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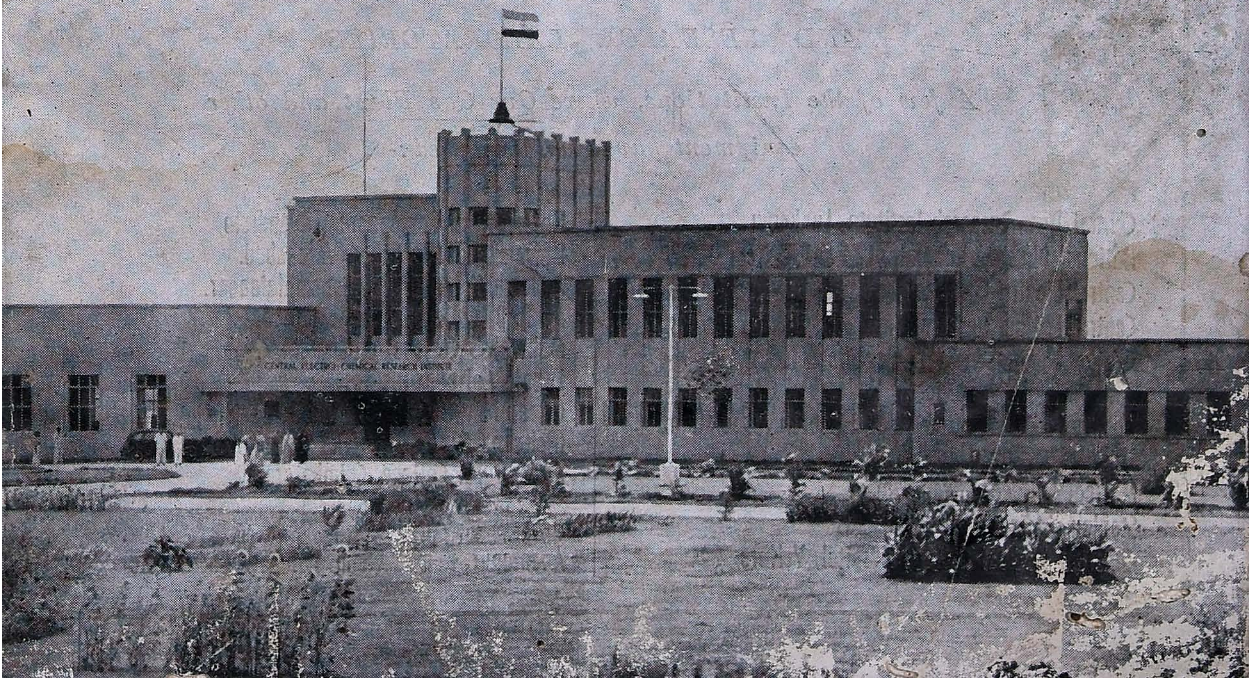

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BULLETIN
OF THE
CENTRAL ELECTROCHEMICAL RESEARCH INSTITUTE
KARAIKUDI

Vol. I. JANUARY 1954 No. 1

CONTENTS

BOOK - REVIEWS :

Take interest, I implore you, in the sacred dwellings which one designates by the expressive term: Laboratories.

Demand that they be multiplied and advanced.

These are the temples of the future—temples of well-being and happiness.

There it is that humanity grows greater, stronger, better.

Louis Pasteur.

Editorial

The starting of a journal for this Institute is an event of more than ordinary significance. It marks the beginning of organized Electrochemical Research in India, and is an expression of faith in the promise of electrochemistry for the future of the country. To-day the border-lines where electricity meets chemistry and the other sciences offer the most exciting fields of exploration, and if we attempt to eliminate all chemical phenomena from chemistry and physics, there is little left of these sciences as we know them. In publishing this modest bulletin we have been actuated by the hope that our early venture may in course of time become worthy to rank with such periodicals of world importance as the Journal of the Electrochemical Society of U. S. A., the Transactions of the Faraday Society of Great Britain and the Zeitschrift für Elektrochemie of Germany. As the Chinese proverb has it "No matter how long the journey the first step must be taken".

The subject of electrochemistry is in its infancy in this country. Workers are few and industries employing trained electrochemists fewer still. There is, however, no cause for dismay, for electrochemical work in other countries had to face similar difficulties in the beginning; the early practitioners of electrochemistry in U. S. A. had little chemical knowledge and the scarcity of chemists in industries was a matter for frequent comment[1]. And when we consider the future, we cannot help being struck by the possibilities of development of what may be regarded as a new science so far as India is concerned. As more and cheaper electric energy becomes available with the completion of the many hydro-electric projects in this country, vistas of large industries consuming hundreds of thousands cf units of current open out before one's eyes. It is, of course, still a dream, but some day it will come true, and the foundation of the Central Electrochemical Research Institute may come to be regarded as the first step in the direction of realising this dream.

The development of electrochemistry has a long history behind it. The invention of the voltaic pile enabled Sir Humphrey Davy and Michael Faraday to make their pioneering studies of the chemical effects of the electric current. Their researches laid the foundations of electrochemical technology. Its large applications in industries, however, came much later. The electrochemical industry is of comparatively modern origin. The construction of the first dynamo for electroplating by Edward Weston (1875) ushered in the Electrochemical era in industry. The next landmark in the history of applied electrochemistry was the electrolytic production of aluminium by Charles. M. Hall in 1886. Thenceforward progress has continued at an amazing pace, and it is stated[2] that to-day the Electrochemical industry in America doubles its consumption of electric power every six years.

The provision in the universities for training in electrochemistry is very meagre, not merely in India, but even in advanced countries like U. S. A. and U. K. A committee appointed by the Electrochemical Society of America to conduct a survey of the need for electrochemists in industry found that the recognized need for electrochemists was only a fraction of the potential demand and even in this situation men with training in electrochemistry were in short supply[3]. The position is immensely more difficult in India and the dearth of suitable men to initiate and guide researches in the various branches of electrochemistry is acting as a severe handicap to rapid progress. It is greatly to be desired that courses in theoretical and applied electrochemistry form an essential part of the curricula of our universities and engineering schools, for there is no more suitable subject to-day for fundamental training in engineering and science.

Notwithstanding, the first sod has been turned; and granted the will and determination on the part of our workers and adequate support from the state, there is every reason to hope that India will also forge ahead and make rapid and important advances in electrochemical research as other countries have been doing, laying the foundations for new and important industries in this country.

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THE CENTRAL ELECTROCHEMICAL RESEARCH INSTITUTE, KARAIKUDI.

ITS GENESIS

The genesis of the Central Electro-Chemical Research Institute, Karaikudi, dates back to the year 1948, when Dr. RM. Alagappa Chettiar, the great philanthropist and educationist of South India made a generous offer of Rs. 15 lakhs and 300 acres of land as donation to the Council of Scientific & Industrial Research for the establishment of this Research Institute at Karaikudi. The Council of Scientific & Industrial Research were already aware of the great part that electrochemical research could play towards the industrial advancement of the country. As early as in 1940, they had sponsored, through the Electrochemical Research Committee and the Graphite & Carbon Electrodes Committee, researches on certain specific problems for investigation in diffirent University Laboratories and other places where some facilities existed. About the year 1944, the Board and the Governing Body of the Council of Scientific & Industrial Research had accepted a scheme for the establisment of a National Research Laboratory for electrochemical research, which was initiated by Dr. S S. Bhatnagar and Dr. J. C. Ghcsh. The Industrial Research Planning Committee of which the late Sir. R. K. Shanmugam Chetty was Chairman also gave their support to that scheme. In such a setting, Dr. Alagappa Chettiar's donation naturally came as a very welcome offer and was accepted by the Governing Body of the Council of Scientific & Industrial Research

In view of the keenness an enthusiasm of Dr. Alagappa Chettiar to bring the scheme to fruition without delay, the foundation-stone of the Institute was laid on the 25th July, 1948 by the Prime Minister, Pandit Jawaharlal Nehru, even before the detailed plans and estimates could be formulated. The Council of Scientific & Industrial Research deputed Dr. A. Jogarao as Officer-on-Special duty. later designated as Planning Officer. A Local Advisory Planning Committee was also constituted with Dr. Alagappa Chettiar as Chairman and the planning Officer as Secretary, and with prominent men of affairs and scientists as Members, to help and advise the Planning Officer in the building and equipping of this Institute and in the solving of certain ancillary problems, such as the development of water-supply posibilities by sinking deep bore-wells, the formation of roads and pathways, the supply of electricity, the designing of the different laboratory and office furniture and work-bench units, the lay-out of various laboratory service-lines, including sanitary and drainage installations etc. Most of the preliminaries having been settled by about the middle of the year 1950, and after fully assuring of the existence of underground water-sources which could be drawn upon by putting down a number of deep bore-wells tenders for building-work were advertised and ultimately the construction work of the main building was entrusted to M/S. The Modern Housing Construction & Properties Ltd., Madras, under the supervision of M/S. Iyengar & Co., Architects Bombay The firm commenced the building operations on the 25th July, 1950, exactly two years after the foundation-stone of the Institute was laid. By the end of the year 1952, the construction of the main building, the installation of essential laboratory services and the procurement of certain laboratory stores end apparatus, workshop machinery and tools, library books and journals etc. were completed The Institute was declared open by Dr. S. Radhakrishnan, Vice-President of the Republic of India, on the 14th of January, 1953. Dr. B. B Dey and Dr. B. K Ram Prasad, who as Scientist-Members of the Local Advisory Planning Committee were intimately associated with the development of this Institute were invited to be the first Director and Associate Director, respectively, of the Institute. Dr. B. B. Dey assumed charge as the Director on the 10th of February, 1953. The completion of the building-works and the commencement of the functioning of the laboratories under the guidance of the Director of the Institute marked the fulfilment of the purpose of the Local Advisory Planning Committee and in the latter's place an Advisory Board was constituted with Dr. Alagappa Chettiar as Chairman and the Director as Member-Secretary.

