direct from a scale attached. The details of the instrument as supplied commercially have been worked out with the greatest care and ingenuity, but space does not permit of a description of these, as this could be given only with a series of diagrams. Full particulars can, however, be obtained from Messrs. J. Wild & Co., Middlesbrough. One great advantage of the pyrometer is that it has an automatic recorder, seven hours of which are always in view, so that all variations for this period can be seen at a glance. It is especially adapted for recording the temperature of hot gases, or of large heated chambers, such as annealing furnaces, and a large number are in use both in Europe and in America.

CHAPTER XIX.

MICROSCOPICAL EXAMINATION OF STEEL.

THE study of the microstructure of steel originated with Dr. Sorby of Sheffield many years ago, but for a considerable time after the publication of his first paper little attention was given to this method of research in this country, although substantial progress was made in Germany by Professor Martens. It is only during the last twenty years or so that British metallurgists have given the subject serious consideration, but during that time it has been extensively studied and progress has been very rapid, so that now the microscopic examination of iron and steel alloys is not only largely carried out in all laboratories devoted to scientific research, but has become an essential part of the work of many of the chemical laboratories connected with our leading steel works.

Microscopic examination not only aids in the determination of the soundness of a steel and the detection of mechanical defects, but also throws much light on its internal constitution and the heat treatment to which it has been subjected, and it is in the latter relation that the greatest assistance may be anticipated from the use of the microscope.

The term "microscopic metallography" has been assigned to the method of microscopical examination of metals in general.

Preparation of Samples.—In order to examine the structure of metals microscopically it is necessary first of all to polish a small section until it is as free as possible from even the most minute scratches.

The details of the methods employed vary with different investigators, but in general principles they are alike, and consist of first filing the sample as dead true as possible, then grinding on an emery wheel, and polishing, either by hand or on revolving wheels covered with emery paper of different degrees of fineness, and finally buffing up on a cloth, kidskin, or similar disc with some fine polishing powder. The details of the methods used by Sorby, Osmond, Roberts-Austen, Arnold, Martens, Stead, Ewing and Rosenhain, Le Chatelier, and Sauveur respectively are given below, and it is most desirable that the student should try the different methods, and adopt the one which he finds gives the best results for the particular material with which he has to deal.

Sorby's Method.—Dr. Sorby used small slices, about $\frac{1}{10}$ of an inch (2·5 mm.) in thickness, and by filing, and grinding on an emery wheel obtained as true a surface as possible. The section was then fixed on a glass plate with a little Canada balsam and the surface further ground by rubbing backwards and forwards on, first coarse, and afterwards finer grades of emery paper fixed on a glass support. Before polishing, the surface of the sample was ground on fine-grained Water-of-Ayr stone, which removes all the scratches left by the emery. The final polishing was then effected by rubbing the sample with some crocus powder and afterwards with the finest levigated rouge on a wet cloth stretched over a flat piece of wood, which was kept moist by adding a few drops of water occasionally during the time of rubbing.

It sometimes happens that the sample contains some portions softer than others, and this method causes the surface to be worn away unequally. In

cases where it was desirable to have all the constituents of the polished surface on precisely the same level and the specimen not rounded at the edges, Dr. Sorby performed the final polishing with dry rouge on a sheet of parchment stretched over a glass plate; some of Dr. Sorby's most beautiful specimens were polished dry and not etched by acid, &c. By the dry method some of the constituents are left quite bright and metallic, whilst others assume a blue appearance.

Osmond's Method.—Osmond pointed out that it is very important that the sample should be cut cold and not in any way distorted. He followed very closely the original method of Sorby, but used a machine for the final polishing, and did not fix the specimens to a support, but simply held them between his fingers. In the final polishing he used a method of "Polish attack," which consisted in rubbing the sample on a sheet of parchment covered with precipitated Calcium Sulphate moistened with an infusion of liquorice, by which means some constituents of the steel were coloured. The liquorice infusion, however, does not keep very long, and attacks the sample unequally when a day or two old. In the *Metallographist* for January, 1900, Osmond recommended the use of a 2 per cent. solution of Ammonium Nitrate as far preferable to the liquorice solution. When he required a polished unetched surface he finished either on cloth or kidskin with some fine polishing powder.

Roberts-Austen's Method.—The method used by the late Sir Wm. Roberts-Austen is almost identical with Osmond's, each sample, after filing and grinding, being polished on emery paper of different grades fixed on wooden discs which are revolved at very high speeds by a motor. The final polishing may be completed either by the "Polish attack" of Osmond, or by a disc covered with cloth or kidskin, and by using the finest rouge or similar powder.

Arnold's Method.—The following are the details of the method employed by Prof. Arnold as communicated to Mr. Stead*:—

The sections employed are $\frac{1}{2}$ inch in diameter and $\frac{1}{10}$ inch thick. Before leaving the machine shop they are finished with a dead smooth file, and the file marks are removed by rubbing on a piece of the finest emery cloth, lightly stretched on a hard-wood board. The remaining operations are carried out on materials stretched by means of rings on machined circular tapered cast-iron blocks.

On No. 1 block is the finest emery cloth from which the coarser grains have been removed by rubbing, first with a marble slab, and secondly, with a piece of smooth hard steel about 1 inch diameter by $\frac{1}{4}$ inch thick. On No. 2 block is stretched thick black unribbed cloth, charged with oil and the finest emery knife polish. On No. 3 block is a piece of the finest wash leather slightly charged, dry, with the best jeweller's rouge. The average times occupied in each stage are as follows:—On emery cloth five minutes, and on Nos. 1, 2, and 3 blocks about ten minutes each. The plan of rubbing off the coarser grains of emery from the commercial papers Mr. Stead finds is excellent, and does away with the necessity of personally preparing fine emery papers, and the author's experience fully confirms this.

Martens' Method.—In the case of steel, the samples are cut about 2 to 5 mm. thick with a saw or file, but for brittle iron and spiegeleisen it is necessary to chip a piece off with a chisel, and then grind it flat on a grindstone or emery wheel. Martens often polishes a large number of specimens at the same time by cementing them in a circle on a plate of glass. For this purpose he uses a cement made by melting together equal parts of beeswax and resin.

