Electric Furnace Reactions under High Gaseous Pressures

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XI. *Electric Furnace Reactions under High Gaseous Pressures.*

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[PLATE 6.]

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INTRODUCTION.

SOME ten years ago the classical work of HENRI MOISSAN laid the foundations of the scientific study of high-temperature chemical reactions.

It is hardly necessary to recall the rapid and extensive development which the subject has since experienced; it must, however, be remembered that the progress has been almost exclusively along technical lines, and even at the present time very little detailed work on the chemical and physical sides of the question has been published.

Doubtless individual inventors have acquired extensive experience and knowledge each of his special branch of the subject, but they have seldom found it advisable to impart the results of their researches.

The field of investigation, even with regard to the purely chemical phenomena occurring in quite ordinary cases, is far from exhausted, and practically nothing is known of the modifications introduced by abnormal conditions, such for instance as the combination of high pressure and high temperature.

The present research has been undertaken with a view to determine the correlation between the physical conditions and the chemical effects in the electric furnace, and more especially to ascertain the direct results produced by high gaseous pressures. In such work, if it is to be of real value, it is necessary to carry out the experiments on as large a scale as the limitations of cost and labour will permit; it is also particularly desirable to provide means for the modification of the various factors within the widest possible range. The chief factors being power, current, electromotive force, and above all, pressure.

In work carried out during preceding years under atmospheric pressure the necessity for employing now one type, now another of electric furnace determined us to select an apparatus suitable for both open and smothered arc, as also for resistance heating.*

The above considerations accentuated the already somewhat difficult task of designing a furnace suitable for high gaseous pressures. For, as already suggested, it would have been of little use to provide for a slight increase of pressure over that of the atmosphere.

The apparatus constructed is capable of employment for electric heating according to the most varied type of furnace, and has frequently been used for pressures as high as 200 atmospheres. We were thus in a position to extend our direct experimental study up to the limits which engineering difficulties set to practical application.

With regard to the experimental work, the first step was to investigate the additional effect of high pressure upon the more characteristic electric furnace reactions, it being obviously advisable to start by repeating the better known preparations, retaining as far as possible all other conditions similar to those at present in use.

It is with a general investigation of this character that our communication has to deal.

To avoid burdening the description of the work with the many numerical results, we have collected these in tables in an appendix to the paper.

DESCRIPTION OF APPARATUS.

Large High-Pressure Furnace.

From what has been said above it will be clear that an apparatus was necessary capable of being adapted to very varied requirements.

This end was met by designing a large steel enclosure of about 20 litres capacity,

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* We have frequently had occasion to divide the enclosure into a number of separate chambers, *e.g.*, for absorbing gaseous products inside the furnace, as also for a condensing chamber in volatilisations and distillations.

provided with various fittings for the introduction or circulation of gas, gauges for measurement of pressure, windows for observation and, finally, with insulated carbon holders leading the current to the inside of the furnace. Within this shell many different forms of furnace, such as vertical or horizontal arc or resistance core, could be built up as illustrated in fig. 11 (p. 438).

The construction of the enclosure will be easily understood by reference to figs. 1 and 2, giving sectional drawings. The shape of the interior is cylindrical, 10 inches diameter by 17 inches long, with hemispherical ends, one of which forms the cover B and is held in place by ten $2\frac{1}{4}$-inch studs (F_1, F_2) which are fixed into a flange of the main forging. The cover is rendered gas-tight by a spigot joint S, packed with lead; it is surmounted by a cast-iron casing H, through which cooling water was circulated.

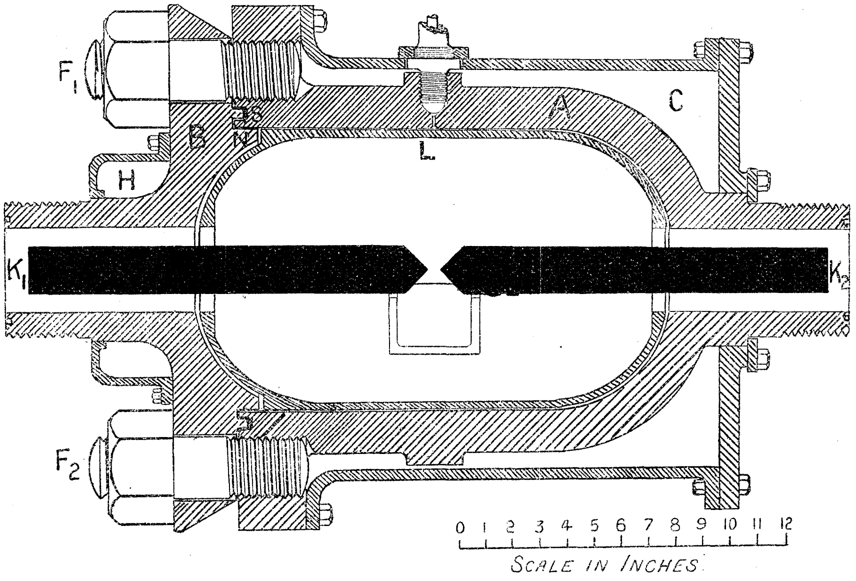

Fig. 1. Sectional drawing of large pressure furnace.

A. Main forging, $1\frac{1}{2}$ inches ruling thickness increased to 2 inches over central belt, through which the various openings are bored.

C. Water jacket surrounding the body of the furnace.

H. Water jacket surmounting the cover.

B. Cover held down by ten $2\frac{1}{4}$-inch studs, the joint being made by a lead ring placed in the spigot groove S. The projection N protects the joint from contact with the hot gases when the furnace is in use and shields it from mechanical injury while the cover is being lifted or replaced.

L. Cast-iron lining.

The main forging A is surrounded by the cast-iron water jacket C.

Both the hemispherical ends of the furnace have projections K_1, K_2 bored out to a distance of 3 inches.

The carbon holders which move in these recesses are thus protected from the direct heat or flame of the furnace. The length of the projections K_1, K_2 is sufficient to allow a feed of 8 inches. To obviate any risk of damage to the main forging by contact with the hot furnace materials, a cast-iron lining L was always used.

When in a horizontal position the furnace rests on four cast-iron feet (not shown in the figure). When vertical it is supported by the cover of the water jacket, the lower carbon feeder passing through a hole cut in a massive wooden stand.

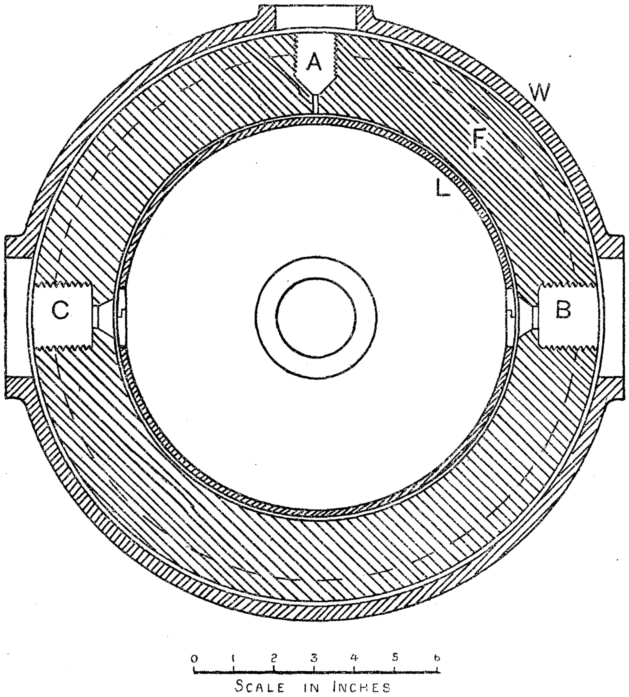

Fig. 2. Transverse section through the centre of the large furnace.

W. Water jacket.
F. Main forging.
L. Cast-iron lining.

The inlet valve is screwed into A, whereas the openings B and C receive the windows shown in fig. 4, outlet valves and gauge connections, or, when required, auxiliary insulated terminals.

The main forging is provided with three openings, as shown in fig. 2, which is a section through the centre of the furnace perpendicular to the axis of the carbons.

The aperture A served to receive the valve through which the enclosure was filled with compressed gas, whereas in most cases one of the windows shown in fig. 4 was screwed into B. The third opening was connected to a pressure gauge and served also, when desired, for the escape of the gaseous products of reaction.

Carbon Feeding Mechanism.

This is shown in detail in fig. 3. A ring B is fitted to each of the projections K of the furnace. To this ring the small cover A is bolted. The joint is made, as in the case of the main furnace cover, by means of the lead-packed spigot V. The cover carries two columns C_1, C_2 surmounted by a yoke Y which is insulated from them by micanite bushes and washers R. To this yoke the main terminals (not shown in the figure) are fixed. The nut N is revolved by means of steel levers inserted in the holes T, and thus the feeding rod S is moved forward. The feather F fits in a groove cut in this rod and prevents it following the rotary motion of the nut.

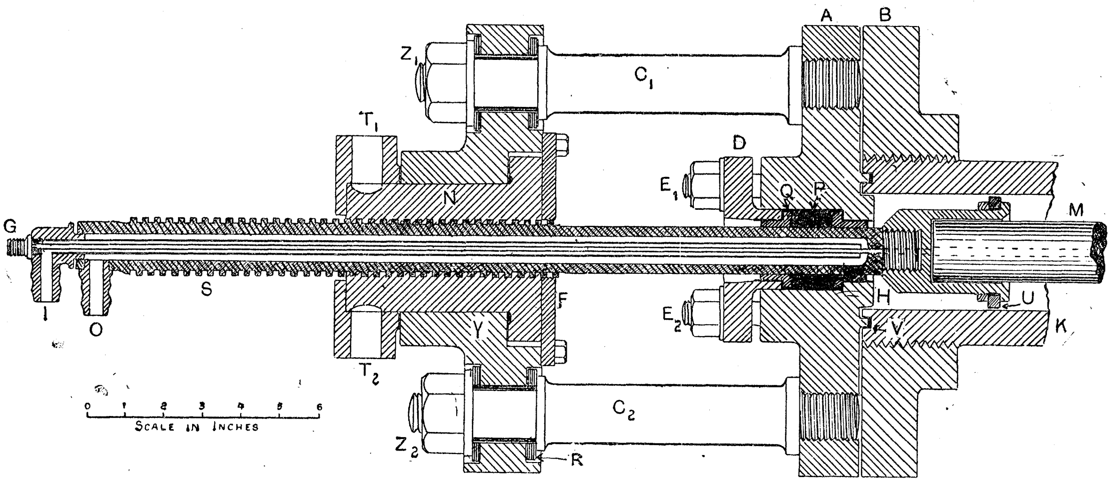

Fig. 3. Carbon feeding mechanism of the large furnace.

The small cover A is bolted down to the ring B, which is fitted on to the projection K, forming part of the main forging.

Two columns, C_1, C_2, support the insulated yoke Y, in which the nut N is revolved by steel levers fitting into T_1 and T_2.

A stream of water flows through the hollow feeding rod S, the nipples I and O carrying the inlet and outlet pipes. A central pipe can be used to pass a flow of compressed gas through the axis of the electrode M.

The glands Q of the stuffing box P are electrically insulated from the cover A.

The electrode M is soldered into the cup-shaped holder H which screws on to the end of the feeding rod.

The feeding rod passes into the furnace through the insulated stuffing box P.

This stuffing box serves the double purpose of making a gas-tight joint and providing insulation sufficiently perfect for the relatively low electromotive force which is generally required with this furnace. As packing, a mixture of asbestos and tallow is used, which in itself assists the insulation.

The stuffing box is compressed by means of the ring D, which presses on the gland Q, but is electrically insulated from it by mica washers and bushes. The inner gland is insulated in a similar manner from the steel cover.

The feeding rod is hollow. The current of water passes into it at I, and flowing through an inner brass tube is delivered at the extremity of the rod and passes back to the outlet O. A gas connection G is also provided by means of which compressed gas can, when necessary, be passed directly into the centre of the furnace through the axis of a hollow electrode (M).

Carbon Holders.

These are of two different patterns, according to the size of the electrode used.

Carbons of 30 millims. diameter or less are held by clamps similar to those used with the smaller furnace, as shown at T in fig. 6 (p. 429).

For larger carbons the limited space available prohibits the use of such an arrangement, which in any case would hardly provide a sufficiently good contact for heavy currents (500 to 1000 ampères).

The larger carbons are therefore electro-coppered at their ends, and soldered into cup-shaped holders, as shown at H (fig. 3). The lip of the cup is fitted with a ring of refractory insulating material, U, which nearly fits the bore of the tube K, and thus protects the stuffing box from flame and dust.

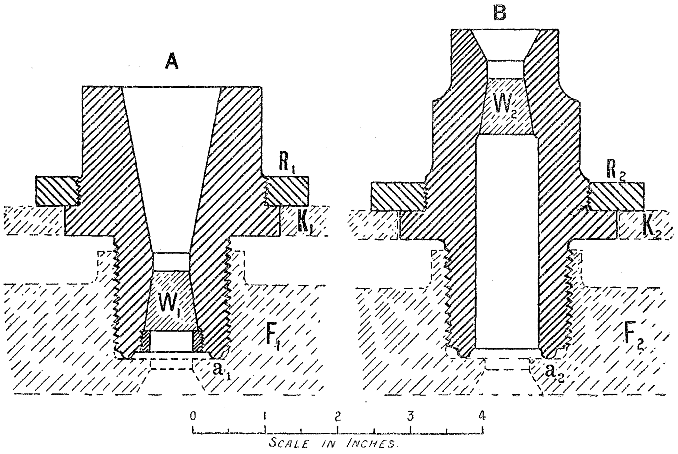

Fig. 4. Windows.

A and B. Gun-metal fittings carrying the glass windows W.
F. Steel wall of furnace enclosure.
a. Gas-tight ring joint.
R. Ring making water-tight joint between the fitting and the water jacket K.
The design A is used when working with arcs of small intensity, and B for larger currents.

Windows.

Although unnecessary for the purely chemical work, it was of considerable importance to be able to observe, project, or photograph the arc itself or its spectrum under conditions of high pressure.* By providing two openings (B and C, fig. 2) diametrically opposite, absorption spectra could be observed during the operation of the arc.

The forms of construction are shown in fig. 4. The window itself consists of a glass or quartz cone (W) $\frac{3}{4}$ inch thick and $\frac{1}{2}$ inch diameter at its smaller end. This

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* "Preliminary Note on the Effect of Pressure upon Arc Spectra," J. E. PETAVEL and R. S. HUTTON, 'Phil. Mag.,' Nov., 1903, vol. 6, pp. 569–577.

transparent cone is forced into the gun-metal fitting after being surrounded with a thin film of cement, and is held in place by a metal ring, the shape of the glass tending to make the joint more perfect the higher the pressure.

The joint between the fitting and the aperture in the main forging is made by the ring *a* turned on the end of the fitting, which presses tightly against a steel ledge, no packing being required.

