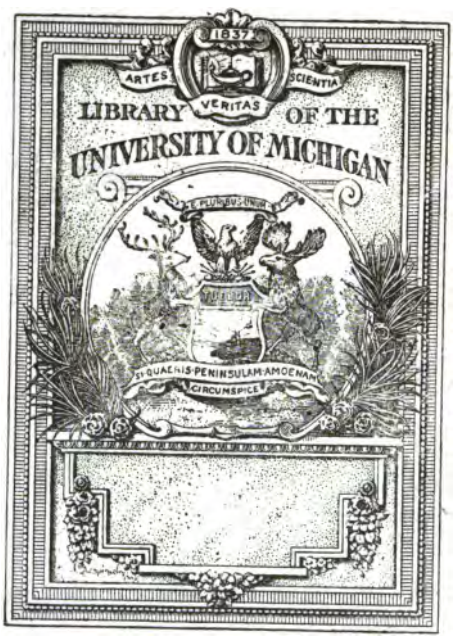
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MICROSCOPIC ANALYSIS OF METALS

BY

FLORIS OSMOND,

CIVIL ENGINEER, PARIS.

EDITED BY

J. E. STEAD, F.R.S., F.I.C.,

MIDDLESBROUGH.

WITH ONE HUNDRED PHOTOGRAPHIC ILLUSTRATIONS AND TWO FOLDING DIAGRAMS.

LONDON:

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1904.

PREFACE

THE accuracy of Mons. Osmond's metallographic work has received universal recognition, as is amply proved by the writings of metallographers in Europe and America.

The clear, charming, and accurate micro-photographs with which he illustrates his writings must be accepted as holding a premier position; and the method he has invented and described as the "polish attack" has placed in the hands of metallographers a most perfect means of revealing some of the hidden and complicated structures of metals and alloys.

Before asking Mons. Osmond to publish an English translation of two of his charming papers on Microscopic Assay, the editor has repeated nearly every one of the Assays given in his work on the "Micrographic Analysis of Carbon Steels," and has in every instance confirmed his observations.

The first paper on "Metallography considered as a Method of Assay" was read before the International Association for the Testing of Materials in 1897 at the Stockholm Congress. It was printed in both French and German, but not in English, owing to the feeble support given to the Association, and the apparent want of interest in its objects by English-speaking races.

That paper is a suitable introduction to the researches of the author, and is also a fitting introduction to the second work on "The Micrographic Analysis of Carbon Steels."

The latter is a second edition of one previously published by the Société d'Encouragement pour l'Industrie Nationale in 1895, but is revised, and much of the author's more recent work included. This second edition was published in 1901 by the same Society.

The author has not only authorised the publication of an English translation, but has contributed a final chapter describing the micro- and photographic apparatus he uses and the method of using it, and has also written a special appendix on Austenite.

It only required this important addition to make the work complete, and it can now be said with justice that there has been no work previously published in English calculated to be so useful to the student in metallographic research. It is, in fact, a standard work on Metallography.

Its unique value is due to the great accuracy of the author's experimental observations. The careful and logical reasoning and the hypothetical conclusions arrived at have their charm, and no one can carefully read them without feeling that they have been made by a master mind, whose one aim is to arrive at the truth.

It is true that some of the hypothetical conclusions have been disputed, and that there are many who do

not accept the author's generalisations regarding the allotropic conditions in iron and steel; it is a fact, however, that they have not been disproved.

As very many metallographers and metallurgists have received them with favour, it is most desirable that English readers should have the data on which they were based, so as to be in a position to form their own opinions.

The scientific use the author has made of his imagination, the constant mental effort to connect cause and effect, has stimulated metallographic research in all intelligent and progressive nations.

Both Mons. Osmond and the editor acknowledge with gratitude the permission for the publication in English of these papers by the Societies before whom they were read.

J. E. STEAD,
Editor.

January 1904.

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PART I.

METALLOGRAPHY

CONSIDERED AS A METHOD OF ASSAY.

DEFINITION.—Metallography, in the widest sense of the word, is the description of the structure of metals and of their alloys. It is not confined to the use of some single instrument—the microscope, for example; although the use of this instrument demands some special training, and for this reason tends to create specialisation and specialists. In reality, in the examination of metals we begin by using our eyes. When our eyes have shown us all that can be seen, we reinforce them with glasses of gradually increasing power, until at enlargements of about 2000 diameters we are arrested by ultra-microscopic mysteries. But the eyes, and the optical instruments which extend their power, are themselves but incomplete means of investigation. They make, so to speak, a preliminary examination; the indications, which the visible characteristics

—viz., shape, colour, or brilliancy — furnish, must be checked by chemical analysis, micro-chemistry, and crystallography, by the determination of physical or mechanical constants: in fine, by every means which permit the differentiation and identification of the substances, so far as these means, unfortunately restricted, are applicable to the subject under consideration.

Comparison with other Sciences.—To better understand the aim and the processes of metallography, we may institute a comparison with other sciences.

Anatomy, for instance, has recognised in the bodies of animals the presence of a certain number of organs.

If we apply to one of these organs—a bone, for example—processes of *ultimate chemical analysis*, we find phosphorus, calcium, magnesium, oxygen, carbon, hydrogen, nitrogen, etc., and we estimate their proportions by weight. We have here information of a certain value. However, the elementary bodies found by chemistry, when placed together in free contact in a flask in the desired proportions, would evidently give us no idea of what a bone really is.

By *proximate analysis* we take a step further and learn how simple bodies combine among themselves to form phosphate and carbonate of magnesium, gelatine, fats, etc. But the fortuitous mixture of these compounds, admitting that we knew them all and that we put them together in the requisite proportions, still does not make a bone. We must have recourse to *histology* to define their structural forms and their respective topographical positions.

Thus we arrive at a precise notion of the organ studied. But the bone, the architecture of which we have discovered, still remains inanimate. It still remains for us to find how the cells, of which we have discovered only the shape and dimensions, assimilate their nourishment or eliminate their excreta—in a word, how they live ; and we find ourselves in the presence of still another science—*biology*. Finally, the vital processes do not always follow their normal course ; they are disturbed by the introduction of extraneous factors, organic or inorganic, microbes or poisons ; the organs may be diseased, and *pathology* is occupied with the causes and effects of these diseases.

The history of the metals and of their alloys is not without analogy with that of living organisms. Fig. 1, which represents one of the possible distributions of the carbide Fe_3C in steel, curiously recalls, at the first glance, a colony of bacilli and cocci. The similarity, in truth, is here quite accidental and superficial. But there are also more profound analogies. Our metals are neither simple bodies nor are they inert.

The ultimate chemical analysis of an alloy does not tell us how the elements present are combined among themselves, or whether they form isolated definite compounds or solutions, homogeneous or not. The proximate analysis which, in addition, is not often possible in the present state of chemistry, does not tell us how the constituents, supposed to be chemically separated, are individually organised or topographically distributed, as compared one with another. Histological

organisation and anatomical distribution, supposing they are known, do not tell us what modifications the actual

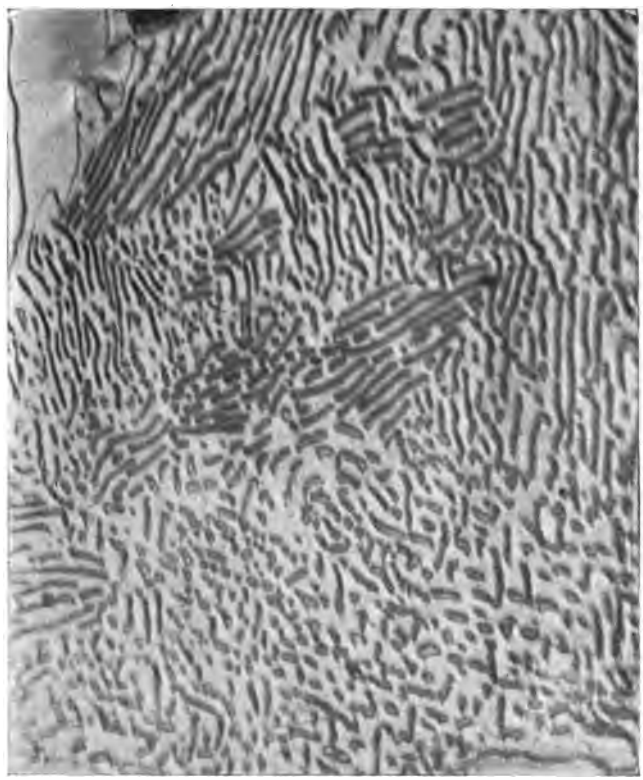


Fig. 1.—CEMENTATION STEEL.]- Polish-attack. * V × 1200 diameters.

condition would undergo under the influence of certain changes of temperature or pressure.

* V = Vertical illumination.

Finally, these normal modifications, supposing them to be known in their turn, might be considerably disturbed by an infinitesimal quantity of some foreign impurity, sometimes inevitable, and the very presence of which is, at times, unsuspected.

SUBDIVISIONS OF METALLOGRAPHY.

Thus we are naturally led to establish in the study of metals several subdivisions quite analogous to those of medical science, and to speak of metallography as **anatomical** (histologic), **biological**, and **pathological**.

The first distinguishes and defines the different constituents of which an alloy may consist by observing their optical characteristics (*colour, brilliancy*), their chemical characteristics (*resistance to reagents, formation of patinas*), or their mechanical characteristics (*resistance to wear, to scarification*), and describes their forms, crystalline or otherwise, measures their absolute or relative dimensions, and examines the planes of weakness (*joints or cleavages*) which separate one from the other, or traverse each of them separately.

The second subdivision is occupied with determining how the composition, form, dimensions, and relations of the different constituents, determined in a fixed state in a given sample, are connected with possible conditions of calorific or mechanical treatment which the alloy under consideration may undergo during the process of its manufacture or employment.

The third subdivision deals with the influence of errors of treatment, and of the presence of impurities

which constitute, as it were, a particular diathesis: at the same time, when effects have once been connected with their causes by a preparatory study, it often allows us to group them together again for the solution of the problems given by practical work.

It remains for these three divisions of metallography to be illustrated by appropriate examples. It will suffice, for this purpose, to make extracts from the important works of scientists who have devoted themselves to the microscopic examination of metals, and who have, for the most part, brought the sum of their experience before the International Association for the Testing of Technical Materials. Hence I shall constantly refer to these works.

ANATOMICAL METALLOGRAPHY.

Bodies chemically homogeneous.—Let us take, as a first example, a simple body—*e.g.*, cast-silver. The photograph (fig. 2) is a reproduction from a design of Professor Behrens,[1] whose work *Mikroskopisches Gefüge der Metalle und Legierungen* is a mine of information. It shows the skeleton of a cube, itself formed of little secondary cubes regularly juxtaposed. In addition, complete cubes, the length of whose sides varies from 0·05 mm. to 2 mm., are also grouped in series, or at least placed in tiers projecting from one corner and from the adjacent portions. It is easy to understand that the cleavages of these cubes are natural surfaces of fracture.

Let us now examine a hammered sample of ingot iron.

[1] Fig. 1, p. 40.

It contains C = 0·125 ; Si = 0·03 ; S = 0·026 ; P = 0·019 ; Mn = 0·31. It is almost pure iron. With the exception of the filaments of carbide and of some small grains of slag, polishing on damp parchment with a little calcium sulphate, followed or not by an attack with nitric acid or tincture of iodine, divides the metal into

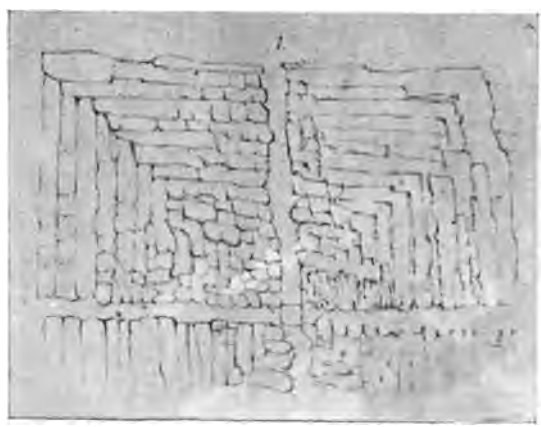


Fig. 2.—CAST-SILVER (after Behrens). Not attacked. V × 20 diameters.

a network of polygonal (fig. 3) sections of polyhedric grains.

These grains are not, properly speaking, crystals at all, since they have neither plane faces nor geometrically regular forms. They are not, however, all identical; polishing on parchment does not wear them away uniformly ; nitric acid and iodine do not attack them equally. These differences are, at least partially, due to the crystalline arrangement of the mass—an arrange-

ment which varies between neighbouring grains, and yet remains constant throughout an individual grain. To bring out this structure, it is advisable to etch rather deeply with dilute and lukewarm sulphuric acid. The grains, cut in sections parallel to two of their axes, show

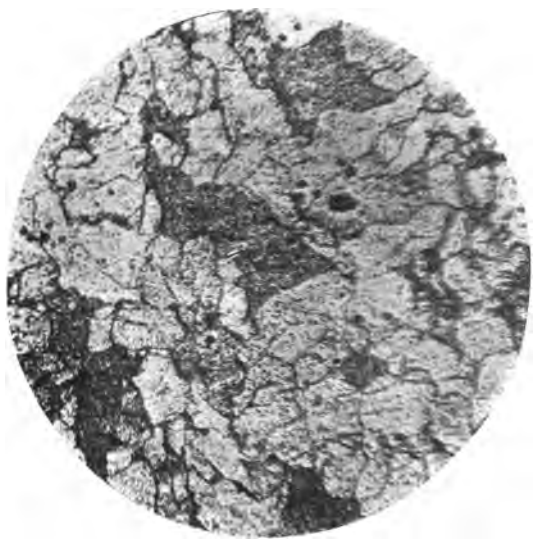


Fig. 3.—FORGED STEEL (0·125 per cent. carbon). Attacked by dilute nitric acid. V × 100 diameters.

little cubes regularly superimposed, very much like those of silver (fig. 4). If the section is parallel only to a single axis, the etched grain seems lamellar (fig. 5); and the exposed structure becomes confused when the section is not parallel to any axis of the grain. We have therefore a double network of weak cleavage planes: one due to those in the mass of the grains themselves, the

other to the demarcation of adjacent grains, variously orientated. These interspaces form a natural locale for the gases which may be set free at the moment of solidification or later, and are ready formed lines along which fissures may open or extend under mechanical

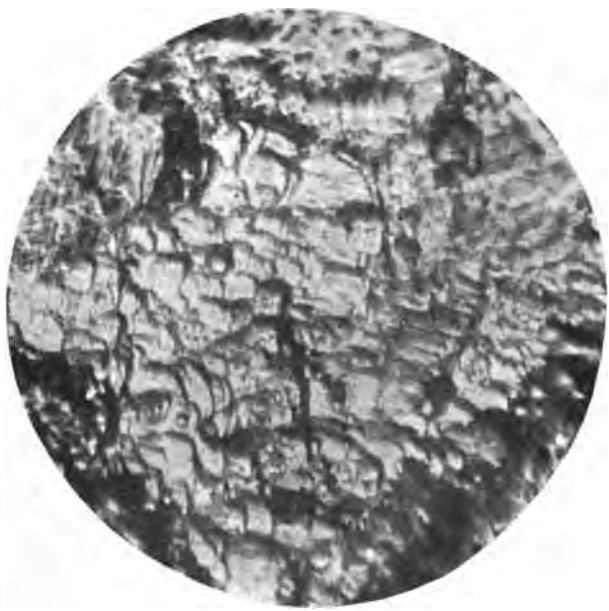


Fig. 4.—FORGED STEEL (0·125 per cent. carbon). Attacked by dilute sulphuric acid. V × 600 diameters.

influence. All the efforts of metallurgists must then tend, if not towards eliminating these sources of weakness, which is impossible, at least towards minimising them by diminishing the proportion of the gases, by checking the liberation of these gases, by preventing

the free play of the crystalline forces, by accelerating cooling, and, in a word, by every means adapted to making the contact surfaces smaller and less discontinuous.

We see that a body, homogeneous in the eyes of the

Fig. 5.—FORGED STEEL (0·125 per cent. carbon). Attacked by dilute sulphuric acid. V × 1000 diameters.

chemist, may be very complex from a structural point of view; but chemically homogeneous bodies are themselves the exception, and we generally have at an early stage to differentiate the chemically distinct constituents.

Bodies chemically heterogeneous.—The simplest case is that in which the constituents do not possess the same colour, which enables us to recognise them at a first inspection of a plain section. Figs. 6 and 7

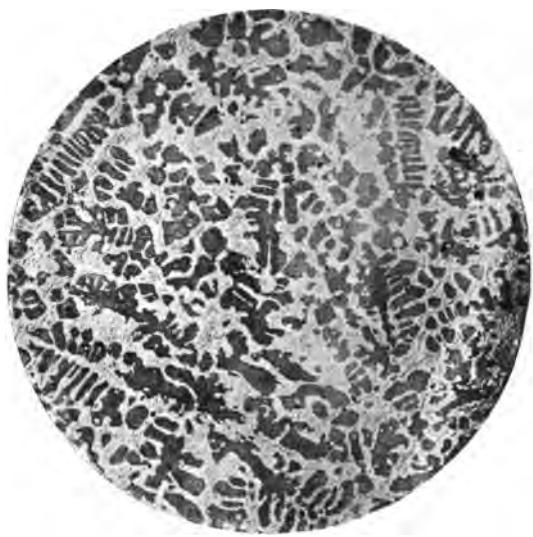


Fig. 6.—GOLD = 85 per cent. ; ALUMINIUM = 15 per cent. Cast. Polished only. V × 1000 diameters.

represent two alloys of gold and aluminium, which contain respectively:

Au = 85 ; Al = 15 (fig. 6).
Au = 54 ; Al = 46 (fig. 7).

Both contain a common constituent which was discovered by Sir William Roberts-Austen, and is of a beautiful purple colour; it is a definite compound of the formula $AuAl_2$, which possesses the very rare property

of having a fusion point above that of the least fusible of its two components. During solidification it first isolates in crystallites, which appear black in the photograph; the spaces are filled up with the material, separating out during the second stage of solidification,

Fig. 7.—GOLD = 54 per cent.; ALUMINIUM = 46 per cent. Cast. Polished only. × 100 diameters.

the proportions and the nature of which vary with the composition of the alloy.

When the constituents, as generally happens in the case of metals, do not possess distinctive colours of their own, we may try to colour them artificially.

Tincture of iodine, for example, will allow us to

distinguish lead, in many of its mixtures, by the formation of its iodide, which is yellow. But the precipitates thus produced by a chemical reaction do not always adhere to the reacting constituent, and very rarely resist the wiping of the surface of the object[1]; it is perhaps best to fix them, as M. Le Chatelier has advised, in a fine covering of gelatine impregnated with a suitable reagent, and applied to the surface which we wish to study. This operation can be performed and followed under the microscope.

A second method consists of forming an adherent patina, too thin to have a colour of its own, but which gives to reflected light a coloration varying with the thickness of the layer. These are the tempering colours. As they do not form on unoxidisable metals, and do not appear simultaneously, other things equal, on metals unequally oxidisable, this process is of frequent application.

In this manner Professor Martens has obtained the beautiful figures of spiegeleisen, which illustrate his first papers,[2] and are one of the first successes of nascent metallography. The metal under consideration is formed of two constituents: a definite carbide $(Fe.Mn)_3C$, and a solution of carbon (or of carbide) in the remainder of the alloy. The definite carbide is relatively slightly oxidisable, and takes, after a certain amount of heating at a certain temperature, a yellow

[1] In such cases when the objects have to be wiped or rubbed with a linen fabric to dry the surfaces.

[2] *Zeitschr. des Ver. Deutscher Ing.*, 1878, Taf. xxiv. and xxv.

colour, for example, whilst the solution has already turned to blue. Colours induced by this method have the great advantage of not being diminished with high enlargements. Mr Wedding,[1] Mr Behrens,[2] and Mr Guillemin[3] have all made frequent use of them; they are a great help in the study of alloys of copper.

When the constituents are of too small dimensions to be discernible even with the microscope, the difference in depth formed by an unequal attack again gives rise to decomposition of reflected light; as the attack becomes deeper, there appear successively (in normal light) yellow, brown, blue, and black colorations, which must not be confounded with the preceding, as they do not show, like them, the presence of a patina. From this we deduce a third method, which extends the power of the microscope to a certain point, but teaches only one fact, viz., that the constituents under consideration are not uniformly acted upon. It is in this manner that we identify sorbite in untempered steels (portions not striated in fig. 8), and troostite in steels tempered half hard at certain temperatures (dark bands of fig. 9).

Colorations, natural or artificial, are not the only resource of anatomical metallography. We can make use of mechanical properties of bodies. M. Behrens has prepared a whole series of needles of regularly varying hardness, with which he scratches the surface

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

[2] *Loc. cit., passim.*

[3] *Commission des Méthodes d'essai des matériaux de construction*, 1st Session, Reports, t. ii. p. 19.

of a polished section [1]; such a needle may scratch one constituent and not another. In the bronzes, the

Fig. 8.—FORGED STEEL (1·24 per cent. carbon). Reheated to 800°. Polish-attack. V × 2000 diameters.

composition of which is represented by the formula

[1] *Loc. cit.*, pp. 21–27.

$CuSn_3$ or $CuSn_2$, Prof. Martens has been able not only to determine, but to measure with the sclerometer, the hardness of each of the structural elements.[1]

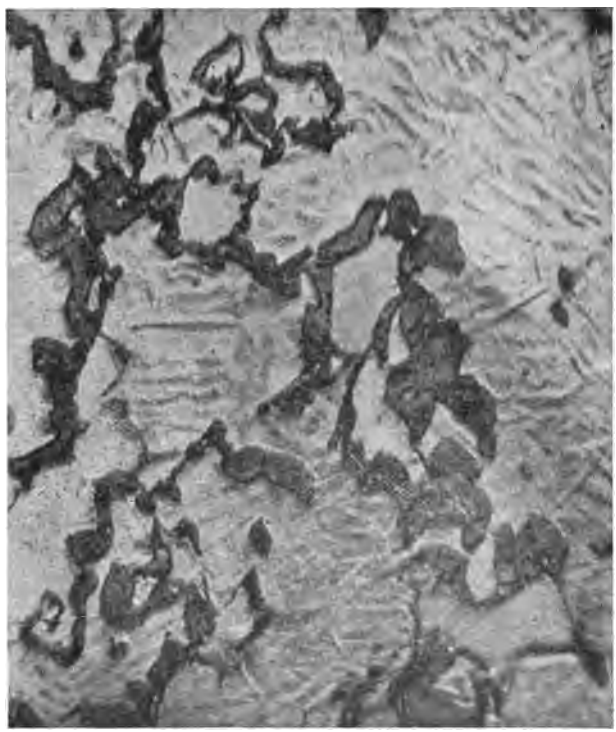


Fig. 9. – FORGED STEEL (0·45 per cent. carbon). Heated, and quenched at 730°. Polish-attack. V × 1000 diameters.

