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THE ELECTRIC VACUUM FURNACE.

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I. *Historical.*—Previous work *in vacuo* at high temperatures has been confined to experiments with quartz and porcelain tubes.

Schuller distilled and sublimed various elements and compounds in evacuated quartz vessels. The boiling-points of many metals have been determined in evacuated porcelain tubes by Kahlbaum, Roth, Siedler, Krafft, Bergfeld, Lehmann and others. In all cases, however, the vessels were subjected to external atmospheric pressure, and the scope of the experiments was limited by the temperature at which they collapsed. Porcelain collapsed between 1400° and 1500°, and quartz at somewhat higher temperatures. Moreover, certain metals and other substances which attack quartz and porcelain could not be investigated.

In the electric vacuum furnace described below, the attainable temperature is limited only by the vaporization of carbon, and crucibles of any material suitable for a particular experiment may be used.

II. *Description.*—The vacuum furnace is a special type of resistance furnace enclosed in a vacuum chamber, with means for continuous cooling of the parts liable to be injured by excessive heating, and so designed that the effect of heat on any substance or the course of any reaction may be studied up to the vaporizing point of carbon, the substance in the crucible being always visible through a window.

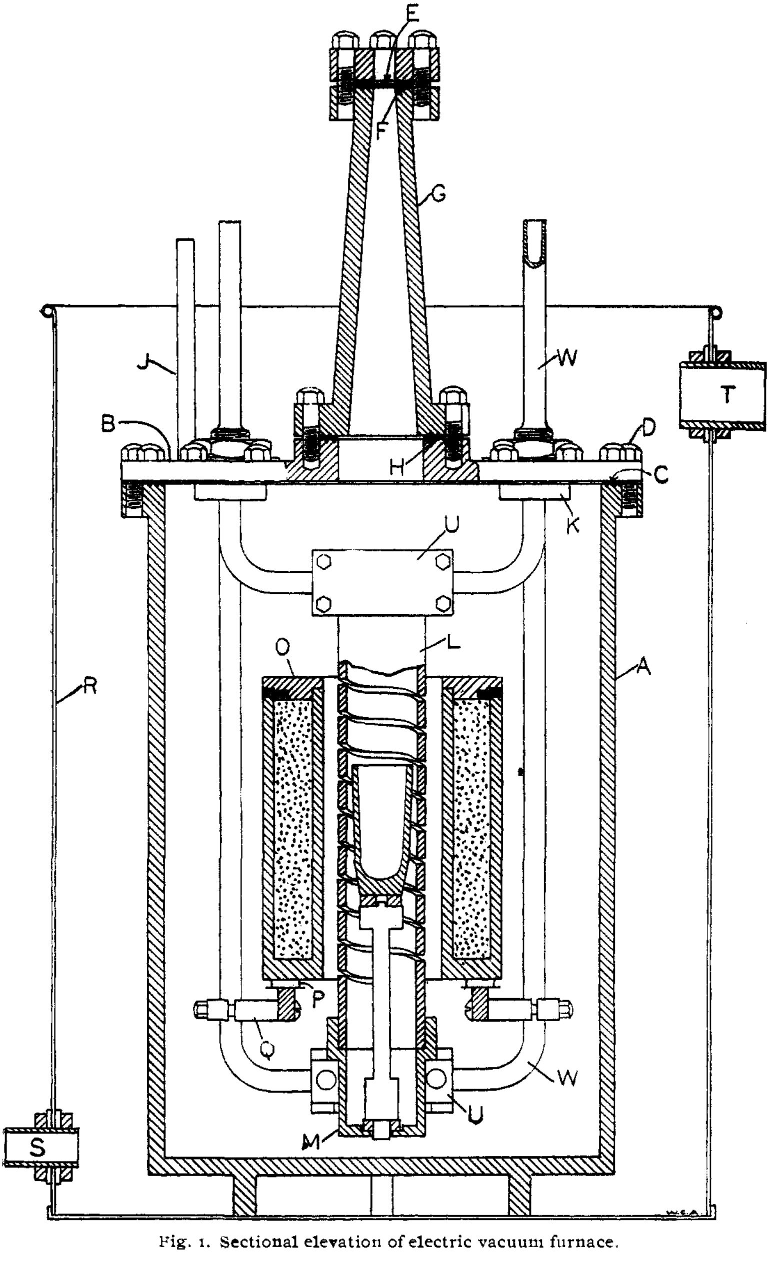

Fig. 1. Sectional elevation of electric vacuum furnace.

A calibration of the furnace to determine the relation of the temperature to the energy makes possible the accurate control of the temperature. The advantages of a vacuum for protecting the heater and the substance being heated are obvious.

It consists of an air-tight metal vessel with tubular electrodes entering the cover. These electrodes are provided with clamps which hold the heater in place and make contact between it and the electrodes. The radiation screen which surrounds the heater to diminish the radiation loss is also held in place by supports attached to the electrodes. The metal vessel stands in a can which serves as a water jacket. When the furnace is running, a constant flow of water is maintained through the water jacket and through the tubular electrodes.

