MAKING THIN SECTIONS OF ROCKS;

I. REGULAR METHODS.

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INTRODUCTION.

In several text books of petrography[2] and of petrographic methods[3] the making of thin sections of rocks is described, the descriptions by Johannsen and by Holmes being especially detailed and based on practical experience. There may be in this, as in any other art, considerable difference in the technique of different workers. Miss Keyes, my laboratory assistant, has had five years' experience in the Petrographic Laboratory of the U. S. Geological Survey in the making of thin sections, in the course of which work many kinds of material have been treated. Her experience there, as well as in the Geophysical Laboratory, has led to some methods in manipulation that differ from the procedures published by Johannsen and by Holmes. It is thought that a description of her methods, which are essentially those in use at the U. S. Geological Survey, may be of practical interest to petrographers.

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SECTIONS OF FIRM ROCKS.

The maker of thin sections has most often to deal with firm, non-friable rocks, so that their treatment will be described first and in greatest detail: in the second part briefer remarks will be made on friable material, to the treatment of which the general procedures apply, modified in details because of their peculiar textures. In Fig. 1 are shown diagrammatic views of a thin section in the process of making: these will be referred to as Fig. 1 (a), (b), etc.

Preparing the chip.[4] A rock chip before grinding should be about one inch in diameter and from one-eighth to one-half

[1] Published with permission of the Director, U. S. Geological Survey.

[2] Rosenbusch-Wülfing, Mikroskopische Physiographie, 5th ed., I, 1, 11-24, 1921. Iddings, Rock Minerals, 1911, 190-194.

[3] Johannsen, Manual of Petrographic Methods, 1914, 572-604; Holmes, Petrographic Methods and Calculations, 1921, 231-249.

These authors give the history of thin sections and many references.

[4] In this paper "chip" denotes the fragment or slice of rock from the time when it is first roughly prepared until it has been mounted on the slide and has undergone the second rough grinding: "section" denotes the thin lamina of rock from this point until the section is finished. "Chip" corresponds to *a* to *c* and "section" to *d* to *f* of Fig. 1. "Slide" denotes the thin glass plate on which the chip is mounted.

an inch thick, with the flat, rough faces approximately parallel (Fig. 1, a). It is best to trim the chip into a roughly circular disk. The use of thin "flakes" of rock for section-making is not advisable. There is no objection to using a sawn slice if the material demands it, but for ordinary compact materials time and labor are saved by using an unsliced chip with rough faces.

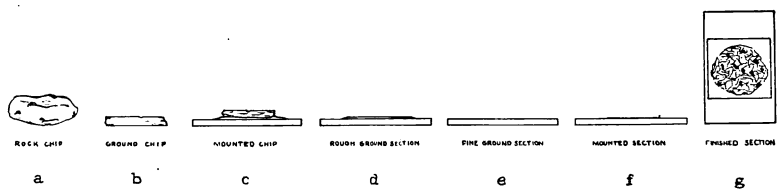

Fig. 1.—Diagrammatic views of the successive stages in the making of a thin section.

First rough grinding. It is a common practice to cement the chip, one face of which has been smoothed, to a small plate of thick glass, before grinding, the thin chip or section being transferred later to the final slide. But I grind the chip very smooth on both sides and to an appropriate thinness, before it is mounted on the slide for the second grinding. This has the advantage of enabling one to mount the slide onto the chip, so that it can be readily seen when the two are properly attached, and that no air bubbles are present. By this method the necessity of transferring the thin section is avoided, a procedure which is difficult, and hazardous to the section.

It is assumed that a revolving metal lap, 9 to 12 inches in diameter, driven by a motor, is used, as is done at the U. S. Geological Survey and at the Geophysical Laboratory; this greatly facilitates and shortens the procedures. If the grinding must be done by hand, the various processes to be described must be modified to meet the different condition.

The first rough grinding is done with No. F carborundum on a "rough lap," which must never be used for fine grinding. The abrasive, mixed with water to the consistency of a thin paste in a shallow container, is applied with the finger tip, and spread, not too thickly, over the revolving lap. A little more is added later from time to time. It is not advisable to apply dry abrasive to a wet lap.

