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FINAL REPORT

IOWA STATE HIGHWAY COMMISSION

RESEARCH PROJECT #70

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PROPERTIES OF CARBONATE ROCKS IN IOWA

-Final Report-

Investigators: Drs. Leo A. Thomas and Donald L. Biggs

Introduction: The Iowa Highway Research Project 70 began in the fall of 1959 with the objective of determining the basic properties of carbonate rocks occurring in the state. It was evident from the beginning that such a study would require several years and that the project would change in emphasis and method as knowledge was gained. During the two years of its operation, Iowa Highway Research Project 70 has allowed the investigators to establish techniques for study of carbonate rocks and to arrive at a better idea of the most efficient scheduling of such work.

This report will serve as the final report of Iowa Highway Research Project 70 and the work utilizing the scientific techniques and organizational policies developed under this project will be carried forward under a new title. The data accumulated to date and conclusions drawn therefrom are appended to this report.

STUDIES COMPLETED

Diffraction Studies

Much of the mineralogical study accomplished by X-ray diffraction studies was summarized in the progress report submitted to the Iowa Highway Commission in March of this year. Additional studies are herewith reported and the data from all rocks examined under HRP-70 in the form of diffractometer traces and X-ray data analysis sheets, are placed on file in the office of the Iowa Highway Commission Geologist.

Petrography

Petrofabrics:- Preferred orientation of crystals in carbonate rocks has been shown to exist in some cases. Previous techniques were limited to studying rocks composed of crystals of relatively large size. Many Iowa carbonate rocks are composed predominantly of crystals near or below the limit of resolution of the polarizing microscope. In order to study the petrofabrics of such rocks, it is necessary to work with X-ray diffraction. During the past year, the project has acquired the equipment for performing such studies. The technique depends only on the precise orientation of the sample for its accuracy, and on any crystalline material provides the data for a unique petrofabric determination in something less than two hours.

Sample Preparation

Oriented cylindrical cores of 5/8-inch diameter and approximately one inch length are cut from rock samples by means of a drill press (Plate I, figure 1). The data obtained from a hemisphere of any known orientation may be rotated into a standard projection, but it is generally most convenient to cut a core normal to the bedding plane. Consequently, the method of plotting and standardizing data which is given below is based on the assumption that the core be cut normal to the bedding plane and that the hemisphere be ground on the upper end of the core. Cores are cemented^{**} into tightly fitting sockets in brass spindles to insure correct axial alignment.

The spindle is placed in a collet chuck assembly which rotates at 81 rpm against the rotation of the lap wheel (Plate I, figure 2). The lap contains three grooves which are, in cross-section, 120° arcs of 0.250-inch radii circles. The outer groove is used with coarse grit for shaping; the inner two grooves with progressively finer grit for finishing. The chuck assembly is manually tilted back and forth through an arc which is normal to the groove at the point of contact and whose center of motion is the center of the hemisphere being generated; i.e., 0.250 inch above the bottom of the groove. The lap wheel height and chuck assembly location are adjustable to allow precise setting because slight errors result in very poor hemispheres. The sample axis may be inclined between 40° and 90° to the lap surface by means of the chuck assembly, so that the surface generated is slightly more than half of a sphere.

Sample Holder

Preferred orientation of crystals in a rock may be determined by the X-ray technique developed by Jetter and Borie¹ (1953). Hemispherical samples (described above in detail) are used to assure constant radiation intensity. The sample holder, which is used with a Geiger counter X-ray diffractometer, allows rotation of the sample about two axes without disturbing camera geometry. The holder moves as an integral part of the goniometer motion, so that the preferred orientation of any desired peak can be studied.

Rotation about the sample (polar) axis x_{∞} is continuous and is synchronized with the strip chart recorder, which moves four inches per minute. Viewed from above the hemisphere, the rotation of the sample is 1 rpm in a counterclockwise direction. x_{∞} may be tilted by intervals between 0° and 90° by means of the slip ring axis x_{∞} , which is parallel to and 0.250 inch from the goniometer axis x_{α} (Plate I, figures 3 and 4). The combina-

A thermal cement, such as Lakeside 70, is satisfactory.

