MICRO-STRUCTURAL CHARACTERISTICS OF STEEL.

FRANCIS SCOTT RICE, STEELTON, PA.

In 1864, Dr. H. C. Sorby, that capable veteran in microscopy, of whom our worthy President spoke last evening, published a paper through the Sheffield (England) Literary and Philosophical Society, entitled, A New Method of Illustrating the Structure of Various Kinds of Steel by Nature Printing, so far as known the first paper treating of the micro-examination of steel. He occupied the field alone for nearly twenty years; but others took up the work, more especially in England, until, in 1891, Mr. J. E. Stead writes: "It is evidently time that this particular branch of research should not be neglected, and that in our works, and all metallurgical laboratories, the microscope should hold a prominent if not a premier position." American workers have taken up the subject, and at the present time the method is used in examination of castings; important pieces, such as car axles; inspection of armor plate for our navy; and, finally, in the study of various commercial forms of steel with a view of improving the product and methods of working. Scientific interest in the study will perhaps prove sufficient to attract more workers, but the commercial importance of the results will surely make the microscope a most valued instrument to the metallurgist in the near future. The following notes may be of interest as recording the experience of some study. As regards the preparation of specimens:

After trying many sizes of sections, the uniform size of three-fourths of an inch in diameter has been adopted as being convenient to mount on ordinary slips; much smaller sections are hard to grind to a plane surface, having a tendency to round on the edge, while in larger surfaces the central part of the section is hard to polish. If the sections are to be submitted to heat treatment after cutting in the lathe, the thickness should be at least three-sixteenths of an inch in order to properly retain heat. After many trials of emery and crocus papers and powders, jewelers' rouge, wheels speeded to over 3,000 revolutions per minute and charged with various polishing and cutting compounds, it is thought that the best sections are obtained by carefully grinding off the surface to a plane by hand on an ordinary quick-cutting oil stone, then on the finest Belgian oil hone, and, finally, polishing on a piece of chamois tightly stretched over a block of wood and charged with peroxide of tin. The last operation must be accomplished with frequent reference to the microscope, and to reach perfect results the peroxide of tin must be levigated and the finest powder only be used for finishing. The peroxide tends to stick to the ground surface of the specimen at first, but may be rubbed off with a damp cloth and the polishing continued with very light pressure until the surface becomes more smooth. Since scratches can never be confounded with structure, a perfect polish is for appearances only. The great object is to get a perfectly clean, sharply cut plane without any distortion or crushing of the softest structure on the surface. Finally, wash thoroughly with alcohol, followed by a little chloroform.

A polished section of steel examined under the microscope by oblique reflected light appears uniformly dark, the polished surface reflecting all rays of light outside of the objective. Seen by direct reflected light, all rays will be reflected to the objective and the surface appears uniformly light. To develop the structure it is, therefore, necessary to etch the polished surface. One method is by use of dilute nitric acid, which is very uncertain and hard to control. A better method is to dip the specimen into concentrated nitric acid of specific gravity of 1.40 or more and then place under a water tap. The concentrated acid has no effect until diluted by the running water, when it etches rapidly for the short time until it is washed off by the water. This application may be repeated a second time if necessary. The surest and most delicate results, however, seem to be gained by use of a saturated solution of iodine in alcohol, diluted with an equal quantity of alcohol, both 95 per cent. pure. While comparatively slow and sometimes requiring many applications before the desired effect is obtained, it can be controlled and gives uniform results. After each etching wash carefully in 95 per cent. alcohol, dry quickly or oxidation will begin at once; polish briskly on chamois and examine under the objective. When satisfactory the section may be mounted on an ordinary slip with Canada balsam, so that it can be used in the common mechanical stage or stored in regular cabinets. A thin coating of vaseline has been found the best protection from oxidation when not under observation, and can be quickly cleaned, when necessary, with a cloth and alcohol. The details of the structure are almost wholly lost if cover slips are used.

