[CONTRIBUTION FROM THE LABORATORY OF ANALYTICAL CHEMISTRY, UNIVERSITY OF MICHIGAN.]

FURTHER STUDY ON THE INFLUENCE OF HEAT TREATMENT AND CARBON UPON THE SOLUBILITY OF PHOSPHORUS IN STEEL.

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ABOUT a year ago we published a paper upon the same subject as that of the present research.[1] At the time of our previous work we did not have a pyrometer on which we could depend, so that we were unable to state with accuracy the exact treatment to which the material under examination had been subjected. Since the time of our previous work we have obtained an accurate Le Chatelier pyrometer so that we have been able to heat specimens up to 1,100°, with probable error of not over 5°.

The heat treatment to which the specimens in the table given below were subjected are as follows: The metal called "annealed" was raised to between 900° and 1,000° and then allowed to cool slowly. The specimens called "quenched from x°" were placed in a porcelain-lined iron tube with the thermal junction of the Le Chatelier pyrometer in contact with the specimen. The portion of the tube containing the specimen was slowly heated by means of a Hoskins' Gasolene Furnace until the desired temperature was reached. The time required for heating the specimens was usually from twenty-five to forty minutes. When the desired temperature was reached, the tube was tilted up so that the specimen dropped directly into ice-water at a temperature of from 4° to 5°. The specimens so prepared were then either drilled or pounded sufficiently fine to enable them to be used for chemical examination.

The method of analysis employed is somewhat different from that described in our previous paper. Instead of treating first with neutral mercuric chloride, then after filtering and washing the precipitated mercury, treating this latter with four per cent. hydrochloric acid, and finally volatilizing the mercury and determining the phosphorus in the residue, we have shortened the

[1] *Am. Chem. J.*, 18, 719.

process and determined only the phosphorus soluble in a slightly acid solution of mercuric chloride. The objections to the process employed last year are principally, the length of time necessary for handling the precipitates, and to a less extent the fact that the amount of phosphorus soluble in hydrochloric acid increases somewhat with the time of digestion, thus necessitating the adoption of an empirical length of time of treatment with the reagent. In the method which we have employed in the present work, we have demonstrated by numerous experiments that the length of time of standing has no appreciable effect on the solubility of the phosphorus in a given sample. A specimen digested one and one-half hours gave 0.096 per cent. soluble, while another sample of the same specimen gave 0.093 per cent. after standing forty hours.

The special solutions employed in the determination of the soluble phosphorus were as follows: The 'two per cent. hydrochloric' is twenty cc. hydrochloric acid (sp. gr. 1.20) made up to one liter. The ferric chloride solution is 8.15 grams of iron wire containing 0.050 per cent. phosphorus dissolved in 1.20 nitric acid, boiled, and evaporated to dryness with excess of hydrochloric acid, sufficient to convert it to ferric chloride. After evaporation to dryness the ferric chloride was taken up with twenty-five cc. hydrochloric acid, transferred to a 250 cc. flask, and made up to the mark. Five cc. of this solution contains five-tenths cc. of free hydrochloric acid and sufficient phosphorus to give five milligrams precipitate of ammonium phosphomolybdate, which amount was deducted from the weight of the precipitates obtained in the course of the work. The ammonium acetate solution was made by dissolving 400 grams of ammonium acetate in 800 cc. of water.

The method employed is as follows: Five grams of the steel is introduced into an Erlenmeyer flask and thirty-five grams of powdered mercuric chloride added; to this is further added 100 cc. of two per cent. hydrochloric acid and the flask tightly stoppered. The whole is allowed to stand, with occasional shaking, for one and one-half hours, or until solution is complete. The solution is then filtered by aid of a pump and the precipitated mercury washed four times with hot water. A convenient method for filtering and washing the precipitated mercury is to

employ a perforated porcelain disk about four cm. in diameter, held in a funnel. On this is placed one filter paper just the size of the disk, and a second one on top being slightly larger. After fitting the upper one by wetting and pressing down firmly a small amount of finely divided asbestos, suspended in water, may be poured on, which will serve to make the points tight and facilitate the removal of the paper and mercury after washing.

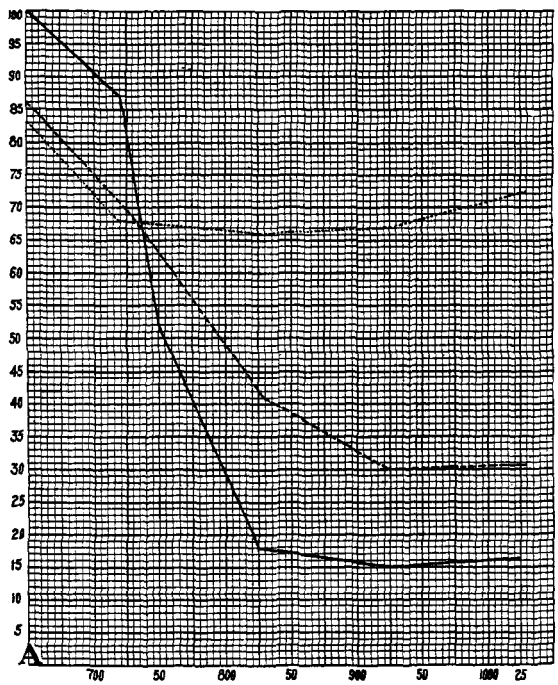
To the filtrate and washings from the precipitated mercury, amounting to 200 to 225 cc., is added five cc. of the ferric chloride solution and fifteen cc. of the ammonium acetate. The whole is then covered in lipless beakers and brought almost to a boil, when the basic acetate carrying ferric phosphate is precipitated. The solution is then filtered and allowed to drain without washing. The filter containing the basic acetate is transferred to the beaker in which the precipitation was made and heated with twenty cc. of strong nitric acid until the solution is complete. After diluting with twice its volume of water, the filter paper is filtered out and washed with hot water, acidified with nitric acid, and the solution boiled down and the phosphorus precipitated with ammonium molybdate. From the weight of the ammonium phosphomolybdate weighed on asbestos funnels, dried at 120°, is deducted five milligrams for the phosphorus contained in the ferric chloride solution used, and the per cent. of the phosphorus soluble is then calculated.