The construction will be better understood from Fig. 1, which is a sectional elevation through the center. The chamber (*A*) and cover (*B*) are castings of gun-metal turned true at the joint. A lead gasket (*C*), $^1/_{16}$ inch thick, forms an air-tight joint when the cover is fastened down with the cap-screws (*D*). The first time the furnace is assembled, the tightening of the screws forces the lead into the annular grooves of the chamber and cover. The ridges thus formed in the lead washer, and suitable reference marks on the cover and chamber, make it possible to replace the parts in the same position after opening the furnace for cleaning or repairs. Leakage, due to porosity of the casting, is prevented by applying a thin coat of solder all over the inner and outer surfaces of the chamber and cover, after turning to dimensions. The chamber has four legs to permit the flow of water underneath. The tube (*J*), through which the air is exhausted, is soldered into the cover.

The window (*E*) is a disc of clear white mica about 0.005 inch thick, clamped between two lead washers (*F*) by means of a brass cap and four cap-screws. The brass surfaces touching the lead washers have annular grooves into which the lead is forced by the pressure. The window tube (*G*) is of gun-metal and is fastened to the cover by six cap-screws, the joint being made tight by a lead washer (*H*).

The electrodes (W) are formed of brass tubing bent into shape. The threaded brass bushings (K, K) which constitute parts of the electrode joints, are soldered to the electrode tubes.

The construction of an electrode joint is shown in detail in Fig. 2. Two lead washers (W_1, W_2), separated by a mica washer, are interposed between the cover and the flange of the bushing. The bushing is insulated from the cover and the upper lead washer (W_2) by the fibre sleeve (S), and the nut (N) is insulated from the cover by the fibre washer (F). The fibre parts are impregnated with paraffin to make them waterproof. By tightening the nut (N), a perfectly tight, electrically insulated joint is obtained. The heater-clamps (U. U), Fig. 1, are of copper.

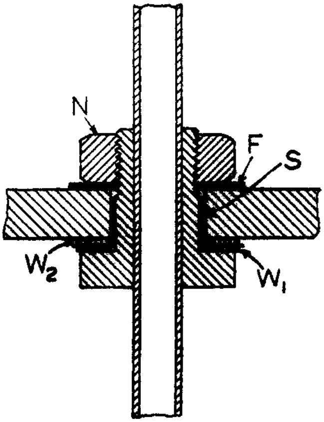

Fig. 2. Section of electrode joint.

The heater (L) is a helix of conducting material such as carbon or graphite. Artificial graphite is preferred for high temperature work because of its freedom from impurities. Metallic heaters can be used for some purposes and have certain advantages, one of which is the inability to absorb air when cold. A heater is made by sawing a slot in a hollow graphite cylinder. Its dimensions and the number of turns are so related that at the highest temperature reached the potential difference across the heater shall not be much more than 50 volts, in order to avoid excessive Edison effect and the tendency to arc across to the screen. Good results have been obtained with a heater 2 inches outside diameter, $1^5/_8$ inches inside diameter, and having ten turns in a length of $7^1/_2$ inches. The graphite cup (M) holds the lava insulating ring on which the crucible support rests. The proximity of this ring to the cold bottom of the furnace prevents it from fusing or being made conducting by heat.

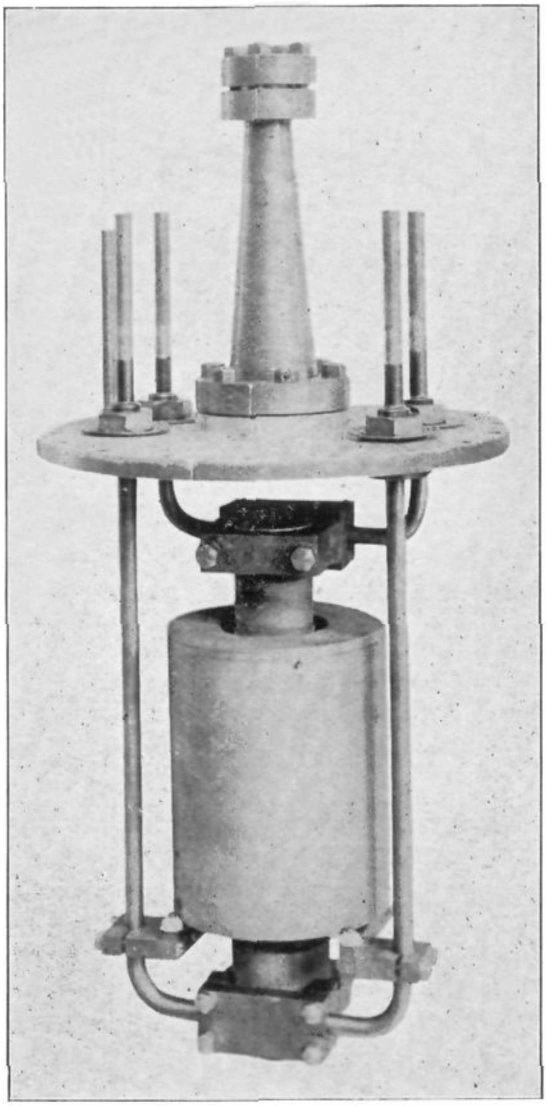

Fig. 3. Electric vacuum furnace.