However, the resistance to scratching does not depend only on the nature of the bodies, but also on their

[1] *Mitteil. aus den K. techn. Versuchsanstalten*, t. viii. p. 236.

absolute dimensions; a layer of glass sufficiently thin may be, if not scratched, at least cut, by the finger nail. The method is only applicable, therefore, to fairly coarse structures. But equivalent information may be obtained, in the case of more delicate structures, by rubbing on a soft layer with the help of very fine powders. Fig. 10 is a photograph of some hard steel (1·24 per cent. carbon) forged into a round bar 12 millimetres in diameter, the forging of which has been finished at about a dull red heat. A transverse section has first been polished; then the polishing has been continued on damp parchment sprinkled with a very small quantity of rouge; the iron is hollowed out, and has left in relief the carbide of iron Fe_3C, which, in the sample under consideration, is divided into a multitude of little rounded grains. The grains of carbide appear dark on a clear ground or light on a dark ground, according as the objective is put a little above or below the focussing point. (The photograph was taken in the first of these positions.) The same result would be obtained by attacking the metal by an acid which etches the iron without appreciably dissolving the carbide; but as the iron, in being attacked, has its polish removed in a very irregular manner, the preparations are often lacking in definition. The two methods can again be combined by replacing the water, in the polishing on parchment, by an extract of liquorice-root, which, by the help of repeated rubbing, slightly dissolves the iron without taking away its polish. The device is useful in bringing out clearly that curious

constituent of slowly cooled steels, the discovery of which does so much honour to Dr Sorby, the initiator of microscopic metallography.[1]

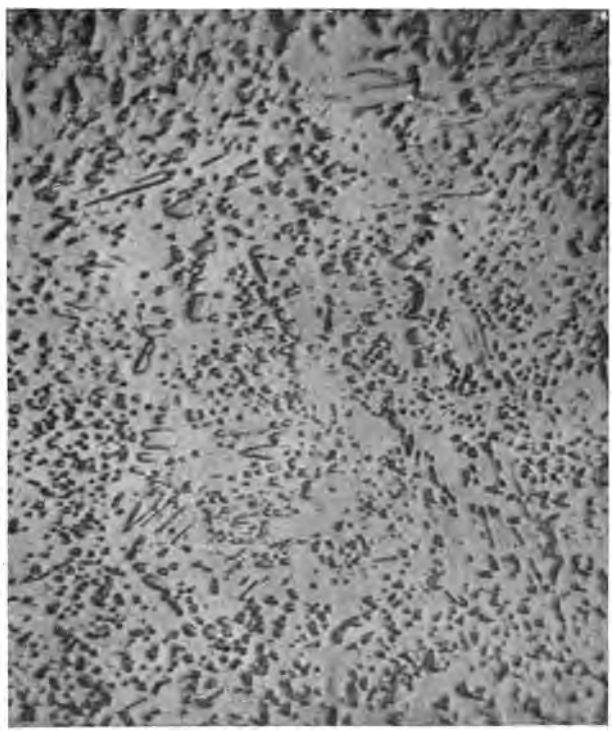


Fig. 10.—FORGED STEEL (1·24 per cent. carbon). Polished in bas-relief. V × 1000 diameters.

This constituent is a mixture of iron and of the carbide Fe_3C in alternate layers, generally curved (fig. 11); the thickness of a pair of layers probably does not

[1] *Jour. of I. and S. Inst.*, 1886, p. 140.

exceed $\frac{1}{1000}$ mm. on an average; it may be much less. The unequal relief of the two components causes, on

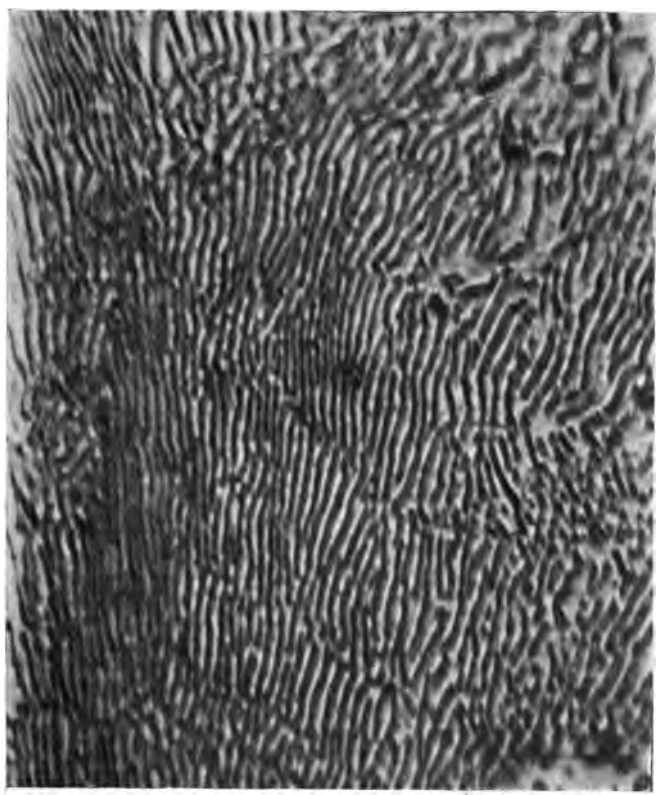


Fig. 11.—CEMENTATION STEEL. Polish-attack. V × 1500 diameters.

account of the decomposition of the light, iridescent reflections which remind one of pearl; whence the

name pearlite, proposed by Mr Howe with the assent of Dr Sorby, and adopted to-day by several writers.

Causes of Error.—The different methods which have just been enumerated, and which serve to distinguish two constituents, do not always give conclusive indications. A chemically homogeneous body may offer a very variable resistance to chemical or mechanical action according to its crystalline arrangement, and according to its greater or smaller degree of compactness. We have seen examples of it above. It is easy enough to mistake for two different constituents two different aspects of a single constituent; and confusions of this kind have certainly been made more than once. The scepticism of certain people in connection with the statements of metallographers has in principle, therefore, some justification; investigators should check themselves by multiplying their experiments, and should only give as facts concordant results obtained from several different methods. But this necessary verification has taken place in certain cases. It has been said, for example, that the aspect of pearlite under the microscope does not indicate the existence of two different bodies, but only the lamellar structure of a single body, with a slighter or deeper attack of the cleavages. The objection in itself is reasonable; but these hard layers of carbide of iron which are seen under the microscope in a section of a steel have been chemically isolated by Müller,[1] by

[1] *Stahl und Eisen*, t. viii. p. 292 (1888).

Abel and Deering,[1] Osmond and Werth,[2] Arnold and Read,[3] and by Mylius, Förster, and Schöne,[4] and it is not possible to-day to deny it an independent existence. As, on the other hand, the carbide Fe_3C, isolated by the best methods from steels cooled very slowly, contains almost the whole of the carbon, it cannot be doubted that the second constituent, ferrite, is almost pure iron. It would no longer be the same after quicker cooling, without it being necessary to go even as far as quenching. Ferrite would then include a certain proportion of carbon under a form chemically discernible, that of hardening carbon. Thus we see that micrography and chemistry constantly help each other. The one indicates the existence of a mechanical mixture, and the other tries to determine its nature; or again, analysis separates certain definite constituents which micrography only locates.

BIOLOGICAL METALLOGRAPHY.

Generalities.—In the preceding section we have seen what methods permit of differentiating the different structures of a chemically homogeneous body, and the different constituents of a chemically heterogeneous mixture. This distinction made, the determination of the forms and dimensions do not present any difficulties, and we are to some extent commencing biological

[1] *Ann. de Chim. et de Phys.*, 5e série, t. xxx. p. 499.
[2] *Ann. des Mines*, 5e série, t. viii. p. 19 (1885).
[3] *Jour. of the Chem. Soc.*, t. lxv. p. 788 (1894).
[4] *Zeitschr. für anorg. Chem.*, t. xiii. p. 38 (1896).

problems—that is to say, connecting the changes of structure with their causes.

The life of a metal or an alloy consists of the transformations, reversible or non-reversible, which such metal or alloy may undergo in the conditions, normal or experimental, of its manufacture or application.

The factors in these transformations are, as for living beings, temperature and pressure; and, as their duration is generally measurable, time should also enter into our reckoning. We shall suppose in the first place that the pressure remains equal to that of the atmosphere.

The changes which may take place in structure and, generally speaking, in all the properties of a metal, when the temperature varies with a given rapidity, are of two kinds: continuous changes and critical changes.

Critical changes are those which correspond to a sudden modification in the relation between a certain property and temperature. On the curves which represent this relation, they are usually indicated by the intersection of two branches.

These critical changes are evidently the most important to be considered, because they divide the whole scale of temperatures into so many intervals in which the body under consideration only undergoes slow, progressive, and, as a result, easily managed modifications. We know also that they indicate a sudden change of energy, which is at the same time the indication and the cause of them.

Knowledge of the sudden changes of energy is there-

fore one of the first requirements in the study of a body.

Every metal undergoes at least two such changes: fusion and volatilisation. These are the changes of physical state.

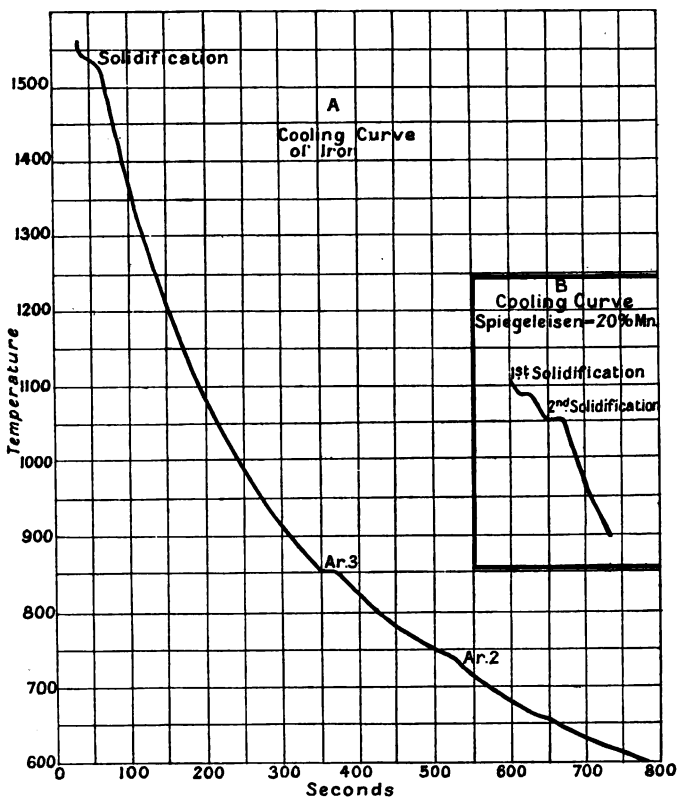
Certain bodies, undoubtedly much more numerous than is generally believed, possess other points of transformation which are called allotropic or isomeric changes according to whether they involve simple bodies (iron, nickel, cobalt, sulphur, etc.), or compound bodies (iodide of silver, nitrate of potassium, etc.).

Iron.—If, for example, pure iron is left to cool, from the temperature of casting, and the temperatures are recorded as functions of time, the curve will indicate three stoppages, or at least three retardations, in the rate of cooling. The first, about 1530°, corresponds to solidification; the second, about 860°, and the third, between 750° and 700°, correspond to two allotropic transformations; and the whole curve presents the general aspect of fig. A.

Spiegeleisen.—If, instead of bodies, simple or compound, but chemically homogeneous, we consider complex bodies, mixtures of several constituents, these mixtures will have as a rule as many points of solidification as the constituents, without speaking of the allotropic or isomeric transformations possible in each constituent.

Spiegeleisen, with 20 per cent. manganese, has two points of solidification: the first about 1085°, the second about 1050° (fig. B). The diagrams of Professor

Martens have also further revealed, in a metal of the same family, two distinct constituents—a definite



Figs. A and B.—Cooling Curve of pure Iron, and 20 per cent. Spiegeleisen.

carbide and a solid solution of the same carbide, more or less dissociated, in an isomorphous mixture of the alloyed metals.

This example carries us back to micrography and helps us to understand its use. Micrography, with the aid of chemistry if needful, will assist us to interpret the curves of cooling, and to give the exact signification to these, at times, mysterious, yet always so important, points of retardation or of stoppage.

Alloys of Silver and Copper.—Let us now pass on from the study of an isolated alloy to that of the group of binary alloys which two given metals may form between them. These studies have lately received fresh impetus from the influence of committees appointed under the auspices of the Institution of Mechanical Engineers in England, and in France of the Société d'Encouragement. If we take for abscissæ the percentage in weight of one of the metals, and for ordinates the temperatures at which the liberations of heat occur during the course of cooling, commencing from the liquid state, we obtain, by joining the corresponding points, the so-called fusibility curves. The aspect of these curves gives very useful information on the chemico-physical relations of the metals considered.

Let us take, for example, a relatively simple case, that of the alloys of silver with copper. The fusibility curve of these alloys was plotted in 1875 by Mr (afterwards Sir William) Roberts-Austen as exactly as was possible at that time[1]; partly corrected by the same observer in 1891 with the aid of the Le Chatelier[2] Pyrometer, and revised lately by Messrs Heycock and

[1] *Proc. Roy. Soc.*, 1875, p. 481.
[2] *Proc. Inst. Mech. Eng.*, p. 555 (1891).

Neville.[1] This curve (fig. C) is therefore known exactly; it is composed of two inclined branches starting from the respective fusion points of copper and silver, and cutting at the temperature 770°, for the composition which corresponds to the formula Ag_3Cu_2. A third branch, horizontal, passes by the point of meeting. This teaches us that copper and silver do not form a

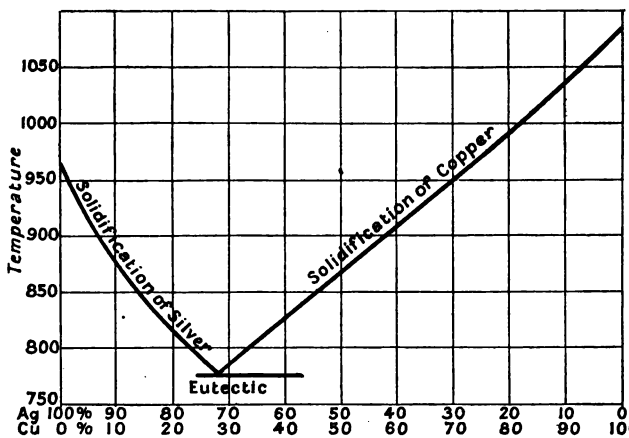


Fig. C.—Equilibrium Curves, Silver and Copper. (Heycock and Neville.)

definite compound or an isomorphous mixture. If the actual theory of solutions established by Mr H. Le Chatelier is correct, one of the branches of the curve corresponds to the commencing solidification of the copper, the other to the commencing solidification of the silver, and the horizontal branch to the simultaneous solidification of the two metals. In other terms,

[1] *Trans. Roy. Soc.*, t. clxxxix. p. 25 (1897).

according to the composition of the alloy, the metal in excess begins to separate out from the solution at a fixed temperature, and continues to be precipitated until the portion remaining liquid contains 72 per cent. silver against 28 per cent. copper; at this point the silver is saturated with copper, the copper is saturated with silver, and the two metals simultaneously solidify at a constant temperature in the form of a mechanical mixture. This is the so-called eutectic alloy, the only one the ingots of which do not liquate, as Levol showed a long time ago.[1]

However, as this alloy of Levol gives by analysis, as exactly as possible, an atomically simple formula (Ag_3Cu_2), and as its surface shows a very clear crystallisation, we hesitate to remove it from the list of definite compounds, and ask if it does not show the theory to be wrong.

Micrography solves the question in a most simple and certain manner. It is enough to prepare a well-polished section on which the two component metals are recognised by their respective colours. We can also, by annealing at a suitable temperature, give to copper a beautiful orange patina, whilst the silver remains white. We thus see that the alloy of Levol is not at all homogeneous. Its structure is identical with that of the pearlite of steel; the yellow metal is distributed, sometimes in fine points, sometimes in straight or curved layers, alternating with the white layers of silver. This alternation after annealing, which causes slight differences of level, gives the known effect

[1] *Ann. de Chim. et de Phys.*, 3e série, t. xxxvi. p. 193 (1852).

of iridescence which the word pearlite recalls. The grains of first solidification can only be suitably brought out under an enlargement of 1000 diameters (fig. 12); they are enveloped in a network of coarser structure, but quite similar (fig. 13). The alloy with 72 per cent.

Fig. 12.—COPPER = 28 per cent.; SILVER = 72 per cent. (Cast.) Heat, oxidation tinted. V × 1000 diameters.

is therefore an eutectic, as the curve of fusibility would lead us to foresee; and the compound Ag_3Cu_2 does not exist.

In the other alloys of the group, accordingly as the amount of silver is above or below 72 per cent., there is

deposited at first either crystallites of silver or crystallites of copper, which increase until the portion remaining fluid attains the desired composition; the eutectic

Fig. 13.—COPPER = 28 per cent.; SILVER = 72 per cent. (Cast.) Heat, oxidation tinted. V × 600 diameters.

solidifies then, at a constant temperature, and surrounds the crystallites, as figs. 14 and 15 show.

M. Charpy has studied several analogous cases.[1]

The samples photographed were melted in the Leclerq and Forquignon furnace, and slowly cooled with the furnace itself. If the cooling is hastened by casting the

[1] *Bull. Soc. d'Encouragement*, 5e série, t. 11, p. 384 (1897).

liquid alloy in a cold metallic ingot mould, the eutectic still shows, after annealing to orange colour, a tendency to the formation of the layers which are indicated by the effects of light; but the layers are not clearly distinguished one from the other, and the general colour

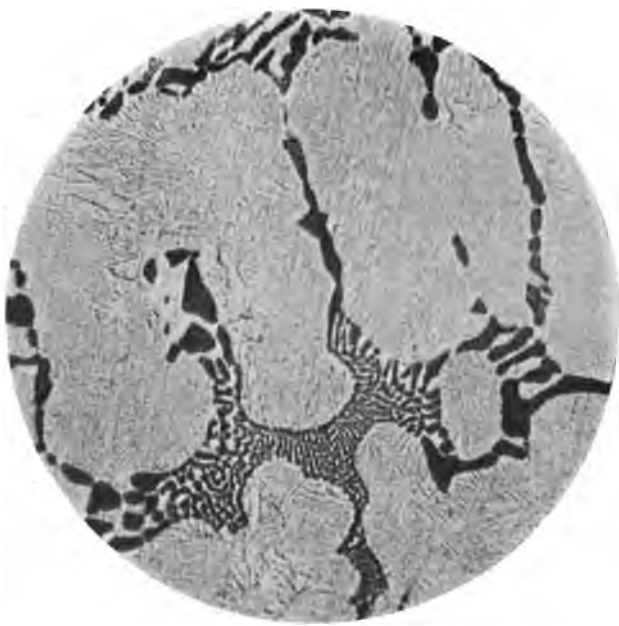


Fig. 14.—COPPER = 15 per cent.; SILVER = 85 per cent. (Cast.) Heat, oxidation tinted. V × 600 diameters.

of the preparation remains nearly uniform with a high enlargement; the two metals have not had time to separate completely.

In fact, the two constituents of the alloys of copper and silver are not pure copper and pure silver, as might

be believed from their respective colours. The copper retains a little silver, and the silver a little copper. We can be assured of this by studying the alloys which contain one of the metals in only a small proportion. The added metal does not commence to appear in

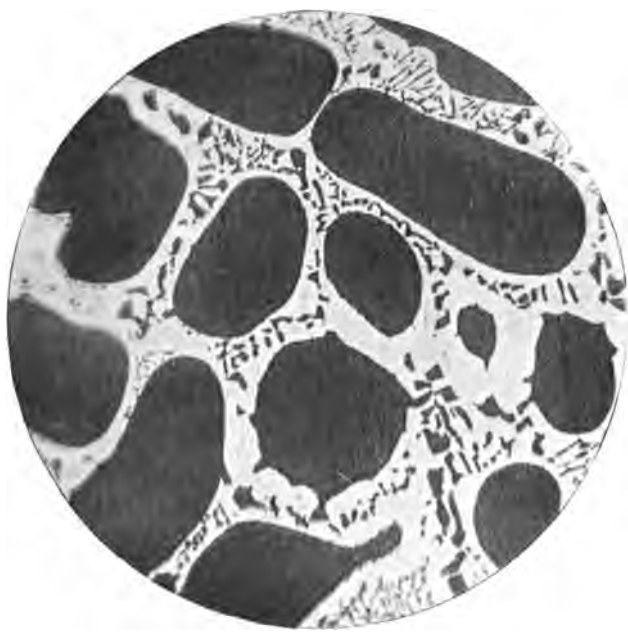


Fig. 15.—COPPER = 65 per cent.; SILVER = 35 per cent. (Cast.) Heat, oxidation tinted. V × 600 diameters.

independent discernible grains, until the proportion of 1 per cent. is exceeded ; and, for a higher amount, which is visible, estimated by the extent of the areas, remains less than the proportion added.

Steels.—This brings us to the conception of *solid*

solution, of which a very conclusive example is furnished by steels.

Let us take for abscissæ the proportion of carbon from 0·0 to 1·6 per cent., and for ordinates the temperatures, as we have already done. For each proportion of carbon, let us point out on the corresponding ordinate the temperatures at which the different

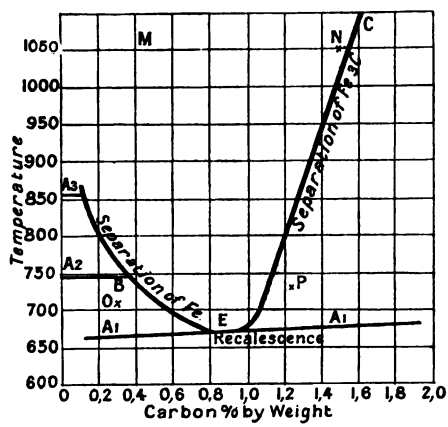


Fig. D.—Equilibrium Curves, Iron and Carbon.

liberations of heat begin: we thus obtain a group of curves (fig. D).

The branches A_3B and A_2B are those of the allotropic transformations of iron—transformations which Bergmann found out, more than a century before their definite discovery, when he said: "*Adeo ut jure dici queat polymorphum ferrum plurium simul metallorum vices sustinere.*"[1] The two branches unite in a single one BE

[1] *De analyse ferri*, p. 4 (after Roberts-Austen, Nature, Nov. 7, 89).

when the proportion of carbon exceeds 0·2 per cent., and this double branch cuts, at the point E, another one inclined in a contrary direction, of which we shall see the significance later. Finally, the horizontal A_1EA_1, passing by the point of intersection, corresponds to the point A of Tschernoff: this is the temperature above which the steel begins to harden by quenching. The passing of the point A is accompanied by a temporary expansion interrupting the regularity of the contraction (Gore),[1] and by a sudden liberation of heat which sometimes can be distinguished by the eye, whence the name of recalescence (Barrett).[2] This phenomenon has been studied from a metallurgical standpoint by the Swedish engineer Brinell[3] in a remarkable work, in which the author has been able to replace the instruments he lacked by a rare power of observation. The point E, where the three branches intersect, has for abscissa, according to Prof. Arnold,[4] the percentage of 0·9 per cent. carbon, or at least, not to be too precise, a percentage between 0·8 per cent. and 1 per cent.

