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Techniques of Study:
Collection, Preparation, and Analysis of the
Paleozoic Stromatoporoidea

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PART E, REVISED, VOLUME 4, CHAPTER 15A: TECHNIQUES OF STUDY: COLLECTION, PREPARATION, AND ANALYSIS OF THE PALEOZOIC STROMATOPOROIDEA

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FIELD OBSERVATIONS AND COLLECTING

COLLECTING IN CARBONATES OF THE REEF FACIES

Most stromatoporoids are preserved in carbonate sediments formed within a reef environment. They are, therefore, most common in unbedded or poorly bedded limestones and dolomites of the reef facies, or in bedded carbonates deposited in adjacent lagoonal or foreslope deposits. In such carbonates, the fossils do not weather free of the matrix and must be extracted, usually in fragments, by breaking the rock. Where the rock is broken in fragments in quarrying, this may not be difficult, but in natural outcrops where the unbedded reefal facies commonly forms smooth-surfaced domes, it may be almost impossible with a geologist's hammer. Where a specimen must be extracted to satisfy a sampling scheme, a portable circular saw with a cement-cutting blade can be used to make grooves around the sample and allow a cold chisel to chip it out. The saw, however, generates much rock dust, therefore the operator should wear a protective mask. Generally, in such host rocks, the collector must be satisfied with fragments that will provide enough material for the two thin sections required for identification.

In many reef outcrops, the shapes of stromatoporoids can be observed only in a random cross section. Because the whole specimen can rarely be collected, the impression of shape that such sections allow should be recorded in notes before collection. The study of stromatoporoids in cores from reef reservoirs in the subsurface involves similar problems, although the regularity of the

core surface may make estimates of shape in three dimensions easier. Samples must be cut from the core with a rock saw. Core storage agencies will generally allow only a small sample to be cut out of the core (for example, a cubic inch every linear foot or 15 ml/0.3 m).

In areas of cold climate, such as high altitudes and latitudes, carbonate outcrops are commonly covered with a thin tufa that obscures fossils. Fresh rock faces recently exposed by frost wedging that show the rock texture better can usually be found in these areas, but the surface may have to be broken with a hammer to reveal the fossils within. Reef textures and fossils are most clearly revealed in outcrops repeatedly abraded by flooding rivers, tides and waves, and winds charged with sand.

COLLECTING IN FOREREEF SLOPES

The carbonates deposited at the margins of Paleozoic reef complexes are commonly affected by pervasive dolomitization that reduces stromatoporoids to so-called ghosts. The faunas of these margins are commonly much better preserved in debris blocks that have slumped from the steep reef front onto the forereef slope (MOUNTJOY & others, 1972; CONAGHAN & others, 1976). Reef blocks several meters across may have traveled several kilometers downslope into basinal deposits and now constitute beds of megabreccia. Well-preserved stromatoporoid faunas have been described from such debris flow deposits (SCRIVASTAVA, STEARN, & MOUNTJOY, 1972; POLAN & STEARN, 1984).

The depositional slope on which benthic organisms (such as stromatoporoids in position of growth) grew can be estimated by

measuring growth axes. If it is assumed that the growth axis of domical and dendroid stromatoporoids is on average vertical (that is, they are geotropic or phototropic), then the divergence between the axis and a line perpendicular to the bedding will indicate the slope on which they grew. The orientation of the growth axis can be determined if the stromatoporoid is exposed in more than one plane and its pole measured with a simple device. A dowel that can be oriented along the growth axis and fixed at one end temporarily with plasticine is attached at right angles at the free end to a flat disk whose strike and dip can be measured with a Brunton compass. From these data, the poles of the bedding and growth axes can be plotted on a stereonet. In deformed beds, the post-depositional tilt of the beds must be compensated for by modifying the poles of growth by the strike and dip of the bed using a stereonet.

