

A History of Iridium

OVERCOMING THE DIFFICULTIES OF MELTING AND FABRICATION

By L. B. Hunt

The Johnson Matthey Group

The use in unmanned space craft of iridium to encapsulate the radio-isotope thermoelectric generators, where temperatures of up to 2000°C have to be withstood over several years of operation, with possible impact velocities of 90 metres per second, has focused greater attention on the remarkable properties of this member of the platinum group of metals. But these same properties of very high melting point and great mechanical strength have been the cause of difficulties in its melting and fabrication over a long period of years. How these problems were tackled and eventually overcome is described in this article.

One of the less well-known members of the platinum group, iridium possesses quite remarkable chemical and physical properties. It is not only the most resistant of all metals to corrosion, insoluble in all mineral acids including aqua regia and unattacked by other molten metals or by silicates at high temperatures, but has a very high melting point and is the only metal to maintain good mechanical properties in air at temperatures above 1600°C. Its great stability can be gauged from its physical properties, outlined in the Table. Its high modulus of elasticity and modulus of rigidity, together with the very low figure for Poisson's Ratio (the relationship of longitudinal to lateral strain), clearly indicate the high degree of stiffness and resistance to deformation that have rendered its fabrication into useful components a matter of great difficulty over the long period since its discovery, but despite these limitations—and despite its high cost—a number of applications have developed in more recent years where mechanical strength is an essential factor in some of the extremely severe conditions nowadays encountered in modern technology. But before success could be achieved in either the melting or the fabrication of iridium there lies a long struggle to be recorded.

For some fifty years after the discovery of platinum in South America and the early investigations of its properties by a number of English, French, German and Swedish scientists, it was not realised that the native platinum they were examining also contained other elements. The first to recognise that a small insoluble residue survived the dissolution of native platinum in aqua regia was the French chemist Joseph Louis Proust, working for a time in Madrid under the patronage of King Carlos IV, but he failed to grasp that other members of the platinum group were present and in 1799 described his black residue as "Nothing less than graphite or plumbago" (1).

Discovery and Characterisation

It was this black residue that greatly intrigued Smithson Tennant shortly after his entry into partnership with William Hyde Wollaston in 1800. The object of this famous enterprise was the production and marketing of platinum in quantity and it was soon decided that while Wollaston should pursue the study of the aqua regia solution of native platinum (so discovering palladium and rhodium) Tennant should concentrate on the insoluble matter. By the summer of 1803 he had begun his investigation, fortunately mentioning to Sir Joseph

Physical and Mechanical Properties of Iridium	
Atomic number	77
Atomic weight	192.22
Crystal structure	f.c.c.
Lattice constant, Å	3.8392
Melting point, °C	2443
Specific gravity (20°C)	22.65
Latent heat of fusion, J/g.	117
Specific heat (0–100°C), J/g. °C	0.134
Thermal conductivity (0–100°C), J/cm.s. °C.	1.48
Vapour pressure at 1500°C, torr	10 ⁻⁸
Specific electrical resistivity at 0°C, microhm. cm.	4.71
Temperature coefficient of electrical resistivity (0–100°C), per °C	4.27 × 10 ⁻³
Electrical conductivity, % IACS	36.6
Thermal coefficient of linear expansion (0–100°C), per °C.	6.8 × 10 ⁻⁶
Hardness, HV (annealed), kg/mm ²	200–240
Modulus of elasticity, E, MN/m ²	516 × 10 ³
Modulus of rigidity, G, MN/m ²	210 × 10 ³
Bulk modulus, K, MN/m ²	371 × 10 ³
Poisson's ratio.	0.26
Typical tensile strength (annealed), 20°C, MN/m ²	490–740

Banks, President of the Royal Society, his observation that the black powder “did not, as was generally believed, consist chiefly of plumbago but contained some unknown metallic ingredients”. Fortunately, because work on the same problem was being undertaken almost simultaneously in Paris, first by the director of the École des Mines, Hippolyte Victor Collet-Descotils, and a little later by Antoine François de Fourcroy and Nicolas Louis Vauquelin at the Muséum d’Histoire Naturelle (2, 3). All three found that the black residue contained a hitherto unknown element but they neither isolated it nor gave it a name.