The results obtained on three specimens of steel are given in the following table:

Name of steel.	Chemical composition. C.	P.	Mn.	S.	Si.	Heat treatment.	Quenching temperature.	Per cent. of phosphorus soluble in acid $HgCl_2$	Per cent. of total phosphorus soluble in acid $HgCl_2$.
Cl 5	0.10	0.119	0.484	....	....	annealed	....	0.099	83.2
Cl 5	0.10	0.119	0.484	....	....		719°	0.081	68.08
Cl 5	0.10	0.119	0.484	....	....		825°	0.079	66.4
Cl 5	0.10	0.119	0.484	....	....		928°	0.080	67.2
Cl 5	0.10	0.119	0.484	....	....		1028°	0.086	72.2
N.S. 5	0.37	0.160	0.820	....	....	annealed	....	0.137	85.6
N.S. 5	0.37	0.160	0.820	....	....		728°	0.110	68.8
N.S. 5	0.37	0.160	0.820	....	....		827°	0.066	41.2
N.S. 5	0.37	0.160	0.820	....	....		923°	0.048	30.0
N.S. 5	0.37	0.160	0.820	....	....		1027°	0.049	30.6

Name of steel.	Chemical composition. C.	P.	Mn.	S.	Si.	Heat treatment. Quenching temperature.	Per cent. of phosphor's soluble in acid $HgCl_2$.	Per cent. of total phosphorus soluble in acid $HgCl_2$.
Car. I	1.22	0.098	0.780	0.068	0.058	annealed	0.098	100.0
Car. I	1.22	0.098	0.780	0.068	0.058	719°	0.087	89.8
Car. I	1.22	0.098	0.780	0.068	0.058	750°	0.051	52.0
Car. I	1.22	0.098	0.780	0.068	0.058	825°	0.018	18.3
Car. I	1.22	0.098	0.789	0.068	0.058	923°	0.015	15.3
Car. I	1.22	0.098	0.780	0.068	0.058	1023°	0.016	16.2

The evident influence of the amount of carbon present and the heat treatment upon the solubility of the phosphorus as given in the last column of the above table is best shown graphically in the diagram given below.



Full line = Carnegie Iron.
Broken line = N. S.
Dotted line = Cleveland.

The above work brings out the fact that phosphorus like carbon is capable of existing in steel in at least two forms and that

the influence of phosphorus upon the physical properties of steel in which it is contained is as much dependent upon the form of combination in which it exists as upon the quantity. This power of phosphorus to exist in two or more forms in steel with the varying influence on the brittleness of the steel according to the form present will, we think, account for many of the apparent inconsistencies in the statements usually made by metallurgists in regard to the behavior of this element. Much remains to be done upon the products of solution of iron and steel along the lines indicated either by the present paper or by that of Mackintosh,[1] or more recently by Juptner's paper, read before the British Iron and Steel Institute, May, 1897, before we shall be able to accumulate sufficient data to enable us to draw reliable conclusions as to the forms in which phosphorus may exist in iron and steel, the conditions under which the different forms are produced, and the influence of the different forms on the physical properties of the metals.

AN ELECTRICAL LABORATORY STOVE.

BY M. D. SOHON.

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THIS apparatus has been designed to economically replace, as far as possible, the ordinary water-baths and gas burners used in the laboratory.

The stove is of copper, preferably cylindrical, about three

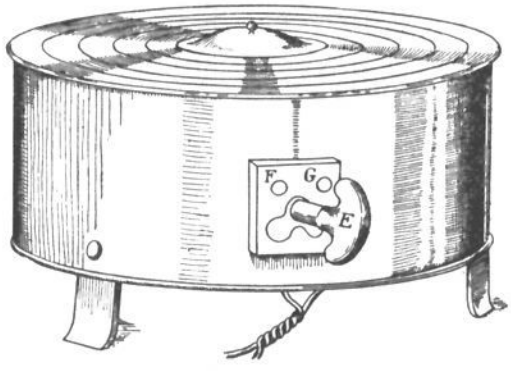

FIG. I.

inches in height, exclusive of legs, and seven and a half inches in diameter. The top consists of the usual concentric rings; the bottom is open. The heating plate *A* is placed two inches

[1] *Trans. Amer. Inst. Min. Eng.*, 14, 385.