CHAPTER VII.

DETAILED EXAMINATION OF SELECTED STEELS.

My experiments have been specially carried out on five samples of the purest classes of steel made industrially, containing varying amounts of carbon, other foreign elements being in small and very slightly differing proportions.

The first of these samples is exceptionally low in carbon. The second represents very soft steels such as are used for plates, etc.; the third, medium hard steels of the composition used for cannon, plates, rails, etc.; the fourth, tool steels; the fifth is a blister steel obtained by cementing extra-soft steel.

The following are their chemical analyses:—

Chemical Composition.	1	2	3	4	5
		Process of Manufacture.			
		Extra-soft Open Hearth.	Medium-hard Open Hearth.	Hard Crucible.	Cemented Steel.
	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.
Carbon . .	0·02	0·14	0·45	1·24	1·57
Silicon . .	0·05	0·045	0·07	0·35	0·023
Sulphur . .	0·02	0·018	0·016	0·012	0·043
Phosphorus .	0·03	0·01	0·036	0·017	0·044
Manganese .	0·25	0·19	0·35	Strong traces.	0·50

In order to follow the transformations of the structure which take place within the range of the critical points, it was necessary first to determine these points. This having been effected, the results are represented in the following table, in compiling which the abscissæ have been taken as the temperatures, and the ordinates as the time, which the thermometer takes to pass through 1° of the Le Chatelier thermo-electric pyrometer (about 10° C.). The necessary figures are shown for the first four samples in the table under the usual notations. The basis of gradation of the pyrometer is always the solidification of sulphate of potassium at 1015° C.

Samples. — Percentage of Carbon.	Position of Critical Points on Cooling.								
	Ar_3.			Ar_2.			Ar_1.		
	Begins about	Maximum between	Ends about	Begins about	Maximum between	Ends about	Begins about	Maximum between	Ends about
Steel with 0·02	°.	855	°.	760	740–720	700	°.	Failed	°.
,, 0·14	840	825–815	800	750	730–720	700	655	650–640	635
,, 0·45	.	.	.	715	695–685	Confused	650		630
,, 1·24	.	.	.	.	.	.	700	675	650

Whilst heating the metal containing 1·24 per cent. carbon, the thermometer shows a lengthened pause at 705° C.; and the metal with 0·45 per cent., a shorter pause at 710° C.

It should be remembered that the point Ar_1 corresponds to Barrett's recalescence point, and to the transformation of the hardening carbon into annealing carbon; while the points Ar_3 and Ar_2 reveal the allotropic transformations of iron itself in the ferrite. It

was seen from experiments made in 1891, and referred to for the first time in January 1892 in a report to the Commission des Méthodes d'Essai "*Sur la méthode du Refroidissement*"—experiments since confirmed by Professor Curie[1]—that these two transformations of iron are distinct, a point which, from my former researches, I had been unable to decide. In other words, the iron[2] is in the state γ above Ar_3, β between Ar_3 and Ar_2, and α below Ar_2. The work of Dr Ball,[3] and that of Professor Curie, even show the possible existence of a fourth allotropic state δ above 1300°.

With the exception of the cementation steel, all my samples were in the first place forged: the first in pieces 6 by 6 millimetres square under conditions unknown to me, the three others in round bars of 12 to 13 mm. diameter, from which have been cut, in the cold state, sections 7 mm. thick. The forging of these was finished at a dark red heat in such a way as to give, without cold hammering, the finest possible grain. In thus taking forged metals to commence with, I aimed at the destruction and effacement of the original heterogeneity which characterises rough cast metals. The structure of the ingots and the transformations caused by hammering, reheating, and quenching, would demand special studies by themselves, and have already been made the subject of an important memoir presented by Professor Martens to the Chicago Congress.

The sections prepared in the manner just described were subjected, under definitely fixed conditions, to

[1] Gauthier-Villars et fils, Paris (1895).

[2] Readers are referred to the recent statements of Professor Carpenter and Dr Rosenhain, *Jour. of I. and S. Inst.*, vol. i. 1913, on Beta iron.—[EDITOR.]