Smithson Tennant, who had the advantage of a much greater amount of residue from Wollaston’s work on platinum, realised that while the Frenchmen suspected the presence of one new metal in the black powder, there were in fact two, namely osmium and iridium, and on 21st June 1804 he presented his paper, a masterpiece of clarity and conciseness, to the Royal Society. For this he was awarded the Copley Medal for that year, the highest honour in their gift, while a little later the French workers fully accepted the priority of his discoveries (4).

Tennant’s comment on iridium was something of a forecast of the difficulties to come:

“It appeared of a white colour, and was not capable of being melted by any degree of heat I could apply”.

XVI. *On two Metals, found in the black Powder remaining after the Solution of Platina.* By Smithson Tennant, Esq. F. R. S.

Read June 21, 1804.

UPON making some experiments, last summer, on the black powder which remains after the solution of platina, I observed that it did not, as was generally believed, consist chiefly of plumbago, but contained some unknown metallic ingredients. Intending to repeat my experiments with more attention during the winter, I mentioned the result of them to Sir JOSEPH BANKS, together with my intention of communicating to the Royal Society, my examination of this substance, as soon as it should appear in any degree satisfactory. Two memoirs were afterwards published in France, on the same subject; one of them by M. DESCOTILS, and the other by Messrs. VAUQUELIN and FOURCROY. M. DESCOTILS chiefly directs his attention to the effects produced by this substance on the solutions of platina. He remarks, that a small portion of it is always taken up by nitro-muriatic acid, during its action on platina; and, principally from the observations he is thence enabled to make, he infers, that it contains a new metal, which, among other properties, has that of giving a deep red colour to the precipitates of platina.

M. VAUQUELIN attempted a more direct analysis of the substance, and obtained from it the same metal as that discovered by M. DESCOTILS. But neither of these chemists have observed,

Early in the course of their famous partnership designed to yield malleable platinum in commercial quantities, it was agreed that while Wollaston should pursue the investigation of the portion of the native metal soluble in aqua regia, Smithson Tennant would study the insoluble portion. The result was the discovery of both iridium and osmium. This shows the opening page of Tennant’s paper given to the Royal Society in 1804

Fourcroy and Vauquelin remarked similarly:

“This metal has appeared to us to be of a greyish white colour, difficult of fusion, of oxydation, and of solution in acid”. (5)

Some years later in 1814 Vauquelin wrote of iridium:

“It appears to be brittle and consequently hard. I cannot give its specific gravity because I have not yet been able to melt it completely”. (6)

First Attempts at Melting

But by this time a new and powerful method of obtaining a high temperature was becoming available. Following Volta's news of his discovery of the electric pile in 1800, a number of British scientists turned their attention to the construction of large batteries to yield a high voltage. Foremost among them was John George Children, who began to build large batteries in his private laboratory at Ferox Hall near Tonbridge in Kent in about 1806, while as is well known Humphry Davy in October 1807 discovered potassium and sodium by means of a discharge from a battery consisting of 250 plates of zinc and of copper each 6 inches by 4 inches. Davy was friendly with Children and he was present at Tonbridge when in August 1808 a piece of platinum wire was successfully melted by the use of a voltaic battery larger and more powerful than any hitherto constructed (7). This success encouraged Davy to build a still larger battery at the Royal Institution, but Children went on to construct an even larger model in 1813.

In that year Smithson Tennant had been appointed Professor of Chemistry at Cambridge, and while giving his mind to the preparation of his lectures in June he wrote to Berzelius, whose friendship with him arose chiefly from a visit the Swedish chemist had paid to Tennant's estate in Somerset in 1812, about the choice of a “galvanic machine”. The letter goes on:

“On the 2nd of next month I am going to Mr. Children's house to see a machine of which the plates are 16 square feet. The object is only to produce a very high temperature and I shall try its effect on iridium, which a flame even intensified with oxygen will not even touch”. (8)

On that historic occasion no less than thirty-eight distinguished scientists, including Davy, Wollaston, Hatchett, William Allen, W. H. Pepys, W. T. Brande, Henry Warburton and William Babington, dined together at Ferox Hall, spent the night there and in the morning witnessed the melting of a small piece of iridium. This was achieved by means of “the greatest galvanic battery that has ever been constructed”, consisting of 20 pairs of copper and zinc plates, each 6 feet long by 2 feet 8 inches, suspended from the ceiling and then lowered into an enormous tank containing 945 gallons of dilute nitric and sulphuric acids (9).