The main aim of this Research Institute is to provide opportunities and to give adequate work-facilities for both fundamental and applied research, with a view to help expansion of the existing industries and the starting of new ones based on judicious exploitation of the natural resources and mineral wealth of the country. With the major irrigation-cum-hydro-electric and thermal power-projects launched by both the Central and the State Governments coming to fruition, large blocks of electric power are expected to become available for the industrial advancement of the country. For the best possible utilisation of the electric power and the available indigenous raw materials, a large amount of exploratory and developmental research work, both on the laboratory and pilot-plant scale, will have to be carried out; and it is in this that one looks forward to the Central Electro chemical Research Institute to play her role. Already, during the brief period of less than an year that has elapsed since the Institute was formally declared open, with only a comparatively small number of research staff who have been appointed, quite a few research investigations have been brought to a successful stage of completion. A notable example is the electrolytic production of high-purity manganese metal from low-grade manganese ore, of which we have unlimited quantities in the country Other examples of work successfully completed are the preparation of calcium gluconate and the preparation of cuprous oxide of approved quality which is an essential anti-fouling paint-ingredient required by the Navy. Several other interesting research investigations relating to anodising and dyeing of aluminium, electro deposition of alloys. reclamation of metals such as copper, tin and lead from scrap, utilisation of lignite (extensive deposits of which are being unearthed at Neively in the South Arcot District) in the manufacture of calcium carbide, preparation of para-aminophenol, salicylaldehyde and other electro-organic chemicals; preparation of high-test hydrogen peroxide, heavy water, potassium chlorate, titanium metal; formulation of electro plating salts, special-purpose primary cells etc.; to mention only a few, have also been taken on hand and these are expected to yield results of commercial significance in the development of some important industries. As the work in the laboratories gradually evolves and progresses more research and technical personnel will be added on to the existing stall of the Institute.

To accomplish its role fully and to fulfil the duties and functions expected of this Institute, its work is organised under five main Divisions viz.,

Division I.	Electro-Metallurgy & Electric Furnace Products.
Division II.	Electrolytic Cells.
Division III.	Electro deposition & Allied Processes.
Division IV.	Fundamental Electro-Chemistry & Electro-Chemistry of Gases.

Division V. Research Utilisation & Industrial Intelligence.

As electrochemical research often involves investigations on a number of border-line subjects, it is essential to maintain proper liaison and co-ordination with work being conducted in allied fields in the other sister research institutes, in particular, the National Metallurgical Laboratory, the Central Glass & Ceramic Research Institute, the Fuel Research Institute, the National Physical Laboratory and the National chemical Laboratory. That will avoid overlapping and unnecessary duplication of effort and will also contribute to achieving the set objectives, particularly in fields of border-line interest such as electro—metallurgy, electro—plating, powder metallurgy, studies of refractories, electrical insulators, corrosion phenomena, colloid and ion-exchange studies, dry cells and storage batteries, polarographic, chromatographic and other electro-analytic methods etc.

The translation of successful laboratory researches to the pilot-plant scale in order to demonstrate their feasibility in actual industrial operation is often beset with unsuspected problems and difficulties of its own, mostly of a technological nature, which cannot be solved by a mere arithmetical stepping up of the work from the laboratory scale to the pilot-plant scale. Hence, a very important place has been given by way of facilities to pilot-plant investigations in the Institute. Besides the usual laboratory services, adequate facilities for D. C. supply, such as large-capacity accumulators, M. G. sets and rectifiers have been provided since the majority of electrochemical operations are based on direct current supply.

It is hoped that the labours and activities of the scientific and technical workers in this Institute will lead to fuller and better utilisation of the available raw materials and electric power for helping in the expansion of the existing electrochemical industries in the country and for the establishment of new ones.

A. Joga Rao

INDUSTRIAL ALLOY PLATING WITH SPECIAL REFERENCE TO PLATING OF THE α-PHASE.

by
T. Banerjee
(National Metallurgical Laboratory, Jamshedpur)

The properties of alloys have been found to depend, in addition to other important factors such as grain size, internal strain, etc. on the type of phase or phases they are composed of. An example may be cited: In the copper-zinc system the β-constituent obtained from about equal amounts of the two metals is less ductile than the α-brass containing 0—37% of zinc at ordinary temperature. However brass containing 60% copper may be rolled hot. But when the percentage of copper in the annealed copper-zinc alloy lies between 32·8—41.0, one gets an alloy which contains 52 atoms to a unit cube and is known as γ-brass. This brass is extremely hard and brittle. Alloys containing γ-phase even in small quantity are devoid of technical importance. The relations between the properties of alloys and their crystal structure are given in detail in "Metallography" by Desch[1], and have lately been discussed in a general way by H. Moore[2].

For the production of alloys of constant composition with special properties the two requirements are: (1) constancy of composition during the period of deposition, (2) Phase or phases in the deposit should be the same as that predicted from the thermal equilibrium diagram. The question of depositing alloys within narrow limits of composition has been taken up quite recently, due to the large demands in various industries. There are certain composition ranges where alloys are attended with special properties, e. g. more corrosion resistant, more adherent to the base metal, more rapidly buffed, more suitable for subsequent plate, and so on.

That the commercial rubber will adhere to no other metal so nicely as to α-brass (70 copper : 30 zinc) has been established (Buchan and Rae [3]).

Stout and Faust[4] found that an electrodeposited Cd-Zn alloy with 60% of cadmium gave the best protection to mild steel. Further according to Stout and Kowarsky[5] a Cd-Zn alloy containing 53% to 63% Zn is practically as protective as cadmium.

Monk and Ellingham[6] deposited Ni-Zn alloy containing 25% Ni whose hardness was about seven times that of electrodeposited tin. More information about the special properties and uses of these electro-deposited alloys with narrow limits of composition can be found in the paper by Faust [7]. But it was not mentioned in any case if the electrolytic deposits contain phases as expected from the thermal equilibrium diagram till when Stillwell and Stout[8] showed that electrolytic alloys obtained by electrolysing solutions of silver and cadmium cyanides *at constant current density* and without stirring were highly heterogeneous and showed the simultaneous existence of several phases.

Banerjee[9] found that the composition of brass deposited with stirring from an electrolyte of which the concentration remains essentially unchanged during the period of electrolysis is determined by the cathode potential and if the latter is kept constant it remains unaffected by the duration of electrolysis. Using a large bulk of the solution and soluble brass anode with good stirring brasses of copper content varying from 0·98 to 99.1% were deposited from the cyanide bath containing copper and zinc. Banerjee and Allmand[10] found that though the composition of the deposit obtained under the conditions mentioned above remains unaffected by the duration of electrolysis the composition limits of these phases do not always correspond exactly to equilibrium. The homogeneity limits of α-and-η-brass are definitely enlarged while that of complexγ-phase is reduced.

Nambissan and Allmand recorded more complicated observations with silver-cadmium electrolytic alloys. But homogeneity was found to exist in the two extreme phases α and ε solid solutions). They of course found that α-zone narrowed down a little while the ε phase was broadened. Banerjee and Allmand[10] advanced a mechanism for these phase-transformations in electrolytic alloys. They further found that these electrolytic alloys approached the equilibrium condition only on prolonged annealing.

When constant cathode potential was used, deposits of intermediate phases did not correspond to equilibrium in many cases. But in the deposition of the two solid solutions at the extreme end of the phase diagram, α, η in the case of Cu-Zn and ,α, ε, in the case of Ag-Cd), the deposits over a larger portion of these ranges, especially in the α-range, were found to be homogeneous. These observations indicated that it may be possible to deposit α-phase whose composition will not be influenced by the duration of electrolysis even when electrolysis is carried out at constant current density. In fact experiments of Nakamura[12] and Banerjee[13] showed that the structure of electrolytic α-brass when deposited from a cyanide bath of large bulk (to keep the composition of the bath more or less unaltered during the period of deposition) at constant current density corresponded to the thermel counterpart over the entire portion of the α-zone in the phase diagram.

Hence a systematic investigation on the electrolytic deposition of various binary α-phases was taken up at the National Metallurgical Laboratory, Jamshedpur. This was thought necessary as in the deposition of homogeneous α-phases for industrials purposes the practice of keeping cathode potentia constant will not be welcomed as the process itself is difficult and costly-requiring much attention of the technical staff.

Ray and Banerjee[14] studied the deposition of various phases of brass from a bath containing copper tartrate and zincate by changing the copper/zinc ratio in the bath and using a constant current density. They found that copper/zinc ratio of a deposit from a bath remains constant when the current density above a critical value is used. From the study of the static potential of those alloys in N-zinc sulphate solution they got definite indication that in the α-brass and η-brass ranges, deposition is homogeneous. In the β, γ and ε ranges however (especially in the γ -range), transformations in the phases resulting in the change of static potential were observed in several cases. It can be expected from the above observation that production of homogeneous α-phase by electrolytic deposition from a large bulk of electrolyte is possible if the optimum range of current density where α-phase of constant composition is deposited during the period of electrolysis is determined previously. Confirmation of the above observation with the help of X-ray study will be taken up soon.

The initial effect arising out of the type of cathode material used has not been taken into consideration in this paper. Initial disturbances, if any, can however be eliminated by flash-plating from a more concentrated bath where the first deposit will be the same α-brass which is desired to be plated from the main plating vat.

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BRASS PLATING WITH SPECIAL REFERENCE TO PLATING FROM NON - CYANIDE BATHS

by
S. K. Ray
(C. E. C. R. I. Karaikudi.)

Brass having a composition of 70% copper and 30% zinc, is known as α - brass and is largely electroplated for decorative and protective purposes and in the "finishing out" of large quantities of steel hard wares. Brass plated articles have come to occupy an important rôle in various requirements of art, industries and utility.

Brass plating still offers the best method for obtaining the strongest bond of rubber to metal, especially steel. It is only by preplating steel with α - brass (70Cu : 30Zn) that rubber can be made to adhere to it. Bonding rubber to metals has, in fact, extended the scope of brass plating on an industrial scale and has been developed and established by Buchan and Rae[1].

Commercially brass is usually deposited from a cyanide bath containing water soluble complexes of sodium cuprous cyanide $Na_2Cu(CN)_3$ and sodium zinc cyanide $Na_2Zn(CN)_4$ with an excess of free cyanide. Several workers [2,3,4,5,6,7] have studied brass-plating from the cyanide bath and the improvements made have brought the process to a stage of commercial success and utility. There are a number of formulae for brass-plating in the literature. The various factors influencing brass-plating which have so far been studied include the copper/zinc ratio in the electrolyte, concentrations of NaCN (free and total), caustic soda, sodium carbonate, ammonia, and sodium bicarbonate, the pH of the solution and the effects of addition agents, current density, temperature and agitation. Attempts have been made to control the composition of the electrodeposited brass by control of the current density.