1 Jetter, L.K. and Borie, B.S., Jr., "Method for the Gualitative Determination of Preferred Orientation." Journal of Applied Physics, 1953, 24:532-535.





Figure 1. Combination of rotations about x_{∞} and $x \not o$ results in the scanning of

circles perpendicular to xa.

Polar net.

Figure 2. Equal-area nets, showing intersections of parallels and meridians taken at 10° intervals.



Schmidt net.



Figure 3. Standard hemisphere.

Figure 4a. Side crosssectional view. The point recorded is the intersection of the normal to the reflecting plane with the extended hemisphere. By convention, the lower half of the sphere is the reference hemisphere.





Figure 4b. Top view. The sample rotates in a counterclockwise direction in its calibrated, stationary holder. If the normal to a crystallographic plane points downward in the direction of the strike, it is recorded at $q = 270^{\circ}$.



Figure 5. Standardization of a polar net. The $\ll = 270^{\circ}$ meridian is located in the strike direction, N 50° E, and a 17° southward rotation about this meridian corrects for the northerly dip of the bed. The dashed lines belong to the Schmidt net below. One quadrant of the standardized net is shown (solid lines). tion of these motions thus results in the scanning of circles on the hemisphere; each circle is normal to the sample axis at its center and represents one value of \emptyset . (Figure 1)

X-ray Study Technique

Vertical and horizontal translations allow alignment of the hemisphere so that its surface is always in focus at the goniometer axis. With $\emptyset = 0$, the sample is translated until the zenith of the hemisphere just touches the aligning knife blade.

Background intensities and the exact goniometer settings of peaks to be studied may then be determined after the recorder is properly calibrated. With 1° divergence and .003" receiving slits, the peak is roughly located by allowing the sample to rotate at any $\not p \neq 0$ while the goniometer is scanning the peak area. The goniometer motion is stopped when a maximum area seems to be reached, and the sample is allowed to rotate to the highest possible intensity. The background intensity for any peak may be determined by interpolation from the intensities on both sides of the peak:

$$B = D_1 \times SF_1 \times M_1 \times K$$

where B is the background intensity in counts per second; D_1 the background height on the strip chart as interpolated between the background height on both sides of the peak; SF_1 the scale factor; and M_1 the multiplier. K depends upon the calibration of the recorder and is constant so long as the calibration operation is performed consistently. Since this work is concerned only with relative intensities, the function of K can be ignored.

The strike direction is set at mechanical zero. For each peak being studied, a complete α -rotation is recorded at $\emptyset = 0^{\circ}$, $\emptyset_1 = 10^{\circ}$, etc., through $\emptyset = 90^{\circ}$. The focus must be checked with the knife blade at each \emptyset -setting and corrected if necessary. The average figure for the complete rotation at $\emptyset = 0^{\circ}$ is used as the intensity of this point.

Plotting of X-ray Data

The X-ray intensities are plotted on either polar or Schmidt equal-area nets at positions corresponding with the intersections of parallels and meridians, each taken at 10° intervals. The formula for intensity determination is:

 $I = D_2 \times SF_2 \times M_2 \times K - B$

where I is the peak intensity in counts per second; D_2 the peak height measured on the strip chart; SF_2 the scale factor; M_2 the multiplier; and B the background intensity. As explained before, K is ignored. Since sample cores are assumed to be cut normal to the bedding planes, it is necessary to translate the points on the net into a standard reference hemisphere to allow comparison.

The generalized method of standardizing an equal-area net is to rotate first about the strike to correct for the dip angle, then to rotate about the vertical axis into standard compass directions. Polar equal-area projection (Figure 2) has the advantage that the second operation is accomplished automatically by the numeration of meridians. The lower hemisphere (Figure 3) was chosen as a standard for this work, and a polar net was employed.