The joint between the fitting and the water jacket is made by means of a gun-metal ring R, which screws on to the fitting itself.

The two types of window differ only in the relative position of the transparent plug. The design shown at A gives a clearer view of the arc, but for very large currents it is advisable to make use of the fitting B, in which the glass plug is more carefully protected from the source of heat.

Valves and Gas Connections.

The types which have already been described* were employed for regulating the flow of the various gases used.

After the construction of the furnace was completed it was tested to 450 atmospheres, and has since been used frequently up to 200 atmospheres gaseous pressure.

At first it was anticipated that so large a joint as that of the main cover would show some leakage at the higher pressures, the total stress on the bolts retaining the cover amounting under ordinary working conditions to over 100 tons. These fears were, however, not realised, the only precautions necessary being to keep the joint perfectly clean and, of course, to tighten up the nuts evenly all round. Occasionally, as the pressure rose, a slight escape of gas was noticed, but this was stopped without any difficulty by tightening up the corresponding stuffing box or joint.

In fact, it may be said that throughout the work no difficulty has been experienced in keeping the apparatus gas-tight.

The furnace is, of course, equally suitable for work *in vacuo*, and has occasionally been used in this way—in connection, for instance, with spectroscopical investigations.

In the course of the present research the electrical conditions have varied widely, in some cases as much as 1000 ampères, in others 500 volts, having been employed without difficulty. The power used in most experiments was between 10 and 15 kilowatts. When it is desired to use high-tension currents (1000–25,000 volts) with this furnace the carbon feeding mechanism of the small apparatus can be used to replace that described above, with which it is interchangeable.

The weight of the enclosure was, of course, considerable, and to facilitate its manipulation a crane was fixed to the main laboratory wall. The crane is regularly employed for the removal of the cover, and serves also to lift the entire furnace and change its position from horizontal to vertical, or *vice versâ*.

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* J. E. Petavel, 'Phil. Trans.,' A, vol. 205, p. 369, 1905.

Small Furnace for High-Tension Currents.

Since there are a considerable number of electric furnace reactions, as, for instance, most gas reactions, for which very high-tension currents are necessary, it was decided to complete the equipment by providing a furnace specially for this class of work.

In this case a large capacity, instead of being an advantage, would constitute a serious drawback, owing to the difficulty of preparing and purifying such a quantity of gas. Moreover, in the case of a high-tension arc, the natural temperature gradient is so steep that the maximum temperature is easily and safely attained even in a small enclosure. The rapid rate of cooling which results from the proximity of the arc to the cold walls of the furnace is also of material advantage.

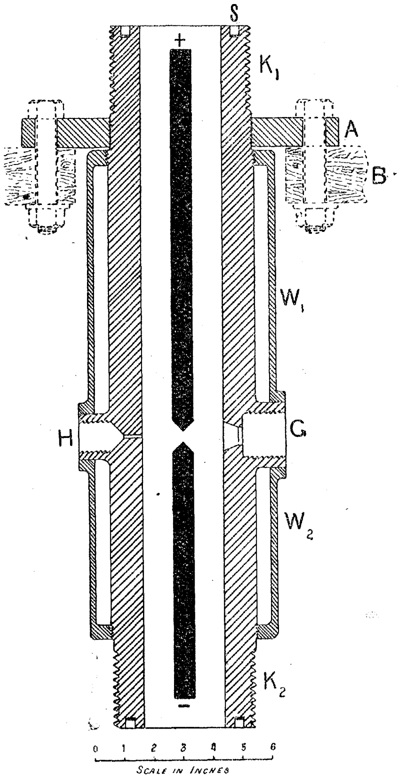

Fig. 5. Sectional view of small furnace.

The body of the furnace is suspended from a cast-iron plate A bolted to a wooden stand B.

The two ends K_1, K_2 are closed by covers which carry the feeding mechanism (see fig. 6). The body of the furnace is surrounded by a water jacket W.

A little below the centre the walls are thickened up to 2 inches and are pierced by two openings. The inlet valve screws into H, and a window (see fig. 4) into G.

The smaller furnace is of one-tenth the capacity of the larger, and consists of a mild steel cylinder about 3 inches internal diameter, with walls $1\frac{1}{4}$ inches thick, surrounded on the outside with a water jacket (see fig. 5). The walls are thickened up a little below the centre, and two openings are bored in the ring thus formed. One of these

(H) serving for the inlet valve, the other (G) for receiving one of the windows we have already described, the size and form of the openings being such as to render all fittings interchangeable with those used in the larger furnace.

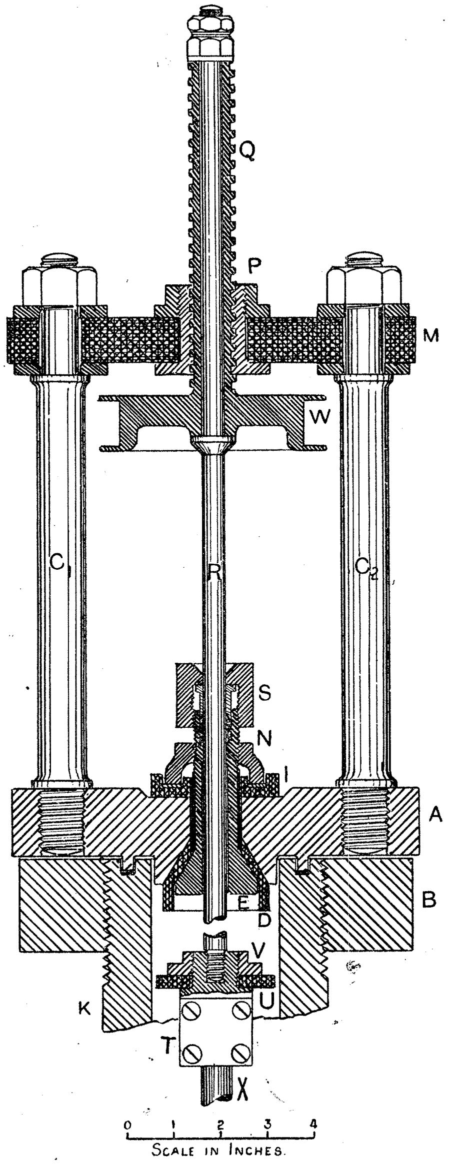

Fig. 6. Carbon feeding mechanism for the small electric furnace.

The ring B screws on to the end K of the furnace and supports the cover A. Into this are screwed the columns C which carry a thick plate of insulating material M.

The feeding screw Q is rotated by means of a cord passed over the pulley W; this screw works in the nut P which is fixed in the centre of the insulating plate (M).

The electrode X is held in a clamp T which is fixed to the feeding rod R.

This rod passes into the furnace through the stuffing box S, which forms the upper part of the central steel plug E. This plug is forced by the nut N against a cupped insulating piece D which fits a recess in the furnace cover.

The chief characteristic of this furnace lies in the means employed for insulating and moving the carbon holders. The mechanism is clearly shown in figs. 6 and 7. A conical hole tapering outwards is bored in the centre of the covers of the cylinder, and receives a cup-shaped insulating bush (D) of ebonite or red fibre. A central plug E is provided with a mushroom-shaped end which fits closely into this cup and is lightly drawn against it by means of the nut N placed on the outside, this nut in turn pressing on the insulating ebonite washer I.

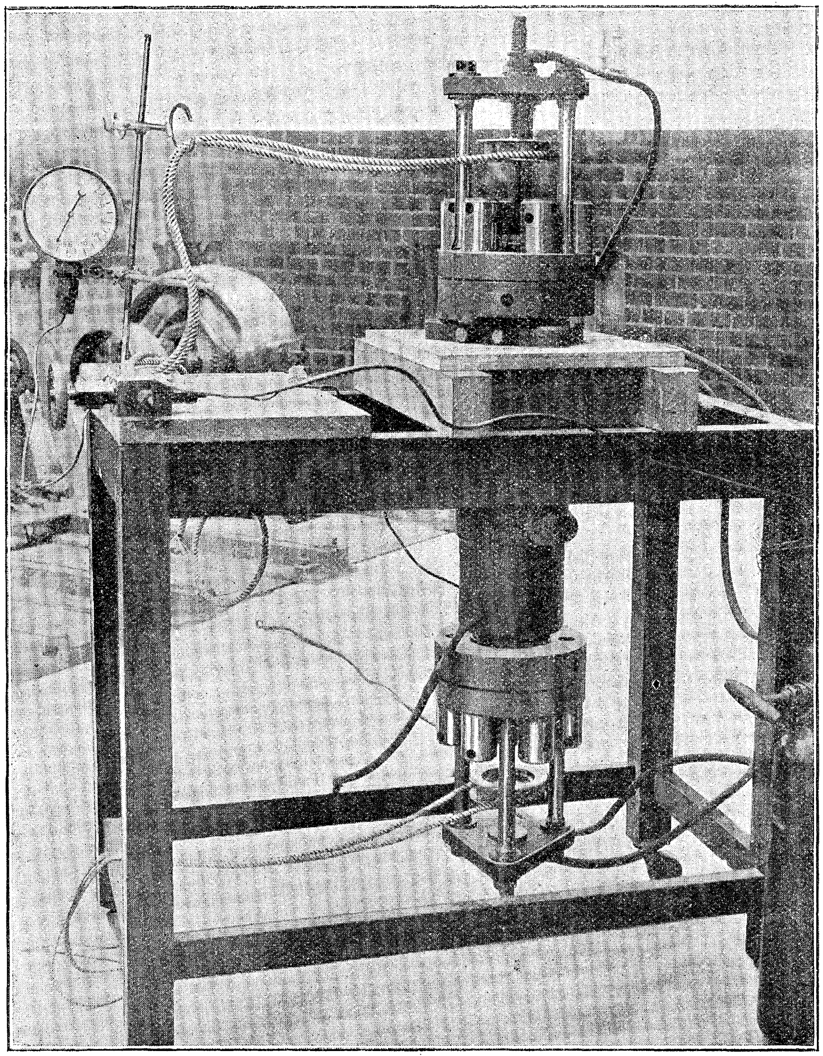

Fig. 7. Small high-pressure furnace.

The gas pressure itself forces down this plug firmly on its seat and secures a satisfactory joint. The upper end of the steel plug contains the necessary stuffing box (S) through which passes the feeding rod R.

From each cover of the furnace project three steel pillars, carrying at their extremities a thick triangular plate M of insulating material. To the centre of this is fixed the nut (P) in which the screw-gear works. This screw-gear is rotated by means of a cord passing over the pulley W. Thus, when the furnace is being used for very high electric tensions, the feeding gear can be manipulated from a safe distance. This form of insulation has been found to work satisfactorily up to 3000 volts. For higher electromotive forces one of the steel feeding rods is removed and replaced by a thick-walled glass tube. The current, which under these conditions is of course very small, is led by a number of fine platinum wires fused through the glass to the electrode, which is supported on the end of this rod.

With this modification the furnace has been used up to 25,000 volts.

Gas Preparation and Compression.

In connection with this research it was necessary to manufacture and compress relatively large quantities of hydrogen, carbon monoxide, ethylene, and nitrogen.

The question has been dealt with fully in a recent publication to which we must refer those specially interested in this branch of the subject.*

GENERAL OBSERVATIONS ON THE ELECTRIC ARC UNDER HIGH GASEOUS PRESSURES.

At the time the present research was started very little information was available with regard to the electric arc burnt in a compressed atmosphere, the investigations having been limited to small arcs and pressures of about 15 atmospheres. The belief was then current that it would prove to be impossible to maintain an arc under gaseous pressures of 100 or 200 atmospheres.

This erroneous conclusion was due to a misconception of the nature of the difficulties which had been encountered by the various workers.†

For spectroscopical investigations it is necessary to obtain a relatively long arc giving a clear flame between the extremities of the electrodes. Such an arc, it is undoubtedly true, can only be maintained in a dense atmosphere by means of an exceptionally high electromotive force.

It should nevertheless be clearly understood that even low-tension arcs will burn without difficulty. At ordinary current densities the entire phenomenon is, however, confined to the crater itself, and a projected image of the arc shows merely the outline of the white-hot crater, and only occasionally is a flash of flame visible on the periphery.

Electrically the arc is still well characterised by its high electromotive force, which instantly disappears if the electrodes are brought into actual contact.

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* HUTTON and PETAVEL, 'Journ. Soc. Chem. Ind.,' 1904, vol. 23, pp. 87 to 93.

† W. E. WILSON and G. F. FITZGERALD, 'Roy. Soc. Proc.' 1896, vol. 60, pp. 377–383.

When the arc is started in a compressed oxidising atmosphere, the current is at first unsteady and the electrodes must be rapidly fed up. Soon, however, a steadier state is reached. If, after such a run, the furnace is opened and the carbons examined, it will be observed that, by the action of the current, the electrodes have been so shaped as to nest one into the other as shown by the dotted lines in fig. 8. In this way a considerable cross-sectional area is produced over which the discharge can occur.

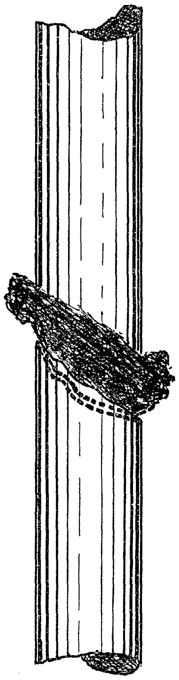

Fig. 8. Configuration of carbon electrodes after use in an inert gas under high pressure.

The end of the negative carbon becomes moulded to the shape of the crater of the positive and projects into it as shown by the dotted lines.

A mushroom-shaped deposit of carbon accumulates on the extremity of the negative electrode.

The E.M.F. of the arc rises as the pressure of the surrounding atmosphere increases* and at the high pressures used in the course of this work it becomes more than double the normal value.

It is, however, the first few atmospheres which produce the greatest effect upon the voltage.

A detailed analytical investigation of the arc would in itself, as can be judged from the great amount of work carried out at ordinary pressures, require considerable expenditure of time and is outside the scope of the present research. We therefore limit ourselves here to the few observations recorded in Table I. (p. 451), in which the behaviour of the arc under certain definite conditions of pressure and current is recorded.

An interesting effect is throughout noticeable. Although the maximum length of arc is so much reduced, the voltage is in all cases abnormally high, and consequently a large amount of power is concentrated in a small space.

Two very distinct types of arc exist. The most usual in these enclosed furnaces is found with a non-oxidising atmosphere such as carbon monoxide or nitrogen.

In such arcs at ordinary current densities the electrical conditions are complicated by the rapid growth of a deposit of carbon, chiefly around the negative electrode and often completely enclosing the end of the positive electrode. The difference between a resistance and an arc is then less marked. The arc flame is not visible, but is replaced by a zone of brightly incandescent carbon; electrically the conditions are ill-defined and difficult to reproduce. It is consequently only during the early part of the experiment that concordant measurements can be obtained. As time goes on the carbon deposit builds itself up, the voltage shows a tendency to rise, and the general appearance

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* DUNCAN, ROWLAND, and TODD, 'Electrical World,' 1893, vol. 22, p. 101, for 6-ampère arcs up to 10 atmospheres pressure.

indicates that the electrical conditions more nearly approach those of a resistance than of a true arc.