The examination of the specimens requires some sort of vertical illuminator. In the Tilghman type, through the reflection of a beam of light from a thin, clear glass disc placed in the optical axis at an angle of forty-five degrees, the reflected rays pass vertically to the back of the objective which acts as its own condenser, and throws a brilliant point of light on the surface. Again reflected back to and gathered by the objective, the image rays mainly pass through the clear disc to the eye. This illumination gives the best results with lower powers, and the photo-micrographs appended were taken with its help. The prism illuminator, as constructed by Zeiss, is a most perfect appliance and most satisfactory for higher powers, from 4.3 mm. focus up. It shuts off one-half of the objective, but the light is ample and the definition beautifully clear. The beam of light for either kind is most conveniently secured from a bull's-eye between lamp and instrument, and for photography the illuminant must be cut down by diaphragms to a round, even circle of light, the smaller in size the better for definition.

The photographic camera used was made to order by a handy cabinet worker and designed after the suggestions of Dr. Van Heurck in his book The Microscope. It is a vertical box of right size inside for 8 x 10 inch plates, arranged with slides for plate holders at different heights from the eye-piece; is supported by four stout legs of sufficient height to allow the microscope to slide under it, and rests on a solid base board. Velvet-lined slides confine the microscope always to the same position; the front and top are hinged so that focusing can be easily accomplished for plates at any height. A simple shutter, working from the outside, controls the exposure. It is perfectly rigid and solid, can be used with immersion objectives, and is convenient to operate. My own is made of cherry and is a solid and rather handsome piece of furniture, with no suggestion of its use in its appearance; but an equally good one can be made of pine lumber for about five dollars. This experience is given as a hint to those who, although wanting a photo-micrographic camera, feel that the expense would be burdensome.

Disregarding any small impurities, all steel is composed of two primary constituents, which are known as ferrite, or the original iron, and carbon, through the presence of which iron becomes steel. Ferrite may occur either segregated and free or in combination, but carbon is always in combination with the iron, forming a carbide with the formula $Fe_3C.$, which is called cementite. Cementite recombines with the ferrite to form pearlyte in all steels normally cooled; but combines in a different proportion to form a different structure, called martensite, if the steel be heated to 800° C. or over; and if the specimen be then suddenly cooled by plunging into cold water or other fluid, this form may be instantly "fixed" and then be available for microscopic observation. Steels cooled, as in ordinary working by the surrounding air, will be termed "normal" as regards heat treatment; those suddenly cooled will be called "quenched"; and those cooled so slowly that the whole mass of metal is practically of one temperature until completely cooled, are termed "annealed." This nomenclature of microscopic constituents and conditions of heat treatment is universally accepted by metallurgists in microscopic work.

Passing to the conditions under which any or several of these constituents appear, and the characteristics of each as regards etching, color, proportion and structure: Ferrite, the larger constituent, will, of course, appear alone in iron, as in Fig. 18. In soft normal steels it appears as crystals, mostly octahedra, as in Fig. 6. As carbon is added it combines with the cementite to form pearlyte, until with 0.80 per cent. of carbon the whole mass is pearlyte, as in Fig. 10, and this is termed the "saturation point," since in steels with greater percentage of carbon the cementite appears segregated, as in Fig. 13. Under etching ferrite is discolored, as in Fig. 7, but by rubbing on chamois is left clear and white, as in Fig. 1; and slightly deeper etching shows a papillar appearance on some grains with highly iridescent effects, as in Fig. 2. Ferrite is the softest of the four constituents.

Cementite occurs only in combination with iron, as pearlyte or martensite, until the carbon percentage is greater than 0.80 per cent., then appearing as plainly outlined cell wall, as in Fig. 13, and with about 1.30 per cent. of carbon as segregated masses, but is always structureless. It is the hardest of the constituents, remains brilliant after etching and has a more metallic appearance than ferrite.

Pearlyte is strictly a condition only of the combination of ferrite and cementite in normal and annealed steels, while the condition of the same combination in quenched specimens is termed martensite. The distinction, however, rests not only on the micro-structural differences, but also on wholly different physical characteristics. Martensite is the condition under which steel exhibits hardness, while pearlyte possesses no such properties. Pearlyte is colored dark by etching and forms from none to 100 per cent. of the mass as the carbon percentage rises to 0.80 per cent. The micro-structure is somewhat uncertain; but in crucible steels and those more thoroughly worked it appears plainly as a laminated structure, resembling little contour maps and formed of alternate plates of cementite and pearlyte, as in Fig. 12. The lamination gives rise to beautiful iridescent effects like mother-of-pearl, whence its name. Since this effect appears in specimens etched only in the slightest degree, I am inclined to believe that we have here to do with a diffraction grating, formed by plates of greater or less thickness and of different reflective power.

