

The Story of the Platinum-Wound Electric Resistance Furnace

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For many years platinum-wound furnaces have been established as a means of obtaining high temperatures without the complication of a protective atmosphere to surround the resistance elements. Indeed, such electric furnaces were first produced commercially in Germany around the turn of the present century. The history of their early development has, however, never been described, and in this paper the author gives a detailed account of their origin and their gradual development into valuable items of laboratory equipment.

Standard reference books are rather coy about the history of the simple platinum-wound tube or muffle furnace and even extensive reviews are not any more revealing (1,2). Certainly the Dictionary of Scientific Biography does attribute its invention correctly (3), but such a reference presupposes prior knowledge. In actual fact, the history of this common article of laboratory furniture is rather fascinating, if only because of the time lapse between the observations that made it possible and its development.

The Contributions by Volta, Children and Pepys

The story effectively starts with the letter, dated 20th March 1800, of Alessandro Volta (1745-1827) describing his famous pile (4), which led to a flurry of interest in things electrical and, in 1809 (5) and 1813 (6), to the development of two large batteries by John George Children (1777-1852). These were enormous: the 1815 development of the 1813 model, for example, had 63 plates of zinc and copper each measuring 6 ft by 2 ft 8 in dipping into 945 gallons of a 3:1 mixture of fuming nitric and sulphuric acids diluted with water, and the flow of electric current was stopped by raising the plates out of the acid by a system of ropes and pulleys. This battery was located at

his father's house, Ferox Hall, near Tonbridge, Kent (7), and was also used by several other Fellows of the Royal Society, including Sir Humphry Davy, for electrical experimentation. For his work with his batteries Children was awarded a Royal Society Medal in 1828. He established, among other things, that the heating powers of metals varied with their specific resistance; but the most interesting observation from our viewpoint was that of William Hasledine Pepys (1775-1856) who was interested in the controversy then raging on whether diamond could convert soft iron into steel on heating (8), or whether the necessary carbon came from an outside source (9,10,11). Use of Children's battery ensured that there would be no source of carbon other than diamond and accordingly (6):

"he bent a wire of pure soft iron, so as to form an angle in the middle, in which part he divided it longitudinally, by a fine saw. In the opening so formed he placed some diamond powder, securing it in its situation by two finer wires . . . All the wires were of pure soft iron, and the part containing the diamond powder, was enveloped by thin leaves of talc [*sic*, presumably mica]. Thus, arranged, the apparatus was placed in the electrical circuit, when it soon became red hot, and was kept so for six minutes . . . On opening the wire, Mr Pepys found that the whole of the diamond had disappeared . . . and all that part that had been in contact with the diamond was converted into perfect blistered steel".



Henry Louis Le Chatelier
1850–1936

Born in Paris, Le Chatelier came into early contact with eminent scientists through his father, then Inspector General of Mines. Educated at the Military Academy, the College Rollin, the École Polytechnique and the École des Mines, he became in 1875–77 a mining engineer in Besançon and from 1877 to his retirement in 1919 was Professor of General Chemistry at the École des Mines: he also held several other teaching posts. Although best known for “Le Chatelier’s Principle”, he had a wide range of interests, including cements, clays and metallurgy. In 1886 he showed that the thermocouple could be made an accurate temperature-measuring device (hence its then name “Le Chatelier’s pyrometer”) and is generally regarded as the father of thermal analysis

So was born the first electrically heated wire resistance furnace, although the heating element was not platinum.

The Lost Years, 1815–1893

Why did nearly a century elapse between the above experiment and the invention of the platinum-wound tube furnace?

The answer is not to be found in lack of technical advance, since continuous current could be obtained from reliable compact batteries before 1840 (12, 13) and the electrical generator had been developed from the rudimentary form of Hippolyte Pixii in 1832 to the commercial model of Zénole Theophile Gramme (1826–1901) in 1870 (14,15). Filament lamps, which employ a somewhat analogous principle for another purpose, developed over the period 1820 to 1878 from early attempts to use platinum wires in a vacuum to usable glass-enclosed lamps (15,16). Moreover, in 1841, James Prescott Joule (1818–1889) quantified the amount of heat

liberated when an electric current is passed through a resistance (17).