ORIENTATION

KOBLUK (1974) measured the azimuths of dendroid stromatoporoids on bedding planes in the Miette Reef Complex in Alberta. He analyzed the results by a chi-square test to show that the stems had a preferred north-west orientation. KOBLUK, BOTTJER, and RISK (1977) measured the proportion between domical stromatoporoids of various sizes that were in growth position and those that were disoriented. They found no difference in mean size between those that were turned over and those in growth position. The toppled or upright position of stromatoporoids has also been measured by KERSHAW (1981) at the Kuppen biostrome in Gotland and by KERSHAW and RIDING (1980) in Devon.

MARLS

In argillaceous limestone successions (marls), stromatoporoids may weather free or be easily extracted from the soft matrix. Such successions are found in the Silurian rocks of Scandinavia and Britain. There the growth forms of stromatoporoids are much

easier to study, and surfaces of the skeletons can be examined in detail. Many of the studies of the relationship of growth form, environment, and taxonomy have been made in these areas (for example, KERSHAW 1981, 1984, 1993; KERSHAW & KEELING, 1994) and are discussed in the chapter on growth form (see *Treatise Online*, Part E, Revised, Volume 4, Chapter 9B).

STATISTICALLY CONTROLLED SAMPLING

Although various research workers have advocated a statistical approach to the study of the distribution of stromatoporoid taxa or shapes in reefs, local conditions rarely make random sampling, a requirement of most statistical tests, possible over a large area. Stromatoporoids on extensively exposed horizontal bedding planes have been divided into quadrats and surveyed as to shape and size over areas of several tens of square meters. Quarry faces and mountain cliffs may expose large vertical sections of a reef deposit but are only rarely accessible for random sampling over extensive horizontal or vertical distances.

Estimates of the proportion or density of various growth forms or types of organisms on a face or bedding plane can be made by drawing random lines, or stretching strings randomly, across a face. The constituents along the line are identified. Either the total length of the line lying upon each constituent is summed, or the line is marked at a regular interval (e.g., every 5 cm), the constituent beneath each mark is recorded, and the number of occurrences is taken as a measure of the relative abundance of each constituent. The latter method, a form of point counting, is the quicker of the two (POLAN & STEARN, 1984). Line intercept transects were also used by EDINGER and others (2002) in their survey of Onandaga reefs. SANDSTRÖM (1998) drew sketches of outcrops on Gotland at 1:5 scale and point counted these sketches to quantify the identity and shape of the stromatoporoids. Because stromatoporoids can rarely be iden-

tified taxonomically on external appearance alone, methods like these that depend on identification without collection and processing do not give information for plotting the distribution of species in a reef.

MAPPING

Detailed maps of the distribution of stromatoporoid shapes and taxa on small representative areas of biostromes and bioherms have been made by many investigators. Only studies in which the occurrence of stromatoporoids is essential, rather than incidental, are mentioned here. KERSHAW (1984, 1990) and KANO (1989, 1990) have published maps showing the distribution of stromatoporoids in the reefs of Gotland. SCHNEIDER and AUSICH (2002) have mapped the distribution of various framebuilders, including stromatoporoids, in the lower Silurian Brassfield Formation of Ohio. FAGERSTROM and BRADSHAW (2002) drew maps of the distribution of Early Devonian stromatoporoids in the reef facies at Reefton, New Zealand. Stromatoporoids are prominent in the maps of Late Ordovician patch reefs in Alabama presented by STOCK and his colleagues (STOCK & BENSON, 1982; CROW & others, 2001).

GENERAL

The usual precautions of labeling and cataloguing that apply to all fossils are not discussed here. Because specimens broken from carbonates rarely are complete or show details of surfaces, wrapping of individual specimens is usually unnecessary, but pieces broken from a single large specimen should be kept together if an approximation of the abundance of the individual taxa in a collection is to be obtained from the contents of the collection bag.

A collection of papers on various laboratory techniques for preparation of fossils published as Paleontological Society Special Publication 4 (FELDMAN, CHAPMAN, & HANNIBAL, 1989) contains descriptions of many procedures relevant to stromatoporoids. A similar collection of papers was

assembled earlier by KUMMEL and RAUP (1965).