Published work on the electrodeposition of alloys of copper and zinc, deals primarily with the deposition of α - brass (70% copper: 30%zinc) which has decorative and industrial uses.

So far the conditions for the deposition of the whole composition range of a binary alloy have not been determined in detail, nor has the internal structure of the whole range of alloys been systematically examined. The deposition of α - brass of constant composition over long periods of electrolysis was studied by Nakamura[8] employing a large bulk of solution and a constant current density; the deposit containing 82.48% Cu had the face-centred cubic structure of α - Brass, identical with that of a thermal brass of the same composition. Kersten[9] obtained deposits which showed in one case an α - and in another an (α-β)-structure, while Kersten and Maas[10], using electrolytes with varying high zinc/copper ratios. prepared brasses with 11.4—21.4% Cu. all of which had the ε-structure; the limits of ε-brass field as given by the thermal diagram for 200° C are 13.4 to 20.6% Cu.

From the same cyanide bath with good circulation of the electrolyte, the composition of which remained practically unchanged during the period of electrolysis, Banerjee[11,12] deposited brasses of various compositions with copper content ranging from 0 . 98% to

99.13% by controlling the cathodic potential and copper/zinc ratio of the electrolyte. He also studied the structure of the deposited brass by X-ray metallography. Such deposits contain either one pure phase or a mixture of two adjacent phases. He observed that the phase composition limit of electrolytic brass is different from that of thermal alloys. The existence range of α-η-ε-brass are definitely enlarged, whilst that of the complex-γ phase is reduced.

Brass can also be plated for industrial purposes from other salt solutions not containing cyanide. The cyanide bath is hazardous for the workers and in carrying out plating from a cyanide bath, certain precautions have to be observed rigorously for the safety of the workers. A few attempts have been made to develop a brass plating bath, free from cyanide but work has been mainly on the fundamental side, and no procedure suitable for industrial application has been devised.

Thon and Pinilla[13] deposited brass from solutions of copper and zinc thiocyanates containig excess of metal thiocyanates. Sukhodskii, Kheiftz and Chapurskii[14] carried out a few preliminary experiments on brass deposition from a bath containing copper and zinc sulphates, tartaric acid and sodium hydroxide. An exploratory study of the deposition of copper-zinc alloys from thiosulphate solutions of cuprous chloride and zinc sulphate was described by Gernes and Montillon[15]. In the deposition of the copper-zinc alloys, the effects of bath temperature and current density on the copper content of the deposit were studied. A number of bath composition and plating conditions have been recommended for the deposition of copper-zinc alloys having the colour and the copper content of ordinary yellow brass.

Ethanolamine complexes of copper and zinc have been used for the co-deposition of copper and zinc on steel, the anodes being of brass, and smooth and adherent deposits of brass of widely varying copper-zinc ratio obtained by Sadagopachari[16], in his preliminary investigations.

The possibilites of the co-deposition of copper and zinc from a solution containing complex oxalates of these two metals have been indicated by A. I. Stabrovsky[17], as the deposition potentials of copper and zinc in their respective solutions are equal through a certain range of current densities; the deposition potentials at different current densities were determined polarographically. Systematic studies are still being made with a view to obtain good deposits of brass on a commercial scale.

Ray and Banarjee[18] studied the deposition of brass of various composition and phases from a bath containing sodium cupric tartrate and sodium zincate by changing the copper/zinc ratio in the bath and using a constant current density. They found that the copper/zinc ratio of the deposit from baths of a particular composition is not altered by further increase of current density above a critical value. This experimental observation was used to develop a commercial brass plating bath to plate α-brass (70 copper: 30 zinc) suitable for decorative and protective purposes and rubber adhesion, the solution being taken in large bulk. The colour, composition and adhesion of brass deposits were studied on mild steel specimen at the optimum range of current density where α-brass (70 copper : 30 zinc) is deposited from a plater's point of view. The influence of various factors on the colour and the composition of the deposits was noted. The results obtained in laboratory scale experiments indicate that commercial scale practice is possible.

In the course of work on other non-cyanide brass-plating baths, the alkaline solution of copper sulphate with glycerine, and sodium zincate has been found suitable. Stabrovsky's[17] observation that the cathodic deposition potentials of copper and zinc in the glycerine complex of copper and sodium zincate respectively are equal through a certain range of current densities indicated the possibility of codepositing the two metals. Preliminary experiments have been completed and confirm this expectation. Brasses of different compostions and in different phases can also be electrodeposited from the proposed electrolyte. Systematic studies are in progress. In the meanwhile, good deposits of brass (70 copper : 30 zinc) for the purpose of a decorative finish have been obtained.

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ELECTROPOLISHING IN RELATION TO ENGINEERING

S. Ramachandran,
(C. E. C. R. I. Karaikudi.)

Electropolishing is the process of obtaining a smooth, bright, mirror-finish on a metallic surface. This is achieved by controlled anodic dissolution of the metal. Systematic studies on electropolishing were first initiated by Jacquet in relation to metallographic studies, eventhough casual observation of this phenomenon had been indicated earlier [1].

The pleasing 'finish' which electropolished surfaces acquire has lead to its adoption in the metal finishing industry. The improvement which this method of treating a metallic surface confers on it in regard to the adhesion of electroplate, wear resistance, and fatigue strength has led to its application in the field of engineering. Therefore the importance of the study of this subject has been well realized, so that in almost all industrially advanced countries the subject is being investigated with great diligence.

A mechanically polished metallic surface is different in structure from the layer beneath it. This surface has an amorphous structure brought about by 'flowing in' of the metal forming what is known as the 'Beilby' layer. Further, metal fragments, broken down crystals, and surface strain are also there. Electropolished surfaces on the other-hand, are free from such surface blemishes, and have surfaces which are "mechanically clean."

A specimen to be electropolished is made the anode of an electrolytic cell, sheets of stainless steel, aluminium or copper being made the cathode. The anode to cathode area varies from 1 : 6 to 1 : 10. The specimen is given a preliminary grind, mechanically polished to an extent and finally electropolished. A variety of electrolytes made up of organic solvent mixed with phosphoric or perchloric acid have been used, the applied voltage being generally low, with high current densities; arrangements also being made for maintaining an uniform temperature. Almost all metals have been eletcropolished for metallographic studies. Mechanical polishing for metallograhic work is tedious, and a skilled job, with low reproducibility. Electropolishing for this type of work is very useful, for this is quick, with high reproducibility, and does not require skilled workmanship.

The pleasing finish, which electropolishing confers on materials has led to the use of this process in giving a very acceptable 'finish' to many automobile parts, those of refrigerators, cutlery, surgical instruments, costume jewellery, novelties etc. Special varieties of steels, copper, brass, aluminium, monel, nichrome, nickel-silver, and silver have been electropolished on an industrial scale with appropriate electrolytes [2, 3].

The usefulness of many materials depends largely on their surface properties. The value of electropolishing in engineering is due to two main characteristics it confers on a surface. It brings out the true crystalline nature of the surface; and secondly an exceedingly smooth surface is brought into existence. The former is responsible for improving the adhesion of an electroplate, and the latter for improving wear resistance and fatigue strength.

Electroplating of metals on other basis material of ferrous or non-ferrous nature is done to prevent corrosion, enhance appearance and improve wear resistance. Whatever may be the object, the adhesion of the plate to the basis metal is of paramount importance

Investigations show that electropolished steel surfaces lead to stronger adhesion of the electroplate than mechanically polished ones. The "mechanically clean" electropolished surface favours crystal growth across metal interfaces. The improved adhesion so brought about is valuable for bearing surfaces[3].

The fatigue strength of alloy-steels such as special half hard Ni-Cr-Mo steels is mainly influenced by the micronotch effect (resulting from microgeometrical roughness), surface stresses and tensile strength. Electropolishing of such steels eliminates microgeometric roughness and therefore the micronotch effect; it removes the surface layer subjected to stresses, and increases tensile strength; all these go to improve the fatigue limit. By reducing microgeometric roughness, this process reduces the friction of a bearing surface and thus enhances its wear-resistance. Electropolishing can be made use of for detecting defective structures not in evidence by ordinary inspection. Because electropolishing preferentially attacks fissures, inclusions etc., surface defects such as grinding cracks are exposed by it. Faulty heat treatment and incomplete tempering leading to structural defects show themselves as matte patches by the side of the polished surface[4].

In apparatus like X-ray tubes where the use of very high voltage is common, electrons are emitted from the cold cathode and such emission is known as cold emission (or high field emission). Cold emission is undesirable for it upsets the normal electronic current, and the current involved may damage the equipment (kinetrons, electron microscope etc.). Cold emissions are the result of surface impurities, and microgeometric roughness. Electropolishing eliminates both, and therefore suppresses cold emission[4, 5].

Grinding and cutting of carbide tools used for machining tough alloys used in jet engines, ordnance etc., consume large quantities of diamond bort which is costly and not easily available. A way of overcoming this difficulty was indicated by a Russian engineer and worked out in greater detail both in Russia and other countries. The technique developed consists in making a revolving wheel the cathode, and the tool to be ground, or cut, or drilled, the anode; the carefully chosen electrolyte being flushed in between them. The application of this electromachining (electro-cutting) process is restricted to small areas as current densities involved are very high[6, 7].

During World War II, brass was in short supply in Germany. Soft iron was therefore used as a substitute for brass in cartridge cases for machine gun ammunition. It was however found that the extraction of the soft iron case after firing was difficult and led to stoppages. It was noticed that if the chambers of machine guns were electrolytically polished soft iron could be used and extraction was easy. It has also been claimed that the life of reciprocating parts of automotive weapons was extended to three to four times their normal life by electropolishing[8].

The process of electropolishing has come to stay as an important industrial process. Recent in origin and starting as an adjunct to metallographic studies, it has developed rapidly. It promises to hold its own in the field of engineering, where surface properties play an important part. However, a word of caution is necessary: the conditions to be adopted are so specific that the application of electropolishing must be taken into consideration even at the designing stage of the material. All materials are not amenable to electropolishing, and so a careful choice of material is necessary to derive the full benefit of electropolishing.