[3] *Jour. of I. and S. Inst.*, part i. p. 85 (1890); and part i. p. 103 (1891).

various heat treatments, and were hardened and annealed. The critical points were afterwards determined, and the polished surfaces were examined under the microscope.[1] As it is necessary to define terms of which the meaning is doubtful, I shall call *reheating* all heating above Ar_1 followed by cooling slow enough not sensibly to displace the transformation points, *whatever may be the time during which the maximum temperature has been maintained*: I shall call *quenching* all cooling quick enough sensibly to displace these same points; and *tempering*, all reheating, after quenching, to a temperature lower than Ar_1, whatever the rapidity of the subsequent cooling. In all reheating trials, the samples, placed between two pads of tightly pressed asbestos, were put into a tube heated in a Leclercq and Forguinon furnace, the heat having been raised, in the space of about half an hour, to the maximum temperature it was proposed to attain: the gas was then turned off, and the samples left to cool spontaneously in the furnace with the door or lid closed.

With all the samples which had undergone heat treatment, care was taken to remove, with a file or grindstone according to the hardness, a thickness of metal of 1 mm. or more, so as to remove the external decarburised metal.

In all cases micrographic analysis was only carried out on cross-cut sections. Analysis of sections cut longitudinally would equally have an interest of its own, but only for the study of deformations produced at red heat—a study which does not form the principal object of these researches.

[1] The temperatures had been measured by the Le Chatelier pyrometer, the solder being pressed between two plates of the same metal.

After these general explanations, the detailed examination of the steels selected may be described.

I.—STEEL WITH 0·02 PER CENT. CARBON.

This metal is of Swedish manufacture, and contained originally 0·07 per cent. carbon. Sir Robert Hadfield, who was kind enough to furnish me with it, has lowered its percentage of carbon to 0·02 per cent. by prolonged annealing in oxide of iron (the process of making malleable cast-iron). The analysis given in the table was made in Sir Robert's laboratory before annealing. The amount of carbon after annealing is only given with reserve. But as polishing in bas-relief shows no cementite, it is clear there is practically no carbon present.

Reheating and quenching the fine structured iron at different temperatures, including white heat, do not modify the initial structure to any appreciable extent; and the description of any one of the preparations can apply equally to all the others.

Preparatory polishing shows abundant inclusions of scoria.

Polishing on rouged parchment and polish-attack begin to show the network of the grains of ferrite; but the structure is shown better and more perfectly after attack with tincture of iodine (three applications of one drop each, just to discoloration). The black spots (fig. 136, V × 100) are the scoriæ. The ferrite is resolved into large grains, very irregular in size and shape, which may split up into more regular grains, smaller and not so clear. The grains are etched very unequally, according to their crystalline orientation.

It seems to follow from this first examination that

the structure of pure iron produced by a prolonged heating at about 900° would remain almost independent of the temperature of heating and the rapidity of cooling, but such a conclusion would be premature. As already stated, we want rather to differentiate the constituents of steel and to determine their mutual relations, than to give a description of any one of

Fig. 136.—IRON (0·02 per cent. carbon). Etched with iodine. V × 100 diameters.

them taken separately. All that can be said is that the transformations of structure of pure iron are slower than those of carburised irons. By varying simultaneously the time and temperature of annealing, Stead has obtained important results (see *Jour. of I. and S. Inst.*, part i. pp. 145–149 (1898), and part ii. pp. 137–154). Elsewhere M. Cartaud and I have written lengthy articles on the Crystallography of Iron.[1]

[1] *Annales des Mines*, Aug. 1900; *Jour. of I. and S. Inst.*, 1906, No. iii. p. 444.

II.—STEEL WITH 0·14 PER CENT. CARBON.