THIN SECTIONS SIZE AND THICKNESS

Since NICHOLSON introduced the method about 1875, stromatoporoid workers have used thin sections viewed in transmitted light to identify these fossils (WELLS in FELDMAN, CHAPMAN & HANNIBAL, 1989). Two sections are required to define the skeletal elements in three dimensions; one parallel to the growth surface (tangential) and the other perpendicular to it (longitudinal). Large thin sections are better than small ones, because they show the local variation of structural elements in the various phases of the skeleton. LECOMPTE (1951–1952) studied sections that were up to 5 cm × 10 cm. However, such large sections are very difficult to make uniformly thin enough to show microstructure clearly. Such sections are also difficult to store. The most useful size for thin sections is 44 mm × 75 mm, as commercially available cabinets for storing 22 mm × 75 mm slides can be modified to hold them. Sections ground to standard petrographic thickness of 30 μm are too thin to show structural elements clearly. The appropriate thickness of the section can only be determined experimentally as it depends on the particular type of preservation but should be such that the structural elements are translucent, their microstructure is clear, their edges are in sharp focus in photographs at ×10 magnification, and the crystal boundaries in the galleries are sharp. Most illustrations that appear out of focus are taken of thin sections that are too thick. Unfortunately, sections of the holotypes of older taxa are commonly too thick to show microstructure clearly.

ADHESIVES

Until the middle of the 20th century, thin sections were made exclusively with Canada Balsam. If the adhesive was properly cooked, such sections were archival, and many in collections of the late 1800s are in pristine

condition. In the 1950s, thermoplastics, such as Lakeside 70, were used to cement the specimen to the slide. These were convenient but were difficult to clear of bubbles. Covering agents used at this time included the commercial product Permount, which proved unsatisfactory because it became opaque after about 20 years. Beginning about 1960, epoxy cements such as Araldite became the choice of many preparators, as, once set, they were impervious to heat or chemicals. Plastic solutions that were allowed to flow over the surface and set were also used to form a clear membrane on the thinned specimen in place of a cover glass. About 1990, adhesives that set by the action of ultraviolet radiation became generally available and proved to be a great convenience for thin-section preparation. The adhesive film between the specimen and glass slide is set by ultraviolet light shone through the glass slide for a few minutes. It sets only under the specimen where not exposed to the air and the excess cement around the specimen can be wiped off with methanol. If the cover glass is to be permanently attached, the same adhesive can be used. Canada Balsam remains the most reliable, long lasting, and easily removable cement for cover glasses.

IMPREGNATION

In stromatoporoids that have been dolomitized, the galleries and pores of the stromatoporoid skeleton are empty, and they trap air bubbles and abrasive in the cements used in making thin sections. The pores must be filled before the specimen is cemented to the glass to exclude these undesirable contaminants. In the traditional method, the specimen is immersed in a low-viscosity, slow-setting epoxy treated with hardener and is placed in a chamber in which pressure can be reduced by a vacuum pump (WELLS in FELDMAN, CHAPMAN, & HANNIBAL, 1989, gave trade names of products). As ambient pressure is reduced, the air escapes from the pores, and the epoxy takes its place. Unfortunately, the low pressure produced by

the vacuum pump may evaporate the more volatile constituents of the epoxy mixture, and the proper proportions of hardener and resin that ensure setting may be modified. If the pores are not interconnected, the impregnating epoxy may fail to reach them all. STEARN (1996) proposed a method using melted paraffin wax to fill the pores on the polished surface and diamond-faced laps to eliminate loose abrasive. Excess wax is scraped from the surface with a blade, and the specimen is cemented to the glass with ultraviolet-setting adhesive.

SERIAL SECTIONS

Successive, parallel, thin sections or polished surfaces cut through a fossil specimen allow it to be reconstructed in three dimensions. Computer programs are available to assist in combining the multiple images into a three-dimensional reconstruction. This technique may involve the destruction of the specimen by grinding it away to produce the successive polished surfaces, or closely spaced thin sections may be prepared by repeatedly cementing the specimen to a microscope slide and slicing it off as close to the slide as possible. The spacing of the sections is as close as the thickness of the blade. This latter procedure was used by STEARN (1997) to prepare a set of serial thin sections to act as neotypes for *Amphipora*. Another method of preparing three-dimensional reconstructions of large specimens of corals that could be applied to stromatoporoids was described by HAMMER (1999). He placed successive polished sections of *Catenipora* on a scanner and used a computer program to produce a three-dimensional image of its growth.