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ELECTROLYTIC MANUFACTURE OF P-AMINOPHENOL

by

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(C. E. C. R. I. Karaikudi)

Introductory The electrolytic preparation of p-aminophenol has been investigated by several workers and there is an extensive literature available on the subject. A survey of the literature shows that a commercially feasible process for the manufacture of this important chemical was evolved in various stages.

Uses. p-Aminophenol finds a variety of uses, the most important of which is as a developer of photographic negatives. A concentrated solution of p-aminophenol is a very good developer which is rapid, soft-working and suitable for tropical climates. It is an intermediate in the production of several important derivatives like Metol (methyl p-aminophenol sulphate), and Glycine (N (p. hydroxy phenyl) amino acetic acid). It is also used in the preparation of a number of dyestuffs, particularly azo and sulphur colours. Among them may be mentioned Chrome Deep Brown 3R, Supranol Yellow RA, Pyrogene Direct Blue RLCF, Hydron Blue BG, Immedial Black, Immedial Green, Katigen Red Brown 6 RN. Under the name of Ursol P it is used as a fur, hair and feather dye which dyes a deep shade of brown by after oxidation. For photographic purposes it is usually marketed as its hydrochloride or stabilised with sodium sulphite under the names of Rodinal, Unal, Kodelan, Citol, Paranol, Perinal and Azol. In the field of pharmaceuticals it is an intermediate for the preparation of the important febrifuges, phenacetin and methacetin.

Historical. p-Aminophenol[1] was first prepared in 1874 by Bayer and Caro by reducing p-nitrophenol with tin and hydrochloric acid. Since then various methods have been developed for its preparation and these can be classified as follows:—

1. Reduction of p-nitrophenol, p-nitrosophenol, p-azo phenol and p-hydroxy azobenzene with active metals and acids like tin and hydrochloric acid, zinc and hydrochloric acid, catalytically with hydrogen and with sodium sulphide, stannous chloride or zinc and sodium hydroxide.
2. Treatment of p-chloro phenol with ammonia under pressure in the presence of a copper catalyst. Technically it has been prepared till recently by the normal Bechamp reduction of p-nitrophenol with iron and a minimum amount of hydrochloric acid. In all these cases, the intermediate like p-nintrophenol p-azo phenol, etc., have themselves to be prepared first by a series of operations from caol tar primaries.

The advantages of the electrolytic method over the chemical method are briefly enumerated below:—

1. The electrolytic method makes use of the cheap and abundantly available nitrobenzene instead of the more expensive p-nitrophenol etc.
2. The reaction can be carried out in a single step and directly gives p-amino phenol.
3. In electrolytic methods, the use of expensive reducing agents like tin, zinc etc., is avoided. The cost of the current used in electrolytic reduction is much lower than that of other reducing agents excepting iron.
4. The electrolytic method yields the product in a high state of purity, since there are no sludges of inorganic by products to be removed.
5. The electrolytic process provides greater ease of control over the operations and the reaction can be carried out to any desired stage by proper adjustment of current density, pH, etc.

Development of the electrolytic process.

Gattermann[3] was the first to describe the electrolytic preparation of p-aminophenol by the reduction of nitro-benzene in warm, concd. H_2SO_4 using platinum electrodes. Later, Haber[4] made a systematic study of the reduction of nitrobenzene and cleared up the existing doubts as to the mechanism of the process. Attempts were made to improve Gattermann's method, notably by Darmstadter[5] and Thatcher[6] who substituted porous carbon cathodes for platinum and obtained a yield of 80%. Caesar[7] tried to use copper gauze cathode and duriron anode in Gattermann's method, without success. During the first world war, Eastman Kodak Co., adopted Thatcher's modification of Gattermann's method for the manufacture of p-amino phenol and Mc Daniel[8] et al have given a detailed description of this process. Mc Daniel found that carbon cathodes gave very poor yields and only platinum metal gave good results. This process was abandoned after some years in favour of the chemical process for the following reasons:

1. The use of strong sulphuric acid is a serious handicap to operation on a commercial scale because of the difficulty of recovering sulfuric acid which is a major item in the cost of production and the excessive upkeep charges due to the corrosion of the electrodes and other equipment.
2. The use of platinum electrodes, entails heavy capital expenditure and requires expert supervision for proper conservation.

The advantages of using dilute sulfuric acid and cheap materials like copper, nickel, lead etc., was obvious and the need was felt for the development of a suitable process which would be commercially feasible. This was developed at first by Hale[9] and Imray[10] but it was not until Dr. Dey[11] and co-workers made new departures that it became practical as a preparative method. The present state of the electrolytic process is due entirely to the pioneering studies of those workers, who describe the use of 20% sulfuric acid of density 112 and amalgamated copper and monel as cathodes. Although this method enjoys the advantages of using dilute H_2SO_4 and cheap materials like copper etc., it is not altogether without drawbacks and these have to be overcome before it can be worked successfully on a commercial scale. The disadvantage in this process is that the optimum current density that can be employed to obtain a fairly good yield of p-aminophenol and a reasonably current efficiency is very low and is of the order of 1 to 2 amp/dm^2 [11]. This will entail the use of very large equipment with consequent increase in capital expenditure, a large floor area. and a low cell capacty. Also in stationary electrodes it is rather difficult to get reproducible results on account of various polarisation factors which come into play.

With a view to improve upon this method and to minimise the time of electrolysis it was decided to try the use of a rotating cathode; it is a well-known fact that the only method to maintain a high current efficiency and to obtain a good yield of the product at high current densities is to employ a high speed rotating disc cathode, and McKee[12] and Gerapcstolou have already reported on the reduction of nitro-benzene under a rotating cathode. The interesting results obtained by Wilson[13] and Udupa who have done further work on this subject, have prompted further investigation of this problem in these laboratories to examine the possibility of translating them to large scale practice and to effect any improvements that may be suggested during the course of the work. A model cell has been designed and fabricated and further work is in progress.

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PROSPECTS OF ELECTROLYTIC PRODUCTION OF MAGNESIUM METAL IN SOUTH INDIA

by

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INTRODUCTION

The production of magnesium metal involves the use of considerable quantities of raw materials and heavy expenditure of electric energy. Hence the plants for the production of magnesium should be located at places where both high grade raw material and cheap electric power are available in close proximity to each other Magnesite ($MgCO_3$) and dolomite ($MgCO_3 \cdot CaCO_3$) are the important carbonate ores. Sea water, although it contains only about 0 135% of magnesium in the form of chloride sulphate and bromide, has been used as a raw material in places where high grade magnesium carbonate ores are not available locally or from import at reasonable prices. Magnesite is the most attractive raw material, especially when it is very pure, as only about three and a half tons of magnesite are required instead of about eight tons of dolomite for the production of one ton of magnesium. About 12-14 kilo-watt hours are consumed per pound of magnesium produced.

Salem in South India is an ideal place for the electrolytic production of magnesium metal due to the following considerations:-

1. There are extensive deposits of high grade magnesite;
2. Electric power can be obtained in large quantities at reasonable rates;
3. Salem is linked by rail and road to many important towns and ports of India and hence transport of the magnesium produced to the principal markets would be cheap;
4. The use of magnesium alloys in air-craft is increasing daily. They are used in the aero-engines, in the manufacture of crank cases, fuselages, fairings, steering parts, petrol tanks, landing wheels propellers etc.. Every air-craft, now built, contains at least half a ton of magnesium and an all magnesium alloy plane is becoming a possibility. The aircraft industry has thus become the biggest consumer of magnesium in the form of its alloys;

The Hindustan Air-craft Ltd., at Bangalore, is very close to Salem and can employ magnesium metal advantageously in the construction of new types of planes, if magnesium alloys are produced at Salem, using the aluminium produced in South-India. Production of magnesium in Salem would lead not only to the expansion of the existing aluminium industry in South India, but also to the installation of additional plant for the production of aluminium at Salem utilising the vast deposits of high quality Shevaroy bauxite of Salem. Shevaroy bauxite is trihydrate or gibbisite and its Bayer treatment would be easier and less expensive than that of Central Indian bauxite. The silica associated with bauxite has to be eliminated by benefication before Bayer treatment, but this would be an inexpensive process. Liquid caustic soda for Bayer treatment can be obtained at Salem economically from the alkali chlorine plant in Mettur. A thermal station employing coke obtained from processed South Arcot lignite as fuel can generate cheap power. The tar and pitch obtained as byproducts in the carbonisation of lignite can be used together with high grade wood charcoal available cheaply at Salem for the manufacture of electrodes.

5. The processes at present employed extensively for the commercial production of titanium & zirconium involve the use of considerable quantities of magnesium Anhydrous chlorides of zirconium and titanium are prepared from zircon and ilmenite available in large quantities in Travancore-sands by well-known methods using mainly the chlorine obtained during the production of magnesium metal from magnesium chloride. Magnesium metal is then reacted with the anhydrous chlorides of titanium or zirconium to obtain the corresponding metal. The magnesium chloride resulting from this process together with fresh amounts produced from magnesite and surplus alkali chlorine is used in turn to obtain magnesium and the process becomes a cyclic one as represented below:-

$$ZrSiO_3 \longrightarrow ZrO_2 \xrightarrow{C+Cl_2}$$

$$ZrCl_4 \xrightarrow{Mg} Zr + 2MgCl_2$$

$$FeO.\,TiO_2 \longrightarrow TiO_2 \xrightarrow{C+Cl}$$

$$TiCl_4 \longrightarrow Ti + 2MgCl_2$$

$$MgCl_2 \longrightarrow Mg + Cl_2$$

Thus the production of magnesium metal in South India would make the economic production of zirconium and titanium possible and create a use for the surplus alkali chlorine of the alkali works.

Zircoium and Titanium in combination with magnesium can be utilised in the construction of air-craft at the Hindustan Air-Craft Limited. The wings of planes can be made with an alloy of magnesium and zirconium to realise the advantages of employing only fewer stringers to prevent buckling at high loads giving more space for fuel tanks and a 30% increase in range. The major use for titanium is also in the field of aviation where high strength, light weight and resistance to corrosion are important. Titanium-magnesium-aluminium alloy has great strength and is light. This can be largely used to replace parts made of steel and heavier alloys in jet planes etc ;

6. Magnesium and its alloys are widely used in the manufacture of radios, telephones etc. The Indian Telephone Industries Ltd., at Bangalore can economically manufacture various parts and derive maximum benefit from a factory established at Salem for the manufacture of magnesium;

7. One of the most promising fields for the untilisation of magnesium is in surface transportation to reduce the dead weight of equipment[1]. The Government Perambur Coach Factory at Madras and also the Hindustan Air-Craft Ltd., at Bangalore can advantageously utilise magnesium in the manufacture of various parts if a factory is established for the production of magnesium at Salem.