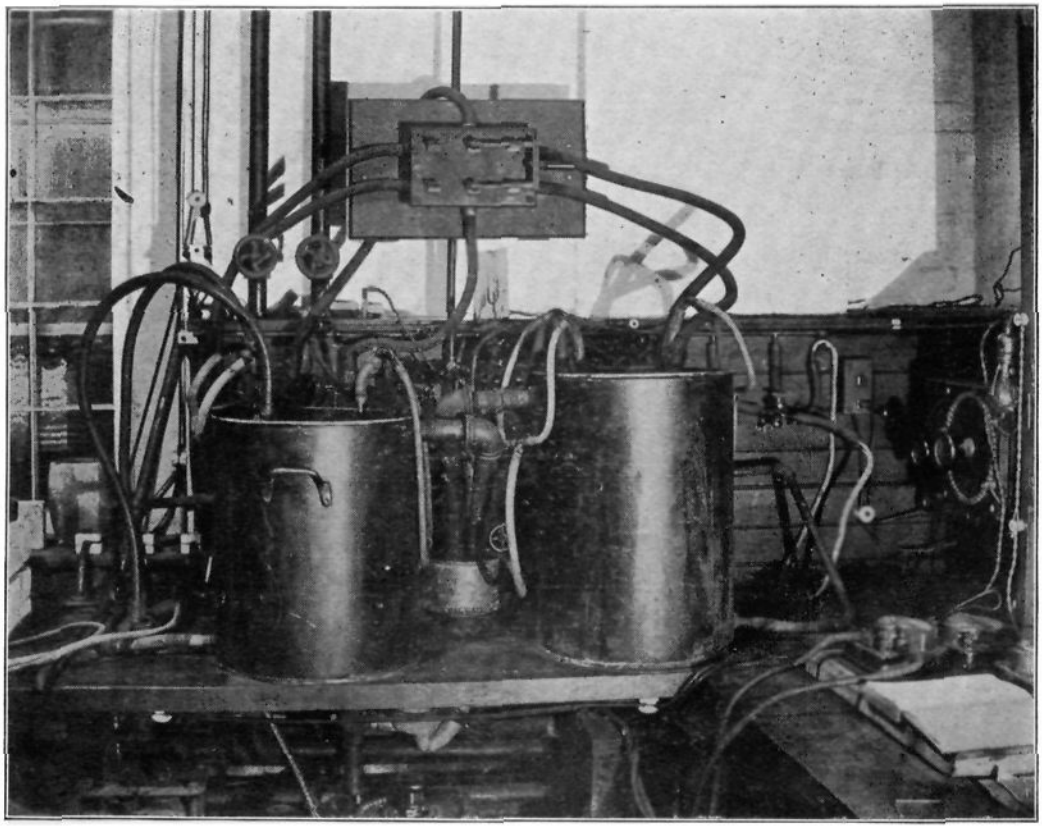

Fig. 4. Electric vacuum furnaces No. 1 and No. 2.

The radiation screen (*O*) is a double-walled cylindrical box of Acheson graphite, filled with graphite powder. Its purpose is to minimize loss of heat by radiation. Owing to the low thermal conductivity of the powdered graphite, the inner wall of the screen becomes nearly as hot as the heater, and then the rate of radiation from the heater to the screen, which depends upon the temperature difference, becomes very small, and equal to the rate at which heat is conducted away through the graphite powder. It was found that with the use of the screen, the melting-point of platinum could be reached with but one-fourth of the energy necessary without it. The screen is supported by the copper arms (*Q*) from which it is insulated by the lava buttons (*P*).

The water jacket (*R*) is a galvanized iron tank provided with an inlet (*S*) and outlet (*T*).

Fig. 3 shows the interior parts of the furnace raised out of the chamber. In Fig. 4 are shown two vacuum furnaces which have had almost constant use for two years. These have no window tubes, but the windows are fastened directly to the covers of the furnaces.

III. *Crucibles and Crucible Supports.*—The choice of material for crucibles is determined by the nature of the substance to be heated and the temperature reached. Graphite crucibles are used when the substance to be heated is not acted upon by carbon, and are conveniently made from solid rods of Acheson graphite.

Magnesia crucibles are frequently used for fusing metals and preparing alloys when the temperature to be reached does not exceed 1650°. These crucibles are made by strongly compressing in a steel mold by means of a hydraulic press, a mixture of finely powdered and sifted magnesia with a suitable binder. A porcelain kiln was used for firing the magnesia before using and for baking the crucibles, the temperature attained being about 1350° C. Crucibles made from magnesia fired at a lower temperature are very liable to crack, owing to the great shrinkage which they undergo between 1200° and 1600°.

Other metallic oxides have been found to be more suitable than magnesium oxide as crucible material. In many cases oxide crucibles must be separated from the graphite support by a refractory metal cap, to prevent reduction at the contact surface.