The point recorded is the intersection with the imaginary lower hemisphere of the line which is normal to the reflecting plane at the focal point. The meanings of α - and \emptyset -values are illustrated in Figure 4. The concentric circles of the net represent \emptyset -values, increasing from $\emptyset = 0^{\circ}$ at the center to $\emptyset = 90^{\circ}$ on the periphery. α -values are measured around the periphery in a clockwise direction. (The strike is recorded at $\alpha = 270^{\circ}$, the direction 90° counterclockwise from the strike at $\alpha = 180^{\circ}$, etc.)

Points are initially plotted on a polar net (drawn on tracing paper) whose meridians have been numbered to place $\alpha = 270^{\circ}$ in the position which represents the strike direction on the standard hemisphere. The polar net is then superimposed on a Schmidt net of the same size, so that $\alpha = 90^{\circ}$ and $\alpha = 270^{\circ}$ of the polar net coincide with the poles of the Schmidt net. The points are then translated along the parallels of the Schmidt net by the number of degrees of dip and in the direction opposite the dip. Figure 5 shows the translation for a bed oriented at N 50° E, 17° N. The standardized net is labeled so that intensities can be recorded on it directly.

Bibliography

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EXPLANATION OF PLATE I

Figure 1. Drill press with sample cores

- Figure 2. Hemisphere grinder
- Figure 3. Unshielded diffractometer showing sample holder in $\emptyset = 0^{\circ}$ position
- Figure 4. Shielded diffractometer



Textural Studies

Carbonate rock textures are a subject of some complexity and interest. Acting upon the assumption that carbonate rock textures are static features that represent the environment of deposition, petrologists have drawn large numbers of conclusions which, on the account of internal inconsistencies, must be considered at least partially in error. In order that the geologist in the field might profit by observations restricted to the laboratory, a study of carbonate rock textures was undertaken.

Texture is defined as the geometrical aspects of the component particle of a rock, including size, shape, and arrangement. The fundamental component particles of a rock are the crystals therein. In the case of uniform size gradation it is easy to describe the texture on the basis of crystal size, shape, and arrangement.

Many carbonate rocks, however, contain crystals of two or more distinct size ranges. Furthermore, crystals in at least one of the size ranges commonly have a characteristic shape and arrangement. Moreover, these latter crystals are commonly below or near the limit of microscopic resolution and are readily observed by the unaided eye or low power magnification only as aggregates in which the individual crystals are not resolved. In addition to the small crystals occurring as aggregates, carbonate rocks commonly contain larger crystals that may also have a characteristic shape and arrangement but, because of the larger size, such shape and arrangement is less common and not so easily seen. In any event, such crystals are much more easily resolved as individuals and are thus treated as single crystals.

Broadly then, the textural elements of carbonate rocks may be divided into two categories: 1) monocrystalline elements - all those crystals that behave as individual crystals, easily resolved by microscopy and lacking, in general, any characteristic arrangement with respect to other monocrystalline elements; 2) polycrystalline elements - the aggregate composed of crystals below the limit of easy microscopic resolution. The micro-crystals composing the polycrystalline element may or may not have a characteristic shape and arrangement within the element.

The monocrystalline elements may be of three or four minerals that are abundant in carbonate rocks, but they are simple in that they are internally homogeneous. The shapes of such monocrystalline elements are variable between the limits of sharp euhedra and completely irregular anhedra. Such elements are commonly too small to be seen by the unaided eye, though they have been observed in masses measured in centimeters. In most instances the arrangement of monocrystalline elements in the rock is an interlocking mosaic of apparently randomly oriented anhedra.

Polycrystalline elements range in size from units a few microns in diameter to masses measurable in centimeters. They occur as fossils, or fragments thereof, as ovoid bodies of concretionary habit, or clots, pellets, or irregular masses. The polycrystalline elements are most commonly composed of calcite, though quartz bodies have been observed; goethite bodies are not uncommon and dolomite bodies are rare. Such elements may or may not be internally structured, but they are distinguished from the monocrystalline elements in that they are composed of large numbers of exceedingly small crystals that behave as a unit. The foregoing will serve to indicate the complexity of the problem. It will be readily appreciated that if both monocrystalline and polycrystalline elements occur in the same rock, certainly the size distribution becomes at least bimodal; probably the shape distribution becomes bimodal, and the number of possible arrangements becomes large. In extreme cases, it may be necessary to discuss separately the textures of monocrystalline and polycrystalline elements and treat them additively to describe the rock texture.