* *Iron and Steel Inst. Journ.*, 1894, vol. i., p. 396.

The plate to which the specimens are attached is then put in a lathe, and the surfaces of the samples are turned until all are of exactly the same level.

In the case of very hard or brittle samples the levelling must be done by grinding.

For the preliminary polishing he uses emery (Corundum), pounded quartz, sand, and oilstone, and for the fine polishing, hard, and soft, polishing rouge. All his grinding and polishing powders are very carefully levigated before use; in fact, he gives this as a *sine qua non* for successful work, and recommends that each operator should prepare his own powders.

For grinding beds he uses plates of glass, cast iron, copper and lead, and for polishing beds plates of glass or pitch, and sometimes soft beds, such as india-rubber, leather, or cloth. The beds are selected according to the hardness of the material to be polished. He states that the pitch beds are best prepared by melting soft pitch, with the addition of a small quantity of resin, and pouring the mixture on to plates of glass. He finds that the time necessary for preparation by this method varies with circumstances and the experience of the operator, at first several days may be necessary to obtain a single polished surface, but, after a little practice, what formerly took weeks to do may be accomplished in a few hours.

Stead's Method.—Stead prefers to have the sections cut about $\frac{3}{8}$ or $\frac{1}{2}$ inch in diameter and about $\frac{1}{10}$ inch thick, and filed as smooth as possible with a dead smooth file. The section is then rubbed on emery paper of the grade known as No. 0 English, and then successively on Nos. 0, 00, and 000 French paper until all but the very finest scratches are removed. Polishing is then finished on wet cloth with diamantine powder, and finally on chamois leather with fine washed jeweller's rouge. This treatment gives results which are quite sufficient for all ordinary work, but for the very finest work under high powers the final polishing is done on wet parchment or kid skin with rouge. Stead* has designed a machine for polishing micro sections, sketches of which are given (figs. 266 and 267). At first he did the whole of his polishing on this machine, but latterly the rubbing on the first three grades of emery paper has been effected by hand, using the paper stretched or glued to blocks of wood about 5 inches square. The smoothest grade of paper and the cloth and chamois leather he still uses on the machine.

Stead usually requires from 10 to 15 minutes for an average sample of the sizes given above.

More recently Stead has designed another machine which can be driven either by hand or power, in which the specimens are held in movable clips over the polishing bed, and can be transferred in the clips to the microscope stand for examination, and, if any further polishing is necessary, can be again placed on the polishing machine. As the specimen is not removed from the clips during examination it can be refixed in the polishing machine without altering the relative position of the surfaces of the specimen and the polishing disc, and so ensures the polishing being continued in the same plane.

Ewing and Rosenhain's Methods.†—These operators rub the specimen on various grades of commercial emery paper from which the coarse particles have previously been removed by rubbing with a piece of flat steel as suggested by Arnold. The specimens are then finished on a rapidly revolving disc, covered with fine wash leather, charged with a thin paste of rouge and water. The rouge used for the finest work is specially prepared by precipitation from a solution of pure Ferric Chloride. In the

* *Iron and Steel Inst. Journ.*, 1894, vol. i., p. 305.
† *Phil. Trans. Roy. Soc., London*, vol. cxciii., p. 353.

case of samples easily tarnished wet polishing is avoided, and either the rouge is used dry or it is moistened with paraffin on the wash leather.

Mons. H. Le Chatelier has made some very important investigations on the best methods of preparing the polishing powders, as upon this, he considers, largely depends the success of the subsequent polishing opera-

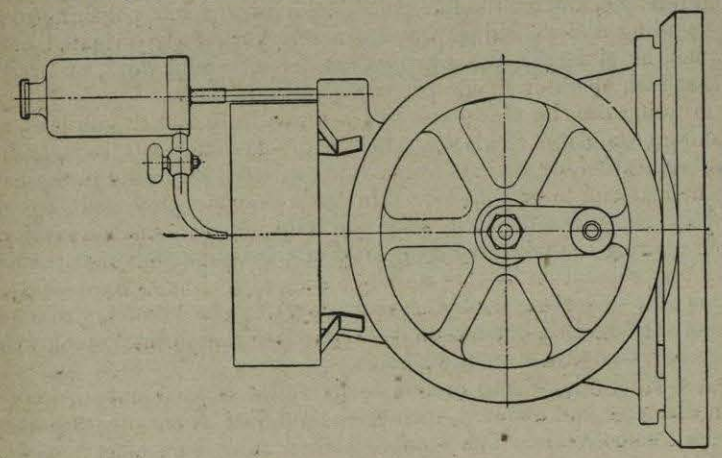


Fig. 267.

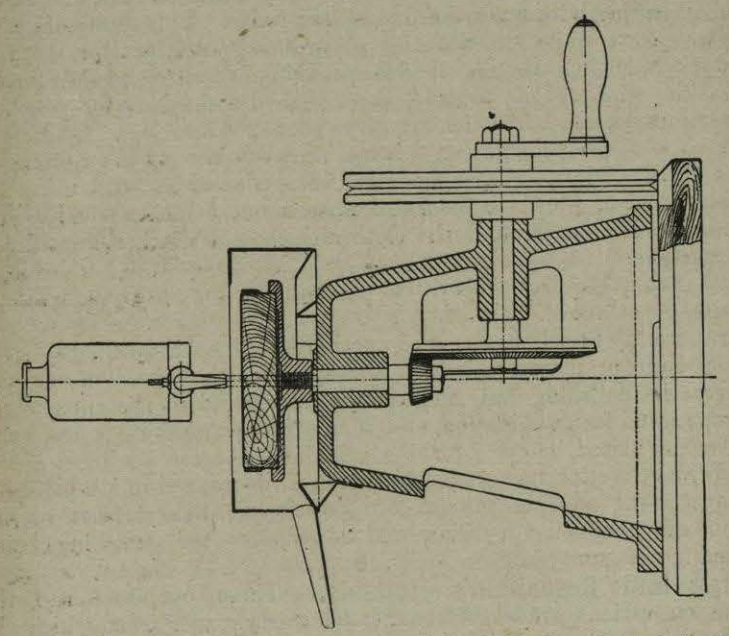


Fig. 266.