Children was not yet satisfied, however, that he had really achieved the melting of a pure specimen of iridium, and early in 1815 he again set about its fusion, holding the metal in a cavity in a piece of charcoal floating on mercury. He had now, acting on a suggestion made by Wollaston, added a second copper plate to each cell so that each zinc plate was now faced by two of copper. This materially increased the power of the battery, and he then recorded in his paper to the Royal Society, read on 15th June, 1815:

“Exp. 8. Pure iridium; fused into an imperfect globule, not quite free from small cavities and weighing 7.1 grams. The metal is white, very brilliant and in its present state its specific gravity is 18.68, which must be much too low on account of the porous state of the globule”. (10)

This great occasion was greatly saddened for those present, however, by the absence of Smithson Tennant who, returning from a five-month tour of France in the February, had met with a fatal accident while on a riding excursion near Boulogne.

No further attempts to melt iridium are recorded for quite a long period, until, in fact 1842, when Robert Hare, Professor of Chemistry in the University of Pennsylvania, reported his success in this direction to the American Philosophical Society. Forty years earlier Hare had devised a hydrogen-oxygen blowpipe with which he had melted platinum, for the first time in reasonable quantity, and over the years he had gradually brought about

John George Children
1777–1852

The enthusiasm for building large electric batteries following Volta's announcement of his discovery infected Children as well as Humphry Davy. In 1831 no less than thirty-eight scientists, including Tennant, were entertained by Children at his home near Tonbridge and witnessed the first melting of a small globule of iridium by the discharge of a colossal battery he had constructed

Photograph by courtesy of the Wellcome Institute Library, London



improvements in the apparatus. His paper opens:

“This communication respects mainly my success in fusing both iridium and rhodium, neither of which in a *state of purity* had been previously fused. It may be supposed that the globule of iridium, obtained by Children's colossal battery, forms an exception; but the low specific gravity, and porosity, of that globule, may justify a belief that it was not pure; and at any rate, the means employed were of a nature not to be at command for the repetition of the process—so that iridium might as well be *infusible*, as to be *fusible only by such a battery*”. (11)

Unlike Children's specimen, Hare's iridium gave specific gravity values of 21.83 and 21.78, amply confirming his claim to high purity. Of its physical characteristics he wrote:

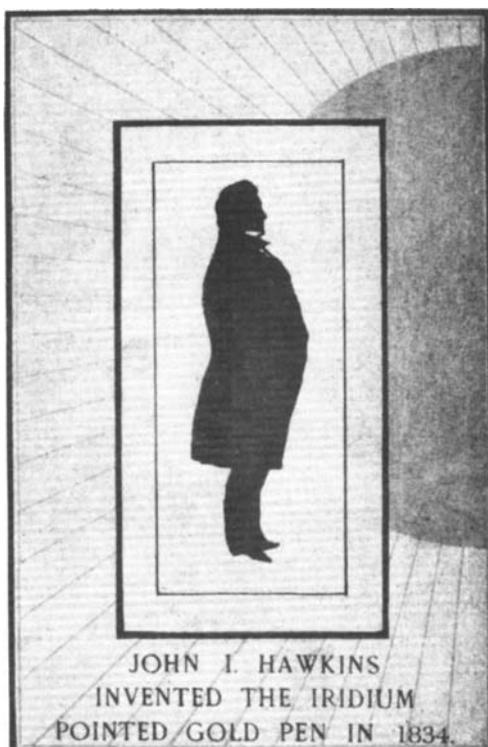
“Although as hard as untempered steel, it is somewhat sectile, since when split by means of a cold chisel, the edge penetrated about the eighth of an inch before a division was effected. By light hammering a corner was flattened without fracture, although under heavier blows the mass cracked. I infer that although nearly unmalleable and very hard, iridium may be wrought in the lathe.”