Because the presence of polycrystalline elements is easily seen, but not analyzed during field examination in most rocks of mixed texture, the textural scheme that has been devised is as follows:

- I. Homogeneous Textures
 - A. Coarsely crystalline
 - B. Finely crystalline
 - C. Microcrystalline
- II. Polymodal textures
 - A. Recrystallization breccia
 - 1. Polycrystalline elements, irregular and very angular. Internal structure lacking.
 - 2. Monocrystalline elements separate polycrystalline elements.
 - B. Ovoid
 - 1. Polycrystalline elements are rounded and may be radially and concentrically structured.
 - 2. Separation of polycrystalline elements by aggregations of monocrystalline elements more or less perfect.
 - C. Poikilitic
 - 1. Polycrystalline elements lacking
 - 2. Monocrystalline elements contained within monocrystalline elements as dolomite rhombs included within a calcite crystal.

Few of the rocks examined fall into class I. Some specimens referred to as lithographic limestones are of uniform texture in that the elements are monocrystalline, equant anhedra near or below the resolution limit of the microscope. Such rocks, however, commonly have some larger elements present and when these occur, the texture immediately passes over into the polymodal class II. No homogeneous coarsely crystalline or finely crystalline textures have been observed in this study.

Recrystallization Breccia

Recrystallization breccia is the textural type designated for those rocks in which the polycrystalline elements are composed of equant, apparently diversely oriented, calcite crystals of very small diameter, usually less than five microns. Monocrystalline elements surround and enclose polycrystalline elements of a wide range of particle sizes. Such textures are shown in the photomicrographs of Plate II. Because of the tendency to ascribe to the discontinuous element, the role of the brecciated fragment, set in a matrix of some homogeneity making up the rest of the rock, such textures have been misinterpreted. It is easily understood that if the amount of polycrystalline elements relative to that of monocrystalline elements varies over a wide range. The role of the brecciated fragment might and probably would be assigned to the monocrystalline elements where few were present, and to the polycrystalline elements. The role of the matrix would of course vary inversely in such an interpretation.

Most, if not all, the monocrystalline elements display some relict texture suggesting that the volume they now occupy was formerly filled by other and smaller crystals much like those composing the polycrystalline elements. It is considered then that the monocrystalline elements are secondary; thus, the texture is largely controlled by diagenetic processes.

Ovoid Rocks

As might be expected from the foregoing paragraph, recrystallization breccias must grade imperceptibly into rocks characterized by ovoid texture. It has been shown in a former report that the ovoid bodies in ovoid rocks are recrystallization products or sub-spherical regions wherein recrystallization has not yet been completed. Because ovoid bodies and their genesis has been previously treated in some detail, the reader is here referred only to Plate III.

Poikilitic Texture

Poikilitic rocks are those wherein crystals of one mineral contain those of another. Several examples are illustrated in Plate IV. By far the most abundant mineral pairs represented by this class are calcite enclosing dolomite, and calcite enclosing calcite. In all occurrences of the latter pair, the enclosed calcite is a microcrystalline mosaic or scattered individual microcrystals of a brown color indicating some impurities either within the crystal structures or coatings at their edges.

Because dolomite and coarsely crystalline calcite are not primary in the rock, the poikilitic texture is considered of diagenetic origin. The microcrystalline calcite is possibly representative of the original texture, though it may be secondary. If the microcrystalline calcite is indeed secondary, the texture that preceeded it has been completely destroyed.

