III. Bakerian Lecture, 1918.—*Experiments on the Artificial Production of Diamond.*

By the Hon. Sir Charles Algernon Parsons, *K.C.B., F.R.S.*

Lecture delivered April 25,—MS. received July 15, 1918.

Contents.

In this paper is given an account of experiments on the artificial production of diamond which I commenced in 1887, and have carried on intermittently till the commencement of the War, when they were interrupted. Although the account is not as full as I could have wished, yet it is hoped that from the description of such experiments as relate to the salient features, followed by a summary of their bearings upon the research, and the conclusions at which we have arrived, together with an Appendix stating briefly the character of about one-third of the total number of experiments, a fair idea may be gathered of this research.

One reason for writing this paper at the present time has been a publication on the same subject by Otto Ruff in 'Zeitschrift für Anorganische Chemie,' vol. 99, pp. 73–104, May 25, 1917, who also referred to the work of Lummer on the apparently molten aspect of the surface of the carbon of the electric arc.

In my paper to the Royal Society in 1888 were described experiments where a carbon rod heated by a current of electricity (fig. 1) was immersed in liquids at pressures up to 2200 atmospheres, and where the liquids, benzene, paraffin, treacle, chloride and bisulphide of carbon, were found to yield deposits of amorphous carbon.

In my paper of 1907 allusion was made to experiments in liquids at a pressure of 4400 atmospheres, and to the distillation of carbon in carbon monoxide and dioxide at this pressure with similar results, also to an attempt to melt carbon at pressures

 [Published December 16, 1919.

up to 15,000 atmospheres, which produced soft graphite, and an experiment where a carbon crucible, containing iron previously heated and carburized in the electric furnace, was quickly transferred to a steel die, and while molten and during cooling subjected to a pressure of 11,200 atmospheres, the analyses showing less crystalline residue than if the crucible had been cooled in water.

It was also emphasized that the pressure of 11,200 atmospheres must be greater than could be produced in the interior of a spheroidal mass of cast iron when suddenly cooled, and that the inference from these experiments was that mechanical pressure is not the cause of the production of diamond in rapidly cooled iron, as had been

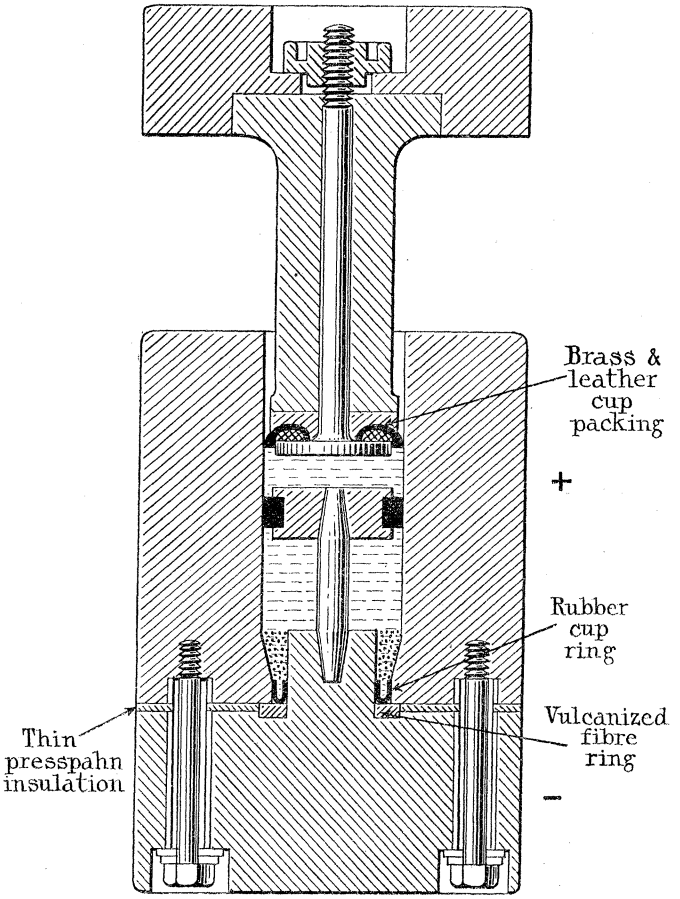

Fig. 1.

supposed by MOISSAN. This conclusion appears to us in the light of our more recent experiments to be one of great importance, and it will be further discussed in this paper.

It may be well to state that, in order to facilitate a clearer view of the bearing of each experiment on the subject, they are not placed always in chronological order. The difficulty of ensuring satisfactory experiments and the elusive character of the analyses must be the excuse for the random character of some of the former. The great majority of the experiments were failures as regards results, but a few have given information that was scarcely anticipated when they were devised.

Several thousand experiments have been made and a much greater number of analyses, generally following the methods of MOISSAN and CROOKES; the more important experiments are described at some length, and in most cases are typical of groups or repetitions of the same experiment with small variations.

The selection has been chiefly determined by their bearing on the general trend of the results of our own work and the work of others.

Those who are familiar with analyses for the detection and isolation of minute particles of diamond will know of the tendency of such particles to float, and to become lost in the frequent washings. To diminish the risk of arriving at erroneous

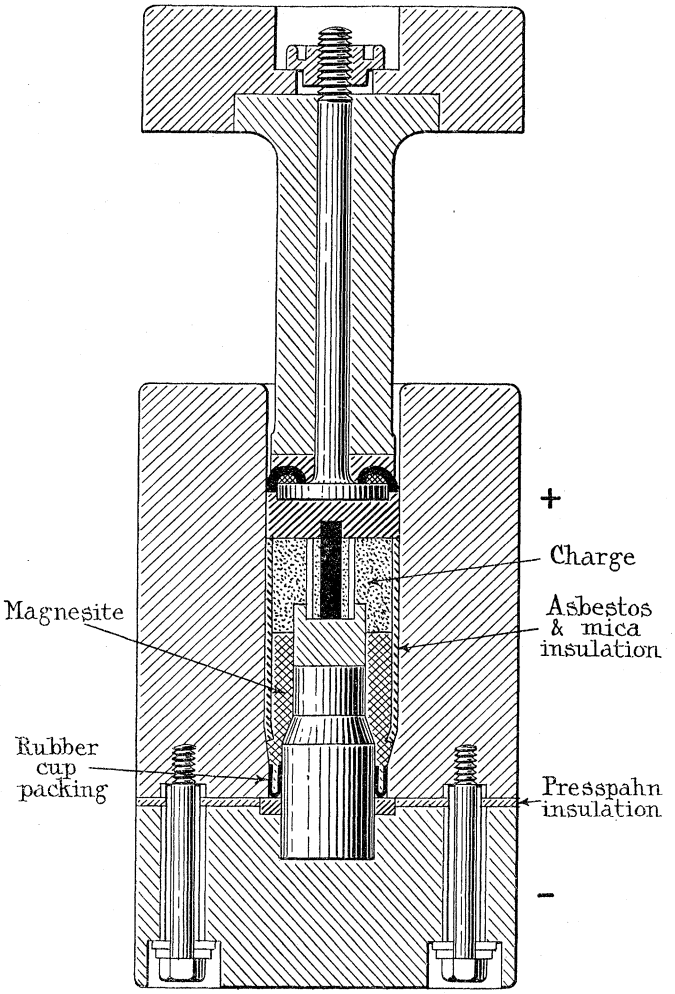

Fig. 2.

conclusions the analyses of the more important experiments have generally been repeated several times.

Experiments under High Pressure.

In the experiments designed to test chemical reactions under high pressure, where the charge was heated by passing an electric current through a central core (fig. 2) small residues of diamond occasionally occurred. A review of these

experiments, however, indicates in most cases an association with iron, whether introduced intentionally, or present from the melting of the poles, or from other causes; allusion to this is made in the Appendix.

Experiments Designed to Melt Carbon under Pressure by Resistance Heating.

In the attempts to melt carbon under pressure by this method (fig. 3) heat was applied for a duration of 5 seconds, sufficient in amount to melt the graphite core six times over, with the result of only altering the structure. RICHARD THRELFALL

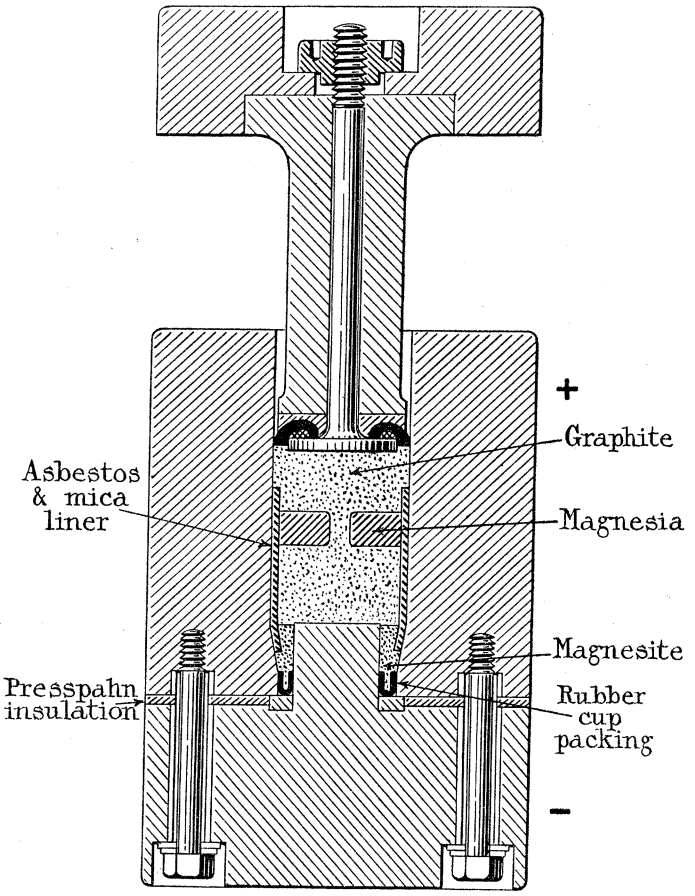

Fig. 3.

independently came to the conclusion from his experiments at about the same time, 1907, that under 100 tons per square inch, graphite, electrically heated, remained graphite.

It appeared, however, desirable further to investigate the possibility of carbon losing its electrical conductivity when approaching its melting point, as alleged by LUDWIG and others, and of thus shunting the current from itself on to the contiguous molten layers of the insulating barrier surrounding it. There had, however, been no indication of this having occurred, even momentarily; the evidence was rather that the graphite core had been vaporized and condensed in the surrounding parts of the

charge, yet it was thought well to repeat the experiment with rods of iron and tungsten imbedded in the core, so that should the temperature of volatilization of the metals under a pressure of 12,000 atmospheres exceed that necessary to liquefy carbon under the same pressure, the presence of these metals might produce a different result. No change however occurred, though in one experiment the pressure was raised to 15,000 atmospheres.

Experiments Designed to Melt Carbon under Pressure by the Rapid Compression of Flame.

A different mode of attack was then arranged, which would ensure that carbon should be subjected to an extremely high temperature concurrently with high pressure, obtained by the rapid compression of the hottest possible flame, that of acetylene and oxygen, with a slight excess of the former to provide the carbon.

The arrangement was as follows (figs. 4 and 5) :—

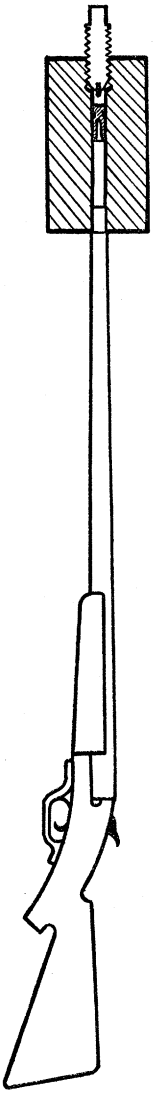

Fig. 4.

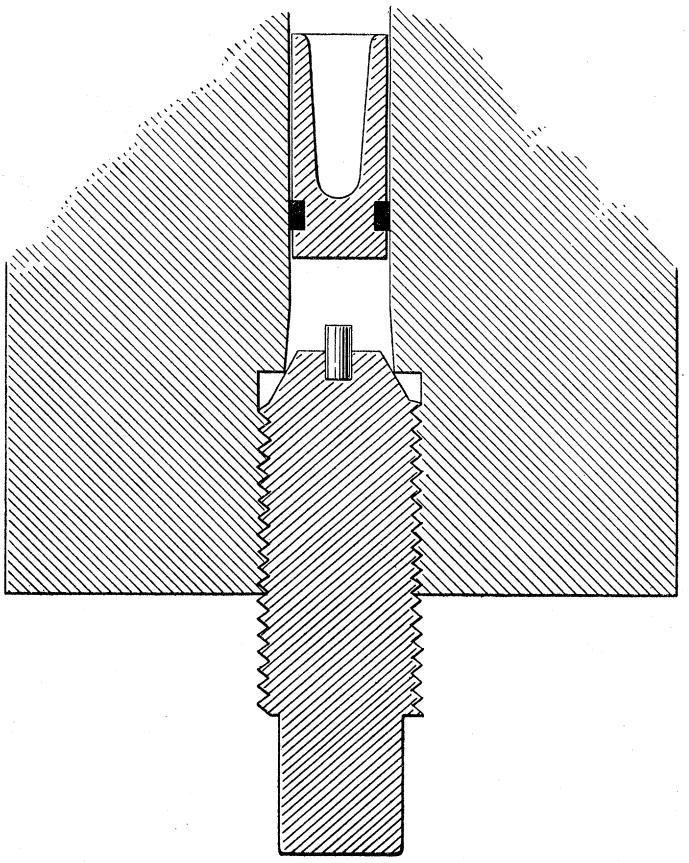

Fig. 5.

