During the work it was found that covering the end of the sliding tube with a red glass of sufficient thickness, most of the substances under examination could be clearly seen against the surface of the capsule. This is due to the fact that their radiating power is less than that of carbon, and also because the temperature of the free surface must be somewhat lower than that of the capsule. This provided a means of observing the change of form, to some extent at least, of the heated substance, and to recognize a sintering or fusing of the substance without removing the capsule. The presence of the fumes which were occasionally developed by decomposition of the substance could also be detected in the same way.

ELECTROCHEMICAL LABORATORY, COLUMBIA UNIVERSITY,
March 15, 1906.

## THE MEASUREMENT OF TEMPERATURE IN THE FOR-MATION OF CARBORUNDUM.

BY S. A. TUCKER AND ALEXANDER LAMPEN.

THE purpose of this investigation was to determine, if possible, the temperature for the formation of carborundum, and its decomposition into graphite and silicon.

When a carborundum furnace built on the lines of a commercial furnace was taken down after a run it was found that fairly sharp lines of demarkation could be observed between the layers of graphite, carborundum (crystallized silicon carbide), and siloxicon (amorphous silicon carbide). If after bringing the furnace to a stationary condition, temperature measurements could be made at different points of the cross-section, the desired temperatures could be easily obtained.

This was accomplished by placing through the centre of the furnace a horizontal graphite tube containing a plug which could be pushed to any desired section and its temperature measured by an optical pyrometer.

### THE FURNACE.

This, as will be seen from the drawing (Fig. 1), was built on the general plan of a furnace used in the manufacture of carborundum,
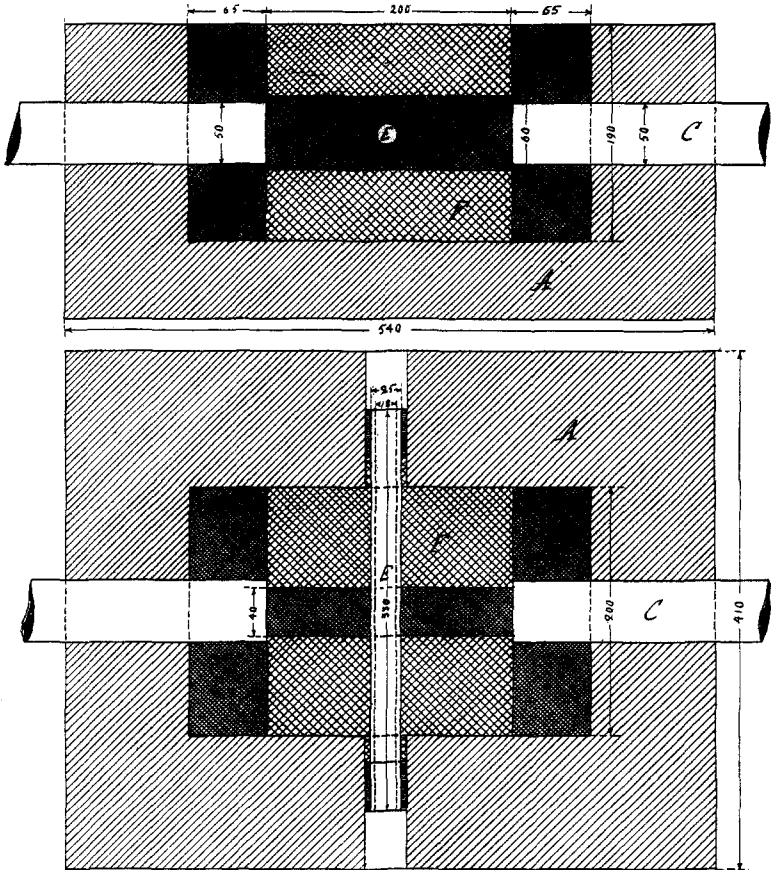
Fig. 1.

though several alterations were made in the proportions of the furnace as well as the dimensions and the form of the core, owing to the voltage used and the particular use to which the furnace was to be put.