8. The demand for magnesium in the production of star shells, tracer ammunition, etc. for defence purposes in India can also be met.

9. The use of magnesium for the production of nodular cast iron, and for the production of alloys containing manganese would help the economical production of not only nodular cast iron but also encourage

the electrolytic production of manganese metal

10. Large quantities of magnesium will also find application in India as anodes in primary dry and wet batteries, water activated batteries etc.. This would eliminate to a very great extent our dependence on foreign zinc for such purposes. Establishment of the following auxiliary industries for the production of component parts of primary batteries would put the manufacture of primary batteries on a sound footing in South India :

    (a) production of manganese dioxide from the manganese ores of South India and of gas absorbing active carbon from cocoanut shell charcoal for use as depolarisers ;

    (b) production of carbon and graphite electrodes from locally available high class vegetable charcoals and pitch and tar from the carbonisation of lignite and wood.

Suggested method for the production of magnesium metal from magnesite at Salem :

The electrolysis of anhydrous magnesium chloride in admixture with sodium chloride, potassium chloride and calcium chloride in proper proportion is the process widely employed for the production of magnesium metal and accounts for about 75% of the metal produced. Although magnesium chloride can be obtained from magnesite by chlorinating calcined magnesite with the byproduct chlorine of the magnesium metal manufacture in the presence of carbon, greater economies in the production of magnesium chloride can be realised by combining the chemical method of manufacturing potassium chlorate from potassium chloride with the production of magnesium chloride from magnesium oxide by the following procedure. Chlorine obtained as a byproduct in the production of magnesium metal together with some fresh amounts of surplus alkali chlorine from Mettur is passed into a suspension of magnesia to obtain a mixture of magnesium chlorate and chloride. The solution is then evaporated to separate out half the amount of magnesium chloride. The liquor is then double-decomposed with the required amount of potassium chloride under proper conditions to precipitate out potassium chlorate. Anhydrous magnesium chloride is then obtained from magnesium chloride solution by well known methods. The total cost of prduction (including cost of electrical energy, operation and maintenance charges, depreciation, interest on capital etc) of potassium chlorate and magnesium metal by this procedure would be lower than the total cost of production of magnesium metal and potassium chlorate by electrolytic methcds separately. [3]

A magnesium plant capable of producing three tons per day would be an economic unit to start with for meeting the present demand from all the various fields of application indicated already. About 5 tons of potassium chlorate can also be simultaneously produced to meet our demand for potassium chlorate in various industries. 3,640 kilowatt years of electrical energy, approximately, would be required to produce 1,000 tons of magnesium. Hence 5,000 kilowatt years of electric energy would be sufficient not only to supply direct current needed for electrolysis but also the electric energy required for calcining magnesia, evaporating the solutions, keeping the electrolyte molten etc..

Electric energy for this purpose can be obtained economically at Salem by the following procedure. Lignite of South Arcot is first subjected to heat treatment out of contact with air at temperatures below 375°C when considerable condensation of the cellulosic or humic constituents with the simultaneous expulsion of oxygen mainly as carbon dioxide and water will take place and the potential energy of the fuel will be concentrated in the residue.[4] The lignite concentrate is then subjected to low temperature carbonisation and the low temperature coke thus obtained is employed for generation of power.

The application of electricity as the heating agent for concentrating lignite and then separately for subjecting it to low temperature carbonisation has several advantages[5] over other methods such as low capital investment, low operating and maintenance costs, small ground area needed; simplicity of design and construction; high thermal efficiency, ability to utilise off-peak electricity, practically automatic operation and control, production of rich low temperature gas, production of easily ignitable coke having uniform burning characteristics etc. 48,000 tons of lignite can utilise 1600 kilowatt years of off-peak electricity and through the intermediate production of 40,000 tons of concentrated lignite, can yeild 28,000 tons of low temperature coke capable of generating 5,000 kilowatt years, 40×10^7 cubic feet of rich gas and 8×10^5 gallons of oil.

The processed rich gas can be used (1) as a very good and cheap substitute for petrol in internal combustion engines, for domestic and industrial heating in the place of high class coal or coke and (2) as a cheap source of methane or after pyrolysis as a cheap source of ethylene and acetylene etc.. The utilisation of methane, ethylene, acetylene etc. in combination with the surplus alkali chlorine available at Mettur in the production of commercially important organic solvents, intermediaries for plastic and synthetic rubbers etc. will encourage the alkali chlorine industry at Mettur From the oil, several fractions can be separated by distillation and industrially important chlorine products obtaind from them economically utilizing the surplus alkali chlorine available at Mettur.

By this procedure, it would be possible to produce magnesium metal at Salem at a total cost (comprising of cost of electric energy, cost of anhydrous magnesium chloride, depreciation, maintenance, wages, salaries etc.) not exceeding the cost of production of magnesium in any place in other countries. Hence the cost of production of titanium and zirconium metals, alloys of magnesium zirconium etc., primary cells and dry batteries, nodular cast iron etc., will compare very favourably with their cost of production in other countries.

CONCLUSION :

In conclusion, it may be stated that the prospects of establishing light metal and other industries vital to civilian needs and national defence in South India are quite bright if the strategic metal, viz., magnesium, is produced at Salem and utilised in South India along the lines indicated in this contribution.

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RECLAMATION OF ALUMINIUM FROM SCRAP

by
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In spite of refinements in practice with the object of minimising metal losses in melting casting and refining, some metal values are unavoidably lost in furnace skimmings, splashings and ladle skimmings etc. Large quantities of scrap in the form of turnings, cuttings, shavings, raspings, dust etc., also arise in the course of fabrication of aluminium utensils etc., from commercial aluminium blanks in various cottage industries. Thus the question of reclamation of pure aluminium from scrap is a very important factor in the national economy. Common contaminants of Aluminium Scrap are grease, oil, copper, iron, silica, manganese, tin, zinc, magnesium and titanium. The grease and allied organic matters can be removed by heating the scrap for 40-50 minutes at about 400°C. Extending the time or raising the temperature causes some oxidation, thereby reducing the quantity of pure metal recovered.

A suitable flux is often the principal requirement for the successful melting of scrap. The flux should be able to dissolve the superficial but tenacious adherence oxide of aluminium. It is very desirable that the flux used is such that is non-deliquescent and cheap, and melts in the temperature range 600-700°C The ability of a flux to reduce the surface tension of globular aluminium particles is another important factor. The density of the flux is also another important consideration. Although pure aluminium melts at 658°C., in practice a considerably higher temperature is required to be attained to melt aluminium as such, on account of the presence of the thin film of oxide which covers aluminium and which behaves as a refractory layer. It is for this reason that certain flux compositions are used for easily melting the aluminium. The fluxes dissolves and thereby remove the oxide film, which floats up and can be separated only by skimming off from the top.

Various methods for the recovery of aluminium-bearing scraps have been suggested in the technical and patent literature.

1. A liquid flux is suggested for covering the scrap during melting. The flux should melt at or below the melting point of Aluminium and it is employed for the purpose of protecting the metal from contact with air and hot combustion products in ordinary melting furnaces.

2. The use of the so called volatile fluxes, for promoting coalescence is also suggested. Volatile salts NH_4Cl, $AlCl_3$ $ZnCl_2$ which vapourise at 350°C., 180°C. and 730°C., respectively, are stirred into a mass of melted scrap consisting of globules of metal mixed with Al_2O_3 and dirt, mainly silicate. The gas evolved lifts up particles and also effects some mechanical stirring. Thus some of the small liquid globules come into actual metallic contact with each other and coalesc. It is also possible that nascent chlorine or HCl formed by the disseciation of the above chlorides may act upon the Al_2O_3 film, forming $AlCl_3$, there by cleaning the surfaces of the globules.

3. According to the report made by the combined intelligence objectives Sub-Committee investigation German Industry, recovery of aluminium from scrap is effected by extractive treatment with mercury. In the treatment of scrap the more soluble impurities such as Sn, Pb, Mg, and Zn are concentrated in the mother liquor of the aluminium-Crystallisation, the less soluble impurities, such as Fe, Si, Ti, Mn, N and Cu going into the residue, after the mercury extraction treatment.
4. According to B. I. O. S. report aluminium electrolytically refined by what may be described as a three layer refining process. In this the melt is maintained in 3 separate layers whose specific gravities are so adjusted that they remain quite separate and distinct unless disturbed. The lowest layer (round the anode) contains the impure aluminium, the middle layer consists of the fused electrolyte, while the refined metal is present in the top layer. The furnace operates at 5 volts and consumes 20 KW. per Kg. of refined aluminium.

On the question of furnace equipment opinion is divided as regards the relative merits and limitations of the rotary the reverberatary types of furnaces. A recent development is the use of the low-frequency induction furnace for melting many kinds of scraps. This equipment is operated now-a-days mainly in Germany and America.

For small scale use a barrel-furnace is suitable. In this bath of salt-flux consisting of 85% NaCl, 10% Cryolite, and 5% KCl is first melted and the furnace is continuously rotated so as to submerge the scrap in the molten flux. It is claimed that loss of metal is thus reduced to a minimum. Before the scrap is melted it is common practice to subject it to a preparatory treatment, in which the scrap is heated to 300° C. in a rotary kiln to dry it and burn off any organic matter. This delivers by a conveyor to a trommel screen and thence the material passes across a magnetic separator which completely separates iron impurities.

After the preliminary treatments indicated above, the scrap is melted by the aid of the chosen fluxes Solid fluxes may be introduced into the melt in various ways viz.,

(a) by stirring,

(b) by wrapping in aluminium foil and plunging into the melt,

(c) by dropping into the melt as solid cakes or tablets having a higher density than the melt.