Fossil Fragments

Fossil fragments perhaps deserve special mention because they are abundant in many carbonate rocks and are sometimes difficult to recognize in thin section. Shells or fragments of shells behave as monocrystalline elements when they are composed of single crystals, as are echinoderm fragments. Shells of most organisms are polycrystalline aggregates having mechanical properties distinct from the rest of the rock and thus behave as polycrystalline elements. They are observed as having been replaced by monocrystalline elements during diagenesis, or as having grown during diagenesis and replaced other parts of the rock.

Several photomicrographs showing the role of fossil fragments are shown in Plate V.

Textural Evolution of Carbonate Rocks

A consideration of chemical equilibria leads to the conclusion that crystals precipitated from a solution, whether by biologic or inorganic agencies, should be of uniform size, composition, and regularity of shape. The exact measure of these parameters is a function of several variables; chiefly temperature, pressure, and composition of the solution and crystalline phases and, in the case of biologic precipitation, the metabolism of the orgnism causing the crystallization. Though some organisms precipitate large crystals of carbonate, most biologically precipitated crystals are small, relatively impure, anhedral crystals of irregular outline. The textures observed in the carbonate rocks are not uniform with respect to either size, purity, or shape of the crystals. The large crystals that are the monocrystalline textural elements, except large biologic crystals as echinoderm parts, contain relicts of smaller crystals (Plate V, Figure 1).

A probable textural evolution then would involve several steps. Beginning with a microcrystalline mosaic of finely divided carbonate crystals recrystallization beginning in a number of isolated localities in the rock would give rise to spots or "eyes" of large carbonate crystals in a finetextured groundmass (Plate VI, Figure 2). Many such large crystals might completely surround fragments of the apparently unaltered microcrystalline mosaic, giving rise to polycrystalline textural elements. The size of the newly engendered monocrystalline elements seems controlled by the frequency with which they encounter other crystals of the same type. This is, of course, directly controlled by the nucleation density of the new phase.

The monocrystalline elements increase both in size and number of individuals at the expense of the polycrystalline elements. Such a sequence of events might then give rise to a rock like that illustrated in Plate VI, Figure 3 -and- Plate II, Figure 1; or, in the event of recrystallization and internal ordering of the polycrystalline elements resulting in an ovoid rock like that shown in Plate III, Figure 1. Further recrystallization might result in the obliteration of the former textures in steps, as shown in Plate VI, Figures 4 and 5, with the eventual production of a more or less uniform texture composed of monocrystalline elements only as shown in Plate VI, Figure 6. Poikilitic textures may occur at any stage in this evolutionary sequence. The sequence would seem reversable at any stage because the texture is clearly dependent on the stability of crystals of given sizes and purity at given temperatures and pressures.

EXPLANATION OF PLATE II

- Figure 1. Recrystallization breccia, Maynes Creek formation, Ferguson, Iowa. 100X PAL.
- Figure 2. Partial development of breccia fragment Gilmore City formation, Humboldt, Iowa. 160X PPL.
- Figure 3. Recrystallization breccia showing relict texture in monocrystalline elements and reorganization of material in polycrystalline elements. Gilmore City formation Humboldt, Iowa. 160X PAL.
- Figure 4. Recrystallization breccia. Iowa Falls formation, Iowa Falls, Iowa. 36X PPL
- Figure 5. Development of recrystallization breccia by inclusion of polycrystalline elements in later formed monocrystalline elements Gilmore City formation LeGrand, Iowa. 40X PPL
- Figure 6. Well developed recyrstallization breccia Iowa Falls formation, Iowa Falls, Iowa. 36X PPL



EXPLANATION OF PLATE III

- Figure 1. Ovoid bodies included in large monocrystalline element displaying large patch of relict texture at center. Gilmore City formation, LeGrand, Iowa. 25X nicols partly crossed.
- Figure 2. Vaguely defined ovoid bodies with few monocrystalline elements between ovoid bodies. 160X nicols partly crossed.
- Figure 3. Ovoid bodies separated by and containing large monocrystalline elements. 25% nicols partly crossed.
- Figure 4. Vaguely defined ovoid body in microcrystalline uniform textured limestone. 100X PPL
- Figure 5. Ovoid bodies in "Pisolitic limestone" St. Louis formation, Mt. Pleasant, Iowa. 36X PAL
- Figure 6. Development of ovoid bodies Gilmore City formation, Humboldt, Iowa. 25X P AL