It is, however, almost inconceivable that a power of 5 or 10 kilowatts (see fig. 10, B and C) can be expended in such a limited volume of solid material without volatilising it; and, as we shall see, the amount of disruption as evidenced by th feed required is under these conditions extremely small.

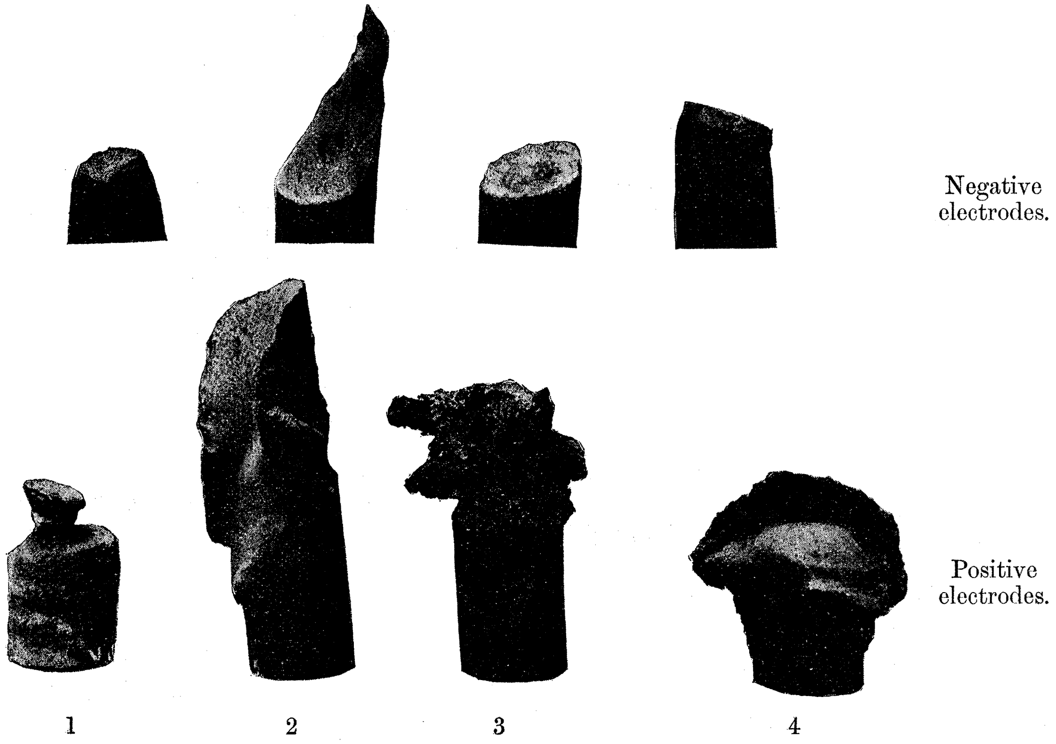

Fig. 9. Photograph of carbon electrodes after use. (Horizontal arc.)

No.	Pressure, atmospheres.	Current, ampères.	E.M.F., volts.	Gas.
1	30	60	60	Originally air, but all oxygen fixed by carbon.
2	30	400	65	Ditto.
3	50	100	100	Coal gas.
4	48	120	130	Originally air, but all oxygen fixed by carbon.

With an exceptionally high current density the arc in a non-oxidising gas at high pressure gives a well-defined flame.

In fig. 10, D, a comparison is given between arcs in carbon monoxide at 11 and 16 atmospheres and an experiment made by Mrs. AYRTON[*] with an enclosed arc at ordinary pressure, using carbons of similar size. It will be seen that the increase of voltage due to an increase of pressure of 15 atmospheres is very considerable. For

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[*] Mrs. AYRTON, 'The Electric Arc,' p. 304.

these high current densities the apparent resistance of the arc under pressure remains positive just as at atmospheric pressure.

An entirely different type is obtained in an oxidising atmosphere, and in this case alone are the results comparable with the well-known conditions of the ordinary open arc.

In fig. 10, E, the E.M.F. of a 150-atmosphere arc of about 2 millims. length is given and compared with measurements made at atmospheric pressure, the positive carbon being 41 millims., the negative 27 millims. diameter.

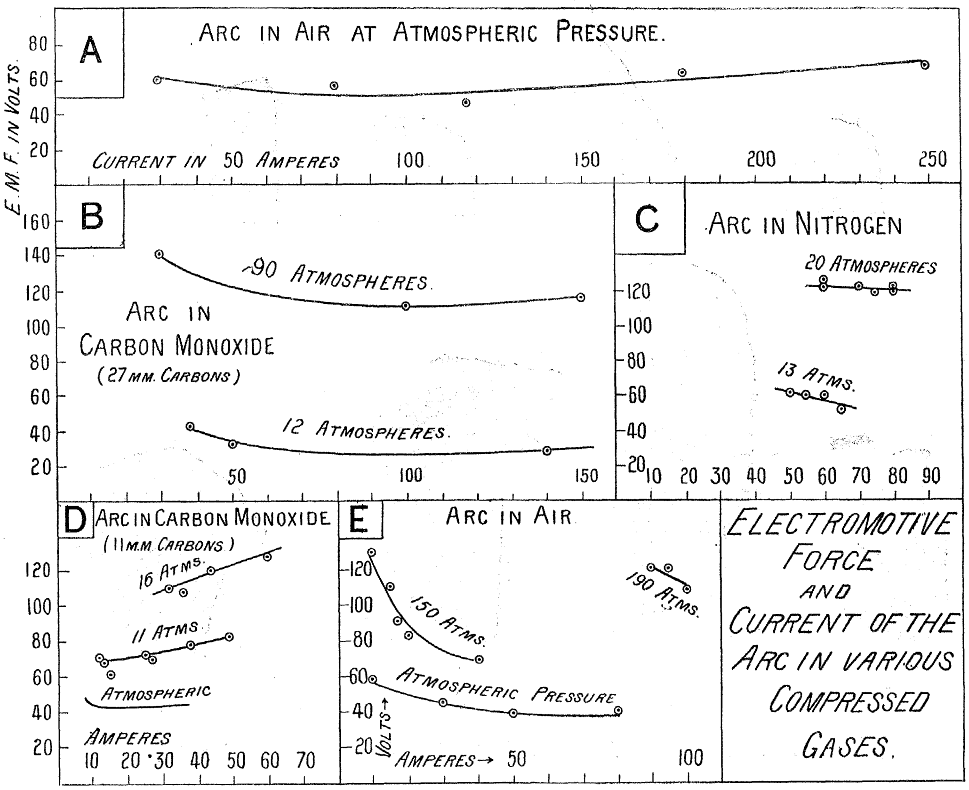

Fig. 10. Curves of the electromotive force and current of the arc in various compressed gases.

The data refer to the conditions existing while a fair proportion of the oxygen was still present; the arc then shows a bright flame which, if the electrodes are fed up rapidly and with regularity, can be easily maintained. Here again the increased voltage observed is due principally, as shown by other experiments, to the first 10 or 15 atmospheres.

For the purpose of general comparison, few data on large current arcs being available, a number of measurements were made with an open arc burning at atmospheric pressure between carbons (positive 41 millims., negative 27 millims.) and maintained at a constant length of 8 millims. The curve thus obtained is recorded in fig. 10, A.

Finally it should be mentioned that, in the course of the chemical work to be described, constant use has been made of "smothered" arcs, as, for instance, in the production of calcium carbide. With arcs of as much as 500 ampères between the extreme limits of our experiments, *i.e.*, from 0·5 atmosphere up to nearly 200 atmospheres, no consistent effect on the voltage which could be ascribed to the influence of gas pressure was apparent.

A noticeable feature in all the experiments carried out in air is the very rapid rate of burning of the electrodes.

To take one instance: with 27-millim. carbons and a 30-ampère arc under a pressure of 29 atmospheres the carbon was consumed at a rate of about 6 millims. per minute, which is nearly twenty times as fast as at ordinary pressures.*

This burning is principally limited to the positive electrode, which in the above experiment was consumed seven times as fast as the negative, the relative rates at ordinary pressures being about as 3 : 1. At a still lower current density the negative carbon shows practically no loss of weight.

Under the normal conditions of most of our experiments, that is with very high current densities, the combustion becomes very violent.

The oxygen thus becomes rapidly exhausted and the subsequent feeding required is relatively very small.

At first carbonic acid is formed which is in turn reduced to carbon monoxide. This reduction occurs also when the arc is run in an atmosphere of compressed carbonic acid. In the latter case an interesting observation was the separation of flocculent carbon which was seen to be moving rapidly in the convection currents. This phenomenon is observed from the commencement of the experiment.

The decomposition of the carbonic acid under these conditions is comparatively slow, in one experiment less than half being decomposed by the end of half an hour.

A somewhat similar process goes on also in non-oxidising gases. Finely divided carbon is deposited in considerable quantities on the cool walls of the enclosure, the weight collected being in fair agreement with the loss from the electrodes. With such gases the atmosphere, however, remained perfectly clear.†

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* B. MONASCH, 'Der elektrische Lichtbogen,' p. 22.

† In cases in which a hydrocarbon gas atmosphere (*e.g.*, coal gas) is employed, the deposition of carbon is augmented by the breaking up of the higher hydrocarbons.

The following analyses give the constitution of the coal gas before and after a run at about 60 atmospheres, which lasted 36 minutes with a power of about 10 kilowatts, a horizontal arc being employed. In this case both electrodes had increased in weight, the positive by 6, the negative by 15 grammes:—

	Before run.	After run.
Hydrocarbon vapours	1·0	0·0
Heavy hydrocarbons	4·2	0·7
CO_2	2·0	0·6
CO	15·0	14·7
CH_4	18·0	24·9
H	42·0	41·8
O	1·7	0·0
N (by difference)	16·1	17·3
	100·0	100·0

The interesting results thus obtained with regard to combustion led us to carry out some experiments on the oxidation of electrodes of other materials, the production of an atmosphere free from oxygen and the oxides of carbon being also of considerable practical importance for the further work we had in view. *A priori* one would be led to believe that copper, iron, or aluminium when heated and fused in highly compressed air (100 atmospheres) would not only rapidly fix the available oxygen, but would do so with sufficient intensity to make the combustion self-supporting.

Repeated attempts were made to produce this result, a summary of which will be found in Table II. (p. 452).

Briefly speaking, we may say that with an iron bar maintained for one hour at a bright red heat and then partially melted by means of a current rising to 1000 ampères, the percentage of oxygen fixed was hardly appreciable.

A similar result was obtained in the case of copper, whereas even aluminium melted in the arc at a pressure of 26 atmospheres only oxidised on the surface, the well-known tenacity of the oxide films being sufficient to prevent the rapid combustion of the metal.

The negative results thus obtained led to some experiments being carried out with oxygen.

An arc was struck between two iron bars of $1\frac{1}{16}$-inch diameter, surrounded by oxygen at 15 atmospheres pressure; as soon as the temperature reached a bright red heat, a vivid combustion commenced on the positive electrode and continued quite steadily, although the current was then cut off.* After the combustion had proceeded a short time the pressure was gradually reduced, the combustion ceasing when the pressure had fallen to about 5 atmospheres. A length of some 5 inches of the bar had by this time been consumed, the loss of weight being 535 grammes, the loss on the negative electrode being only 15 grammes. The product of the reaction was collected in a crucible placed for this purpose under the arc, and on analysis proved to be magnetic oxide (Fe_3O_4).

It is worthy of note that although the partial pressure of the oxygen in the experiments previously referred to was considerably above that required in the case of the pure gas, the combustion was not merely insufficient to maintain the temperature required for continued combustion, but even with the assistance of the arc the total iron burnt was almost negligible. The result is due probably to the high effective heat conductivity which is characteristic of compressed gases, the evolution of heat due to oxidation being, in the diluted gas, insufficient to overcome this cooling effect.†

It was thought also that oxygen might easily be removed from the atmosphere by the introduction of successive small quantities of hydrogen, care being taken to always

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* E. FRANKLAND, 'Journ. Chem. Soc.,' 1864, vol. 17, pp. 52–55, describes an interesting case of the combustion of iron in compressed oxygen.

† J. E. PETAVEL, 'Phil. Trans.,' A, vol. 197, pp. 229–254, 1901.

keep below the explosive limit, a small arc being maintained to effect the combination. Under these conditions, however, the water vapour produced was rapidly converted into carbon monoxide and hydrogen under the action of the arc. The method had, therefore, no advantage over the direct combustion by carbon, and was abandoned.

In many of the cases in which a carbon arc was maintained after all the oxygen of the air had been fixed, small traces of hydrocyanic acid were detected. In the above experiment, in which hydrogen was present in considerable amount, the formation was much increased.*

On the Formation of Calcium Carbide.

The production of calcium carbide, constituting a simple and typical example of electric furnace reactions, was considered a suitable subject for the first series of experiments.

From previous experience we were impressed with the necessity of maintaining the greatest possible uniformity in the conditions under which the furnace was operated.

Above all it was desirable to avoid the variations introduced by the use of different forms of furnace construction, and therefore for all the experiments dealt with in this section we have employed the simple type represented in fig. 11, A, the dimensions of the furnace, the size of the electrodes, the weight and constitution of the reacting mixture being kept the same and, as far as possible, the factors under consideration varied only one at a time.

The building up of the furnace entailed the use of the enclosure in a vertical position as shown in Plate 6, fig. 1. The furnace cover having been removed, by means of a crane installed for the purpose, the cast-iron liner is raised and deposited on a separate stand where it is prepared for the experiment. To protect the bottom of this receptacle from the direct action of the arc, a layer of powdered retort carbon is first introduced, which thus constitutes the lower electrode. The mixture of lime and carbon (about 10 kilogs.) is then filled up around a paper tube which serves to keep a central passage free for the upper electrode.

If there is any doubt as to the perfect desiccation of the raw materials, the cast-iron pot with its contents must be maintained at a red heat for some hours before it is placed in the enclosure. This not only ensures more consistent results, but renders it possible to follow the progress of the reaction by a measurement and examination of the gases generated. The cover of the enclosure is lowered carefully into position, the carbon electrode sliding into the cylindrical space which has been reserved for it.

After the bolts have been tightened up and the desired quantity of gas introduced, the arc is started by lowering the upper electrode, which then comes in contact with the carbon bed beneath it.

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* H. Hoyermann, 'Chem. Zeitung,' 1902, vol. 26, pp. 70, 71; J. Gruszkiewic, 'Z. für Elektrochemie,' 1903, vol. 9, pp. 83–85; H. Auer, 'Acad. Sci. Buda-Pesth,' 1904.

During the experiment the voltage and current are read at frequent intervals; the watts, at these instants, plotted on a curve enable the total kilowatt hours supplied to the furnace to be obtained by integration.