Martensite is a differing condition of the same constituents, as in pearlyte, caused by heating. In specimens properly heated and quenched its proportion of the mass increases with the carbon percentage until it forms 100 per cent. with 0.20 per cent. carbon; this proportion of the mass remains constant until about 1.00 per cent. of carbon is added, when free or segregated cementite appears. Combining in these different proportions it forms a material of corresponding hardness, and with etching deepens in color proportional to the amount of carbon from a slightly yellow to almost perfectly black color. It may be added that martensite is the key to the whole tool steel industry, and an immense amount, in fact, nearly all of the micro-structural work done has been to approve or disapprove the various theories of the hardening of steel; whoever solves the micro-structure of martensite will not only settle the theories, but place tool steel manufacture on a sure and scientific basis.

It is anticipated that micro-structural study of almost every variety of steel product will be taken up in the immediate future. As mentioned, it is used in examination of armor plate, doubtless by showing how thoroughly and to what depth a carbon absorption from the outside has hardened a milder plate. It is also hoped that it will prove a quick and sure method, both for manufacturer and engineer, in the production and inspection of structural steels, leading to a more homogeneous and reliable product, and safely allowing the use of steels of higher carbon and correspondingly greater tensile strength ; decrease the weight of bridges and add to the safety of property and human life. The study has proved a most interesting and absorbing one, although prosecuted in the intervals of a very busy life, and perhaps in no field of microscopic research are there more important results promised from a scientific standpoint ; and abundant remuneration will also come to the successful investigator. The presentation of these few very imperfect results to this Society is a great pleasure, and it is hoped that interest in the subject may be awakened both in individuals and the Society. All the figures herewith shown are from permanent enameled bromide prints of a series of negatives taken under the same conditions as regards plate, exposure and so nearly as possible development. The magnification is x200 diameters, and each view shows an actual area of 1-100 inch or 0.25 mm. Pieces marked "same as" are actually cut from same bar and supposed to be of identical chemical composition. Several failures are shown also, as examples of what to expect from imperfect manipulation.

BIBLIOGRAPHY.