However, the reference by Powell and Schroeder (15) and Jarvis (16) to the 1820 work of Warren de la Rue must be treated with caution as de la Rue was then only 5 years old. Comments by Grove (*Phil. Mag.*, 1840, **16**, 338–339) suggest the reference might be to de la Rive, and presumably to Gaspard (1770–1834), since the first scientific paper of Auguste (1801–1873) appears to have been published in 1822.

Lack of interest is likewise not a tenable answer as two reviews which appeared about the turn of the century reveal a continuous interest in certain types of electric furnace. Thus, Street (1) in 1895 considered such furnaces could be divided into two main classes: “les uns ... basés sur l’incandescence d’une fraction résistante de circuit et les autres sur l’emploi de l’arc électrique”, the first referring only to systems where the resistance of the contents was the source of heat. Minet (2) in 1905



**Augustin Georges Albert Charpy
1865–1945**

Born in Oullins, France, the son of a naval captain, Charpy was educated there and at Lyon before entering the *École Polytechnique* in Paris. He remained there, after graduating in 1887, until he had acquired his doctorate for work on the volumes and densities of salt solutions. At this time he got to know Moissan, Le Chatelier and Osmond. In 1892 he moved to the *Laboratoire Central de l'Artillerie de Marine*, in 1898 he joined the *Compagnie de Chatillon-Commentry*, where he rose to be *Technical Director*, and in 1919 he became *Deputy Director of the Compagnie des Aciéries de la Marine and Professor at both the École des Mines and the École Polytechnique*. While most of his work was concerned with the study of alloys, he was also responsible for introducing scientific methods of problem-solving into the companies he was concerned with

lists three stages of development, that in the period 1808 to 1886 being attributed to "laboratory furnaces", but again refers only to the types recognised by Street. In his final complex classification of furnaces, a type not discussed in the text is given as a "solid resistance not in contact with the material" and under this he has "iron wire round the reaction chamber" and "platinum wire" associated with the names "Charpy (1893)" and "Nernst (1900), Howe (1900)" respectively but with no references. In general, therefore, it would appear that electric furnaces were developed with essentially technical and industrial applications in mind—and Street's two classes would fit these requirements.

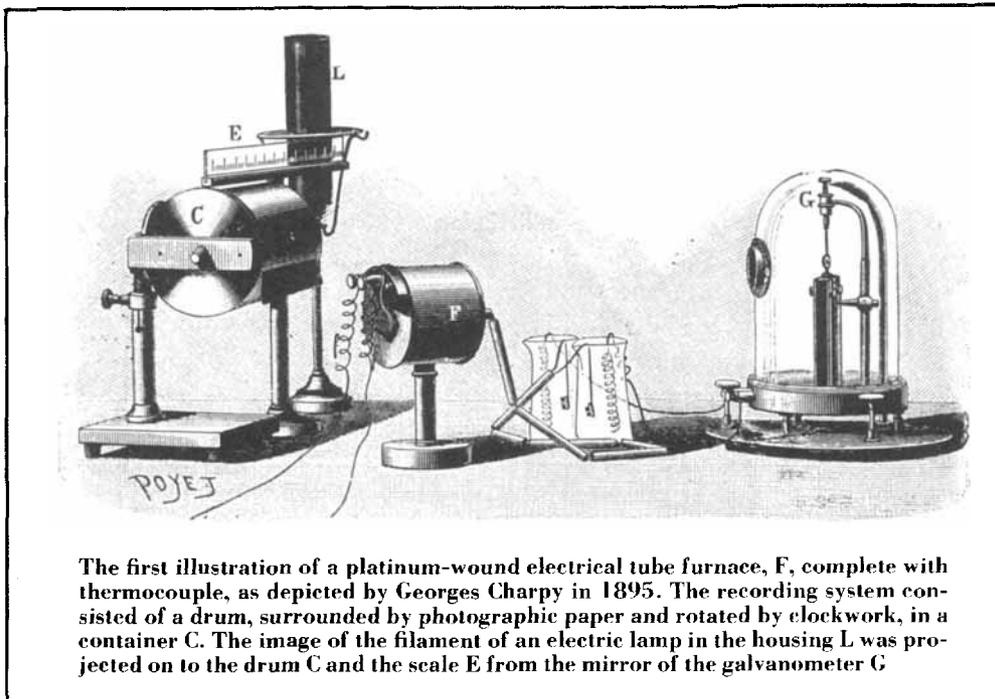
Another factor in the delay was undoubtedly the use of coal gas. With the introduction of the Bunsen burner in 1853 (18) gas became the normal source of heat in laboratories and small gas furnaces and muffles (19,20) were adequate for most laboratory purposes. Electricity, in contrast, was not readily available until into the

twentieth century and even then the position varied from laboratory to laboratory, as illustrated by the use of gas heating for thermogravimetry as late as 1925 (21).