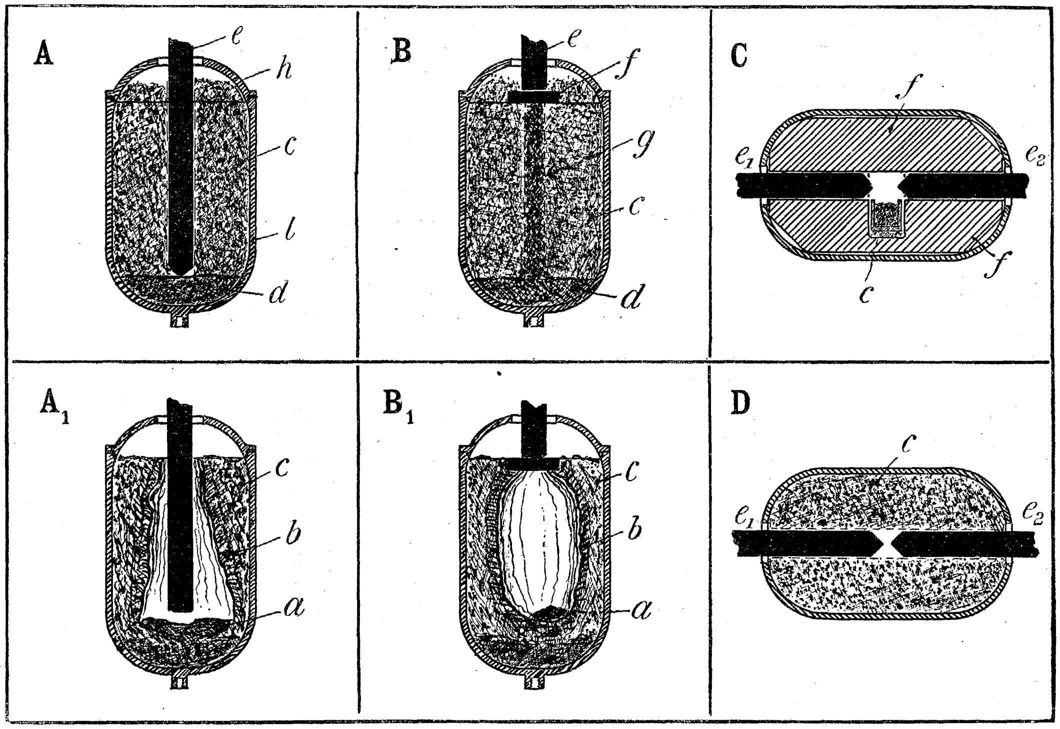

Fig. 11. Sectional views of various types of electric furnace.

A. *Smothered arc* (*before run*).

l is the cast-iron liner in which the charge was placed. *h*, cover of same. *e*, vertical carbon electrode (41 millims. diameter). *d*, granular carbon bed forming the lower electrode. *c*, charge.

A_1. *Smothered arc* (*after run*).

a, ingot of fused product. *b*, fused and fritted material forming walls of cavity. *c*, unacted-on material.

B. *Resistance* (*before run*).

e, carbon electrode. *f*, graphite end-piece leading current to core. *g*, resistance core of granular material or carbon rod, or other solid "resistor." *c*, charge. *d*, granular carbon bed or other form of lower electrode.

B_1. *Resistance* (*after run*).

a, ingot of fused product. *b*, fused and fritted material forming walls of cavity. *c*, unacted-on material.

C. *Horizontal arc: radiation heating.*

e_1, e_2, electrodes. *f*, walls or jacket of heat insulating material. *c*, charge in carbon or other crucible.

D. *Smothered arc with two carbons embedded in the material* (used horizontal or vertical position).

e_1, e_2, electrodes. *c*, charge.

As the reaction proceeds, the pressure in the furnace, due to the evolution of carbon monoxide, rises rapidly and the fused carbide collects upon the bed of carbon and soon accumulates sufficiently to come in contact with the end of the electrode. The sharp drop of the electromotive force thus produced serves to indicate that the time has arrived to raise the carbon slightly.

There is no difficulty in maintaining these smothered arcs even at the highest pressures, and the regulation of the power is quite a simple matter. Currents of some 500 ampères have frequently been employed in the course of this work and maintained as long as desired.

It is very noticeable that, contrary to experience with such arcs when maintained at atmospheric pressure, the upward rush of the gases through the finely divided charge causes very little displacement of material. For a given rate of reaction the velocity of the gas currents is of course roughly in inverse proportion to the working pressure. At high pressures, therefore, the gaseous products of reaction rise at a relatively slow speed and percolate through the mixture without disturbing it. When, on the other hand, the enclosure is evacuated the projection of the material is very marked and causes considerable trouble.

An extremely low consumption of the carbon electrodes is characteristic of these enclosed furnaces, the deterioration of the electrodes being so slight that they can be repeatedly used. In general practice the loss in weight of the electrodes is an important question and in favourable cases is still between 1 and 3 per cent. of the output of carbide. The consumption in ordinary furnaces on a laboratory scale is still larger, whereas in the present experiments the loss has always been so small as to be hardly appreciable.

We may therefore conclude that the corrosion is not due to the dissociation of the lime as suggested by GIN,[*] but is to be ascribed to atmospheric oxidation.

A summary of the more important experiments will be found in Table III., whereas Table IV. gives the detailed observations referring to one typical case.

Before discussing these results it may be well to consider briefly the mechanism of the chemical reaction upon which the formation of the carbide depends.

It is generally stated that carbon first commences to react readily with lime when the latter reaches its melting point, the production of carbide below this temperature being limited and of little practical importance.[†]

In a careful investigation of ROTHMUND[‡] it has however been shown that a definite equilibrium exists at about 1600° C., as represented by the equation

$$\mathrm{CaO} + 3\mathrm{C} \rightleftharpoons \mathrm{CaC_2} + \mathrm{CO},$$

the reaction tending to go from right to left at higher, from left to right at lower

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[*] G. GIN, 'Z. für Elektrochemie,' 1902, vol. 8, p. 397.

[†] H. MOISSAN, 'Comptes Rendus,' 1904, vol. 138, pp. 243–245.

[‡] V. ROTHMUND, 'Z. für anorg. Chemie,' 1902, vol. 31, p. 136.

temperatures than this. The formation of carbon under these conditions has also been observed by A. Frank.*

From this it would at first sight appear that, if the carbon monoxide resulting from the formation of calcium carbide were retained in the furnace and the pressure allowed to accumulate, the reaction would soon come to a standstill.

Our experiments are, however, in direct opposition to this conclusion, proving that the temperature prevailing in the furnace is sufficiently far above the point of equilibrium to preclude the inverse reaction so long as the heating is continued.

On the other hand, the above considerations alone might lead one to suppose that, already at any temperature above 1600° C., the formation of carbide would progress rapidly to completion, provided only that free exit were allowed for the gaseous products of reaction. It must, however, be remembered that the process is endothermic, and can therefore only proceed at a pace measured in terms of the rate at which energy is being supplied to the furnace.

We have carried out a large number of experiments specially to study the effect of the presence of carbon monoxide upon the yield (see Table III.).

In these and in other cases the resulting product was submitted to a careful examination and analysis.

The sectional view shown in fig. 11, A_1, gives an idea of the general appearance of the furnace after the run.

The furnace contents consist of (*a*) ingot of fused calcium carbide, (*b*) fritted mass surrounding the central cavity, (*c*) residual unacted-on material. These were separately collected and weighed, then parted and sampled, and subsequently analysed in the manner described below.

Generally speaking, the central lump represented the entire yield of carbide, although small quantities of acetylene were sometimes obtained from the fritted mass.

The yields recorded are all calculated from the amount of acetylene produced. The gas evolved was always carefully analysed, as the possibility presented itself of the formation of other carbides or free calcium metal. The amount of impurity was, however, invariably found to be insignificant. The outside unfused material was examined, but gave no appreciable evolution of a combustible gas when acted upon by water, or even by dilute hydrochloric acid.

The ingots of carbide showed a good crystalline fracture. The purity of the lump was, as might be expected, below that of a good grade technical product, but increased as the rate of power expenditure rose, and, curiously enough, was entirely independent of the presence or absence of carbon monoxide.

When the carbon monoxide was retained in the furnace the ingot frequently showed on its upper surface a thin coating of bright graphite, giving it a metallic appearance, and in some few cases narrow strata of graphite plates occurred within the mass itself.

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* A. Frank, 'Z. für angew. Chemie,' 1905, vol. 18, p. 1733.

The experiment detailed in Table IV. was one of those in which all the gaseous products of reaction were retained in the furnace until long after the completion of the run.

Fig. 12, which refers to this experiment, is reproduced, since it is typical of many of those dealt with in the section.

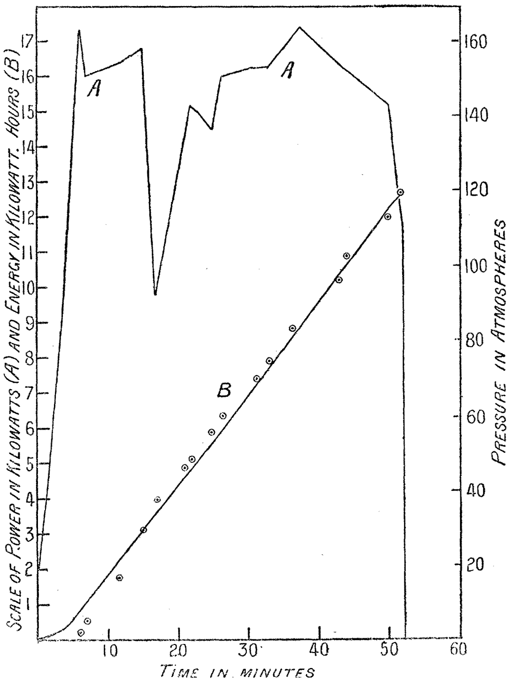

Fig. 12. Energy, power, and pressure curves of a calcium carbide experiment.

The curves refer to the experiment dealt with in Table IV., in which the furnace was started at atmospheric pressure and all the gas generated was retained.

Curve A represents the power in kilowatts at each instant.

Curve B is obtained by integration of A, and gives therefore the total energy expended up to any given time.

The readings of the pressure gauge on the furnace, taken at intervals, are marked on the diagram, and fall nearly upon the energy curve.

The power at each instant during the run is given in curve A, whilst the total energy expended up to any given moment, obtained by an integration of this, is given at B.

In all the experiments the pressure after the first few minutes rose at an almost constant rate. When the pressure observations are plotted to a suitable scale they coincide with the integrated power curve. In the figure their position is indicated, and the concordance can thus be clearly seen.

The interpretation is not so simple as it appears, for there are two disturbing factors which must be taken into account: firstly, the thermal loss, which increases as the hot zone widens out, and, secondly, the variation of the average temperature of the furnace, the effect of which will superpose itself upon the actual gas evolution and thus augment the pressure readings. Apparently these two factors counterbalance each other.

From the analysis of the furnace gas [see Table IV. (3)] a re-absorption of carbon monoxide is clearly indicated. The fall of pressure after the end of the run cannot of course form the criterion, as its amount will vary with the distribution of temperature in the furnace. The considerable decrease in the *percentage* of the carbon monoxide can, however, only be ascribed to re-absorption, and the results are thus so far in agreement with those of the observers mentioned above.

That this absorption is not due entirely to physical causes is shown clearly by comparison with a similar series of analyses carried out in the case of carborundum (Table VII.).

There is little doubt that the graphitised surface of the ingot, referred to in a preceding paragraph, is a result of the recombination of the carbon monoxide. The instant the power is cut off, the temperature of the molten mass begins to fall rapidly, and it is during this period that the back reaction chiefly occurs. As soon as the product has frozen, further attack is limited, for the ingot of carbide is of a very compact and impermeable texture, and is protected by the graphite film.

Such a "skin reaction" would doubtless become predominant were the experiments carried out on a few grammes of material, but when dealing, as we are here, with larger quantities, the total loss is too small to influence appreciably the result. This is shown by the fact that the yield is not increased when the carbon monoxide is let off immediately upon stopping (*cf.* Table III., C 26).

The further question, as to whether the presence of an atmosphere of carbon monoxide *during* the run has an unfavourable effect on the efficiency of the process, is also answered in the negative by a comparison with furnaces operated at atmospheric pressure, but otherwise under identical conditions.

These unexpected results called for more detailed study, and a number of experiments were undertaken in which provision was made for carrying away the carbon monoxide as soon as it was formed. The first method which suggested itself was to remove the gaseous products of reaction by dilution with some inert gas, which was alternately introduced and discharged, the pressure in the furnace being made to fluctuate between two fixed values. The record of such an experiment with coal gas will be found in Table III. (C 22), showing, if anything, a decreased yield.

A more efficient method of washing out the carbon monoxide was then devised. A hollow carbon electrode was brought into use, and during the entire run a constant stream of pure hydrogen was injected directly into the reaction zone of the furnace. The current of gas was also maintained during the cooling, the quantity of gas employed in each experiment being some 2000 litres. The pressure in the furnace was regulated and kept constant by one of the valves placed on the side of the enclosure, through which the required amount of gas was allowed to escape.

In view of these experiments, arrangements had been made for communication between those engaged in operating the furnace and the worker in charge of the compressor. The gas connections were so disposed that the gas could be delivered either directly into the furnace or into a receiver communicating therewith. Gauges in the furnace room indicated the pressure on the pump, and also the working pressure of the enclosure.

Most gases had of course to be simultaneously manufactured and compressed, but coal gas, drawn directly from the mains, was occasionally used for simply washing out of the products of reaction.

The result of the circulation was not to increase, but considerably to decrease the efficiency of the carbide formation. It occurred to us, however, that the low yields might be ascribed to the thermal losses entailed by the specific heat and the relatively high conductivity of the hydrogen used for dilution.

In order to be quite free from such objections, it was decided to remove the carbon monoxide as fast as it was formed by means of a pump, and carry out the reaction under a partial vacuum.

To protect the pump from the large quantities of finely divided material, which are carried away with the stream of gas, a number of scrubbers and filters were used. When the furnace is operated at full power, the gas generated by the reaction amounts to some 30 litres per minute, and, although an exceptionally powerful vacuum pump was available, it was only possible to maintain the vacuum at about 30 to 40 centims. of mercury. The average yield obtained in the vacuum experiments does not materially differ from the results already given.

We are therefore justified in concluding that, however contradictory it may seem, even a concentrated and compressed atmosphere of carbon monoxide has no deleterious effect upon the formation of calcium carbide.

Having entered so fully into the important question of the influence of carbon monoxide, it is necessary to deal very briefly with other sides of the question.

Generally speaking, within wide limits (between 5 and 20 kilowatt hours) the total power consumption does not affect the efficiency of the process.

The influence of pressure *per se* has not resulted in any marked change in the chemical or physical nature of the products, neither can a considerable decrease in the yield be traced to this cause. Such variations in the purity or richness of the carbide as have been noticed are attributable only indirectly to pressure, being accounted for by the increased thermal losses in high pressure gases.

