[CONTRIBUTIONS FROM THE HAVEMEYER LABORATORIES OF COLUMBIA UNIVERSITY, NO. 126.]

AN ELECTRICAL RESISTANCE FURNACE FOR THE MEASUREMENT OF HIGHER TEMPERATURES WITH THE OPTICAL PYROMETER.

BY ALEXANDER LAMPEN.

Received May 16, 1906.

THE plan adopted was to heat a small quantity of the material or substances under investigation to a required temperature, and after being kept at this point for a certain time, withdraw and examine it for any physical or chemical change taking place, and then if necessary place again in the heated zone of the furnace, the temperature of which was raised to a known degree.

By repetition of this operation with an increase of temperature by known intervals the desired change in the material could be reached with an approximate knowledge of the temperature which had produced this change.

Such a furnace should fulfil the following requirements:

(1) To consist of materials which should withstand a sufficiently high temperature without destruction.

(2) The temperature zone to which the substance is exposed should permit a variation to wide limits, and be easily regulated.

(3) To enable a direct measurement of the temperature of the substance by sighting with an optical pyrometer on it, or on a surface raised to the same temperature.

(4) To enable an easy and rapid removal and replacement of the substance during the run.

(5) To come as nearly as possible to the optical properties of a "black body."

As it was desired to work up to temperatures of approximately 2500° C. the only available material for the furnace was carbon. From the ease with which Acheson graphite can be machined, this article was exclusively used for the construction of the working parts of the furnace.

After several experiments with furnaces of varying construction, the plan finally adopted was that shown in Fig. 1. The enclosing walls *A* of fire-brick were lined with a mixture *B* of equal parts of fire-clay and siloxicon to prevent fusing. The terminals, *C*, were Acheson graphite electrodes, the outside parts of which were wrapped with sheet asbestos, *I*, to prevent oxidation.

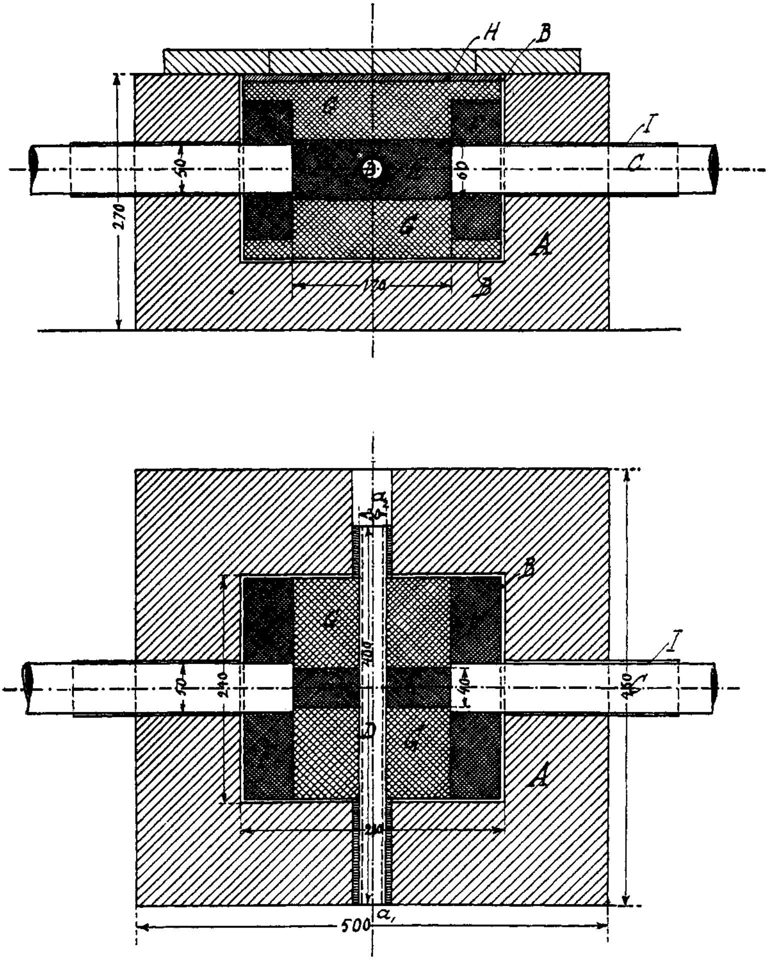

Fig. 1.

A graphite tube, *D*, 400 mm. long, 30 mm. outside, and 23 mm. inside diameter, was placed transversely across the furnace, through holes provided in the walls. The ends of this tube were also wrapped in sheet asbestos, and luted with the above mixture.