The manner of holding the chip is a matter of personal preference, but the hand should never be cramped. I press it

down with the tips of the first three fingers. The motion is in general "circular."

With experience the chip may be ground so that the two faces are uniformly flat and parallel, to a thickness of 2 to 3 millimeters (Fig. 1, b), the thickness depending on the character of the material. Time is thus saved because of the coarseness of the abrasive, and less grinding is required after the chip is mounted, when the grinding is more difficult and there is danger of destruction of the section. Also, the thin, uniformly ground chip may be heated more evenly than a rough one in the process of mounting.

Next, the chip is thoroughly scrubbed in water, with a stiff hand brush. The rough edges particularly should be treated, because the coarse powder is more liable to adhere to them. It is of the utmost importance that all abrasive be removed before the fine grinding, for a single coarse grain in the finer powder will cause scratches and may tear the section to pieces. The hands also, therefore, must be thoroughly scrubbed before the fine grinding.

First fine grinding. A lap, separately mounted, is desirable for the fine grinding. If only a single shaft is available, great care must be taken that none of the coarse abrasive from the rough grinding lap is transferred to that used for the fine grinding.

This operation is to produce a smooth flat chip preparatory to mounting it on the slide, consequently only one face need be ground, for which carborundum powder, "15 minute" or No. 400, should be used. It is well to mark the rougher face by holding it for a few seconds against the edge of the revolving lap, thus making a shallow groove. If the rough face is unmarked the chip may inadvertently be mounted with the rough, instead of the smooth, face against the slide, an error impossible to correct later. If the ground chip has sharp, irregular corners it is well to trim them off with end-cutting pliers, as they may lead to the tearing away of the section later.

Mounting the chip. An appropriate size for the glass slide is that now adopted in the U. S. Geological Survey, namely 25 by 45 millimeters, the thickness being one millimeter. The slide and the finely ground chip, with the smooth (unmarked) face up, are placed on the mounting table,[5] which is heated by

[5] Convenient dimensions for the mounting table are: length 14 inches, width 5½ inches, thickness ¼ inch, height 8 inches, height of Bunsen burner 5½ inches. The plate is best of brass.

a Bunsen burner to a temperature of 80° to 100°C. The slide should previously have been numbered on the reverse side with a diamond point, to correspond to that of the rock specimen. It should be marked before the chip is mounted, because pressure may cause the chip to "spring" from the glass (shown by the appearance of "Newton's colors").

When the slide and the chip are thoroughly hot, a little previously boiled balsam is applied with the "balsam stick" to the upper smooth face of the chip and to the center of the glass slide. The balsam should form a thin film on both chip and slide. Experience will soon teach one about the correct amount to apply.

I have tried the method recommended by Holmes,[6] of spreading *raw* balsam over the whole surface of the slide and the chip and then cooking it on the mounting table, but do not approve of it. It is my experience that, while it is not advisable to stint oneself in the use of balsam for mounting, it is important to avoid the use of an over-supply. If it is spread over the whole slide "from end to end"[7] it is very difficult to squeeze out the excess from between the chip and the slide. Furthermore, a thick layer of balsam beneath the chip may, in the final stages of grinding, so mislead one as to the actual thickness of the section, that it may be destroyed. By applying a moderate amount of balsam, as described above, the balsam that is then pressed out forms a ridge or frame (Fig. 1, c) around the edges of the chip, which serves as sufficient protection when it is being ground. If several sections are being made at the same time, a sufficient amount of balsam should be "cooked" beforehand.

When the balsam placed on the chip and on the slide has become well melted, the slide is picked up from the table with the fingers and placed, balsamed face down, on the smooth, balsamed face of the chip, with the faces parallel, using but slight pressure. The slide with the chip attached is then removed from the table and placed, with the chip underneath, on the work bench, which may advantageously be covered by a sheet of plate glass in front of the mounting table. Using the butt end of the balsam stick, the slide is pressed firmly down onto the chip, the pressure being continued with a quick tapping motion until the excess balsam has been squeezed out and there are no bubbles. With the transparent glass slide

[6] Holmes, A., Petrographic Methods and Calculations, 239, 1921.
[7] Cf. Holmes, p. 239.

uppermost it is easy to see when all bubbles are eliminated. If bubbles still persist, the whole process must be repeated. It is not necessary to apply much pressure to the slide after the excess balsam has been squeezed out, nor to continue the pressure until the slide and chip are cold.