STEAD'S POLISHING APPARATUS.

tions, and also the length of time occupied. He also adapted Schloesing's method for the analysis of Kaolin to the washing and classification of the various materials used. The following description of the method is taken from Le Chatelier's paper in the *Metallographist** :—

"Mr. Schloesing treats the powders which he desires to classify with water containing 1 part of Nitric Acid in 1,000 parts of water, in order to

* *Metallographist*, vol. iv., No. 1.

dissolve the Carbonate and Sulphate of Lime and other salts which might be present. After standing a few hours, with occasional stirring, the mixture is allowed to settle. The powder falls rapidly to the bottom, and the clear liquid above it can be easily decanted. This liquid is now replaced by distilled water, and the mixture stirred; it is then again allowed to settle, and decanted. After a few similar operations, when all the acid has been removed, the settling takes place more slowly, and the liquid remains milky. The conditions are now favourable to proceed with the levigation, and the operation is further facilitated by adding 2 cubic centimetres of Ammonia to each litre of water, as it helps the suspension of the finest particles. Decantation is now resorted to, at stated intervals, by means of a syphon having a hook at its extremity, so as to avoid disturbing the portions which have settled.

"By treating 10 grains of powder in a 1-litre flask, nine-tenths of the liquid may be syphoned off without fear of disturbing the deposit.

"The decantations are made at the following intervals—fifteen minutes, one hour, four hours, twenty-four hours, and eight days.

"The deposit left after the first decantation contains all the coarse grains unsuitable for polishing. The second deposit, resulting from the first decantation, after one hour's settling, yields a substance which is not yet very homogeneous, but which may serve to start the polishing. The third deposit constitutes a good polishing powder for hard metals. It is, however, the deposit collected between the first and eighth day which constitutes true polishing powder. Instead of waiting eight days for the formation of the last deposit, it may be precipitated immediately after the removal of the 'twenty-four hours' powder by adding some Acetic Acid to saturate the Ammonia. The powder, still remaining in suspension, is then deposited after a few hours. The only objection is that some very fine particles, which would have remained suspended after eight days, are also precipitated.

"Once prepared, the powders must be carefully kept and contamination by any foreign matter avoided.

"A method which has given me very good results consists in converting them into a paste by mixing with soap. To do this, a piece of very dry Castile soap should be shaved by means of a very clean knife. The flakes of soap thus obtained are added to the polishing powder, when the latter is still wet, in the proportion of 1 part of dry soap to 10 parts of the wet powder. The mixture is melted in a water-bath, and allowed to cool, stirring all the while, until the mass begins to thicken; it is then poured into tin tubes similar to those used for keeping oil paint. After complete cooling the tubes are closed, and in this way the paste can be preserved very conveniently."

Le Chatelier has found that the substances which give the most satisfactory results are: commercial flour of emery; Oxide of Chromium (prepared by igniting Ammonium Bichromate); Alumina (obtained by the calcination of Ammonium Alum); and Oxide of Iron (from the strong ignition of Oxalate of Iron in the air). Alumina is considered to be very much superior to the others, both as regards results obtained and the expenditure of time.

Le Chatelier recommends, as supports for these powders, albumen, leather, cloth, velvet, and felt.

The method followed by him in polishing his samples is—first, to rub on commercial emery papers of various grades until all but the finest scratches are removed. This part of the polishing is done by hand. For the final polishing he uses first a disc covered with an emery paper, prepared with albumen and the washed powder which settles out after the second decan-

tation. Next, a felt or cloth disc upon which is placed some soap paste made with Alumina, which settles out between one and four hours; and finally, a disc of wood or ebonite to which is glued cloth or velvet covered with soap paste made from the Alumina deposited after twenty-four hours. The last three discs are rotated at as great a speed as possible, and for this purpose he uses a small machine made by Mons. Grauer, of Paris, worked by means of a treadle, and having the discs in a vertical position thus avoiding grit and dust settling on them. The discs have to be kept constantly moistened, and the sample is rotated in an opposite direction to that of the disc in order to avoid the rounding of the edges and the tearing open any small cavities or pits which may exist in the sample. M. Le Chatelier gives about five minutes as the time of polishing. He uses various reagents for etching, and recommends, amongst others, absolute alcohol containing 10 per cent. of gaseous Hydrochloric Acid to which from 1 to 5 per cent. of Anhydrous Copper Chloride has been added.

Sauveur's Method.*—Sauveur practically follows the method of Le Chatelier, except that he uses a paste made of tripoli and Castile soap for polishing. For general work he considers the treatment with rouge may be omitted, and altogether the entire polishing operation may consist of only three treatments. The machine is one of his own design, and carries four discs, which revolve in a vertical plane. Water is fed on near the centre, the rapid revolution of the discs causing it to be distributed over the whole surface. The machine is driven by a motor, and one operator can polish two samples at the same time. For a sample $\frac{1}{4}$ inch square, five or six minutes is sufficient for the whole operation.

The author uses a combination of several of the above methods. After sawing cold, the specimen, which is usually about $\frac{3}{8}$ to $\frac{1}{2}$ inch square and of suitable thickness, is filed as smooth as possible, and is then rubbed by hand on three grades of emery paper fixed to wooden blocks. For general work it is then only necessary to give one further treatment, which is effected with Alumina soap paste on a revolving cloth disc driven by a motor. For finer work rouged kidskin is used, when as fine a polish as can be desired is obtained in ten to fifteen minutes, according to the size and nature of the sample. In many cases excellent results have been obtained by Osmond's "Polish attack."