A very light piston made of tool steel was carefully fitted to the barrel of a Duck-gun of 0·9-inch bore; the piston was flat in front, lightened out behind, and fitted with a cupped copper gas check ring, the cup facing forward; the total travel of the piston was 36 inches. To the muzzle of the gun was fitted a prolongation of the barrel, formed out of a massive steel block, the joint being gas-tight. The end of the bore in the block was closed by a screwed-in plug made of tempered tool steel, also with a gas-tight collar. A small copper pin projected from the centre of the plug to give a record of the limit of travel of the piston.

The gun was loaded with 2 drachms of black sporting powder, which amount had been calculated from some preliminary trials. The barrel in front of the piston was filled with acetylene and oxygen, with a small excess of acetylene. It was estimated that this mixture would explode when the piston had travelled about half-way along the bore; when fired the piston travelled to within $\frac{1}{8}$-inch of the end, as had been estimated, giving a total compression ratio of 288 to 1.

Result.—The surfaces of the end plug, the fore end of the piston, and the circumference of the bore up to $\frac{3}{8}$-inch from the end of the plug had been fused to a depth of about 0·01-inch and were glass hard, the surface of the copper pin had been vaporized and copper sprayed over the surface of the end plug and piston.

The end plug showed signs of compression, and the bore of the block for $\frac{3}{8}$-inch from the plug was enlarged by 0·023-inch in diameter, both deformations indicating that a pressure of above 15,000 atmospheres had been reached. A little brown carbon was found in the chamber, which was easily destroyed by boiling sulphuric acid and nitre with no residue. There was a small crystalline residue from the melted layer of the end plug, from which was isolated one non-polarizing crystal, probably diamond, but too small to identify with absolute certainty.

Considering the light weight of the piston and the short duration of the exposure to heat, also the small diameter and volume of the end clearance space, the observed effects would seem to indicate that a very abnormal temperature had been reached, many times greater than exists in the chambers of large guns. There was, however, no evidence of any melting and re-crystallization of the free carbon present. In the Appendix is given a calculation from which it seems that the temperature reached was probably above 15,250° C.

Experiments with High Velocity Bullets.

As it seemed desirable to try the effect of still higher pressures, a rifle, 0·303-inch bore, was fitted with a specially strong breech mechanism by RIGBY, capable of withstanding a charge of cordite 90 per cent. in excess of the service charge.

The gun (fig. 6) was fixed in a vertical position on the wall of the armoured press house, with its muzzle 6 inches from a block of steel, in which a hole

0·303-inch diameter had been drilled to a depth somewhat greater than the length of the bullet, and in alignment with the bore of the gun; the trigger was pulled by a string from without. Cylindrical bullets of steel with a copper driving band were used, shorter than the service bullet, and about one-half of the weight, some with cupped noses to entrain material, some with coned noses to match the bottom of the hole in the block. The velocity with 90 per cent. excess charge was estimated to be about 5000 ft./secs.

The substance to be compressed was placed either at the bottom of the hole when the coned-nose bullet was used, or over the mouth of the hole when the cupped-nose

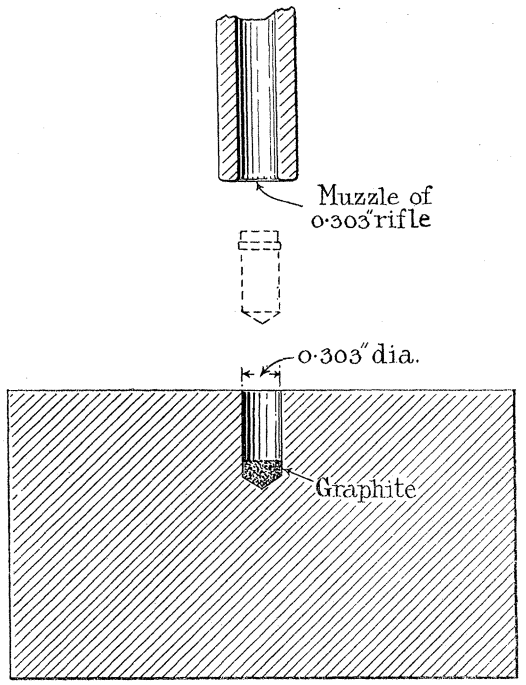

Fig. 6.

bullets were used. Some of the bullets were of mild steel, but those with cupped noses were of tool steel.

The substances placed in the hole are given in the Appendix, and included graphite, sugar carbon, bisulphide of carbon, oils, &c., graphite and sodium nitrate, graphite and fulminate of mercury, finely divided iron and fine carborundum, olivine and graphite. After each shot (fig. 7) the bullet and surrounding steel were drilled out, and the chips and entrained matter analysed.

Several experiments were also made with a bridge of arc-light carbon just over the hole, raised to the limit of incandescence by an electric current, and the shot fired through into the hole at the moment the carbon commenced to vaporize, as observed in a mirror from without. Also an arc between two carbons was arranged just over

the hole (fig. 8) and the shot fired through it, as also through a crucible of carbon with a very thin bottom containing a little molten highly carburized iron.

Of all these experiments the only ones that yielded a reasonable amount of residue were one made with graphite wrapped in tissue paper, the bullet, however, in this case having grazed the side of the hole, and thus producing some molten iron by the friction, as also the shots through the incandescent bridge, where again some molten metal would probably occur. The residues were in all cases exceedingly small and not more than would be produced from a small amount of iron melted, carburized and quickly cooled. There was no evidence of any incipient transformation of carbon in bulk into diamond that could be detected by analysis.

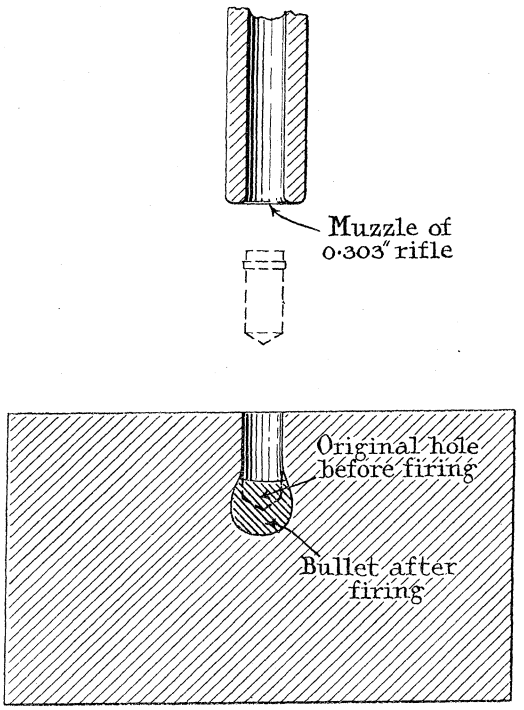

Fig. 7.

A bullet was also fired into a long hole, 0·303-inch in diameter, bored in a steel block and filled with acetylene gas, retained by gold-beater's skin over the mouth, thus repeating the flame experiment (but in this case without oxygen) on a small scale with the intensest pressures available. The residue was nil.

The pressure on impact of a steel bullet fired into a hole in a steel block which it fits is limited by the coefficient of compressibility of the steel, and with a velocity of 5000 ft./secs. is about 2000 tons per sq. inch. Measurements made from a section through the block and bullet (fig. 7) showed that the mean retarding force on the frontal face, after impact till it had come to rest, was about 600 tons per sq. inch.

Several experiments were made by substituting a tungsten-steel block, and a hole tapering gently from 0·303-inch at the mouth to 0·125-inch at the bottom, and using a mild steel bullet, which on entry would be deformed and a greatly increased velocity imparted to the nose. Progressively increased charges were used, and even with relatively small charges the block cracked on the second round. With the 90 per cent. excess charge, the block always split on the first shot, but this probably occurred after impact, and not till the full instantaneous pressure had been exerted, which was estimated to be greater than with the plain hole, probably over 5000 tons.

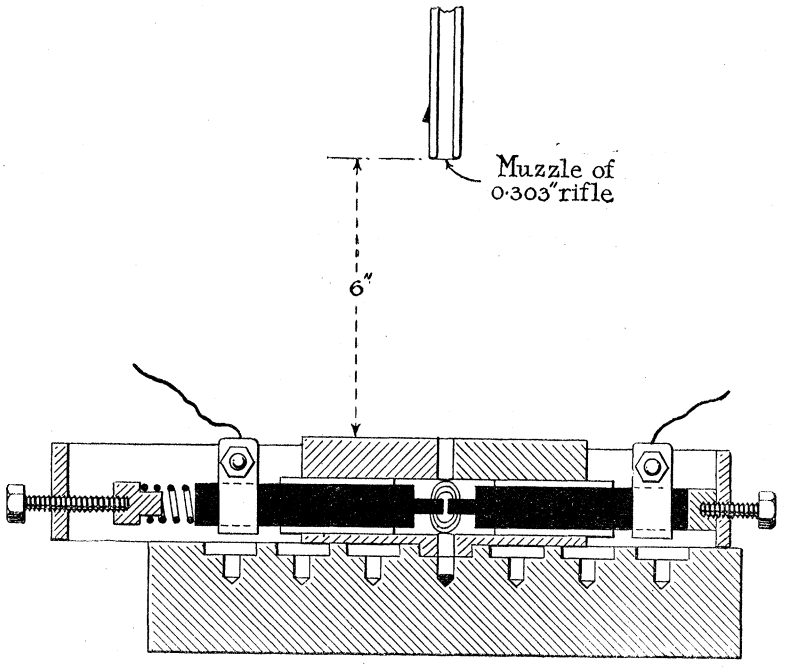

Fig. 8.

Only graphite was placed at the bottom of the hole in these latter experiments, and the analysis yielded nothing.

Experiments on Pressure in Cast Iron when Cooled.

It has been generally assumed that iron rich in carbon expands on setting, and that this supposed property is a contributory cause in the formation of diamond.

Several experiments were made by pouring iron saturated with carbon from the electric furnace through a narrow git into a very massive steel mould, closed at the bottom with a breech screw (fig. 9). When cold, the breech screw was easily removed, and there was no sign of any appreciable pressure having come on the threads. Not being sure that, because of capillarity, the corners of the mould had been quite filled, a steel mandril was, immediately after pouring, forced down the git-hole by a press giving a fluid pressure in the mould of 75 atmospheres. The observed pressure

on the breech screw appeared not to have exceeded this pressure. Highly carburized iron, therefore, does not expand with any considerable force on setting.

The reason why a lump of cast iron thrown into a ladle of molten metal first sinks to the bottom and soon rises and floats on the surface is probably that cast iron is about seven times stronger in compression than in tension. Therefore when a sufficiently thick layer of the cold metal has been heated the interior is torn asunder by the expansion of the outer skin, and the specific gravity of the whole mass is diminished. (See Mr. WRIGHTSON's paper "On Iron and Steel at High Temperatures," with discussion, 'Journal of the Iron and Steel Institute,' No. 1 for 1880.)

We may therefore safely conclude that when iron is suddenly cooled, the only compressive bulk pressure that is brought to bear on the interior is that arising from

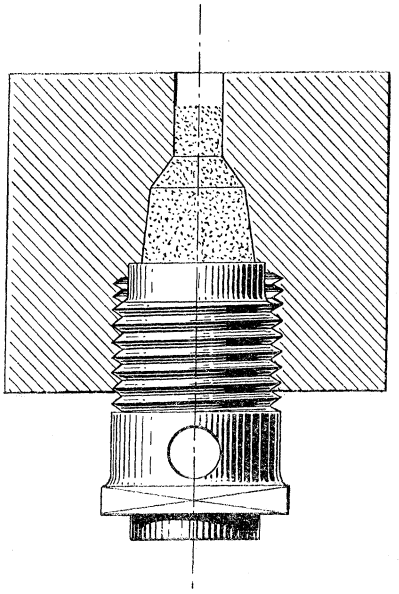

Fig. 9.

the contraction of the outer layers after setting, and with highly carburized iron this can only be small because of the low tensile strength of the metal.

Gases Ejected from Cast Iron on Setting.

As bearing upon the question of the possibility of the occluded gases playing a part, MOISSAN was the first to observe that spherules or small spheres of iron with cracks and geodes never contained diamond. We have made experiments by pouring highly carburized iron, alloys and mixtures on to iron plates, the cooling taking place from one side only, and under such conditions no diamond results; in fact it only occurs when the ingot or spherule is cooled on all sides nearly simultaneously, so that an envelope of cold metal is formed all over before the centre sets.

Since my paper in 1907, the experiment of heating iron in a carbon crucible and transferring it to a steel die and subjecting it to 11,200 atmospheres pressure has been repeated, and it has been found that if the iron is allowed to set before the pressure is applied the amount of diamond is much greater than if pressed when very hot and molten, and that it is then about the same as when the crucible is cooled in water. The only reason that suggests itself to account for this is, that when pressure is applied while the iron is very hot some of the latter permeates the carbon of the crucible, and because of the greater specific heat and lesser conductibility of the carbon, the iron next to and in the carbon remains molten after the ingot has been cooled by direct contact with the steel cup on the face of the plunger. Thus, when cooling, the occluded gases have a free exit from the ingot, through the molten metal (which is pervious to gas) into the carbon of the crucible, and are not retained in the ingot to the same extent as when it is set and enclosed in an envelope of colder iron impermeable to the gases before pressing.

The experiments of BARADUC MULLER ('Iron and Steel Institute, Carnegie Scholarship Memoirs,' 1914, p. 216), on the extraction of gases from molten steel, showed that steel is permeable to gases down to 600° C.

Other Experiments.

The action of water on carbide of calcium, and of concentrated sulphuric acid on sugar for 6 hours under pressure of 30,000 atmospheres were tried; in both cases amorphous carbon was formed and no diamond.

