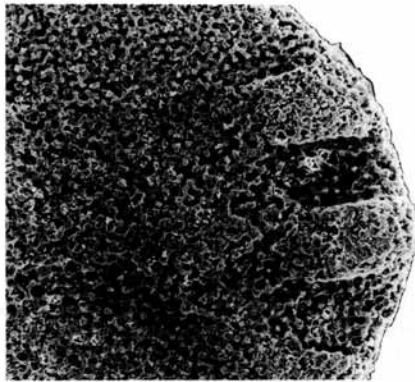
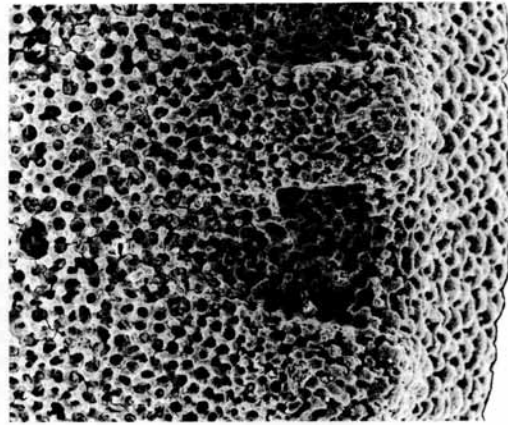
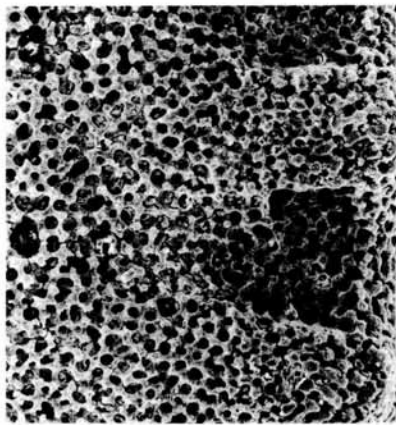
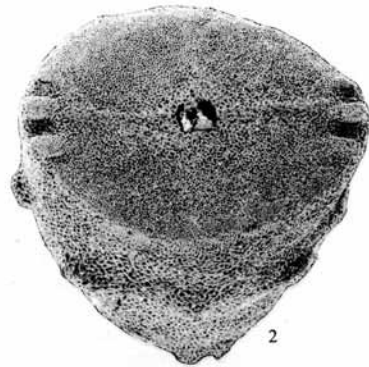
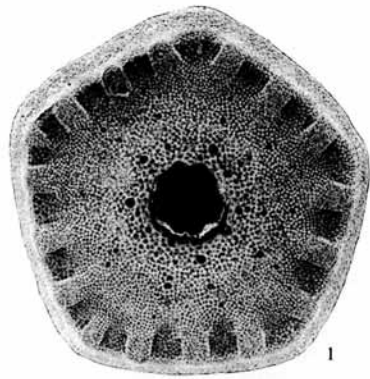
2. A slightly depressed area less than 30 μm deep bounded by the fulcral ridge and the aboral margin. By analogy with Recent crinoids, this area in Palaeozoic inadunates has been identified as the aboral ligament fossa which housed the extensor ligament bundles. It is floored by galleried stereom (pore diameters typically 7 μm ; porosity approximately 35%). The pores are subrounded and arranged in a crude rectilinear pattern. In most specimens (but not the figured example) a distinct small deeper ligament pit occurs just aborally of the mid-point of the fulcral ridge.

3. Two wide subequal depressions, typically less than 30 μm deep, adoral of the fulcral ridge and on either side of its mid-point, which have been identified as interarticular ligament fossae. They are floored by galleried stereom in which the trabeculae and pores are conspicuously wider than elsewhere on the articular surface. Pore diameters generally range from 10 to 15 μm ; the porosity is approximately 40%.

EXPLANATION OF PLATE 94

Figs. 1, 3. Fluoridized pentagonal columnal (TCD 19861), from the shale above the Charlestown Main Limestone, St. Monance, Fife (Lower Carboniferous; Brigantian age). 1, slightly oblique view of the articular surface, $\times 45$. 3, stereopair of the crenularium and outer part of the areola, located at about 7 o'clock on fig. 1, $\times 230$.