Crucibles of iron, copper, fire-clay, porcelain and thorium oxide have been used for special purposes, the latter substance withstanding a higher temperature than anything else tried except graphite.

In Fig. 1, a crucible and graphite support with a metal cap are shown partly in section.

IV. *Accessory Apparatus. Vacuum Pump.*—A double-cylinder Geryk oil-pump is used with the cylinders in series. It gives a good vacuum in the furnace fifteen minutes after starting. The pump is connected to a brass main-tube having soldered branches to which the furnace, gauge, and air inlet valve are connected by means of heavy rubber pressure-tubing. The joints are made tight by brass sleeves, the ends of which are filled with a wax mixture. Brass stop-cocks were first used, but were later replaced by mercury-seal glass cocks which did not leak.

Vacuum Gauge.—A closed mercury gauge of the manometer type was used at first, but it gradually became soiled and an uncertain capillarity error was introduced, which made it unreliable for accurate measurements. To avoid this, the gauge is now connected through a tube containing glass wool and potassium bicarbonate crystals to retain dust and acid vapors, with an auxiliary tube containing a drying agent.

Electrical Apparatus.—The current is furnished by a single-phase alternator in connection with a step-down transformer. Regulation of the current is effected by means of rheostats in the generator field circuit. The measuring instruments are of the Thomson inclined-coil type. The voltage is measured directly, the current being first reduced by a current transformer.

V. *Operating Conditions. Vacuum.*—When all joints have been properly tightened, the pressure can be reduced to a fraction of a millimeter of mercury. If leaks are suspected, the furnace is closed, the jacket is filled with water and compressed air is forced into the chamber. Leaks are shown by air-bubbles. If due to defects in the casting, they can be repaired by soldering, or if not too large, by painting with pyroxylin lacquer. Leaks at the electrode-joints may occur when a new furnace is first assembled, but these disappear when the nuts are tightened sufficiently. The vacuum becomes somewhat poorer as the temperature in the furnace rises, especially with a new heater and screen, owing to the escape of gases from the graphite or from the substance which is being heated, but on maintaining the temperature at a definite point, the vacuum finally improves up to the starting value.

A high vacuum can be maintained even at the vaporizing point of graphite.

Description of Run.—The crucible is placed on the support and centered so that it does not touch the heater, the window tube is then fastened down, the washer and contact surfaces having been first carefully cleaned. When a good vacuum has been established the current is switched on and the water allowed to flow at such a rate through the jacket that it comes out barely warm, the water-level being above the window joint. Water is also passed through the electrode tubes, the outflow being allowed to play against the side of the window tube. In most cases the current should be raised gradually, so that the behavior of the heated substance can be followed.

The substance or article being heated is observed through the mica window, colored glass being used to protect the eyes at temperatures above 1100° C. At 2500° C. and above, three or more thicknesses of blue glass are necessary to make it possible to distinguish the outlines of the substance, because of the intensity of the light.

When the desired result has been obtained, the circuit is opened. From one to three hours are allowed for cooling, according to the temperature reached, then the water is let out of the jacket, air is admitted to the furnace, the window tube is unfastened and the crucible is removed.

VI. *Temperature Estimation.*—Owing to the small total heat capacity of the heater and screen, the temperature very soon reaches a constant value for each value of the energy used, and it is therefore possible to calibrate the furnace so that the relation of temperature to energy is known.

For this purpose the melting-points of copper and platinum were taken as fixed points, and the amounts of energy required to maintain these temperatures continuously were determined, the procedure being as follows:

Some pieces of electrolytic copper were fused in a graphite crucible so as to obtain oxygen-free copper with the melting-point 1084° (Holborn and Day). The current and voltage were read just when fusion began. The current was then diminished until the copper solidified and then gradually raised about one ampere every five minutes until the copper melted again. This procedure was repeated a number of times, and the product of the lowest average values of the current and voltage was taken as the energy corresponding to the melting-point of the copper. Fusion of the globule was characterized by the appearance of a dark spot on top, this being the only part of the globule which reflected no light vertically upward. Freezing of the globule was accompanied by the disappearance of the dark spot when the copper crystallized and the surface was no longer smooth.

A piece of chemically pure platinum wire was heated in a small cup of thorium oxide inside of a graphite crucible. The current was very gradually raised as the melting-point was approached, this having been approximately determined previously. The readings were taken while beads of melted platinum were slowly forming on the wire. The melting-point of platinum was taken as 1780° C. (Le Chatelier).[1]

To find a mathematical relation between the energy and temperature the observed values were tried in various well-known formulae, and it became evident that only an exponential equation would answer.

The expression chosen was $y^n = ax$, in which y represents the temperature of the crucible above that of the room, x is the energy in kilovolt-amperes, and n and a are constants. For convenience the equation was used in the form $(y—20)^n = ax$, or $n \log (y—20) = \log a + \log x$ in which y is the actual temperature centigrade of a crucible at the middle of the heater, the average temperature of the water in the jacket at the start, 20°, being taken as the temperature of the surroundings.