HANNAY'S experiments were repeated, where paraffin and dipple-oil with the alkali metals, especially potassium, were sealed in steel tubes and subjected to a red heat for several hours. The analysis gave no diamonds; in fact it became apparent that when hydrocarbons or water were relied on to produce pressure, the latter could only exist for a short time at the commencement, for when a red heat was reached the hydrogen escaped through the metal, and the oxygen combined with the steel.

We did not analyse the steel tubes themselves. Many experiments were however tried with central heating under the press at 6000 atmospheres, and nothing was obtained of interest with the substances used by HANNAY, unless, as previously mentioned, some iron was present. FRIEDLANDER'S experiment was repeated, where a molten globule of olivine, in a reducing flame, or with carbon added, was stated by him to contain minute diamonds. An experiment was made with molten olivine in a carbon crucible in a wind furnace stirred with a carbon rod, with and without an electric current passing between the rod and crucible.

Many experiments were also tried at 6000 atmospheres under the press with central heating with olivine associated with carbon, hydrocarbons, bisulphide of carbon, water, &c., also with blue ground from Kimberley instead of olivine. The results of the analyses were in all cases negative, except occasionally when metallic iron was present. Thus in some cases the olivine or blue ground was partially

smelted by the heating carbon rod or by the associated hydrocarbons, &c., when such were added, and iron globules were formed. In these, diamond was occasionally found when cooling was rapid and they were centrally situated in the charge.

Very Quick Cooling.—To test the action of very quick cooling a carbon crucible of 2-inch internal diameter charged with iron, sugar carbon, 2 per cent. silicide of carbon, well boiled by resistance heating under atmospheric pressure and 2 per cent. of iron sulphide added, was quickly placed on asbestos mill-board resting on a steel table frictionally held in the bore of the 4-inch mould, below being placed 2 lbs. of carbon dioxide snow, and the plunger quickly brought down by the press, subjecting the whole to 6000 atmospheres pressure. When taken out the crucible was intact, the contents had divided into a lower portion consisting of a large grained crumbling mass of graphite admixed with granules of very hard iron, in the centre a rounded pillar of white iron equally hard. The cooling seemed to have been unusually rapid.

The experiment was repeated, the crucible being charged with iron, sugar carbon, 5 per cent. manganese, 5 per cent. cobalt, 2 per cent. silicide of carbon, boiled, and 2 per cent. iron sulphide added.

It was also repeated with water instead of carbon dioxide snow. The result of all these experiments was similar to the first. No diamond was found in any part.

An experiment which seemed to give practically instantaneous cooling was as follows:—A small carbon crucible containing iron, with traces of silicon, aluminium, calcium, magnesia and sulphur, was floated on a carbon block on a bath of mercury, all contained in a vessel exhausted to 2 mm. absolute. The crucible was heated by an arc from an upper carbon, the holder passing through a stuffing box. When the crucible was sufficiently hot and the contents carburized, the upper carbon was thrust down, submerging the crucible under the mercury; the cooling was almost explosive and instantaneous—the finely divided iron and graphite on analysis yielded no diamond.

Extremely rapid cooling does not, therefore, seem to be a direct cause in the production of diamond.

Experiments at Atmospheric Pressure.

A convenient method of studying the effect of the association of other elements with iron on a small scale uncontaminated by the vapours of a furnace lining suggested itself, and a series of experiments were made as follows:—A deep iron dish was packed tightly with Acheson graphite with a slight dimple in the centre to hold the ingot, above, graphite was filled in loosely to a depth of half an inch covering the ingot. An arc was struck by a carbon on to the ingot submerged in the loose graphite. When the iron was well boiled the surrounding graphite with the ingot in it was dug out entire and thrown into a bowl of mercury covered with water.

The results showed that, using ordinary mild steel, no diamond ever occurred on analysis, but that a small percentage of silicon is absolutely essential; small

percentages of aluminium, magnesium, calcium, one or all are important; sulphur, manganese, and cobalt increase the yield, nickel appeared to be a disadvantage. An alloy of iron and 10 per cent. manganese, 10 per cent. cobalt, and 5 per cent. silicon gave out much gas when cooled slowly, and on quick cooling in water and mercury most of the spherules were burst and shredded.

Finally about 1 to 3 per cent. of the other elements added to iron appeared to give the best results and the spherules were not then burst.

An experiment was made by letting the ingot remain in the bed till it had quite set, hard enough to handle with the iron spoon, and then, cooled in water and mercury, it gave a fair diamond residue.

Experiments on the Conversion of Diamond to Graphite.

A clear octahedral diamond was placed in a small carbon crucible and packed loosely with Acheson graphite and heated for 10 minutes to about 1400° C. The diamond was coated with a firm layer of graphite.

After two prolonged treatments with fuming nitric acid and potassium chlorate, alternating with boiling sulphuric acid and nitre, the opaque coating was removed and there remained a blackish translucent skin. When fractured the interior was unaltered and perfectly transparent.

A piece of bort somewhat laminated, after the same treatment, showed the laminations separated by cracks starting from the outside. Upon breaking, the interior surface of the fissures showed an incipient change to graphite, but less rapid than on the outside surface. There was a sinuous pitting, deepest near the outside and diminishing inwards. The substance of the bort between the fissures was unaltered.

The change of diamond to graphite under the conditions described is gradual, the surrounding gases, carbon monoxide, carbon dioxide, nitrogen, hydrogen, and also vapour of iron (as an impurity in the graphite) singly, or collectively, probably play a part, and further investigation as to this seems to be desirable.

Sir JAMES DEWAR, in 1880, heated a diamond in a carbon tube to a temperature of 2000° C., while a flow of pure hydrogen was maintained through the tube. The diamond soon became covered with a coating of graphite ('Proceedings of the Royal Institution').

A clear diamond plunged into molten iron saturated with carbon at about 1400° C. for 5 minutes was deeply pitted. When removed from the iron small globules of iron adhered to the surface and the pits appeared to occur at these spots.

A clear diamond was disintegrated by cathode rays, the temperature by pyrometer being 1890° C., the splinters were quite black and opaque, but after several prolonged treatments with fuming nitric acid and potassium chlorate, alternating with boiling sulphuric acid and nitre, the coating that remained was a dusky grey, but

semi-transparent, the gas present being chiefly hydrogen. (Paper by PARSONS and SWINTON, January 16, 1908, 'Roy. Soc. Proc.,' A, vol. 80.)

In this latter experiment the surface action appeared to be much less in proportion to the incipient change of the under layer to graphite, and the impression is that at 1890° C. the temperature of bulk transformation is being approached, also that carbon monoxide, carbon dioxide, nitrogen, hydrogen, and iron, one or more, act as catalysts in the change of diamond to graphite.

Experiments on the Oxidation of Alloys of Iron when Molten.

Iron was melted in a carbon crucible and highly carburized; when it had somewhat cooled, the other elements were added, in small percentages of aluminium, silicon, calcium, magnesium, manganese, iron sulphide, collectively and in some cases singly; the crucible was then removed from the furnace and superheated steam blown through a carbon tube into the metal; energetic action took place and much heat was evolved; on analysis, after destroying the graphite, a bulky transparent crystalline residue remained.

With aluminium alone the crystals were chiefly crystallized alumina, and with the other elements the spinels and other crystals were produced; all were transparent and colourless, but when chromium was added some rounded crystals occurred resembling pyrope. When submitted to sulphur dioxide and carbon dioxide the result was the same, but less residue was produced. Under the microscope there appeared to be a small proportion of very small crystals like diamond; these burnt in oxygen. When the bulky residue was placed in a test-tube with the double nitrate of silver and thallium, and the density adjusted so that a diamond floated midway between the top and bottom, there collected into its immediate neighbourhood after a time an amount of the small crystals which was estimated to be about 5 per cent. of the total residue.

One prolonged treatment of hydrofluoric acid had no apparent effect on the bulky residue, and it required so many treatments to destroy it that we failed to isolate the very small particles whose size did not exceed $\frac{1}{20}$ mm.; they were probably lost by flotation. These experiments were repeated many times with the same result, but they merit further investigation, with steam under high pressure and conditions favourable to the formation of larger crystals.

Note.—MARSDEN observed in silver the association of black diamond with crystalline alumina, silicide of carbon, &c., 'Roy. Soc. Proc.,' 1880.

Experiments in Vacuo.

The presence of diamond in some meteorites suggested a series of experiments under various degrees of vacuum up to the highest obtainable.*

* Also an impression suggested itself in 1907 that hydrogen had an adverse effect on the formation of diamond.

It is probable that some meteoric matter may have been melted by collision or ejected into space in a molten state and cooled by radiation, and that under such conditions the absence, or diminution, of occluded gases might be a factor conducive to the crystallization of carbon.

One of the 4-inch diameter pressure moulds (fig. 10) was used in a preliminary experiment as the container. The crucible was turned out of a $1\frac{1}{2}$-inch carbon rod, and so formed on a stem that the electric current heated the bottom and sides equally. The cover was similarly formed and its holder was electrically connected with the container, but free to move vertically and to rest its weight on the crucible,

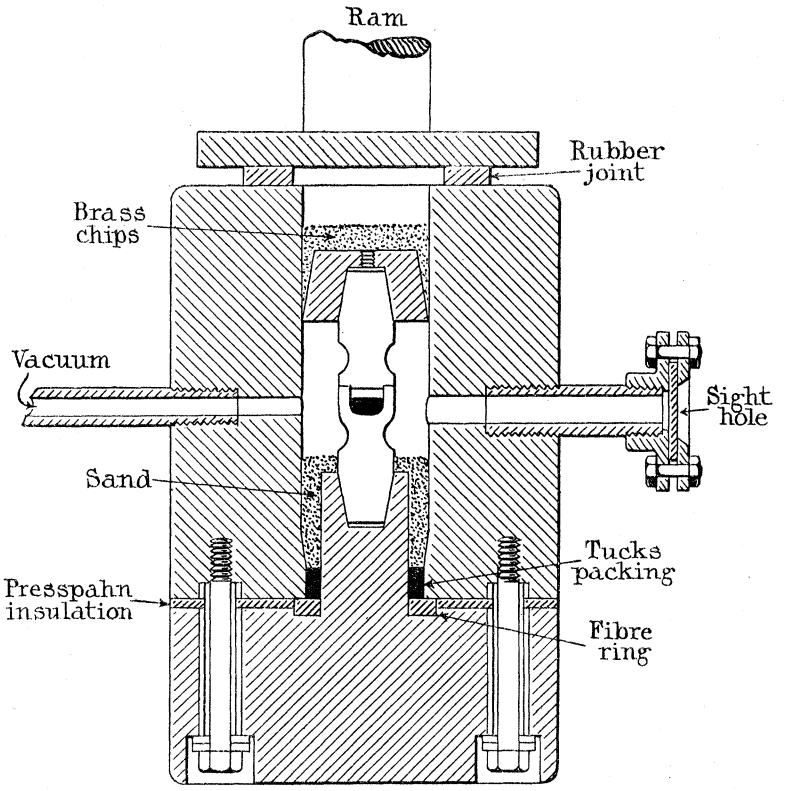

Fig. 10.

electrical connection to the container being made by a layer of brass or iron turnings resting on the holder. A current of 1000 amperes at 16 volts sufficed, and the temperature was observed through a glass window at the side of the container.

The crucible was charged with reduced iron and lampblack. The Geryk pump evacuated the container to $\frac{3}{8}$-inch mercury absolute; current was turned on for 15 seconds, the vacuum fell to 3 inches, when it had risen again to $\frac{3}{8}$-inch current again turned on. This was repeated three or four times, finally current was applied for 30 seconds and the vacuum again fell to 3 inches. The gas was drawn off and collected, it amounted to a total of $\frac{1}{2}$ gallon at atmospheric pressure and

consisted of 95 per cent. carbon monoxide, 1 per cent. hydrogen, 2 per cent. hydrocarbon, 2 per cent. nitrogen.

The carbon which formed the crucible and cover contained a large percentage of silica, but the carbon monoxide was produced chiefly by the action of sand (of which there was a thick layer on the bottom of the container to protect the insulating joint from iron spilled from the crucible) on the carbon of the stem of the crucible. About one half of the iron had been evaporated, and there remained an ingot about the size and shape of a broad bean. It contained rather large graphite crystals and was easily broken. The analysis gave the largest residue of diamond in proportion to the amount of iron of any of our experiments, the largest crystals being 0·7 mm. in length.

This experiment was repeated several times with the same result. The time of cooling of the crucible, from switching off the current to the temperature of setting, was 15 seconds, and probably sufficiently rapid to allow of a skin to be formed around the ingot before the centre was solidified, for the configuration of the crucible and cover were such as to ensure nearly equal and simultaneous cooling on all sides of the ingot. At the time, vacuum was erroneously thought to be the chief contributory cause and not the presence of carbon monoxide in large proportion.

High Vacuum Experiments.

The molecular pump not having yet been evolved, a powerful pumping system was arranged, consisting of three steam-jet exhausters in series, the last ejector of the series discharging into a jet condenser with separate air and water pumps, the former assisted by a steam jet. The two steam-jet exhausters nearest to the exhausted chamber were fed with highly superheated steam at 200 lbs. pressure, and the suction pipe to the chamber was 4 inches in diameter—the chamber 2 feet 6 inches diameter—of spherical shape (fig. 11). A vacuum of $\frac{1}{6}$ mm. absolute could be reached.

The crucible was placed on a large block of carbon, resting on the base of the chamber, and forming the bottom pole. The cover was insulated from the chamber, and through an oil-sealed gland passed a 2-inch brass rod, carrying a crown holder, with four 2-inch carbons which rested on the lip of the crucible for resistance heating. An observation window was placed at the apex of a long iron cone, projecting from the side of the cover, which gave a good view of the crucible and its contents. The whole of the chamber was submerged in a tank of water, up to the level of the gland in the cover.

