PART III.

THE MICROGRAPHIC ANALYSIS OF CARBON STEELS.

CHAPTER I.

ROUGH POLISHING.

Preliminary Operations.—Neglecting the study of fractures, to which, unfortunately, we can apply only a feeble degree of 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 found that, even by following these rules with the greatest possible care, we neither 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.

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,

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

the paper and the glue must themselves be free from particles likely to scratch the soft iron.

Preparation of Emery Papers. — 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 do not matter). 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]

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

Albumen	72 c.c.
Water	24 ,,

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

In addition to the first rubbings, which should efface the file marks, and for which commercial papers fully suffice, I usually rub the 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 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 (which is the only part which can be used) by levigation. M. Henrivaux, the well-known manager of the Saint-Gobain Glass Works, has spared me this task by giving me a sample of oxalate rouge, which is excellent, and 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 Steiglitz, 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 confuse 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 at the present time three-fourths of the labour 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]

[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 Dr 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 Dr Stead (*Jour. of I. and S. Inst.*, part i. p. 292 (1894); *Metallographist*, vol. iii. p. 220, July 1900).

There is or should be finally obtained a distinctly specular surface, even when the metal is not homogeneous, provided that the cloth be not too wet, or that the rouge be not 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, is 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 portion of the work to present.

CHAPTER II.

FINE POLISHING

includes three successive operations:—

Polishing in Bas-Relief.
"Polish-Attack."
Etching.

Polishing 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 the unequal relief of its constituents. This is what I call polishing in bas-relief. Sorby,[1] Martens,[2] and Behrens[3] 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 the desired result, it is necessary to polish on a substratum, elastic enough to allow of the production of inequalities, and delicate enough to show the finest

[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.

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]

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 following

[1] It would be interesting, for the further development of the method, to draw up a table of powders of sufficient fineness and of graduated hardness like the mineralogical scale of Mohs.

device. 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.

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 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 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 Ivan Werlein who directed my attention to it as an example of certain prejudices of workmen. But workshop recipes, although sometimes

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

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 liquorice extract by macerating 10 grammes 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. It has also a very serious drawback: like all vegetable products, it 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 solutions, sometimes too strong, sometimes not strong enough.

It is necessary, therefore, to have a more reliable reagent. This I have found, with the help 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 neither too wet nor 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 inspect the sample periodically, in order to stop when the desired effect is obtained: 200 rubbings backwards and forwards often suffice.

Etching.—After the polish-attack, it is best to rub the sample again with rouge on the revolving disc, in order to decolorise it and to efface the reliefs. The examination is completed by etching with chemicals. The reagents which it is possible to use may be divided into three classes: acid solutions, solutions of the halogens, and salt solutions.

The final result of the complete solution of carburised irons in the principal acids is comparatively well known, but the initial processes of a very incomplete attack by a dilute acid remain more obscure, and are probably more complex. 2, 5, and 10 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), and 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 2 and 20 per cent.[1] By immersing the sample in concentrated acid, in which it becomes at first passive, and subsequently sprinkling it with plenty of water, 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

[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.

with absolute alcohol, or a very slow and irregular attack will be obtained from which no conclusions can be drawn. An ordinary tincture is suitable. I apply it by drops 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 made if needful. The first application is often sufficient and sometimes even a little too energetic, and in such cases a solution twice diluted can then be used, as recommended by Sauveur.

Copper sulphate, bichloride of mercury, gold chloride, and potassium platinochloride—salts which replace the dissolved iron by a deposit of their specific metal—have not given very encouraging results, but the number of experiments being necessarily limited, I may easily have missed favourable conditions, and would not wish to dissuade anybody from repeating the trials.

I now only retain the following reagents, tincture of iodine, and, accessorily, nitric acid, and, in particular cases, hydrochloric acid and picric 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 oil-immersion objective has to be used.

With the aid of these three successive operations—polishing in bas-relief, polish-attack, and etching,—and by the reactions to which they give rise, we can define the primary constituents of carbon steels.