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Birmingham Section.

Meeting held at Birmingham University, on Thursday,
March 5th, 1914.

MR. H. T. PINNOCK IN THE CHAIR.

A CHEAP FORM OF ROTATING CATHODE AND ANODE FOR RAPID ELECTROLYTIC ANALYSIS.

BY ERNEST A. LEWIS.

When the rapid method of electrolytic analysis was introduced the apparatus usually contained from 20 to 40 grms. of platinum. I believe that a rotating anode containing about 5 grms. of platinum and made largely of glass is about the smallest weight of platinum recommended. The continuous advance in the price of platinum makes it necessary for technical work to use cheaper metals if possible, and to reduce the weight of platinum in the anode to a minimum. The use of silver and amalgamated brass wire gauze cathodes has been recommended, especially for the assay of zinc. The rotating cathode which I use for zinc determinations is made of copper gauze, 20 meshes to the inch, supported by stiff copper wire which projects from the side and helps to cause vigorous circulation of the liquid. The anode is made of $\frac{1}{4}$ in. glass rod, bent to the shape shown in the diagram, 14 inches of platinum wire of the size 1 grain equals 1 inch, are wound round the glass as shown. The platinum wire is connected to some electrical D.C.C. bell wire and the joint is made perfect by a small piece of platinum foil being wrapped round the joint, which is then wrapped round with adhesive electrical tape. The total weight of platinum is about 1 gm. The shape of the anode causes the liquid to thoroughly mix. The apparatus has been used successfully for determining zinc in alloys after the removal of copper, tin and lead. The current used being 2 amperes in a caustic soda solution.

If an ammoniacal solution or acetic acid solution is used the cathode must be of silver or the whole of it must be silver plated.

The apparatus has also been used for determining copper rapidly from a solution containing about 6 c.c. of strong nitric acid and 10 c.c. of strong sulphuric acid in about 150 c.c. of solution; using 3 amperes and heating to about 70° C. before putting in the copper cathode 0.5 gm. of copper is deposited in about 1 hour. It may seem rather unscientific to use copper cathodes to determine copper, but in practice it is quite satisfactory; if the solution is diluted to about 300 c.c. by washing in, about $\frac{1}{2}$ hour before the end, with cold water, the cathode can be rapidly lifted out and washed without any risk of re-solution; of course the liquid can be syphoned off if preferred, but this complicates the operation. After the cathode is weighed it can be used again for 4 or 5 determinations, when the deposited copper can be carefully dissolved off in dilute nitric acid. It is, of course, not possible to ignite a copper cathode, but it is not necessary. The copper cathodes are easy to make, they cost less than 6d. in material, and the anode only costs about 8s. The anode will safely carry a current of 3 amperes.

I endeavoured to plate a cathode with platinum, but the experiment was not successful, owing to the ease with which platinum is deposited on copper or silver by simple immersion. It is my intention to make a silver cathode and hope to be able to plate it with a thin coating of gold, and then to deposit a thin layer of platinum on the gold. If this is successful the silver cathode will be to all intents and purposes equal to a platinum cathode, and at a fraction of the cost.

It is also possible to use the current direct from the Corporation mains, without the use of accumulators. An 8 lamp accumulator charging board is used, and by the use of 8, 10, and 32 C.P. carbon lamps, giving $\frac{1}{2}$, $\frac{2}{3}$ and $\frac{3}{4}$ ampere respectively, a current varying from $\frac{1}{4}$ ampere to

4 amperes can be obtained. The whole apparatus, including motor, cost about £5.

DISCUSSION.