It is seen that the whole of the diagram exactly recalls the diagram of fusibility of the alloys of silver with copper. On the other hand, micrographic research has shown that the branch BE corresponds to a separation of practically pure iron (ferrite), the branch EC to a separation of carbide Fe_3C (cementite), and the horizontal branch to the simultaneous separation of

[1] *Phil. Mag.*, 4e série, t. xxxviii. p. 59 (1869).
[2] *Loc. cit.*, t. xlvi. p. 472 (1873).
[3] *Jernkontorets Ann.*, p. 9 (1885).
[4] *Proc. Inst. Civ. Eng.*, t. cxxiii. p. 127 (1895-6).

the two constituents in alternating layers (pearlite). In other terms, steel above BEC is a homogeneous solid solution of the carbide Fe_3C in an allotropic form

Fig. 16.—FORGED STEEL (0·45 per cent. carbon). Polish-attack. V × 1000 diameters.

of iron: during slow cooling this solution deposits either iron or carbide when it is saturated with one or the other, and thus it reaches a definite composition,

that of the eutectic; and this is always the same whatever may be the starting-point. These changes take place in just the same manner as in liquid solutions,

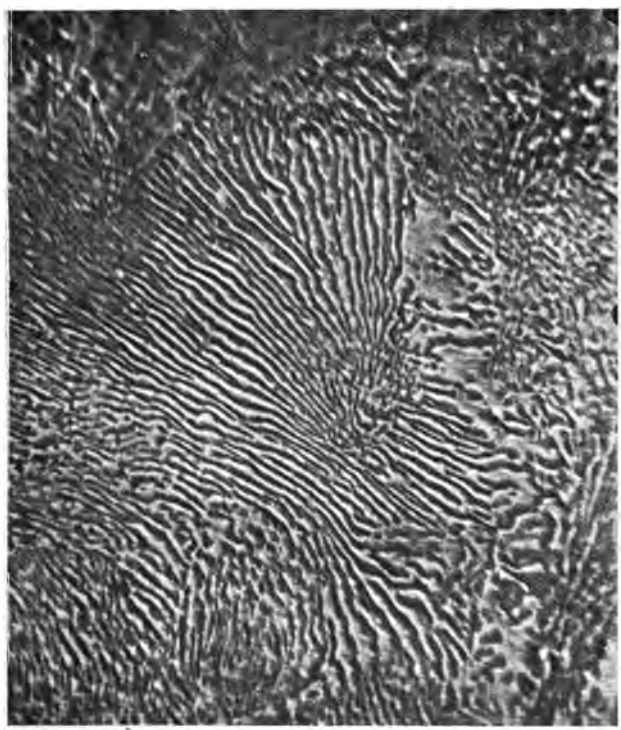


Fig. 17.—FORGED STEEL (1·00 per cent. carbon). Reheated to 800°. Polished in bas-relief. V × 1000 diameters.

and the allotropic transformation plays the same part as solidification. Slowly cooled steels then are formed of a mixture of ferrite and pearlite (fig. 16), of pure

pearlite (fig. 17), or a mixture of pearlite and cementite (fig. 18), according as the proportion of carbon is inferior, nearly equal, or superior to the proportion in

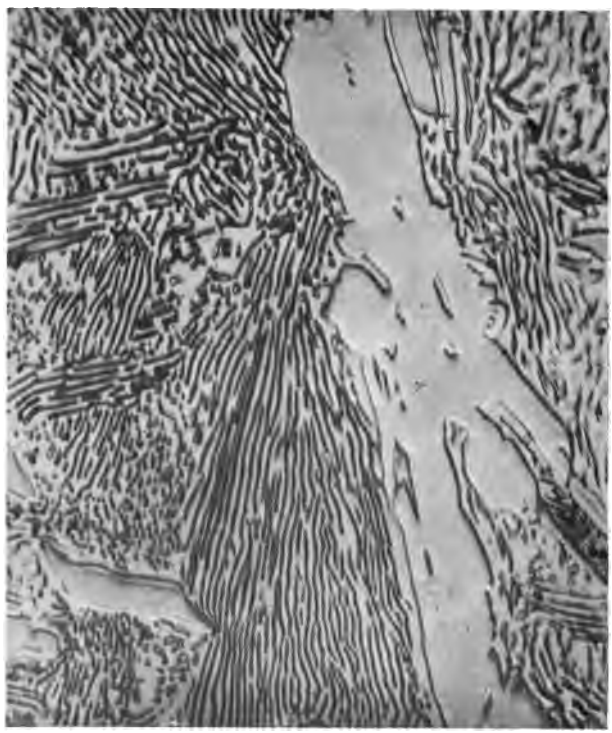


Fig. 18.—CEMENTATION STEEL (1·57 per cent. carbon). Polish-attack. V × 1000 diameters.

the eutectic—a proportion which may also vary in presence of other foreign bodies. Thus the microscopic observations of Dr Sorby correspond so exactly to the

thermal phenomena, that the former might have led to the prediction of the latter if the theory of solution had been known sooner.

If, instead of leaving the steel to cool quietly, it is quenched in a cold liquid, the transformations which would be produced during slow cooling are replaced, at least in part, because they have not had time to be completed in the interval of temperature in which they were possible. The carbon partially retains the state of dissolved carbon which it possessed at higher temperatures: it is said that it remains in the state of hardening carbon; and as the existence of a dissolved body requires that of a solvent, it appears necessary, although this idea is energetically disputed, that the iron also should be regarded as partially retaining an allotropic form. The theory of tempering, enunciated so brilliantly by Professor Akerman,[1] still remains in existence: modern researches have extended and completed it without destroying it.

These phenomena are illustrated in the structure.

If the quenching is done above the curve BEC, and as energetically as possible—in iced water, for example—ferrite, pearlite, and cementite disappear. Two cases are possible according as the proportion of carbon is inferior or superior to that of the eutectic. As an example of the first, let us quench a sample of steel with 0·45 per cent. carbon at the temperature 1050°. The commencement of the quenching is then represented by the point M of fig. D. We obtain a constituent probably homo-

[1] *Jour. of I. and S. Inst.*, p. 504 (1879).

geneous, formed of needles, parallel in the same region, which frequently cut each other parallel to three principal directions (fig. 19). This is martensite. Other

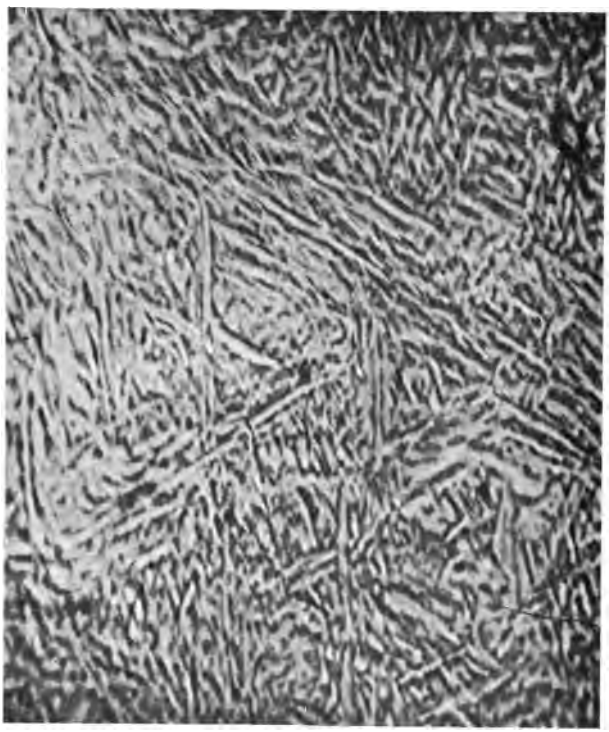


Fig. 19.—FORGED STEEL (0·45 per cent. carbon). Heated to 825°, and quenched at 720°. Polish-attack. V × 1000 diameters.

things equal, the needles of martensite become smaller and less distinct according as the eutectic is approached; the hardness simultaneously augments up to its

maximum. After that the mass does not remain homogeneous. A steel with 1·5 per cent. carbon, tempered

Fig. 20.—CEMENTATION STEEL (1·57 per cent. carbon). Heated to nearly 1050°, and quenched in ice-cold water. Polish-attack. V × 1000 diameters.

at 1050° (point N of fig. D) in iced water, separates into two constituents (fig. 20). One of the two, desig-

nated by the name austenite (this is the clear part of the figure), possesses the unexpected property of being scratched by a sewing-needle: the hardness diminishes then with the amount of carbon, when this proportion exceeds a certain limit.

If we retard the quenching until the temperature has fallen below BEC but is still above A_1E, there will naturally be found in the chilled metal the constituents which have already been isolated during the slow cooling before the commencement of the quenching. Thus, steel with 0·30 per cent. carbon, quenched at 720° C. (point O of fig. D), will show white ferrite at the side of striated martensite (fig. 21); the steel with 1·24 per cent. carbon, quenched at 735° (point P of fig. D), will contain cementite, in relief, which appears black under the conditions of illumination adopted, as compared with the martensite which surrounds it (fig. 22).

Below A_1E the structure is fixed, chilling commenced from a point in this region remaining without effect.

Bronzes.—The knowledge of the bronzes is much less advanced than that of steels. The complete curve has been determined by Mr Stansfield [1] (fig. E). Below are shown together the curves which represent the variations, in a function of the chemical composition, of several of the physical properties :—the electro-motive force (after Laurie), the electric conductivity (after Lodge), the calorific conductivity (after Calvert and

[1] *Proc. Inst. Mech. Eng.*, p. 269, plates 41 to 43 (1895).

Johnson), the hardness (after Martens), induction (by the Hughes Balance), tenacity (after Roberts-Austen),

Fig. 21.—FORGED STEEL (0·30 per cent. carbon). Heated to 900°, and quenched at 720°. Polish-attack. × 1000 diameters.

and the elongation (after Thurston). The majority of these curves are borrowed from Mr Stansfield. Many

of them seem to indicate the existence of the definite compounds $SnCu_3$ and $SnCu_4$, to say nothing of isolated

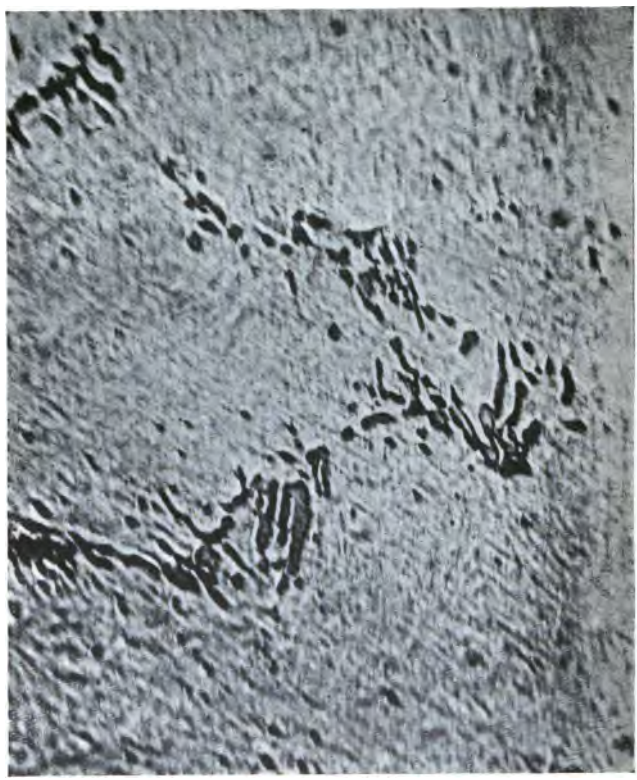


Fig. 22.—FORGED STEEL (1·24 per cent. carbon). Heated, and quenched at 735°. Polish-attack. V × 1000 diameters.

interruptions. Behrens,[1] by the study of the micro-structure, has arrived at the idea of definite compounds

[1] *Proc. Inst. Mech. Eng.*, pp. 70–91.

still more numerous; and M. H. Le Chatelier[1] thinks

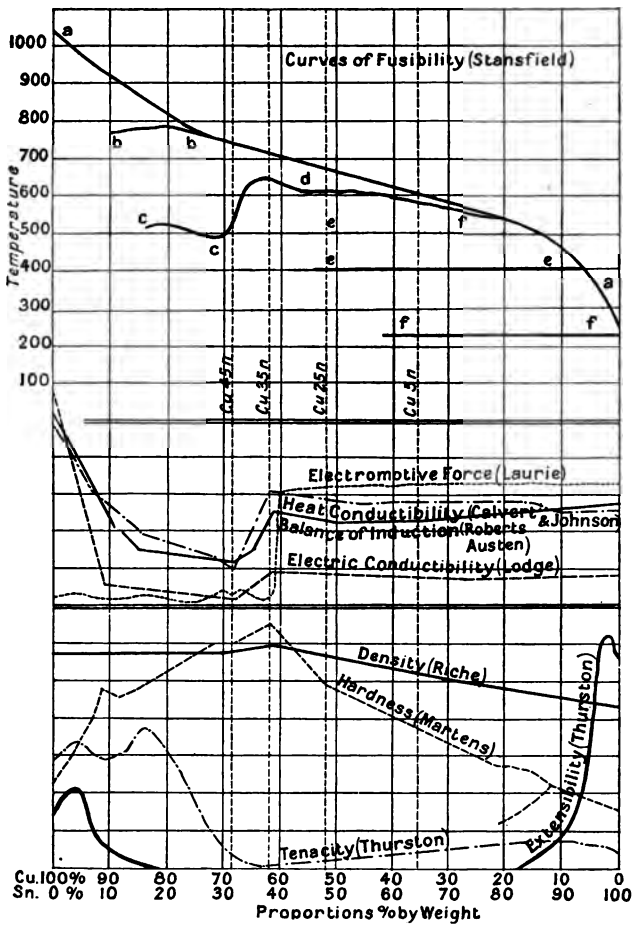


Fig. E.—Equilibrium Curves and Physical Properties of the Alloys of Copper and Tin.

[1] *Bull. Soc. d'Encouragement*, 4e série, t. x. p. 388 (1895).

he has isolated $SnCu_3$ by proximate chemical analysis. However, the alloy of this composition shows two liberations of heat. In fact, there is not, perhaps, a single one of the conclusions drawn from a series of experiments which is not contradicted or at least challenged by the conclusions drawn from another series. When we seek to reconcile the divergences, we have to struggle with inextricable difficulties, and the writer will not risk augmenting a number of premature interpretations.[1] The aspect of the curve of fusibility alone shows the complexity of the question: certain alloys have three, and as many as four, points of transformation or of solidification, the position and sometimes the existence of which may be connected with the rapidity of cooling. Fortunately the problem is now solvable, thanks above all to micrographic analysis, which alone furnishes as many equations as unknowns, and which has already given, through MM. Guillemin, Behrens, and Charpy, very interesting results. The solution is now only a question of time, patience, and method.

Whilst awaiting it, there will be seen with interest several micro-photographs of bronzes obtained from beautiful preparations by M. Guillemin. The structure has been revealed in every case by heating so as to obtain colours; the dark parts of the photographs are those which are the first to take the oxidation tints.

[1] Messrs Heycock and Neville, in their Bakerian lecture given before the Royal Society on The Constitution of the Copper Tin Alloys, Series A, vol. 202, pp. 1-69, 1903, have most fully dealt with this subject.

A first series comprises bronzes cast in ingot moulds in the form of small bars, with a square section of 15 by 15 mm., in which the percentage of tin varies from 9 to 33 per cent. All the photographs are enlarged 100

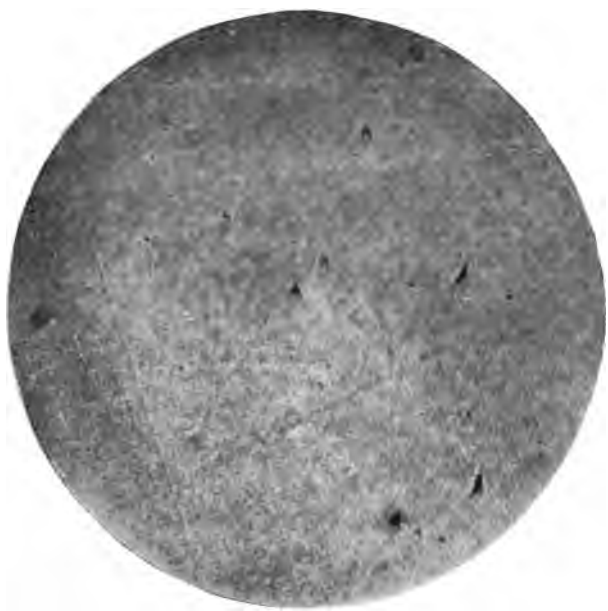


Fig. 23.—BRONZE, containing 9 per cent. Tin, cast in an ingot mould. Heat, oxidation tinted. V × 100 diameters.

diameters, and would not show further details with even higher enlargements.

The bronze with 9 per cent. of tin (fig. 23) appears homogeneous; at least, the process of investigation shows it as such. There is probably only one point of fusion, for it is just at this percentage that the line BB of the

second point of solidification on the curve of Mr Stansfield stops.

The bronzes with 11, 16, and 19 per cent. of tin (figs. 24, 25, and 26) show increasing proportions of a second constituent around the crystallites of the first. Its

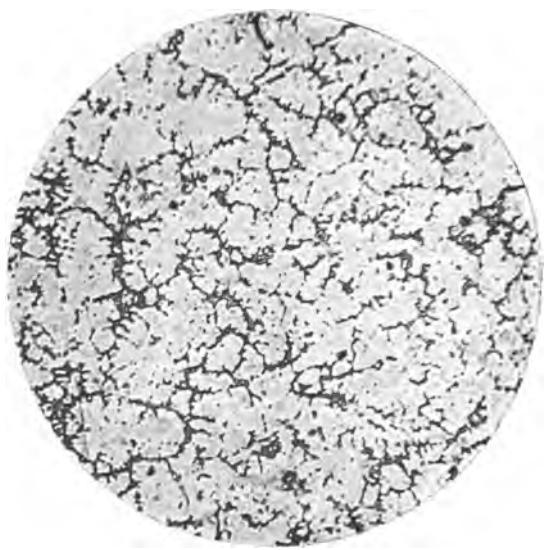


Fig. 24.—BRONZE, with 11 per cent. Tin, cast in an ingot mould. Heat, oxidation tinted. V × 100 diameters.

point of solidification is probably indicated by the line *bb* of the diagram.

With 33 per cent. of tin the alloy again becomes homogeneous (fig. 27), and subdivides into jointed polyhedrons, although the composition does not correspond to a definite composition, and is comprised of $SnCu_3$ and $SnCu_4$.

Another series of photographs shows the influence of the rapidity of cooling on a bronze with 19 per cent. of tin.

A sample has been cast in sand and cooled slowly (fig. 28, 100 diameters). The crystallites of first solidification have acquired better defined forms than in the

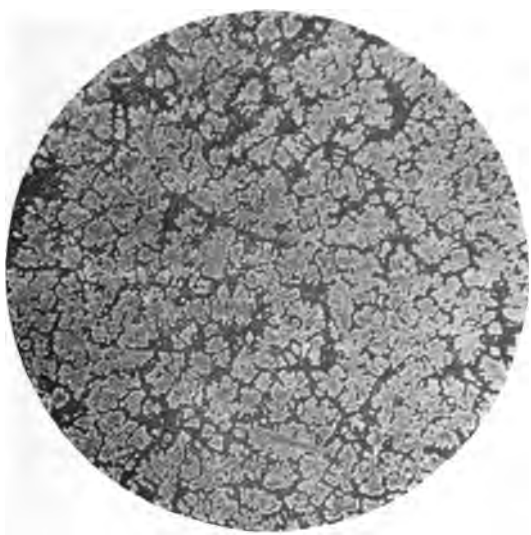


Fig. 25.—BRONZE, with 16 per cent. Tin, cast in an ingot mould. Heat, oxidation tinted. V × 100 diameters.

similar sample cast in an ingot mould (fig. 26). Again, the matrix of the second solidification is no longer homogeneous; it is divided into two elements of which only one is coloured by oxidation, as is seen on a more enlarged photograph (fig. 29, 500 diameters).

A second sample of the same composition has been

Fig. 26.—Bronze, with 19 per cent. Tin, cast in an ingot mould. Heat, oxidation tinted. V × 100 diameters.

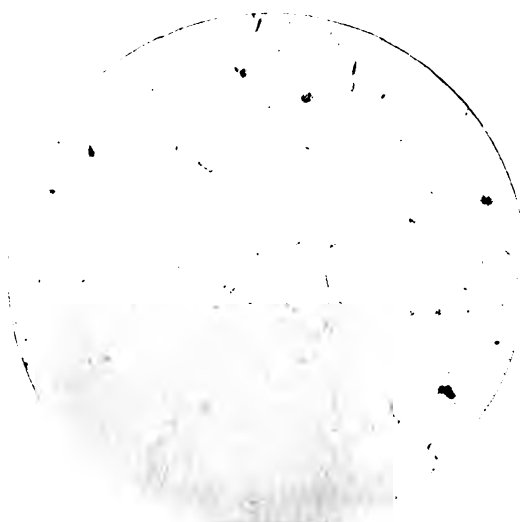


Fig. 27.—Bronze, containing 33 per cent. Tin, cast in an ingot mould. Heat, oxidation tinted. V × 100 diameters.

Fig. 28.—BRONZE, with 19 per cent. Tin, cast in sand. Heat, oxidation tinted. V × 100 diameters.

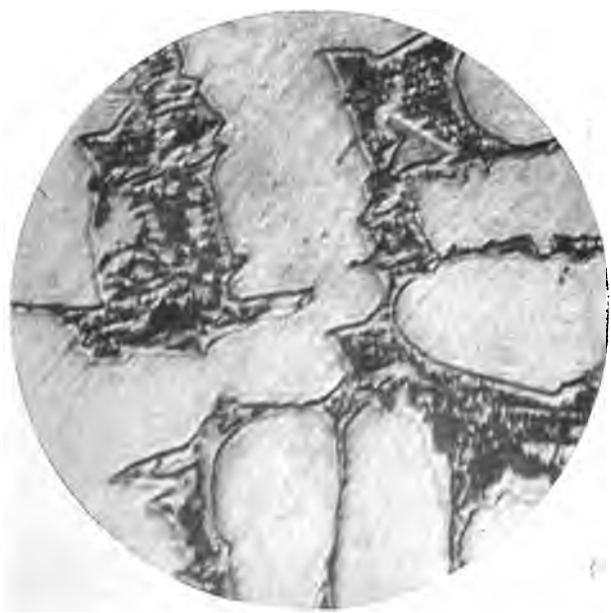


Fig. 29.—BRONZE, with 19 per cent. Tin, cast in sand. Heat, oxidation tinted. V × 500 diameters.

tempered at a dull red heat in water at 50°; the general structure appears to have remained nearly the same if we look at the enlargement of 100 diameters (fig. 30). Under an enlargement of 500 diameters (fig. 31), it is seen that the matrix of second solidification cannot be resolved into two parts. The transformation prevented

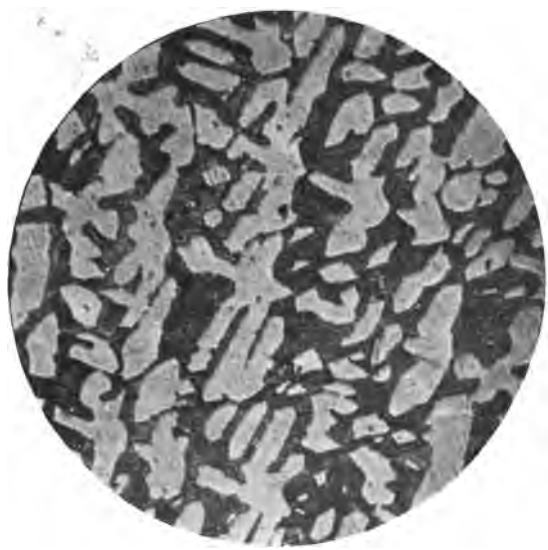


Fig. 30.—BRONZE, with 19 per cent. Tin, quenched from dark red in water at 50°. Heat, oxidation tinted. V × 100 diameters.

by quenching is probably that which the line CC of the diagram indicates. This line CC appears then to correspond, not to the solidification of a liquid properly so-called, but rather to the resolution of a solid solution.

Influence of Pressure.—Hitherto we have not introduced the element of pressure; it must, however,

have influence during the operations of forging, or simply during cooling, on account of the contraction of the exterior layers which are the first to cool. One of the effects of pressure is to displace the points of transformation, raising them or lowering them according to the sign of concomitant change of volume. This

Fig. 31.—BRONZE, with 19 per cent. Tin, quenched from dark red in water at 50°. Heat, oxidation tinted. V × 500 diameters.

effect is not very marked on the points of fusion; it appears to be much more so on the points of molecular transformation. M. Mallard and Le Chatelier have thus lowered by more than 100° the isomeric

modification of the iodide of silver.[1] Sir W. Roberts-Austen[2] has lowered, by the same means, the recalescence; and it is probable that the pressure determined at first by the contraction, and then by the change of state and of volume of the iron, plays, as Professor Åkerman thinks, a great part in quenching.