The average values found by the experiments described were:

Temperature.	Kilo-volt-amperes.
(20°)	(0.0)
1084°	1.909
1780	4.953

From these values the constants were calculated:

$$n = 1.895$$
$$\log a = 5.4552$$
$$a = 285200$$

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[1] Recent work gives the melting-point as 1710° by the thermoelectric couple (Harker: Chem. News, 91, 250 (1905); Holborn and Henning: Ber. Berl. Akad. 1905, p. 316) and 1745° by optical methods. Nernst and Wartenberg: Verh. deut. phys. Ges. 1906, p. 48.—EDITOR.

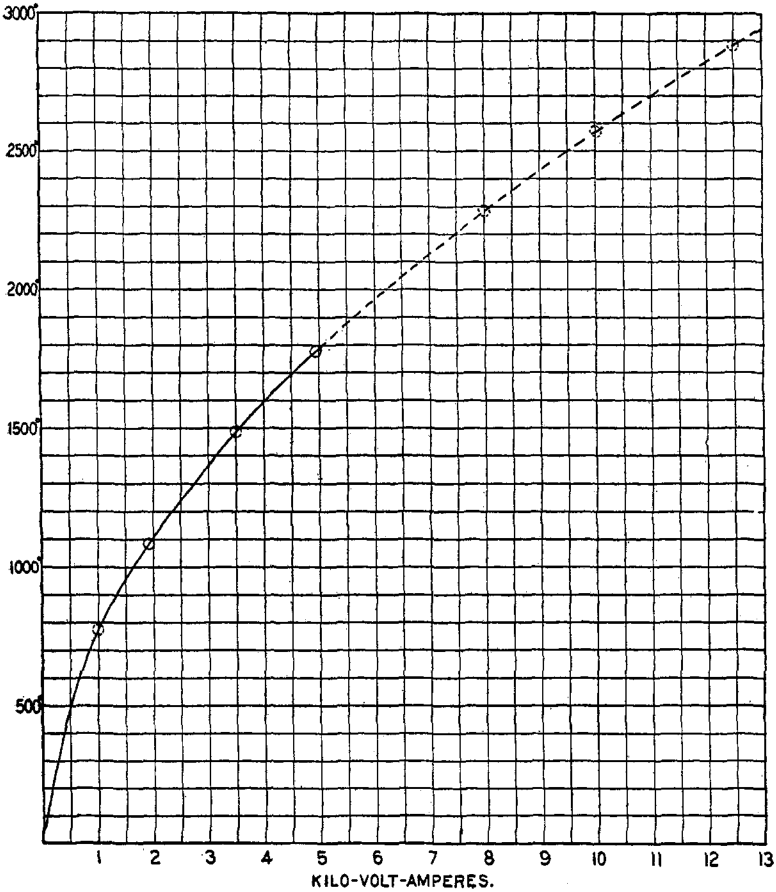

Fig. 5. Curve showing relation of temperature to energy in electric vacuum furnace.

The curve (Fig. 5) was plotted from the results obtained, the dotted portion being continued through extrapolated points calculated from the equation.

Accuracy of Calibration.—The principal source of error was the slight variation of the generator voltage. The variation at the melting-point of platinum was 4.915 to 4.992 K. V. A., corresponding to an uncertainty of 25° in temperature.

If the equation holds at higher temperatures, the error thus introduced into the constants will make measurements in the neighborhood of 3000° uncertain by 50°.

I hope later to check these calculations by an optical pyrometer, which should give accurate results, since any substance heated in the furnace, being enclosed by walls at the same temperature, will radiate like a black body.

The calibration up to 2000° is at least sufficiently accurate for practical purposes. In one experiment, the furnace was running constantly day and night for ninety hours, keeping the temperature very close to 1800°.

(No correction was made for the small current which flows through the water between the electrodes.)

VII. *Edison Effect.*[1]—In one of the first forms of the furnace, the radiation screen was supported by the lower clamp, and the crucible support was also in electrical contact with the clamp, as shown in Fig. 6, the need for further insulation being then unknown. With such an arrangement a rise of current and a

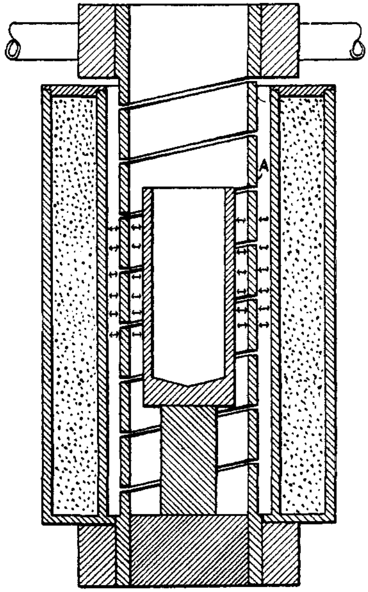

Fig 6. Arrangement of furnace No. 1, giving single-ring "Edison effect."