Iron and iron alloys were boiled and allowed to cool slowly by radiation, or were rapidly quenched by admitting water through a large valve from the tank into the vacuum vessel. The iron and carbon vapour from the boilings deposited dust and globules on the cover and sides and bottom of the chamber. A very small diamond

residue generally resulted from the small iron globules, and also from the dust, but never anything from the ingot remaining in the crucible.

In several experiments water was admitted, which played directly on the crucible, the upper carbons resting on the rim prevented its upsetting by the force of the water, and still there was no residue. In one experiment the carbons were lifted and the charge flowed out, forming spherules of varying size in the water. There was a very small diamond residue from these spherules.

In one experiment a crucible was filled with iron and carbon and closed by a tight carbon cover, a hole bored in the side of the crucible, a massive block of iron placed

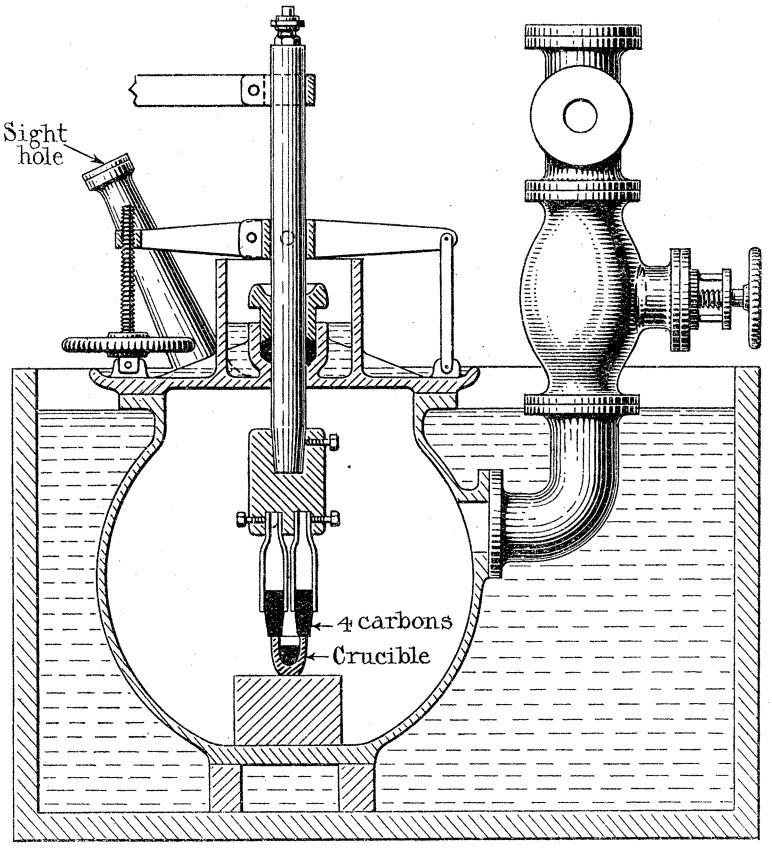

Fig. 11.

close opposite the hole and the crucible boiled, the vacuum being under 1 mm. No crystallised residue was found in the deposit on the iron block from this high velocity jet of vapour of iron and carbon.

In another experiment a powerful electro-magnet was provided with poles to give a concentrated field, and an arc struck between two carbons, arranged to burn within this field and regulated from without by hand. There was an iron block upon which the arc directed by the field could play and condense its carbon vapour. The analysis gave no diamond.

It was thought that the vapour from boiling iron saturated with carbon might, by the action of bisulphide of carbon, cause a crystalline deposit, but all the experiments to this end yielded no results.

Experiments under X-ray Vacuum.

Experiments were made under X-ray vacuum in a new chamber of cast iron with very thick walls to absorb the heat, exhausted through an 8-inch diameter suction by a large molecular pump alongside, in series with a dry, high speed, two stage, pump, 12-inch diameter pistons, and last of the series a 3-inch+2-inch compound

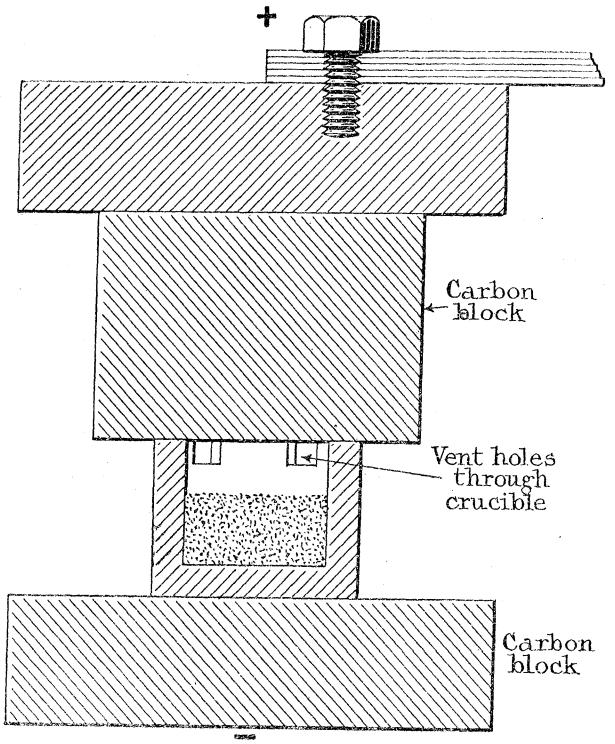

Fig. 12.

Fleuss. The crucible was resistance-heated as before (fig. 12). No diamond was produced in any of these experiments, except in those where iron, sand, and other elements, with or without sulphur, were first heated and well boiled in the carbon crucible at atmospheric pressure, and after cooling transferred to the vacuum furnace and re-heated by resistance under X-ray vacuum; violent ebullition occurred owing to the liberation of occluded gases, and many iron spherules were ejected, which cooled by radiation and conduction where they fell; diamond was found in these, which burnt in oxygen, but no diamond was ever found in the ingot remaining in the crucible.

It occurred to us to try the effect of great mechanical pressure accompanied by heat upon small particles and powders, the interstices being exhausted to a high vacuum.

Several experiments were made in the press under a mass pressure of 3000 atmospheres.

A layer of cast-iron turnings resting on a layer of carborundum grit, the exhaustion being effected through a hole in the side of the mould covered by a perforated steel plate within the layer of grit, heat was applied as usual by a central carbon rod.

Analysis yielded some thin crystal plates from the grit which had lain in the line between the cast iron and the suction outlet at the grid, and also from the layer of grit which had lain against the cast-iron turnings which had become heated but not melted by the central carbon rod.

To ascertain the cause of the occurrence of these plates, experiments were made without bulk pressure on the concentrated action of the gases given off from cast-iron turnings heated up to a good red, and drawn by a high-vacuum pump through carborundum grit placed in a silica tube heated by a gas burner at the centre of its length to dull red. These yielded similar crystal plates.

Control experiments showed that no similar plates existed in the untreated grit.

It was also found that the cast-iron turnings would not produce this effect on a second heating unless they had been subjected to CO at atmospheric pressure for some hours. Carbon monoxide, sulphur dioxide, cyanogen, hydrogen, nitrogen, oxygen, nitric acid gas, chlorine, ammonia, ammonium oxalate vapour, ammonium chloride, acetylene, coal gas, produced no plates.

These plates resemble diamond very closely in appearance and form of crystallization, they do not polarize, and some have triangular markings; they will not, however, burn in oxygen at 900° C., and are completely destroyed by chlorine purified from oxygen and water vapour at 1100° C.; their specific gravity is about 3·2, they are therefore not diamond.

Note.—Recent experiments have shown that carbon monoxide passed over molten iron sulphide and then over carborundum grit below red heat at atmospheric pressure also produces these plates, and that if coal gas is substituted for carbon monoxide no plates are formed. Also that only a few of the grains produce plates.

The composition of the grains is—

Carborundum	36·56
Iron oxide and alumina	44·09
Lime	10·45
Magnesia	5·57

Summary of Experiments and Conclusions.

The experiments have shown that all the hydrocarbons, chlorides of carbon, and oxides of carbon tested, deposit amorphous carbon or graphite on a carbon rod

electrically heated at any pressure up to 4400 atmospheres, and in a few experiments up to 6000 atmospheres.

That at 15,000 atmospheres carbon and graphite electrically heated are either directly transformed into soft graphite or are first vaporized and then condensed as such.

While the experiment of rapidly compressing a mixture of acetylene and oxygen and the production of temperatures much in excess of that necessary to vaporize carbon, accompanied by a momentary pressure of about 15,000 atmospheres, confirms the conclusion that the negative results obtained in the attempts to convert graphite into diamond by electrical heating are not due to lack of temperature; on the other hand, the presence of minute crystals in the molten layer of the steel of the end of the barrel subjected to high gaseous pressures of carbon monoxide, carbon dioxide, and hydrogen appears to be connected with the other experiments bearing upon the inclusion of gases in metal as a factor in the production of diamond.

The experiment of firing a high velocity steel bullet with cupped nose through vaporizing carbon into a hole in a block of steel has tested the effect of a momentary pressure of about 300,000 atmospheres on carbon initially near its melting-point, and probably raised by adiabatic compression by another 1000° C.

The fact that only a very few minute crystals resembling diamond were produced (probably from the iron) raises the question as to whether the duration of the pressure is sufficient to start a transformation of graphite to diamond which can be detected by analysis. We have distinct evidence that, with iron as the matrix, the time is sufficient to form very small crystals which can be identified with some certainty, so it therefore seems reasonable to conclude that there was no incipient transformation in bulk, and that however long the pressure of 300,000 atmospheres were applied, it is extremely doubtful if any change would occur.

The pressure of 300,000 atmospheres is between one quarter to one half that obtaining at the centre of the Earth, but vastly greater pressures exist at the centre of the larger stars, and are produced by the collision of large bodies in space; these pressures are many thousands of times greater, and whether they would effect the change it is impossible to predict. On the other hand, a heating effect on large masses of iron might be produced by collisions, and owing to the heat generated by adiabatic compression of the central portions, some of the mass would be melted and subsequently cooled on release of the pressure, so that if heating and cooling under pressure are alone necessary for the production of diamond large stones might result. These considerations, though of interest as bearing upon the presence of diamonds in meteorites and also indicating a possible origin of natural diamond, are of no practical value to us because the pressures required are entirely beyond our reach. There are, however, other considerations arising out of the experiments of Marsden, Moissan, and Crookes, as well as our own, which seem to give some hope of solutions of the problem at issue which lie within the means at our disposal.

A repetition has been made of many of the experiments in which diamond is claimed to have been produced. These have given negative results in all cases except where iron has played a part, as for instance when olivine, being partly reduced by carbon or a reducing flame, small spherules of iron are produced and may, if the mass is quickly cooled, be found to contain diamond.

The repetition of MOISSAN'S experiments under a variety of conditions and pressures has not only confirmed his results but has thrown, it is hoped, additional light on the causes operating to produce diamond in iron.

The experiments under high pressure in steel moulds, where heating of the charge was effected by a central core through which current was passed, enabled HANNAY'S experiments with dipple oil to be tried under much higher pressures, and more thoroughly than is possible with steel tubes in a furnace.

The Appendix gives some indication of the many substances and chemical reactions tested. The results were chiefly negative. The few that were favourable were generally attributable, as has been said, to the presence of iron. It was noticed that the iron seldom contained diamond unless when so situated in the charge as to cause equal cooling on all sides, and it will be remembered that the experiments under atmospheric pressure showed this condition to be essential for the formation of diamond.

In some of the experiments of this group considerable gaseous pressure existed up to 6,000 atmospheres, but it is doubtful if in these the right kind of gas was present or a sufficiency of heating or carburization of the iron occurred. On the whole, therefore, it would appear that all, or nearly all, the chemical reactions as such, under pressures up to 6000 atmospheres, have given negative results.

The experiments on very rapid cooling would seem to dispel the theory that carbon can be caught in a state of transition, and to lead us to the conclusion that quick cooling is not in itself a cause of the occurrence of diamond in rapidly cooled iron.

MOISSAN observed that when the spherules of granulated iron were cracked, or contained geodes, no diamond was ever found in them, and he attributed this to want of mechanical pressure. The experiments we have made not only corroborate this fact, but they tend to show, we think conclusively, that the cracks in the spherules act by allowing a free passage for the occluded gases to escape, and the geodes by providing cavities in which the gases can find lodgment without much gaseous pressure occurring in the metal.* Further, the experiments have shown that iron when it sets does not expand with appreciable force, and that the only compressive forces that are brought to bear on the interior are those arising from the contraction of the outer layers.

Our experiments further show that when a crucible of molten iron is subjected to pressure more than three times as great as can be produced by these contractile forces, the yield of diamond is not increased. On the other hand, when the

* Conversely they may act to allow gases to enter the metal.

conditions of the experiment operate to imprison the occluded gases, then the yield of diamond is about the same as if the crucible had been plunged into water, while if the conditions are such as to allow a free passage through the skin of the ingot, the yield is at once diminished, even though the bulk pressure on the ingot is the same.

The experiment, on compressing acetylene and oxygen, has shown that minute crystals, probably diamond, are produced almost instantaneously in the molten surface of metal exposed on one side to gases consisting of carbon monoxide, carbon dioxide, and hydrogen at very high temperature and at 15,000 atmospheres. Sir WILLIAM CROOKES' experiment described in his lecture before the British Association at Kimberley in 1905 is somewhat analogous; cordite with a little additional carbon was fired in a chamber, the pressure reaching 8,000 atmospheres, a few crystals of diamond were found and isolated; this result CROOKES attributed to the melting of the carbon under the temperature of explosion and crystallization under the pressure on cooling.

Under the conditions of the experiment there would be a considerable amount of the surface of the chamber melted and swept into the products of the charge by the turbulence of the explosion, and the spherules of iron would thus be carburized and cooled while still under heavy pressure.