PATHOLOGIC METALLOGRAPHY.

This branch of Metallography is occupied, as its name indicates, with what might be called the diseases of metals.

Fig. 32 represents a network of fissures in steel quenched at too high a temperature. Professor Wedding[3] has wisely directed his studies towards faults of this kind.

In fig. 33 we see how simple polishing reveals the inclusion of slag in steel. These inclusions are, in fact, very frequent: they are not very troublesome when the grains of slag are little and scattered; they may, however, become very dangerous if the slag forms extensive layers cropping out on the exterior surfaces, for they then constitute causes likely to lead to fracture, which will gradually spread from place to place. According to Prof. Arnold, sulphur in the state of sulphide of iron would cause analogous inclusions.

Mr Stead[4] has recently shown, by an ingenious pro-

[1] *Comptes rendus*, t. xcix. p. 157 (1884).
[2] *Proc. Inst. Mech. Eng.*, p. 124 (1893)
[3] *Stahl und Eisen*, t. xi. p. 879 (1891).
[4] *Jour. of I. and S. Inst.*, 1897, part 1.

cess, the influence of these fragile filaments in a malleable body. A plate, having been previously polished and suitably attacked in order to show the structure, is then bent until the commencement of rupture is produced. It is immediately seen that the

Fig. 32.—FORGED STEEL (1·24 per cent. carbon). Quenched from white heat. Polished. V × 20 diameters.

lines of rupture follow the slag in puddled iron, the cementite in cement steel, and the phosphide of tin in tin which contains phosphorus.

Foreign bodies and their chemical compounds do not always form these independent networks, which are sometimes useful, sometimes obnoxious, and the in-

fluence of which may be so great on the mechanical properties. They may also remain invisible and dissolve,

Fig. 33.—CINDER IN A STEEL. Polished. V × 1000 diameters.

modifying, although so attenuated, the state and the qualities of the metallic mass.

In every case the structure acquired from a cooling

of fixed rapidity may always be altered by an elevation of temperature, which redissolves the separated compound or causes the dissolved bodies to move.

For example, fig. 34 shows the structure of a little bar of gold alloyed with 0·2 per cent. bismuth and cast

Fig. 34.—GOLD, containing 0·2 per cent. Bismuth, cast in an ingot mould. Attacked by nitro-hydrochloric acid. V × 17 diameters.

in an ingot mould; it is composed of elements largely prismatic, normal to the surfaces of cooling. After annealing for some minutes at about 250°, the prisms become subdivided into a large number of little polyhedrons, distinguished by the variations in their brightness according to the incidence of the light with respect to the crystalline organisations (fig. 35).

The fusion of the eutectic alloy of gold and bismuth might have been reached, and it is easily conceived that the recrystallisation of the mass has taken place, by a series of solutions and precipitations in succession, but the existence of a liquid is not necessary. An alloy

Fig. 35.—GOLD, containing 0·2 per cent. Bismuth, reheated to nearly 250°. Attacked by nitro-hydrochloric acid. V × 17 diameters.

of gold with 0·2 per cent. antimony, cast in an ingot mould like the former (arising from the researches of Sir W. Roberts-Austen [1]), is represented by figs. 36, 37, before and after annealing at 250°. Annealing has

[1] *Trans. Roy. Soc.*, clxxix. p. 339 (1888), and clxxxvii. 417 (1896).

rendered the structure almost amorphous, although the more fusible of the alloys of gold and antimony do not melt below 440°.

We do not know *a priori* if such changes would be good or bad in a particular case, but we have learned

Fig. 36.—GOLD, containing 0·2 per cent. Antimony, cast in an ingot mould. Attacked by nitro-hydrochloric acid. V × 17 diameters.

that the structure and, accordingly, the properties of the refractory metal may be largely transformed by reheating at a relatively low temperature in the presence of a small proportion of certain impurities. This is a fact which commands serious attention.

The distinction between morbid accidents and

physiological phenomena is not always so obvious as in the preceding examples. It may result from a convention founded on experience. If a certain mechanical and calorific treatment gives to a certain metal the best possible qualities, the metal thus prepared will be

Fig. 37.—GOLD, containing 0·2 per cent. Antimony, reheated to nearly 250°. Attacked by nitro-hydrochloric acid. V × 17 diameters.

regarded as healthy. And, conversely, the metal prepared by different processes might be regarded as diseased.

Here are some samples of steel, containing 0·45 per cent. of carbon, which have been, after forging, annealed respectively at the temperatures of 750°, 1015°, and

Fig. 38.—FORGED STEEL (0·45 per cent. carbon). Reheated to 750°. Polish-attack. V × 100 diameters.

Fig. 39.—FORGED STEEL (0·45 per cent. carbon). Reheated to 1105°. Polish-attack. V × 100 diameters.

1390°. The three samples are mixtures of ferrite and pearlite, but how different! In the first (fig. 38) the two constituents are in masses of small size irregularly mixed; in the second (fig. 39) the pearlite tends to form nuclei, which the ferrite surrounds with a continuous and ramified network; in the third (fig. 40)

Fig. 40.—FORGED STEEL (0·45 per cent. carbon). Reheated to 1390°. Polish-attack. V × 100 diameters.

this structure is more accentuated, and the grains of pearlite have become so large that the photograph, at an enlargement of 100 diameters, does not show a single one of them whole. The aspect of the structure and the absolute dimensions of the grains are, as Mr Sauveur[1] has notably shown, characteristic of the calorific treatment of steel, and, accordingly, of the

[1] *Trans. Amer. Inst. Min. Eng.*, t. xxii. p. 546 (1893).

correlative mechanical properties. Microscopic study allows us to recognise, after the event, the calorific treatment which has been adopted, and to correct it if necessary.

Having thus dealt with the constitution and diseases of metals, it remains for us to perfect our means of safeguarding the former and of curing the latter, in order to secure, as far as possible, permanent strength for our structures. Such are the aims common alike to the metallurgical and to the engineering members of the International Society for the Testing of Materials. Towards the attainment of this end metallography offers a new means of diagnosis, the methods of manipulation improve daily, and the utility of which appears more and more evident.

PART II.

GENERAL METHOD FOR THE MICROGRAPHIC ANALYSIS OF CARBON STEELS.

CHAPTER I.

ROUGH POLISHING.

Preliminary Operations.—Neglecting the study of fractures, to which, unfortunately, we can apply only feeble magnification, the first operation which has to be performed when examining a given sample is to obtain a plane section. Assuming that such a section has been prepared in the workshop by means of a lathe, file, saw, grindstone, or any chilled tool, according to the hardness of the metal, it remains to polish the cut surface.

General Remarks on Polishing.—A professional polisher, on being questioned, would tell you that he passes the section first over a fine emery wheel, and then upon a disc of buff-leather covered with English rouge moistened with water, and turning with a velocity of about 2000 revolutions per minute. The result

would be satisfactory for hard quenched steels, but not so for soft or annealed steels. The workman answers this criticism by saying that the faults are inherent in the metal, and that there is no better process.

It is necessary, therefore, to do one's own work. The technical handbooks on polishing give but feeble assistance. To obtain any useful information, we must consult metallographers. Dr Sorby[1] and Professor Martens[2] have from the first given good technical rules for the preparation of metallic surfaces. Still, it is soon seen that, even in following these rules with the greatest possible care, we do not either at the first attempt, nor sometimes after many trials, arrive at the desired results. This is because polishing is still an art, the theory of which is unknown, and because success depends on numerous causes and on apparently trivial differences in the quality of the polishing materials and the manner of using them. Each person has therefore to serve his own apprenticeship, which the experience of others may shorten, but cannot entirely supersede.

This remark is made to warn my readers against premature discouragements. I shall now proceed to describe the process of polishing I have finally adopted.

Preparatory Polishing.—This process is in principle the same as that of the workman, viz., roughing off by emery, and finishing with rouge; but it is the details which determine the results.

[1] *Jour. of I. and S. Inst.*, 1887, p. 255.
[2] Glaser's *Annalen*, t. xxx. p. 201.

In default of a mechanical motor, I substitute, (following the example of Dr Sorby) for the emery grindstone of the workshop, increasingly fine emery papers placed on glass. These papers must be of regular grain; the powder must be sufficiently adherent not to be detached by the rubbing, so that wear takes place in scratches and not by "pits" filled with dust, as happens when the powder is non-adherent; lastly, the paper and the glue must themselves be free from particles likely to scratch the soft iron. Commercial emery papers rarely fulfil all these conditions, so that it is best to make one's own papers. For this purpose I buy levigated "120-minute" emery—that is to say, emery which has taken 120 minutes to settle in a vessel of water (the dimensions of which I do not know). I carefully mix this with water, and collect the deposits formed at the end of increasing periods of time in precipitating glasses. The following is an example of the behaviour of washed emery under these conditions:—

In 0·5 litre of water;				deposited in less than			1	minute:	16	per cent.
„	„	„	„	from	1	to	2	„	13	„
„	„	„	„	„	2	„	5	„	8	„
„	„	„	„	„	5	„	10	„	6	„
„	„	„	„	„	10	„	20	„	14	„
In 1·5 litres of water;				„	20	„	40	„	8	„
„	„	„	„	„	40	„	60	„	11	„
„	„	„	not deposited in				60	„	24	„
									100	

It will be seen that the commercial powder is not exactly homogeneous. The classified powders obtained are, after drying, diffused in a mucilage of albumen,

such as is used for the preparation of sensitised paper and spread on paper of the best quality. The sheets thus prepared are not much to look at, and show all the brush marks; but they are good, and will last a long time.[1]

In addition to the first rubbings, which should efface the file marks, and for which commercial papers fully suffice, I usually rub my sample backwards and forwards 100 times on each of the above 1–2, 2–5, 5–10, 10–20, and 20–40 "minute" papers successively. It is advisable to change the direction of rubbing at each change of paper, in order to detect whether the scratches of the preceding paper have been effaced. The finest papers are not used, as the ochre-coloured powder, which probably contains more hydrated peroxide of iron than emery, roughens metals, and dulls them instead of polishing them.

When the emery papers are good, the polishing can be finished off at once with English rouge, without rubbing with any intermediate powder. Commercial rouges are, like the emery powders, not always of a suitable quality. The so-called "Steel" rouges are too hard and coarse for unquenched steels, but the "Gold" rouges are generally better; still it is usually

[1] Here is a recipe for this paste, which I have taken from a treatise on photography:—

Albumen, ...	72 c.c.
Water, ...	24 c.c.

Beat to a froth, and, after 12 hours, strain through a fine-meshed sponge. Gelatine scratches the iron.

necessary to wash them. You must, therefore, prepare your own rouge, carefully avoiding the presence of any grit. This is done by calcining copperas at as low a temperature as possible, and separating the finest part of the product by levigation, which is the only part which can be used. M. Henrivaux, the well-known Manager of the Saint-Gobain Glass Works, has removed this difficulty by giving me a sample of his oxalate rouge, which is excellent, and has proved very useful. I sprinkle this rouge on a piece of cloth with a fine nap, doubled and stretched on the cast-iron table of a small horizontal polishing machine. The machine in question comes from Germany, where I had seen it in Professor Marten's laboratory at Charlottenburg; it is made by Fuess, at Steglitz, near Berlin, and costs £3. The sample is held in the left hand and pressed on the cloth, whilst the handle is turned with the right hand at the rate of about 200 revolutions a minute; the corresponding circumferential velocity of the polisher is, in round figures, 2·50 metres per second.

Before polishing, the cloth must be slightly wetted, just sufficiently to prevent the rouge from forming an adherent film on the metal. Towards the end of the operation the amount of water may be increased, the pressure on the sample being at the same time diminished.

When the preparation with emery paper has been effective, the very fine striations are rapidly effaced; more trouble is experienced in getting rid of little hollows, common chiefly in soft steels, which seem to arise from tearing, and which it is important, however,

to remove, so as not to confound them with natural porosities. To give an idea of the polishing required with rouge, at least 1000 turns of the handle must be given for polishing hard quenched steels, and 2000 for annealed steels.

It is hard work, but to-day three-fourths of the work can be avoided by substituting alumina for rouge, following the advice of Le Chatelier,[1] who has thus rendered very considerable service to metallographers.[2]

There is obtained finally (or should be obtained) a distinctly specular surface, even when the metal is not homogeneous, provided that the cloth is not too wet, or that the rouge has not been used too sparingly. But this surface usually only leads to the detection of the presence or absence of slag, cracks, or porosities; plain polishing is not, in fact, by itself a very fruitful means of investigation, but, in the majority of cases, simply preparatory to the application of some other process of micrographic analysis.

The following chapter gives a description of the special process which it is the object of this work to present.

[1] *Bulletin de la Société pour l'Encouragement de l'Industrie Nationale*, 5e série, t. vi. p. 365, Sept. 1900. Diamantine powder, used for many years by Mr Stead, also consists of washed alumina.

[2] Here and elsewhere I simply describe my own processes, without pretending to give them as a model to be followed. The conditions under which I work compel me to seek, above all, the greatest possible simplicity of appliances. But if a mechanical motor can be used, it would be best to make use of it. Le Chatelier's example can be followed profitably, or that of Mr Stead (*Journ. of I. and S. Inst.*, 1894, part i. p. 292; *Metallographist*, t. iii. p. 220, July 1900), who has so ably described his machines and processes.

CHAPTER II.

GENERAL METHOD.

This method includes three successive operations:—

1. **Polishing in bas-relief.**
2. **"Polish-attack."**
3. **The action of suitable chemical reagents.**

1. **Polish in Bas-relief.**—When a non-homogeneous body is polished, its different constituents tend to wear away unequally, according to their specific properties and actual dimensions; and it is possible, under suitable conditions, to show the structure by unequal relief of its constituents. This is what I call polish in bas-relief. Dr Sorby,[1] Professor Martens,[2] and especially M. Behrens,[3] have thus obtained, sometimes by accident, sometimes by design, interesting metallic preparations. The method in question is not therefore new: I have only endeavoured to make its use more systematic and its management easier. To obtain a desired result, it

[1] *Loc. cit.*

[2] *Stahl und Eisen*, t. xii. p. 406, and plate vi. figs. 34 to 38.

[3] *Récueil des travaux chimiques des Pays-Bas*, t. x. p. 261 ; and *Das Mikroskopische Gefüge der Metalle und Legierungen*, passim.

is necessary to polish on a substratum, elastic enough to allow of the production of inequalities, and delicate enough to show the finest details. That which has succeeded best with me is parchment, the value of which Dr Sorby first discovered. It is stretched damp on a piece of well-planed wood, and fixed by nails. To use it, it is again damped at the place where the rubbing is to be done, and sprinkled with English rouge, not in excess. The small quantity of powder put on is rubbed strongly on the parchment; then the board is put under a water-tap, and washed while it is rubbed, so that only the finest particles of rouge, or those which have penetrated into the pores of the parchment, are retained. On this *rouged parchment* the metallic surface is rubbed, adding from time to time some drops of water when needed. The hard constituents appear much more quickly in relief, because their relative resistance to wear is greater and their dimensions larger: 500 to 2000 rubbings, backwards and forwards, covering about 8 centimetres, are generally sufficient.

The finest rouge is, however, still rather coarse, and it is occasionally useful to continue the polishing, on moistened parchment, with precipitated sulphate of lime. I have also used, sometimes with much success, more often with inexplicable failure, precipitated sulphate of baryta. Certain details are then revealed; but the work is long and tedious, and, happily, it is not often necessary.[1]

[1] It would be interesting, for the further development of the method,

The section once prepared, we have now to discriminate under the microscope between its raised portions and its cavities. Success is ensured, provided that the preparation is uncoloured, by the aid of the following artifice, which probably is not unknown, although I have nowhere found it described. The luminous rays are strongly diaphragmed; the objective is placed a little below the focussing point, and is slowly raised. The reliefs, which at first appear brilliant and yellowish on a relatively darker ground, gradually become dark on a bright ground; the cavities present inverse appearances so perfectly, that two photographs of the same preparation, taken one a little below and the other a little above the mean focussing point, are almost positive and negative to one another.

2. **Polish-Attack.**—The second operation, to which I have given the name "polish-attack," consists of reinforcing the mechanical action of the chemically inert powder by the action of a liquid reagent, which is inert by itself, but which becomes active owing to the friction.[1] Singular effects are sometimes obtained by this means. Ammoniacal water, for example, not only does not oxidise steel, but preserves it from oxidation. Hence I was much surprised one day, when polishing a piece of soft steel with dilute ammonia and barium sulphate, to see the steel become slowly and successively

to draw up a table of powders of sufficient fineness and of graduated hardness like the mineralogical scale of Mohs.

[1] Compare Daubrée, *Études Synthétiques de Géologie Expérimentale*, Dunod, Paris, 1879, p. 268 *et seq.*

tinted with all the tempering colours in their usual order, and in such a way that the hard constituent was always a shade in advance of the soft mass: direct heating had never given such a well-marked result. I have not yet discovered the reason for this occurrence. The reagent which, for some time past, I have used for polish-attack, is an aqueous extract of liquorice-root popularly known as "coco." It was M. Ivan Werlein who directed my attention to it as an example of certain prejudices of workmen. But workshop recipes, although sometimes curious, often embody the results of actual observation: I therefore tried coco, with the preconceived idea that it might be a lubricant of the same kind as soap-suds, which would perhaps make the rubbing easier and so reduce the manual labour. The result did not justify my surmise; it was found, instead, that when used on parchment in conjunction with rouge or precipitated sulphate of lime, this extract intensified the relief polishing and tinted certain constituents, either to the exclusion of others or with different degrees of rapidity.

I prepare the extract by macerating 10 grms. of chopped-up root in 100 c.c. of cold water for 4 hours, and then filtering. The solution quickly spoils and becomes more and more active, and it has a very serious drawback: liquorice-root, like all vegetable products, varies in different samples. According to its source, its age, its state of preservation, and other unknown circumstances, it gives, although prepared under similar conditions, very variable solu-

tions, sometimes too strong, sometimes not strong enough.

It is necessary, therefore, to have a more reliable reagent. This I have found, with the aid of M. Cartaud, in a 2 per cent. solution, in water, of ammonium nitrate. This solution acts exactly like coco, but more quickly, provided that the parchment is not too soaked or too dry. There are, at first, certain practical difficulties in the way, but when these have been overcome good results are obtained. It is necessary to look at the sample periodically, in order to stop when the desired effect is obtained: 200 rubbings backwards and forwards are often sufficient.

3. **Chemical Attack.**—After the polish-attack, it is best to rub the sample again with rouge on the revolving disc, in order to decolourise it and to efface the reliefs. The examination is completed after making a chemical attack.

The reagents which it is possible to use may be divided into three classes: acids, halogens, and salts.

The final result of the total solution of carburetted irons in the principal acids is comparatively well known, but the initial processes of a very incomplete attack by a dilute liquid remain more obscure, and are probably more complex. 10, 5, and 2 per cent. hydrochloric acid (the sample either being bound or not to the positive pole of a Grenet element), 10 per cent. sulphuric acid, 10 per cent. oxalic acid, 10 per cent. normal chromic acid (Abel's reagent), hydrochloric acid in absolute alcohol (Marten's reagent), do not seem to me to

present advantages over nitric acid, the much more general use of which will be criticised further on. We can make use of, according to circumstances, solutions of nitric acid at 36° Baumé, or of aqueous dilutions of 20 and 2 per cent.[1] By immersing the sample in concentrated acid, in which it becomes inactive at first, and then sprinkling it with plenty of water, M. Sauveur has obtained very homogeneous attacks even on large surfaces.

The halogens, which attack iron, completely separating the whole of the carbon, have a simpler action than the acids. I am quite satisfied with the use of tincture of iodine. This tincture should not be made with absolute alcohol, or a very slow and irregular attack will be obtained from which no conclusions can be drawn. That bought at the chemist's is suitable. I apply it by touch and by successive additions of a drop for every square centimetre of surface. It is left to act until it is decolorised, and, after examination, a further addition is added if needful. The first application is often sufficient and sometimes even a little too much, and in such cases a solution twice diluted can then be used, as recommended by M. Sauveur.

Copper sulphate, bichloride of mercury, gold chloride, and potassium platinochloride—salts which replace the dissolved iron by a deposit of their metal—have not given me very encouraging results, but the number of

[1] The percentage of acid is reckoned in fractions of the total volume for liquid acids, and in weight by comparison with the water for solid acids.

experiments being necessarily limited, I may easily have missed favourable conditions, and I would not dissuade anybody from repeating the trials.

I now only retain the following reagents, viz., tincture of iodine, and, accessorily, nitric acid, and, in particular cases, hydrochloric acid. After attack, I wash with alcohol in the case of tincture of iodine, and with water and alcohol for the other reagents, and wipe with fine dry linen. Drying under a jet of compressed air is preferable whenever feasible. The drying is omitted if an immersion objective has to be used.

With the aid of these three successive operations—viz., polishing in bas-relief, polish-attack, and chemical attack—and by the reactions which they furnish, we shall now try to define the primary constituents of carbon steels.

CHAPTER III.

PRIMARY CONSTITUENTS OF CARBON STEELS.

I CALL every structural element a primary constituent when its micrographic reactions permit it to be considered as a species. These constituents, not including the slag, which is an impurity, nor the graphite, which does not appear normally in the usual conditions of the manufacture and treatment of steels, are really six in number. But the list is necessarily provisional, like that of the chemical elements, and for the same reasons. The progress of science brings discoveries, duplications, and confirmations.

FERRITE.

The chief constituent of carburetted iron is the iron itself, pure, or at least relatively pure, for absolute purity is a myth. When iron is considered as a structural element of a complex whole, it is convenient to give it a particular name, and I have adopted that of "*Ferrite*," happily coined by Professor Howe in conformity with mineralogical nomenclature.

By polishing in bas-relief, ferrite at first assumes a specular polish. If this operation be long continued, it granulates and forms polyhedric grains, of which some are slightly in relief and some are in depression above and below a given level.

Polish-attack produces the same effect, but more rapidly.

Tincture of iodine leads again to the same results from the first application under the conditions previously defined. On continued etching, certain grains begin to colour yellowish-brown, more or less deeply. The extremities of the grains are traced by dark lines in intaglio.

As for nitric acid, it also marks the joints of the grains very well, but it colours or does not colour the preparation according to circumstances so complex and confused as to justify a special investigation. If you place under the microscope, lighted by the vertical illuminator, a polished section of extra-mild steel in a bath of 2 per 1000 nitric acid (2 volumes of acid at 36° Baumé in 998 of water), and if you continue the attack for half an hour, you see certain grains of ferrite become yellow and brown progressively, whilst others become dull without being coloured, or remain practically unaltered. The patina of the browned grains generally remains after wiping. On making the same experiment in 2 per cent. acid, the ferrite takes beautiful colours, differing in different grains and often in the same grain. These colourings are analogous to those which are produced by heating in air,

and we obtain yellow, brown, purple, blue, green, or colourless grains with continual transformations. The patina acquired at the moment it is brought out of the liquid is more or less deteriorated by wiping. It is difficult to follow with the microscope the too rapid attack of 20 per cent. acid: but an acid of this degree of strength often furnishes a preparation, after wiping, the ferrite of which is more or less eaten into, but not coloured. These facts lead one to think that the attack commences by the formation of a fine pellicle of oxide or of some other body, which pellicle tends to be dissolved in the bath. According as the formation of this patina at a given point appears more quickly or more slowly than its dissolution, the tints do or do not appear; and when they do, they can, under suitable circumstances, run up or down their ascending or descending scale. The phenomena are so complex that you cannot foresee them exactly, even in the best studied cases. Nitric acid, though it may render excellent service with known steels—*e.g.*, for determining the size of the grain and the final temperature at which it has been worked—is then, for micrographic analysis, a dangerous reagent which has led me personally more than once into error, before I had thoroughly fathomed its manner of action. To say the least, its indications ought to be controlled by other means. This remark applies equally to all the acids.