Now Hare had obtained some of his supplies of iridium from Johnson and Cock (as Johnson Matthey was known from 1837 until 1845) and although no published paper exists to support the contention, it is virtually certain that, as will be seen a little later, as early as 1837 iridium had been melted in the Hatton Garden

refinery. In any case by 1846, when Michael Faraday was studying the magnetic properties of a wide range of metals and compounds, his records contain the following entry:

“2368. Iridium.—Mr. Johnson supplied me with several preparations of iridium. The oxide, chloride and ammonio-chloride were magnetic; and so was a sample of the metal. One specimen of the metal, which seemed to be very pure, was scarcely at all magnetic, and on the whole I incline to believe that iridium does not stand in the magnetic class”. (12)

But the melting of iridium in any appreciable quantity had to await the work of Henri Sainte-Claire Deville and his partner Jules Henri Debray in Paris in the late 1850s. Their design of a furnace constructed from two cylindrical, hollowed out blocks of lime and fired by coal-gas and oxygen was first used to melt platinum on a large scale in 1857 (13), and the British rights in their patent were at once acquired by Johnson Matthey. A little later the Russian government, impressed with their new procedure for refining native platinum, sponsored Deville and Debray by setting up for them



**John Isaac Hawkins
1772–1865**

This pictorial tribute appears in a small book, "The Everlasting Gold Pen and How it is Made", published in 1892 by F. Mordan & Co. A native of Taunton in Somerset he spent his early years in America, inventing among other things the upright piano. Returning to England in 1803 he followed a successful career as a consulting engineer and patent agent, being elected a Member of the Institution of Civil Engineers in 1824. He was the first to introduce the iridium pen point

Illustration by courtesy of Mr. Michael Finlay

larger furnaces and oxygen plants. From the Russian Imperial Mint they were supplied, in February 1860, with some eight kilogrammes of crude iridium-containing material among other residues, and "after three and a half months of incessant work" they were able to return large quantities of platinum ingots and sheet, together with an ingot of iridium weighing 1.805 kilogramme (14). To achieve this fusion they had to consume, for one kilogramme of iridium, more than 300 litres of oxygen and

pure hydrogen, coal gas being useless for their purpose.

Johnson Matthey similarly put the process into effect, and were able to show in the International Exhibition in London in 1862 a melted ingot of iridium among their numerous other exhibits of the platinum metals. The report of the Juries contains the following item:

"An ingot of iridium, hitherto regarded as infusible, now melted into a lump of twenty seven and three quarter ounces."

In 1879 George Matthey presented a detailed account of the refining of the platinum metals in the course of a paper to the Royal Society (15) of which he was shortly afterwards elected a Fellow. His procedure for the separation, refining and then melting of iridium was "an operation of extreme delicacy" and much too lengthy for reproduction here, but some measure of his achievement may be gained from his statement that while the metal should, if perfectly pure, possess a specific gravity of 22.39, the highest value he had yet been able to attain was 22.38.

The Earliest Applications

Turning now to the earliest applications of iridium we have to retrace our steps to the 1830s and to the ingenuity of an extraordinarily versatile English engineer. This was John Isaac Hawkins, born in Taunton in Somerset in 1772 the son of a watch and clockmaker. At an early age he emigrated to America to study at Jersey College in Pennsylvania—the institution which became Princeton University in 1896—and then turned his mind to mechanical and chemical engineering and also to the improvement of musical instruments, filing a patent in 1800 for the invention of the upright piano, a development that brought him to the attention of President Jefferson. In 1803 he returned to England in order to claim a legacy and here he embarked on a successful career as a patent agent and consultant, his further inventions including a pantograph and then, together with one Sampson Mordan, the use of small particles of "diamond, ruby or other hard substances" as pen points, the nibs then being made of horn or

tortoise-shell (16). The advent of the gold nib was also due to Hawkins, who spent many years seeking a suitable material to make a fine and hard point. Then in 1833 he learned that some twelve years earlier Wollaston had provided samples of rhodium and of the native alloy of iridium and osmium to Thomas Charles Robinson, the famous maker of chemical balances and drawing instruments, requesting him to prepare pens with each material. (Robinson, of Devonshire Street, off Portland Place in London, lived quite close to Wollaston and had provided him with a balance incorporating improvements proposed by the purchaser.) Hawkins' information was that Robinson had duly made a few pens with rhodium points (actually an alloy of rhodium and tin) but had returned the iridium alloy as too hard to be wrought into pen points. This stimulated him to investigate further, and by 1834 he had succeeded in producing the first gold pen, this tipped with two specks of iridium. Wollaston had died in 1828, and Hawkins had therefore to seek other sources of iridium.