The tube was so placed within the asbestos bushings that it allowed of expansion without tending to fracture.

The core, *E*, and the layers, *F*, around the free ends of the terminals, *C*, consisted of powdered coke.

The filling mass, *G*, was crushed charcoal and on top of this was a thin layer of carborundum, *H*, in order to prevent the burning of the charcoal. The furnace was covered with fire-bricks, and the walls carefully luted with fire-clay to keep the air out of the inner parts.

Fig. 2 gives the necessary details of the working parts. *D* is the transversal tube. The substance which is to be heated is introduced into a small graphite capsule, *L*, and then placed in the end, c_2, of a second tube, *K*; this is then slipped into tube *D* at a_1. The diameter of tube is of such size as to slip easily within tube *D*.

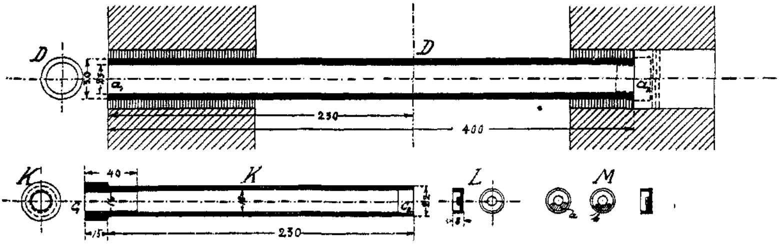

Fig 2.

When the current is passed through the furnace the hottest part of the fixed tube will be in the core, and the temperature decreases towards the ends. By regulating the current, and moving the sliding tube, *K*, in a proper manner, the desired temperatures may be easily reached and maintained.

The temperature readings are taken through the end, c_1, by sighting with the pyrometer on the capsule. To remove the substance the sliding tube is pulled out and the capsule removed with a carbon rod. During the runs the end, a_2, of the tube *D* was shut with a well-fitting graphite plug and the opening in the wall closed with asbestos and luted with fire-clay to prevent air circulating in the tube.

The substance was introduced into the capsule so that it occupied the lower part of it only as seen at *M* (Fig. 2).

When in powder it was slightly packed to a layer of 0.5 to 1 mm. thickness (*a*); if in lump, a piece of the same thickness was placed as indicated by *b*.

This was in order to get uniform readings as the upper half of the capsule supplies the light for the pyrometer field, and thus radiations from a graphite surface were investigated independent of the substance which was heated.

The choice of the material, *G*, for filling was somewhat restricted as it was essential that it be a poor conductor of heat and electricity and should not contain constituents which on decomposition by heat would give rise to fumes, and thus interfere with pyrometer measurements, and should stand the highest temperatures without fusing or reacting upon the graphite tube.

Many different materials were tried for this purpose, and charcoal was found to be the only one fulfilling these requirements.

The length of the fixed tube, *D*, was a matter of considerable importance, because that portion passing through the charcoal *G*, should be long enough to admit a considerable drop of temperature from the core, and also the end, a_1, must be far enough from the centre of the heat so that it can be kept below a red heat. In spite of this some oxidation at the mouth of the tube did take place, which could be probably entirely avoided by using some water-cooling device at this point. The inner tube, *K*, was coated with a thin layer of siloxicon two-thirds of its length from c_1 to prevent its oxidation when removed hot from the furnace.

The currents used were alternating, 150–450 amperes at 15–20 volts.

During the first runs of a new furnace some difficulty was experienced from fumes in the tube interfering with the pyrometer observations. These were probably derived from decomposition of impurities in the tube or the surrounding bodies. After a few runs these fumes were no longer observed, the impurities which gave rise to them having been volatilized.

In order to ascertain how much the radiation from the capsule differed from that of a black body a series of comparative temperature measurements were made with a Wanner optical pyrometer and a Siemens and Halske thermocouple. The latter instrument was standardized up to about 1050° C. and had a scale up to 1400° C. The couple was introduced into the sliding tube, the temperature regulated and kept constant for a while, the couple then quickly removed and a reading taken with the optical pyrometer. The current which supplied the standard lamp of the Wanner instrument was kept constant by means of a rheostat and milliammeter.

TABLE I.

The junction of the couple bare and nearly touching the capsule.

Siemens and Halske.	Wanner.
1105 C	1116 C
1135	1142
1175	1183
1205	1208
1210	1211

TABLE II.

The junction of couple placed in a clay tube, the bottom of which was about 1 mm. thick and in contact with the capsule.

Siemens and Halske.	Wanner.
1275	1296
1310	1330
1373	1388

As far as these results go, it shows that the optical properties of the apparatus did not deviate appreciably from those of a "black body."