The grains of ferrite are chiefly fragments of cubical crystals which develop around independent centres of crystallisation, and are mutually limited by roughly

plane surfaces. Their structure can be revealed by the prolonged action of nitric acid diluted with varying proportions of water (Andrews[1] and Stead[2]), by the

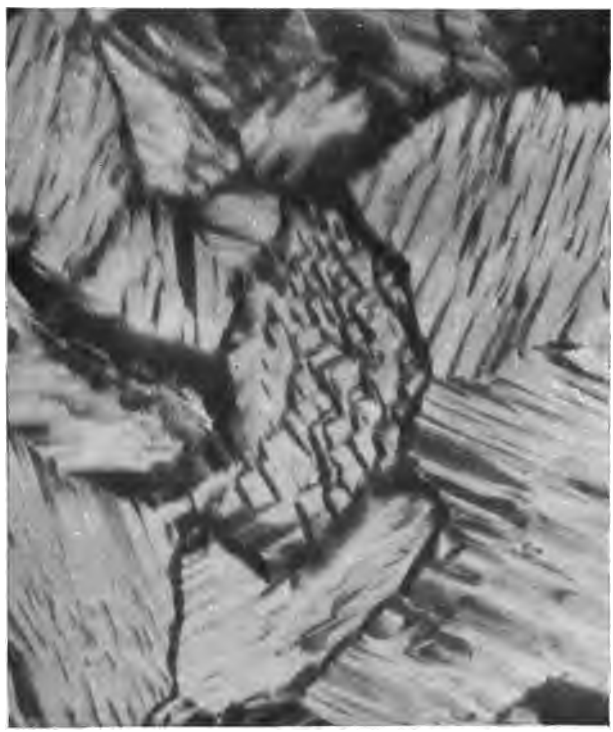


Fig. 41.—FERRITE. Strongly etched. Showing the orientation of the crystals. V × 400 diameters.

double chloride of copper and ammonia (Heyn[3]), or,

[1] *Engineering*, lx. p. 88, 1895.
[2] *Jour. of I. and S. Inst.*, 1898, part i. p. 145.
[3] *Mittheilungen aus den k. technischen Vers.*, 1898, pp. 310–331.

better still, by a sufficiently long submersion in lukewarm 20 per cent. sulphuric acid, followed by cleaning in nitric acid. After such treatment, the surface of a certain grain may be formed parallel to either the face or the edge of a cube, or in any direction whatever. The corrosion figures on this grain show squares, parallel lines, or have a confused aspect. Fig. 41 (vertical light, $\times 400$), which in reality represents the figures of growth of iron reduced from chloride of iron by hydrogen below 850°, gives a good idea of these corrosion figures. But, interesting as it may be in itself, the crystallographic study of iron does not enter into this notice. Micrographic analysis proposes rather to define ferrite as a whole without revealing details, and the reagents indicated have been chosen to this end.

CEMENTITE AND PEARLITE.[1]

The second constituent of steels is Carbide of Iron, foreseen by Karsten and Caron, and isolated more or lest intact by F. C. G. Müller, Sir Fr. Abel, Osmond and Werth, Arnold and Read, and lastly, by Milius Förster and Schöne, and by Moissan. The results, taken together, tend to give this carbide the formula Fe_3C, the iron being capable of being replaced, in any proportion whatever, in special steels, by manganese or chromium. Professor Howe calls it "*Cementite*," and the carbon which is therein found combined in a definite composition

[1] I have recently used with success Mr Igersky's reagent, consisting of 5 per cent. picric acid in alcohol, and find it excellent for developing the structure of pearlite.—*Ed.*

has received the name of *Carbon of Cementation* (Caron), *Annealing Carbon* (Osmond and Werth), and *Carbon of the Normal Carbide* (Ledebur). It is, in fact, in cementation steels, and more generally in all steels cooled very slowly, that the cementite aggregates in particles of the largest size, and where it is the easiest to study.

Dr Sorby has shown that cementite can be obtained in two distinct forms—first, in rectilinear lamellæ relatively thick, which can be superposed like leaves (*Independent Cementite*): these groups often trace a continuous network, which divides the mass into grains, which are thus isolated from each other. Second, in finer lamellæ, usually curved, those of the same group nearly parallel and interstratified with lamellæ of ferrite.[1] This mixture has been named "*Pearlite*" by Professor Howe, because it shows, with oblique light, the rainbow colours of mother-of-pearl, when the polishing or attack has eaten away to a convenient depth the soft and attackable lamellae of ferrite. (This optical phenomenon has long been known, and has been explained by Fresnel.)

Sorby has since proclaimed that hard steels are mixtures of pearlite and independent cementite, soft steels mixtures of pearlite and ferrite, and, for an intermediate carbon content, the whole mass is composed of pearlite alone. This percentage of carbon has been fixed by Professor Arnold[2] at 0·90 in pure steels, which

[1] *Jour. of I. and S. Inst.*, 1886, p. 140; and 1887, p. 255.
[2] *Proc. Civ. Eng.*, t. cxxiii., 1895–6, part 1.

is practically exact, although it cannot be considered as having an absolute value. Not only does it vary with the amount of strange bodies present, but it is sufficient to examine a steel incompletely cemented and in which the carbon decreases gradually from the surface to the centre, in order to see that pure pearlite, unassociated with ferrite or independent cementite, is not reduced to a single line, but occupies a strip, the size of which corresponds to a variation of about 0·20 per cent. in the value of carbon between the appearance of the ferrite and that of the cementite.

Doubts are raised as to the identity of the independent cementite and the cementite of the pearlite. Sorby was doubtful on this point, and I for a long time have shared this uncertainty. But the analyses of Professor Ledebur[1] have established the indentity of its chemical composition. Messrs Arnold and Read state that they have distinguished in the residues obtained on attacking reheated steels by the Weyl[2] method, two carbides of different appearance—one white and shining, the other grey and dull. If this is so, then the difference of aspect can be explained as follows: viz., that the thin bright plates are compact and united, whilst the dull ones are badly packed together and rugged.

The fact of these two forms of cementite having the same genesis, seems, as we shall see presently, to indi-

[1] *Stahl und Eisen*, t. viii. p. 742, and t. xi. p. 294; *Bull. Soc. d'Encour.*, 4 série, t. vii. p. 665.

[2] *Jour. Chem. Soc.*, t. lxv. p. 788 (Aug. 1894).

cate unity. However, the hypothesis of several isomeric cementites, enunciated by Professor Campbell,[1] is not altogether excluded.

The characteristic property of cementite is its hardness, in the mineralogical sense of the word. Dr Müller[2] compares it to that of felspar (No. 6 in the scale of Mohs), which seems to me correct. The variable figures, 45 to 47, indicated by Professor Behrens[3] are explained—the harder by the presence of chromium, the softer by the state of disaggregation to which allusion was just now made. This hardness, superior to that of all the other constituents of carbon steels (including hardened and annealed steels), allows the cementite to be easily isolated by polishing in bas-relief,[4] provided that it is not scattered in a softer bed, in a state of division which exceeds the power of the microscope to define, and permits of no other control than that of immediate chemical analysis.

Polish-attack, so far as I have tried it, does not colour cementite.

Tincture of iodine, applied eight times, always under the conditions stated above, does not colour it, and leaves it shining with a silvery whiteness under perpendicular illumination.

The effect is the same, at ordinary temperatures, with nitric acid of 20 per cent. strength acting for

[1] *Jour. of I. and S. Inst.*, 1899, part ii. p. 223.

[2] *Stahl und Eisen*, t. viii. p. 292, 1888.

[3] *Op. cit.*

[4] Polishing with emery is enough to bring out the principal characteristics.

40 seconds at least, and 2 per cent. acid for a much longer time.

Figs. 42 and 43 (V × 1000), of a cementation

Fig. 42.—CEMENTED STEEL (1·57 per cent. carbon). Polish-attack. V × 1000 diameters.

steel containing about 1·60 per cent. carbon and prepared by polish-attack, show mixtures of cementite and pearlite. In fig. 42 the pearlite is exceptionally distinct, the lamellæ of cementite being often thick

enough to be measured. Photography, then, presents them in the form of threads relatively clear, bordered on the right and left by dark strips corresponding

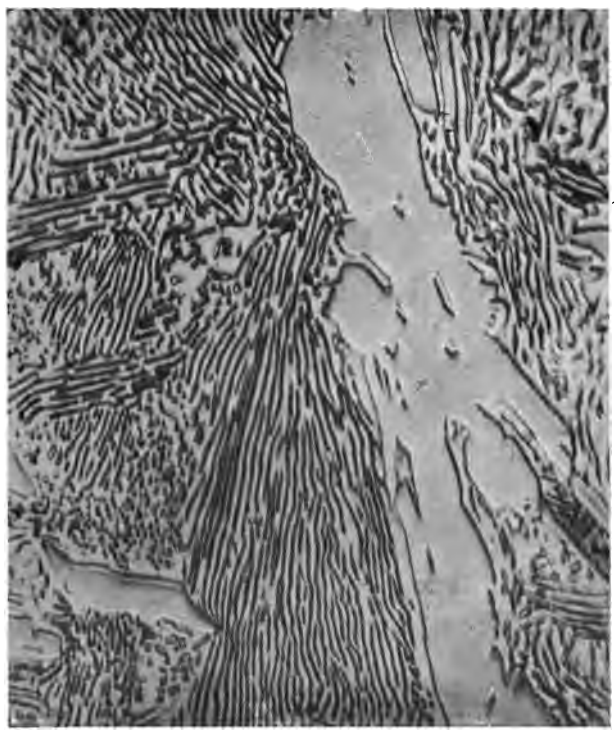


Fig. 43.—CEMENTED STEEL (1·57 per cent. carbon). Polish-attack. V × 1000 diameters.

to the rounding of the edges by polishing. The clear bands between the threads of cementite are ~~pearlite~~. The independent cementite is a little less ferrite

clear than the ferrite, but, as has been said, the respective clearness depends upon the focussing of the microscope. In fig. 43 the pearlite is very fine;

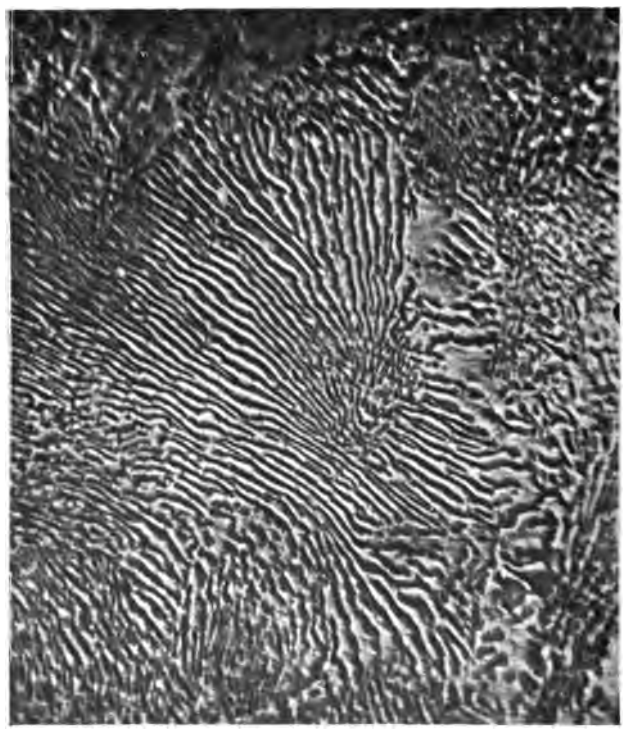


Fig. 44.—PEARLITE in 1·00 per cent. carbon steel. Forged, heated, and cooled slowly from 800° C. Polished only. V × 1000 diameters.

the lamellæ of cementite no longer have a horizontal surface, and are reduced to little rounded points and edges indicated by a simple dark track around the

point, the interstratified ferrite remaining clear. The independent cementite is in a semi-tint, and is distinguished by its form and dimensions—much greater in this case than those of the lamellæ of ferrite.

Fig. 44 is an example of pure pearlite in a steel of 1·00 per cent. carbon, forged, then reheated to 800° C., and submitted to simple polishing in bas-relief (V × 1000).

SORBITE.

The types of pearlite just described are perfectly clear. This is not invariably the case. It often happens, either in polishing in bas-relief, in polish-attack, or in the attack by tincture of iodine, that part of the pearlite is free, while others are differently coloured in yellow, brown, and blue. Often very fine and deep lamellæ of cementite are found, which are more or less continuous: this is what fig. 45 (polish-attack, V × 1500) ought to show, if the feebleness of the original photograph has been sufficiently reproduced, which is doubtful. But, of the other strips of pearlite, a single one cannot be seen with any magnification: the surface nearly uniformly tinted on the same grain is only slightly granular (fig. 46: polish-attack, V × 1500). In this last case I think that there is a distinct constituent, primarily by definition of a new order, because it cannot be resolved into two others, and I call it "*Sorbite*," in honour of the first pioneer of metallography.

Figs. 45, 46, and 47 have been taken of a steel with 1·00 per cent. carbon, forged to 12 mm. round, then reheated to 860°, and cooled in about half an

Fig. 45.—SORBITIC PEARLITE (lamellar) in 1·00 per cent. carbon steel. Polish-attack. V × 1500 diameters.

hour: it is the same steel as that of fig. 44. Sorbite is found side by side with pearlite in the same samples, and in fig. 47 one can see the juxtaposition

of the free pearlite, and of the pearlite passing to sorbite. This circumstance, and the fact that the immediate chemical analysis has always, in similar

Fig. 46.—SORBITIC PEARLITE (granular) in 1·00 per cent. carbon steel. Polish-attack. V × 1500 diameters.

steels, isolated the greater part of the carbon in the state of cementite, seems to prove that there is not, from a physico-chemical point of view, a great

difference between pearlite and sorbite. But sorbite may be obtained side by side with pearlite, by hastening the cooling without quenching, or by quenching a

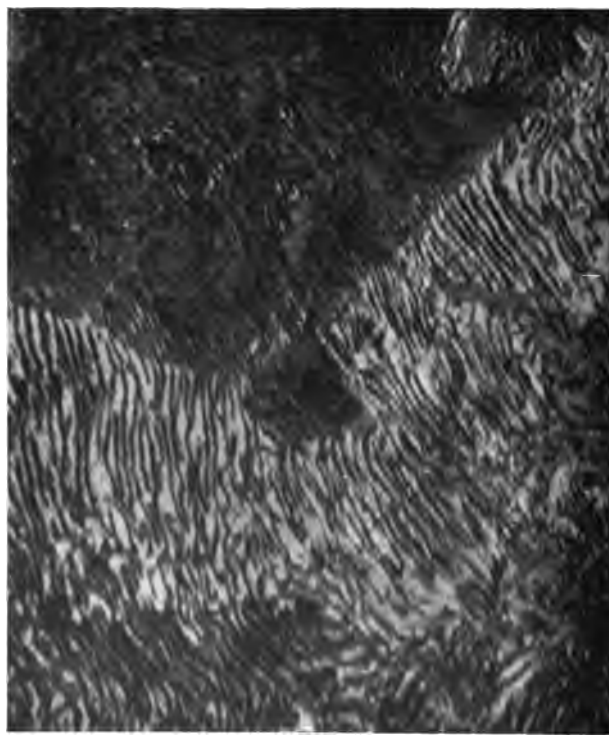


Fig. 47.—PEARLITE passing into SORBITE in 1·00 per cent. carbon steel. Polish-attack. V × 1500 diameters.

steel near the end of the critical interval, or, again, by reheating a quenched steel to about the same critical interval. For all these reasons sorbite may be

considered as pearlite which has not been able to separate into ferrite and cementite by reason of lack of time, or from some other cause, and it seems to be true that it ought to contain a little more "hardening" carbon than free pearlite.

It has been said that sorbite is an unimportant constituent, and several authors have not distinguished it from pearlite. I think that this is wrong, and for this reason, that in the first edition of this work[1] I did not give, with sufficient clearness, ideas which were perhaps slightly confused. But if we remember that sorbite, although it can only remain present in annealed steels up to a certain point, is essentially characteristic of "negative" quenchings, and that this procedure considerably improves the mechanical qualities of the steel, it would undoubtedly appear as legitimate and as necessary to distinguish sorbite from pearlite as it is to distinguish steels cooled naturally in air from steels which have been submitted to "negative" quenching (such as oil-hardening), double quenching, or tempering above blue heat. In my opinion the present methods in the manufacture of rails, etc., will very probably appear primitive in the future, and I hope that the greater quantity of pearlite in our steel will be replaced in future practice by sorbite.

From the point of view of micrography, sorbite is characterised by the absence of striæ and by the property of colouring rapidly by polish-attack or by tincture of iodine, after the application of the first drop of

[1] The First French Edition is, of course, that referred to.

tincture, even when that is diluted with its own volume of alcohol. It is easily seen in figs. 42 and 43 that all the transitions between sorbite and pearlite occur as the separation of the cementite is more or less advanced.

MARTENSITE.

The fourth constituent is that which is generally obtained by quick quenching in cold water from a temperature a little superior to the maximum temperature of the critical interval; it is not new, but the structure of it was not known. Dr Sorby[1] speaks, but with great reserve, of an extremely fine grain. Professor Behrens[2] mentions, in certain samples which he has examined, the existence of a soft polygonal network; but he has subsequently admitted that this network is the result of a beginning of superficial decarburisation. The researches of Professor Martens[3] apply to steels more or less soft, and show the unequal distribution of carbon in these metals, but not the ultimate structure of the hard portions. I published, in 1891,[4] a photograph of medium hard quenched steel, which showed in oblique light, after slight etching by nitric acid, a clear enough wavy appearance; but I misinterpreted this observation. In short, as the grain of the fracture diminishes by quenching so far as to disappear entirely to the eye in steels of high carbon content, we were

[1] *Jour. of I. and S. Inst.*, 1887, p. 276.
[2] *Op. cit.*, p. 151.
[3] *Trans. Amer. Inst. Eng.*, t. xxiii. p. 59.
[4] *Jour. of I. and S. Inst.*, 1891, part i. pl. viii.

convinced that the structure of quenched steels tended towards an amorphous state. It is, however, as we shall shortly see, very crystalline. A good example is

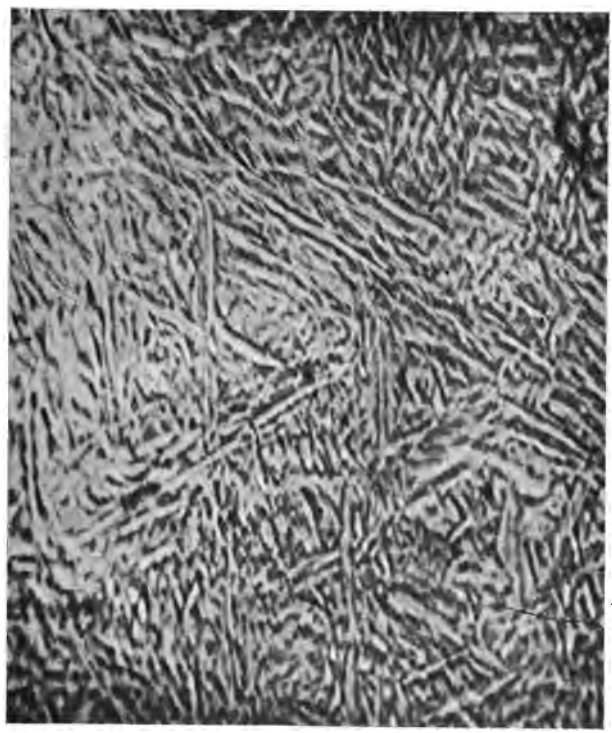


Fig. 48.—MARTENSITE in 0·45 per cent. carbon steel. Polish-attack. V × 1000 diameters.

afforded in steel with 0·45 per cent. carbon, heated to 825°, which, after slow cooling to 720°, is quenched in a freezing mixture at – 20°. The structure only appears

vaguely after polishing in bas-relief, from which it follows that the hardness of the mass is about uniform: polish-attack produces a good development (fig. 48, V × 1000). There are then seen faintly engraved groups of needles, or, rather, of rectilinear fibres, arranged parallel and sometimes separated by grains of a vermicular or worm-like character. Three systems of fibres, respectively parallel to the three sides of a triangle, cross frequently in the same region, and characterise, according to the opinion which M. Michel Lévy has given me, crystallites of the cubic system. I shall call this constituent "*Martensite*," after the name of Professor Martens, who has founded in Germany, since 1878, an independent centre of metallographic research, and has since followed these studies with much perseverance, talent, and success.

Martensite, isolated by polish-attack, is generally colourless, or only a pale yellow. On the other hand, the first drop of tincture of iodine causes a yellow, brown, or black coloration, according to the amount of carbon, the fibres always coming out bright.

In the presence of these inequalities of coloration and attack, there is doubt whether martensite really is a primary constituent. But the observed facts can be equally explained by the juxtaposition of fibres and the tendency of these fibres to throw the carbon out of their organism. The most recent facts tend to support the second hypothesis, without, however, eliminating all the doubt. It is thus in previously polished samples of steels, with very variable contents of carbon, that the

characteristic forms of martensite appear by simply annealing in an atmosphere of hydrogen.

Whatever it may be, martensite keeps its form as well in the carburised regions hardened by quenching extra soft steel as in the hardest steels, with this difference only, that, when the metal contains less carbon, the needles are longer and better differentiated, other things being equal. These are the forms which characterise it and enable it to be recognised under variations in its hardness: it is certainly not a definitely segregated combination of carbon and iron: it represents the crystalline organisation, under the influence of carbon, of one of the allotropic modifications of iron. However, the forms in which we find it through rapid cooling may be pseudomorphic, and I do not put them forward as a sufficient argument in favour of the real presence of the corresponding allotropic variety in the hardened metal.

Professor Howe has proposed for quenched steels (considered from a structural point of view) the name of "**Hardenite**." This name, on account of its very etymology, cannot be used to mean a constituent of variable hardness, and this is why I have adopted that of Martensite. But it may be useful to keep the word Hardenite, and to restrict it to Martensite saturated with carbon; and in this well-defined sense it is now used.

TROOSTITE.

The fifth constituent has been obtained by quenching steels during the critical interval $Ar_{1\cdot2\cdot3}$—that is to say, during the course of transformation. Suppose we take a metal with 0·45 per cent. carbon, heated to 825°, cooled slowly to 690°, and then quenched in water at ordinary temperature. Polishing on rouged parchment then shows on it projections in relief, fragments hollowed out, and, between the two, a border of variable size and of intermediate hardness. After the polish-attack, it is evident that the hard projections are martensite, and the soft parts hollowed out ferrite. As for the interposing borders, they are yellow, brown, blue, or black: but these colours form irregular marblings and develop less quickly than in the case of sorbite (fig. 49, V × 1000). Their structure is almost amorphous, slightly granular, and mammillated. In a hard steel, quenched during the critical interval, the ferrite is replaced by pearlite, itself surrounded by sorbite.

Tincture of iodine, either on the first or second application, acts like the polish-attack.

By calling this fifth constituent "*Troostite*" I have done homage to one of our French masters, who has rendered eminent services to metallurgical science.