MR. J. E. STACEY JONES said he had used almost identical cathodes, with the exception that the gauze was replaced by plain foil. He had used brass sheet cathodes, about 32 or 34 standard gauge, and although he had used them chiefly for electrolysis in cyanide solutions, with gold, silver, and other metals, he saw no reason why they should not be used in ordinary laboratory work. Such cathodes were effective and had the advantage that they could be made easily and quickly. The brass foil required little cleaning and retained its brightness. Gold cathodes were effective but not gold anodes, because they would be ineffective if it were necessary to remove the deposit with *aqua regia*. In cathodes gold sheet of 15 c., of about platinum thickness, was cheap and serviceable. Copper could be removed by nitric acid. The objection with gold, however, was that one could not adopt ignition because 15 c. melted at a low temperature. Still a superior 14-carat gold cathode could be obtained for about £1, as compared with £10 for a platinum cathode. The method of obtaining electricity from the Corporation mains by carbon lamps might be convenient, but it was highly expensive.

London Section.

Meeting held at Burlington House, on March 2nd, 1914.

MR. E. GRANT HOOPER IN THE CHAIR.

AN APPLICATION OF CALCIUM CARBIDE IN THE FORMATION OF ALLOYS.

BY W. R. HODGKINSON, PH.D.

THE powerful "reducing" action exerted by acetylene on some salts has suggested that calcium carbide would under suitable conditions be useful in some metallurgical

processes. In some early experiments a mixture of carbide and borax, or carbide and sodium chloride was found to be a most efficient de-oxidising flux for copper and some copper alloys—as bronzes—cupro-nickel, &c., perfectly bubble-free castings being obtained. About one ounce of carbide to the usual amount of borax, was quite sufficient for each 10 lb. pot of metal when added before the last stirring. With bronzes, bronzes, or cupro-nickel, a good time to add the carbide-borax was found to be at the first melt when the last portions of copper, &c., were being added.

A considerable number of metallic oxides are reduced to the metallic state by carbide, but unless the metal is very fusible or a large amount of flux—borax—sodium or magnesium or barium chlorides—be employed, the infusible calcium oxide and any separated carbon or free carbide hold up the metal in shots or globules.

Attempts to reduce some of the rarer oxides, as those of cerium, zirconium, &c., and form alloys with copper failed at ordinary coke-furnace temperatures.

It appeared probable, however, that the halides of these rarer metals might be reduced by carbide which would at the same time form a calcium halide capable of acting as a flux. Trials with copper, lead, silver, zinc, and other metallic halides proved that, at suitable temperatures, reduction was complete and the calcium halide (chloride) formed a fairly efficient flux but the separation of the metal was retarded by the liberated carbon. The addition of salt, barium chloride, or borax (none of which has any action on carbide) overcame this difficulty.

The liberated carbon sometimes takes the graphitic form. Experiments with cerium chloride (CeCl_2), carbide, and metallic copper showed that cerium was reduced and taken up by the copper, alloys containing 6 per cent. and more of cerium being obtained.

It may here be noted that a convenient method of obtaining metallic chlorides in a state ready for further metallurgical work is to drop a mixture of metallic oxide and ammonium chloride into a red hot crucible, in small portions at a time. A very slight excess only of the ammonium chloride is necessary. In most cases the chloride is obtained in the melted state.

Two methods have been employed for obtaining alloys; one in which the main constituent—say copper—was melted and a mixture of the halide of the alloying metal and carbide then added; and, a better plan, where both, or all, metals for the alloy were in the form of halides roughly mixed with the calculated amount of carbide and in the case of low melting point metals some additional flux—as borax or salt. For alloys of high melting point, such as nickel and manganese, the carbide alone acted perfectly.

The chlorides and fluorides of most of the common metals react energetically with carbide provided the starting temperature is high enough—somewhat like the aluminium thermite reaction. As far as has been ascertained, the best starting temperature is one a little higher than the melting point of calcium chloride or other halide employed. The results have certainly been better where the carbide—metallic halide mixture has been introduced in successive moderate quantities into an already hot crucible than by slowly heating up a cold charge.

Employing this method—the hot crucible—no difficulty has been found in making copper—manganese or copper—nickel of almost any desired content of Mn or Ni, using manganese chloride or nickel chloride and either copper turnings or copper chloride. Similarly with nickel—manganese, or cobalt—manganese, both metals as chlorides.