A. Forged Metal.—After polishing in bas-relief (fig. 137, V × 100), numerous scattered threads, more or less distorted, project and appear relatively dark in the photograph, taken a little above the mean focussing point. These are the strips of pearlite or sorbite, or

Fig. 137.—Forged Steel (0·14 per cent. carbon). Polished in bas-relief. V × 100 diameters.

even single layers of cementite. Professor Wedding developed them by heating till they assume the colours of tempering, and described them under the name of crystalline iron.[1] The remainder is ferrite. By prolonged polishing on parchment with sulphate of lime and water, little by little a network of seams is developed, cutting out the ferrite in contiguous polyhedric grains, leaving in slight relief the strips

[1] *Jour. of I. and S. Inst.*, p. 187 (1885).

of pearlite which are almost always external (fig. 138, V × 100), and rarely the centre of the grains. The grains themselves do not form a specular surface: certain of them are slightly in intaglio, others slightly in relief, as the relative variation in definition with the position of the objective proves, which is easily detected by raising or depressing the object-glass of the microscope.

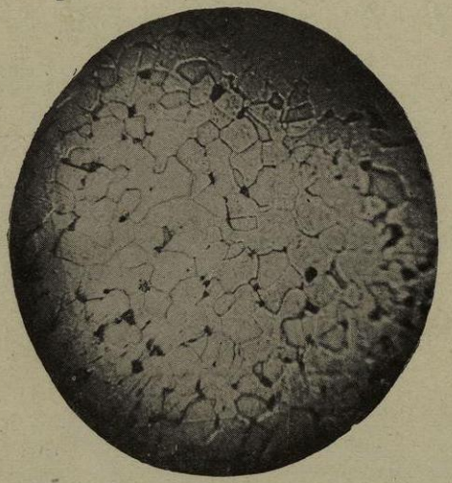

Fig. 138.—Forged Steel (0·14 per cent. carbon). Polished with calcium sulphate and water. V × 100 diameters.

After attack by four successive applications of tincture of iodine (fig. 139, V × 100), some grains take a yellow or brown colour. The pearlite forms dark spots. After another attack of 12 seconds by 20 per cent. mixture of nitric acid in water, some deeply engraved grains are mixed with the remaining bright grains. The pearlite is no longer clearly distinguished from enlarged seams. In examining a fixed part of the preparation, and following it grain by grain in the

different phases of the analysis, as much by photographs as by superposed drawings in the camera, I have not found regular relations between the reliefs

Fig. 139.—Carbon 0·14 per cent. Etched with tincture of iodine. V × 100 diameters.

obtained by polishing, the colorations by iodine, and the etching by nitric acid.

The crystalline orientation of the grains with relation to the surface is not then the sole cause of the

observed facts. We may still suspect an unequal distribution of foreign bodies in the different grains, and also a difference in compactness in relation to the movements of the occluded gases.

It will be noticed that the method of analysis applied has allowed us to detect exactly, in the structure of very soft steel, the joints and filaments

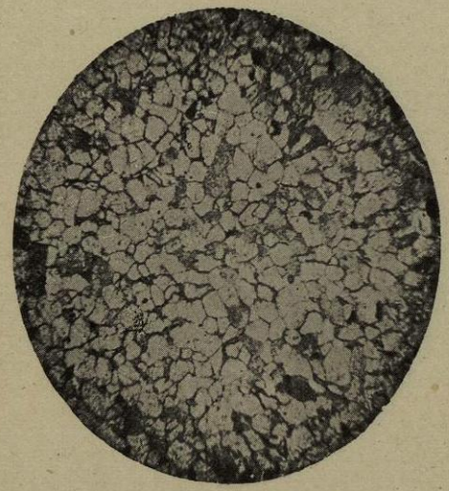

Fig. 140.—Carbon 0·14 per cent. Reheated to 750° C. Etched with 20 per cent. nitric acid. V × 100 diameters.

of pearlite which the "attack," particularly with rather strong nitric acid, tended to confuse under a uniformly black appearance. The distinction once made, the use of 20 per cent. nitric acid is very convenient for the rapid determination of the mean size of the grains on quickly polished specimens.

B. Influence of Reheating.—In proportion as the temperature of heating is increased, other conditions being equal, the polyhedrons of ferrite enlarge, slowly

at first, while maintaining their shape. Above 1000° they become irregular, and tend to lengthen into groups of juxtaposed bands. The pearlite generally remains exterior to the arrangement of ferrite, and stratifies in seams.