Analysis of these facts suggests that the main causes of delay in the introduction of the wire-wound furnace were the ready availability of laboratory gas furnaces, the familiarity of scientists with gas and possibly the fact that highly accurate temperature control was not then considered essential for many of the operations performed.

**Le Chatelier, Osmond,
Moissan and Charpy**

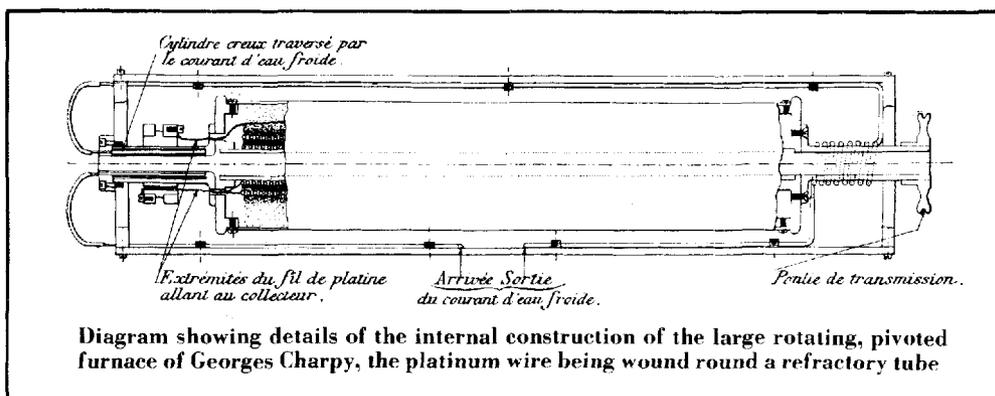
In the late 1880s, however, interest in metallurgy grew, and convenient temperature control became a reality in 1886 when Henry Louis Le Chatelier developed the thermocouple as an accurate temperature-measuring device (22), thereby confounding earlier prognostications (23). At about this period too, Floris Osmond (1849–1912) carried out his extensive



metallurgical investigations (24) and Ferdinand-Frédéric-Henri Moissan (1852–1907) was involved in related studies. It was in this climate that Augustin Georges Albert Charpy entered l'École Polytechnique in 1887 (25,26). Becoming an Assistant in the Chemistry Course in 1889, he gained his doctorate in 1892 and then, not surprisingly as Henri Moissan had presented his papers to the Academy of Sciences from 1891 on (27), he commenced metallurgical studies at the

Laboratoire Central de l'Artillerie de Marine. His first paper on this subject, published in 1893 and dealing with the effect of annealing on the physical properties of brass, contains the comment (28):

“Pour effectuer le recuit on employait un four chauffé par une spirale de platine traversée par un courant électrique, ce qui permet d'obtenir facilement des températures constantes: les températures étaient mesurées avec un pyromètre thermo-électrique Le Chatelier” [that is a thermocouple].



This is the first record in the literature of a wire-wound electric resistance furnace—and the winding was platinum, not the “iron wire” mentioned by Minet (2), whose notes, regrettably, are not always accurate. Berthélemy was thus quite correct in stating in 1947 (26) that “il a construit le premier four à résistance électrique, à éléments de platine, qui nous paraît très simple, mais qui fut et qui reste un outil extrêmement précieux pour les mesures”. For a study of the allotropic forms of iron, in 1894, Charpy used a similar furnace of which he gives more details (29). The platinum wire was wound around a refractory tube 20 cm diameter by 60 cm long, giving a zone 20 cm long in which the temperature, as measured by a thermocouple, remained constant for several hours over the range 500 to 1300°C. The furnace tube was insulated with asbestos, the current was regulated by a rheostat and the furnace was pivoted so that it could be quickly tilted vertically to allow the iron bar being heated to fall into the tempering bath without its temperature falling significantly in transit.