In an account of his early efforts, given in 1875 by an American pen manufacturer, John Foley, who had received it directly from the former, the following passage occurs:

"In the early stage of the business, namely in March, 1834 Mr. Hawkins says, 'I procured the native alloy of Iridium from Mr. Johnson of Hatton Garden, London, who allowed me to select from his small stock of a few ounces such particles as suited my purpose, at thirty shillings an ounce. Mr. Johnson continued to supply me at that price till July, 1835, when I had picked out all that would suit and he said that he did not expect any more for some time. I had then only enough to make three dozen Pens, and knew not where to procure more. I, therefore, went to the British Association for the Advancement of Science, which met at Dublin on the 10th of August, 1835, to inquire of the great Chemists of the time, expected to be there assembled, where I could be supplied with the precious material. On asking Dr. Dalton of Manchester, Dr. Thomas Thomson of Glasgow, Dr. Daubeny of Oxford and many other eminent Chemists, present at the meeting, where I could procure the substance, each, without communicating with any of the others, answered that I could obtain it of Mr. Johnson, Hatton Garden, London.' " (17)

Hawkins sold the secret of his technique to an American in 1836, but manufacture of the iridium-tipped pens was continued by Francis Mordan.

In the United States several manufacturers entered the gold pen business, but the one that is remarkable in our present connection was G. W. Sheppard of Detroit who moved in about 1842 to Cincinnati. Some ten years or so later he engaged a boy whom he had noticed working in a neighbouring drug store, one John Holland. Sheppard carried out many experiments with the oxy-hydrogen blowpipe and with electric batteries in attempts to melt iridium but without success. In 1858 Holland became a partner in the business and then, on Sheppard's death in 1862, the sole owner and manager (18). He continued the experimental work with no greater degree of success until 1880 or thereabouts, when he was faced with a demand for much larger pieces of the iridium alloy for a new type of pen. Simultaneously he observed that a specimen of iron ore very high in phosphorus content melted much more readily than other samples and he quickly heated a small piece of iridium to a white heat and then threw into the crucible a piece of phosphorus. When the fumes had cleared away he poured out the contents of the crucible and found "to his joy and amazement a white, compact, hard metal" (19).

For use as pen points this material was perfectly satisfactory, but if other uses were to be found for the metal it was obviously necessary to remove the phosphorus. It was at this point that Holland invoked the help and advice of William Lofland Dudley, Professor of Chemistry at the local medical college in Cincinnati. Dudley tackled this problem with great enthusiasm, quickly removing the phosphorus by repeated heatings with lime, but equally quickly developing an interest in finding new applications for iridium. This enthusiasm led to the formation of the American Iridium Company based upon Holland's activities but with Professor Dudley as general manager.

The activities of the company received quite a considerable amount of publicity. John

William Lofland Dudley
1859–1914

Professor of Chemistry in Vanderbilt University in Nashville, Tennessee, from 1886 until his death, Dudley had spent the previous five years teaching chemistry at the Medical College in Cincinnati and it was here that his help and advice were sought by John Holland who had succeeded in melting iridium by adding phosphorus to the charge in the crucible. Dudley plunged into the the metallurgy of iridium with great enthusiasm and contributed a number of papers dealing with its properties and uses

Photograph by courtesy of the Photographic Archives, Vanderbilt University



Chatelier in 1886, platinum versus rhodium-platinum, but clearly this could not be employed to measure temperatures higher than those approaching the melting point of platinum. One of the great pioneers in the measurement of such temperatures was Sir William Chandler Roberts-Austen, Chemist to the Royal Mint and Professor of Metallurgy at the Royal School of Mines, and in the course of one of his numerous lectures, given to the Royal Institution in 1892, he referred to other metals with higher melting points being available:

“Thus iridium will only just melt in the flame produced by the combustion of pure and dry hydrogen and oxygen. By the kindness of Mr. Edward Matthey, a thin rod of iridium has been prepared with much labour, and it can be used as a thermo-junction with a similar rod of iridium alloyed with 10 per cent of platinum. The junction may be readily melted in the electric arc, and by this means a temperature may be registered which careful laboratory experiments show to be close to 2000°C”. (24)

This problem of measuring very high temperatures was taken up a few years later by Wilhelm Carl Heraeus, the founder of the well-

known firm in Hanau. In the closing years of his life he devised an electrical resistance furnace in which the main element was a tube of pure iridium, this furnace being used among others by Nernst to determine high temperatures in 1903. It was also supplied to the Physikalisch-Technische Reichsanstalt for the same purpose, together with thermocouples consisting of pure iridium against 10 per cent ruthenium-iridium (25). This combination was found, over the ensuing years, to require frequent calibration because of the oxidation of ruthenium, and numerous couples were tried based upon combinations of tungsten, molybdenum and tantalum, all of which are of course subject to severe oxidation. Then, in 1933 Otto Feussner, also of the Heraeus company, carried out a thorough investigation of the iridium-rhodium system and proposed a thermocouple of pure iridium against 40 per cent rhodium-iridium, thus enabling temperatures up to 2000°C to be measured in air (26). This combination is still in use at the present time.