Finally, we hope that the general methods of following the course of the reaction by a measurement and analysis of the gaseous products will be as useful when applied to other problems as they have been in this special case.

ON THE FUSION OF SILICA.

When quartz is subjected to a high temperature it attains the vitreous condition and becomes sufficiently plastic to be moulded and shaped.

It was shown some years ago that silica can be heated in direct contact with carbon and brought to a plastic state without marked reduction occurring.

The heating was effected either by radiation from an electric arc or by placing the material around a carbon core* through which the current was passed.

At ordinary pressures it is, however, impossible to maintain any considerable quantity of silica in a really liquid state.

When the power expenditure in the core is increased a rapid volatilisation sets in which effectually puts a limit to the temperature.

It seemed probable that more satisfactory results could be attained by carrying out the fusion under a considerable pressure in the enclosure described above.

Details of the principal experiments will be found in Table V. A resistance furnace of construction similar to that shown in fig. 11, B, was first employed, the central core of granular carbon being replaced by a carbon tube held in two massive graphite terminal pieces to which the current was led. This core was arranged centrally and surrounded by pure quartz sand, the experiments being carried out in air at 50 and 100 atmospheres.

Thick-walled hollow cylinders, 25 centims. long and 15 or 20 centims. external diameter, were in this way easily obtained.

At first sight more complete liquefaction seemed to have occurred. Upon fracture, however, the material was found still to contain innumerable small gas bubbles, giving it a translucent appearance and tending to show that the fluidity had not been much increased.

Proof of the diminished volatilisation of the material was, however, given by the absence of a deposit of condensed silica vapour, as also by the very small formation upon the core of carborundum, both of which are evident at atmospheric pressure.

It was then decided to study two modifications of the *régime*, either of which seemed likely to give improved results.

The well-known ease with which hydrogen passes through heated silica led us to believe that if a compressed atmosphere of this gas were employed any bubbles imprisoned at the moment of fusion would disappear, leaving the glass clear. None of our experiments, however, verified this assumption. Not only is the occlusion of the gas apparently unaffected, but from the nearly explosive violence with which the

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* HUTTON, 'Mem. Manch. Lit. and Phil. Soc.,' 1901, vol. 46, No. 6, pp. 1–5; also 'Trans. Amer. Electrochem. Soc.,' 1902, vol. 2, pp. 105–111.

material splits when subjected to shock there is little doubt that each minute cavity still retains gas under a considerable pressure.*

The small weight of vitrified product is ascribed to the high cooling effect of hydrogen, but the properties of the material were similar to those noted in the earlier work.

Finally, an attempt was made to attain the highest possible temperature by concentrating the heat of a powerful arc in the centre of a mass of sand, the equipment of the furnace being of the type shown in fig. 11, D.

No difficulty was experienced in maintaining an arc of some 10 kilowatts for an hour or more, the electromotive force rising to 120 volts under a pressure of 180 atmospheres.

A hollow sphere of 18 centims. external diameter and weighing about 5 kilogs. was obtained, which, however, was no more transparent than with the less intense heating.

On the Formation of Carborundum.

In 1893 Acheson discovered that a mixture of carbon and silica heated around a resistance core readily produces a carbide of silicon to which the name of carborundum was given. Its preparation is now carried out on a large scale. It was therefore of some interest to study the reaction, first under the usual conditions, then under high pressure.

We have prepared some quantity of this material in the laboratory and found it to differ from the commercial product only in the smaller size of the crystals. The amorphous variety invariably occurs in considerable amount surrounding the crystalline layers.†

In carrying out the reaction in the pressure furnace the mixture of sand and carbon was disposed around a core of granular retort carbon as shown in fig. 11, B.

To insure a uniform cross-section this resistance core was tightly packed into a thin brass tube which was placed in the axis of the furnace and supported between the two graphite electrodes. As the mixture when heated becomes somewhat conducting, it is advisable to line the furnace with a thin insulating layer of sand or other material in order to avoid a short circuit through the iron.

In these experiments the gas generated by the reaction was retained in the furnace and the pressure allowed to accumulate up to some fixed limit (100 atmospheres).

A typical example of such a run is given in Table VI. As will be seen, the brass

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* Relative to this subject an interesting investigation has been carried out by Arthur L. Day in America. Using lower pressures, which, however, were only applied after a high temperature had been attained, he has prepared a glass containing relatively few air bubbles. 'Science,' N.S., 1906, vol. 23, pp. 670–672.

† See also S. A. Tucker and A. Lampen, 'Journ. Amer. Chem. Soc.,' 1906, vol. 28, pp. 853–858.

tube fuses within the first few minutes; the power can then be rapidly increased, and the temperature of formation of carborundum is soon attained.

There is little doubt that the production of carborundum is the result of the interaction of the vapour of silica and the highly heated granular carbon. From the previous work on quartz we were therefore led to anticipate that under pressure the reaction would not occur very readily.

Several experiments confirmed this impression. An examination of the furnace after the run showed, in every case, that, though the mixture of sand and carbon had attained a sufficiently high temperature to effect the fusion of the quartz to a considerable depth, thus agglomerating the mixture, only a small quantity of carborundum was formed and that immediately around the central core.

Another distinctive feature of the pressure experiments is the almost entire absence of the amorphous variety of carborundum. The reaction progresses at a slow rate, but apparently uniformly, the resulting pressure being, as in the case of calcium carbide, a linear function of the time (see fig. 12).

In order to study more in detail the progress of the reaction, an experiment was planned in which the whole of the gas generated was retained in the furnace.

As is shown in Table VII., analyses were made at intervals both during the run and in the subsequent cooling period. From these it is clear that in this case there is no inverse reaction. The high absorbing power of carbon for carbon monoxide as compared with hydrogen fully accounts for the slight decrease in the percentage of the former.*

On the Direct Reduction of Alumina by Carbon.

The methods used in practice for obtaining aluminium from its ores are indirect and inefficient.

The preparation involves a lengthy and complicated purification of the oxide, followed by its electrolysis in a bath of cryolite. Early work showed that where aluminium alloys are required they could be obtained by a simple method involving the reduction of alumina by carbon, but the process has never been successful for the production of the pure metal. Up to the present time opinion seems to be divided as to the effect of heating alumina and carbon together in the electric furnace.

Several authorities definitely state that alumina is irreducible by carbon,† whilst others affirm that it is quite easily reduced.‡

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* Dewar, 'Roy. Soc. Proc.,' 1904, vol. 74, pp. 122–127.

† W. Hampe, 'Chemiker-Zeitung,' 1888, 12, 391. S. A. Tucker and H. R. Moody, 'Journ. Soc. Chem. Ind.,' 1901, 20, 970.

‡ Cowles see W .P. Thompson, 'Journ. Soc. Chem. Ind.,' 1886, vol. 5, p. 206; W. Borchers, 'Elektro-Metallurgie,' 3te Aufl., 1903, p. 102.

MOISSAN,* taking an intermediate position, asserts that the two materials only react when in the form of vapour.

The question, which for many reasons is of considerable importance, has never received the detailed investigation which it deserves.

Our experiments at atmospheric pressure, as we shall see, pointed to the fact that a well-marked thermal reaction does take place, but not until the fusing point of alumina is reached.

HÉROULT,† while admitting that reduction occurs, attributes it to electrolytic action. Having carried out some experiments in which the reacting substances were heated by radiation alone and in which good yields of aluminium bronze were obtained, we contend that the assumption of electrolysis is by no means necessary.

No information was available as to the temperature of vaporisation of metallic aluminium, but various observations led us to believe that a large proportion of the reduced metal was lost by volatilisation and subsequent combustion where the furnace gases come in contact with the air.

The high-pressure furnace seemed to us therefore particularly suitable for studying this question, the advantages to be gained consisting firstly in the complete protection of the products from oxidation, and secondly in the decreased volatilisation which might be expected under the high gaseous pressures.

Some of the experiments tried under pressure to study this problem are given in Table VIII., details of one experiment being reproduced in Table IX.

By a cursory inspection of Table VIII. the two following facts may at once be deduced:—

(1) That in the resistance furnace neither aluminium nor its carbide is produced.

(2) That on the other hand all arc furnaces give a more or less marked reduction; although it will be noticed (in section B) that the product chiefly occurs as carbide of aluminium.

In several cases small malleable lumps of the metal were condensed in the powdered material surrounding the fused product.

From this it would appear that the required conditions for which we are searching had for some short period been accidentally fulfilled—these conditions being the rapid removal of the metal vapour from the reduction zone and its condensation under circumstances which precluded carburisation.

The idea that, by reducing the partial pressure of the carbon monoxide by a circulation of hydrogen or coal gas, more favourable results would be attained led to the experiments quoted in Table VIII., C and D. From these we infer that the reaction is considerably favoured by a dilution of the carbon monoxide. It is further noticeable that this precaution results in an increase in the relative quantity of

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* H. MOISSAN, 'The Electric Furnace,' London Ed., ARNOLD, p. 184.

† P. L. T. HÉROULT, 'Eng. Pat. 16853,' 1887; also 'Congrès intern. des Mines et de la Métallurgie' (Paris), 1900.

aluminium metal, although it is still accompanied by a considerable amount of the carbide.

It therefore became evident that further work at high pressures must be preceded by a more detailed study of the conditions of reduction. The several problems which arise may briefly be stated as follows :—

(1) At what temperature does alumina first show signs of reduction by carbon ?

(2) In the production of aluminium alloys, what is the function of the auxiliary metal in facilitating the reduction of alumina ?

(3) What precautions are necessary to limit the formation of carbide and increase the production of metal ?

Since it is well known that alumina cannot be reduced under ordinary circumstances in the Moissan furnace, it was thought advisable to see whether, by carrying out the reaction in an atmosphere of hydrogen, a definite indication of reduction could be obtained. The Moissan furnace was, of course, modified to exclude the use of limestone and the accompanying production of carbon monoxide.

As will be seen from Table X., A, a negative result was obtained.

As a means of limiting the temperature of reaction, calcium fluoride was introduced, but no signs of reduction were apparent at the boiling-point of the bath. From these and similar negative results at lower temperatures, which it is unnecessary to record, we assumed as a working hypothesis that the temperature of reduction of alumina is above the boiling-point of aluminium metal under atmospheric pressure.

The hypothesis we confirmed by experiments (Table X., B) in which special precautions were taken to protect the material from access of air and to provide a condensing chamber in which the vapours were cooled down before their exit from the furnace. The deposit so obtained showed unmistakeable evidence of the presence of finely divided aluminium.*

It therefore became necessary to devise some better means for indicating the production of any metal vapour.

A method which suggested itself to us, and one which has proved of considerable usefulness, was the employment of a bath of molten copper, on the surface of which the reaction mixture was placed. The copper served as an absorbent for any aluminium vapour liberated.

To determine the lowest temperature at which reduction occurs, a series of experiments was carried out. Small carbon crucibles were used to contain the mixture. These were heated either in a carbon tube furnace, or, for higher temperatures, more conveniently by embedding them in a granular carbon resistance. From the summary of these experiments in Table X., C, it will be seen that the minimum temperature of reduction coincides fairly sharply with the melting-point of alumina, and is not appreciably lowered by the introduction of either fluor spar or lime as a flux.

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* See also C. F. Mabery, 'Amer. Chem. Journ.,' 1887, vol. 9, pp. 11–15.

By referring again to Table VIII. it will be found that this view is substantiated by a comparison between the arc and resistance experiments.

In the latter the yield is always extremely low. This may be explained by the fact that as the inner layer of mixture approaches its fusing point it flows away by gravity, and, ceasing to transmit the current, is not maintained at the requisite temperature for marked reduction to occur.

We come now to that curious apparent contradiction of facts which has for so long puzzled investigators in this field; namely, that though aluminium bronze and ferro-aluminium can be so readily produced, no process exists by which the metal itself can be obtained from the oxide, except by indirect means. What is, then, the function of the auxiliary metal? It has been suggested that a marked chemical affinity exists between the aluminium and the metal with which it alloys, the evidence in support of this being the high heat evolution which is noticeable when aluminium is added to the metals in a molten state.

It must, however, be remembered that under ordinary conditions the fused metals contain dissolved oxide, and it therefore seemed worth while to carry out a preliminary investigation of this question.

Upon adding aluminium to molten copper in a thoroughly reduced condition, there is no visible evidence of a reaction, and such pyrometric measurements as were made sufficed to show that no considerable amount of heat could have been evolved.

Thus we feel justified in concluding that the copper or other metal serves chiefly to condense and dissolve the aluminium, and does not itself take part in the primary chemical reduction of the oxide.

A secondary function of the auxiliary metal is, however, possible. It occurred to us that the absence of aluminium carbide, when reduction is effected in the presence of other metals, might be explained by some chemical action of the aluminium carbide upon the copper or iron or one of their oxides.

An investigation of this matter has been undertaken by J. N. PRING,* whose results clearly show that at the temperatures we are considering, namely, at or above the melting-point of alumina, aluminium carbide reacts with either the oxide or the metal, forming an alloy.

The third problem, viz., the limitation of the formation of carbide, seems to be the most difficult to solve.

As we have seen, the metal may be considered to exist in the form of vapour at the moment of its reduction. Owing to the well-known affinity of aluminium for carbon monoxide,† it is obviously important to remove this gas as completely and rapidly as possible.

A method of reducing the partial pressure of the carbon monoxide has been dealt with above, and we have found it important to lead the gas used for dilution directly

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* J. N. PRING, 'Trans. Chem. Soc.,' 1905, vol. 87, p. 1530.

† GUNTZ and MASSON, 'Comptes Rendus,' 1897, vol. 124, p. 187.

to the seat of reaction by means of a hollow electrode, the stream of gas thus not only effectively diluting the carbon monoxide, but serving to carry forward the metallic vapour into a zone more favourable for its condensation.

Even in the absence of carbon monoxide, carburisation can occur by direct union of the metal with solid carbon.

Some unpublished work of W. H. PATTERSON carried out in this laboratory has, however, shown that in the absence of carbon monoxide this reaction only occurs above a bright red heat, thus explaining the results already quoted in which the metal was obtained, although doubtless it had not altogether escaped contact with carbon.

We are therefore in the following position: we have proved the facility with which the direct reduction of alumina by carbon can be effected, and have shown that the minimum temperature at which it can occur is already sufficiently high for the metal to be produced in the form of vapour.

Future work must be directed towards the application of high pressure for reducing the vaporisation of the metal at the temperature of reaction, the rapid removal or dilution of the carbon monoxide by a stream of inert or reducing gas, and a modification of the *régime* to facilitate the condensation and prevent the collected metal from flowing into a bed of highly heated carbon.

Thus the necessary conditions for the successful direct reduction of alumina by carbon seem to be fairly well defined, the outstanding problem being chiefly a matter of the arrangement and construction of the furnace.