After several experiments the following construction was adopted:

The retaining walls of fire-brick, $A$, enclosed a space 330 mm. long, 200 mm. wide, and 190 mm. deep. The terminals of Acheson graphite, $C$, were 50 mm. in diameter and placed at a distance of 200 mm. between the ends. A tube, $E$, bored out of Acheson graphite, with a length of 330 mm., and outside diameter of 25 mm., and inside diameter of 18 mm., was put transversely

through the centre of the furnace with the ends projecting into holes provided in the brick retaining walls and luted with fire-clay. Thin plates of sheet iron were placed against the ends of the terminals which were surrounded with a packed layer of powdered coke, $D$. The space between the plates was filled with charge $F$, to a proper height, and a core of powdered coke, $B$, 40 mm. wide and 60 mm. deep was made between the terminals by means of iron plates. The furnace was then filled up with the charge and all the plates withdrawn. The charge, of which about 8 kilos was used, consisted of:

Coke ................................. 34.2 parts by weight.
Sand ................................. 54.2 " " "
Sawdust............................. 9.9 " " "

Finally a graphite plug 17.5 mm. in diameter and 9 mm. long was slipped into the tube and the furnace was ready for a run.

It was found after several preliminary trials that a good yield of carborundum could be obtained with 12 kilowatts running about two hours.

PYROMETER.

This instrument was by Wanner, and was used with all the precautions indicated by Waidner and Burgess in their work on optical pyrometry.[1]

The current which supplied the standard electric lamp was kept constant by means of a milliammeter and variable resistance throughout the work. Before using the instrument it was checked up with a standardized thermocouple to temperatures around 1300° C. and was found to vary but slightly. At the higher temperatures a black glass is interposed between the instrument and the incandescent body, and this point was found to be in close agreement with the open instrument. It may be said that on using the instrument without control of the current which supplies the standard lamp, no accurate measurements can be obtained.

EXPERIMENTAL.

The run is started with 130 volts alternating current, the maximum voltage of the generator. The current is hardly appreciable at first but as the resistor begins to warm up gases are evolved, and should be lighted at once to prevent explosion.

[1] "Optical Pyrometry," by C. W. Waidner and G. K. Burgess, Bureau of Standards, Washington, D. C., 1905.

Some time is taken in getting the current to the required point by varying the field of the alternator.

The pyrometer was set up in a fixed position so that by sighting through one end of the graphite tube the radiations from the plug would illuminate the field completely.

The first run made in this way had for its object the determination of the maximum temperature of the furnace which would give a good yield of carborundum, and clearly defined layers, and was found to be about 2300°.

If too high a temperature is attained, the layers will, of course, be pushed further to the sides of the furnace, and fusion of the retaining brick walls is likely to result, which changes the shape of the furnace to such an extent that no properly defined layers can be distinguished. Therefore, in the succeeding experiments the temperature of the furnace was raised until the plug in the centre of the core indicated a temperature of nearly 2300° C. and this temperature maintained constant for this point throughout the run.

The temperature was slowly raised during about two hours and then kept at its maximum until the condition of the furnace could be considered stationary, i. e., the reaction practically finished and the layers fixed. The decreasing evolution of gases indicated when this point was reached. Then the plug was pushed by means of carbon rods to the desired position and given time to acquire the surrounding temperature. The pyrometer was sighted on it and the temperature recorded.

Between readings the ends of the tube were closed with pieces of sheet asbestos.

The following table shows the results of a run as recorded:

| Time. | Volts. | Amperes. | Position of the plug. | Temperature. |
|---|---|---|---|---|
| 10.25 | 130 | ..... | Centre | ...... |
| 10.50 | 55 | 130 | " | ...... |
| 11.00 | 47 | 205 | " | 1664° C. |
| 11.10 | 45 | 230 | " | 1766 |
| 11.20 | 44 | 240 | " | 1865 |
| 11.25 | 42 | 260 | " | 2030 |
| 11.30 | 42 | 260 | " | 2120 |
| 11.35 | 41.5 | 270 | " | 2175 |
| 11.40 | 41 | 265 | " | 2175 |
| 11.45 | 41 | 280 | " | 2175 |
| 11.50 | 41 | 300 | " | 2270 |
| 11.55 | 40.5 | 300 | " | 2250 |
| 12.00 | 40.5 | 300 | " | 2260 |