Tantalum, titanium, zirconium, and other metals which form salts of the types, K_2TaF_6 or K_2TiF_6 , have been reduced and alloyed with copper. From one product containing above 20 per cent. Ta most of the tantalum segregated out during cooling and was left as a metallic powder on solution of the alloy in dilute acid.

From manganese chloride alloys have been obtained with tin, bismuth, antimony, and lead, employing these metals in the metallic state. The first product with lead, using the metal, contained 5.6 per cent. Mn: using PbCl_2 , higher proportions of manganese were obtained. (These products from the microstructure seem to be suspensions of metallic manganese in lead.)

The tin and bismuth “mixtures” or solutions of manganese are only so far interesting that with more than 10 per cent. Mn they show decided magnetic properties.*

In cases where iron and aluminium free alloys are required the carbide reduction may be of value. Only in the cases of nickel, cobalt and iron with manganese did there appear to be any carbon taken up. A manganese-cobalt alloy for instance, contained, Mn 38. Co. 59 and graphite 3 per cent.

The method and some of the products are being further investigated.

Specimens of alloys, such as Cu-Mn, Cu-Ce, Cu-Ta, Cu-Ti, Bi-Mn, Sn-Mn, Sb-Mn, Co-Mn, Ni-Mn, prepared by the process, and having in most cases atomic proportions of each element, and micro-photographs, were shown at the meeting.

I am indebted to Capts. Hunter and Macrae for analysis and other help.

DISCUSSION.

Mr. E. GRANT HOOPER said Professor Hodgkinson had struck out a new line in this utilization of calcium carbide in the preparation of alloys. He thought the paper introduced an interesting and he trusted an important departure in connection with the preparation of alloys.

Professor HODGKINSON said he was reserving an account of the properties of some of the alloys. He only spoke of the method because he thought it would be useful, and he was sure there was room for its development. There was a difficulty. Some of the halides were volatile, necessitating a careful adjustment of temperatures, but he thought it was possible in most cases to arrange matters and obtain the product with little loss.

Meeting held at Burlington House, on Monday April 6th, 1914.

PROF. W. R. HODGKINSON IN THE CHAIR.

THE VISCOSITY OF RUBBER SOLUTIONS:

BY R. GAUNT, M.Sc., PH.D.

(Scientific and Technical Department, Imperial Institute).

Considerable interest attaches to the viscosity of rubber solutions, not only because they are colloidal solutions, but also on account of the suggestion put forward first by Axelrod¹ and afterwards by Schidrowitz and Goldsborough² that the viscosity of a rubber solution stands in intimate relationship with the quality and technical value of the rubber.

Schidrowitz³ regards the so-called “nerve” of rubber as due to three factors:—(a) Mechanical structure as indicated by the film structure of fine hard Para, and brought about by the method of conglutination; (b) physical structure or colloidal aggregation of the rubber particles; (c) chemical structure or molecular complexity.

The mechanical structure of crude rubber is largely broken down in the washing and mixing operations, and is therefore only a small factor in the quality of manufactured rubber. Schidrowitz suggests that viscosity measurements connote the physical and chemical structure of rubber although the exact relation is uncertain. He states⁴ that within the same species viscosity measurements are a means of determining strength and vulcanising capacity; comparing species with species, however, this does not hold directly.

Beadle and Stevens⁵ however, found that although some relationships can be detected, the general results of viscosity determinations are not in accordance with those obtained by vulcanisation.

* Their properties will be described in a further communication.

¹ Gummi Zeitung, 1905, 10, 1053, and 20, 105.

² This Journal, 1909, 22, 3, “The Rubber Industry,” p. 260. (The International Rubber and Allied Trades Exhibition, Ltd., 1911).

³ “Rubber,” p. 163 (Methuen and Co., 1911).

⁴ “Rubber,” p. 165.

⁵ “The Rubber Industry,” pp. 260 and 270.