1. H. C. Sorby: On a New Method of Illustrating the Structure of Various Kinds of Steel by Nature Printing. Sheffield Literary and Philosophical Society, February, 1864.
2. H. C. Sorby: On the Microscopical Structure of Meteorites and Meteoric Iron. Proceedings of the Royal Society, Vol. XIII., p. 333, and British Association Report, Part I., p. 139, 1865.
3. H. C. Sorby: On Microscopical Photographs of Various Kinds of Iron and Steel. British Association Report, Part II, p. 189, 1864.
4. H. C. Sorby: On the Microscopical Structure of Iron and Steel. Dr. Lionel Beale's How to Work with the Microscope, 4th Ed., pp. 181–183, 1868.
5. A. Martens: Ueber die Mikroskopische Untersuchung des Eisens. Zeits. des Ver. Deuts. Ing, Vol. XXI., pp. 11, 205 and 481, January, May and November, 1878; also Vol. XXIV., p. 397, August, 1880.
6. H C. Sorby: Lecture delivered in the Firth College, read October 20, 1882. The Engineer, Vol. LIV., p. 308, October 27, 1882.
7. A. Martens: Ueber die Mikroskopische Untersuchung des Eisens. Verhandl. des Ver. zur Beförderung des Gewerbfleisses, Sitzungsberichte, p. 233, 1882.
8. J. C. Bayles: Microscopic Analysis of the Structure of Iron and Steel. Trans. of the American Institute of Mining Engineers, Vol. XI, p. 261, 1883.
9. O. Dolliak: Beiträge zur Mikroskopie der Metalle. Mittheil. über Gegenstände des Artillerie und Geniewesens, Heft 9. p. 467, 1883.
10. A. Martens: Erläuterungen einer in der kön. Bergakademie zu Berlin befindlichen Sammlung von 120 Schliffen zur Darstellung des Mikroskopischen Gefüges verschiedener Eisen und Stahlsorten. Berlin, 1884.
11. F. Osmond et J. Werth: Structure Cellulaire de l'Acier Fondu. Comptes rendus de l'Academie des Sciences, Vol C., p 450, February 16, 1885.
12. F. Lynwood Garrison: The Microscopic Structure of Iron and Steel. Trans. American Inst. of Mining Engineers, Vol. XIV., p. 64, 1885.
13. H. Wedding: The Properties of Malleable Iron, deduced from its Microscopic Structure. Journal of Iron and Steel Institute, p. 87, 1885.
14. F. Osmond et J. Werth: Theorie Cellulaire des Proprietés de l'Acier. Ann. des Mines, 8th Series, Vol. VIII., p. 5, July to August, 1885.
15. F. Lynwood Garrison: The Microscopic Structure of Car Wheel Iron. Trans. American Inst. of Mining Engineers, Vol. XIV., p. 913, 1886.
16. H. C Sorby: On the Application of Very High Powers to the Study of the Microscopical Structure of Steel. Journal of Iron and Steel Institute, p. 140, 1886.
17. H. Wedding: Die Mikrostructur des Gebrannten Eisens. Stahl und Eisen, Vol. VI., p. 633, October, 1886.
18. H. Wedding: Die Mikrostructur des Eisen. Stahl und Eisen, Vol. VII., p. 82, February, 1887.
19. F Lynwood Garrison: Microscopic Structure of Steel Rails. Trans. American Inst. of Mining Engineers, Vol. XV., p. 761, February, 1887.
20. A. Martens: Ueber das Kleingefüge des schmiedbaren Eisens, besonders Stahls. Stahl und Eisen, Vol. VII., p. 235, April, 1887.

21. H. C. Sorby: The Microscopic Structure of Iron and Steel. Journal of the Iron and Steel Institute, p. 255, 1887.
22. H. Schild: Die Neuesten Forschungen auf dem Gebiete der Mikroskopischen Untersuchung von Stahl und Eisen. Stahl und Eisen, Vol. VIII., p. 90, February, 1888.
23. H. Wedding: Zusammenhang zwischen der Chemischen Zusammensetzung und dem Kleingefüge einerseits und der Leitungsgüte des Telegraphendrahtes anderseits. Mittheil. aus den kön. technischen Versuchsanstalten, Ergänzungsheft I., p. 6, 1888.
24. H. Wedding: Ueber Fortschritte in der Lichtabbildung des Kleingefüges von Eisen und die Herstellung von Schliffen. Stahl und Eisen, Vol. IX, April, 1889.
25. A. Martens: Ueber die Mikroskopische Untersuchung des Kleingefüges von Eisen. Stahl und Eisen, Vol. IX, p. 393, May, 1889.
26. F. Osmond: Le Fer et l'Acier. Lumiere Electrique, Vol. XXXV., p. 256, February 8, 1890.
27. H. Wedding: Das Kleingefüge des Eisens. Mikroskopische Originalphotographien mit Erläuterungen Berlin, 1891.
28. Sir F. A. Abel: Presidential Address. Journal of the Iron and Steel Institute, No. 1, p. 18, 1891.
29. F. Osmond: Note on the Micro-structure of Steel. Journal of the Iron and Steel Institute, No. 1, p. 100, 1891.
30. H. Behrens: Sur la Structure Microscopique et sur la Trempe de l'Acier et de la Fonte. Recueil des travaux chim. des Pays Bas, Vol. X., p. 261, 1891.
31. H. Wedding: Das Gefüge der Schienenköpfe. Stahl und Eisen, Vol. XI., p. 879, November, 1891.
32. A. Martens: Die Mikrophotographische Ausrüstung der kön. Mechanisch-Technischen Versuchsanstalten. Mittheil. aus den kön. Technischen Versuchsanstalten, Heft 6, p. 278, 1891.
33. P. H. Dudley: Microscopic Structure of Steel. Journal of the New York Microscopical Society, October, 1891.
34. A Martens: Ueber Einige in der Mechanish-Technischen Versuchsanstalt ausgeführte Mikroskopische Eisenuntersuchungen. Mittheil. aus den kön. Technischen Versuchsanstalten, Vol. X., p. 57, 1892.
35. A. Martens: Die Mikroskopische Untersuchung der Metalle. Glaser's Annalen, Vol. XXX., p. 201, 1892.
36. H. Behrens: Revue Générale des Sciences Pures et Appliquées. Vol. III., p. 343, May 15, 1892.
37. A. Martens: Das Gefüge der Schienenköpfe. Stahl und Eisen, Vol. XII., p. 406, May 1, 1892.
38. H. Wedding: Das Gefüge der Schienenköpfe. Stahl und Eisen, Vol. XII., p. 478, May 15, 1892.
39. A. Martens: Das Gefüge der Schienenköpfe. Stahl und Eisen, Vol. XII., p. 530, June 1, 1892.
40. G. Guillemin: Analyse Micrographique des Alliages. Comptes rendus de l'Academie des Sciences, Vol. CXV., p. 232, July 25, 1892.
41. Tetskichi Mukai: Studien über Chemisch-Analytische und Mikroskopische Untersuchung des Manganstahls. Freiberg, 1892.
42. F. Osmond: Microscopic Metallography. Trans. of the American Institute of Mining Engineers, Vol. XXII., 1894.
43. A. Martens: The Micro-structure of Ingot Iron in Cast Ingots. Trans. of the American Institute of Mining Engineers, Vol. XXII., 1894.