EXPLANATION OF PLATE IV

- Figure 1. Poikilitic intergrowth of calcite and dolomite. Bed 29 LeGrand quarry, LeGrand, Iowa. 100X PAL
- Figure 2. Subhedral dolomite crystal contained in single calcite crystal, location as Figure 1. 400X PAL
- Figure 3. Poikilitic intergrowth of calcite with dolomite Maynes Creek formation, LeGrand, Iowa. 160X nicols partly crossed.
- Figure 4. Poikilitic intergrowth inside a fossil fragment Gilmore City formation, LeGrand, Iowa. 40X nicols partly crossed.
- Figure 5. Intergrowth at vein wall Gilmore City formation, Humboldt, Iowa. 160X PPL
- Figure 6. Poikilitic intergrowth of large calcite crystals containing smaller calcite crystals. Eagle City formation, LeGrand, Iowa. 160X nicols partly crossed.



EXPLANATION OF PLATE V

- Figure 1. Echinoid fragments in matrix of large calcite crystals "cross bedded" Gilmore City formation, LeGrand, Iowa. 25X PPL
- Figure 2. Fossil fragments showing complete replacement of parts of fossils by monocrystalline elements. 25X nicols partly crossed.
- Figure 3. Recrystallization of fossil wall. Large crystals (white) of calcite occurring in the wall of a gastropod shell. Note development of crude ovoid bodies. Gilmore City formation, Humboldt, Iowa. 160X PPL
- Figure 4. Recrystallization within the walls of a coral. Gilmore City formation, Humboldt, Iowa. 25X PPL
- Figure 5. Central part of Figure 4. 100X PPL
- Figure 6. Echinoid fragments surrounded by polycrystalline elements including ovoid body containing dolomite rhombs. Eagle City formation, LeGrand, Iowa. 40X PPL



EXPLANATION OF PLATE VI

- Figure 1. Relict texture in large crystal Gilmore City formation. LeGrand, Iowa. 100X nicols partly crossed.
- Figure 2. Large calcite crystals in "Pisolitic bed" St. Louis formation Mt. Pleasant, Iowa. 36X PAL
- Figure 3. Polycrystalline elements in various stages of replacement by large (light) monocrystalline elements. Gilmore City formation Humboldt, Iowa. 25X nicols partly crossed.
- Figure 4. Relict ovoid bodies Eagle City formation LeGrand, Iowa. 25X nicols partly crossed.
- Figure 5. Ovoid relicts in uniform dolomite Maynes Creek formation LeGrand, Iowa. 40X PPL
- Figure 6. Uniform finely crystalline dolomite mosaic. Maynes Creek formation LeGrand, Iowa. 160X PAL







Stratigraphic Studies

The mineralogy and stratigraphic relationships of three quarry sections are indicated in the geologic sections attached. The mineralogy and stratigraphic section of the Eagle City formation and Iowa Falls formation at Iowa Falls, Iowa, have been transmitted in the thesis titled PETROLOGY OF THE HAMPTON FORMATION AT IOWA FALLS, IOWA, by James R. Mason.

Future Studies

Beyond the compilation of data that was the objective of HRP-70, the analysis of the data here collected and its integration with other studies will greatly benefit the investigators in their understanding of the problems of carbonate rock genesis and diagenesis. Technical papers stemming from this source will be deposited with the geology branch of the Iowa State Highway Commission at such times as become appropriate in the future.

Schroeder quarry, Marquette, Iowa Basil Giard Claim No. 1--Clayton County.



Scale 1" to 10'

.ogged: Thomas, L.A. 6/9/61

Grace Hill Quarry NE_4^1 , SW_4^1 Sec. 31, T. 75 N, R. 8 W.



* Bed numbers Ia. Hwy. Comm.

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DOUDS STONE CO. MINE NWL SEL Sec. 25, T. 70 N. R. 11W.

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