On 25th November 1892 the Council of La Société d'Encouragement pour l'Industrie Nationale offered a grant of 3000 francs for studies on the tempering and annealing of steel to Floris Osmond, who at first accepted but later withdrew as his research had taken a different direction. Thereupon the Council approached Georges Charpy who, with the approval of his superiors at the Laboratoire de la Marine, entered into an agreement with the Society in June 1894. After visiting his laboratory, a Committee of Council, who were most impressed by his research, handed over to Charpy the 3000 francs previously set aside for Osmond (30). On 25th January 1895, Charpy described his work to a meeting of the Society and this was published in their *Bulletin* for June 1895 (31). This remarkable piece of work for such a short period deals not only with the tempering of steel in detail but also describes several platinum-wound electric resistance furnaces and was very highly praised by the Society's assessors, who refer to “la profondeur de ses recherches, la vigueur et la précision de

ses méthodes et l'originalité de ses procédés d'investigation” (30). Apparently Charpy also exhibited one of his furnaces at the meeting on 25th January; as this excited great interest (32), one wonders why it is not referred to in Street's review (1) and how Minet (2) got his facts wrong.

The first illustration of such a furnace and its associated temperature measuring and recording arrangement is reproduced herewith; Charpy describes its construction as follows:

“[Le four] comprend un tube en terre réfractaire de Om,20 de diamètre et 10 centimètres de long. Autour de ce tube, est enroulé un fil de platine de Omm,5 de diamètre recouvert lui-même d'une épaisse couche d'amianté. Le tout est enfermé dans un cylindre métallique porté sur un pied . . . Avec un courant de 6 ampères environ, on arrive rapidement et d'une façon très régulière à une température de 1200° et 1300°”.

A rather more complex furnace was used by Charpy in his studies on the tempering of steel. It consisted of a platinum-wound refractory tube 60 cm long by 2.5 cm diameter insulated with asbestos inside a brass tube sealed with end-pieces. The refractory tube was extended at each end by cooled bronze tubes forming axles set in bearings fixed to the outer frame: one axle had a pulley attached and the other the electrical contacts. This arrangement was rotated by an electric motor attached to the outer frame and again the whole system was pivoted so that it could be rapidly brought to the vertical position, allowing the metal bar sample to drop into the tempering bath. Temperature profiles, obtained using a thermocouple, showed that the sensibly “constant-temperature” region tended to become smaller as the temperature increased up to 1000°C, although, even at this temperature, the variation was less than 10°C over 15 cm in the centre of the tube. Temperature control was by manual adjustment of a rheostat. The winding used consisted of four platinum wires 7 m long and 0.15 mm diameter arranged in parallel; two wires were wound directly on the refractory tube and were covered with a sheet of asbestos on which were wound the other two. In an

appendix to the paper, Charpy gives instructions for constructing a similar furnace 40 to 50 cm long but without the rotating arrangement or water-cooled ends, indicating that this can be used when great temperature precision is not required. In this furnace, constructed for Charpy by M. Jobin, precision instrument maker, Paris:

“Une feuille de cuivre double l'intérieur du tube, et facilite l'égalisation de température . . . Une dérivation montée sur les spires centrales du fil de platine (dans la région où la température est uniforme) communique avec un volt-mètre assez sensible; si l'on connaît l'intensité du courant qui traverse le fil de platine, on a . . . une mesure approximative de la résistance . . . [et] on arrive facilement à évaluer la température à 15° ou 20° près”.

Thus, not only did Charpy construct the first platinum-wound furnace in 1893, but in the next year he arranged it on a pivot so that it could be turned rapidly from the horizontal position to the vertical, and in 1895 he produced a rotating, pivoted version with water-cooled ends that seems complex even today. He also used part of the winding as an inbuilt platinum resistance thermometer for less precise work. Clearly he was an engineer of resource and ingenuity and it is little wonder that, later in 1895, he was awarded a gold medal of the Société d'Encouragement pour l'Industrie Nationale for his efforts (33).