The cost of most of the apparatus, which was specially constructed for the above research, was defrayed by funds awarded by the Government Grant Committee of the Royal Society. We have also been materially assisted, so far as the gas preparation plant is concerned, by Messrs. Brunner, Mond & Co., Ltd., and the Tudor Accumulator Co., Ltd.

With regard to running expenses, the work has been much facilitated by the kind way in which the ample resources of the Physical Laboratory of the Manchester University have been placed at our disposal.

In concluding, we desire to express our heart-felt gratitude to Professor ARTHUR SCHUSTER for the never-failing interest and encouragement which he has given us during the several years over which the research has extended.

TABLE I.—E.M.F. and Current of Carbon Arc under Pressure (see fig. 10).

A.

In air at atmospheric pressure.
+ve 41 millims.
−ve 27 millims.
Length of arc 8 millims.*

ampères	volts	
40	60	silent.
80	56	
118	46	
180	63	
250	66	

B.

In carbon monoxide.
Both carbons 27 millims.
Length of arc 1 to 2 millims.

(1) At 12 atmospheres.

ampères	volts
38	42
50	32
140	27

(2) At about 90 atmospheres.

ampères	volts
30	140
100	110
150	115

C.

In nitrogen.
Both carbons 27 millims.
Length of arc about 2 millims.

(1) At 13 atmospheres.

ampères	volts
50	60
55	58
60	58
65	50

(2) About 20 atmospheres.

ampères	volts	
60	120	abnormal running.
60	124	
70	120	
75	117	
80	117	
80	120	

D.

In carbon monoxide.
Both carbons 11 millims.
Length of arc 1 to 2 millims.

(1) At 11 atmospheres.

ampères	volts
12	70
13	68
15	61
25	72
27	70
38	79
48	82

(2) At 16 atmospheres.

ampères	volts
32	110
36	108
44	120
60	128

E.

In air.
+ve carbon 41 millims.
−ve carbon 27 millims.
Length of arc about 2 millims.

(1) Pressure, atmospheric.

ampères	volts	
10	57	hissing.
30	43	
50	37	
80	40	

(2) At 150 atmospheres.

ampères	volts
10	130
15	110
17	90
20	82
40	68

(3) At 190 atmospheres.

ampères	volts	
90	120	abnormal running.
95	120	
100	108	

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* In A only the length of the arc is the distance between the point of the negative of the edge of the crater of the positive. In all other experiments it is the distance of feed required to produce actual contact between the two electrodes. Solid carbons were used throughout.

TABLE II.—Oxidation of Metals in Air under High Pressures.

Experiment No.	
A 1, 3, and 4	Crucible filled with copper, iron, or aluminium, heated for half-an-hour in furnace of type fig. 11, C, under carbon arc. Pressures, about 30 atmospheres. Power, 5 to 10 kilowatts. Metals fused, but the oxidation as shown by the gas analysis was practically limited to the carbon.
A 5	Arc between iron electrodes. Pressures, 27 to 75 atmospheres. Metal at end of electrode apparently violently boiling, but after more than an hour still over 20 per cent. oxygen. Subsequently iron bar maintained at bright red heat by current of 1000 ampères for about 2 hours without entering into combustion. Oxygen at end of run over 20 per cent.
A 6	Iron electrodes, $1\frac{1}{16}$ inches diameter, in oxygen at 15 atmospheres. Points of electrodes heated by 300-ampère arc. Vivid combustion started. Current at once cut off. Positive electrode continued burning until pressure was reduced to 5 atmospheres. Total iron burnt, 550 grammes.
A 7	Rod of iron, $\frac{3}{8}$ inch diameter, $1\frac{1}{2}$ inches long, between iron electrodes in air at 100 atmospheres, maintained at bright red heat by powerful current and then fused without starting combustion. Over 20 per cent. oxygen after run.
A 13	High-tension arc (1000 volts) between thin iron rods. Pressure, 110 atmospheres. Combustion not started.
A 12	Arc between copper electrodes at 92 atmospheres. After run, 20·5 per cent. oxygen.

Table III.—Summary of the Principal Experiments on the Production of Calcium Carbide.

Experiment No.	Total energy.	Average power.	Initial pressure.	Maximum pressure.	Product, grammes, CaC_2 from analysis.	Yield, grammes per kilowatt hour.	Observations.
	kilowatt hours	kilowatts	atmospheres	atmospheres			
C 29	3·84	11·5	Atmospheric	30	283	73·7	CO from reaction retained in furnace.
C 27	6·2	12·0	Idem	50	504	81·4	Idem.
C 58	9·26	13·2	Idem	42	703	75·9	Idem.
C 61	12·6	14·5	Idem	120	1088	86·7	Idem.
C 23	15·0	15·8	Idem	15	1258	84	Pressure kept at 15 atmospheres, excess of CO being let off.
C 26	6·5	13·9	Idem	48	391	60·2	CO retained during run, but let off at once on stopping.
C 22	14·9	14·2	10 coal gas	25	1017	68·3	Circulation of coal gas by alternately admitting and blowing off gas from furnace enclosure. Total coal gas, 1420 litres.
C 65	9·75	12·7	50 hydrogen	50	405	41·5	Circulation of hydrogen through hollow carbon electrode. Total hydrogen, 1705 litres.
C 71	12·4	9·78	50 hydrogen	59	238	19·2	Idem. Total hydrogen, 2420 litres.
C 66	6·48	11·7	Atmospheric	Minimum 30 centims. Hg absolute	455	70·3	Vacuum. Maintained below ½ atmosphere during entire run.
C 67	15·8	17·2	Idem	Minimum 35 centims. Hg absolute	1385	87·7	Vacuum. Idem.
C 73	13·5	13·5	Idem	Atmospheric	1437	106·4	Atmospheric pressure.

TABLE IV. (1).—Details of one Typical Calcium Carbide Experiment (C. 61).

The large furnace enclosure was used in the vertical position (see Plate 6, fig. 1).

Iron liner filled as shown in fig. 11, A.

Lower electrode (– ve), bed of 2 kilogs. retort carbon.

Upper electrode (+ ve), a 41-millim. carbon rod, 32 centims. long.

Weight with holder, 1657 grammes. Loss of weight during run, less than 5 grammes.

Charge: about 8600 grammes of a mixture of selected Buxton lime and petroleum coke (100 CaO : 65 C). Lime all passed through a 20-mesh sieve, petroleum coke 60-mesh. The vessel with its contents was heated for some hours to a red heat before the experiment to ensure complete drying of material.

Initial pressure, atmospheric.

The gas capacity of the furnace when charged was found to be 11·6 litres per atmosphere.

All the gaseous products of reaction were retained during the experiment and for 42 hours after completion of same, with the exception of the small quantities required for gas analysis at intervals as recorded below.

The E.M.F. at the terminals of the generators was varied as required and was usually 5 to 10 volts above that on the furnace.

TABLE IV. (2).

Time.	Current.	E.M.F.	Power.	Pressure.	Observations.
minutes	ampères	volts	kilowatts	atmospheres	
start	70	27	1·89	—	
2	200	21	4·20	—	
4	300	30	9·00	—	
6	360	48	17·3	2	
7	380	42	16·0	5	
12	400	41	16·4	17	1st sample of gas taken for analysis.
15	350	48	16·8	30	
17	140	70	9·8	38	
22	240	63	15·1	46	
23	300	50	15·0	48	2nd sample of gas taken for analysis.
25	360	40	14·4	55	
27	340	47	16·0	60	
32	300	54	16·2	70	3rd sample of gas taken for analysis.
34	280	58	16·2	75	
37	300	58	17·4	84	
43	280	58	16·2	96	
44	240	67	16·1	103	4th sample of gas taken for analysis.
50	220	69	15·2	113	
52	200	58	11·6	120	5th sample of gas taken for analysis.
stop	—	—	—	—	Temperature of outside of the main enclosure at stop, 40° C.*

* At 30 minutes after stop the cover attained a maximum temperature of 65° C., falling in 2½ hours to 30°.

TABLE IV. (3).—Record of Analyses of Furnace Gas.

Sample No.	Time after stop.		Furnace pressure.	CO_2.	CO.	H.	CH_4.	N.
			atmospheres	per cent.	per cent.	per cent.	per cent.	per cent.
1	During run, see previous table		17	1·6	85·1	8·2	1·1	4·0
2			48	1·2	85·3		13·5	
3			70	0·5	86·5		13·0	
4			103	0·3	89·0		10·7	
5			120	1·1	89·6	7·4	0·9	1·0
	h.	m.						
6	0	8	92	3·4	85·3		11·3	
7	0	25	55	1·7	80·1		18·2	
8	0	39	43	2·7	77·9		19·4	
9	0	58	34	1·3	76·1	14·8	4·9	2·9
10	1	13	30·5	2·0	74·0		24·0	
11	2	43	21·5	1·5	71·1	16·7	6·2	4·5
12	3	40	18·5	1·8	69·1	19·1	7·2	2·8
13	22	0	15	1·4	66·9		31·7	
15	42	0	12	1·4	65·6	20·2	8·3	4·5

In this, as in most other cases, the furnace gas was found to contain a small percentage of iron carbonyl.

TABLE IV. (4).

The appearance of the furnace after run was similar to that shown in fig. 11, A_1.

The products from the different zones of the furnace were collected separately and submitted to analysis with the following results:—

	Gross weight.	Weight of CaC_2 from analysis.
	grammes	grammes
Fused lump.	2090	1088
Fritted mass round lump . .	1030	negligible
Fritted mass from base . . .	770	"
Unacted-on mixture	6570	"

Table V.—Summary of the Principal Experiments on the Fusion of Quartz.

Experiment No.	Type of furnace.	Total energy.	Average power.	Gas.	Working pressure.	Observations.
		kilowatt hours	kilowatts		atmospheres	
D 62	Resistance. Carbon tube, 4 centims. external diameter, length, 22 centims., between graphite terminals	8·34	10·0	Air	100	Product, a tube 23 centims. long, 4·5 centims. internal diameter, quite detached from core. External diameter about 8 centims. Weight of vitrified product, 2000 grammes.
D 68	Resistance. Carbon tube, 3 centims. external diameter, length, 21·7 centims., between graphite terminals	12·9	9·94	Air	100	Weight of vitrified product, 4100 grammes, for greater part of its length adhering to carbon core. At top blown out, forming a cup about 10 centims. internal diameter.
D 72	Resistance, as in D 68	11·3	9·14	Hydrogen	50	Weight of vitrified product, 2100 grammes, moulded around core.
D 74	Arc. 100 ampères at 120 volts, between carbon electrodes (fig. 11, D)	9·52	8·94	Hydrogen	180	Hollow sphere of vitrified material, 18 centims. external diameter, weighing 4900 grammes.

TABLE VI.—Details of One Typical Carborundum Experiment.

Large furnace enclosure used in vertical position (Plate 6, fig. 1).

Iron liner filled as shown in fig. 11, B.

Central resistance core of granular retort carbon packed in thin-walled brass tube 30 centims. long, 2·6 centims. diameter.

Electrical connection at top and bottom of core made by graphite discs 5 centims. thick and 8 centims. diameter.

Charge: mixture of 7½ kilogs. white Calais sand and 4½ kilogs. finely ground retort carbon; calcined shortly before experiment.

Iron pot lined with thin sheet of asbestos.

Initial pressure atmospheric; gaseous products of reaction retained up to about 100 atmospheres, then let off and pressure maintained constant.

E.M.F. at the terminals of the generator varied as required, and usually 2 to 3 volts above that in the furnace.

Time.	Current.	E.M.F. on furnace.	Power.	Pressure.	Observations.
minutes	ampères	volts	kilowatts	atmospheres	
start	220	4·0	0·88	Atmospheric	
1	480	7·0	3·36	—	
3	600	7·0	4·20	—	
8	600	6·0	3·60	—	
12	840	8·0	6·72	—	
13	1000	8·5	8·50	—	
14	500	12·0	6·00	—	Sudden increase of resistance due to fusion of thin brass tube.
16	550	22·0	12·1	1	
18	530	29·0	15·4	8	
19	410	38·0	15·6	13	
21	520	31·0	16·1	20	
22	520	29·5	15·3	26	
23	520	29·5	15·3	33	
25	500	30·5	15·2	43	
27	470	32·5	15·3	52	
30	370	40·5	15·0	71	
34	300	45·0	13·5	93	
37	340	41·0	13·9	108	Started letting gas off into gasometer.
38	360	41·0	14·8	—	
39	380	40·0	15·2	—	
41	410	38·0	15·6	107	56 litres had been collected in gasometer.
44	440	36·0	15·8	105	269 litres had been collected in gasometer.
46	470	35·0	16·4	101	
49	470	33·0	15·5	108	
52	470	33·0	15·5	107	
55	470	33·0	15·5	102	

TABLE VI.—Details of One Typical Carborundum Experiment (continued).

Time.	Current.	E.M.F. on furnace.	Power.	Pressure.	Observations.
minutes	ampères	volts	kilowatts	atmospheres	
58	490	32·0	15·7	106	
62	490	32·0	15·7	100	
65	490	32·0	15·7	—	
67	480	32·0	15·4	104	
70	480	32·5	15·6	—	
73	480	32·5	15·6	103	
76	480	32·5	15·6	103	Stop.

Total gas production during the run calculated to be 840 litres at 0° C. and 760 millims.

Product closely adhering to central core formed a fritted mass cylindrical in shape, about 15 centims. diameter and 27 centims. long.

Total weight, 3600 grammes. Inner layer about 2 centims. thick, consisting of crystalline carborundum surrounded by thin sheath of the amorphous variety. The outer layers on analysis found to consist of agglomerated carbon and sand, containing only small percentage of carborundum.

Granular carbon core had been graphitised, but contained no carborundum.

TABLE VII.—Record of Furnace Gas Analyses in a Carborundum Experiment.

Sample No.	Time.	Furnace pressure.	CO_2.	CO.	H.	CH_4.	N.
	minutes	atmospheres	per cent.	per cent.	per cent.	per cent.	per cent.
1	From start, 21	26	5·2	70·0	—	24·8	—
2	34	55	3·7	83·3	—	13·0	—
4	62 (stop)	122	2·9	89·0	4·2	0·7	3·2
5	After stop, 5	120	4·9	86·2	—	8·9	—
6	20	94	4·5	85·6	—	9·9	—
7	36	80	5·7	83·8	—	10·5	—
8	61	67	6·4	82·7	—	10·9	—
9	154	46	5·4	83·5	6·3	1·2	3·6

TABLE VIII.—Summary of Principal Experiments on the Reduction of Alumina by Carbon under High Pressure.