Second rough grinding. The cooled, mounted, chip is reground on the revolving rough lap with No. F carborundum. Practice alone will enable one to judge of the thinness to which a chip may safely be ground on the rough lap, but rough grinding should not be continued after the rim of balsam around the edge of the chip has begun to wear smooth, Fig. 1, d. A single application of coarse abrasive powder to the section after this protecting rim has been broken will almost always result in the loss of the section.

Second fine grinding. After the second rough grinding the very thin lamina of rock is called a section. The slide and attached section are to be washed thoroughly, and the section is again ground on the rotating fine lap, "15 minute" carborundum being used. (Fig. 1, e.)

It is at this stage that the greatest care must be taken. The slide must be turned frequently so that all parts of the section face may be equally acted on by the lap. The section must be examined under the microscope frequently for correct thinness, because a slight touch after the final thinness has been reached may grind the section entirely off the slide.

For this grinding the abrasive powder should be mixed with water and applied as a thin paste. It may be applied frequently during the earlier stages of the grinding, but it is not advisable to apply fresh and, therefore, sharper abrasive when the section is nearing its final thinness, else it may scratch or destroy the section.

With careful manipulation and the steady application of uniform pressure very thin sections should be obtained. For most purposes the section should be from 0.03 to 0.04 mm. thick. The interference colors of quartz and the feldspars may usually be taken as indicating the proper thickness: these should be light gray of the first order. Sections of cryptocrystalline or very fine-grained rocks should be even thinner, as thin as it is possible to make them so as to bring out the minute textures: on the other hand, sections of fossils and fossil woods must be much thicker in order to show their micro-structure.

Final hand grinding. The section should again be thor-

oughly washed preparatory to the final grinding. This is done by hand on a square of finely ground plate glass. At the U. S. Geological Survey the glass plate is specially prepared by grinding it well over the whole surface on the revolving lap with fine carborundum. The ground glass plates usually found in commerce are too coarse for the best work. The glass plate should always be kept covered when not in use.

A very fine abrasive must be used for this final grinding. "Washed" flour emery or "60 minute" carborundum may be used, but the abrasive, of whatever kind, must pass through 200-mesh silk bolting cloth. Most satisfactory results have recently been obtained with "Sira abrasive," a very fine alumina powder, each grain of which is a separate rounded crystal, not a sharp fragment. This abrasive is made according to the specifications of the British Scientific Research Association, and may be obtained from R. and J. Beck, 68 Cornhill, London, E. C.

Comparatively little abrasive need be used for the final hand grinding, and it should be applied to the plate by the finger tip. The purpose of this final grinding is not to produce a thinner section, but to smooth and bring out clearly the texture of a section already thin. If it is found necessary, because of the texture of the rock, to discontinue the second fine grinding on the rotating lap before the section is thin enough (that is, while the quartz or feldspar still shows bright colors between crossed nicols), the slower hand grinding on the plate may be continued until the proper thinness has been obtained.

Drops of clear water, applied from time to time with the finger tip, sufficiently lubricate the grinding plate. The section should be pressed against the plate by placing the finger tips of one or both hands over the center of the slide, using a circular motion. As in the case of the second fine grinding, the section must be examined frequently under the microscope.

The statement often made that sections are to be *polished* should be corrected. Polishing involves rubbing the finely ground surface on cloth or felt-covered laps with such materials as rouge, tin dioxide (putty powder), or chromic oxide, the result being a glossy surface, which tends to obscure the texture. The surface of a thin section should be smooth but dull, so that the slight relief of the different minerals may be clearly seen.

The small remnants of the rim of surplus balsam are removed

with turpentine applied with a camel's hair brush. The slide should then be held upright on a piece of blotting paper to allow the turpentine to drain off. The slide and the section should never be wiped with a cloth, as small fragments of lint and fiber are likely to become attached to the surface.