In the acetylene-oxygen experiment there is a molten surface with reducing gases on one side at high pressure, and on the other metal impervious to gases. In CROOKES' experiment the globules of metal are surrounded by gases at high pressure. In both cases the metal has solidified with the occluded gases imprisoned by the high external gaseous pressure, for we have seen that the pressure of occluded gases in highly carburized iron when quickly cooled cannot exceed about 1000 atmospheres.

The experiments under vacua from 75 mm. up to X-ray vacua have shown generally that as the vacuum is increased the yield of diamond in the crucible is diminished, and that below 2 mm. none has been detected. But when alloys previously boiled at atmospheric pressure are quickly heated up under high vacuum violent ebullition takes place, from the large volume of gases liberated, and some of the contents are ejected into the vacuum chamber before they have had time and sufficient temperature to part with their occluded gases, and diamond occurs in the spherules so ejected.

The gases occluded in cast iron which are given off when heated *in vacuo* have been investigated by H. C. CARPENTER and others, and the relative amounts of the constituents are found to vary widely according to the previous heat treatment and the nature of the gases in contact with the metal while molten and during cooling; they are carbon monoxide and carbon dioxide, hydrogen and nitrogen.

H. C. CARPENTER ('Journal of Iron and Steel Institute,' 1911) states that, when heating up a bar of cast iron *in vacuo* in a silica tube, "After the twenty-fifth heat

it was noticed that in the water-cooled areas of the quartz tube a lustrous black ring had formed. On being strongly heated, some of this, evidently carbon, burnt off, leaving a white film, presumably silica. This seems to show that a volatile silico-organic compound, containing carbon, hydrogen, and silicon, was evolved from the iron on heating."

It would appear from our experiments that probably a ferro-silicon carbonyl is given off from the iron, for, as has been said, we observed a corrosive action on carborundum by the gas evolved from iron borings at red heat under a high vacuum, and the same action was produced by gaseous ferro-carbonyl, and also by carbon monoxide, previously passed over molten iron sulphide at atmospheric pressure.

Let us consider what happens in an ingot or spherule when rapidly cooled simultaneously on all sides. It is first surrounded by a thin coat of solidified metal which, below 600° C., is impervious to gases. As the coat thickens layer within layer, more and more gas is ejected by the solidifying metal, and its semi-solidified centre, still pervious to gas, receives the charge. As this process progresses the pressure may rise higher and higher, though there may be a limit to the pressure against which the metal is able to eject gas when setting. All we, however, know is, that the mechanical strength of the ingot or spherule places a limit of about 7000 atmospheres on the gaseous pressure, and, as we have already mentioned in the case of some iron alloys, most of the spherules are split or shredded, with an appearance consistent with this view.

CROOKES' microscopical examination of diamonds with polarized light supports this view. In his lecture at Kimberley, in 1905, he states: "I have examined many hundred diamond crystals under polarized light, and with few exceptions all show the presence of internal tension.

"On rotating the polarizer, the black cross most frequently seen revolves round a particular point in the inside of the crystal; on examining this point with a high power we sometimes see a slight flaw, more rarely a minute cavity. The cavity is filled with gas at enormous pressure, and the strain is set up in the stone by the effort of the gas to escape."

It seems therefore probable, or indeed almost certain, from the accumulated evidence, that the chief function of quick cooling in the production of diamond in an ingot or spherule is to bottle up and concentrate into local spots the gases occluded in the metal which, under slow cooling, would partially escape and the remainder become evenly distributed throughout the mass.

As to the condition in which the gases exist within the iron at temperatures above 500° C. little is known, though at 200° C. and at 180 atmospheres MOND has shown that iron penta-carbonyl is formed. The intimate contact between the occluded gases and other elements, metals or carbides, must favour complex interactions as cooling takes place. Such actions might be concentrated by the heat flow across the metal on quick cooling.

It appears probable that concentration of gaseous pressure causes certain reactions which bring about an association of carbon atoms in the tetrahedral form—against their natural tendency to assume the more stable form of graphite.*

The necessity of subjecting the iron to a temperature above 2000° C. before cooling would seem to imply the necessity of carbides of the other metals, such as silicon, magnesium, &c., being present to insure the necessary chemical reactions with the gases at high pressure within the ingot.

In reviewing all our experiments, the greatest percentage of diamond occurred when the atmosphere around the crucible consisted of 95 per cent. carbon monoxide and 1 per cent. hydrogen, 2 per cent. hydrocarbons, 2 per cent. nitrogen, the mean pressure in the vessel being about 1 inch absolute of mercury. The weight of diamond we estimated to be about $1 \div 20{,}000$ of the weight of the iron. If we, for the moment, assume a volume of carbon monoxide at atmospheric pressure equal to 0·69 that of the iron, the weight of carbon contained in it equals that of the diamond.

For the following reasons it would appear that the formation of diamond in rapidly-cooled iron takes place when it is solid or in a plastic condition, or even at a still lower temperature. The rapid pitting of a diamond in highly carburized iron just above its melting point is so pronounced that the largest diamond hitherto produced artificially would be destroyed in a second or two if the iron matrix were molten. The production of diamond was obtained in an ingot rapidly cooled after it had set sufficiently hard to be handled in a spoon. A similar result was obtained in the case of a crucible placed in the die and subjected to 11,200 atmospheres pressure after the contents had set. MOISSAN found the diamonds to occur in the centre of the ingots both in the case of iron and also of silver.

It has been seen that iron is permeable to carbon monoxide and hydrogen at temperatures above 600° C., and there appears to be no reason why the concentration of the occluded gases should not take place within the mass as effectively at 600° C. as at higher temperatures, provided that they cannot escape. The most probable temperature, however, may be the point of recalescence at 690° C.†

It would appear that the function of the impervious metal coating thrown around the ingot by quick cooling might be better effected by gas of the same composition as that which the metal ejects on cooling, the pressure being sufficient to ensure that the gaseous pressure around the ingot shall be equal to, or greater than could occur on quick cooling. Such a substitution might result in a larger gaseous content and a larger proportion of the ingot being brought into a suitable condition for the formation of diamond, and the yield might thereby be increased. Some gradations

* It also appears that the conditions may operate to the exclusion of some gas or element inimical to the formation of diamond from certain parts of the metal, viz., the graphite liberated and the cooled metal of the outer layers may absorb some gas or element from the inner portion of the ingot and leave none for the central portion.

† These conditions may also operate to exclude some gases from certain portions of the metal.

of temperature might still be found necessary to concentrate the reactions. It seems however probable that the rate of cooling might be so much prolonged as to obtain much larger crystals and a larger total yield.

The presence of crystals of silica, alumina and magnesia and the spinels and pyrope associated with diamond in rapidly cooled iron alloys, and also when oxidized by steam and some other gases, appears to have a bearing upon the presence of similar crystals usually found in association with diamond, and to be compatible with the conclusions of BONNEY that eclogite is the parent-rock of the diamond in South Africa. It seems probable that both the eclogite and the diamond may have been crystallized nearly simultaneously from an iron alloy.

MOISSAN, after a recital of the geological conditions existing in the South African pipes (see 'Four Electrique,' p. 115), came to the conclusion that diamond was not a vein mineral, but must have been evolved in the midst of a plastic mass; and he concludes that iron at high pressure must have been the matrix. Our experiments, however, seem to show that bulk pressure on the metal does not play a part, but that the previous heat treatment, the impurities in the iron and the condition of the gases within the metal, are the important factors.

It is interesting to note that in the best experiments the yield of diamond in rapidly-cooled iron has reached $1 \div 20{,}000$ of the weight of iron, whereas the weight of diamond obtained from the blue ground of the South African mines is only $1 \div 5{,}400{,}000$. This comparison appears to be confirmed by the relative rarity of microscopic diamonds we have found in the many analyses we have made of blue ground and of the conglomerate from Brazil.

Thus in cooled iron there may be more than 270 times as much diamond as exists in the bulk average of blue ground.

In conclusion, I desire to express my obligations for kind assistance and advice to Sir DUGALD CLERK, Prof. JEANS, Mr. STANLEY COOK, Mr. CAMPBELL SWINTON, and to many other friends, as also to Mr. H. M. DUNCAN.

From 1906 to 1908 inclusive, the late Mr. TREVOR CART assisted me in the arrangement of the experiments and was responsible for most of the analyses until the time of his death.

From January, 1911, to August, 1914, Mr. H. M. DUNCAN acted as my assistant and analyst and has given valuable help in the collection and tabulation of the whole of the work. During the preceding and intervening periods the analyses were made in the laboratory at my house.

APPENDIX.

ABRIDGED SCHEDULE OF EXPERIMENTS.

(A.) UNDER PRESSURES GREATER THAN ATMOSPHERIC.

k.w.m. = kilowatt minutes. Nil in Result column means no diamond formed.
tons = pressure per square inch.

From every experiment several samples were selected representing different parts of the ingot or mixture and analysed separately.

(See figs. 1, 2, and 3.)

Experiment.	Result.
4-inch mould; iron tube core, filled with ferrous oxalate, marble round, iron disc to bring current to top carbon, and graphite on top, 10 tons pressure, 100 k.w.m. total heating, heating $\frac{1}{4}$ minute.	Nil.
2-inch mould; carbon rod core, layer of silicon carbide, then calcium carbide, then carbon, iron nose piece, 80 k.w.m. total heating, 10 tons pressure, heating 2 minutes.	Nil.
4-inch mould; carbon rod core, water round, then marble, 15 tons pressure, 30 k.w.m. total heating, heating $\frac{1}{2}$ minute.	Nil.
4-inch mould; $\frac{7}{8}$-inch carbon rod core surrounded by marble bush-ring with $1\frac{1}{2}$-inch hole and fitting mould on outside, naphthalene and iron filings surrounding rod below ring, anthracene and naphthalene surrounding rod in hole and above to maintain fluid pressure, iron plate to bring in current to top of rod, 10 tons pressure, 70 k.w.m. total heating, soft carbon produced, heating $1\frac{1}{2}$ minutes.	Nil.
Same as last, but oxalic acid and ferrous oxalate instead of naphthalene, with iron filings below marble ring and also between carbon rod and ring. Anthracene above ring as before to maintain fluid pressure, $\frac{1}{2}$ ton gaseous pressure when somewhat cooled and ram released, carbon eaten away, 400 k.w.m., duration 10 minutes.	Nil.
4-inch mould; carbon core, charcoal round, then marble, crushed arc light carbons on top, 25 tons pressure, 60 k.w.m. total heating, heating 34 seconds, maximum current 14,000 amperes, 11 volts.	Nil.
4-inch mould; carbon core, marble round lower part, crushed carbon and perforated iron disc to bring current to top carbon round upper part, 1 lb. carbon dioxide, snow on top covered with graphite and iron chips, 15 tons pressure, 60 k.w.m. total heating, about $\frac{1}{2}$ ton gas pressure when somewhat cooled and ram released, found to contain about 50 per cent. CO_2, 10 per cent. CO, and hydrocarbon burning with luminous flame, heating 2 minutes.	Nil.
2-inch mould; graphite neck, magnesia piece bridge, 100 tons pressure, 4 k.w.m. total heating, little gas produced, heating 25 seconds.	Nil.

Experiment.	Result.
2-inch mould; $\frac{9}{16}$-inch carbon rod, titanium oxide bridge piece, 30 tons pressure, 10 k.w.m. total heating, carbon fused, heating 11 seconds.	Two good crystals.
4-inch mould; graphite neck, marble bridge piece, 20 tons pressure, 60 k.w.m. total heating, heating $\frac{1}{2}$ minute.	Nil.
2-inch mould; graphite rod core, calcium carbide plus 10 per cent. sulphur packed round, 24 tons pressure, $2\frac{1}{2}$ k.w.m. total heating, heating 25 seconds.	Nil.
Ditto plus carbon tetrachloride	Nil.
2-inch mould; carbon core, ferric oxide and 20 per cent. sugar charcoal round, graphite on top, 24 tons pressure, 2 k.w.m. total heating, 1 ton gaseous pressure when somewhat cooled and ram released, heating 5 seconds.	Nil.
4-inch mould; graphite core, charcoal and 10 per cent. arsenic round, 20 tons pressure, 13 k.w.m. heating, heating 10 seconds.	Nil.
4 inch mould; graphite core plus 5 per cent. sulphur, willow charcoal round, 20 tons pressure, 16 k.w.m. total heating, heating 10 seconds.	Nil.
4-inch mould; carbon rod core, sand below around lower pole piece, carbon tetrachloride to give fluid pressure throughout whole, perforated iron disc to bring current to top carbon, 10 tons pressure, 3 k.w.m. total heating, about $\frac{1}{2}$ ton gaseous pressure when somewhat cooled and ram released, soft amorphous carbon and silicide of carbon formed, heating $1\frac{3}{4}$ minutes.	Nil.
2-inch mould; iron core, potassium ferrocyanide round, graphite on top, 40 tons pressure, 1 k.w.m. total heating, heating 4 seconds, cone melted, current interrupted.	Nil.
4-inch mould; carbon rod core surrounded with aluminium carbide, carbon dioxide snow and charcoal, 10 tons pressure, 18 k.w.m. total heating, heating 5 minutes.	Nil.
4-inch mould; carbon rod core, reduced iron and carbon bisulphide round, 10 tons pressure, 20 k.w.m. total heating, heating 3 minutes, action complete.	Nil.
2-inch mould; aluminium rod core, wood charcoal round, 20 tons pressure, 6 k.w.m. total heating, heating 1 minute.	Nil.
2-inch mould; magnesium rod core, carbon round, 20 tons pressure, 6 k.w.m. total heating, heating $1\frac{1}{4}$ minutes.	Nil.
4-inch mould; 1-inch magnalium rod core, carbon round, 20 tons pressure, 40 k.w.m. total heating, heating $1\frac{1}{2}$ minutes.	Nil.
2-inch mould; $\frac{5}{8}$-inch calcium rod core, carbon round, 20 tons pressure, 3 k.w.m. total heating, short circuit producing some iron which would be rapidly cooled, heating 8 seconds.	A few crystals.
4-inch mould; $\frac{5}{8}$-inch sodium rod core, carbon round, 10 tons pressure, 4 k.w.m. total heating, 4 tons of gaseous pressure when ram released, heating 20 seconds.	Nil.
2-inch mould; carbon rod core, sodium chloride round and graphite on top, 20 tons pressure, 1 k.w.m. total heating, heating $\frac{3}{4}$ minute, carbon rod eaten.	Nil.