The method of carrying on the observations was as follows:

The sliding tube provided with an empty capsule was introduced into the furnace, the current thrown on, and the temperature readings taken at intervals. When the temperature of the centre of the furnace had become somewhat higher than the desired temperature the tube was removed and the empty capsule replaced with one charged with the substance under investigation. The sliding tube was now gradually pushed into the fixed tube, all the time following the rising temperature with the pyrometer, until the desired point was reached, and this temperature was maintained by slight alteration of the position of the inner sliding tube. If the pyrometer gave any indication of a tendency to rise above this point, the sliding tube was withdrawn a short distance and *vice versa*. After a sufficient time the sliding tube was gradually pulled out to give it time to cool, the capsule removed, examined and replaced, or another capsule ready charged introduced and heated as above described. The removing, replacing and reheating could be carried out in from three to five minutes. It will thus be seen that a great many determinations can be made in a comparatively short time.

The following examples illustrate the mode of working and the results obtained:

SILICON CARBIDE.

A mixture of sand and coke corresponding to the reaction $3C + SiO_2 = 2CO + SiC$ was reduced to fine powder and raised to the temperatures as given below. To determine when the reaction had taken place, the mixture was heated over a Bunsen burner to remove excess of carbon, and then treated with boiling concentrated hydrofluoric acid to remove the unchanged silica. The resulting greenish gray amorphous residue indicated the presence of silicon carbide.

TABLE III.

Temperature.	Time of heating.	Results.
1500	5 minutes	Sintered, no reaction.
1500	10 "	"
1540	10 "	"
1600	10 "	Traces of carbide.
1700	10 "	More carbide.
1800	10 "	Homogeneous mass, mostly carbide.
1900	10 "	Homogeneous mass, mostly carbide.
2000	10 "	Small crystals in amorphous mass.
2100	10 "	Mass of crystals.

Some carbide crystals (carborundum) were introduced and heated as follows:

2100° C.	10 minutes	No change.
2200	10 "	"
2240	10 "	Graphite.

This shows that the reaction point between the above substances is somewhere round 1600° C. The formation of crystallized carbide or carborundum begins between 1900 and 2000° C. and its decomposition into silicon and graphite between 2200° and 2240° C.

The former point has been previously determined in this laboratory to be 1950° C. and the latter 2220° C.

In order to determine more closely the reaction point between carbon and silica, a mixture of pure materials was prepared and heated with small temperature intervals. The first indications of the reaction could be detected at 1615° C., whereas at 1600° C. no carbide could be found with certainty.

CALCIUM CARBIDE.

A fine powdered mixture of coke and calcium oxide (commercial) according to the reaction

$$3C + CaO = CaC_2 + CO$$

was heated at 100° C. intervals for ten minutes.

TABLE IV.

Temperature.	Results.
1500° C.	Slightly shrunk, no gas on addition of water.
1600	More shrinkage, no gas.
1700	Gas evolved with water, an odor of crude acetylene.
1800	The mass fused, abundance of gas with water.

Next, a piece of commercial calcium carbide was heated to:

1750° C.	Traces of sintering.
1780	Partly fused.
1800	Completely fused.

A proper mixture of pure calcium oxide and carbon was heated at small temperature intervals for ten minutes The presence of calcium carbide was determined by dropping a piece of the mixture into ammoniacal cuprous chloride solution, which gives a red precipitate with acetylene.

TABLE V.

Temperature.	Results.
1700° C.	No carbide detected with certainty.
1725	Traces of carbide.
1740	More carbide.

MELTING TEMPERATURES OF SOME REFRACTORY MATERIALS.

A rough determination of the melting-points was carried out for a number of refractory materials used in this laboratory.

TABLE VI.

Fire-clay (Henry Maurer, N. Y.):	
1350° C.	No change.
1400	Fused down.
Fire-brick (Henry Maurer No. 2 A.):	
1550° C.	Partly fused.
1600	Completely fused.
Porcelain from crucible (Dresden):	
1600° C.	Edges rounded.
1650	Fused down.
Piece of pure quartz:	
1650° C.	Edges rounded.
1700	Fused.
Magnesia brick with high percentage iron :	
1900° C.	Edges rounded.
2000	Completely fused.
Magnesia fused in electric arc :	
2000° C.	Fused.

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.

[CONTRIBUTION FROM THE HAVEMEYER LABORATORIES OF COLUMBIA UNIVERSITY, NO. 125].

THE MEASUREMENT OF TEMPERATURE IN THE FORMATION OF CARBORUNDUM.

BY S. A. TUCKER AND ALEXANDER LAMPEN.

Received May 16, 1906.

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,