Even in the conditions of quenching stated, troostite is not an absolutely necessary constituent: it may be absent, the more so when the steel is very soft. In hard steels it passes insensibly to sorbite, while its boundaries with martensite are absolutely definite.

Another means of preparing troostite consists of quenching the steel, at a temperature higher than that of the critical interval, in a liquid less active than cold

Fig. 49.—TROOSTITE and MARTENSITE in 0·45 per cent. carbon steel. V × 1000 diameters.

water—for instance, in oil or in boiling water. With cold water we get the same result if we increase the sizes of the pieces. In short, it is enough to diminish the

energy of the quenching in order to eliminate the martensite structure: and we get troostite. But the hardness is not proportionately diminished, and the troostite, like martensite, may pass through nearly the whole scale of the hardness of steels, according to the content of carbon and the rate of cooling.

If the dimensions of the pieces submitted to quenching are so great that the cooling is not uniform—for example, in the case of a steel of 1 per cent. carbon—one would obtain martensite on the surface, troostite in the centre, and a mixed intermediate zone.

Tempering also tends to transform martensite to troostite.

AUSTENITE.

If we consider all the circumstances which usually affect the hardness of steel after quenching—viz., the content of carbon, the initial temperature of quenching, and the chilling power of the bath—not only is the hardness of the martensite not increased when the carbon exceeds a certain amount, but next to it is seen a new constituent, much softer, which can be scratched with a sewing-needle and by apatite, perhaps even (but this is doubtful) by fluor-spar. I have dedicated this constituent to Sir W. Roberts-Austen, and called it "*Austenite.*"

To obtain it, the temperature of the steel must be above 1000°, and the temperature of the quenching bath a little below or just at 0° C., and the proportion of carbon must exceed 1·10 per cent. With carbon steels I have never obtained pure austenite, but mixtures

with martensite or hardenite. The amount of austenite increases with the amount of carbon from 1·10 per cent., when there is none, to about 1·60 per cent. or 1·80 per cent., when there may be 70 per cent. Beyond that, no matter what is done, it produces a separation of cementite, so that the proportion of dissolved carbon is diminished, and consequently the proportion of austenite no longer increases.

Contrary to what one would expect, austenite differentiates badly from hardenite by polishing in bas-relief, particularly if there is abundance of it.

Polish-attack does not colour it, but in the long run eats into it.

Tincture of iodine at once colours hardenite and austenite. The colorations of the two constituents are generally different, and each of them is homogeneous in the same grain; but they have nothing characteristic, and vary in neighbouring grains. For instance, a blue coloration may belong to hardenite in one grain, and to austenite in another.

The best agent for attack is dilute hydrochloric acid (10 per cent. solution), which colours hardenite and not austenite (fig. 50, V × 250). The attack requires several minutes, and does not influence each grain with the same speed: there is more regularity obtained by having the specimen connected, by means of a platinum wire, with the positive pole of a bi-chromate cell, a strip of platinum placed in the acid being connected with the negative pole. In this way the specimen becomes the anode, and the platinum the cathode.

Fig. 51 represents the same preparation magnified 1000 diameters after polish-attack. The hardenite has been coloured brown, which has allowed a good photo-

Fig. 50.—AUSTENITE and HARDENITE in 1·57 per cent. carbon steel. Polish-attack. V × 250 diameters.

graph to be taken; but there is an abnormal fact—the coloration only appears on a little part of the borders. This point has probably been accidentally tempered.

It is seen that the hardenite forms barbed laminæ with, generally, a mesial rib, slightly visible on the photograph. These laminæ are often parallel in two

Fig. 51.—AUSTENITE and HARDENITE in 1·57 per cent. carbon steel. Polish-attack. V × 1000 diameters.

principal oblique directions in the interior of the same grain. Austenite constitutes the remainder.

Another property quite characteristic of austenite is

that at a very low temperature it is transformed, with increase of volume, into hardenite. It is sufficient to immerse in liquid air for a few minutes a previously

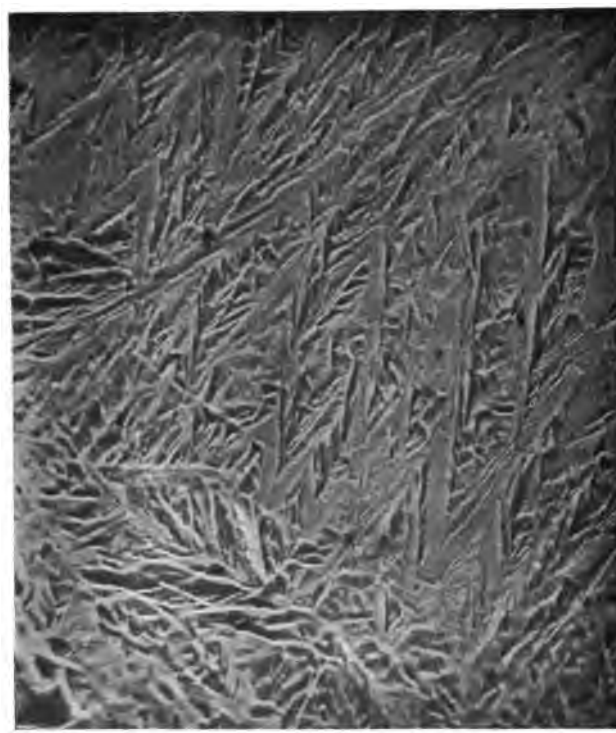


Fig. 52.—AUSTENITE STEEL after plain polishing and placing in liquid air. V × 250 diameters.

polished section containing austenite to produce the change: the austenite swells in slight relief above the hardenite (fig. 52, V × 250).

CHAPTER IV.

MICROGRAPHIC IDENTIFICATION OF CONSTITUENTS.

BEING now acquainted with the six constituents of carbon steels and the manner in which each of them behaves when submitted to the various methods of treatment described, we can resume the general course of the analysis in its application to the micrographic separation of these constituents, putting aside the preliminary operations of simple polishing.

1. Polishing in bas-relief on moistened rouged parchment: examining the preparation under the microscope.
2. Using the polish-attack on parchment, with sulphate of lime moistened with infusion of liquorice-root, or a 2 per cent. solution of nitrate of ammonia: examining under the microscope after every 500 passes to and fro if liquorice-root is used, and every 100 if nitrate is used. A certain amount of personal experience is indispensable. Variations in the pressure used in the operation of polishing have a very marked influence.

The presence or absence of coloration divides the constituents into two groups, intermediate between which is martensite.

a. CONSTITUENTS NOT COLOURED: Ferrite, Cementite, Martensite, or Austenite.

b. CONSTITUENTS COLOURED: Martensite, Troostite, or Sorbite.

Martensite, which only takes a yellowish tint, is recognised by its crystalline forms. An inexperienced observer might confound it with pearlite, especially in an oblique light, because it may exhibit the same phenomena of iridescence, and because its elements of structure may be of the same order of magnitude. The distinction is not, however, difficult. The principal needles of martensite are rectilinear, and frequently cut each other; whereas the layers of pearlite are mostly curvilinear, and never intersect.

Ferrite and cementite are distinguished by their very unequal degrees of hardness: after polishing, the former will be recessed, and the latter will be in relief with respect to all the other constituents. The mixtures of hardenite and austenite are only imperfectly resolved by polish-attack; but as the forms of hardenite are very characteristic, the least difference of level is a sufficient indication to a practised eye.

In the second group, troostite colours more slowly and less uniformly than sorbite; but the true criterion is that troostite is the companion of martensite, whilst sorbite is that of pearlite.

Iodine divides the constituents into two groups:—

a. CONSTITUENTS WHICH ARE NOT COLOURED: Ferrite or Cementite.

b. CONSTITUENTS WHICH ARE COLOURED: Sorbite, Troostite, Martensite, or Austenite.

In the first group the ferrite granulates, while the cementite keeps its polish. Ferrite subdivides into polygonal grains and the cementite often into plates, if these two constituents are not very much divided. In the second group sorbite colours more quickly than troostite, and troostite more quickly than martensite or austenite. The tone and intensity of the coloration always vary with the amount of carbon and the quantity of reagent employed. Martensite and austenite colour simultaneously but differently, in such a manner that the structure is well revealed.

This seems to me the best process to follow, and the one which leads most surely to the object in view. I do not wish, however, to induce anyone to believe that the proposed method equals in security and precision that of petrography or chemical analysis. In the first place, the various constituents of steel are not attacked in an absolute manner by the various reagents: neither are they equally attacked *during a given time.* Again, none of these constituents are exactly defined bodies. *Pure* ferrite does not exist, and we are obliged to consider it as nearly pure iron, which is vague. Cementite has indeed a formula, but it may be diluted with, or dissociated in, sorbite. There are not always distinct

limits between martensite and troostite, neither are there between troostite and sorbite, nor between sorbite and pearlite, in steels of certain hardness; neither is there between martensite and ferrite if the amount of carbon approximates to nil, and is distributed equally throughout the whole of the mass at the moment of quenching. One has frequently to deal with these forms of transition, which are the despair of all who attempt classification. But the classifications are no less necessary, and the divisions which I believe I have been able to establish will allow me, at least I hope so, to show more clearly how the structure of steels varies with their amount of carbon, and how it is transformed under the influence of heat and according to the rate of cooling.

CHAPTER V.

DETAILED EXAMINATION OF SELECTED STEELS.

My trials 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 cementation 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.	Extra-hard Cementation.
	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 by the curves in fig. 1, constructed in the same manner as I have employed in my former publications[1]: in which the abscissæ are the temperatures, and the ordinates the time, which the thermometer takes to pass through 1° of the thermo-electric pyrometer of M. le Chatelier (about 10° C.). The necessary figures are shown for the first four samples in the following 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 with 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.

[1] *Transformations du fer et du carbon dans les fers, les aciers et les fontes blanches*, Baudoin, Paris, 1888.

It should be remembered that the point Ar_1 corresponds to the recalescence point of Barrett, 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 mentioned 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 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,[2] and that of Professor Curie, even show the probable 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

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

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

of the ingots and the transformations caused by hammering, the reheating and the 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 various calorific 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 to sensibly 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 to sensibly 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 a calorific

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

treatment, care was taken to remove, in the cold, before polishing, 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.

After these general explanations, we approach the detailed examination of the steels selected.

I.—STEEL WITH 0·02 PER CENT. CARBON.

This metal is of Swedish manufacture, and contained originally 0·07 per cent. carbon. Mr 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 Mr Hadfield's laboratory before annealing. The amount of carbon after annealing is only given reservedly. But as polishing in bas-relief shows no cementite, it is clear there is practically no carbon present.

Reheating and quenching 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 53, V × 100) are the scoriæ. The ferrite is

Fig. 53.—IRON (0·02 per cent. carbon). Etched with iodine. V × 100 diameters.

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 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, Mr Stead has obtained important results (see *Jour. of I. and S. Inst.*, 1898, part i. pp. 145–149, and part. ii. pp. 137–154). In another direction M. Cartaud and I have written a long article on the Crystallography of Iron in the *Annales des Mines*, Aug. 1900.

II.—STEEL WITH 0·14 PER CENT. CARBON.

A. Forged Metal.—After polishing in bas-relief (fig. 54, V × 100), numerous scattered threads, more or less distorted, project and appear relatively dark on the photograph, taken a little above the mean focussing point. These are the strips of pearlite or sorbite, or 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,

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

cutting out the ferrite in contiguous polyhedric grains, leaving in slight relief the strips of pearlite which are almost always external (fig. 55, 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 ob-

Fig. 54.—FORGED STEEL (0·14 per cent. carbon). Polished in bas-relief. V × 100 diameters.

jective proves, which is easily detected by raising or depressing the object-glass of the microscope.

After attack by four successive applications of tincture of iodine (fig. 56, 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 en-

graved 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 obtained by

Fig. 55.—FORGED STEEL (0·14 per cent. carbon). Polished with calcium sulphate and water. V × 100 diameters.

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 can 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 make out exactly, in the structure of very soft steel, the joints and filaments of pearlite

Fig. 56.—CARBON 0·14 per cent. Etched with tincture of iodine. V × 100 diameters.

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

Fig. 57.—CARBON 0·14 per cent. Reheated to 750° C. Etched with 20 per cent. nitric acid. V × 100 diameters.

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 practically sufficient precision. The three figures—

Nos. 57, 58, and 59—represent, with the usual enlargement of 100 diameters, and after attack by 20 per cent. nitric acid, the same forged steel reheated to 750° (57), reheated to 1015° (58), and reheated to 1330° (59).

C. Influence of Quenching.—One sample was heated to 960°, a temperature at which the point Ac_3 is

Fig. 58.—CARBON 0·14 per cent. Reheated to 1015° C. Etched with 20 per cent. nitric acid. V × 100 diameters.

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. 60, V × 100). Analysis shows that these inclusions are now no longer of pearlite, as in the annealed steel;

Fig. 59.—CARBON 0·14 per cent. Reheated to 1330° C. Etched with 20 per cent. nitric acid. V × 100 diameters.

Fig. 60.—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.

they are not scratched by a sewing-needle: they consist of martensite.

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°,

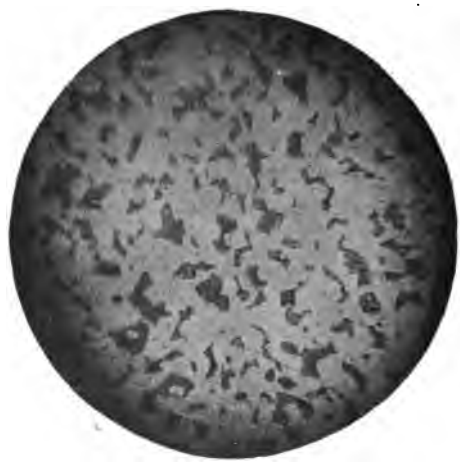


Fig. 61.—CARBON 0·14 per cent. Reheated to 960° C., cooled to 770°, and quenched in water at 15° C. Polish-attack. V × 100 diameters.

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. 61, V. × 100), 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 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.

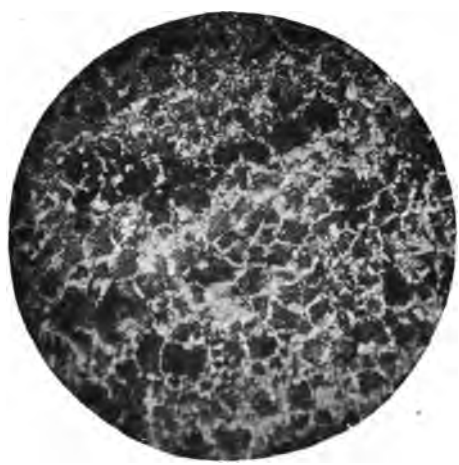


Fig. 62.—CARBON 0·14 per cent. Reheated to 1000° C., and quenched in water at 15° C. Polish-attack. Iodine or nitric acid. V × 100 diameters.

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 intermediate between

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

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

Fig. 63.—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. 62, 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 rondelle 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. 63, 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

Fig. 64.—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.

light shows it sometimes dark, sometimes 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. 64, 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 Messrs 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. 65, V × 1000). The pearlite conforms to the general description which has been given of it. Attack resolves the ferrite into grains.

B. Influence of Reheating.—I shall pass rapidly over this question, which I have treated elsewhere. According as the temperature of reheating is raised, the pearlite forms polyhedrons more and more regular, which the ferrite envelops with a network more and more perfect, and into the interior of which it thrusts parallel ramifications.

Fig. 66 (V × 100) represents the metal simply forged. After annealing at 1015° (fig. 67, V × 100), 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. 68, 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. 69) of the feeble enlargement of 20 diameters is necessary. Here the light is oblique, and, accordingly, the appearances to which we are accustomed are inverted: it is the ferrite which shows

black, and the pearlite light. In photographs taken from objects illuminated in vertical light, it is, on the contrary, the ferrite which is white. Returning to fig.

Fig. 65.—FORGED STEEL. CARBON 0·45 per cent. Polish-attack. V × 1000 diameters.

68, the envelopes of ferrite and their ramifications are easily seen: these envelopes are divided by an axial line, which is perhaps not sufficiently clear in the

Fig. 66.—CARBON 0·45 per cent. Reheated to 750° C. Polish-attack. V × 100 diameters.

Fig. 67.—CARBON 0·45 per cent. Reheated to 1015° C. V × 100 diameters.

Fig. 68.—CARBON 0·45 per cent. Reheated to 1390° C. V × 100 diameters.

Fig. 69.—CARBON 0·45 per cent. Reheated to 1390° C. Oblique illumination. × 20 diameters.

reproduction. These are the lines of contact which sometimes open into veritable fissures, where the introduction of oxidising gases at raised temperatures may form a layer of scoriæ when this state is obtained. Burnt steel cannot be restored by any heat treatment, and always keeps its brittleness.

There are no metals where the temperature of heating inscribes itself more clearly than in steels of medium carbon content.

C. Influence of Quenching.—

(*a*) *Heating to variable temperatures, followed by quenching at the same temperature in water at 15°.*

This series of assays will allow us to follow the diffusions of carbon during the heating.

One plate was heated and quenched at 730°. Polishing in bas-relief isolates a large broken-up network of ferrite. Polish-attack shows, round this ferrite, a border, more or less wide, of troostite, coloured brown with blue spots (fig. 70, V × 100; 49, V × 1000). The rest is martensite, but, in the preparation photographed at 1000 diameters, the resolution of this last constituent has purposely not been pushed very far, in order that the troostite could be better distinguished.

The distribution of martensite and troostite is very irregular. Fig. 71 (V × 100) shows another region where the troostite is much more abundant than in

fig. 70. We see that at about 20° above the inverse point of recalescence the carbon was not yet diffused throughout the whole mass of the steel.

At 1000°, on the contrary, the diffusion of carbon is practically complete. Steel quenched at this temperature takes almost throughout a nearly specular polish on rouged parchment, but the sample is split to the

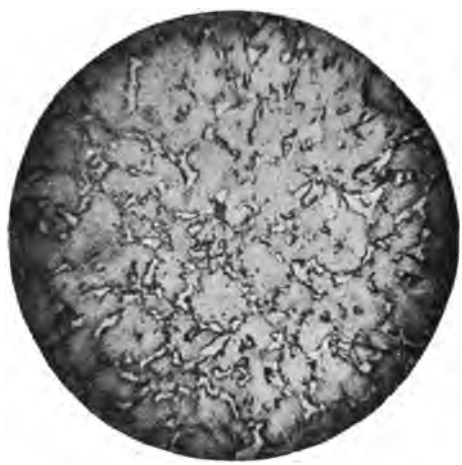


Fig. 70.—CARBON 0·45 per cent. Reheated and quenched from 730° C. Polish-attack. V × 100 diameters. See also Fig. 49.

centre along a diameter. The whole mass is in the state of martensite, except in certain places where, in the neighbourhood of an inclusion of slag, remains of troostite are found, together with traces of ferrite. The needles of martensite are longer and more regularly grouped than in the sample quenched at 730°. The boundaries of the crystalline groupings, besides generally

being much confused, correspond to the places of minimum compactness, which nitric acid etches black by a deeper attack, and which may be transformed into veritable cracks. The cracks, microscopic or not, may equally follow the direction of the needles. There exists, then, in quenched steel a veritable network of surfaces of weakness: this network must be the result of the

Fig. 71.—CARBON 0·45 per cent. Reheated and quenched from 770° C. Polish-attack. Shows more troostite than Fig. 70. V × 100 diameters.

combination of maximum tension, during the rapid contraction, with the planes of minimum resistance created by the actual structure. It is clearly enough shown by placing the polished specimen under a layer of water a few millimetres deep, and allowing one, two, or three drops successively of concentrated nitric acid to drop upon it through the water—a process of attack

used by Mr Werth, and which certainly prevents the solution of the hardening carbon. The polyhedric elements appear to approach to a pentagonal dodecahedron, like the grains of soft annealed steels. These are the grains of quenched steel: rudimentary grains when the temperature of quenching has been just sufficient, but which, in proportion as this temperature

Fig. 72.—CARBON 0·45 per cent. Reheated to, and quenched from, 1225° C. Polish-attack and nitric acid. V × 800 diameters.

is raised, become larger and larger and more and more distinct. These are they which appear in fractures.

A third sample was heated and quenched at 1225°. The diffusion of carbon is naturally more complete than it was in that quenched at 1000°, and with better reason. The martensite is then the only constituent, but the crystallites in it are better and more clearly developed. Their needles are easily resolved by polish-

attack (fig. 72, V × 800). A beautiful wavy appearance is also obtained in an oblique light with small magnification (fig. 73, V × 20). This preparation readily lends itself to the study of the origin of the grain which has been described above. All the varieties of surfaces of weakness can be found there:—1st, the crack visible to the naked eye (fig. 74, V × 20.

Fig 73.—CARBON 0·45 per cent. Reheated to, and quenched from, 1225° C. Polish-attack and nitric acid. × 20 diameters. Oblique illumination.

polish-attack); 2nd, the line of least resistance at the common edge of two crystalline developments (fig. 72, already described); 3rd, the line of least compactness or of greatest tension, which, in crossing a crystalline region following a different direction from that of natural cleavages, shows its presence by a deeper attack of a certain width between the isolated needles

Fig. 74.—CARBON 0·45 per cent. Reheated to, and quenched from, 1225° C. Polish-attack. V × 20 diameters.

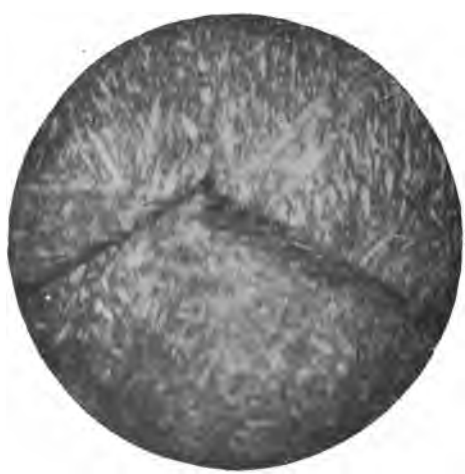


Fig. 75.—CARBON 0·45 per cent. Reheated to, and quenched from, 1225° C. Etched by tincture of iodine. * O × 800 diameters.

* O means oblique illumination ; V, vertical.

(fig. 75, V × 800: attack by iodine). Finally, fig. 76 (V × 100: also attack by iodine) allows one to trace with oblique light the course of a fissure usually, but not always, coinciding with the borders of the crystalline portions of the same orientation.

A fourth sample, heated and quenched at 1325°,

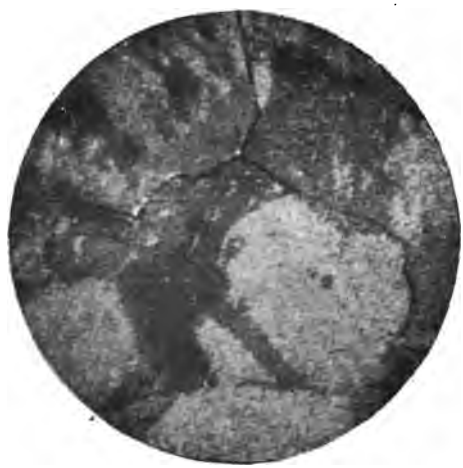


Fig. 76.—CARBON 0·45 per cent. Reheated to, and quenched from, 1225° C. Etched with tincture of iodine. O × 100 diameters.

only differs from the preceding by a new growth of crystallites, and demands no special attention.

(*b*) *Heating at a constant temperature (845°), followed by quenching at variable temperatures in water at 15°.*

The constant temperature of heating—825°—has been chosen so as to obtain the complete diffusion of carbon and the perfect accomplishment of all the

transformations, whilst avoiding to the greatest extent possible the formation of a network of surfaces of weakness. This series of assays will allow us to follow the concentration of the carbon during the cooling, as we have just now followed its dissemination during the heating, and to show how this concentration takes place here entirely in the zone of the critical points.