Experiment.	Result.
2-inch mould; $\frac{3}{4}$-inch carbon core bored and $\frac{3}{16}$-inch iron rod placed inside, sodium chloride packed round, 20 tons pressure, 8 k.w.m. total heating.	Nil.
2-inch mould; aluminium coil core, wood charcoal round, phosphorus at base, 6 tons pressure, 3 k.w.m. total heating, heating $1\frac{1}{2}$ minutes.	Nil.
4-inch mould; 1-inch aluminium rod core, drilled centre $\frac{3}{8}$-inch filled with carborundum charcoal around, 20 tons pressure, 50 k.w.m. total heating, heating 1 minute.	Altered carborundum.
2-inch mould; carbon rod core, phosphorus and carborundum round, graphite on top, 20 tons pressure, 8 k.w.m. total heating, heating 14 seconds.	Nil.
4-inch mould; magnesium and silicon carbide core, magnesia round, graphite top and bottom, 20 tons pressure, 57 k.w.m. total heating, heating 2 minutes.	Nil.
4-inch mould; 1-inch carbon core, boric anhydride round, 20 tons pressure, $22\frac{1}{2}$ k.w.m. total heating, heating $5\frac{1}{2}$ minutes.	Nil.
9-inch mould; 2-inch carbon core surrounded by wood charcoal, poles water jacketed, 10 tons pressure, 400 k.w.m. total heating, heat melted lower pole and made hole into water cavity causing explosion, molten iron shot out on to floor and walls, heating $4\frac{1}{2}$ minutes.	Several good crystals from ejected iron spherules.
4-inch mould; 1-inch carbon rod core, drilled $\frac{3}{8}$-inch hole, filled with calcium carbide, sand top and bottom, 30 tons pressure, 50 k.w.m. total heating, heating 20 minutes.	Nil.
4-inch mould; $\frac{1}{2}$-inch carbon core, sand, carbon silicide, and calcium carbide in layers round it, 30 tons pressure, 40 k.w.m. total heating, heating 8 minutes.	Nil.
Same arrangement of mould, but calcium carbide and carbon silicide only, plus small amount of sulphur, 30 tons pressure, 7 k.w.m. total heating, some iron present from bottom poles, and also CO produced, heating 1 minute.	Several good crystals which burnt in oxygen.
4-inch mould; $\frac{1}{2}$-inch carbon core, sand top and bottom, $\frac{1}{2}$-inch layer of calcium sulphide in middle, 28 tons pressure, 30 k.w.m. total heating, some iron present from bottom pole, also CO produced, heating 20 seconds.	Some good crystals, some burnt in oxygen.
Same, but layer of calcium carbide, sulphur added to sand	Many crystals which burnt in oxygen.
4-inch mould; $\frac{5}{8}$-inch carbon rod core, sand top and bottom, calcium oxide in centre, plus 5 per cent. ferric oxide, 30 tons pressure, 47 k.w.m. total heating, heating 15 minutes.	Nil.
4-inch mould; $\frac{9}{16}$-inch carbon rod core, slaked lime top and bottom, sand in middle, 30 tons pressure, 14 k.w.m. total heating, heat of 10 to 20 kilowatts applied for 30 seconds, four times, with 30 seconds intervals.	Nil.
4-inch mould; slaked lime top and bottom, sand, salt (10 per cent.) in middle, 30 tons pressure, 8 k.w.m. total heating, heating 35 seconds.	Nil.

Experiment.	Result.
4-inch mould; $\frac{9}{16}$-inch carbon core, slaked lime and sodium carbonate on the bottom, silver sand in middle, slaked lime on top, 30 tons pressure, 770 k.w.m. total heating, heating 30 minutes.	Nil.
4-inch mould; $\frac{9}{16}$-inch carbon core, sand and sodium hydroxide 5 per cent., 25 tons pressure, 40 k.w.m. total heating, iron melted from bottom pole, heating 30 minutes.	Some crystals from centre which burnt in oxygen.
Same experiment plus 10 per cent. lampblack, 25 tons pressure, 12 k.w.m. total heating, heating $1\frac{1}{2}$ minutes.	Nil.
4-inch mould; carbon core, containing 5 per cent. lime, sand round, 25 tons pressure, 8 k.w.m. total heating, heating $1\frac{1}{2}$ minutes.	Nil.
4-inch mould; carbon rod core with sodium carbonate, sand round as before, 25 tons pressure, 6 k.w.m. total heating, heating 25 seconds.	Nil.
4-inch mould; carbon core, olivine, graphite, and water round, 10 tons pressure, 12 k.w.m. total heating, charge blew out at base, the pellets would be charged with CO and H from water and carbon, heating 2 minutes.	Pellets of iron shot out, which gave several good crystals.
4-inch mould; iron rod core, olivine, rubber shavings and vaseline round, 10 tons pressure, 9 k.w.m. total heating, heating 45 seconds.	Nil.
4-inch mould; carbon core, pyrope round, Dippel's oil and paraffin poured on top, covered with iron and graphite, 26 tons pressure, 11 k.w.m. total heating, heating 15 seconds.	Nil.
4-inch mould; 1-inch carbon core, graphite and water round, 20 tons pressure, 8 k.w.m. total heating, heating 15 seconds.	Nil.
4-inch mould; 1-inch carbon core, iron oxide, lampblack and water round, 10 tons pressure, 18 k.w.m. total heating, heating $1\frac{1}{2}$ minutes, much gas produced.	One good crystal from iron buttons.
4-inch mould; $\frac{3}{4}$-inch core, magnesite round, iron turnings on top, then carbon bisulphide, 20 tons pressure, 8 k.w.m. total heating, heating $1\frac{1}{2}$ minutes.	Nil.
4-inch mould; 1-inch carbon core, 2-inch paper tube, filled with iron filings, sand round, cast-iron borings on top, carbon and tetrachloride of carbon above, 10 tons pressure, 20 k.w.m. total heating, action very vigorous, solid iron core formed round carbon, and the whole of the sand was permeated with deposited carbon and iron chloride, heating 4 minutes.	Nil.
2-inch mould; $\frac{3}{8}$-inch carbon rod core, 1-inch paper tube filled with carborundum and iron sulphide, sand and sulphur round, 10 tons pressure, 6 k.w.m. total heating, silicon formed, heating 22 seconds.	Nil.
2-inch mould; $\frac{1}{4}$-inch iron rod core, olivine and 5 per cent. carbon round, 100 tous pressure, 4 k.w.m. total heating, heating 1 minute.	Nil.
2-inch mould; $\frac{3}{8}$-inch carbon rod core, $\frac{1}{8}$-inch iron in centre, sodium carbonate round, 100 tons pressure, 4 k.w.m. total heating, heating 10 seconds, charge detonated, mould swelled $\frac{1}{8}$-inch in diameter, charge blew out top and bottom.	One or two crystals from iron.

Carbide mixture = CaC_2 1, SiO_2 2, Al_4C_3 3, FeS 16, Mg 2 parts by weight.

Experiment.	Result.
4-inch mould; iron sulphide core, carbide mixture, and wood charcoal round, 20 tons pressure, 10 k.w.m. total heating, heating $1\frac{1}{4}$ minutes.	Nil.
$4\frac{3}{4}$-inch mould; iron sulphide core, carborundum, caustic soda, and sulphur round, 20 tons pressure, 30 k.w.m. total heating, heating $3\frac{3}{4}$ minutes.	Some crystals.
$4\frac{3}{4}$-inch mould; iron sulphide core, carborundum, caustic soda, sodium carbonate and sulphur round, 15 tons pressure, 30 k.w.m. total heating, about $\frac{1}{2}$ ton of gaseous pressure when somewhat cooled and ram released, heating 3 minutes.	Some crystals.
$4\frac{3}{4}$-inch mould; iron sulphide core, carborundum, sodium carbonate and sulphur round, 15 tons pressure, 30 k.w.m. total heating, heating 8 minutes.	Nil.
$4\frac{3}{4}$-inch mould; iron sulphide core, carborundum and caustic soda round, 15 tons pressure, 25 k.w.m. total heating, about 2 tons of gaseous pressure when ram released, heating 6 minutes.	Nil.
$4\frac{3}{4}$-inch mould; iron sulphide core, carborundum, calcium fluoride and sulphur round, 20 tons pressure, 25 k.w.m. total heating, about 1 ton of gaseous pressure when ram released, heating $1\frac{1}{2}$ minutes.	Nil.
$4\frac{3}{4}$-inch mould; carbon core, paper tube round it filled with iron fluoride, silica round, 20 tons pressure, 40 k.w.m. total heating, heating 1 minute.	Nil.
4-inch mould; carbon rod core, 2-inch paper tube filled with carborundum and sodium, outside paper tube packed with marble, graphite on top, 20 tons pressure, 25 k.w.m. total heating, sodium carbide and silicon formed, heating 1 minute 5 seconds.	Nil.
4-inch mould; $\frac{3}{4}$-inch carbon rod core, 3-inch paper tube containing sodium and carborundum, calcium carbide round, 26 tons pressure, 18 k.w.m. total heating, heating 1 minute.	Nil.
4-inch mould; $\frac{3}{4}$-inch carbon rod core, iron filings round, carborundum on top, then sodium and mercury covered with iron filings, 26 tons pressure, 80 k.w.m. total heating, heating 1 minute.	Nil.
4-inch mould; $\frac{1}{2}$-inch carbon rod core, $1\frac{1}{8}$-inch paper tube containing iron filings, carbon round, sodium on top, 26 tons pressure, 30 k.w.m. total heating, heating 40 seconds.	Nil.
4-inch mould; $\frac{1}{2}$-inch carbon rod core, $1\frac{1}{8}$-inch paper tube containing iron, barium oxide and strontium oxide, carborundum round, sodium on top, 26 tons pressure, 50 k.w.m. total heating, heating $2\frac{3}{4}$ minutes.	Nil.
4-inch mould; filled with marble, pressed, $1\frac{1}{2}$-inch hole drilled in it, $\frac{1}{2}$-inch carbon rod in centre, iron, carbon, carborundum and titanium round, sodium on top, 26 tons pressure, 37 k.w.m. total heating, heating $3\frac{1}{4}$ minutes.	Nil.
4-inch mould; filled with bauxite pressed to 20 tons, $1\frac{1}{2}$-inch hole drilled in it, $\frac{1}{2}$-inch carbon rod in centre, iron, lampblack, carborundum and bauxite round, sodium on top, 26 tons pressure, 30 k.w.m. total heating, heating $4\frac{1}{2}$ minutes, charge blew out.	Some crystals from ejected iron.

EXPERIMENTS.	RESULT.
4-inch mould; packed with dry alumina, $1\frac{1}{4}$-inch hole, $\frac{1}{2}$-inch carbon rod core, iron, lampblack, carborundum, ferro manganese and thorium nitrate round, sodium on top, 26 tons pressure, 20 k.w.m. total heating, heating 1 minute 10 seconds.	Nil.
4-inch mould; packed with carborundum, $1\frac{1}{4}$-inch hole drilled in it, $\frac{1}{2}$-inch carbon rod core, $\frac{3}{4}$-inch paper tube filled with iron and ferro vanadium, carbon and iron round, sodium on top, 26 tons pressure, 20 k.w.m. total heating, heating 1 minute.	Nil.
4-inch mould; packed with anhydrous ferrous oxalate, $\frac{1}{2}$-inch hole drilled, $\frac{1}{4}$-inch iron rod core, sodium round, ferrous oxalate on top, covered with graphite, 26 tons pressure, 25 k.w.m. total heating, heating 3 minutes.	Nil.
4-inch mould; packed with sodium chloride and carborundum, $\frac{5}{8}$-inch carbon rod core, 26 tons pressure, 25 k.w.m. total heating, heating 4 minutes.	Sodium carbide and silicon formed.
4-inch mould; alumina, rouge, magnesium chloride and calcium chloride, pressed; $1\frac{1}{4}$-inch hole, $\frac{5}{8}$-inch carbon rod core, carborundum in hole, 26 tons pressure, 15 k.w.m. total heating, heating $1\frac{1}{4}$ minutes.	Green carborundum produced.
4-inch mould; packed with carborundum, calcium carbide, aluminium carbide, sodium chloride, magnesium chloride, sulphur, iron filings; $\frac{5}{8}$-inch hole drilled, carbon rod put in, carbon tetrachloride poured on top, covered with graphite, 26 tons pressure, 50 k.w.m. total heating, heating 13 minutes, charge blew out, mould dilated.	Some crystals from centre and also from ejected matter.
4-inch mould; packed with alumina, $\frac{1}{2}$-inch hole, $\frac{1}{4}$-inch iron rod core, lead peroxide and carborundum placed in hole and on top, covered with iron filings, 26 tons pressure, 8 k.w.m. total heating, heating 1 minute.	Nil.
4-inch mould; packed with alumina, 1-inch hole, $\frac{1}{4}$-inch iron rod core, rouge and carborundum in hole, 26 tons pressure, 50 k.w.m. total heating, heating 3 minutes.	Some amorphous carbon and silicate of iron.
4-inch mould; packed with carborundum and 4 per cent. sodium carbonate, pressed at 16 tons, 1-inch hole drilled, $\frac{5}{8}$-inch carbon rod core, carborundum and sodium carbonate placed in hole, 26 tons pressure, 15 k.w.m. total heating, heating 3 minutes, formed grey solid which detonated when struck.	Sodium carbide and pale green carborundum.
4-inch mould; $\frac{1}{2}$-inch carbon rod core, iron sulphide and sulphur round, then sodium silicate and lampblack, 26 tons pressure, 50 k.w.m. total heating, heating $2\frac{3}{4}$ minutes.	Nil.
4-inch mould; 1-inch carbon rod core, sodium silicate, alumina, rouge, magnesia, and lime round, 26 tons pressure, 40 k.w.m. total heating, heating $1\frac{1}{2}$ minutes.	Nil.
4-inch mould; packed with artificial pyrope, 1-inch hole, $\frac{1}{2}$-inch carbon rod core, carborundum and sodium peroxide placed in hole, 26 tons pressure, 8 k.w.m. total heating, heating $3\frac{1}{2}$ minutes.	Nil.
4-inch mould; filled with sand, 1-inch hole, $\frac{1}{2}$-inch carbon rod core, filled hole with potassium nitrate and carborundum, 26 tons pressure, 15 k.w.m. total heating, heating 1 minute.	Nil.