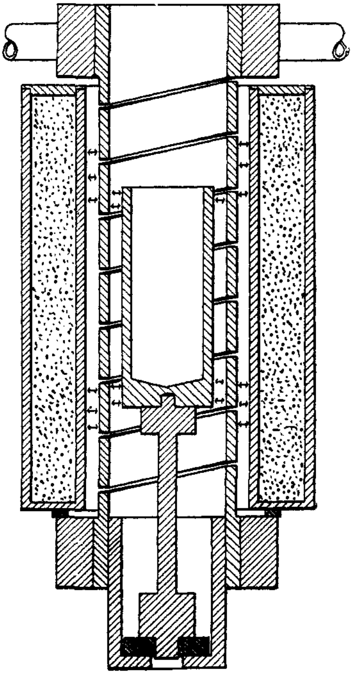

Fig. 7. Arrangement of furnace No. 1, giving double-ring "Edison effect."

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[1] T. A. Edison, 1884. Preece: Pr. Roy. Soc. 38, 219 (1885): Fleming: Pr. Roy. Soc. 47, 118 (1889).

simultaneous fall of voltage was observed as the temperature approached 2000° C. and an arc would soon start at the point *A*, Fig. 6. This was found, by the arrangement shown in Fig. 8, to be due to a discharge taking place across the space between the heater and the screen. The heater being at a slightly higher temperature than the inside of the screen, an excess of current flowed across the space in such a direction that the heater was negative, and amounted to two amperes at times. The arrangement was first calibrated by passing a direct current through the shunt and meter in parallel, and noting the reading of the meter for each value of the current.

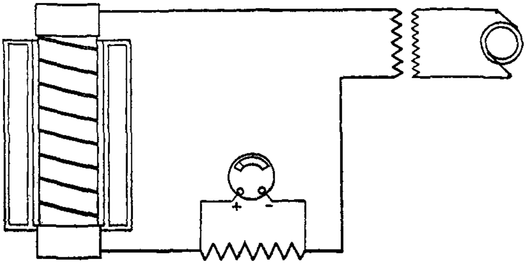

Fig. 8. Connections for showing "Edison effect."

In a later form of the furnace, with the screen and crucible support both completely insulated from the heater (Fig. 7), a similar variation was observed but of much smaller magnitude, and there was now no tendency to arc across. The relative variation in the two cases is shown in Fig. 9. The paths of the discharge in the two cases is shown by the arrows in Figs. 6 and 7, and by the black coatings found on the heater, screen and crucible after a high temperature run.

The ease with which ionization takes place at high temperatures is thus shown in a striking manner.

In Fig. 10, *b* and *c* show the deposit produced on the heater and crucible at high temperatures, with the arrangement shown in Fig. 6. The location of the arc is shown in *a*, Fig. 10, the black deposit being brushed off to show clearly the disintegration which has taken place. Fig. 11 shows two crucibles with the double-ring deposit obtained with the furnace arranged as in Fig. 1 or Fig. 7.

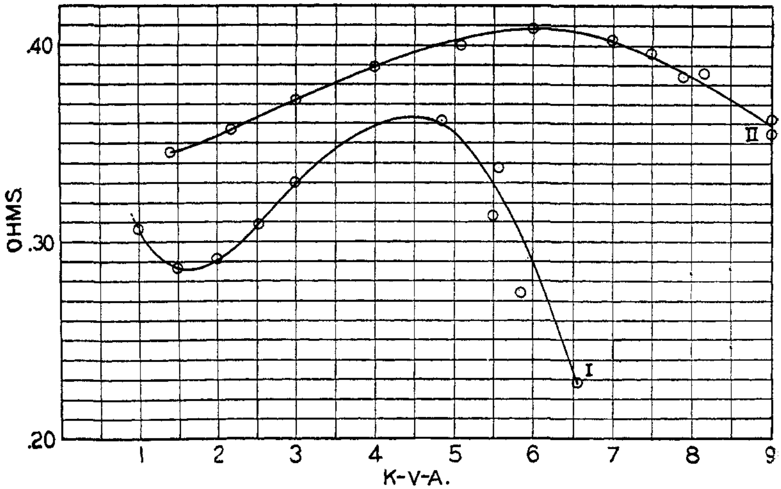

Fig. 9. Curves showing variation of resistance of heaters with temperature, for two arrangements of furnace shown in Figs. 6 and 7.

To pass a discharge through a rarefied gas as in a "vacuum tube" requires a certain fall of potential, mainly at the cathode. During the passage of the discharge, the cathode is disintegrated, the detached particles moving normally from the surface of the cathode, and depositing on the nearest surface.

It is well-known that the cathode fall diminishes as the temperature of the cathode is raised.[1] When the cathode becomes hot enough to have an appreciable vapor-pressure, the glow discharge becomes an arc.[2]

We therefore have in the furnace a complete analogy to the phenomena in a vacuum tube. The points between which the small arrows are placed become alternately anode and cathode to the alternating current, which leaks between them through the ionized gas, so that the erosion takes place on both surfaces. The gradual apparent lowering of the resistance of the heater with rise of temperature shown by the curves in Fig. 9, is due to the increasing leakage of current across the space where the arrows are. With the furnace arranged as in Fig. 7 there are two discharges in series. Between the two rings there was not a great enough potential difference to maintain two discharges.

Two explanations are possible for the presence of the rings.