Development of Structure.—In the great majority of cases before the sample is examined under the microscope it is advisable, and generally necessary, to etch the polished surface to develop the structure, and various reagents have been suggested for this purpose.

Nitric Acid Etching.—Sorby used dilute nitric acid, and for ordinary work this gives good results, the strength of the acid being varied to suit the requirements of the case. Sir William Roberts-Austen for most work used a 1 per cent. nitric acid solution in alcohol; Osmond used a solution of 20 and 2 per cent. respectively of 1·33 specific gravity acid; Arnold recommends 1 part of nitric acid of 1·2 specific gravity in 49 parts of water, or 1 part acid in 199 parts of water, and for some purposes a still more dilute solution. Sauveur etches by immersing the specimen in nitric acid of 1·42 specific gravity for a few seconds, when the iron assumes the passive state. It is then well washed under a good stream of water, when a very slightly and evenly etched surface is obtained, free from the deposit of Carbon which is formed when dilute acids are used. By repeating the process any desired depth of etching may be obtained. Campion† also

* *Metallographist*, vol. iv., No. 4.

† *West Scot. Iron and Steel Inst. Journ.*, vol. vii., No. 2, 1899.

strongly recommends this method of etching, and the author has also found it to give excellent results. When nitric acid, either dilute or strong, is used the specimen, after well washing in water, should be rinsed in alcohol and afterwards wiped with a soft cloth moistened with benzene. It is then ready for examination under the microscope.

Iodine Etching.—Osmond recommends, for general work, tincture of iodine of B.P. strength, and prefers this to nitric acid, while Le Chatelier also prefers this reagent. The latter places some of the iodine solution on the polished surface, and gently rubs the surface with the finger-tip in order to obtain uniform etching and to prevent the constituents in the steel unacted upon from becoming tarnished. Stead prefers, for general work, tincture of iodine made by adding 1·25 grams of iodine and 1·25 grams of potassium iodide to 1·25 c.c. water, then adding alcohol to make the total volume 100 c.c. Sometimes he uses a tincture of one-fourth the above strength. He applies the iodine by placing 1 drop per sq. centimetre on the polished surface and leaving it until the liquid is decolorised, when the specimen is washed in water and alcohol and dried in a current of hot air. This treatment can be repeated until the structure is developed to the required extent. Ridsdale, for the determination of the size and definition of grain, uses a half-saturated solution of iodine in alcohol, immersing the specimen for one or two minutes and afterwards washing in water and methylated spirit or alcohol. The specimen is dried by heat or is lightly rubbed with chamois leather.

Etching with Picric Acid.—This reagent was suggested by Igevsky,* who uses a 5 per cent. solution in alcohol. It gives most excellent results, one of the chief advantages being that the Ferrite is not attacked or coloured, and the mottled appearance so frequently noticed after etching with nitric acid is avoided; it develops the structure with great clearness both in hard and annealed steels, is probably one of the best reagents yet suggested, and is very largely used.

Other Methods of Etching.—The "Polish attack" of Osmond with precipitated sulphate of calcium and liquorice infusion or 2 per cent. solution of ammonium nitrate on parchment is really a combined polishing, staining, and etching process. Martens also uses an alcohol-ether solution of hydrochloric acid. He has also shown that, by heating, the different constituents in the steel assume various colours, by which they may be distinguished under the microscope. Stead has used this method of heat tinting with much success for the examination of phosphoric steels, and has stated that this is the only way at present in which Phosphides and Carbides can be distinguished from one another when present in steel. The following directions for heat tinting are taken from Stead's paper, "Practical Metallography," read before the Cleveland Institution of Engineers in February, 1900:—

"In preparing the specimen for heat tinting, the bright surface should be well rubbed with a clean piece of linen or chamois leather; care must be taken to avoid any condensation of water on the surface; this is best secured by warming the specimen to 90° or 100° C. before giving it a final vigorous rubbing with the linen or chamois leather, and before it cools it should be placed on the iron plate. A sheet of iron, 6 inches square, placed over a Bunsen burner is all that is required for heat tinting. The section is placed in the centre, and the polished surface watched until the proper tint is obtained. Practical experience will soon enable one to find the best tint for any particular metal or alloy. It is advisable to heat gradually and examine periodically under the microscope, and stop when the structure appears to be most perfectly coloured. After each heating

* *Stahl und Eisen*, 1903, vol. xxiii., p. 120.

the section should be placed in a dish containing Mercury, so as to cool it rapidly and check further oxidation. If it is desired to photograph the heat-tinted object, it is most important in many cases to stop the heating when the object assumes a pale yellow tint, for although the contrasts to the eye are not so great, they are quite great enough for the sensitive photographic plate."

Saniter* has proposed a method of hot etching with fused chloride of calcium in order to develop the structure of steel at high temperatures. The calcium chloride is melted in a platinum crucible and heated to the desired temperature. The sample to be etched is placed in the fused mass and allowed to attain the same temperature. When this has occurred the whole is rapidly cooled by plunging the lower part of the crucible into cold water, &c. The adhering chloride is then dissolved off.

Various other etching media have been suggested from time to time, such as chromic acid mixture, sulphuric acid, potash, potassium sulphate, &c., but those described above are the ones most generally used. The best method to adopt for developing the structure depends entirely upon the nature of the material under examination, the previous treatment to which it has been subjected, and the particular structure it is desired to examine. In some cases it may be necessary to compare the structure of the same section which can be developed by different methods. Probably picric acid or the combined polishing and staining method of Osmond is the best to use for developing the structure of the various microscopic constituents under very high powers, especially in the case of annealed steels. For the development of structure, and in cases where measurement of size of grain is required, 1 per cent. of nitric acid in alcohol, tincture of iodine, and strong nitric acid, with subsequent washing, all give very good results. Considerable experience is required in order to know which reagent will give the best result in particular cases, and nothing but repeated examination of various samples, etched by different methods, will enable the student to decide which is the best to use in any particular case.