Experiment.	Result.
$4\frac{1}{4}$-inch mould; packed with sand, coke, sawdust and salt pressed, $\frac{1}{2}$-inch hole drilled, $\frac{1}{2}$-inch carbon rod core covered with iron filings and graphite, 26 tons pressure, 28 k.w.m. total heating, heating $1\frac{1}{2}$ minutes, about 1 ton of gaseous pressure when somewhat cooled and ram released.	Green carborundum non-polarizing.
Same as the above plus 20 per cent. ferrous oxalate, 5 tons pressure, 30 k.w.m. total heating, about 3 tons of gaseous pressure when somewhat cooled and ram released, heating 1 minute 40 seconds.	Many green and also clear non-polarizing plates.
Same as above plus zinc dust, $\frac{3}{4}$ ton pressure, 28 k.w.m. total heating, heating 3 minutes, about 1 ton of gaseous pressure when somewhat cooled and ram released.	Green and yellow plates which would not burn in oxygen.
$4\frac{1}{4}$-inch mould; three-fourths full of CO_2, snow pressed hard, sand, coke, sawdust and salt put on top, carbon rod core, 15 tons pressure, 9 k.w.m. total heating, heating $5\frac{1}{4}$ minutes.	Nil.
2-inch mould; $\frac{1}{2}$-inch carbon rod core, carborundum and chromium oxide packed round, 20 tons pressure, 12 k.w.m. total heating, heating $\frac{3}{4}$ minute.	Green carborundum and silicon produced.
2-inch mould; $\frac{1}{2}$-inch carbon rod core, ferrous oxalate and magnesium packed round, 20 tons pressure, $\frac{1}{2}$ k.w.m. total heating, a few rounded particles of hard carbon, charge blew out after 2 seconds heating, repeated three times with same result, should be further investigated.	Nil.
2-inch die; melted tin and rubber poured into die, crucible containing molten iron placed on top and pressed to 18 tons.	Nil.
$\frac{3}{8}$-inch diameter air-hardened tungsten steel die; graphite and 7 grains of fulminate under 230 tons pressure, die heated 180° C., fulminate exploded and retained in mould.	Nil.
$\frac{3}{8}$-inch diameter air-hardened tungsten steel die; graphite and 15 per cent. potassium chlorate placed in press, and pressure increased till mixture detonated at about 200 tons per sq. inch.	Nil.
$\frac{3}{8}$-inch diameter air-hardened tungsten steel die; graphite and saltpetre and three fulminate caps, heated with burner to above temperature of detonation of fulminate under 230 tons pressure, nothing escaped.	Nil.
Melted iron in carbon crucible, added bismuth, poured into steel mould and pressed to 20 tons.	Nil.
$1\frac{1}{2}$-inch tempered steel die; sodium in bottle surrounded with mixture of carborundum and lampblack, added water, and pressed at 100 tons for 10 minutes.	Nil.
$1\frac{1}{2}$-inch tempered steel die; lithium in bottle, carborundum and water, 50 tons pressure for 15 minutes.	Nil.
$1\frac{1}{2}$-inch tempered steel die; sodium in bottle, carborundum and water, 40 tons pressure, temperature of die raised to 250° C. by means of gas burners.	Nil.
$1\frac{1}{2}$-inch tempered steel die; potassium in bottle and carbon bisulphide, 60 tons pressure, die heated by gas to 250° C.	Nil.
$1\frac{1}{2}$-inch tempered steel die; ferrous oxalate and sodium, 100 tons pressure for $4\frac{1}{2}$ hours.	Nil.

Experiment.	Result.
1½-inch tempered steel die; gum arabic and phosphoric oxide, 60 tons pressure, heated by gas for 1 hour to 250° C., black cindery deposit produced.	Nil.
1½-inch tempered steel die; lead peroxide and carborundum grit mixed with lead fluoride, 50 tons pressure, heated by gas for 1 hour, red leaflets of lead produced.	Nil.
1½-inch tempered steel die; sodium peroxide, carborundum No. 6 grit and sodium chloride heated 45 minutes to 200° C., 50 tons pressure.	Nil.
1½-inch tempered steel die; sodium peroxide, carborundum and artificial pyrope, heated by gas ¾ hour to 200° C. at 50 tons pressure.	Nil.
1½-inch tempered steel die; calcium carbide and glass bulbs filled with water, pressure 50 tons, time 30 minutes.	Nil.
Ditto plus a small amount of sodium.	Nil.

DUCK-GUN EXPERIMENTS.

(See figs. 4 and 5.)

Experiments.	Result.
Fired piston on to charge of graphite and cotton wool placed at end of barrel which contained air at atmospheric pressure; propellant, 20 grains black powder.	Nil.
Same as above, but barrel filled with oxygen; propellant, 40 grains black powder.	Nil.
Same as above, but barrel filled with oxygen and acetylene, and propellant 57 grains of black powder.	Small crystals in skin of piston and end plug, probably Moissan effect.

Calculation of the Temperature Reached on the Compression of Acetylene and Oxygen Experiment.

By Stanley S. Cook.

The temperature reached may be estimated from the final pressure, which the observed deformation of the block and plug indicates to have been in the neighbourhood of 100 tons per sq. inch. But it must be remembered that there is a change of molecular volume as a result of combustion. Thus the mixture which, as C_2H_2 and 5 (O), has $3\frac{1}{2}$ molecular volume, would on combustion to $2CO_2$ and H_2O have only 3 molecular volumes. The final temperature deduced from the pressure will therefore depend upon the extent to which chemical combination has taken place.

The original mixture being at atmospheric pressure and a temperature of 290° C. absolute, a pressure of 100 tons per sq. inch after compression to $\frac{1}{288}$ of its original volume would indicate a temperature of 15,250° C. If, however, complete combustion has taken place, this same pressure would correspond to a temperature greater in ratio of $3\frac{1}{2}$ to 3, viz., to 17,700° C. The actual temperature must therefore have been something between these two values.

(B.) RIFLE EXPERIMENTS.

(See figs. 6, 7, and 8.)

EXPERIMENT.	RESULT.
0·303 bullets were fired into holes, 0·303-inch diameter (in some cases of tungsten steel air hardened, tapering to $\frac{1}{8}$-inch at the bottom) in steel blocks; in the holes were placed the following substances:—	
Coarse carborundum; No. 6 grit, which after the experiment was found to be crushed to small splinters.	Nil.
Carborundum (very fine grained) and sodium; carborundum unaltered	Nil.
Carborundum and iron; very fine grained carborundum and finely powdered iron, the carborundum appeared slightly whitened, otherwise unaltered.	Nil.
Calcium carbide and sulphur; calcium carbide all destroyed and calcium sulphide formed.	Nil.
Carborundum (very fine grained) and nickel filings; no whitening of the carborundum.	Nil.
Carborundum and sodium peroxide	Sodium silicate and amorphous carbon formed.
Bort; somewhat crushed	No change.
Fired bullet through carbon arc into hole in steel block	Nil.
Fired bullet through carbon rods arcing in a bed of graphite contained in fire clay crucible, above the hole in steel block.	Nil.
Graphite and sodium peroxide	Graphite destroyed.
Fired bullet through white hot iron plate into hole containing carbon rod heated white hot by resistance heating.	Nil.
Fired bullet into hole containing graphite in steel block whilst arcing between top of hole and a carbon rod to produce vapour of iron.	Rounded fragments of graphite.
Calcium carbide and paper (wet); amorphous carbon formed	Nil.
Cotton wool; a little amorphous carbon formed	Nil.
Potassium chlorate and sugar carbon	Nil.
Carbon bisulphide and rubber	Nil.
Carbon bisulphide and sodium bismuthate	Nil.
Carbon bisulphide and potassium chlorate	Nil.
Mineral oil	Nil.
Carbon bisulphide	Nil.
Sodium bismuthate, benzene, and carbon tetrachloride	Nil.
Graphite and paper; bullet struck side of hole in block, producing a small amount of molten iron.	A few crystals.
Through white hot carbon-bridge over hole electrically heated to point of vaporization.	A few crystals.
Graphite and iron	Nil.
Graphite and fulminate caps.	Nil.
White hot graphite, contained in small crucible previously heated in arc furnace and placed above hole.	Nil.
Red hot graphite as above	Nil.
Graphite and fulminate in paper	Nil.

Experiment.	Result.
Graphite, naphthalene, and fulminate	Nil.
Through carbon crucible, containing white hot highly carburized iron.	Nil.
Graphite, saltpetre, and fulminate	Nil.
Iron, caps, and carbon bisulphide	Nil.
Sugar carbon	Nil.
Sugar carbon plus two caps	Nil.
Sugar carbon plus two caps plus potassium chlorate	Nil.
Sugar carbon plus two caps plus potassium nitrate	Nil.
Sugar carbon, two caps, potassium chlorate and iron	Nil.
Sugar carbon, sulphur and reduced iron	Nil.
Cotton wool soaked in solution of potassium nitrate and dried	Nil.
Iron filings and fulminate	Nil.
Cotton wool soaked in HF., iron and two caps	Nil.
Cotton wool, iron fluoride and caps	Nil.
Sodium fluoride, sugar carbon and fulminate	Nil.
Sodium fluoride and cotton wool	Nil.
Cotton wool, carbon bisulphide and iron filings	Nil.
Cotton wool, carbon bisulphide and fulminate	Nil.
Gun cotton, carbon bisulphide, fulminate and iron	Nil.
Sodium fluoride, iron, fulminate and sugar carbon	Nil.
Sodium fluoride, iron, fulminate, gun cotton, sugar carbon and iron chloride.	Nil.
Sodium fluoride, iron chloride, gun cotton, and graphite	Nil.
Rouge, reduced iron, sugar carbon and carbon bisulphide	Nil.
Rouge, reduced iron, sugar carbon and sulphur	Nil.
Aluminium, rouge, graphite and chlorate	Nil.
Rouge, iron fluoride, chlorate, graphite and caps	Very small residue; doubtful.
Bullet fired into barrel filled with acetylene and screwed into steel block containing cavity, mouth of barrel closed by gold beater skin.	Nil.

(C) EXPERIMENTS AT ATMOSPHERIC PRESSURE.

All experiments allowed to cool by radiation only unless otherwise stated.

Experiment.	Result.
Aluminium, magnesium, carbon, iron, ferric oxide, olivine, and boric anhydride fused in wind furnace.	Nil.
Aluminium carbide fused in carbon crucible by arc, iron added and then sand, heated for 3 minutes. Ingot of iron obtained and a greenish-blue slag ($Al_2O_3 . SiO_2$) charged with carbon.	Nil.
Aluminium carbide melted and run into molten sand in carbon crucible	Nil.
Aluminium carbide and iron heated in carbon crucible	Nil.
Iron pipe packed with graphite melted in electric furnace and then dropped into sulphuric acid with layer of mercury on bottom.	Nil.
Calcium silicate fused in electric furnace, graphite added and molten iron dropped on to it.	Nil.

Various Moissan experiments when quickly cooled gave good results.

Experiment.	Result.	
Phosphate of iron, covered with olivine in carbon crucible, heated by arc	Nil.	
Olivine, iron and graphite, melted in carbon crucible by electric arc . .	Nil.	
Iron, carbon and chromium, 1 per cent., fused in carbon crucible, cooled in water.	One crystal, 0·5 mm. long, which burnt in oxygen.	
Melted olivine in carbon crucible, added calcium carbide, covered with a layer of sand, fused and allowed to cool slowly.	Nil.	
Fused sodium carbonate and sand, added calcium carbide.	Nil.	
Fused sodium carbonate, silica, sodium chloride, calcium carbide and graphite.	Nil.	
Melted iron in carbon crucible, then forced naphthalene vapour through the melt with arc still on, cooled in water.	A few crystals.	
Carbon bisulphide vapour blown through molten iron charged with carbon, cooled in water.	Doubtful.	
Superheated steam blown through carbon tube into cast iron melted by arc in carbon crucible, cooled in water, large crystalline deposit round carbon tube.	About 10 per cent. burnt in oxygen.	
Same experiment repeated many times and carbide mixture* added while cooling before steam applied.	Same result.	
Iron 20 per cent., magnalium 20 per cent., calcium carbide 30 per cent., sand 30 per cent., melted in carbon crucible by arc and then water added.	Same result.	
Iron melted in carbon crucible, aluminium carbide and silica added, stirred and then sulphur dioxide blown through for half an hour, quenched in water.	Same result.	
Steam and benzene blown through carbon tube into silicon carbide and iron melted in carbon crucible by arc.	Nil.	
Poured iron highly charged with carbon through narrow git into massive steel mould closed at bottom by breech screw, no mechanical pressure exerted on breech screw.	—	Fig. 9.
Did same with plunger forced down git; no pressure from expansion on setting.	—	
Passed CO through molten iron in carbon crucible, poured melt into the above mould.	Nil.	
Melted iron, added 5 per cent. zinc and poured into the above mould. .	Nil.	
Poured molten carburized iron into molten zinc	Nil.	
Olivine fused in small carbon crucible, stirred with a graphite rod, and oxycoal gas blowpipe played on the molten surface, covered melt with graphite, and cooled slowly.	Nil.	
Melted olivine and 10 per cent. graphite in carbon crucible, oxycoal gas blown through, then water poured on.	Nil.	
Same as above but "blue ground" in place of olivine, granite-like mass formed.	Nil.	
Iron melted in carbon crucible, bismuth added, cooled in water.	Nil.	
Heated iron and carbon, and plunged yellow diamond into it when near setting point.	Etched and pock marked.	