44. A. Sauveur: Micro-structure of Steel. Trans. of the American Institute of Mining Engineers, Vol. XXII, 1894.

45. H. Behrens: Das Mikroskopische Gefüge der Metalle und Legierungen. Hamburg, 1894.

46. F. Osmond: Méthode générale pour l'Analyse Micrographique des Aciers au Carbone. Société d'Encouragement pour l'Industrie Nationale, Paris, May, 1895.

47. H. M. Howe: The Hardening of Steel. Journal of the Iron and Steel Institute, No. 2, 1895.

48. H. H. Howe and Albert Sauveur: Further Notes on the Hardening of Steel. Journal of the Iron and Steel Institute, No. 1, 1896.

49. Albert Sauveur: The Micro-structure of Steel and the Current Theories of Hardening. Trans. of the American Institute of Mining Engineers, September, 1896.

50. Thomas Andrews: Microscopic Internal Flaws, Inducing Fracture in Steel. Octavo, pp. 52. London: E. & F. Spon. 1896.

The above list is believed to be exhaustive up to 1895, but is incomplete from that time to date.

PLATE I.

Fig. 1. Open hearth steel, normal, carbon 0.08 per cent. Attention is called to the double cell walls plainly shown, which seem to indicate that each cell has its own individual envelope ; observed for the first time, so far as known. Ferrite is white ; pearlyte is dark portion.

Fig. 2. Same as Fig. 1, quenched. Crystalline ferrite, covered with iridescent papillæ.

Fig. 3. Same as Fig. 1, annealed.

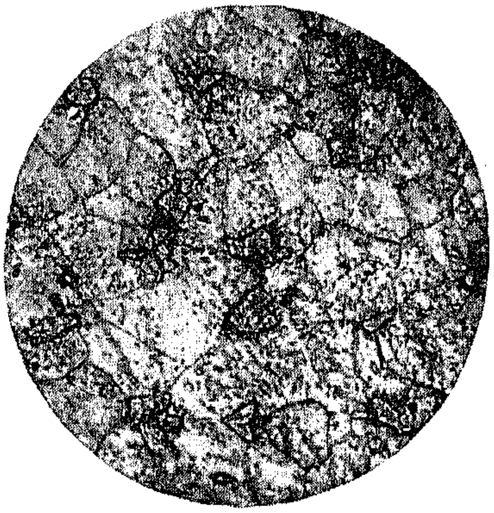
Fig. 4. Open hearth steel, normal, carbon 0.30 per cent. Theoretically should be ferrite 63 per cent. (light), pearlyte 37 per cent. (dark). The negatives are not retouched, as the polishing scratches shown will bear witness.