Until recently the largest area of growth in

the usage of iridium has been for crucibles for growing oxide single crystals for use in computer memory devices and in solid state lasers. The crystals, such as gadolinium gallium garnet and yttrium gallium garnet, are grown by melting pre-sintered charges of mixed oxides under oxidising conditions at temperatures up to 2100°C. However, long before such devices could be imagined or foreseen, one distinguished chemist had discovered the valuable properties of iridium crucibles. This was Sir William Crookes, the founder of *The Chemical News* and President of the Royal Society from 1913 to 1916, who contributed a paper to that society in 1908, "On the Use of Iridium Crucibles in Chemical Operations" (27). The paper opened:

"I should like to draw the attention of chemists to the great advantages of using crucibles of pure iridium instead of platinum in laboratory work. Through the kindness of Messrs. Johnson and Matthey I have had an opportunity of experimenting with crucibles of wrought iridium, and have used one for several months in the usual operations of quantitative analysis in my laboratory. Iridium is as hard as steel, and the crucible is almost unaffected by any mechanical treatment that can reasonably be applied to it."

Crookes, well in advance of his time, went on to report that his iridium crucibles, one of which still survives in the company's possession, were resistant to the fusion of many fluxes, including caustic soda, and were un-

attacked by molten lead, zinc, nickel, iron and gold.

These crucibles were lovingly prepared by Henry Andrew Kent, a man of near genius in things mechanical, who had joined the company in 1875 and who had worked closely with John Sellon in developing the fabrication side of the business. Nowadays the technique of argon arc melting followed by hot rolling makes it possible to produce ductile and malleable iridium in sheet form with a very minimum of impurities. Such sheet can then readily be formed into crucibles of capacities up to 4 or 5 litres.

But the outstanding application of recent years, and one that could never have been envisaged in the wildest dreams of those earlier workers, had to await the development of both nuclear physics and space travel. To provide an independent but reliable source of electric power for space missions radioisotope thermoelectric generators have been used since the Voyager probes were launched in 1977, the fuel consisting of plutonium-238 dioxide connected to thermocouples. The fuel is in the form of spheres and these are encapsulated in iridium to provide a secure shield of exceptionally high melting point and great strength (28). The possibility of this spectacular technique finding other applications is a matter for the future.



Iridium crucibles are now greatly in demand for the growing of oxide single crystals at temperatures around 2000°C but as long ago as 1908 a number of small crucibles were made in iridium by Johnson Matthey at the request of Sir William Crookes. One of these crucibles still survives in the company's possession

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Platinum in Early Instrumentation

The History and Preservation of Chemical Instrumentation

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An unusual and intriguing compilation of papers presented to the History of Chemistry Symposium of the American Chemical Society in September 1985, this publication first emphasises the importance of instruments in chemical industry as well as in the laboratory. The eighteen papers, only two of which have to do with platinum, then deal with instruments ranging from the very simple and oldest—blow-pipe analysis—all the way to the most modern, even robotics. In the former case a long paper by W. B. Jensen of Rochester, New York, rightly describes Johan Gottlieb Gahn as the supreme master of blowpipe analysis and refers to his introduction of platinum wire to replace a gold or silver spoon at a time when platinum was only just becoming available. One of the most fascinating contributions comes from John Burnett, then of the Science Museum,

London, but now with the Royal Scottish Museum, on "The Use of New Materials in the Manufacture of Scientific Instruments, c. 1800-c. 1920". Among many metals and alloys developed or used for specific purposes, the author mentions Seebeck's discovery of thermoelectricity in 1821, this leading gradually on to the introduction of the platinum: rhodium-platinum thermocouple by Henri Le Chatelier just a hundred years ago. The first attempt to make a platinum resistance thermometer was made by Sir William Siemens in 1871, but its successful use had to await the researches of Hugh Longbourne Callendar in the Cavendish Laboratory in the 1880s.

The other papers discuss a wide range of instruments, and the volume forms a tribute to the skill and ingenuity of instrument makers past and present.

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