Mounting the cover glass. The slide, with the section on it uppermost, is placed on the mounting table, which is heated to nearly 100°C. A few drops of refined balsam, filtered through paper, are placed on the center of the section which is slowly heated in order to "cook the balsam, and spread it over the section. During the heating the balsam is to be tested frequently to ascertain when it is of the proper consistency. The most satisfactory method is to remove a minute globule of the hot balsam on the end of the mounting needle and press it, when cool, on the thumb nail. It is of the proper consistency when the globule is so hard that it is barely flattened by the nail, but not so hard that it breaks into pieces.

A cover glass, 22 mm. square, well washed in warm soap suds, rinsed well, and dried with a soft cloth, is picked up by one edge in the forceps and warmed slightly by passing it quickly two or three times through the Bunsen burner flame. It is then placed on one edge in the balsam at one side of the section, the tip of the mounting needle, held in the other hand, being placed close to the other side of the section and resting on the slide. The cover glass is now gently lowered until it is slightly above the needle tip, when the forceps are removed and the cover allowed to drop on to the needle. This is then gently withdrawn and the cover glass is now in place. The slide and section are allowed to stay on the mounting table for a few moments, until the balsam has spread over the whole under surface of the cover glass.

The slide is then removed from the mounting table and, while held in the hand, the cover glass is lightly pressed down on the section with the mounting needle, it being moved slightly to and fro to drive out bubbles and squeeze out the surplus balsam. If the rock is friable and cracks are seen or appear in the section, or if the rock has been previously heated in balsam to harden it, no pressure should be applied to the cover glass, as a slight touch will cause the section to disintegrate. If not too much balsam has been used the cover glass will be very close to the section when the needle tip is removed and should therefore require little or no pressure. (Fig. 1, f.)

When the section has cooled, the surplus hardened balsam

around the edges of the cover glass is removed with a hot knife blade, care being taken not to spring the cover. The slide is then finally washed in alcohol and dried with a soft cloth. (Fig. 1, g.)

Preparation of balsam for mounting. For the successful preparation of thin sections it is of the utmost importance, indeed essential, that the balsam used for mounting the chip on the slide be of the proper consistency. If the balsam is too soft the section will not adhere properly to the slide and grains of coarse abrasive will probably become embedded, leading to disaster during the second fine grinding. If the balsam is too hard, through overcooking, which is more likely to happen than undercooking, the protective rim of balsam will break away during the second fine grinding, and the unprotected edges of the section will then rapidly be broken off, leading to the disintegration and loss of the section. Even if the section is not lost it will not adhere properly to the slide, as the overcooked balsam tends to spring and buckle.

The raw balsam is heated, best on an electric hot plate, in a shallow pan with a flat bottom,[8] filled nearly to the top. The pan is to be reserved for this purpose. The balsam must not be stirred, as this introduces air bubbles, and must not be allowed to boil. At frequent intervals during the heating a small drop should be removed on the end of the mounting needle and tested against the thumb nail as already described. If the balsam is heated insufficiently the mass will be sticky and soft, whereas if it is heated overmuch it will be very brittle and dark colored. It is of the proper consistency if the cold hardened balsam can be just indented with the end of a finger nail. When the properly cooked balsam is cold, the surface is marked into small squares, and the mass turned out on a clean sheet of paper by tapping the upturned bottom of the pan. It is then broken up and the fragments preserved in a closed box.

For use in mounting, a piece is attached to the "balsam stick" by touching with the heated end. The "balsam stick" is a steel or brass rod, about 8 inches long and one-eighth of an inch in diameter.

The raw, "natural" Canada balsam, used for mounting the chip and for hardening friable materials, may conveniently be purchased in 500 cc. (1 lb.) bottles. The specially refined raw balsam, used for mounting the cover glass, is most con-

[8] The pan used at the U. S. Geological Survey is about 6 inches in diameter and 1 inch deep.

veniently contained in flexible metal tubes, of about 30 cc. capacity.

II. SPECIAL METHODS.

Transferring the section. Many ordinary slides, not made of plate glass, present slight irregularities of surface that may affect the uniformity in thickness of sections made on them, and thus seriously interfere with certain optical studies of minerals in thin section under high powers. For such purposes the chip should be mounted on a thin slide of polished plate glass, which may be specially obtained from various dealers in optical supplies.