A nondestructive technique using computer tomography to delineate the interior of a stromatoporoid has been tested by BEUCK and others (2008). The C-T scan allowed the authors to reconstruct the trace of a boring in a stromatoporoid skeleton from Gotland in three dimensions. Differences between the physical properties of the boring and stromatoporoid skeleton allowed its reconstruction, but the

method does not reveal the internal structure of the stromatoporoid.

REFLECTED LIGHT

Nearly all thin sections of stromatoporoidea are best observed in transmitted light at magnifications of $\times 10$ to $\times 50$, but some dolomitized specimens show much more detail in reflected light against a white background. Lights are directed at the thin section surface, about 45° from the plane of the section. Photography under these conditions is difficult, as the level of the light reflected and contrast are low.

ULTRATHIN SECTIONS

In sections of several tens of micrometers thickness, the high birefringence of calcite makes resolution of the crystal boundaries within the structural elements difficult. To examine this aspect of the microstructure of corals, LAFUSTE (1970) introduced the technique of polishing the face of the specimen that is to be adhered to the slide and grinding it carefully to a thickness of two or three micrometers. At this thickness, the interference colors of calcite under crossed polars are grey and yellow. LAFUSTE's work in the 1970s and 1980s was largely applied to tabulate and rugosan corals and convinced him that his slides showed the preservation of original biocrystals. Many of the elongate calcite crystals had a shape he referred to as dented (*bosselfure*) with small embayments down their length. The technique was applied to stromatoporoidea by STEARN and MAH (1987) to investigate the nature of the specks in structural elements (see *Treatise Online*, Part E, Revised, Volume 4, Chapter 9D, Fig. 2,2). MISTIAEN (1994) illustrated many ultrathin sections of stromatoporoidea in his discussion of the density of the skeleton.

STATISTICAL EVALUATION OF TAXONOMIC DIFFERENCES

Relatively little work has been done on specifying the variability of the stromato-

poroid skeleton statistically or on using the parameters that define this variability to distinguish between species or other taxa. FAGERSTROM and SAXENA (1973) used statistical tests to assess whether the variability within a single section of *Syringostroma sherzeri* was representative of the whole of the skeleton. FAGERSTROM (1982) made extensive measurements of the structural elements of specimens and calculated similarity coefficients to distinguish between and to group taxa of stromatoporoidea from the Detroit River Group. STEARN (1989) recorded the intraspecific variability of stromatoporoidea and related organisms in terms of Simpson's coefficient of variability. The most extensive use of statistics to distinguish between species has been by STOCK and BURRY-STOCK (STOCK & BURRY-STOCK, 1998, 2001; STOCK, 1991, 1997) who have applied multivariate procedures to separate species in large collections from the Lower Devonian of New York. They used cluster analysis in an exhaustive study of 103 specimens of *Habrostroma* to distinguish the two species, *H. centrotum* and *H. consimile*, and to rate by canonical correlation analysis which of the skeletal features were most useful in distinguishing them (STOCK & BURRY-STOCK, 2001). Research into stromatoporoidea phylogeny using concepts of cladogenesis has been limited, probably owing to the small number of skeletal characters that these fossils present for analysis. The only cladogram of stromatoporoidea genera published so far is based on 16 characters of the labechiids (WEBBY, 1994).

CATHODOLUMINESCENCE

If thin sections are uncovered, their microstructure can be investigated under the microscope by cold cathode luminescence. This technique is particularly suitable for assessing the degree of alteration of the skeleton and delineating the crystal boundaries (KERSHAW, 1994). The reasons why certain calcite crystals luminesce with different colors is still unclear, but most carbonate workers believe it is due to slight

impurities in their crystal lattices. KERSHAW's studies (1994) confirmed that different stromatoporoids secreted skeletons of aragonite or high magnesium calcite with various proportions of magnesium.