Figs. 2, 4. Fluoridized *Platycrinites* columnal (TCD 19864), from the shale above the Charlestown Main Limestone, St. Monance, Fife (Lower Carboniferous; Brigantian age). 2, oblique view of columnal, $\times 38$. 4, stereopair of part of the fulcral ridge and culmina, from the left side of fig. 2, $\times 150$.



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4. A slightly raised area extending from the adoral groove to the mid-point of the fulcral ridge and separating the interarticular ligament fossae. This area bears a very weak medial groove which ends short of the fulcral ridge. The stereom of the raised area is galleried (pore diameter 6–8 μm) along the margins and more open (pore diameter 10–15 μm) and less regular along the median groove.

5. Well-marked, unequal, 'rabbit-ear'-shaped depressions on either side of the ambulacral groove, which have been interpreted as flexor muscle scars. They are floored by distinctive dense labyrinthic stereom (pore diameters mostly less than 4 μm ; porosity approximately 20%). The surfaces of the fossae are formed by blunt-ended trabecular rods projecting upwards (Pl. 95, fig. 3).

All well-preserved specimens can be seen to have three pores 20–30 μm in diameter adoral of the fulcral ridge on both articular surfaces. Two of them lie along a line normal to the bisectrix of the angle of the adoral groove; the third is between the other two, closer to the fulcral ridge.

Many of the features observed are similar to those reported by Lane and Macurda (1975) for the Pennsylvanian cladid inadunate *Aesiocrinus magnificus*. The upward projecting trabecular rods of the 'rabbit ear' fossae were probably sheathed with a thin connective tissue layer to which the muscle fibres were attached, as illustrated for the Recent crinoid *Annacrinus* by Roux (1974b, pl. 1, figs. 6–7). An unusual feature of the Scottish brachials is the presence of the three canals interpreted here as aboral nerve canals. In Recent crinoids there is only one canal in the brachials; Lane and Macurda (1975) showed that in *Aesiocrinus* a 'double-barelled' nerve canal was present. We have found the 'double-barelled' arrangement in a number of different inadunate brachials, but the triple canal has only been found in the ossicles described here. We are unable to identify the brachials. They clearly were from a cladid inadunate. We have recovered axillary brachials, which show that the rays were branched and that the first dichotomy was above the first primibrachial.

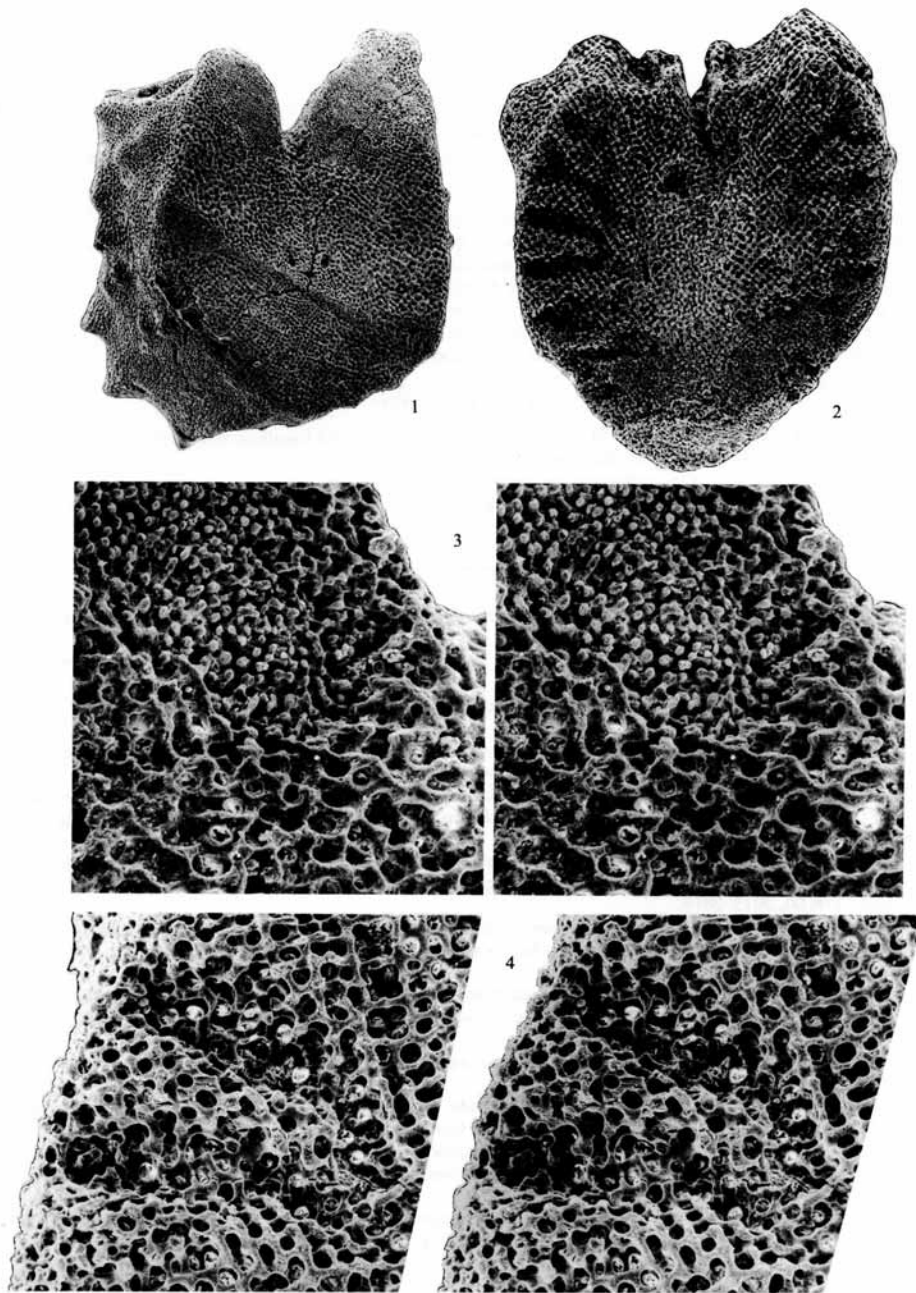
Flexible brachial (TCD 19870–3) (Pl. 95, figs. 2, 4)