After adding the fluxes the molten bath gets a cover of dry dross on the top. The dross is removed, before pouring the metal for casting into ingots In practice a relatively high pouring temperature but a low pouring speed is found to give the best compromise and freedom from surface defects and "trapped-gas" porosity. The less the momentum of the flowing stream, the less will be the penetration and the shorter the distance the bubbles have to rise for escape.

Temperature Control is very important. Too High Temperature may result an excessive gas absorption, over-oxidation and coarse-grain structure. The best pouring temperature range is 700°C to 750°C. Once the temperature of the molten metal is allowed to rise too high, the bath becomes gassy and the only remedy in this case is to remelt the ingots and cast again.

Work on the reclamation of aluminium was taken up in this Institute at the instance of a local aluminium utensils' manufacturing firm in Madurai, who desired the large quantities of the aluminium scrap accumulating in their works for reutilisation in the fabrication of the utensils which they found was not quite successful and in connection with which they sought this Institute's aid. After several preliminary experiments involving the use of different flux-compositions formulated for the purpose, a suitable flux has ultimately been evolved which has been found to give satisfactory results in the reclamation of the aluminium scrap. The flux comprises of indigenous, inexpensive and easily available ingredients. The aluminium reclaimed from the scrap was found to be quite satisfactory for fabrication of utensils and for other treatments such as chemical brightening, anodising and dyeing by the conventional methods in the same manner as commercial aluminium sheets.

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ELECTROLYTIC REDUCTION OF SALICYLIC ACID TO SALICYLALDEHYDE

by

H. V. K. Udupa
(C. E. C. R. I., Karaikudi)

Aromatic acids have been reduced to the corresponding alcohols fairly easily by electrolytic methods. However stopping the reduction of the acids at the intermediate aldehyde stage itself presents an interesting problem. This has been done in the case of salicylic acid and benzoic acid, and it is worthwhile investigating the general applications of the method for the preparation of aromatic aldehydes from the corresponding acids which are fairly cheap and readily available.

Salicylaldehyde is manufactured on a substantial scale as an intermediate in the synthesis of the important perfume and flavouring material, coumarin. Two methods are currently employed in industry, of which the older one consists in heating phenol with choloroform and alkali according to what is known as the Reimer-Tiemann reaction. The ortho isomer is separated by steam distillation from the ortho and para isomers produced simultaneously. The second method encountered in industry is the oxidation of ortho-cresol, analogous to the process by which benzaldehyde is made from toluene.

Reduction of an acid to the corresponding aldehyde is attended by many difficulties and the only process of practical significance consists in reduction of the sodium salt of the acid in aqueous solution with sodium amalgam which may be continuously made electrolytically. This method has decided advantages over the Reimer-Tiemann method in that it gives the single isomer desired and at the same time gives a better yield.

Several examples are cited in literature for the preparation of benzaldehyde and salicylaldehyde by this method (1, 2, 3, 4, 5) as well as by the purely chemical method (6, 7,) wherein metallic sodium is introduced directly into mercury to produce the amalgam. The earliest reference to the electrolytic reduction of salicylic acid to salicylaldehyde seems to be a process patented by Hugo Weil in the year 1906 (5). His method consisted in using benzene for extracting the aldehyde as it was formed. Two years later Carl Mettler (2) described a process similar to Weil's whereby the sodium salts of benzoic and salicylic acids where reduced with a 30 to 50 percent yield of the corresponding aldehydes. In a later contribution Weil (1) described a process using 20 percent amalgam, and a 60 percent yield of Salicylaldehyde was claimed. Davies and Hodgson (6) claimed a 64 percent yield.

The work of Tesh and Lowy (4) is by far the most complete study in this field. The most significant contribution made by these authors is the use of sodium bisulphite to combine with the aldehyde and prevent its further reduction. A 55 percent yield has been claimed by these workers using this technique.

Rutovskii and Korolev (3) studied the work of Weil as well as that of Tesh and Lowy and reported that the methods gave yields only upto 34 percent of theory, only if all the experimental conditions were strictly observed. Kawada and

Yosida have reported a similar procedure adding borax to the catholyte in addition and claimed an 80 percent yield.

This important intermediate is thus reported to be obtained in such widely varying yields, that a reinvestigation of the problem to obtain a practical yield of 80 percent seemed worthwhile. When the problem was undertaken for investigation only 28 percent yield of the aldehyde was obtained following the procedure of Tesh and Lowy, although recently May and Kobe[8] had shown that nearly 50 percent conversion could be obtained by using a special type of stirrer rotating only at 250 r. p. m. The work of the Japanese workers was carefully repeated but the yield was not reproduced.

After careful investigation details of the process for obtaining 55 percent yield have been worked out[9]. Further investigation to reduce the cost of production of salicylaldehyde is still being continued and there is reason to believe that such a possibility is in sight. A few of the more important aromatic acids have also been reduced but not with much success. This aspect of the problem is also being studied and the details of the full investigation will be published later.

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CUPROUS OXIDE BY ELECTROLYSIS

by

S. Sampath

(C. E. C. R. I., Karaikudi)

Cuprous Oxide has been classified as an essential raw material by the Government of India and it is mostly in demand as an ingredient in antifouling compositions. Besides its use in marine paints, there are other purposes for which cuprous oxide can be profitably utilised. In the form of dust or spray, cuprous oxide is a valuable fungicide. It is also used as a protective agent for seeds to insure a high degree of germination. If India takes to using cuprous oxide as a fungicide in future, substantial tonnages (nearly 1000 tons a year) will find an outlet in the agricultural trade. To a minor degree, cuprous oxide finds use in the colouring of ceramic glazes and glass and as a disinfectant for swimming pools and ponds. The most recent application of cuprous oxide is in the form of a deposited film for the rectification of alternating current. The country's present annual requirement of 25 tons of cuprous oxide, for painting the bottoms of ships to inhibit the growth of marine organisms, is almost entirely met by imports. Taking into consideration the fact that the pigment is selling at Rs. 3/- per pound, the production of cuprous oxide in India will result in considerable national saving. Further economy can be effected if the copper scrap available in the country is utilised for producing cuprous oxide.

Cuprous oxide was prepared by the electrolytic method for the first time by Lorenz in 1896 [1] and the first patent was taken by Luckow [2] slightly later. Very few papers [3,4,5,6,7] have been published on the subject. Except for the bare outline of the method, all the more important technical process details are covered by patents [8,9]. Hiralal [7] has tackled the problem from a purely academic viewpoint. The physical and chemical characters of the oxide formed in the anode compartment have been studied under conditions of working which differ from those obtaining in industrial practice.

Investigations were taken up, therefore, to prepare by purely electrochemical methods high grade cuprous oxide from copper scrap. With more attention being paid in recent times to particle size and purity meeting the requirements of the paint industry, the electrolytic method has now gained in popularity in spite of its being costly and requiring very close control of operating conditions This method is based on the anodic oxidation of copper in an alkaline salt solution and yields cuprous oxide of the highest purity. The advantages of the method are the flexibility of operation and the ease with which it can be adopted to prepare products of varying properties as demanded by different consumers unlike the older thermal method where copper is oxidised under controlled conditions. The finer sizes which are useful in agricultural applications are yellow in colour and the coarser varieties tend to be red. Mention can here be made of a patented process [8] for the preparation of yellow cuprous oxide in which a protein containing colloid is continually added during electrolysis.

The problem of the electrolytic preparation of cuprous oxide consists in purifying the scrap and oxidising the refined copper to get cuprous oxide.

The scrap is melted, cast into plates and refined in an acid-copper sulphate bath. The refined copper is then anodically oxidised in an alkaline sodium chloride bath. The various conditions which govern the purity and other properties of the pigment are temperature, composition of the electrolyte and current density. These were carefully studied during the investigation. Optimum conditions have been worked out using a glass cell in which the electrodes of copper are kept separated by a diaphragm to prevent cathodic deposition of spongy copper. During electrolysis the alkalinity developed in thecatholyte has to decontrolled and maintained at an optimum level. The product formed is filtered, washed and stabilised against oxidation before drying. Though the U. S. Navy specification 52 C4C describes two grades of cuprous oxide of purity 97% and 90% respectively, the Indian specification IS: 70 of 1950 stipulates a minimum purity of 96% Cu_2O for the pigment, the rest being mostly Cu and CuO. These impurities must also be reduced to the minimum as free copper aids in the corrosion by sea water and cupric oxide does not possess antifouling properties. The pigment is expected to be bright red in colour. Samples prepared under the conditions standardized in these laboratories satisfied the stringent requirements of the Navy and compared favourably in point of purity and colour with samples imported from U. K.

A larger cell capable of producing one pound of cuprous oxide in 4 hours has been constructed. It successfully reproduces the results obtained on the laboratory scale.

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ELECTROLYSIS OF SODIUM SULPHATE

by

P. S. Desikan and V. Aravamuthan

(C. E. C R. I. Karaikudi)

The electrolysis of Sodium Sulphate for the production of Caustic Soda and Sulphuric acid is of considerable interest to India not only as a measure to make good the deficiency in Sulphuric acid production but also as an additional source of Caustic Soda. A minimum of 50,000 tons of Sodium Sulphate will be available annually from Sambhar Lake and Didwana in Rajputana. About six tons of Sodium Sulphate are obtained daily as a waste bye-product of the Viscose Rayon Industry in one of the factories in South India alone. There are at present three viscose factories in India. There is thus an abundant supply of raw material to justify research in this field.

The electrolysis of Sodium Sulphate can be conducted in two ways,

(i) by using two diaphragms and two solid electrodes and

(ii) by using a mercury cathode with only one diaphragm.

Mercury cells were used successfully on an extensive scale during World War II by the Germans. The main advantages of the mercury cell system are lower consumption of electrical energy and yield of concentrated high grade Caustic Soda. However, these advantages are overshadowed by the high initial capital cost needed for the mercury cells.