Attempts to detect organic matter within the skeleton of stromatoporoids by stimulating fluorescence in ultraviolet light under the microscope showed no response from thin sections (C. W. STEARN, unpublished data). Stromatoporoids, like scleractinian corals, seem to have been able to secrete skeletal carbonates free of organic matter. However, CLARK (2005) reported organic matrix dispersed through a stromatoporoid skeleton.

SCANNING ELECTRON MICROSCOPY

The relationship between the arrangement of crystals and the structure and microstructure of the stromatoporoid skeleton can be studied on polished surfaces that have been etched or on broken surfaces with the scanning electron microscope (SEM). The technique was described by STEARN (1977). Although other workers polished the specimen highly and etched it with weak acids such as acetic or formic, STEARN (1977) found that good results were obtained by grinding with 600 grain silicon carbide and etching with 10% hydrochloric acid for 10 seconds. The specimen surface is then coated with a metallic film (usually gold-palladium) or carbon and placed in the SEM. The relief produced by the differential etching is imaged by the microscope at magnifications up to the tens of thousand times, but for most microstructural studies, magnifications of a few hundred times are most useful (see *Treatise Online*, Part E, Revised, Volume 4, Chapter 9D, Fig. 2, 1, Fig. 11–15). To test whether textures seen in etched specimens are artifacts of the preparation process, specimens may be fractured and the broken surface examined. Some investigators, to insure that the fracture is random and not guided by fine pores and cracks, have soaked

the specimen in a penetrating liquid of very low viscosity (such as ethyl ether) and immersed it in liquid nitrogen to freeze the liquid before fracturing the specimen (STEARNS & MAH, 1987).

Direct comparison of transmitted light images with scanning electron micrographs of the same part of the specimen is difficult. STEARN (1977) described a technique of cutting a disk about 5 mm in diameter from a thin section with an abrasive jet charged with alumina, such as those used to excavate small fossils. The disk is photographed at high and low powers in transmitted light and marked with a reference mark (such as a scratch or depression) that will appear in the electron microscope. It is then prepared for the SEM in the usual way, and the area that was photographed at high power is located in the scanning electron image by reference to the mark. However, comparison of light microscope and SEM images is not easy, because the specimen in the SEM is tilted at an angle, chosen by the operator, to the electron beam, foreshortening its image in the direction of tilt, and the photograph is an inverted mirror image of the scanning electron micrograph. Scanning electron micrographs of stromatoporoids have been published by STEARN (1977, 1989), STEARN and MAH (1987), and RUSH and CHAFETZ (1991).

GEOCHEMISTRY

The original skeletal composition of Paleozoic stromatoporoids and related living hypercalcified sponges has been studied through analysis of the structural elements for strontium, magnesium, lead, and rare earth elements. Results of these studies are further discussed in the chapter on skeletal microstructure and mineralogy (see *Treatise Online*, Part E, Revised, Volume 4, Chapter 9D). The results have been obtained largely through microprobe x-ray fluorescence and laser-ablation plasma mass spectrometry.

Biologically secreted aragonite is enriched in strontium and may contain up to 9000 ppm Sr²⁺. RUSH and CHAFETZ (1991)

supported their conclusion that the original mineralogy of Devonian stromatoporoids was high magnesium calcite with microprobe analyses of Sr^{2+} and Mg^{2+} . MALLAMO (1995; MALLAMO & STEARN, 1991) made cross plots of Sr^{2+} and Mg^{2+} from microprobe analyses of living corals, recently calcitized corals, and stromatoporoids of various ages. He found that high values of Sr^{2+} in the structural elements of Ordovician labechiids relative to that of the gallery cements justified the conclusion of an original aragonite mineralogy. Younger stromatoporoids do not show the elevated Sr^{2+} and probably secreted high magnesium calcite.

ROSENHEIM and others (2004) found that the strontium-calcium ratio in living *Ceratoporella* was an indication of the temperature at which the aragonite skeleton was secreted, but this method has not been applied to fossils. WEBB, WORHEIDE, and NOTHDURFT (2003) measured the distribution of rare earth elements (REE) in stromatoporoids from the Devonian of the Canning Basin, Australia, and the living sponge *Acanthochaetetes*. The proportion of REE in the stromatoporoid was similar to that of sea water and suggested that its skeletal composition was originally calcite. LAZARETH and others (2000) measured lead in recent *Ceratoporella* to assess its relationship to environmental changes.