Microscopic Apparatus.—Space will not permit of a detailed description of the microscope, and for this the student must be referred to a standard treatise on the subject, but there are several points in connection with the microscope to which it is desirable to call attention, and some special accessories used in examining metallic sections which it will be necessary to describe. Any good microscope stand may be used—preferably arranged to be used in a horizontal position—and it should have a sensitive fine adjustment. The base should be sufficiently large and weighty to render the instrument perfectly steady for the use of high power objectives. Metallic objects, being opaque, cannot be examined by transmitted light, and consequently special illuminating apparatus has to be used even for moderately high powers.

Illuminating Apparatus.—The illumination used may be either vertical or oblique, and in many cases it is desirable to examine the specimen by both. For low power objectives the light may be thrown on to the object by means of reflectors placed *below* the object glass, but for high powers they must be placed *above*, that is between the object glass and the eye-piece of the microscope. For examination with low powers the Sorby-Beck reflector is the most useful type of illuminator, and by its use either vertical or oblique illumination can be obtained.

The construction of this illuminator is shown in fig. 268. It consists of a parabolic reflector to which is attached a little silver mirror, with a face at an angle of 45°, which can be turned away from or under the object glass by means of the milled head, A. When oblique illumination is required,

* *Iron and Steel Inst. Journ.*, 1897, vol. ii., p. 123.

the mirror is thrown outside into position shown by the dotted lines (fig. 268), and the specimen is examined by light reflected from the silvered parabola, S. When direct illumination is desired the little mirror is turned back into position shown in fig. 269, so that it partly eclipses the object glass, but leaves room for the horizontal rays, L, reflected from the mirror on to the object to be reflected back from its surface into the microscope.

In the case of oblique illumination the horizontal rays of light, L (fig. 270), falling on the parabola are reflected at various angles upon the object below, and if the surface is perfectly flat none of this light is reflected back into the microscope, and consequently such a surface appears quite black, and cannot be seen with the microscope. If, however, one constituent is in relief as the light falls upon one side of the projecting portion, the light is reflected back into the microscope, and it appears brilliantly illuminated, leaving the other side in shade. Oblique illumination is therefore very useful for determining which constituent is in relief, and which part of a structure is broken up.

When direct illumination is required for low powers, instead of using the Sorby mirror, a very simple arrangement suggested by Stead* will be found to give excellent results. The device is shown in fig. 271. A piece of blackened cardboard is fixed behind the object in a vertical position, and a cover-glass, *g*,

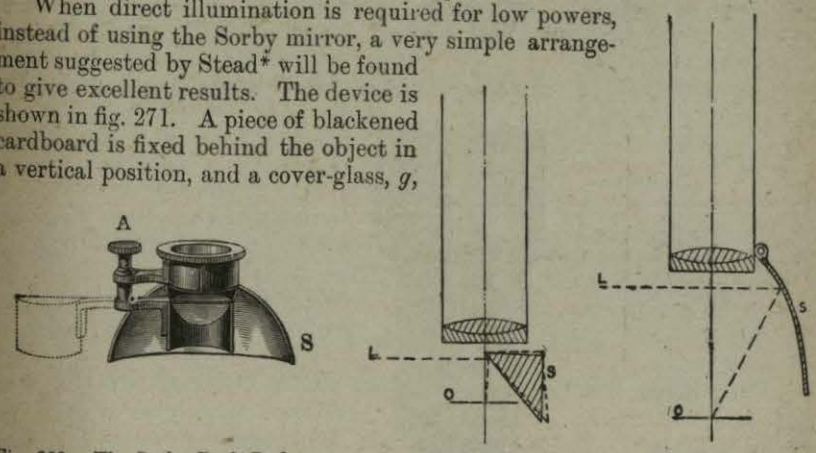


Fig. 268.—The Sorby-Beck Reflector. Fig. 269. Fig. 270.

is placed over the object at an angle of 45°, both being temporarily attached to the slip holding the object with plastic wax. The horizontal rays of light are reflected down by the cover-glass on to the object, and then back from the object through the cover-glass into the microscope, as shown in the sketch.

For high powers above $\frac{2}{3}$ of an inch, direct illumination is always used, and Beck's illuminator is a very useful form. It is exactly similar in principle to that last described except that the reflector, which is a cover-glass, is above the object glass, as shown in fig. 273. It consists of a metal collar or tube in which is fixed on a pivot a cover-glass which acts as a reflector, and which can be rotated to any angle by the milled head shown, light being admitted to the reflector through an opening, A, in the metal tube (fig. 272). The collar is screwed into the microscope tube, and the objective then screwed on to the bottom of the collar, so that the reflector is in the tube of the microscope. The reflector is turned to an angle of 45°, rays of light, L, admitted through the opening, *a*, fall upon the reflector, G (fig. 273), are reflected down on to the object, and then up from its surface through the object glass and reflector to the eyepiece. The above illustrations are from blocks kindly lent me by my friend Mr. Stead, who had them specially prepared to illustrate his paper on "Practical Metallography." Professor Martens and others use

* "Practical Metallography," *Proceed. Cleveland Inst. of Engineers*, Feb. 26, 1900.

a right angled prism fixed in the microscope tube in the same manner as the cover-glass in the Beck reflector.

Fig. 275, for which I am indebted to Mr. Carl Zeiss, shows this form of illuminator, and the sketch in fig. 274 explains how the light is reflected.

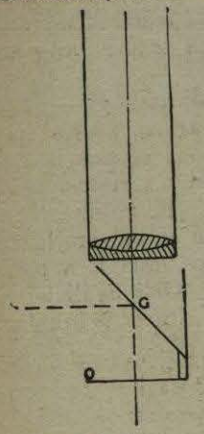


Fig. 271.

Beck's Illuminator.

Fig. 272.—Reflector.