If such thin plate glass slides are not procurable, the chip, after the first fine grinding, is to be mounted on a small (about an inch) square of ordinary plate glass, and about 3 to 4 mm. thick, ground to the requisite thinness, and then transferred, an optically true section, to an ordinary glass slide. The method of transfer is as follows:

The square of plate glass on which the section has been mounted and ground, and the glass slide to which it is to be transferred, are placed on the warm mounting table and a drop or two of filtered balsam is squeezed out from the tube onto the center of the empty slide. When the balsam on the slide has been heated to the proper consistency, tested as before by cooling a globule on the end of the mounting needle and tapping it against the thumb nail, and when the square of plate glass has been sufficiently heated to melt the cementing film of balsam and free the section, the square of plate glass with the section attached is lifted from the mounting table with the forceps, and held slanting above and close to the balsamed area of the slide. The tip of the mounting needle is applied gently to the upper edge of the section, slowly and gently moving it to the lower edge of the glass square, from which it is pushed carefully onto the balsam on the slide, into which it sinks, leaving an upper thin coating of balsam sufficient for the attachment of the cover glass. If, in this process, the balsam becomes stiff through cooling, the slide, held in the forceps, is passed quickly several times through the flame, care being taken that the balsam does not catch fire.

The cover glass is mounted on the transferred section in the manner previously described. When the lower edge is placed in the balsam it is well to drag up some of it onto the lower

face of the cover glass by a pulling movement, so that it may more readily spread over the slide, with less danger of the formation of bubbles.

It is best not to transfer material that is friable or that has been heated with balsam before being ground, as is described later, because the slight reheating melts the cementing balsam and often causes the section to disintegrate. If, however, the transfer of such material, or of a section in which cracks have developed, is necessary, it may best be accomplished as follows. The cover glass is mounted on the section, while still attached to the square of plate glass. The balsam on the slide to which the section is to be transferred having been heated almost to the proper consistency, the square of plate glass is replaced on the mounting table. A few drops of carbon tetrachloride[9] should be applied to the cover glass to keep it cool and the balsam immediately beneath it from melting, while the balsam below the section is being slowly heated. When the section is free from the slide, the cover glass with the section attached to it is lifted with the forceps and placed in the balsam on the slide. It is best to use no pressure, or as little as possible, on the cover glass to get the section into place.

If the rock is not firm and compact, or if it contains minerals that are soluble in water or altered by heating, a method adapted to the special case must be employed. Two of the more important of such cases will be treated here very briefly.

Fragile rocks. If the rock is soft and friable, such as shale, tuff, and some sandstone and limestone, it is necessary to harden, or rather consolidate, the chip before grinding. This is best done by heating Canada balsam on an electric heater, if possible, in a shallow, flat-bottomed vessel, to such a consistency that it is brittle when cold. The rough rock chip, which should be thick enough to hold together, is also thoroughly heated and, held in the forceps, is immersed in the molten balsam for several seconds and removed. Two or three such immersions will be sufficient to carry the balsam into the pores of most rocks. If necessary the chip may be immersed in the balsam and heated until all air bubbles have disappeared.

[9] Benzene may be used, but care must be taken to prevent the ignition of the benzene in the bottle.

It sometimes happens that seemingly firm and coherent rocks, which may be rough ground with ease, break into fragments during the first fine grinding. A fresh, rough ground chip of such material should either be hardened as has been described above, or thoroughly heated, and the face that is to be fine ground rubbed with a piece of hardened balsam such as is used for mounting the chip. The piece of balsam is best held in the fingers, and the gentle rubbing continued until the balsam melts and penetrates the flat chip, which takes only a few seconds. Either of these processes fills the cracks and crevices with balsam, so that fine grinding and mounting may be done with little risk of losing the section.

Rocks altered by heat. In making sections of rocks that contain such minerals as gypsum, sulphur, and zeolites, the temperature at all stages of the operation must be kept as low as possible.

Some of them may be hardened sufficiently by soaking in a solution of balsam in ether, contained in a wide-mouthed bottle, with a close-fitting glass stopper. The balsam should be hardened, as described, broken into small fragments, and dissolved in the ether. The rock chip is allowed to remain in the solution until it is thoroughly saturated. This may take three or four days, the length of time being dependent on the texture and hardness of the material. The ether evaporates very quickly after the chip is removed from the solution, leaving the pores filled with hardened balsam, and the chip may be ground as described above.