Fig. 77.—CARBON 0·45 per cent. Quenched at 690° C. Polished in bas-relief. V × 100 diameters.

A first rondelle has been quenched at 720°—that is to say, before the beginning of the double critical point $Ar_{3\cdot2}$ (see curve, fig. F)—and after a slow cooling from 825° to 720°. Polish-attack gives almost throughout martensite, and, in some regions (only where the carbon was more scarce), a residue of ferrite accompanied by troostite occasionally in filaments.

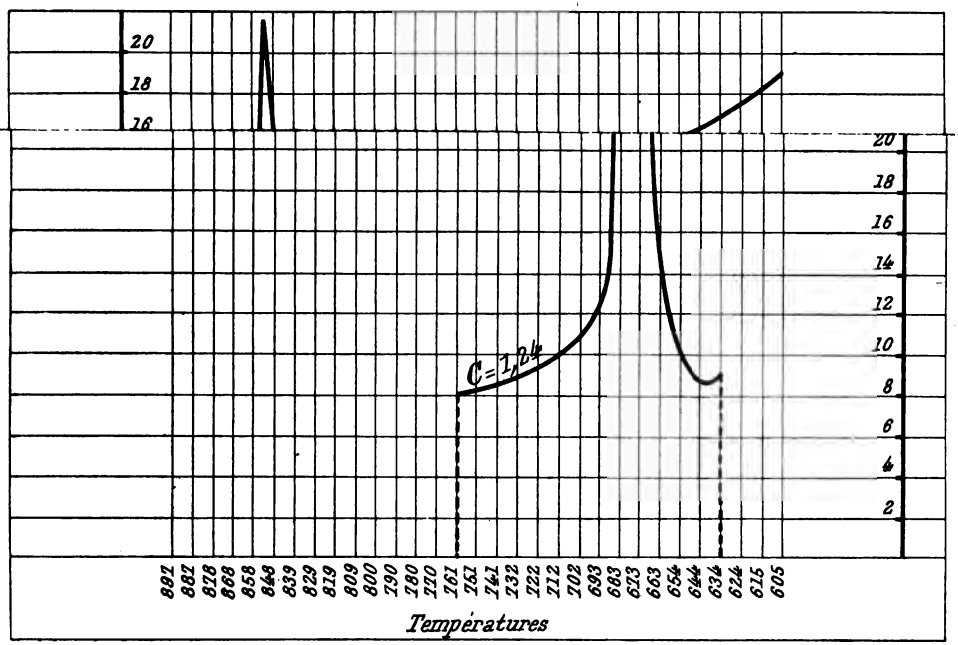


Fig. F.—Critical Points of Steel.

[*To face page* 136.

A second sample was quenched at 690°, towards the maximum of $Ar_{3\cdot2}$. The ferrite has become more abundant, and polish in bas-relief shows it in very numerous strips, which, however, do not yet join together. Liquorice-root, iodine, and nitric acid very nearly reproduce the structure which we have already

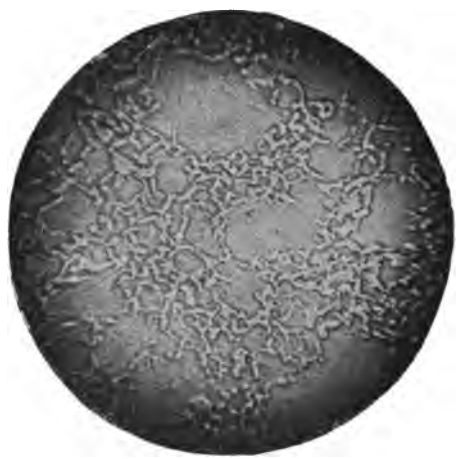


Fig. 78.—CARBON 0·45 per cent. Quenched from 670° C. Polished in bas-relief. V × 100 diameters.

found in the same steel heated and quenched at 730°.

A third sample was quenched at 670° between the maximum of $Ar_{3\cdot2}$ and the beginning of Ar_1. The general structure is still of the same type, but the isolated strips are united, and now form a thick and continuous cellular network easily developed by polishing in bas-relief (fig. 78, V × 100).

A fourth sample was quenched at 650° during the course of the recalescence Ar_1. The area of ferrite has again become what it was in the forged metal, and the appearance of the preparation polished in bas-relief is still the same. But attack for about two seconds by 20 per cent. nitric acid colours certain grains black and others only yellow (fig. 79, V × 100). Some are

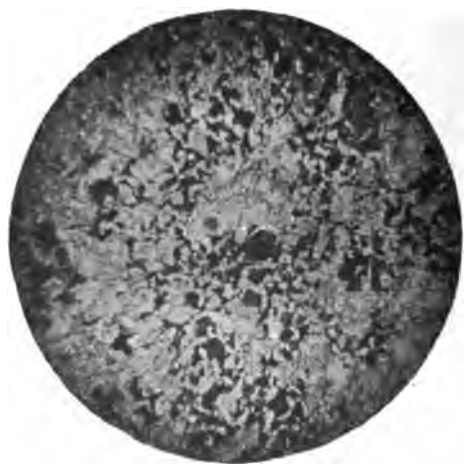


Fig. 79.—CARBON 0·45 per cent. Quenched from 650° C. Etched with 20 per cent. nitric acid. V × 100 diameters.

pearlite, that is to say, non-hardened steel; the others martensite, that is to say, hardened steel. These latter are attacked first at the centre, which is more clearly organised than the edges.

The recalescence passed, say at 640°, quenching does not produce any visible effect, and we return to the structure of the forged steel.

(*c*) *Heating at a constant temperature* (825°), *followed*

Fig. 80.—CARBON 0·45 per cent. Reheated to 825° C., and quenched on cooling at 720° C. in a freezing mixture. Polish-attack. V × 1000 diameters.

by quenching at a constant temperature (720°) *in different media.*

The baths chosen were:—

1. A freezing mixture at $-20°$ C.
2. Water at 15° C.
3. Water at 85° C., the effect of which is analogous to cold oil.
4. Melting lead.

We have seen from what has preceded that, after previous heating at 825°, to make sure of the diffusion of carbon, and quenching at 720°, the hardened metal was almost exclusively formed of martensite. It is also martensite, and with better reason, after quenching in a freezing mixture.

But the mild quenchings at 15° C. admit the formation of a network of ferrite almost as if the quenching had been done at a lower temperature in cold water. Fig. 80 (polish-attack: V × 1000) shows in white the network of ferrite, the remainder taking a marbled patina of different colorations. The needles of martensite have nearly disappeared, while the layers of pearlite have not had time to isolate. We have here to deal with forms of transition between troostite and sorbite.

(*d*) *Influence of Tempering.*

If one tempers, from blue on the one side to brown on the other, a plate quenched at 720° in cold water (after preliminary heating to 825°), polish-attack still allows one to catch a glimpse of the forms of martensite, but only vaguely and feebly. To obtain fig. 81

(V × 1000), it has been necessary to rub for a very long time, and to exaggerate the contrasts by strongly

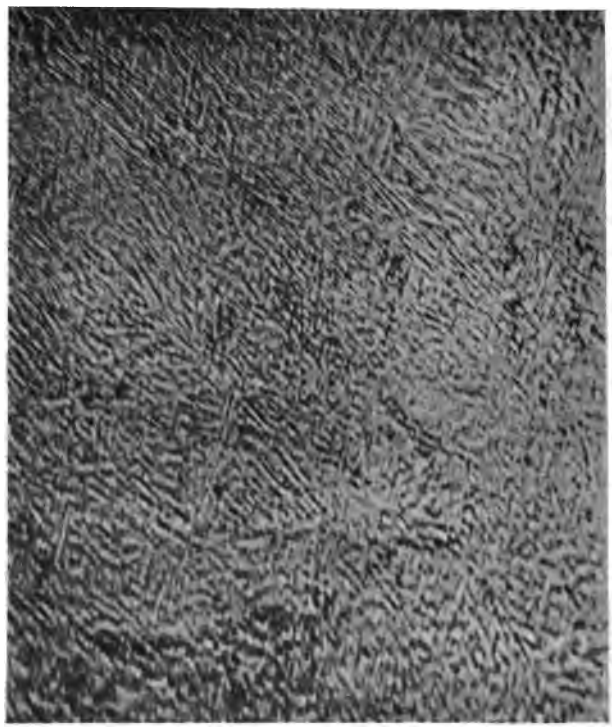


Fig. 81.—CARBON 0·45 per cent. Reheated to 825° C., and quenched from 720° C., and tempered to between blue and brown colour. Polish-attack. V × 1000 diameters.

diaphragming the light. Attack with nitric acid shows the triangles of martensite a little better, at least in some places. But the forms of martensite are here

only the almost effaced image of the anterior structure, and no longer correspond to the actual state of the associations of iron and carbon. Chemical analysis

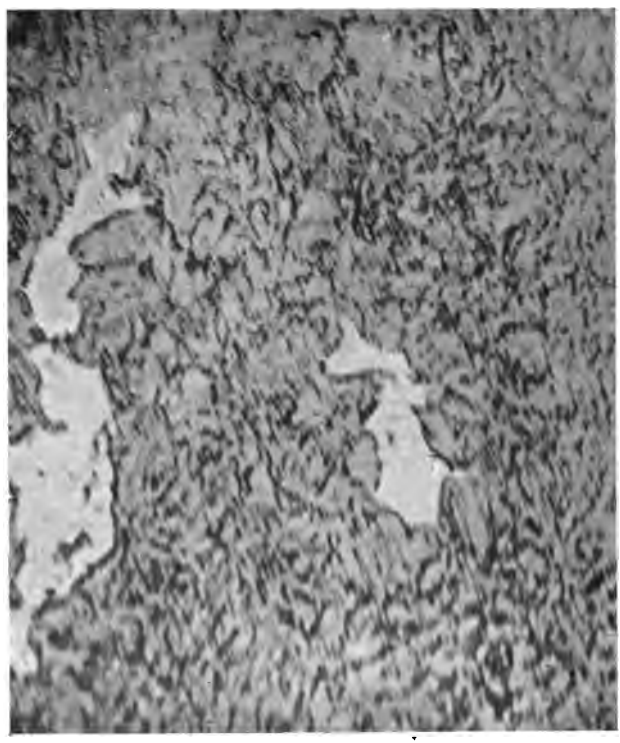


Fig. 82.—CARBON 0·45 per cent. Reheated to 825° C., and quenched from 720° C., and heated to 670° C. Polish-attack. V × 1000 diameters.

teaches us that cementite has been reconstituted: but it is in such a state of division that the microscope cannot identify it. A drop of iodine gives the whole

preparation different colours mixed in the most complete disorder.

On taking the tempering to 670°, we bring a little order into this chaos. The ferrite and cementite tend to separate, the former collecting into badly defined grains, around which the cementite is mixed up with sorbite (fig. 82: polish-attack: V × 1000). But the separation remains very imperfect.

Another rondelle, reheated and tempered at 600° after quenching at a white heat, is very instructive. There are met with side by side a structure resembling a sort of fish-bone, well-preserved remains of the hardening structure, the grains of ferrite are encircled without any sharp demarcation by a network of sorbite, and a region in a state of transformation where the new organisation is installed in the still visible framework of the old.

IV.—STEEL WITH 1·25 PER CENT. CARBON.

A. Forged Metal.—There is accidentally found, towards the axis of the specimen examined, a harder part which is reproduced in the majority of the samples.

This hard region is a good example of pearlite of the type shown in fig. 44, p. 86. In the rest of the plate the cementite has collected, probably by a forging terminated well below Ar_1, into spheroids of variable size (fig. 83: polish in bas-relief: V × 1000). In a harder steel forged under the same conditions, the

spheroids of cementite form parallel fibres, due to the forging, like the fibres of scoriæ in puddled iron.

B. Influence of Reheating.—Towards 750° the

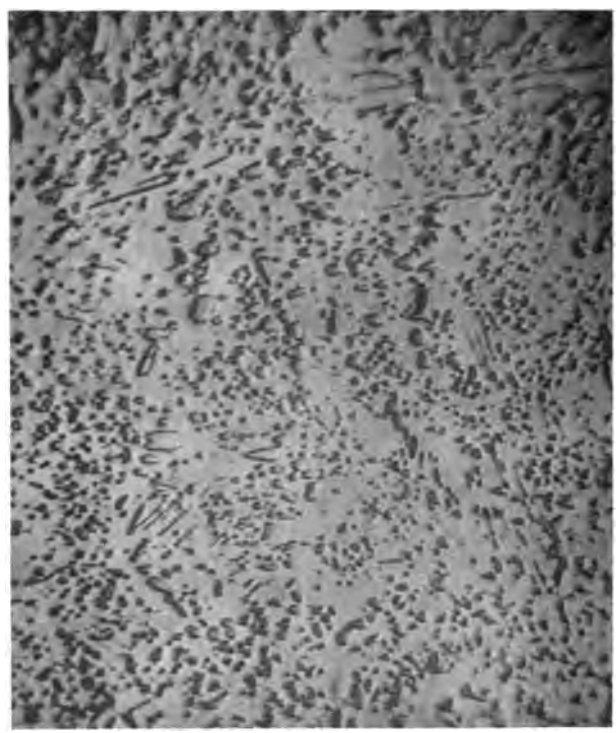


Fig. 83.—CARBON 1·25 per cent. Forged steel. Polished in bas-relief. V × 1000 diameters.

peculiar mixture we have just described, which we call *granulated* pearlite, is transformed to normal pearlite, passing more or less to sorbite, together with strips of independent cementite.

By reheating to 1015°, the hard nucleus is burnt and divided into beautiful polyhedrons by a continuous and cellular network of cementite. At 1330° the mass is altogether burnt: the polyhedrons are of great size and frequently separated by a narrow border of ferrite, probably due to the introduction of oxidising gases. As for the remainder, the principal mass is always pearlite, whatever the temperature of annealing may be: only, the higher this temperature has been, the more the isolated parts tend to develop.

The best means for this class of steels of determining by metallography the temperature to which they have been heated, or that at the end of forging, is to make an attack, after plain polishing by an immersion in nitric acid at 36° Baumé. The attack is limited by the passiveness of the iron: it would, however, be too strong if it were a question of studying the details of the pearlite. But, for exactly the same reason, it furnished in an oblique light, under the small enlargement of about 50 diameters, a wavy appearance so much the more developed as the temperature sought is raised.

C. Influence of Quenching.—A rondelle was heated and quenched at 735° in water at 15°. Hardening at this temperature produces fractures.[1] Three

[1] The whole secret of hardening without cracking appears to be in quenching before the end of the transformations (during the heating) or after their finish (during the cooling). But that is easier said than done when the eye is the only guide. Hence the necessity for specialists. Again, these specialists are often found in error, when the point to which they have been accustomed is changed. Then they declare that the new steel is bad.

fissures visible to the naked eye start from a common point near the centre, and lead towards the edge without reaching it. These fissures are broken,

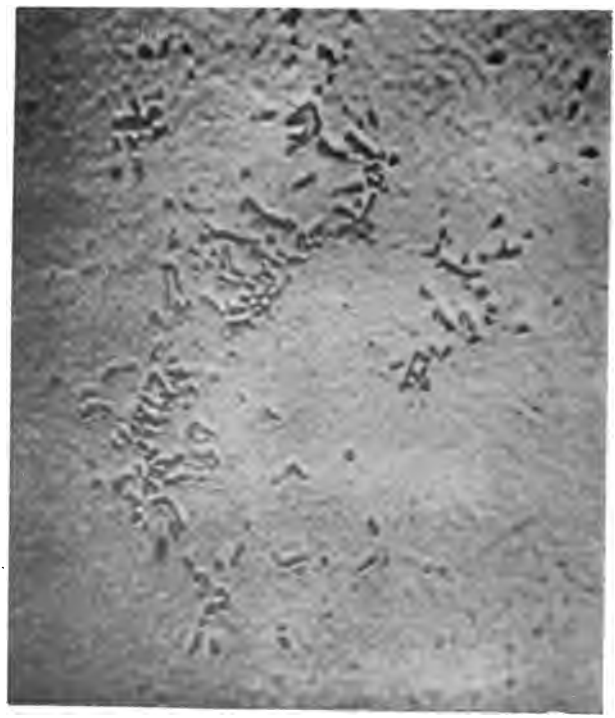


Fig. 84.—CARBON 1·25 per cent. Reheated to 735° C., and quenched in water at 15° C. Polish-attack. V × 1000 diameters.

like those of fig. 74, and are often divided into isolated regular polyhedrons. Polishing on rouged parchment leaves in relief strips harder than quenched steel, and these remain bright after a 5-seconds' attack

by 20 per cent. nitric acid. These are the residues of cementite.

As for martensite, which is in this case hardenite, it keeps its usual forms and shows them by known characteristics; but the needles are smaller and less distinct than we have previously found (fig. 84: polish-attack: V × 1000).

Fig. 85.—CARBON 1·25 per cent. Quenched from 1050° C. Polish-attack. V × 250 diameters.

The cementite which has separated a little above the critical point is dark on the photograph. The presence of this cementite about the point Ac_1 is an interesting fact which will be useful in drawing conclusions.

If the quenching be done towards 1050° or above, it remains no longer cementite, and austenite appears (fig. 85: V × 250). On this photographic preparation, which in reality is from another slightly different

steel, the structure has been developed by polish-attack. The austenite was slightly in intaglio, and the photograph, taken a little below the mean focussing point, shows it black while the hardenite is clear. It is seen that this last form of barbed needles, whose principals are often parallel to two or three directions, dominate on a given grain.

Quenching during recalescence, always in water at 15° C., gives a mixture of isolated transformed grains, and grains not transformed, with others of intermediate character. Those not transformed are naturally hardenite with a surplus of independent cementite; those completely transformed before the quenching took place are pearlite. The hardenite is surrounded by troostite, the pearlite by sorbite, and the passage from troostite to sorbite takes place in insensible degrees.

V.—STEEL WITH 1·57 PER CENT. CARBON.

A. The Natural Cemented Metal.—The description of this has been given previously (pp. 84 and 85), accompanied by figs. 42 and 43.

B. Influence of Quenching.—

(*a*) *Negative Quenching.*

The cementation finished, a few bars 12 or 13 mm. in diameter were drawn from the cementation furnace at maximum temperature and cooled in the air. For bars of this dimension and hardness, cooling in the air can be

considered a negative quenching. The micro-structure (fig. 86: polish-attack: V × 1000) shows the lamella

Fig. 86. — CARBON 1·57 per cent. Cemented steel drawn from a cementation furnace and cooled in air. Polish-attack. V × 1000 diameters.

of clear cementite in parallel groups, and the remainder is sorbite beginning to separate into pearlite.

(*b*) *Positive Quenching.*

On heating and quenching at a temperature higher than $Ac_{3\cdot2\cdot1}$ and lower than about 1000°, the cementite is incompletely dissolved at the beginning of the quenching, and it is found in the principal mass of hardenite, which constitutes the quenched metal.

If the quenching is done from a temperature of about 1050° in iced water or in a freezing mixture of snow and common salt, or in mercury cooled below zero, only traces of cementite are left, in the shape of very fine parallel barbs at the edges of the grains: around them and attached to these same edges there may be little cores of troostite. All the surplus—that is to say, nearly the whole of the preparation—is constituted of a mixture of hardenite and austenite, which has been described on pp. 99–102, and illustrated by figs. 50, 51, and 52.

The variation of the temperature of quenching between about 1050° and 1300° does not appear to have any marked influence. But if, the initial temperature of quenching being above 1050°, a bath of warm water is used—say at 70°—the independent cementite is isolated, and consequently austenite is no longer present. Austenite can still be obtained with water at a temperature of 15° to 20° when using very small specimens; but the result is uncertain, the more so that oxidation is rapid at the high temperatures needed, and the decarburisation of the specimen is great. It is a much surer method for the preparation of

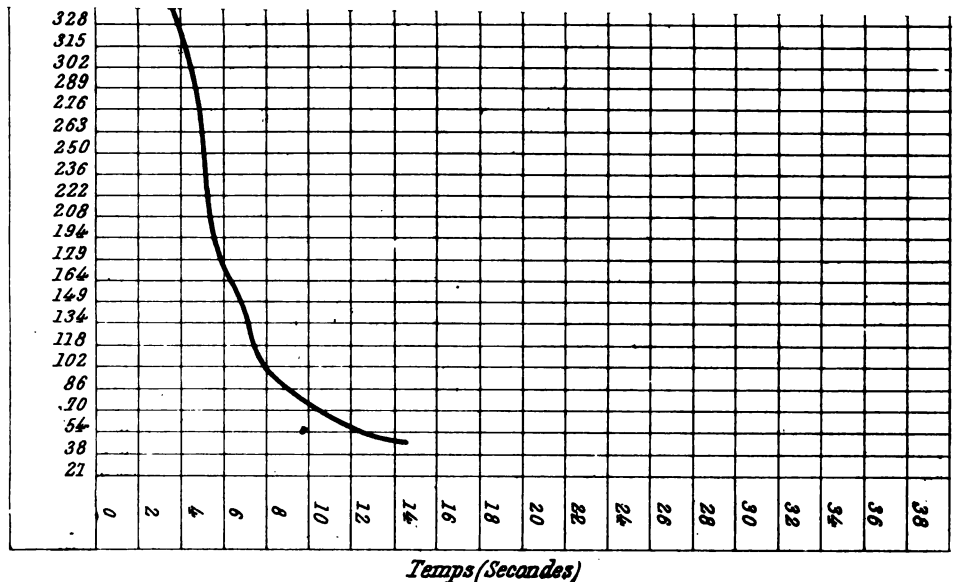


Fig. G.—Heating Curve of Harden Steel containing 1·57 per cent. Carbon.

[*To face page* 151,

austenite to quench in iced water pieces 6 to 7 mm. thick. In every case the structure must be examined on a section sufficiently removed from the surface if the heating has been done in an oxidising atmosphere. Thus we see that it is convenient that the crystalline structure before quenching should be as gross as possible. The best process to obtain austenite is to take the steel direct from the cementation furnace at maximum temperature and quench it directly in iced water. In this manner, Mr Grobot, Director of the Assaily Steel-works, has been good enough to prepare for me the samples which have led to the discovery of austenite.

C. Influence of Tempering.—Two bars quenched in iced water from about 1050° were placed, with a Le Chatelier couple between them, in a porcelain tube enveloped in a covering of asbestos. The whole was placed in the muffle tube of a Mermet furnace (previously heated to a constant temperature) and then heated. The results were plotted on a chart, and indicated the successive periods necessary for the temperature to rise 1° on the pyrometer. These are given in the diagram (fig. G). It is seen that, under the conditions of the experiment (other conditions giving different results), the rate of heating shows three accelerations. The first, towards 140°, is feeble, and therefore doubtful; the second begins towards 275°, and, increasing progressively just towards 400°, reaches a very distinct maximum between 400° and 500°, and ends rapidly between 500° and 540°; the third is less pronounced, and was not reproduced in another trial.

It begins towards 610°: then at about 700° we find the triple point known as $Ac_{3\cdot2\cdot1}$.

Knowing in this manner the main temperature of the course of tempering, four samples, previously quenched from 1050° in iced water, were tempered at increasing temperatures under the same conditions of heating, and the micro-structures were studied after tempering. The micro-structure after quenching is given in figs. 50 and 51, and described in connection with the definition of austenite.

The tempering of the first samples was stopped at 275°, the steel being of pale yellow colour—that is to say, before the commencement of any great acceleration in the recalescence. Hardenite, which before tempering could not be scratched by a sewing-needle and was not coloured by polish-attack, is now slightly scratched—less than austenite—and is browned by the polish-attack with liquorice-root. Thus figs. 50 and 51 are reproduced (pp. 100 and 101).