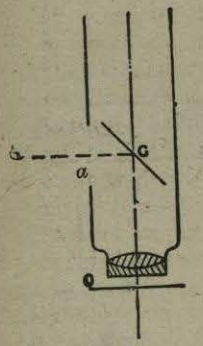
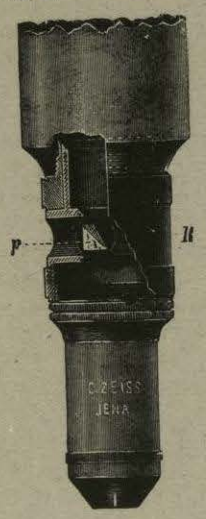


Fig. 273.



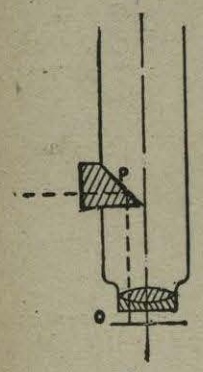


Fig. 274.

Fig. 275.

Light for Illumination.—For ocular examination any ordinary oil lamp may be used with good results up to about 150 diameters. A Welsbach gas burner or acetylene lamp is, however, preferable, and may also be used for photographing up to about 250 diameters, but for very high magnifications the electric arc is necessary for good and rapid work. Between the source of light and the illuminator a bull's-eye condenser should be placed to concentrate the rays. There are many forms of cameras to be obtained for photomicrography, and they may be made to work in either a horizontal or vertical position. The camera with which all the photographs that follow were taken was a horizontal one similar to that shown in fig. 276.

In taking a photograph the object should be first carefully focussed under the microscope in the usual way, and without removing the eyepiece, the hood of the camera pushed over the latter, and the camera extended to give the magnification required. The final focussing is best done on a plain glass plate instead of the usual ground glass camera screen, and, a No. 1 eyepiece being placed on the centre of the glass, the fine adjustment micrometer screw is turned until a very distinct image of the object is seen. If the illumination is good the focussing can be done directly on the ground glass screen without an eyepiece and excellent results obtained. The glass screen is then replaced by a slide with a photographic dry plate in position, the slide drawn and an exposure made. The time of exposure will depend upon the magnification required, the source of illumination, the rapidity of the plate, and to some extent upon the object, and it is a matter of experiment to determine what length of exposure gives the best results for the particular conditions. Using an arc lamp and plates

of medium speed, the author finds from six to ten seconds exposure sufficient for magnifications of 100 diameters, and for 1,000 the exposure will vary from 20 to 30 seconds. The photographic plate may be developed in the usual way.

When purchasing a microscopic outfit it is far cheaper in the end to go to one of the first-class makers, and the following list of microscopic accessories may be considered necessary for good all-round work, although a beginning can be made with less :—

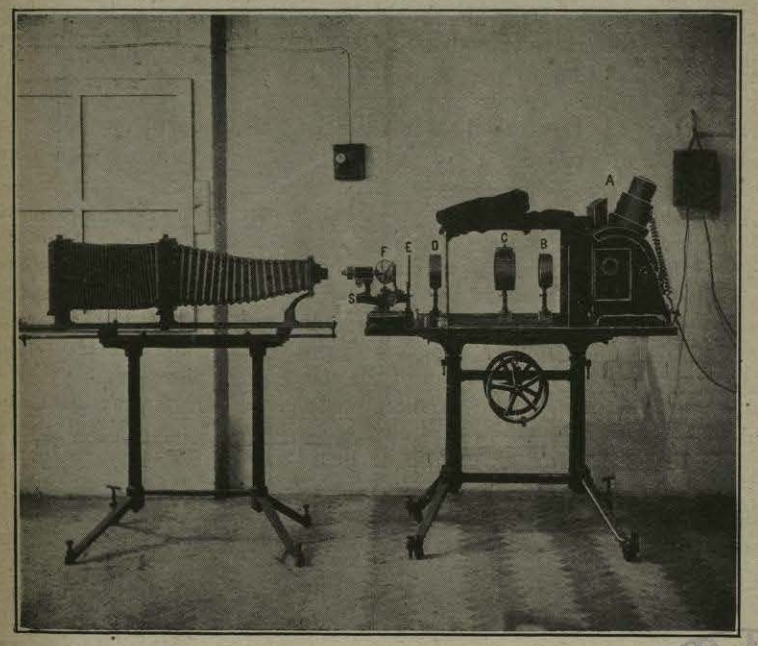


Fig. 276.

Microscopic Accessories.

1 microscope stand of any reliable maker.
Objectives.—1 planar objective 35 mm.
2 projecting aplanats 35 mm. and 70 mm.
1 apochromatic 16 mm.
1 oil immersion 3 mm.
Eyepieces.—Huyghenian, No. 2, for general examination.
Projection, Nos. 2 and 4, for photography.
Some suitable source of illumination.
1 Beck reflector for low powers.
1 Zeiss or Nachet prism reflector.
1 micrometer slide (1 mm. divided into 1,000 parts).
1 camera specially designed for micro work, and if of considerable length an arrangement for working the fine adjustment from a distance, such as "Hook's Key," must be fitted.

Microscopic Examination.—The microscopic examination of steel samples may be considered under three heads :—(1) General examination for mechanical defects ; (2) examination for size of grain and general crystalline structure ; (3) examination to determine the microstructure and to identify the micro-constituents present and their distribution through the steel.

Examination for Mechanical Defects.—In this examination the surfaces to be examined must *not* be ground down, as this would remove small cracks or flaws. The best way is to rub with very fine emery paper (No. 000) afterwards on paper No. 0000, and finally on rouged chamois leather or kidskin. A somewhat pitted surface will be left, suitable for examination under a low power. If nothing is visible the specimen should be immersed in a 1 per cent. solution of nitric acid for a few seconds, and well washed, first with water, then limewater, and then water again. After rubbing with the rouged chamois leather, it should be again examined. If nothing is visible, repeat the treatment; if still no cracks or defects are visible, treat again, this time using 10 per cent. acid for about a minute. If nothing now appears, surface cracks and flaws may be considered absent. The above treatment is very useful when examining the running heads and surfaces of rails and tires, &c. This examination is always made with a very low power.