Experiments have been conducted in this Institute to devise simple types of two-diaphragm cells, capable of being quickly and easily installed, employing cheap and efficient diaphragm materials from indigenous sources on a cottage scale. The laboratory scale set up consisted of a lead-silver alloy anode, stainless steel cathode, ceramic diaphragms and containers of wood coated inside with anticorrosive material, or glass and the like. Considerable economy in electrical energy has been achieved on a laboratory scale by regulating inter-electrode distances and temperature. The result of laboratory scale experiments show that the electrolysis of Sodium Sulphate can become a commercial success in India in places near a rayon factory where the effluent from the cathode and anode compartments can be used as such without further concentration in the manufacturing process. Such a plant will eliminate to a very great extent the dependence of the rayon industry outside on sources of Caustic Soda and Sulphuric acid. Further experiments are being conducted on a laboratory scale to test the feasibility of installing Sodium Sulphate electrolysis plants at places away from the rayon factories but near the sources of raw materials to meet the demand for Caustic Soda and Sulphuric acid from other industries.

PRIMARY WET CELLS

by

V. Aravamuthan

(C. E. C. R. I. Karaikudi)

Primary batteries are regarded as a source of small currents and many primary wet cells have been devised since Volta communicated his epoch-making discovery of the voltaic pile and Crown of Cups to the Royal Society of London on March 20th 1800. The credit for having devised a primary wet cell simple in construction and capable of giving constant voltage with single fluid electrolyte goes to Georges Leclanche. Following on the announcement of the Leclanche primary wet cell in 1868 no fewer than 20,000 of his cells were installed on rail roads and telegraph lines. Leclanche employed manganese dioxide and carbon mixture as a depolariser and found that the best results were obtained when the upper portion of his depolariser was moist without being thoroughly wet, thereby giving a clue to the fact that atmospheric air had a part to play in depolarisation and he was making use of air depolarisation. This observation has now been translated into action commercially, in the "Eveready" and "Lecarbone" "AD" air depolarised cells in which atmospheric air circulates through a porous carbon cathode and acts as an effective and cheap depolariser.

Experiments have been conducted successfully in this Institute to

1. find cheap and effective solid, liquid, gaseous and mixed types of depolarisers from the materials easily available in India
2. work out the optimum conditions for employing aluminium and magnesium as anode materials in the place of zinc
3. substitute cheap electrolytes like commercial common salt for costly electrolytes like ammonium chloride
4. effectively combine a cheap depolariser with an electrolyte in a single fluid cell and find an electrolyte which can also act as a depolariser
5. choose cheap and efficient diaphragms which can be made on a cottage scale from indigeneous sources, and
6. make new wet cells lighter than those in common use.

New types of cheap primary wet cells, having the advantages indicated above are being tested for their suitability for telegraphy, telephony, railway signal systems etc., with the expectation that it may be possible to replace the comparatively costlier leclanche wet cells which are now universally used by these new cells.

Notes & News

Committees & Conferences

An Advisory Board for the C. E. C. R. I. has been constituted consisting of Dr. RM. Alagappa Chettiar, Chairman, Dr. J. C. Ghosh, Mr. M. Ananthasayanam Ayyangar, Dr. S. S. Bhatnagar and Dr. B. K. Ram Prasad with Dr. B. B. Dey as the Member-Secretary. This committee held its first meeting on the 1st October, 1953 under the chairmanship of Dr. RM. Alagappa Chettiar.

Dr. B. B. Dey served as a member of the Four-man Experts Committee appointed by the Government of Madras for examining the modified scheme of elementary education of the State under the chairmanship of Dr. Parulekar of Bombay. The recommendations of the Committee have since been accepted by the Madras State Government.

Colloquia.

Colloquia on the following subjects on which investigations have been carried out in this Institute were held. The reading of a paper on the problem under review was followed by active discussion in which all members of the scientific staff took part.

1. Anodizing and Dyeing of Aluminium by Shri B. A. Shenoy.
2. Preparation of p-Amino Phenol by Cathodic reduction of nitrobenzene by Shri G. S. Krishnamurthy.
3. Electrolytic preparation of cuprous oxide from Copper scrap by Shri S. Sampath.
4. Electrolytic preparation of Potassium chlorate by Sri N. Subramaniam.
5. Production of Electrolytic Manganese by Shri Y. D. Prasada Rao.
6. Batteries by Shri V. Aravamuthan.
7. Utilization of Scrap Aluminium by Shri P. V. Deshmukh.
8. Electrodeposition of Brass from Non-Cyanide bath by Shri S. K. Ray.
9. Electrolytic preparation of Calcium Gluconate by Miss V. Sarada Menon.

Symposium:

A symposium on Electro-chemical processes and their Applications to Indian Industries will be held on the 27th and 28th March 1954 at the Central Electro-Chemical Research Institute, Karaikudi. The symposium will be inaugurated by Dr. S. S. Bhatnagar, Director of Scientific & Industrial Research. The subjects to be discussed at the symposium are:

1. Electro-thermal processes-metallurgical and non-metallic industries;
2. Electric furnace technology;
3. Electrolytic processes-inorganic and organic chemical industries;
4. Electric batteries;
5. Electro-deposition and allied processes;
6. Electric discharges and their applications;
7. Electro-analysis and their applications;
8. Miscellaneous.

Contributors may deal with the fundamental and theoretical or applied aspects of any of the subjects mentioned above. Papers may be forwarded to the convenor, Dr. A. Joga Rao, CECRI, Karaikudi.

Addresses:

The Hon'ble Shri V. V. Giri, Union Minister for Labour addressed the staff when he visited the Institute on 4—1—54. While speaking to the staff, he gave an account of the earlier attempts at planning in India, laying emphasis on the importance of comprehensive planning for the future development of the country and exhorted the scientific workers to aim at being leaders of scientific thought and action and helping in the reconstruction of the country in a manner that posterity might be proud of.

Staff:

The following members of the staff have left us and we wish them all the best of luck.

1. Mr. K. Pullaiah.
   Junior Laboratory Assistant 14-11-53
2. Mr. B. Hanumantha Rao.
   Junior Scientific Assistant 3-12-53

We also wish bonvoyage and all success to Mr. VR. Ramanathan, Senior Scientific Assistant, proceeding to U. K. for higher studies.

Social & Personal:

The C. E. C. R. I. club was inaugurated by Dr. S. S. Bhatnagar on 25-12-52 and is now housed in the main building of the Institute. The club provides facilities to its members for playing volley ball, badminton and ring tennis on the C. E. C. R. I. grounds. Efforts are being made to construct a tennis court on the Institute grounds. Facilities for indoor games are also available.

The staff club has started a reading room and library, containing periodicals and books of general interest. It is still in an embryo stage and it is proposed to increase the number of books gradually.

The opening ceremony of the C. E. C. R. I. canteen was performed by Dr. J. C. Ghosh on the C. E. C. R. I. grounds on 1st October 1953. The need for this canteen had been keenly felt by the staff of the Institute and the establishment of this canteen on a co-operative basis has been a matter of general satisfaction.

A total sum of Rs. 900/-has been received uptodate as donations to the staff club.

Visitors

1.	Dr. J. C. Ghosh, Director, India Institute of Technology, Kharagpur.	1-10-53
2.	Mr. Raymond Currier of New York.	23-10-53
3.	Mr. M. A. Manickavelu Naicker, Revenue Minister, Government of Madras.	3-11-53
4.	Hon. Shri V. V. Giri, Union Minister for Labour.	4- 1-54
5.	Shrimathi M. Chandrasekhar, Union Deputy Minister for Health.	6- 1-54
6.	Shri M. V. Krishnappa, Union Deputy Minister for Food and Agriculture.	6- 1-54
7.	Shri K. D. Malaviya, Union Deputy Minister for Natural Resources and Scientific Research	25- 1-54

It is with deep regret that we note the death on September 16th 1953 at Red Bank, New Jersey, of Dr. Colin G. Fink, at the age of 71.

Dr. Fink was Professor in charge of Electrochemistry at Columbia University and Secretary of the American Electrochemical Society for many years. In the words of Joseph S. Smatko, sectional editor of the Electrochemistry section of the American Chemical Abstracts, Dr. Colin G. Fink earned the title of MR. ELECTRO CHEMISTRY in America. In Dr. Fink's death the Electrochemical Research world has lost a great worker and friend.

Book Reviews

The Economics of Chemical Production, by A. K. Madan (A. MacRae & Co., Ltd. Bombay-London-Calcutta), 1953. pp. 130. Price: Rs. 10/-; Sh 18/-; $ 2.50.

Shri. A. K. Madan is the distinguished author of the well-known work "The Economic Prospects of Chemical Industries in India", first published in the year 1949. His present book "The Economics of Chemical Production" maintains the same high standard as the author's previous work; and there is no doubt that it will serve as a valuable guide to prospective industrialists, chemical technologists and research workers, in placing before them a clear perspective of the relative importance of the different prerequisites for the establishment and organisation of chemical industries on sound and progressive lines. As the author himself has pointed out in his preface, this book may as well appear under the title "The Problems of Industrial Economics" with special reference to the chemical industry.

The titles of the eleven chapters of this book, mentioned below, indicate the breadth of the material covered, in a remarkably terse and trenchant manner within the brief space of about a hundred pages, excluding nearly 30 pages of appendices which are also full of factual information.

Chapter I.	Introduction - which indicates the growth of the industries in the past and the requirements of industry in general.
Chapter II.	The Finance and Capital Investment.
Chapter III.	The Location of Industry.
Chapter IV.	The Problem of Transport.
Chapter V.	The Labour and Its welfare.
Chapter VI.	The Standardization of Products.
Chapter VII.	The Organisation of Marketing.
Chapter VIII.	Controls and Trade Prices.
Chapter IX & X.	The Economic and Tariff Policy.
Chapter XI.	Government and production.

In every one of the eleven chapters, certain essential aspects which play almost a decisive role for the success of an industry, but which are often apt to be under-rated, are rightly given a degree of emphasis which they deserve. Useful information, for example, is presented in chapter VII, as regards "modes of supply", "payment—practices", "packing standards", "Storage of chemicals" etc., questions which are commonly, though erroneously, treated as comparatively "unessential"! This indicates what meticulous care the author has taken in presenting, without omission, all the significant factors which must be reckoned if an industry is to develop along healthy lines. The numerous data presented in the appendices at the end of the book cover what may perhaps be termed "vital" statistics for industries. They include facts and figures relating to the production of sulphuric acid, caustic soda, soda-ash, chlorine, bleaching powder, super-phosphate, bichromates, ammonium sulphate, alcohol ("power" alcohol, rectified spirit, denatured spirit), glycerine, glue, copper sulphate, liver extract, paints and varnishes, coal etc. The appendices also include up-to-date factual information as regards the annual imports of heavy chemicals, drugs and medicines, dyeing and tanning substances, paints and painters' materials, chemical manures, explosives etc., and the exports of chemicals and allied preparations. The information persented, relating to the schedules of import-duties on chemical and allied products, the railway freight-rates and shipping-tonnage scales is also of special relevancy in a book dealing with the economics of chemical production.