Experiment No.	Total energy.	Average power.	Initial pressure.	Maximum pressure.	Product, grammes Al metal.	Product, grammes Al_4C_3.	Observations.
	kilowatt hours	kilowatts	atmospheres	atmospheres			
A. RESISTANCE FURNACE (see fig. 11, B), CO RETAINED.							
G 19	16·0	9·6	63 air	157	Insignificant	Insignificant	Granular carbon core graphitised and containing much alumina fused around its particles.
G 41	2·5	9·0	6 nitrogen	25	Insignificant	Insignificant	Core of soot and alumina surrounded by pure alumina; to ascertain effect of fine division and intimate mixing. Tube of fused alumina around core, but no evidence of reduction.
G 44	3·2	6·5	Atmospheric	17	Insignificant	Insignificant	Experiment similar and confirmatory to G 41.
B. ARC FURNACE (see fig. 11, A), CO RETAINED.							
G 17	9·4	7·8	Atmospheric	44	5·0	47·7	Central cavity formed (see fig. 11, A_1) having, at bottom disc of fused reduction product resting on graphitised pillar of carbon.
G 32	4·6	8·2	Atmospheric	35	13·0	102	Similar to G 17.
C. RESISTANCE FURNACE, CO DILUTED AND REMOVED.							
G 18	16·0	16·0	26 hydrogen	110	3·3	20·9	No diluting gas added during run. Furnace type, fig. 11, B. Resistance core granular carbon, after run no core remaining, appearance being similar to fig. 11, B_1.
G 77	11·4	11·7	55 hydrogen	100	3·2	60·1	Current of hydrogen through hollow electrode. During run, 900 litres; during cooling, 420 litres. Furnace type, fig. 11, D, with core of alumina and carbon between the two carbons.
D. ARC FURNACE, CO DILUTED AND REMOVED.							
G 20	7·2	17·2	39 coal gas	133	46	262	No diluting gas added during run. Some lime added to mixture. Fused product contained large amount of graphite flakes. Furnace type, fig. 11, A.
G 21	8·0	16·0	15 coal gas	25	49	106	The furnace was alternatively filled with coal gas to 25 atmospheres and gas blown down to 15 atmospheres; 600 litres coal gas during run, 1200 litres during cooling period. Some lime added to mixture. Little graphite formation. Furnace type, fig. 11, A.
G 24	14·6	13·4	60 coal gas	90	31·8	198	Current of coal gas continuously passed through furnace at a rate of about 0·5 litre per second, pressure being maintained approximately constant at its maximum value. Furnace type, fig. 11, A.
G 33	9·1	9·8	15 coal gas	30	46	228	*Régime* of gas circulation similar to G 21. During run, 800 litres; during cooling about 800 litres also. Furnace type, fig. 11, A.
G 36	2·0	8·0	3 hydrogen	5	6·5	45	Reduction of alumina by calcium carbide. No diluting gas added during run. Furnace type, fig. 11, A.
G 75	15·3	9·8	50 hydrogen	70	17·7	130	Current of hydrogen through hollow carbon electrodes. During run, 900 litres; during cooling, 600 litres. Furnace type, fig. 11, D.
G 76	6·3	7·9	40 hydrogen	80	23·5	76·2	*Régime* of gas circulation similar to G 75. 510 litres during run; 450 litres during cooling. Furnace type, fig. 11, D.

N.B.—In all these experiments the mixture contained less than the theoretical amount of carbon.

TABLE IX. (1).—Typical Experiment on the Reduction of Alumina by Carbon (G 33).

The large furnace enclosure was used in the vertical position.

Iron liner filled as shown in fig. 11, A.

Lower electrode, bed of 2 kilogs. retort carbon.

Upper electrode, a 41-millim. carbon rod 32 centims. long; weight with holder, 1650 grammes.

Charge: about 5500 grammes calcined alumina (Brit. Al. Co.), mixed with 1130 grammes petroleum coke (60-mesh).

Initial pressure, 20 atmospheres coal gas.

Gaseous products of reaction washed out with coal gas (see p. 442), the gas being let off about every 10 minutes until the pressure had fallen to 15 atmospheres, a fresh supply of coal gas being at once forced in, raising the pressure to 25 or 30 atmospheres.

TABLE IX. (2).

Time.	E.M.F. on dynamo terminals.	E.M.F. on furnace.	Current.	Power.	Observations.
minutes	volts	volts	ampères	kilowatts	
start	60	42	200	8·4	Initial pressure, 20 atmospheres coal gas.
3	50	30	300	9·0	
5	47	30	340	10·2	
8	80	70	150	10·5	Gas blown off down to 15 atmospheres, coal gas admitted to 25 atmospheres.
14	80	70	150	10·5	2nd lot of gas let off as before.
16	65	57	200	11·4	
20	62	45	250	11·3	3rd lot of gas let off as before.
23	56	42	250	10·5	
26	55	42	250	10·5	4th lot of gas let off.
31	60	48	220	10·6	
34	—	—	—	—	5th lot of gas let off.
41	60	50	180	9·0	
43	—	—	—	—	6th lot of gas let off.
45	60	50	180	9·0	
56	60	50	180	9·0	
stop	—	—	—	—	

TABLE IX. (3).

The total volume of coal gas used during the experiment was 800 litres, and about the same volume during the subsequent cooling period.

Product: central fused lump surrounding the vertical electrode, to which it adhered; had a weight of about 1560 grammes, and had the appearance of fused alumina containing some aluminium carbide.

The fused lump contained 228 grammes of aluminium carbide and 36 grammes of aluminium, some of which was found in the form of plates after crushing.

The outside fritted material, about 1125 grammes in weight, contained a further 9 grammes of aluminium in large pieces. No reduced material was found in the outer layers of the charge.

Total yield was therefore 228 grammes aluminium carbide 45·7 grammes aluminium.

TABLE X.—Experiments on the Reduction of Alumina by Carbon at Atmospheric Pressure.

A. Radiation Heating from Arc.

Experiment No.	E.M.F. on furnace.	Current.	Duration.	Product.	Observations.
	volts	ampères	minutes		
G 45	40	140	5	Fused Al_2O_3. No Al metal or carbide	Mixture corresponding to $Al_2O_3 + 2C$ placed under arc on alumina bed. Furnace enclosed. Hydrogen circulation.
G 46	25	240	14	Fused lump of Al_2O_3. No Al metal or carbide	Similar experiment to G 45.
G 47	40	300	10	No aluminium or carbide	CaF_2 used as flux (28 per cent.) with same mixture as above. Heated until very rapid vaporisation of fluor spar. Furnace, ordinary Moissan type. Material in carbon crucible.
G 48	42	300	10	No aluminium or carbide	Larger percentage of fluoride (61 per cent.), otherwise similar experiment to G 47.

B. Resistance Core of Mixture.

Experiment No.		Product.	Observations.
G 61	Total energy, about 1 kilowatt hour Power, 4 kilowatts	Fused lump of alumina. No Al or carbide	Cross-section of furnace hearth, 130 sq. centims.; length, 20 centims. Charge, consisting of central core of alumina and carbon surrounded by pure alumina. Thick iron plates served to cover furnace. No aluminium condensate obtained.
G 62	Total energy, 5·8 kilowatt hours Power, 7 kilowatts	Condensate, but negligible Al in fused lump	Similar construction to above, but cover fitting air-tight, and provided with long condensing chamber. Escaping gas deposited sublimate containing finely divided aluminium.

TABLE X.—Experiments on the Reduction of Alumina by Carbon at Atmospheric Pressure (continued).

C. Approximate Estimation of Temperature of Reduction.

Experiment No.	Mixture.	Temperature estimated.	Result.	Observations.
G 55	A. 10 grammes ($Al_2O_3 + 2C$) + 40 grammes Cu	M.P. Ni . . .	No appreciable reduction	Experiment in carbon tube furnace with careful adjustment of temperature.
	B. 10 grammes ($Al_2O_3 + 2C$) + 40 grammes Cu	M.P. Pt . . .	No appreciable reduction	Experiment in carbon tube furnace with careful adjustment of temperature.
	C. 10 grammes ($Al_2O_3 + 2C$) + 40 grammes Cu	Above M.P. Pt, but below M.P. Al_2O_3	No appreciable reduction	Experiment in carbon tube furnace with careful adjustment of temperature.
G 57	A. 8 grammes ($Al_2O_3 + 2C$) + 25 grammes Cu + 10 grammes CaF_2	Above M.P. Pt, but below M.P. Al_2O_3	No appreciable reduction	Experiment in carbon tube furnace with careful adjustment of temperature (mixture well fused).
	B. 72 grammes ($Al_2O_3 + 2C$) + 25 grammes Cu + 8·5 grammes CaO	Above M.P. Pt, but below M.P. Al_2O_3	No appreciable reduction	Experiment in carbon tube furnace with careful adjustment of temperature (mixture well fused).
G 58	A. 10 grammes ($Al_2O_3 + 2C$) + 50 grammes Cu	Just above M.P. of alumina	46 grammes aluminium-bronze (6·6 per cent. Al)	Crucible containing mixture embedded in granular carbon, covered with a second crucible containing alumina. Latter showed no sign of fusion.
	B. 10 grammes ($Al_2O_3 + 2C$) + 50 grammes Cu	Considerably higher temperature	49 grammes aluminium-bronze (7·8 per cent. Al)	Similar construction alumina in upper crucible also fused. All the mixture either combined or volatilised.

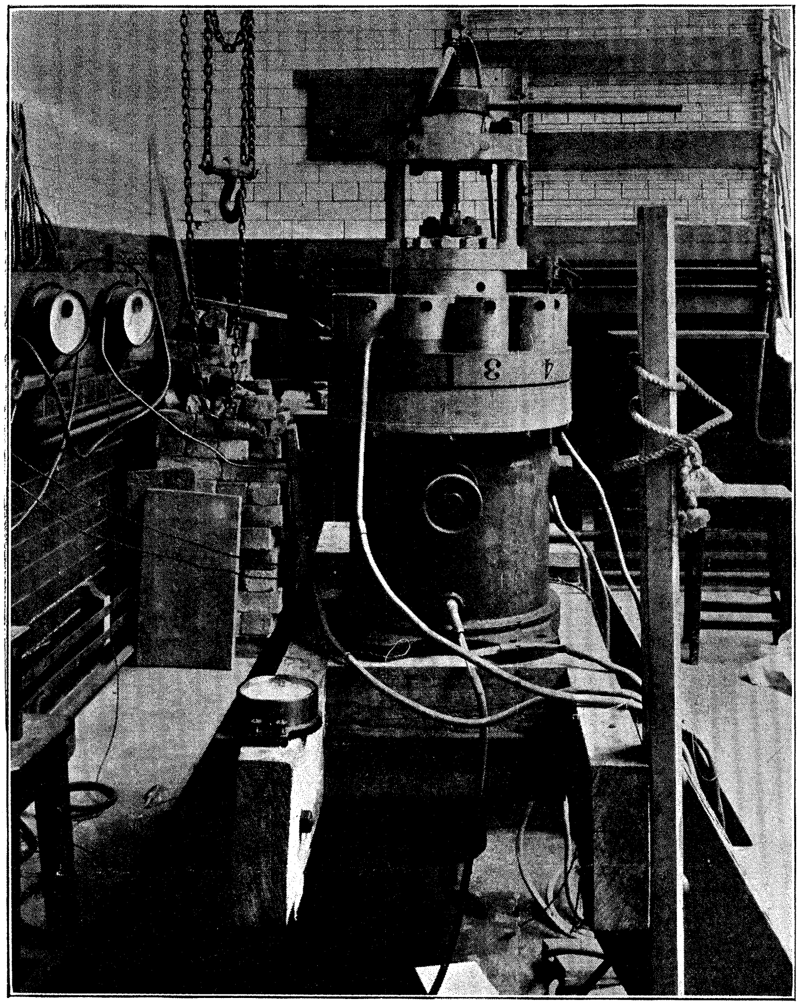

Fig. 1. Large high-pressure furnace (vertical position).

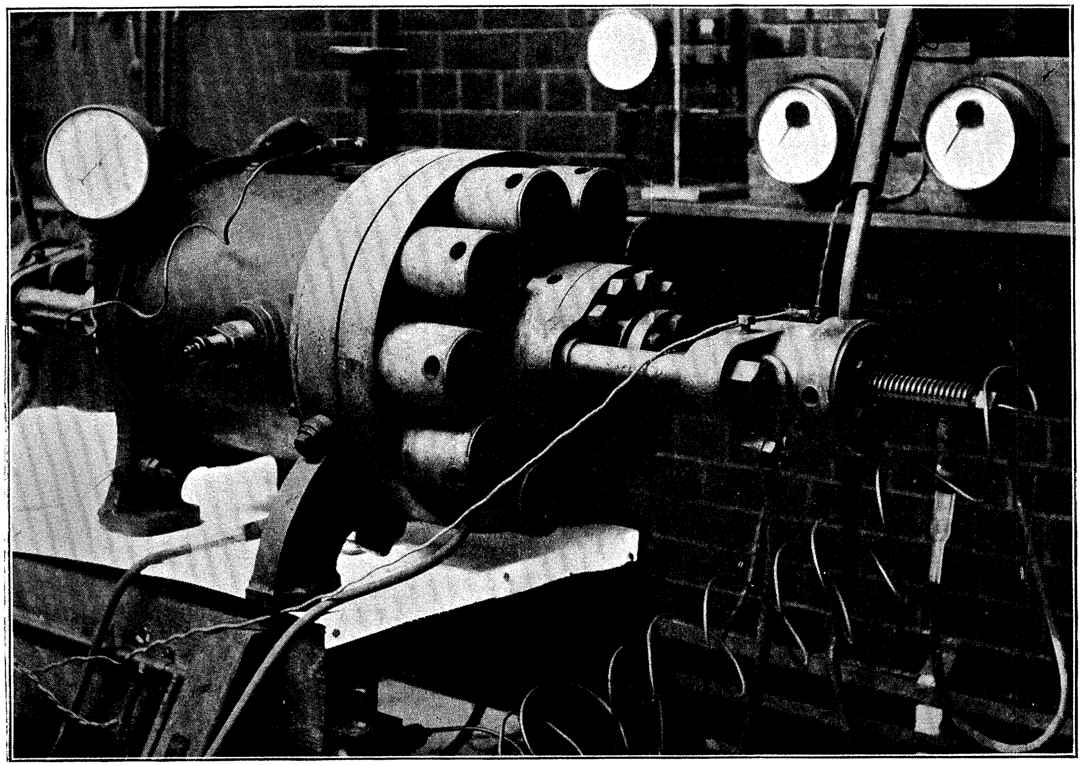

Fig. 2. Large high-pressure furnace (horizontal position).