Brachials of this kind are moderately abundant in the sample, but there is considerable variation in the ratio of width to height; possibly more than one crinoid species is represented. The proximal articular surfaces are extended aborally into patelloid processes and the distal surfaces each have a fossa into which the process fits. In the specimen illustrated (Pl. 95, fig. 2), the lateral margins of the articular surface are crenulate with steep-sided culmina approximately 100 μm high. There is no fulcral ridge, but a fulcral ridge is present on some larger specimens. The stereom of the articular surface occurs in three different arrays. Over most of the surface, excluding the area around the patelloid process and a narrow median area extending aborally from the adoral groove, the pores are quadrangular to round, the diameters of 13–20 μm , and the porosity is approximately 45%. The trabeculae on either side of the aboral/adoral axis of the brachial are oriented at similar angles to the median line and produce a markedly rectilinear pore pattern. The stereom pores visible in side view are approximately the same dimensions as on the articular surfaces, so that the trabeculae form a regular cubic framework. In the median region, aboral of the adoral groove, the pores are slightly reduced in size and the regular arrangement of the pores is lost. On the aboral part of the articular surface around the patelloid process, the stereom is much denser (pore diameter 5–8 μm ; porosity less than 30%) and less regularly arranged.

EXPLANATION OF PLATE 95

Figs. 1, 3. Fluoridized inadunate crinoid brachial (TCD 19867), from the shale above the Charlestown Main Limestone, St. Monance, Fife (Lower Carboniferous; Brigantian age). 1, slightly oblique view of proximal articular surface, with pinnule facet to the left, $\times 48$. 3, stereopair of the left side 'rabbit ear' fossa and adjoining interarticular ligament fossa, $\times 300$.

Figs. 2, 4. Fluoridized flexible brachial (TCD 19870), from the shale above the Charlestown Main Limestone, St. Monance, Fife (Lower Carboniferous; Brigantian age). 2, view of the proximal articular surface, $\times 48$. 4, stereopair of culmina, located near the middle of the left margin in fig. 1, $\times 180$.



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These flexible brachials cannot be more closely identified; all the Carboniferous flexible ossicles encountered in this study have had remarkably similar microstructure.

Most authors (for instance, Van Sant and Lane 1964, p. 51) have suggested that flexible crinoids had only ligamentary articulations. Whether ligament fibres penetrated all the 'cubic' structured stereom or were restricted to certain areas is not certain, but the former arrangement seems more likely.

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