For a given steel, the sizes and shapes of the grains are characteristic of the temperature of annealing with

Fig. 141.—Carbon 0·14 per cent. Reheated to 1015° C. Etched with 20 per cent. nitric acid. V × 100 diameters.

practically sufficient precision. The three figures—figs. 140, 141, and 142—represent, with the usual enlargement of 100 diameters, and after attack by 20 per cent. nitric acid, the same forged steel reheated to 750°, reheated to 1015°, and reheated to 1330° respectively.

C. Influence of Quenching.—One sample was heated to 960°, a temperature at which the point Ac_3 is certainly passed; cooled slowly to 670°, that is to

say, between Ar_1 and Ar_2; and then quenched in water at 15°. The polishing on rouged parchment leaves in relief numerous inclusions rather thicker, less clear, and not so well shaped as those of the annealed steel (fig. 143, V × 100). Analysis shows that these inclusions are now no longer of pearlite, as in the annealed steel; they are not scratched by a sewing-needle: they consist of martensite.

Fig. 142.—Carbon 0·14 per cent. Reheated to 1330° C. Etched with 20 per cent. nitric acid. V × 100 diameters.

Iodine or nitric acid would show that the remainder is ferrite in polyhedric grains, exactly similar to that of the slowly cooled metal starting at the same temperature.

A second sample was heated like the first to 960°, and quenched at 770°—that is to say, between the points Ar_2 and Ar_3. The hard grains projected by polishing in bas-relief, distinguished by being coloured slightly yellow by polish-attack (fig. 144, V × 100),

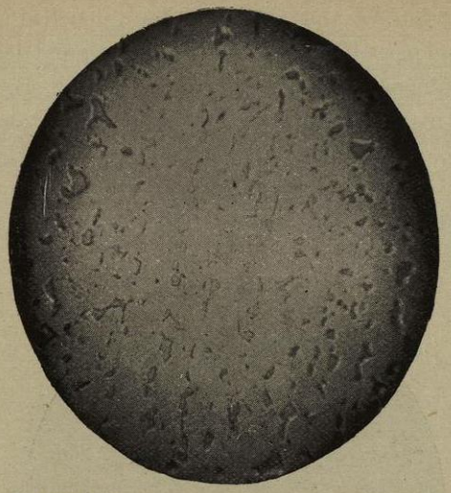

Fig. 143.—Carbon 0·14 per cent. Reheated to 960° C., cooled to 670°. and quenched in water at 15° C. Polished on rouged parchment. V × 100 diameters.

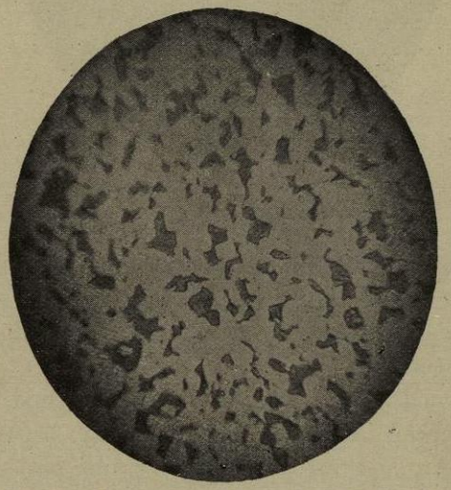

Fig. 144.—Carbon 0·14 per cent. Reheated to 960° C., cooled to 770°, and quenched in water at 15° C. Polish-attack. V × 100 diameters.

are much larger than in the preceding sample; but their hardness hardly exceeds that of fluor spar, because the carbon in them is more diluted. If, after this polish-attack, or after attack by tincture of iodine, we examine them under greater magnification, we see that they form parallel needles which liquorice infusion has etched, and which iodine detaches clearly on a