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[1] Cunningham: Phil. Mag. 9, 193.

[2] Hittorf: Ann. Physik. 21, 90 (1884).

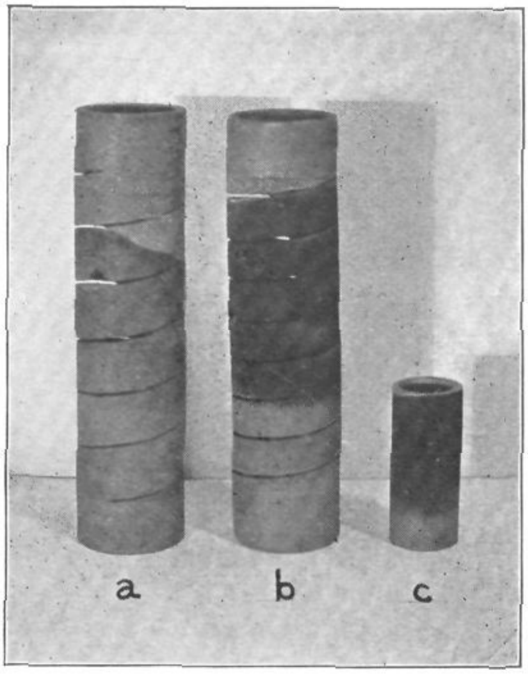

Fig. 10. Two heaters and crucible showing single-ring "Edison effect," and arc spot.

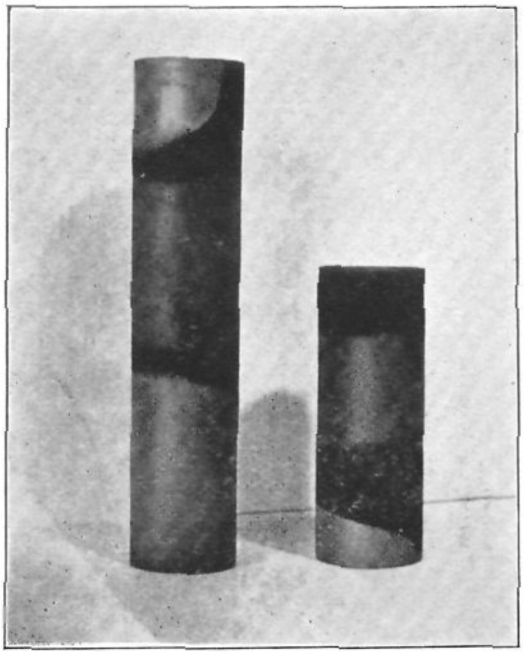

Fig. 11. Two crucibles showing double-ring "Edison effect."

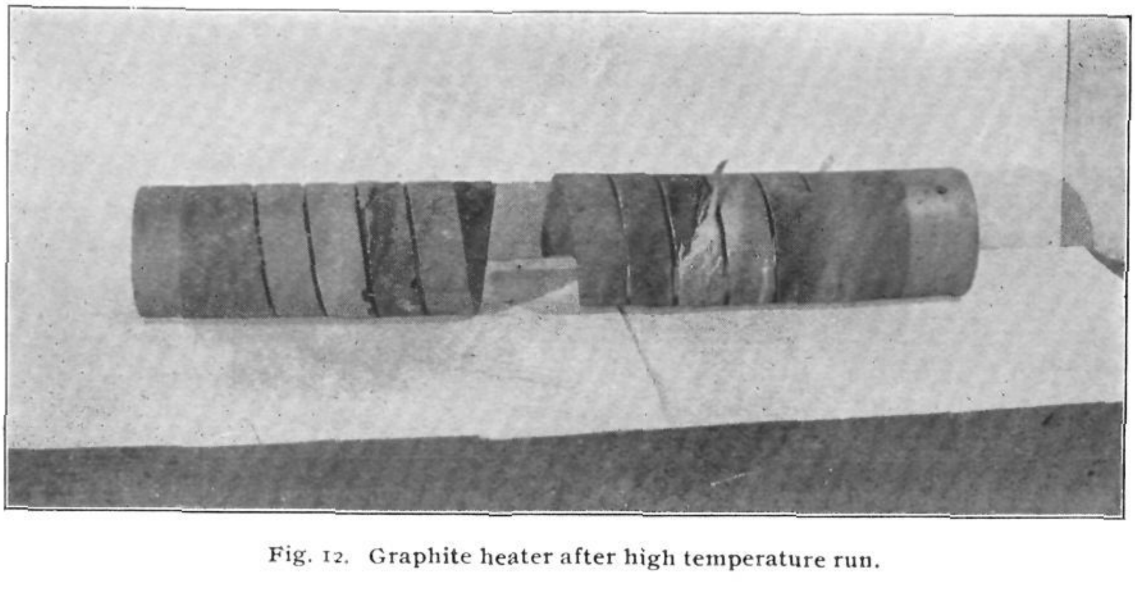

Fig. 12. Graphite heater after high temperature run.

(1) A certain minimum potential difference is required to produce a discharge at a given temperature, just as there is for an arc. This minimum is then just half that across the unblackened space on the heater between the rings, and lies between 10 and 15 volts.