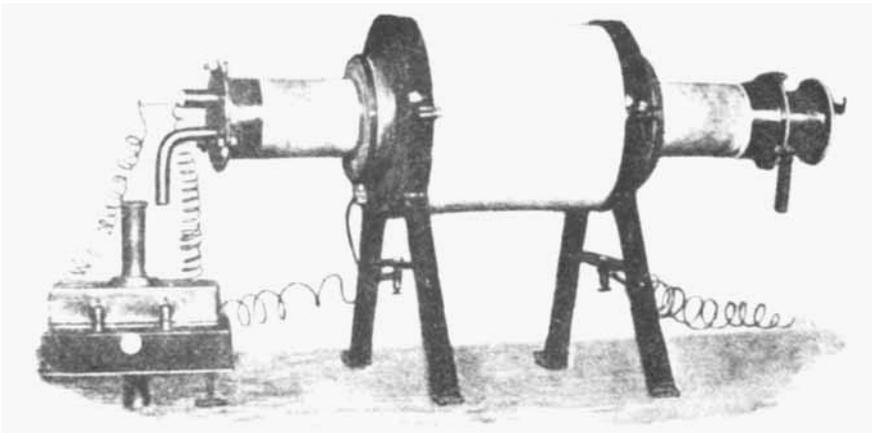
Developments in the Period to 1900

An interesting electric resistance furnace with a spherical cavity, heated by coils of iron wire, rotating on axles and fitted with an inner iron sheath was constructed by William Chandler Roberts-Austen (1843–1902) in 1897 and was used for calibrating thermocouples against an air thermometer (34). Iron wire could not give the temperature stability or reproducibility of platinum—although Le Chatelier, by packing the space around an iron winding with charcoal, claimed long life when used up to 1000°C (35)—but Robert-Austen's furnace was as carefully constructed as Charpy's and was perfectly adequate for the

purpose for which it was made. In 1899, Holburn and Day constructed a tube furnace, also for calibrating thermocouples against an air thermometer, using a winding of nickel wire (36). Base metal windings were thus introduced at an early stage; obviously these were cheaper than platinum, but a contributory factor may have been the difficulty of obtaining pure platinum wire. Many of the papers at that period comment on the fact that specially purified metal must be used for windings and claim that the iridium, which was apparently then a normal impurity, tended to evaporate during heating, contaminating the furnace. Although this seems most unlikely, furnace tubes certainly tended to become conducting after a period of use and poor quality materials gave rise to much trouble. For example, in 1898 a batch of commercial rhodium for the construction of thermocouple wires was found to contain 30 per cent iridium (37) and, up to at least 1908, the quartz present in British porcelain refractory tubes (but not in Berlin porcelain) gave rise to spurious thermal analysis effects (38).

Early Commercial Manufacture

Platinum-wound laboratory resistance furnaces, both tube and muffle, were first produced commercially by the firm of W.C. Heraeus, Hanau, Germany, around 1900. Repeated heating and cooling caused the thin platinum wire spiral on early models to expand and separate in places from the ceramic tube with resultant non-uniformity of temperature distribution and development of local hot-spots where the wire eventually fractured. To overcome this, Heraeus in 1902 developed a furnace using a very thin platinum ribbon (0.007 mm thick) in place of the wire (39). This ribbon required only about one-sixth of the amount of platinum, that is 6 g for a furnace 20 cm long (40). As the coils were laid 1 mm apart there was virtually a complete platinum cylinder around the ceramic tube. Because of their advantages of uniformity of heat supply and long winding life, these furnaces were highly regarded and were extensively used during the



One example of the furnaces supplied by W. C. Heraeus, Hanau, Germany in 1902, the element consisting of thin foil platinum windings. This particular furnace has an extra long tube sealed with mica end-pieces and is fitted with thermocouple and galvanometer for work in controlled atmospheres

early part of this century. An example is illustrated herewith (41).

Heraeus claimed that furnaces with tubes 2.5 cm in diameter could be heated to 1400°C in about 5 minutes and could attain 1700°C without fusion of the winding (39). The refractory material available for the tube, however, became electrically conducting above about 1500°C. Haagn (42) confirmed that, for 6 to 8 hours' operation, the practicable maximum was 1500°C, although the furnace could be heated to 1700°C for a short period without permanent damage.