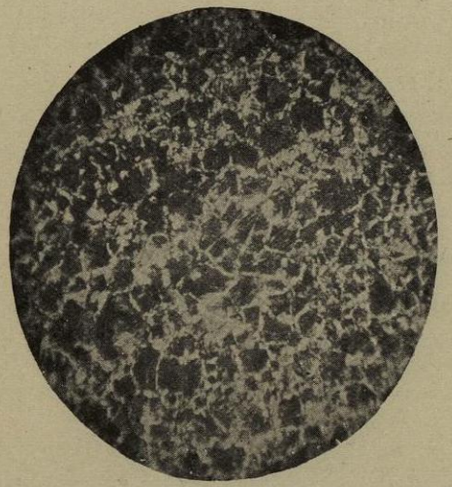

Fig. 145.—Carbon 0·14 per cent. Reheated to 1000° C., and quenched in water at 15° C. Polish-attack. Iodine or nitric acid. V × 100 diameters.

browner ground. Two groups of needles frequently cross in the same region. These are the characteristics of martensite. Strips of troostite may be also found. As for the principal soft mass, it is still ferrite in grains. The same preparation acted upon by 20 per cent. nitric acid hardly distinguishes it from the annealed metal.

A third sample was also heated to 960°, and quenched at 820°—that is to say, during the course of the transformation Ar_3. Its structure is inter-

mediate between that of the preceding and that of the following, and needs no special description.

The fourth disc was heated and quenched at 1000° (that is, above Ar_3), in water at 15°. The ferrite is still scanty: it shows, after polishing on rouged parchment, a cellular network in intaglio, of which the nuclei are coloured by iodine or nitric

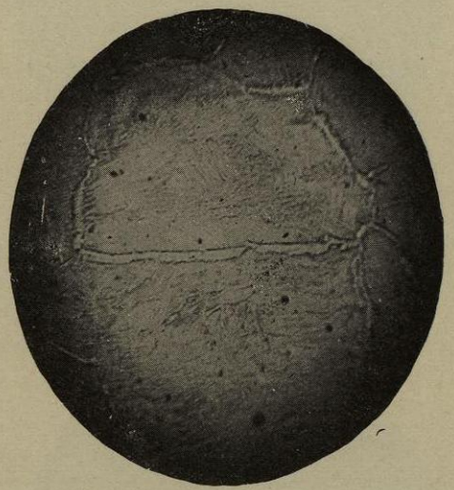

Fig. 146.—Carbon 0·14 per cent. Reheated to 1340° C., and quenched in water at 15° C. Polished in bas-relief. V × 100 diameters.

acid (fig. 145, V × 100). The relief of these nuclei of martensite is feeble, for their mineralogical hardness does not much exceed that of iron. Examination with greater enlargement, after polish-attack, shows that ferrite is resolved into long grains, to which are bound the soft needles of martensite.

Lastly, a fifth disc was heated and quenched at 1340°, a little above the critical point discovered by Dr Ball. The structure appears well on polishing in

bas-relief. Ferrite, properly so-called, forms no more than a thin envelope round large size polygons (fig. 146, V × 100). The interior of these polygons is martensite with its crystalline forms remarkably developed, but martensite passing to ferrite on account of its small quantity of carbon. The view in an oblique light shows it sometimes dark, sometimes

Fig. 147.—Carbon 0·14 per cent. Reheated to 1340° C., and quenched in water at 15° C. Etched with 20 per cent. nitric acid. Oblique illumination. × 100 diameters.

brightly illuminated, according as the inclined faces of parallel needles reflect the incident rays through or behind the objective, and in turning the preparation under the microscope the same polygon is seen to light up and to go dark in turns, like a lighthouse with a revolving light. Attack with 20 per cent. nitric acid (fig. 147, oblique light, × 100) clearly shows, in the same place, the arrangement of elements

following the three sides of a triangle, and the shapes of crystallites of the cubic system.

If the respective areas occupied by the martensite and ferrite are measured on the five samples, we get the following approximate results, viz.:—

Temperature at which the metal is quenched in water at 15° C.	Per cent.	
	Martensite.	Ferrite.
Quenched at 670° between $Ar_1 - Ar_2$.	14	86
,, 770 ,, $Ar_2 - Ar_3$.	24	76
,, 820 middle of Ar_3.	46	54
,, 1000	61	39
,, 1340	90	10

These figures give an idea of the structural composition of steel at the initial temperature of quenching. But it must not be forgotten that the concentration of martensite can take place during quenching in water at 15°, and further experiments should be made to ascertain the effect, on the steel, of much more rapid quenching.