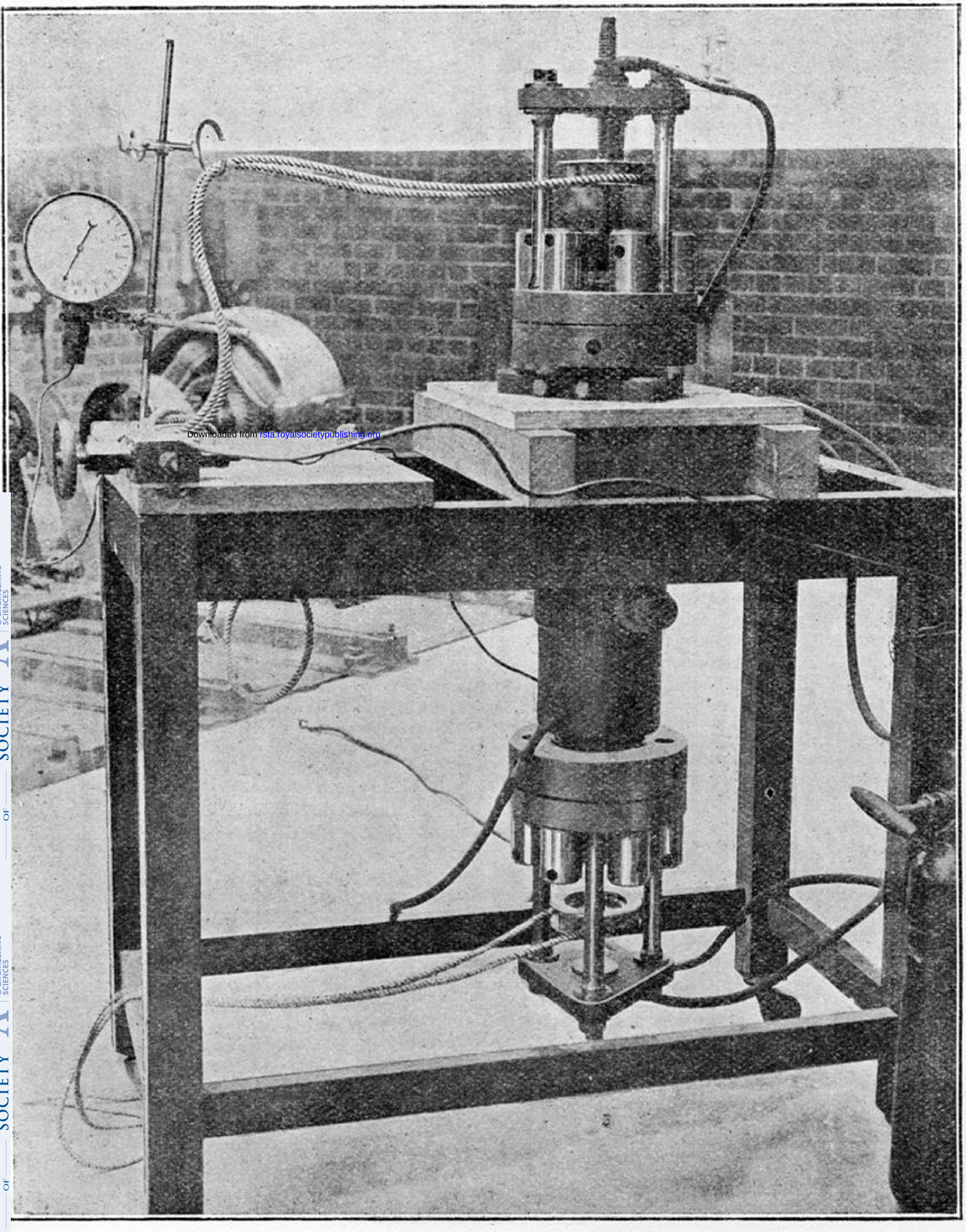

Fig. 7. Small high-pressure furnace.

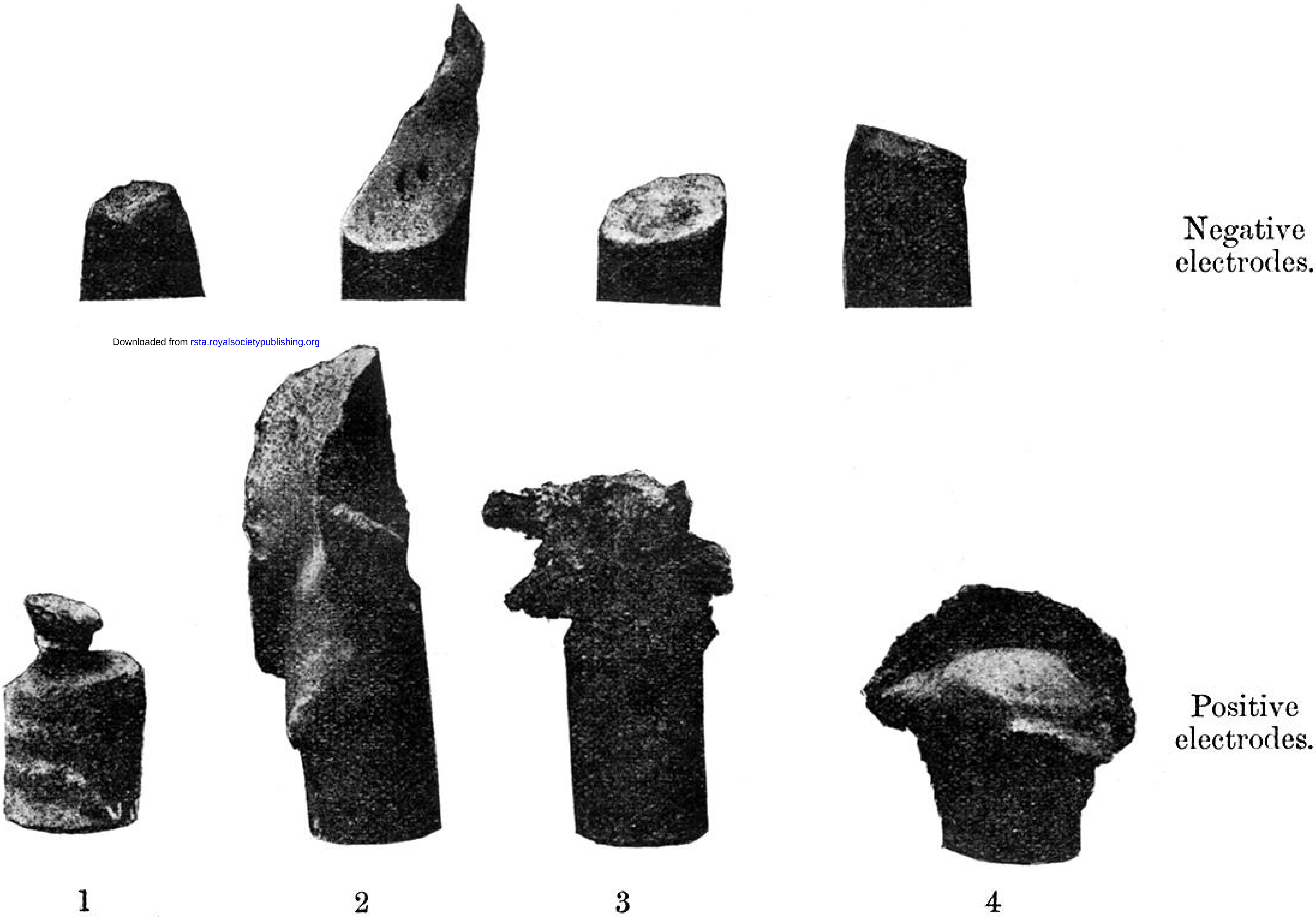

Fig. 9. Photograph of carbon electrodes after use. (Horizontal arc.)

No.	Pressure, atmospheres.	Current, ampères.	E.M.F., volts.	Gas.
1	30	60	60	Originally air, but all oxygen fixed by carbon.
2	30	400	65	Ditto.
3	50	100	100	Coal gas.
4	48	120	130	Originally air, but all oxygen fixed by carbon.

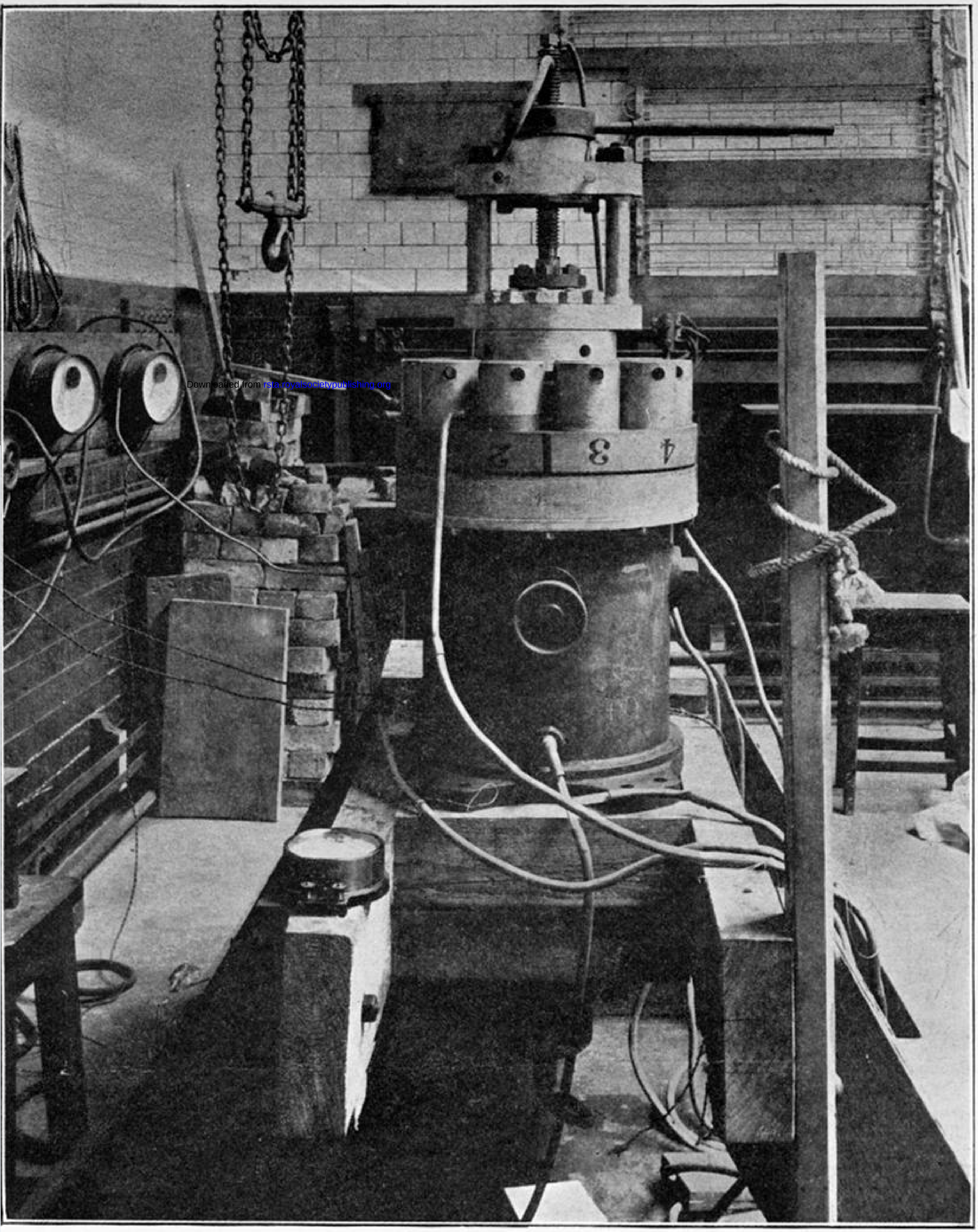

Fig. 1. Large high-pressure furnace (vertical position).

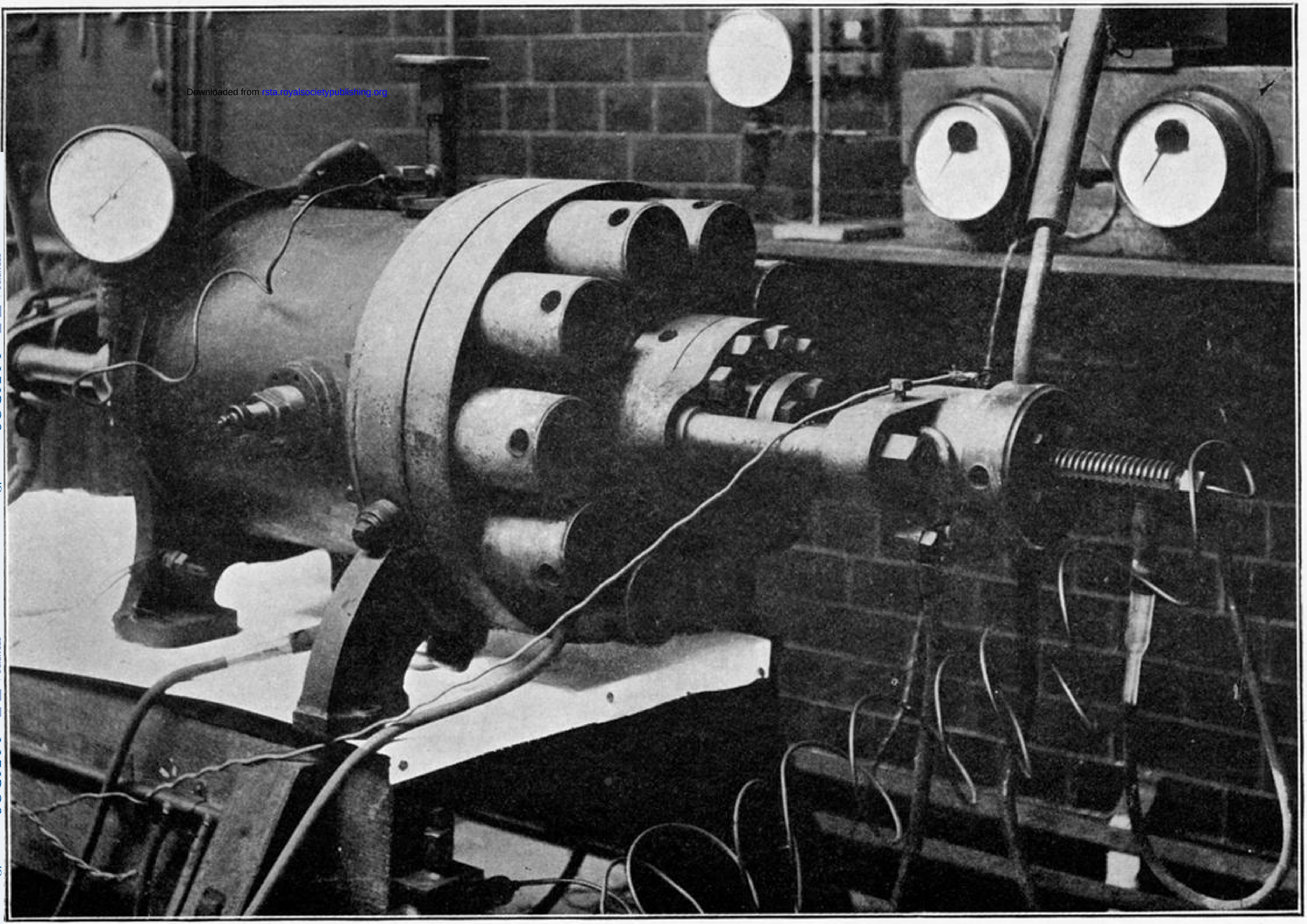

Fig. 2. Large high-pressure furnace (horizontal position).