Later Developments

Despite the ready availability of these furnaces, many laboratories still preferred to make their own, sometimes for financial reasons but sometimes because the sizes available were inappropriate for the work to be performed. For example, in 1902 the Physics Division at the National Physical Laboratory, Teddington, constructed two large electric furnaces for a McLeod, gas thermometer reading up to 1200°C, two large furnaces for thermocouple comparison and two smaller furnaces for miscellaneous work. By 1904 the Thermometry

Department of the Physics Division had 5 wire-resistance furnaces. The winding material is not known with certainty but it would appear to have been platinum or nickel; manganin or eureka wire then being used for low-temperature studies.

Graded windings, to lengthen the uniform-temperature zone, appear to have been first introduced with nickel wire in 1903 (43). In 1904, at the U.S. Geophysical Laboratory, where much early development took place, platinum-iridium windings were placed inside the furnace tube to obtain temperatures 200°C higher than those obtainable with a conventional system (44); in 1908 this arrangement was further improved by using graded windings along with supplementary end-coils to give temperatures varying by not more than 1°C over the central 20 cm of a tube 30 cm long by 6 cm internal diameter (45). Within fifteen years from its introduction, therefore, the wire-wound resistance furnace had developed into a precision tool and its technology was highly advanced.

The usual sources of power for these furnaces were high-capacity accumulators of 70 to 100 volts output, or occasionally dynamos, as

these provided a more reliable output than mains electricity, where this was available (43,44). By 1912, however, local mains supplies were apparently sufficiently stable in some places to be useful (46). Power consumption is only very rarely stated, but seems to have varied enormously—for example, that of the Heraeus range is quoted as 400 to 2000 watts (42), but 3000 and even 4000 watts are mentioned elsewhere (44,47).

Temperature control was normally effected as described above by Charpy (31), that is using visual observation of the galvanometer deflections and manual adjustment of a rheostat. Temperature indicators for use with thermocouples were introduced at an early stage and in the 1900s control at a fixed temperature was effected by using the needle of the indicator as a relay contact. The first automatic control system for continuously increasing temperature seems to have been introduced in 1912 (46); despite the mechanical nature of this, the linearity of the temperature/time curve is impressive.

Replacement of gas furnaces by electric ones was generally slow. Evidence in the literature suggests that by 1906 gas and electricity were fairly equally utilised, whereas by 1909 electricity had probably become more common. Yet the preference for gas was very strong in some countries and gas furnaces remained in use for purposes where electricity would have been more convenient well into the 1920s (21). Moreover, visual temperature observation with manual control persisted until the 1950s (48).

Perhaps it is because of this gradual transition that the history of the platinum-wound electric resistance furnace has been so neglected.

Acknowledgements

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An Historic Platinum Still

Preserved for a great many years in the care of the Society of Chemical Industry in London, a severely damaged small platinum still was recently returned to Johnson Matthey for restoration to something like its original condition. Made for Dr. Rudolph Messel in the 1870s, it was used in the development of his process for making fuming sulphuric acid, or oleum, then much in demand by the growing British dyestuff industry. Messel, born in 1848, had originally come to London as secretary to Professor H. E. Roscoe after studying chemistry in Zürich, Heidelberg and Tübingen, but then joined William Stevens Squire of Dun Squire & Company, this firm later being succeeded by the well known acid manufacturers Spencer Chapman and Messel.

Messel's complicated process involved the heating of sulphuric acid in a platinum still to decompose it into water, sulphur dioxide and oxygen, condensing out the water and then passing the mixed gases in stoichiometric proportions—a concept later shown to be quite unnecessary—over finely divided platinum contained in a heated platinum tube to form sulphur trioxide. This was then dissolved in sulphuric acid to give oleum. This replaced the old-established lead chamber process and had the further advantage that it eliminated catalyst poisoning from the raw materials he had previously tried in his attempts to operate the



contact process originally devised by Peregrine Phillips in 1831.

A British patent, No. 3278 of 1875, was filed for the process by Messel's partner Squire, and on April 20th 1876 a short paper was read to the Chemical Society, again by Squire, "On the Manufacture of Sulphuric Anhydride".

Messel became managing director of the company in 1878, holding this position until his retirement in 1915, by which time some thousand tons a week of oleum were being produced by the process he had originally developed with this experimental still.