In the process of mounting such material on the slide, preparatory to the second rough and fine grindings, the rock chip should be on the part of the mounting table that is not so warm, and balsam is applied to the slide placed at the center of the table, where it is hottest. The slide is placed on the chip after the balsam has melted and cooled somewhat, but before it has begun to harden. If mounted in this way, the chip is subjected to very little heating.

In mounting the cover glass, the slide, with the chip attached, is again placed on the far end of the mounting table, the flame lowered, and a drop or two of balsam applied to the chip. The slide is then very slowly and carefully heated at the low temperature, until the balsam is of the proper consistency, which may take ten or fifteen minutes, when the cover glass is mounted in the usual way.

If very volatile or hydrated substances are present, which

would alter even under the low temperatures of this procedure, the cover should be mounted by applying to the section a drop or two of a solution of balsam in xylol at the end of a glass rod. This solution is best obtained already prepared from a dealer in optical supplies. A section mounted in this way should remain for several days on a flat surface at room temperature, to allow the balsam to dry as much as possible before the section is handled. It takes a long time for cover glasses mounted with the solution of balsam in xylol to become firmly cemented: even after several years the balsam has been found to be so soft that a slight pressure will cause the cover to move.

Rocks containing water-soluble minerals. In making thin sections of rocks that contain minerals which are soluble in water the only change necessary in the methods described is the use of thin oils in the grinding processes. Almond, olive, corn, or neatsfoot oil may be used. For grinding on the revolving lap it is best to thin the oil with benzene, but on a stationary plate they may be used undiluted. Glycerine, which is recommended by Holmes,[10] I have found to be too thick for use with the revolving lap, although it may be used with the stationary plate. Benzene, kerosene, or turpentine are to be used for the washings, which must be done rapidly, as balsam is soluble in these media.

Use of bakelite. Ross[11] has suggested the use of bakelite, the trade name for a phenol-formaldehyde condensation product, in hardening friable rocks, and he has obtained good results with clays, bentonite, and tuffs. His method consists, in brief, in impregnating the rock with bakelite varnish and changing this to bakelite by prolonged heating, usually at temperatures of 70° to 90°. In the course of an investigation of methods for making thin sections of ancient Roman mortar and cement it has been found that bakelite can be used successfully for the hardening of friable materials that contain minerals altered by heat and minerals soluble in water. In the former case the bakelite varnish is hardened at temperatures not above 45° or 50°C., although the time needed for the operation is lengthened considerably, to a week or more. The results of this study will appear later.

[10] Holmes, A., Petrographic Methods and Calculations, 1921, p. 246.
[11] Ross, C. S., A method of preparing thin sections of friable rock, this Jour., 7, 483, 1924.

ABRASIVES.

There are many abrasives on the market suitable for the making of thin sections of rocks, and the choice is largely to be determined by one's personal preference. At the U. S. Geological Survey carborundum has been employed almost exclusively, but for the final grinding on a plate I have found that "Sira," a recently introduced alumina abrasive manufactured in England, is more satisfactory. It is important that the size of grain of the abrasive used for the several stages be fairly uniform, a condition not always met in the abrasives on the market. For this reason the "finest" powder, used for the final hand grinding, should again be sifted prior to use. A list is appended of the grades of several kinds of abrasives which correspond approximately to the size of grain appropriate to the various processes.

A. *Coarse,* for rough grinding, should pass through a 120 to 150 mesh brass sieve: No. F carborundum, No. F crystolon, No. 1F lionite, 120 mesh alundum.

B. *Fine,* for fine grinding on lap, should pass through 124 mesh silk bolting cloth: 15 minute carborundum (now called No. 400), 302 emery, 120 crystolon.

C. *Finest,* for final hand grinding, should pass through 200 mesh silk bolting cloth: 60 minute carborundum (now called No. 600), No. 65F crystolon, Sira abrasive.

In conclusion I would express my appreciation of the instruction and advice given me by Mr. F. S. Reed and by Mr. J. L. Mergner while in the Petrographic Laboratory of the U. S. Geological Survey, and to Dr. H. S. Washington for his assistance in the preparation of this paper.

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