III.—STEEL WITH 0·45 PER CENT. CARBON.

A. Forged Metal.—Analogous steels have been several times described by Sorby, Martens, Wedding, and by the author. They consist of a mixture of ferrite and pearlite, the latter more or less approaching sorbite according to the conditions of cooling. It is by polish-attack that the best preparations are obtained (fig. 148, V × 1000). The pearlite conforms

to the general description which has been given of it. Attack resolves the ferrite into grains.

Fig. 148.—Forged Steel. Carbon 0·45 per cent. Polish-attack. V × 1000 diameters.

B. Influence of Reheating.—I shall pass rapidly over this question, which I have treated elsewhere. According as the temperature of reheating is raised,

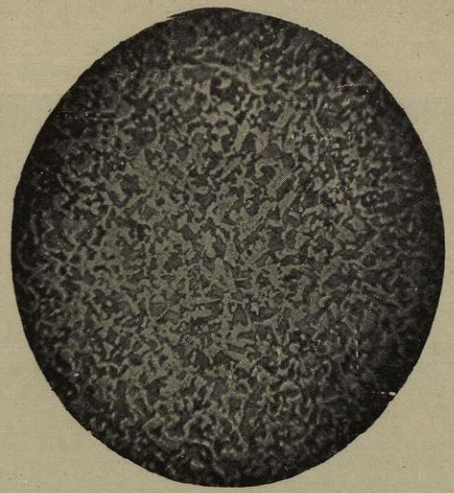

Fig. 149.—Carbon 0·45 per cent. Reheated to 750° C. Polish-attack. V × 100 diameters.

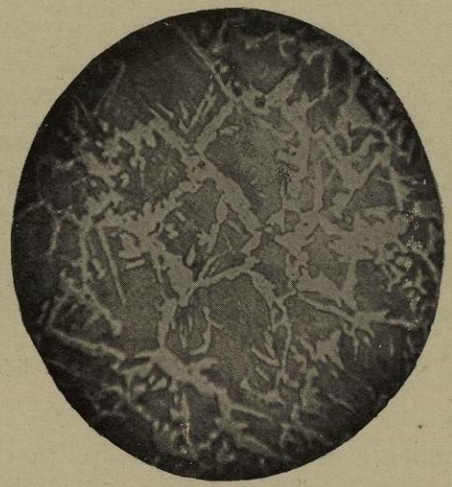

Fig. 150.—Carbon 0·45 per cent. Reheated to 1015° C. V × 100 diameters.

the pearlite forms increasingly regular polyhedrons, which the ferrite envelops with a network more and more perfect, and into the interior of which it intrudes parallel ramifications.

Fig. 149 (V × 100) represents the metal simply forged. After annealing at 1015° (fig. 150, V × 100),

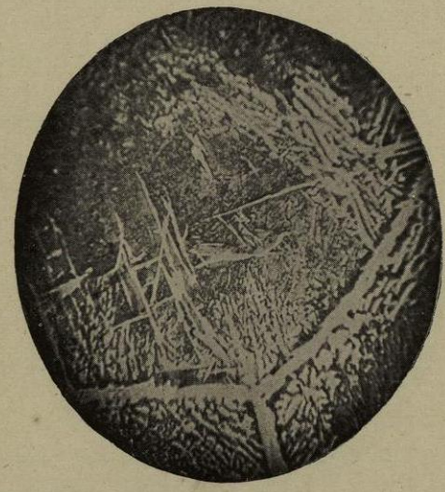

Fig. 151.—Carbon 0·45 per cent. Reheated to 1390° C. V × 100 diameters.

the structure becomes cellular, ferrite forming the envelopes.

After reheating to 1390°, the metal is what is usually described as "burnt." The polyhedrons have become so large that fig. 151, magnified 100 diameters, taken towards the common apex of 3 polygons, does not show a single entire grain. To give an idea of the whole, a photograph (fig. 152) of the feeble enlargement of 20 diameters is necessary. Here the light is oblique, and, accordingly, the