Identification of microdolomite by morphology in scanning electron micrographs as an indication of original magnesium calcite composition in Ordovician stromatoporoids has led to contradictory results (YOO & LEE, 1993; TOBIN & WALKER, 1998).

ISOTOPE STUDIES

NORRIS and CORFIELD (1998) collected a series of papers on the use of isotopes in paleontology.

To isolate a carbonate sample for isotope analysis of the skeleton from that of the galleries, a micropositioning stage driven by stepping motors and connected to a computer is used (DETTMAN & LOHMANN,

1995). A structural element in a polished thin section is drilled out with a dental drill 20 μm wide to a depth of 50 μm . To get a sample large enough for the mass spectrometer (10 μg), about 4 mm along the length of the structural element (e.g., a lamina) must be drilled out.

MALLAMO (1995) has applied analyses of oxygen and carbon isotopes in the stromatoporoid skeleton to the problem of whether the organisms were photosymbiotic. Because photosynthesis preferentially fixes ^{12}C , it increases the $^{13}\text{C}/^{12}\text{C}$ ratio in the skeleton but has only a minor effect on the oxygen isotopes (SWART, 1983). FRYKMAN (1986) plotted the C and O isotopes in stromatoporoids from Gotland but did not discuss the significance of the results for these fossils.

The proportion of O isotopes in the skeletons of modern corals is sensitive to temperature, and changes in the ratio of $^{18}\text{O}/^{16}\text{O}$ across the growth axis have been used to define annual increments. BOEHM and others (2000) have applied this technique to the skeletons of living hypercalcified sponges, but so far application of this technique to stromatoporoids to determine paleotemperatures has not been reported.

PHOTOGRAPHY

In 19th century works, the illustrations are engravings produced by lithography. While most of these illustrations are fair representations of the thin sections from which they were drawn, writers (e.g., STEARN, 1993) have commented that they cannot find the part illustrated in the plate in the type thin sections. In some publications (e.g., PARKS, 1936; GALLOWAY & ST. JEAN, 1955, 1957; GALLOWAY, 1960), the photographs are retouched, typically by whitening out details that the author decided were of secondary origin. The microstructures of such illustrations are rarely accurate representations of the nature of the specimen and in worst cases are misleading. Such retouching has

not been practiced in recently published papers.

Standard methods of photomicrography have been used in illustrating stromatoporoids. Although various magnifications have been used, the standard magnification of 10 for macrostructure and 25 for microstructure has been widely adopted and allows easy comparison between taxonomic descriptions. To increase depth of focus and uniformity of focus across the picture, the thin section can be placed in an enlarger and projected onto film. The image from the enlarger can best be captured on slow orthochromatic emulsions (for example, the now unobtainable Kodak 7302 or 5302), but such products are now difficult to find as manufacturers are discontinuing production of black and white films. To increase depth of focus in producing the negative, the initial magnifications should be kept low, typically $\times 3$, and the $\times 10$ image produced by enlarging the negative $\times 3.3$ onto paper. To save effort, some paleontologists have published negative prints produced by projecting the thin section directly onto printing paper rather than film. To compare such illustrations with those produced as photomicrographs, one must make a mental adjustment that the darker areas on the photograph would be lighter (less opaque) when the section is seen under the microscope.

Recording images with a digital camera or scanning photographs produced from film and paper allows the image to be stored in various memory devices, such as hard disks, zip drives, compact discs, or memory cards and manipulated for size, brightness, and contrast on a computer. As a result, these digital techniques have largely replaced film and paper methods, and all the illustrations in this volume have, at some stage, been digitized, although many were originally recorded on film and later scanned. So far, paleontologists have not confronted the problem that electronic manipulation of images may mislead readers as to the true state of the specimens, to the same extent that retouching photographs could mislead an earlier generation.

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