Fig. 5. Same as Fig. 4, supposed to be quenched, but evidently not heated enough or quenched with sufficient suddenness, since the whole mass should appear as martensite.

Fig. 6. Same as Fig. 4, annealed. Ferrite (light) and pearlyte (dark) crystals apparently with ferrite cell walls.

PLATE I.

1



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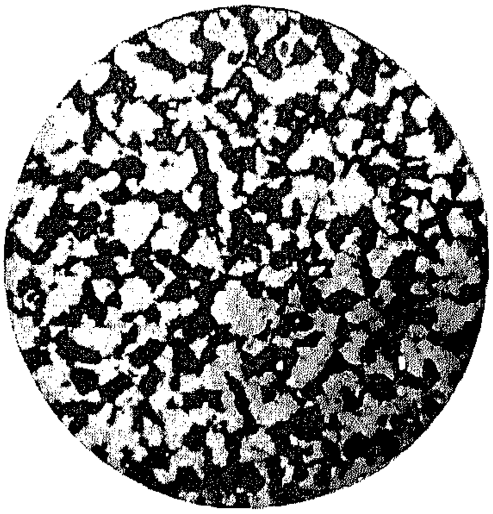

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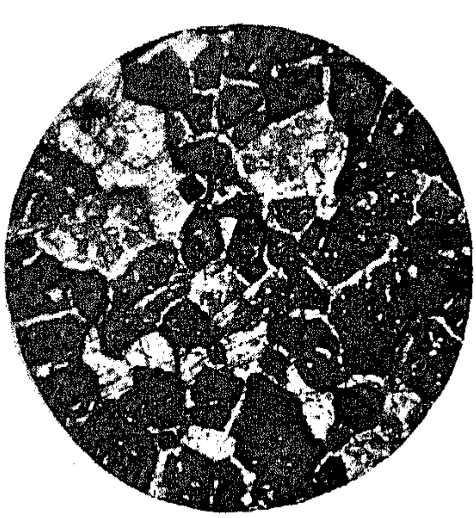

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PLATE II.

Fig 7. Open hearth steel, normal, carbon 0.55 per cent. Theoretically should show ferrite 30 per cent., pearlyte 70 per cent. The darkest part, as shown, is the ferrite after etching, but without polishing on chamois, after which it would appear white. The pearlyte shows lamellar structure in lighter portions.

Fig. 8. Same as Fig. 7, quenched. Whole mass of martensite.

Fig. 9. Same as Fig. 7, annealed. Crystalline structure, but is thought that the specimen cooled too rapidly to allow full crystallisation to be accomplished.

Fig. 10. Crucible steel, normal, carbon 0.90 per cent. Whole mass of lamellar pearlyte. A very fine specimen of pure high grade carefully worked steel. Ingot was hammered before rolling.

Fig. 11. Same as Fig. 10, quenched. Whole mass of martensite, but apparently of finer grain than Fig. 8.

Fig. 12. Crucible steel, normal, carbon 0.97 per cent. Whole mass of pearlyte, with small points of segregated cementite. Lamellæ distinctly shown.

PLATE II.

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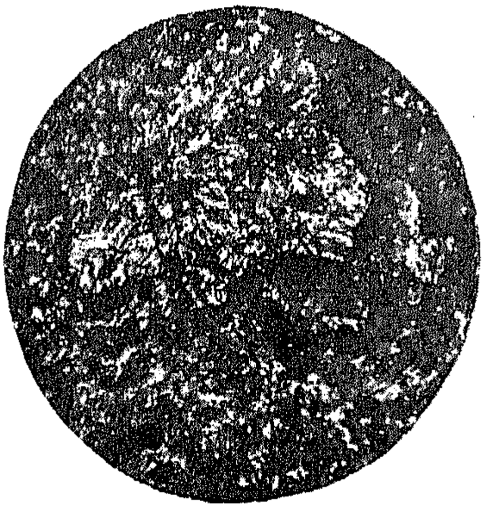