The tempering of the second sample was stopped at 395° at the beginning of the recalescence, the surface of the metal being blue. The former structure is slightly revealed by polishing in bas-relief. The differences of level are very feeble, but they are inverted, the austenite now being rather in relief on the hardenite: a scratch with a sewing-needle is nearly regular, perhaps a little more marked on the barbs. Polish-attack easily colours these same barbs, and borders them with a jagged strip very strongly coloured. It leaves a residue of austenite covered with numerous spots and cleavages

visible only under very strong magnification parallel to the directions of the needles of hardenite (fig. 87, V × 250).

The tempering of the third sample was stopped at 495°—that is to say, at the end of the maximum of the recalescence. By polish-attack the primitive structure

Fig. 87.—CARBON 1·57 per cent. Quenched from 1050° C. in ice water, and reheated to 395° C. Polish-attack. V × 250 diameters.

remains confusedly recognisable (fig. 88, V × 250), but the demarcations greatly efface it.

The tempering of the fourth was done at 620°. Polish-attack gives a coloration nearly homogeneous, on which the old structure can still be distinctly seen.

Altogether, tempering progressively transforms har-

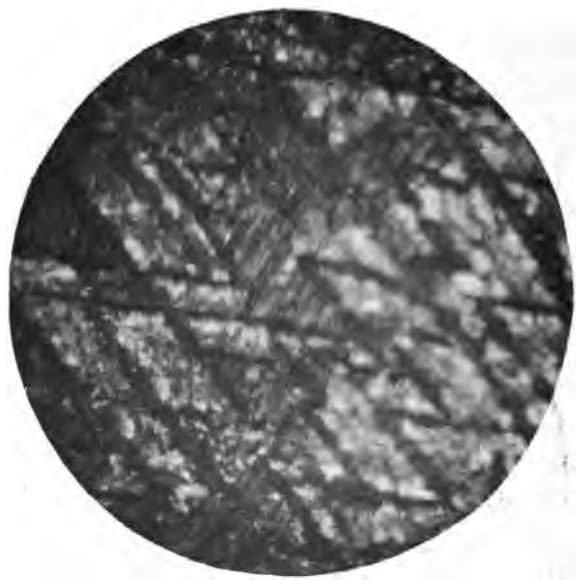


Fig. 88.—CARBON 1·57 per cent. Quenched from 1050° C. in ice water, and reheated to 495° C. Polish-attack. V × 250 diameters.

denite and austenite—the first more quickly than the second—to troostite, then to sorbite.

CHAPTER VI.

CONCLUSIONS.

Theoretical Conclusions.—When the first edition of this work was published, the author was not fully prepared to draw all the conclusions possible. The principal conclusion was that the assimilation of quenched steel to solid solutions was placed beyond all doubt, and most convincing examples of the assimilations of solid solutions to liquid solutions were found.

But the progress of physico-chemistry was needed in order to understand this conclusion in all its clearness. An article by Le Chatelier,[1] and a discussion on a paper by Sauveur on "The Micro-structure of Steel and the Current Theories of Hardening,"[2] the conference of the author at the Congress of Methods of Testing held at Stockholm in 1897, the fourth and principally the fifth report of Roberts-Austen to the Alloys Committee of the Mechanical Engineers,[3] several essays of Juptner,[4] and lastly, an important work of Professor Roozeboom,[5] mark the principal records.

[1] *Revue Générale des Sciences pures et appliqués*, 15 Jan. 1897.
[2] *Trans. Amer. Inst. Min. Eng.*, t. xxvii. p. 584.
[3] *Proc. Inst. Mech. Eng.*, Feb. 1897 and Feb. 1899.
[4] *Stahl und Eisen*, 1898, fasc. 11/2/3/22.
[5] *Bull. Soc. d'Encour.*, Nov. 1900.

Omitting the introductory work, I shall consider things from the point at which they are to-day after the work of co-ordination of Roozeboom. Not that these facts are absolutely definitive; but if there are any difficulties to clear up, the modifications which become necessary will probably be of secondary importance.

Fig. H shows diagrammatically the curves of transformation (or equilibrium) of carburetted irons between 0 per cent. and 2 per cent. carbon. The abscissæ are the carbon, and the ordinates the temperature. AQ is the curve of the commencement of solidification, after the experiments of Roberts-Austen and Stansfield.

The curve AE represents the end of solidification, and has not been determined experimentally: it has been introduced by Professor Roozeboom as a result of his theoretical hypothesis constantly verified in similar cases.

ES shows the commencement of the separation of the cementite. Its existence is proved by the fact that in steels of considerable carbon content the cementite does not appear when quenched below a certain temperature, and this has been confirmed by the thermal method in Roberts-Austen's trials.

The branch GO from the point G (Ar_3) shows the transformation of the γ iron to β iron, and the commencement of the separation of ferrite in soft steels.

MO from the point M (Ar_2) is the transformation from β to α, or, more precisely, the beginning of the transformation which really is progressive.

OS from the point where GO and MO meet gives the transformation from γ to α, and the commencement of the separation of ferrite in steels of ordinary carbon content.

The point S, where OS and SE intersect, is a eutectic point. The horizontal line PSK passing through this point corresponds to the recalescence and a simultaneous deposit of ferrite and cementite, as pearlite.

These four branches GO, MO, OS, and PSK have all been determined by several concordant experiments.

Now the different branches of diagram, fig. H, divide the total area into partial areas, which bear the name "*domains*," and which we will define.

AESOG is the domain in which the iron, in the γ state, contains the carbon, free or otherwise, in solid solution. This solid solution is crystalline. From the study M. Cartaud and I have made, these crystals appear to be fragments of cubes mutually limited and intercepted by numerous lamellæ of twin crystals, or in a less perfect state of crystallites of the same type we meet with in martensite, in non-magnetic manganese steels, and certain steels high in nickel. We give them the name of "mixed γ crystals."

The γ crystals are associated with—

1. A liquid residue rich in carbon in the domain QAE.
2. The cementite in the domain ESK.
3. Free β iron „ „ GOM.
4. „ α „ „ „ MOSP.

Below PSK we have the domain of mixed ferrite and cementite which divides into two sub-domains:—ferrite and pearlite on the left of the vertical ST passing through the eutectic S, and pearlite and cementite on the right of this vertical.

It is now easy to trace the transformations, as much physico-chemical as structural, in a steel of given carbon content. It is sufficient to trace as abscissæ this carbon content, and to follow it in connection with the branches of the diagram.

We will choose as nearly as possible the same steels whose micro-structure we have studied above.

I.—STEEL WITH 0·14 PER CENT. CARBON.

The solidification starts at b_1 and finishes at c_1. From c_1 to d_1 the metal is in the state of mixed γ crystals. Quenching gives pure martensite. At d_1 we have the transformation of γ iron to β iron—that is to say, the β iron separates from a solid solution. The separation is progressive, and the solid solution equally increases in carbon: it contains at the point e_1 a percentage equal to the abscissæ MO. Beginning at e_1, the β iron transforms to α iron, while the solid solution continues to enrich in carbon, the α iron separating out. This continues until the strength of the solution reaches the value PS, practically about 0·85. In f_1 the solid solution transforms to pearlite, on account of the simultaneous separation of the very divided ferrite and cementite.

The examination of the micro-structure is, as we have seen, in accordance with these deductions, and, except on one point, the steel with 0·14 per cent. carbon quenched between c_1 and d_1 ought to be pure martensite, whereas we really have a mixture of martensite with a proportion of ferrite crossing at d_1 towards c_1. If, then, this ferrite is not isolated during the little time the cooling lasted, and if it really existed in the metal at the initial temperature of quenching, we must add to the diagram a line like AR starting from the point of solidification of pure iron and corresponding to a separation of γ iron from the solid solution (fig. I). One can still imagine a similar branch starting from the critical point of Ball and corresponding to a transformation of δ iron to γ iron. This requires elucidation.

II.—STEEL WITH 0·45 PER CENT. CARBON.

The solidification commences at b_2 and ends at c_2. Between c_2 and d_2 the metal remains in the state of mixed γ crystals, and quenching yields pure martensite. At d_2 the iron begins to change to the α state, and the separation continues progressively just to f_2, the solid solution correspondingly enriching in carbon to 0·85 per cent. At f_2 this solid solution transforms to pearlite, as above.

In this case the experiment is in absolute agreement with the micro-structures.

III.—STEEL WITH 0·85 PER CENT. CARBON.

We have here only a single transformation at S of the mixed crystals to pearlite. We find pure martensite in hardened steel between c_3 and S, and pure pearlite below S, whatever the method of cooling. This is also what occurs practically.

IV.—STEEL WITH 1·57 PER CENT. CARBON.

Solidification begins at b_4 and ends at c_4. Methodical tests have not been made of this region, the existence of which was never suspected before the intervention of Roozeboom. I have two or three sections which were quenched under the necessary conditions, and I notice that the micrographic indications of troostite are visible all along the joints. It would not be impossible for these parts to represent the liquid residue at the moment of immersion. But this is hypothetical.

Between c_4 and d_4 we know that the quickest quenching, or that which keeps them best *in statu quo*, gives a mixture of austenite and hardenite. Nothing in the diagram indicates the presence of a new constituent. Professor Roozeboom has investigated this matter, and has proposed very ingenious explanations. Another explanation may be given without the formation of a new phase. It must be noticed that the martensite cannot represent the *intact* mixed γ crystals: they keep their crystalline forms well, but, since it is

magnetic, part of the iron must return to the α state. Austenite, on the contrary, which one has reasons to consider non-magnetic, can represent *intact* mixed γ crystals. That granted, we see that the greatest proportion of carbon which can be in solid solution in iron is 1·80 to about 2 per cent.—a quantity insufficient by itself to maintain, on account of the osmotic pressure involved, the whole of the iron in the γ state, so rapid is the cooling. The transformations then will commence, but they will not commence at random. They begin with cleavage planes of mixed crystals, and continue until the increase of volume, resulting from a partial transformation from γ iron to β iron, would have added to the osmotic pressure of the dissolved carbon a mechanical pressure sufficient to arrest the transformation of the γ iron throughout the remainder of the mass. The hardenite, the rivers of which can correspond, and in reality do correspond, to the cleavages, represents the quantity of iron which the partial transformation has necessitated to suppress the transformation of the surplus — that is to say, of the austenite. And that proportion of hardenite would be as much greater as the amount of total carbon is less, in forming the sole constituent, when the osmotic and mechanical pressure are together insufficient to maintain entirely in the γ state an aliquot fraction of the mass.

Between d_4 and S_4 cementite separates progressively: the amount of dissolved carbon diminishes correspondingly. Quenching between these two points will give

mixtures of cementite and hardenite, with eventually a quantity of intervening austenite.

At last, below f_4 there is no longer a mixture of pearlite and cementite.

In the above conclusions we have not found a place for troostite or sorbite, nor is this necessary if these constituents are transition forms. The existence of these is inevitable, since the transformations require a certain length of time, and they are of great practical importance on account of the mechanical properties which they confer on steel; but the diagram which shows the definite states of equilibrium does not show them.

However, if we remember that troostite does not usually mix with martensite, and if we consider the conditions of its genesis, we can suppose that it corresponds to some transitory association between β iron and carbon. It is a question which requires investigation.

In conclusion, we have just seen that, except for a few remaining difficulties which require further research, the results of the study of the micro-structure of steel are in perfect accord with the deductions to be drawn from the diagram of equilibrium curves. Taken alone, this diagram could be constructed in its main lines, as quenching fixes the initial state and suppresses the ulterior transformation, at least in part.

On the other hand, the diagram of equilibrium curves could have been constructed independently of micrography—*e.g.*, by the thermal method. In fact, it has been by the co-operation of the two methods that they have been controlled and, in certain circumstances, completed.

Practical Conclusions.—Setting aside all theoretical speculation, we have seen that—

1. The temperature of heating,
2. The temperature of quenching,
3. The rapidity of cooling,

that is to say, the principal circumstances of the calorific treatment of steels, reveal themselves in the variations of structure, with a precision which the mere inspection of fractures is certainly far from furnishing. But to get any industrial results from these facts, it would be necessary to have the different aspects of the structure correlated with the corresponding mechanical properties.

The practical application of metallography to Metallurgists and Engineers requires, then, for each interesting metal, a preparatory study. But, when this study is made, it will be easy to determine with sufficient exactness the calorific treatment undergone by a finished article or piece of metal, to see if this treatment, which is of first importance, has conformed or not to given specified rules, to rectify it if needful, and to take the responsibility in case of rejection. Mr Albert Sauveur, who edits that excellent special review *The Metal-*

lographist, had already, when engineer to the Illinois Steel Company, organised a regular system of micrographic analysis.[1]

For industrial applications the complete method which has been described in this paper will not, as a rule, be necessary. Like the general rules of chemical analysis, this method allows, according to circumstances, simplifications suggested by experience. It is a method of investigation which may be laborious in the case of original research, but which will, eventually, be much simplified.

[1] *Trans. Amer. Soc. of Min. Eng.*, t. xxii. p. 546.

APPENDIX I.

APPARATUS EMPLOYED FOR MICRO-PHOTOGRAPHY.

THE whole of the appliances figured are by Nachet & Fils, of Paris, and they give complete satisfaction (fig. K).

The microscope is a present-day model for the examination of transparent substances.

A total reflection prism is fixed above the objective to illuminate opaque bodies. That arrangement, however, has the inconvenience of reducing the utility of the microscope, and microscopes such as are made by Nachet and others at the present time are certainly to be preferred.

The microscope stands on a table of suitable height, under a movable verticle camera which slides between two uprights. I consider the verticle to be very much superior to the horizontal camera for studying metals. The chief reason for this is, that pieces of varying shape, seldom terminated by two parallel planes, have to be examined. In that case the mounting of the specimen is done very quickly and correctly by putting a little soft wax (sculptors' modelling cement) upon a glass plate and embedding the specimen in the wax. The

levelling is managed by means of two pieces of glass

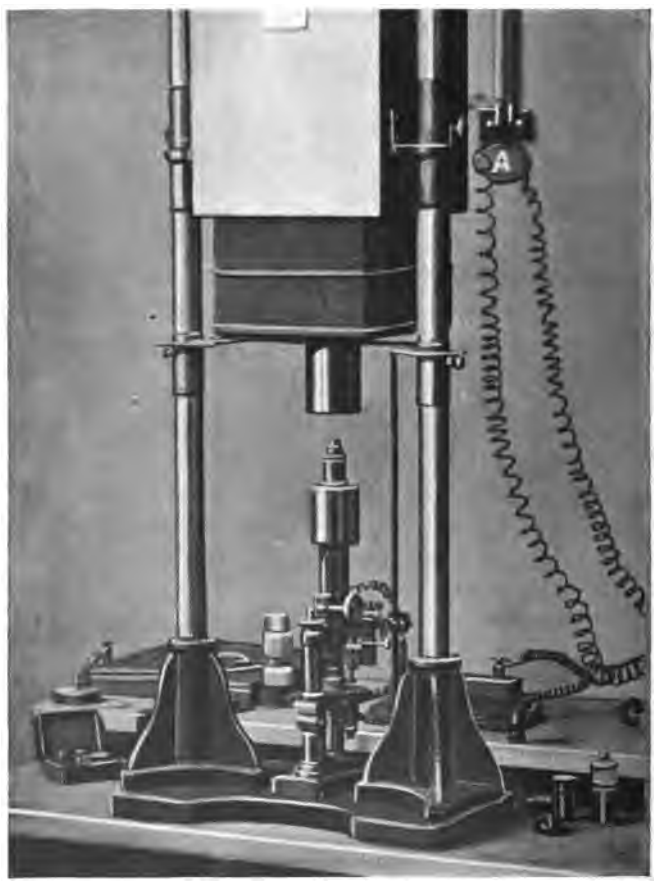


Fig. J.

tube of equal height placed one on each side of the specimen.

A sample so mounted would often move if it were placed vertically, and it would be necessary then to replace the wax by a cement which would harden, or by cutting the metal so that it has two parallel planes. Besides, with the vertical camera the microscope is in the best position for use, and when an interesting point is noticed and the photograph of the specimen required, the camera is simply let down and joined to the fixed socket of the microscope.

The micrometer focussing screw has a forked aluminium lever, and this is worked with a rod.

The space between the uprights gives freedom for the movement of the camera. The only inconvenience, which is, however, not serious, is that it is necessary to stand upon a stool to bring the camera into focus for photographing.

Nachet's No. 2 eye-piece is generally used, and, when measurements are required, a micrometer eye-piece.

For photographing, the ordinary eye-piece is replaced by one for projection. According as the draw-tube is extended or shortened, and as the projection eye-piece itself is more or less extended, the magnification is modified, the extension of the camera being constant.

To obtain the least magnification with each objective, the projection eye-piece is removed and a velvet tube introduced into the draw-tube to avoid reflection upon the photographic plate.

The set of objectives comprises Nachet's Nos. 0, 2, 3, 5, 7, and 9.

These objectives are generally used to give the following respective magnifications, with suitable adjustment of the camera:—

0	. . .	6 to 20	diameters.
2	. . .	20 „ 50	„
3	. . .	50 „ 150	„
5	. . .	150 „ 400	„
7	. . .	400 „ 600	„
9	. . .	600 „ 2000	„

No. 7 is not so suitable as the others for the examination of opaque bodies, and one can do without it. All the others are necessary. Nos. 0 and 2 are not used with the prism. It is therefore removed, if vertical light is used, and replaced by a cover glass placed at 45° between the specimen and the objective. The objective No. 0 is mounted on the socket of the camera as in ordinary cameras, and not upon the microscope.

For illumination I have, for several years, used exclusively a good 6-volt electric lamp, with facilities for increasing to 8 volts. The lamp is fed by accumulators, and a varying resistance is interposed in the circuit. For examining specimens a mild light is preferable, and is not so wearying to the eyesight. A resistance is therefore interposed in the circuit, and about 4 volts used. For photographing the voltage is increased to 8. This consumes 3 ampères.

The prism can be lighted directly by the lamp without the introduction of a condenser, and all colour is thus avoided; but owing to refraction there are a few

shadows cast upon the surface, and these shadows are rather troublesome where the specimens are very brilliant and uniform. Under such conditions a magnifying glass is placed between the lamp and the opening of the microscope, avoiding focussing on the prism.

A vertical partition with two movable shutters must be arranged in front of the prism, to do away with or, at least, diminish any colour.

The length of time for exposures is but little longer with No. 9 objective than with No. 3, for the same extension of the camera. The exposures may vary from ½ to 20 minutes, according to the magnification required for a given objective, the distance of the lamp, and the opening of the partition. The colour and the brilliancy of the specimen have naturally a great influence.

No definite rule can be laid down for the time any object must be exposed, and it needs long experience to expose correctly, and even then it is easy to be mistaken.

The photographic plates I usually employ are Lumière's orthochromatic plates for yellow and green screens. A green screen should give good results, especially for photographing, but it has the inconvenience of lengthening the time of exposure.

APPENDIX II.

THE RELATIVE SOFTNESS OF AUSTENITE.

As there have been repeated expressions of doubt as to the reliability of the method of determining the relative hardness of austenite compared with that of hardenite, I have pleasure in more fully explaining how the demonstration was effected.

I first obtained a bar of steel containing 1·55 per cent. carbon in its interior, and a gradually decreasing amount towards the outside.

A small piece of it was forged into a rectangular form having the dimensions 19 mm. × 9 mm. × 10 mm. This was heated to 1050° C., and was then quenched in mercury at a temperature of −9° C. When cold, one of the surfaces was ground down to remove the outer layer where superficial decarburisation had been effected during the reheating. It was then polished, but was not subjected to any attack by etching reagents. A pointed sewing-needle was then repeatedly drawn across it from end to end with even pressure.

The photograph (fig. K), taken when the object was illuminated by oblique rays of light, indicates clearly enough that the needle scratched the steel in the region

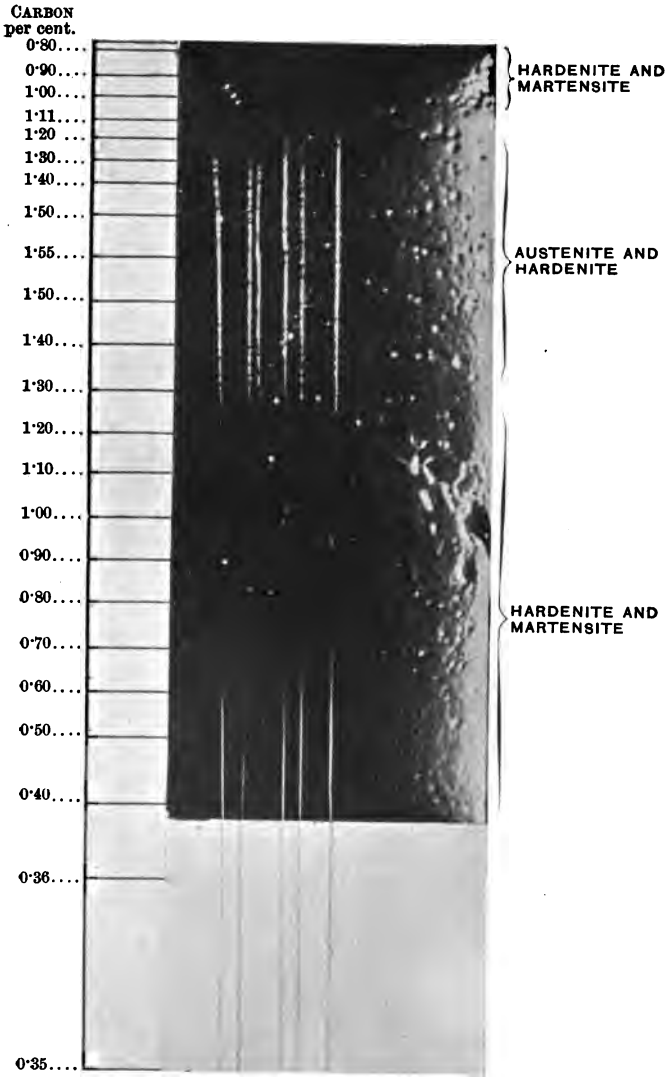

CARBON
per cent.
0·80....
0·90....
1·00....
1·11....
1·20 ...
1·30....
1·40....
1·50....
1·55....
1·50....
1·40....
1·30....
1·20....
1·10....
1·00....
0·90....
0·80....
0·70....
0·60....
0·50....
0·40....
0·36....
0·35....
HARDENITE AND MARTENSITE
AUSTENITE AND HARDENITE
HARDENITE AND MARTENSITE

Fig. K.

where the carbon was between 0·37 per cent. and 0·70 per cent., and that it would not scratch it where the carbon varied between 0·70 per cent. and 1·20 per cent. It commenced again to scratch it feebly in the region of 1·25 per cent. carbon, and continued to scratch it with a gradually increasing depth till 1·45 per cent. carbon was present, and apparently continued to scratch it with equal ease till the carbon reached the maximum of 1·55 per cent. carbon, after which the material gradually got harder and harder with the diminishing carbon, until at a point where it approached 1·15 per cent. carbon it was too hard for the needle to make any impression.

Judging from the relative distinctness of the scratches, it is clearly shown that in the region of highest carbon the metal is more readily scratched than where it is only 0·37 per cent.

The central portions where the needle readily scratches contain austenite.

The question as to the relative hardness of the needle does not arise, for the scratches were all made by the same needle, and they all indicate beyond the slighest shade of doubt that austenite is considerably softer than hardenite. It resembles, in some of its properties, Hadfield's manganese steel after quenching, for both can be scratched with a needle, although they cannot be cut or machined by the hardest tool steels; and austenite steel is relatively little susceptible to magnetic influences compared with hardenite, but not to the same extent as manganese steel.

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