| Time. | Volts. | Amperes. | Position of the plug. | Temperature. |
|-------|--------|----------|-----------------------|--------------|
| 12.05 | 40.5 | 300 | Centre | 2270 |
| 12.09 | 40.5 | 300 | 2 cm. from centre. | 2248 |
| 12.13 | 40.5 | 305 | 3 cm. " " | 2205 |
| 12.18 | 40.5 | 300 | 5 cm. " " | 2088 |
| 12.23 | 41.0 | 300 | 7 cm. " " | 1925 |
| 12.29 | 41.0 | 300 | 9 cm. " " | 1758 |
| 12.32 | 41.0 | 295 | Centre. | 2260 |
| 12.39 | 41.5 | 290 | 2 cm. from centre to other side | 2236 |
| 12.41 | 42.0 | 295 | 3 cm. " " | 2194 |
| 12.47 | 42.0 | 290 | 5 cm. " " | 2073 |
| 12.52 | 42.0 | 285 | 7 cm. " " | 1897 |
| 1.06 | 43.0 | 290 | 9 cm. " " | 1740 |

After cooling, the furnace was taken down and a section made, exposing the tube on which measurements of the positions of the layers were then taken. These measurements defining the limits of the different products combined with the temperature measurements in the above table resulted in the following curve (Fig. 2):
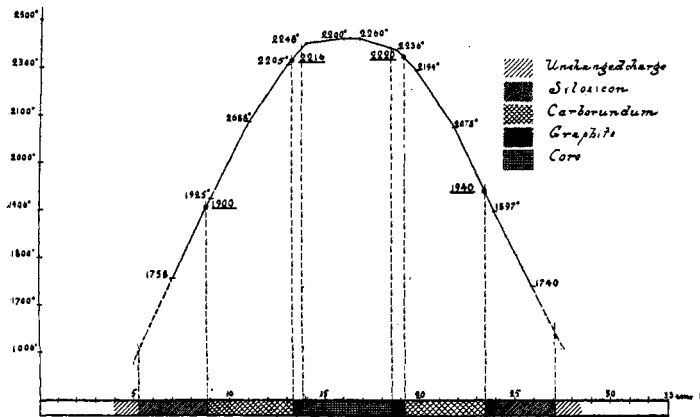


Fig. 2.

This curve gives as the decomposition temperature of carborundum into graphite and silicon 2218° C., and as its formation temperature, *i. e.*, the point where the amorphous turns into crystalline, about 1920° C. From another experiment the decomposition point of carborundum was found to be 2223° C., and the formation point 1980° C. The line between siloxicon and carborundum is always less defined than that between carborundum and graphite. This explains partly at least, the variations in the determinations of the latter point. As a limit was

considered the section where crystals could yet be found in the amorphous mass.

As an average may be taken for the formation point of carborundum 1950° C. and as its decomposition point into graphite and silicon 2220° C.

ELECTROCHEMICAL LABORATORY, COLUMBIA UNIVERSITY,
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## VOLUMETRIC METHOD FOR THE ESTIMATION OF CARBON IN IRON AND STEEL WITH THE USE OF BARIUM HYDROXIDE.[1]

BY JAMES A. AUPPERLE.
Received May 16, 1906.

THE method which I am about to describe has been found to compare very favorably with results obtained by gravimetric methods, with the advantage of being more rapid. It is based upon the following data:

*First.*—Barium hydroxide can be titrated with acids in the presence of barium carbonate (which is neutral or faintly alkaline to phenolphthalein), and no carbonic acid will be lost.

*Second.*—Carbonic acid is indicated by phenolphthalein, and when any barium carbonate is dissolved by standard acid during titration, the fact will be shown by the change in color.

*Third.*—Barium hydroxide can be titrated with acids without filtering off the barium carbonate, providing the acid is run into the mixture by a prolongation of capillary tube attached to the burette and extending well into the solution that is being titrated.

The reason for using the capillary tube is that if any carbonic acid be liberated in the lower portion of the beaker, it would be reabsorbed by the upper portion containing barium hydroxide.

Hydrochloric acid has been found to give the best results, and an acid a trifle less than $N/5$ is used. This is prepared by mixing 15 cc. of acid (1.20) and 1000 cc. of water. One cc. will approximately equal 0.001 gram carbon.

The barium hydroxide solution is prepared from 31.5 grams of Merck's crystallized salt $(Ba(OH)_2,8H_2O)$ dissolved in 1000 cc. of boiled water. When both acid and alkali are at room temperature, and without waiting for the suspended barium carbonate to settle completely, a measured portion is titrated with acid, and the remaining barium hydroxide is then diluted with water,

[1] Read before the Indiana Section of the American Chemical Society at Lafayette, Ind., May 12, 1906.