A method which very often gives valuable results for rails, tires, axles, and similar sections is to cut a complete cross-section of the article to be examined, and, after covering one face and the edges with wax or acid-proof varnish, to immerse it in a 20 per cent. solution of sulphuric acid kept at a temperature of 60° C. for a quarter of an hour, and then well wash in water. Slag flaws, blowholes, rolling cracks, &c., are generally made clearly visible. In some cases half an hour's action of the acid may be necessary.

Steels containing a large amount of Sulphur or Phosphorus often show deep, uneven pits and seams after treatment in this way, and the information obtained is frequently of great assistance in determining where to cut the sections for the subsequent microscopic examination.

Samples of wire rod which have failed, very frequently exhibit a central crack or hollow cavity when treated by this method, especially such samples as break with what is known as a "cuppy" fracture.

Examination for Size of Grain.—For this examination the sample must be well polished to remove all scratches due to grinding, and Stead recommends etching with a 20 per cent. nitric acid solution. Ridsdale prefers to immerse the specimen for one or two minutes, in a half saturated solution of iodine in alcohol, and the author has obtained very good results with Sauveur's method of dipping in strong nitric acid and then washing in a stream of water. Any of the above methods of etching will give good results for this purpose, and after well washing in water and spirit the sample is ready for examination. Both oblique and vertical illumination should be used, and comparatively low powers are required, a magnification of from 50 to 100 diameters generally being quite sufficient, or in cases of exceptionally coarse structures a much lower magnification. Such an examination will generally reveal any coarse crystallisation, and will often give a clue as to the heat treatment to which the steel has been subjected, which must be confirmed by further examination under higher powers. As the size of the grain will vary with the heat treatment it is often important to measure this, and keep a record for reference to compare with similar sections. The simplest way of doing this is to use a micrometer eyepiece, as the average diameter of the grains can then be read off direct from the microscope. Another plan is to take a photograph of the sample at a given magnification and afterwards photograph the divisions of a stage micrometer slide at the same magnification, and place the photograph of the slide over that of the sample when the size of grain is readily obtained. A third method is to use a camera lucida with the microscope and go over the image with a planimeter and thus obtain the grain measurement. In drawing conclusions from the size of grain in any piece of steel the sectional area of the steel sample must be carefully considered, as it is the relation to this sectional area rather than the absolute size of the grain which is important.

Examination to Determine Microstructures.—This examination is by far the most difficult, as it involves the identification of various microstructures and constituents, and where these are not clearly defined, or where they merge the one into the other, considerable experience is necessary to determine the exact structure and distinguish the particular constituents. In preparing the sample, it is polished and etched by one of the methods described and mounted upon a slide with some plastic material. As a rule, direct illumination is best for this examination, and high powers are in many cases necessary, some structures not being resolved under 1,000 diameters, while for others 1,500 diameters are desirable, although for most work a magnification of 500 or 600 diameters is sufficient.

The only way to acquire an exact knowledge of the microstructure of steel is to carefully prepare specimens until the student can readily identify the chief constituents and structures, and so gradually gain experience, no amount of examination of photographs giving him the necessary knowledge. The following are the four chief microstructures which are now fully recognised, viz.:—Ferrite, Cementite, Pearlite, and Martensite, and some of these are usually found in all steels, whether quenched or unquenched.

There are three other Carbon conditions identified by the microscope—viz., Sorbite, Troostite, and Austenite—which are found only in steels within certain limits of composition, and which have been subjected to special heat treatment.

Ferrite (the name suggested by Howe for the crystals of practically pure iron, or, as Sorby calls it, free iron) is the chief constituent of soft steel that has been slowly cooled, or quenched below the critical point, Ar_1. It is described as crystallising in polyhedric grains, which are interfering crystals of the isometric system, mostly cubes and octahedra. On etching the polished surface of a nearly carbonless iron with a solution of iodine or nitric acid, or, better still, picric acid in alcohol, it will be found that the Ferrite remains white and brilliant, or takes only a slightly mottled appearance. On polishing a steel, the Ferrite, being softer than the other constituents, wears away sooner, so that the final result leaves the other constituents of the steel standing in relief on the worn-down Ferrite. This constituent in the *free state* is practically absent from steels containing above ·9 of Carbon.

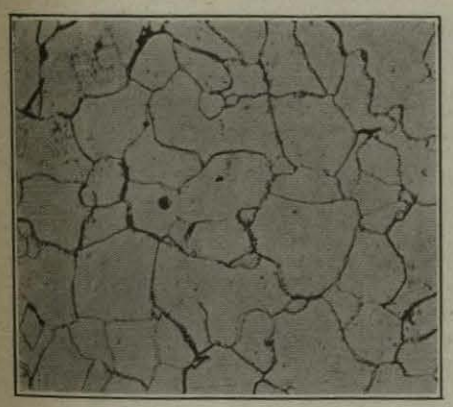


Fig. 277.—Ferrite or nearly Carbonless Steel. Magnification, 150 diameters. The junctures of the Ferrite grains are clearly shown, and this is characteristic. In some cases Ferrite appears as a white body, no junctures being visible, when the surface is only slightly etched.

It must be remembered that these Ferrite crystals, although they have the appearance of, are not necessarily pure iron, but may contain Silicon, Phosphorus, &c., in fact, any metalloids or impurities in the steel except Sulphur and Carbon, which do not appear to penetrate the Ferrite. Fig. 277 is a photograph of steel containing ·057 per cent. of Carbon, and consists almost entirely of Ferrite.

Cementite.—This constituent is undoubtedly a Carbide of Iron, and its composition would appear to be expressed by the formula Fe_3C. It occurs in bands, or segregated masses in high Carbon steel, is intensely

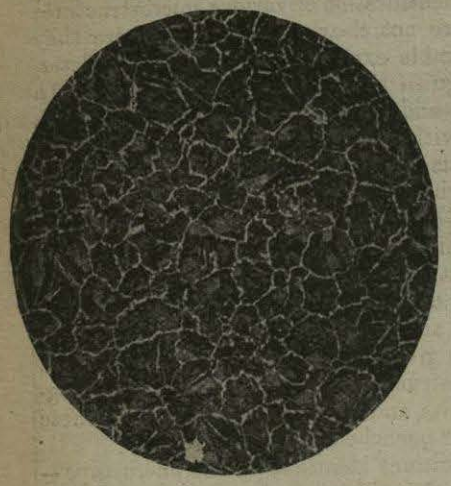


Fig. 278.—Steel containing 1·306 per cent. of Carbon. Magnification, 150 diameters. The white thread-like bands surrounding the dark areas are the Cementite.