(2) The resistance along the path taken by the discharge may be less than the resistance of the heater between the rings.

The second explanation seems inadequate, as it does not account for the complete absence of deposit between the rings. I consider the first explanation more satisfactory.

In the type of furnace shown in Fig. 1 the Edison effect is much reduced by using a heater of lower resistance, so that a lower voltage is required to reach a given temperature, and by making the space between the heater and screen wider. Both the screen and the crucible support are insulated from the heater. The black rings of deposit are now produced only at the highest temperatures (2500° to 3000°).

VIII. *Applications and Results.*—The furnace is especially useful for the fusion or heating of substances at temperatures beyond the range of the ordinary gas-furnace but which cannot be conveniently handled in the arc-furnace because of the excessively high and unmanageable temperature and the presence of carbon vapor. The temperature can be kept constant within less than 25° for any length of time. It makes many experiments possible at high temperatures in the absence of chemically active gases.

Above 2200° work is somewhat restricted by the lack of a crucible material which does not fuse or vaporize, or is not attacked by carbon or the substance to be heated.

Metals obtainable only in the form of a powder containing impurities can be brought into a pure compact state by fusion *in vacuo*, as the non-volatile impurities form a separate layer, and the volatile ones can pass off. Other substances which cannot be heated in air without oxidizing can be fused *in vacuo* without difficulty, unless they vaporize below their melting-points. Aluminum oxide, platinum and other refractory metals have been fused. Magnesium oxide, iron, silicon and aluminum have been vaporized.

Alloys of exact composition can easily be prepared by fusing their components together. In this way a number of iron alloys have been made.

Chemical reactions, syntheses and reductions can be carried on with great facility under direct observation. Gas-producing reactions can be followed closely by means of the manometer, which shows the temperature at which the reaction begins, and the point at which the reaction is over.

The calibration of an optical pyrometer could be easily performed by comparing it with an electrical resistance pyrometer or a thermocouple heated in the furnace, since any substance heated in the furnace, being enclosed by walls at the same temperature, will give "black body" radiation.

Melting-points of metals, compounds, fire-brick, and other refractory substances can be determined within a few degrees by reference to the calibration curve of the furnace, and still greater accuracy could be obtained with an optical pyrometer.

Substances could be distilled for purification or separation. Magnesium, zinc and cadmium have been thus distilled.

IX. *Vaporization of Graphite, and Life of Heaters.*—At high temperatures the middle of the graphite heater slowly vaporizes, and the sublimed graphite condenses on the colder parts of the heater and radiation screen. Finally the heat tends to become more localized at some thin spot, which rapidly wastes away until a break occurs and an arc starts. This point is indicated by a sudden *drop* in voltage, because the arc so extends itself that one or more turns are short-circuited.

Fig. 12 shows a broken heater with the partly loosened skin of sublimed graphite. This sublimed graphite has an almost silvery whiteness, and under a low-power microscope shows a botryoidal surface.

The average life of a heater at different temperatures is shown by the following table:

Temp.	Life.	
2500	9 hours.	These results are for hand-sawed heaters.
2700	3¼ "	Machine-cut heaters being more uniform will probably have a somewhat longer life.
2900	2 "	
3000	1 "	
3100	¾ "	

Since carbon vaporizes from the anode of a carbon arc at about 3700° C., at atmospheric pressure, it is to be expected that *in* *vacuo* it would vaporize at a much lower temperature. It is therefore very probable that the temperature measurements calculated from the extrapolated part of the curve are fairly accurate, and not overestimated, as had been feared.

In this connection it is of interest to note that Sir Wm. Crookes[1] has calculated from certain assumptions that carbon has zero vapor pressure at 3000° Abs., or approximately 2700°, which is about the temperature (estimated from the calibration curve) at which a more rapid wasting of the heater becomes noticeable.

X. *Conclusion.*—I believe that this furnace opens up a wide field for investigation of high temperature phenomena under exact and easily controlled conditions, and hope that eventually it will enable us to become familiar with the properties of substances up to extremely high temperatures.

I expect soon to be able to give the results of a number of interesting investigations which are now in progress.

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ELECTRIC SMELTING AT SAULT STE. MARIE, ONTARIO.[2]

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INTRODUCTION.

THE disability under which the middle provinces of Canada are placed as regards the upbuilding of an iron and steel industry on account of the necessity of importing the required metallurgical coke either from the United States or the extreme east or west of Canada engaged my attention shortly after assuming the duties of my present position. Considering the fact that the provinces of Ontario and Quebec are supplied with extensive deposits of iron ore and water-powers, the utilization of these water-powers in the production of electric energy in substitution of the energy resulting from the combustion of carbon in blast-furnaces suggested itself as a possible solution of the problem of economically producing pig iron without the use of metallurgical fuel.

Fortunately, the necessity of profitably employing the electric

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[1] Pr. Roy. Soc. A 76, 458 (1905).

[2] An address delivered at Ithaca, N. Y., before the American Chemical Society, Saturday, June 30, 1906.