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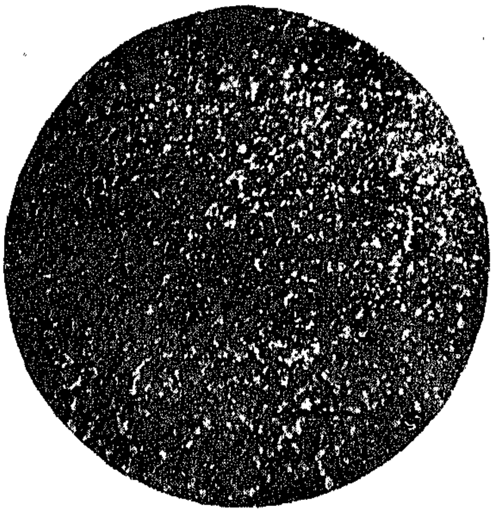

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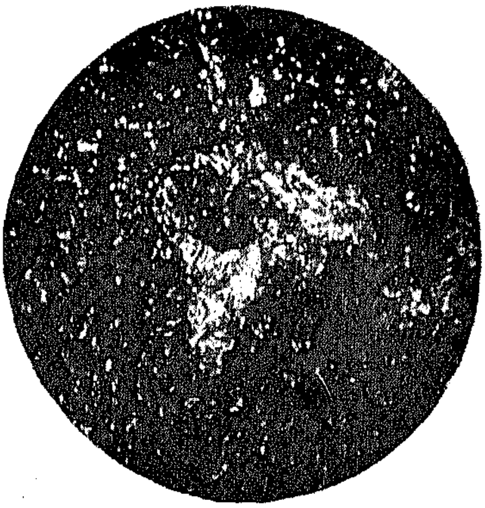

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PLATE III.

Fig. 13. Open hearth steel, normal, carbon 1.00 per cent. Meshes of net pearlyte, outlined with free cementite walls.

Fig. 14. Same as Fig. 13, quenched. Martensite, the free cementite walls of Fig. 13 being partially absorbed by the martensite.

Fig. 15. Open hearth steel, normal; carbon not known, but about 0.15 per cent. A section of rivet after the specifications of a leading railway and, therefore, a typical example of the best class of mild structural steel.

Fig. 16. Longitudinal diagonal section of iron wire from the main cables of old Niagara suspension railway bridge. No carbon present and dark spots are streaks of so-called "cinder." After forty years use this wire shows an average of 95,000 pounds tensile strength per square inch. Manufactured in England.

Fig. 17. Cross section of same as Fig. 16.

Fig 18. Longitudinal section of same as Fig. 16.

PLATE III.

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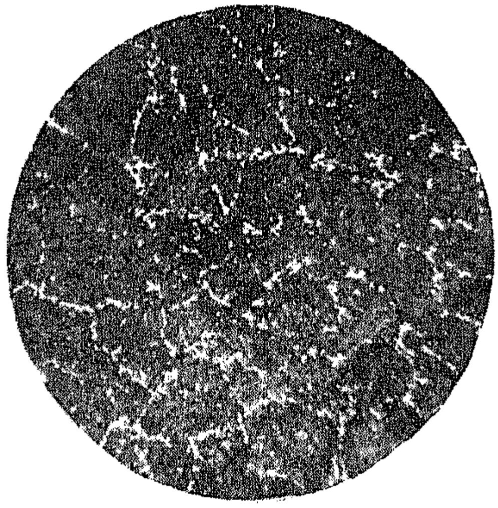

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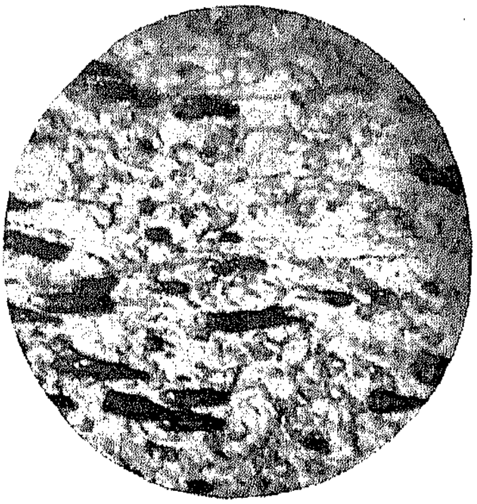

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