* *Carbide mixture* = CaC_2 1, SiO_2 2, Al_4C_3 3, FeS 16, Mg 2 parts by weight.

Experiment.	Result.
Same experiment with ferro-titanium	Etching deeper.
Iron melted in carbon crucible, aluminium carbide, iron sulphide and silica added, steamed, quenched in water.	Crystals which burnt in oxygen.
Heated iron, magnesium, strontium oxide and calcium, cooled in water .	Nil.
Heated iron, tin, bismuth, antimony, and ferro-vanadium, cooled in water.	Nil.
Heated iron, lead, silver, copper, cooled in water	Nil.
Silica 4, magnesia 5, rouge 1, parts by weight, carborundum and iron melted in electric furnace on graphite bed.	Octahedra (spinels) which would not burn in oxygen.
Melted a round steel file by arcing under oil	Nil.
Heated chromium oxide and carborundum in carbon crucible, cooled in water.	Nil.
Fused calcium carbide and added potassium chlorate, cooled in water . .	Nil.
Passed hydrogen for 1 hour over carborundum No. 6 grit, heated cherry red, grit unaltered.	Nil.
Passed CO for 1 hour over carborundum No. 6 grit, heated cherry red, grit very little altered.	Nil.
Passed iron pentacarbonyl over carborundum No. 6 grit, heated nearly red for 3 hours, the grit was much etched.	Several plates, thought to be correct at the time, but would not burn in oxygen.
Passed iron pentacarbonyl over magnesium (redness) 1 hour	Nil.
Passed iron pentacarbonyl over magnesium carbonate (redness) $\frac{1}{2}$ hour .	Nil.
Passed iron pentacarbonyl over magnesium oxide (redness) 1 hour . . .	Nil.
Passed iron pentacarbonyl over carborundum No. 6 grit and sodium chloride (redness) 1 hour, the grit was etched.	Nil.
Passed iron pentacarbonyl over carborundum No. 6 grit, sodium chloride and iron filings, orange heat 20 minutes.	Nil.
Passed the following over carborundum No. 6 grit contained in a silica tube heated to varying temperatures along its length from just red to white heat for periods varying from 20 minutes to $1\frac{1}{2}$ hours; cyanogen, nitrogen, nitric acid gas mixed with iron pentacarbonyl; chlorine gas; ammonia gas; CO_2 and CO; ammonium chloride vapour; sulphuretted hydrogen; acetylene; coal gas.	All nil.
Passed iron pentacarbonyl over the following substances, heated in a silica tube for varying lengths of time; magnesium silicide; silicon, magnesium and calcium alloy; graphite; ferro silicon; ferro silicon and magnesium alloy; ferro silicon, magnesium powder and sodium chloride; iron sulphide; ferrous carbonate; ferrous ammonium sulphate; sodium; potassium and magnesium sulphates; calcium silicate; magnesium silicate; cadmium; artificial pyrope; ferro manganese; silicon and manganese; chromium; silicon, manganese and carbon alloy; finely divided nickel; sodium silicate; nickel and sand.	All nil.
50 per cent. ferro silicon in silica tube heated to 1300° C., carbon bisulphide vapour passed over it for half an hour.	Nil.
Silicon heated to 1100° C. in silica tube, carbon bisulphide vapour passed over it for half an hour.	Nil.

Experiment.	Result.
Passed CO over iron sulphide melted in salamander crucible in wind furnace. Top of crucible closed by massive iron lid containing on side inlet tube for CO and in centre 1 inch hole with perforated bottom, hole fitted with No. 6 grit.	Many clear plates, some with triangular markings.
Same experiment repeated many times	Would not burn in oxygen.

(D) EXPERIMENTS IN VACUUM.

(See figs. 10 and 11.)

All experiments allowed to cool by radiation only unless otherwise stated.

Experiments.	Result.
4-inch mould; heated reduced iron and lampblack in carbon cup, pressure varied between $\frac{3}{8}$-inch and 3-inch absolute. Cooling by radiation only. Large amount of gas produced containing CO 95 per cent., hydrogen 1 per cent., hydrocarbon 2 per cent., nitrogen 2 per cent. (Fig. 10.)	Large residue of crystals which burnt in oxygen.

High Vacuum Experiments under pressures varying from 10 *mm. to* $\frac{1}{6}$ *mm. absolute.*

Heated iron and 10 per cent. sugar carbon in salamander crucible for half an hour by arc on top, vacuum about 1 mm.	Nil.
Iron, sugar carbon and iron sulphide heated in carbon crucible 15 minutes, then water allowed to enter through tap causing spherules to be ejected, vacuum about 1 mm.	10 per cent. of residue from ejected spherules burnt in oxygen.
As above, but carbon bisulphide instead of water	Nil.
Magnesia, lampblack and tar heated in carbon crucible	Metallic magnesium and magnesium carbide produced.
Alumina and borax fused in wind furnace then heated in carbon crucible in vacuum.	Clear transparent alumina.
Alumina, lampblack and tar heated in carbon crucible	Aluminium carbide formed.
Aluminium carbide, calcium carbide, magnesia, iron, carbon and tar, baked and placed in crucible, resistance heated in vacuum furnace, crucible of sulphur either side to give off sulphur vapour in chamber.	Nil.
Crucible filled with iron and carbon, closed by carbon cover, hole bored in side of crucible and massive iron block placed opposite hole, contents boiled, deposit of carburized iron on block analysed, vacuum about 0·25 mm.	Nil.
Arc deflected by electro-magnet on to iron block, deposit on block analysed, vacuum about 10 mm.	Nil.
Tungsten steel packed round with carbon in carbon crucible and heated to ebullition.	Nil.
Iron, lampblack, Prussian blue heated in carbon crucible by resistance heating.	Nil.
Carbon crucible containing potassium ferrocyanide, lampblack and reduced iron, melted by resistance heating.	Nil.

Experiment.	Result.
Carbon crucible containing sand, sulphur and iron, heated under 1 mm., and coal gas admitted during slow cooling.	Nil.
Iron sulphide, carborundum and magnesia, heated in carbon crucible . .	Nil.
Reduced iron and carborundum, heated in carbon crucible	Nil.
Calcium and carborundum, heated in carbon crucible	Carborundum decomposed to coke-like mass.
Iron, carbon and garnet, heated in carbon crucible	Nil.
Iron, carbon, olivine and iron sulphide, heated carbon in crucible . . .	Nil.
Carborundum, heated white hot in carbon crucible and carbon bisulphide admitted through tap to top of crucible.	Carborundum decomposed.
Reduced iron and lampblack, heated in carbon crucible, sand and sulphur round.	Nil.
Ferrous oxalate, boric acid, sugar carbon and iron, heated in carbon crucible.	Nil.
Sodium carbonate, carborundum and iron, heated for 20 minutes at bright red in iron crucible.	Nil.
Carbon and iron placed in carbon crucible, carborundum grit on top, moderate heat.	Nil.
Carbon and iron chips placed in carbon crucible, massive iron lid with hole bored in it, filled with No. 6 grit, temperature of crucible bright orange, temperature of lid and grit not exceeding dull red heat.	Several crystal plates from top of tube which resembled diamond and burnt in oxygen.
Carbon with reduced iron and rouge in small crucible, same as previous experiment, heated ten minutes bright red.	Nil.
Carbon, iron and carborundum, heated to bright red 50 minutes . . .	Nil.
Calcium and carborundum, heated for 15 minutes to bright red, carborundum No. 6 grit in tube on top.	Nil.
Carborundum, iron and rouge heated 20 minutes to bright red	Doubtful.
Iron, carbon and lime heated 20 minutes to bright red	Nil.
Ferro-vanadium, carborundum, lampblack and iron heated 15 minutes bright orange.	Nil.
Iron pole arcing on iron in crucible rod carborundum on top	Nil.
Carbon pole arcing on iron rod placed in crucible containing iron filings at bottom and carborundum on top.	Several crystal plates very like diamond.
Carbon pole arcing on iron and magnesium, carborundum on top . . .	Nil.
Nickel and carbon heated to orange for 15 minutes, carbon crucible containing No. 6 grit on top.	Nil.
Heated ferrous chloride and carborundum to bright red for 20 minutes, No. 6 grit in tube on top.	Nil.
Gases from heated iron filings in steel bottle passed through silica tube containing carborundum No. 6 grit heated to a bright red.	Several plates as before.
Repeat with same filings gave nothing. To test if due to exhaustion of filings made following experiment:—	
Passed CO over the previously used iron filings, 30 minutes, placed filings in crucible, and heated in vacuum dull red, 20 minutes, carborundum No. 6 grit in hole in massive iron lid on top.	A few plates.

Experiment.	Result.
Heated iron filings and sodium carbonate No. 6 grit in tube on top (bright red) 30 minutes.	Nil.
Heated iron and sodium chloride (bright red) 20 minutes carborundum No. 6 grit in hole in cast-iron lid on top.	Nil.
Heated ferro silicon (15 per cent.) in carbon crucible till most of contents of crucible had volatilized, analysed dust from walls of vacuum pot and ingot remaining in crucible.	Nil.
Heated iron, ferro silicon, ferro manganese, carbon and calcium carbide in carbon crucible.	Nil.
Heated iron, iron sulphide, carbon and calcium carbide, cooled slowly in vapour of carbon bisulphide.	Nil.
Heated iron, carbon, calcium carbide and ferro titanium in atmosphere of carbon bisulphide, pressure $\frac{1}{3}$ mm. absolute.	Nil.
Heated iron, ferro silicon, and ferro titanium in atmosphere of carbon bisulphide, pressure $\frac{1}{3}$ mm. absolute.	Nil.
Heated iron, ferro titanium and ferro silicon	Nil.
Heated iron and carborundum in atmosphere of carbon bisulphide, pressure $\frac{1}{3}$ mm. absolute.	Nil.
Heated reduced iron and lampblack, calcium metal placed near crucible to absorb nitrogen, pressure $\frac{1}{10}$ mm. absolute.	Nil.

EXPERIMENTS IN X-RAY VACUA.

(See fig. 12.)

Experiment.	Result.
Heated cast iron in carbon crucible to vaporising point, cooling by radiation only.	Nil.

The following were all heated to temperature of vaporisation and then allowed to cool by radiation only:—

Heated calcium carbide	Nil.
Heated ferro-silicon	Nil.
Heated calcium carbide and No. 6 grit	Nil.
Heated nickel and No. 6	Nil.
Heated aluminium, magnesium, calcium carbide, and No. 6 grit . . .	Nil.
Heated ferro-silicon, nickel, and No. 6 grit	Nil.
Heated nickel, iron sulphide, and No. 6 grit	Nil.
Heated ferro-titanium and No. 6 grit	Nil.
Heated iron sulphide, zinc, and No. 6 grit	Nil.
Heated ferro-silicon, sand, and lampblack	Doubtful.
Heated reduced iron, sand, and lampblack to point of ebullition at atmospheric pressure, then re-heated under vacuum, much ebullition of gas from metal and many iron spherules thrown out of crucible.	Large yield of rounded particles and plates from ejected metal. These all burnt in oxygen. Contents of crucible yielded nothing.

Experiment.	Result.
Heated samarskite, zircon, iron, carborundum, No. 6 grit, and lampblack	Nil.
Heated reduced iron, sand, and lampblack	Nil.
Heated silicon and carbon	Nil.
Heated olivine, sand, iron, and lampblack	Nil.
Heated magnesia, sand, iron, and lampblack	Nil.
Heated glass, sand, carbon, and iron	Nil.
Heated ferro-silicon, sand, carbon, and iron	Nil.
Heated silicon, iron phosphide, iron, and carbon	Nil.
Heated silicon, manganese, carbon, and iron	Nil.
Heated reduced iron, rouge, sand, and carbon	Nil.
Heated reduced iron, sand, aluminium, and carbon	Nil.

BULK PRESSURE AND VACUUM APPLIED TO A GRANULAR MASS.

Experiment.	Result.
4-inch mould; 1-inch carbon rod with $\frac{1}{4}$-inch iron rod down centre, coarse carborundum round, the vacuum exit tube through side of mould protected by perforated steel plate and drawing the gases from the carborundum, iron turnings on top to bring current into rod, 40 tons pressure, 72 k.w.m. vacuum 0·5 mm. absolute.	Carborundum next to exit tube and layer next iron turnings whitened, and some crystalline plates.

SEALED TUBE EXPERIMENTS.

Experiment.	Result.
Heated sodium, calcium carbide and water in sealed iron tube at 400° C.	Nil.
Heated sodium carbonate and carborundum in sealed iron tube to red heat for 50 minutes, sodium silicate and amorphous carbon formed.	Nil.
As above, but heated for 2 hours 10 minutes at an orange heat, not so much carbon and silicate formed as in previous experiment.	Nil.
Sealed tube containing ferrous oxalate and magnesium powder, heated dull red $\frac{1}{2}$ hour.	Amorphous carbon.
Sealed tube half filled with olivine, graphite, sugar and water, heated to full red for 1 hour.	Nil.