Fig. 279.—Same as 278. Magnification, 1,500 diameters. This shows the white bands of Cementite much enlarged, and that the dark areas which they more or less surround are laminated Pearlite.

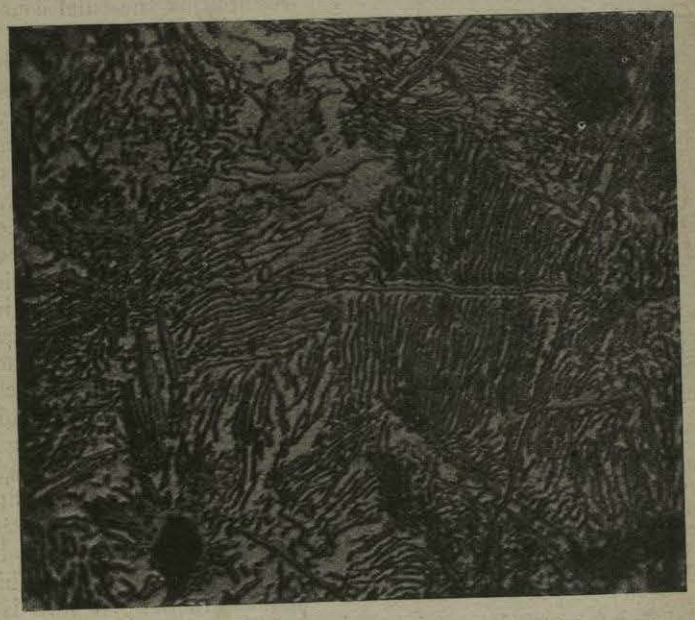


Fig. 280.—Another high Carbon steel showing the Cementite segregated in masses associated with Pearlite. The white raised portions are Cementite, the dark laminated portions Pearlite. Magnification, 1,000 diameters.

brilliant by direct illumination under the microscope, but perfectly black by oblique illumination. It often occurs as thin bands surrounding the Pearlite

grains. It is intensely hard, and remains bright even after repeated attacks with iodine, nitric acid, or picric acid. It cannot be scratched by a needle, whilst Ferrite can. This constituent is abundantly present in high Carbon, unhardened steel, but is practically absent in the *free state* from steel containing under ·9 per cent. of Carbon.

It is not necessarily pure Carbide of Iron but may contain other Carbides present in the steel. Figs. 278, 279, and 280 are characteristic photographs of Cementite as it appears under low and high powers respectively.

Pearlite is an intimate mixture of Ferrite and Cementite. It occurs in two varieties—the lamellar and the granular. The lamellar, or laminated, so-called because it is made up of very thin plates or lamellæ of Ferrite and Cementite alternately, is found in steel very slowly cooled from a high temperature, and especially in annealed steel. It takes its name from the play of colours seen after etching, suggestive of mother-of-pearl. The granular variety consists of fine globules of Cementite in a matrix of Ferrite, chiefly found in steels which have been reheated to a low temperature; the Pearlite in steel annealed at just under Ar_1 is granular. Pearlite appears to be practically the sole constituent of unhardened steel, containing from ·8 to ·9 per cent. of Carbon.

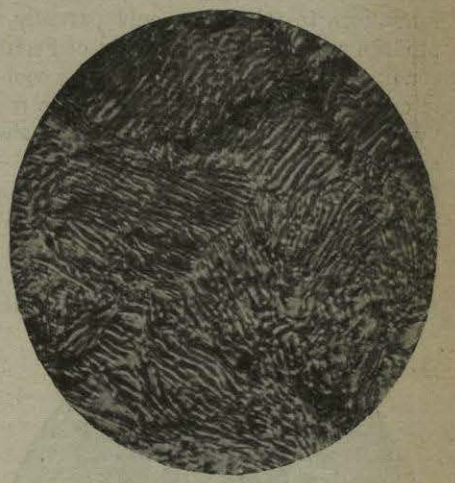


Fig. 281.—Steel containing ·871 C. Magnified, 1,500 diameters. Showing typical structure of laminated Pearlite.

Fig. 281 is a photograph of steel containing ·87 per cent. of Carbon under a high power, showing the typical structure of laminated Pearlite.

Martensite.—This is the microstructure which is characteristic of steels quenched suddenly from high temperatures. Under the microscope it appears like a system of interlacing crystalline fibres. The real nature of Martensite is still in dispute, but it may best be considered as the first stage in the transformation of Austenite.

On quenching small samples of steels containing from ·2 to ·8 per cent. of Carbon from above Ar_3, the structure apparently consists entirely of Martensite.

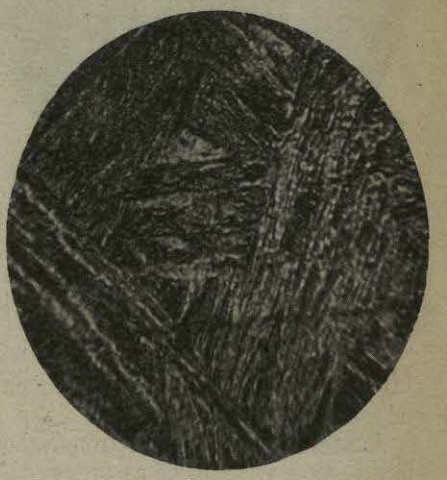


Fig. 282.—Steel containing ·468 C quenched from a temperature of 820° C. in water at 15° C. consisting entirely of Martensite. The interlacing crystalline structure is characteristic of Martensite.