This book can be confidently commended to University students, industrialists and technologists and in fact, to all those interested in the starting or expansion of Indian chemical industies.

A. J. R

International Council of Scientific Unions, 1952, Vol. VI (Reports of the proceedinsg of the Sixth General Assembly held at Amsterdam on October 1st to 3rd, 1952 (Ed) pp. 157) E. J. M. Stratton, Cambridge University Press.

The Book is a report of the procedings held under the aegis of the International Council of Scientific Unions at Amsterdam in October 1952. The reports which are mostly in French, survey past achievements in the various fields of science and suggest measures for promoting scientibic activities through international co-operation, for example the allocation of funds to facilitate the international movement of scientists, to subsidise scientific expeditions and to arrange symposia where scientists can collate their views and findings.

The International Council of Scientific Unions is an organisation whose function is to promote international co-operation and activities which favour the development of science. It supplements and co-ordinates the activities of various national scientific academies. Some of the more important of its activities are the promotion of international travel by scientists, organisation of general and special scientific meetings of an international character; the solution of financial and technical difficulties obstructing the prompt publication and universal dissemination of scientific discoveries and ideas, and the imparting of scientific knowledge to young people. The Union works in close co-operation with the U. N. E. S. C. O. to which it owes a great deal for its financial aid and with which it shares certain responsibilities in the field of scientific research, such as the allocation of grants-in-aid for scientific purposes.

It is gratifying to note that countries behind the so called "iron-curtain" are represented in this assembly and their members have taken a very active part in the proceedings - which makes the organization truly international in character.

The address of Prof. H. A. Kruyt, President of the Division of Sciences in the Royal Netherlands Academy, who welcomed the Assembly on behalf of the council, is at once a challenge and an inspiration; a challenge to those skeptical about the value of any organised activity, and an inspiration to younger scientists all over the world.' Says Prof. Kruyt "Your organisation has a very special charm for a scientist; it includes only scientists; the academies are the organisation of the outsatnding scientists in each country; the unions are the foci for activity of national organisations which have been founded and are run by scientists. Therefore one feels happy in these bodies, knowing they enjoy the autonomy so necessary for the free development of science: Scientists discuss scientific problems with other scientists, unhamperd by administrative and political influences........................ one feels here being "entre nous", one knows that any contribution to a discussion doos not seek anything else than the development of science........... I always feel that pure spirit of scientific unselfishness that one misses so often in politically influenced circles".

G. S. Krishnamurthy

Industrial Brazing: H. R. Brooker and E. V. Beatson First edition. pp. 344 London; Iliffe & Sons Ltd., 35 Sh.

Although a large volume of information and data lies scattered in technical literature on this subject, the book 'is offered as the first one of its kind.' This is really a reference book giving in a small compass of three hundred and odd pages a wealth of detail drawn mainly from the experience of the authors.

The origin, principles and scope of brazing is given in the first chapter, and is followed by the second dealing with heating methods and equipment. Brazing materials–brazing alloys and fluxes are discussed, in the third and the fourth chapter describes the design and strength of brazed joints. The six chapters following give the different types of brazing in practice viz., torch, furnace, induction resistance, salt bath and dip brazing. A separate chapter is devoted to the brazing of aluminium in view of the peculiar properties of the metal and its alloys. Following this is the twelfth chapter devoted to special applications and the final chapter deals with selection and inspection. The chapters are so written that each can "stand well on its own", and thus "relieve those who consult a single section from the necessity of making too many references to the other parts of the book". Valuable data are given in numerous tables and graphs, and well chosen photographs facilitate a quicker grasp of the process of brazing.

The chapter on torch brazing is of particular interest to us in India. It is encouraging to learn that costly and specialized equipment is needed only for the continuous production of very large numbers of a given assembly, that there are only a few engaged in such work; and that for those who want to produce a hundred or a few thousand brazed joints "torch brazing holds its own and it is probably true that there are more joints brazed by hand than by all the other methods added together" (pp. 159)

No detail has escaped the authors who describe with picturesqueness the "lazy way" of applying the flux (pp. 168) to the part to be brazed and the right way to do it.

The get-up of the book leaves nothing to be desired, and it deserves a place in all workshops.

S. Ramachandran

Copper in Instrumentation: Copper Development Association, pp. 152 Kendal Hall, Radlett, Herts.

This publication is No. 48 in the series issued by the Copper Development Association. It deals with the uses of copper and its alloys in certain types of measuring and control instruments. These are considered under three main heads: pressure and strain responsive instruments, electromagnetic instruments and temperature responsive instruments.

The book covers the use of copper and its alloys in both ordinery industrial instruments and in the ancillary equipment used for automatic control processes The discussion of the functions of an individual instrument or controller has been split into three stages: the conversion or detection stages. where the sensitive elements detect the change in the variable to be measured or controller, an intermediate stage, where the output of the sensitive element is amplified or transmitted and the final stage, which may be the movement of a pointer over an instrument scale, a pen over a recording chart, or the initiation of a control operation.

Although this book is concerned to a great extent with the materials used in instrumentation, the broader view has not been neglected and the student as well as the practising instrument engineer will find much to interest him.

No hesitation is felt in recommending the book to all concerned with instrumentation as a most useful addition to their technical library.

K. Kameswara Rao

Chemistry of Carbon Compounds; Ed. E. H. Rodd. Vol. 11 Alicyclic Compounds. pp. xi - 488 (Elsevier Publishing Company: New York etc., 1953).

This series of volumes on organic chemistry will be greatly welcomed by all research workers in the field. In addition to being full of information for beginners in research work in any particular field, the books also contain the most authoritative information and guidance on facts needed by all organic chemists. In fact, next to 'Organic Reactions' which are unique in providing experimental techniques to the organic chemist, these books are the best available for ready reference. They surpass 'Richter' and 'Beilstein' in so far as the latest information is included in all branches of organic chemistry under particular heads in a single volume.

Volume II deals with alicyclic compounds and provides a good deal of recent information in this field. It is unnecessary to deal with various chapters in detail here except to point out that they are all very comprehensive. The volume is not dull and drab with a catalogue of reactions and references but has the easy trend of a text-book which makes it interesting to read through.

Although it is somewhat difficult to reconcile oneself to the presence of the chapters on 'Carotenoids' and 'Rubber' in this particular volume, their inclusion is perhaps justified by the comprehensive nature of this work.

Further volumes in this series will be eagerly awaited by all organic chemists.

H. V. K. Udupa.

Abstracts

Selected Literature on Polarography. Lewis J. A., *Industrial Chemist*, 29, 172-9 (1953).

Important books, periodicals and selected bibliography on the subject are indicated.

Electro-Deposition apparatus for student laboratories. Elving, Hayes, and Mellor. *J. Chem. Edn.*, 30, 254-256 (1953).

An easy circuit with the different components for electro-analytical work is given. Constructional details are presented.

Chlorine and Caustic in Italy: Amalgam cell production Kenyon R. L. and Gallone P. *Ind. Eng. Chem.* 45, 1162 (1953). P. J. Wilson, Jr. (C. A. Sept. 10, 1953, p. 8553) Describes the installation of the de Nora cell in Italy and its operation; conditions of operation in other countries are different from those in the U. S.

Cell for electrolysis of alkali chloride solutions: Solvay & Cie, Ger. 810,274, Aug. 9 1951 — M. Mannheimer, C. A. Aug. 25, 1953 p. 7921

Cells with Hg-amalgam flat-pool cathodes and flat solid anodes are described. These cells are particularly suitable for flowing Hg cathodes and for flow of the electrolyte mainly between the electrodes. Rapid flow of the brine and introduction of the brine under higher than atm. pressure into the cells are special characteristics of the construction.

The Current distribution of the Krebs amalgam type chlorine cell: Hine, F., Oyo Butsuri 22, 9-11 (1953); Hideaki Chihara (*C. A.* Sept. 25, 1953, p. 9185).

A mathematical and theoretical analysis is presented for the electric current distribution over the graphite anode of the above mentioned cell used in the electrolysis of NaCl solution. The best position for the horizontal anode in the cell is suggested.

Mercury Cell chlorine and caustic: Saunders, H. J., Gardiner, W. C., and Wood S. L., *Ind. & Eng. Chemistry*, 45, 1824-35, 1953.

A staff industry collaborative report on the process used at Mathieson Alabama Chemical Corporation's Plant.

Chlorine Production with Mercury Cells: Recent Developments: Thomas, L. R. *Chemical & Process Engineering* 34, 312-14, (1953)

The basic principles and characteristics of present day mercury cells are described, and increase in the use of such cells is predicted.

Developments in the Production of chlorine with special reference to mercury cells: Thomas, L. R., *Industrial Chemist*, 29, 489-497 (1953).

A review of the basic principles, underlying the design of mercury cells for the production of chlorine, with a description of the various types of cells which have been developed for this purpose. A comparison of the advantages of the mercury and diaphragm cells is also given.

Industrial Production of Chlorine: Gordon, *J.*, *Chem. Engineering*, 60, 187—193, (1953).

In a review of the various methods of making chlorine on an industrial scale, mention is made of the electrolysis of hydrochloric acid and of the electrolytic reduction of cupric chloride, which is later regenerated by air-oxidation in the presence of hydrochloric acid.

Electrolytic Production of chlorates: Earnest W. K. and Karr E. H., U. S. 2,628,935, Feb. 17, 1953; *C. A.* 47, 15826g.

A solution of sodium chloride containing $NaClO_3$ and $Na_2Cr_2O_7$ is subjected to the action of chlorine and electrolysed at a pH of 6.7-6.9. The electrolyte is continually withdrawn to separate the chlorate and the spent liquor is returned to the system. The dichromate is used to regulate the pH since it reacts with the alkali formed in the cell to form chromate, which is reconverted to dichromate in the chlorination step. The process is cyclic. A typical electrolyte contains NaCl 150, $NaClO_3$ 350, $Na_2Cr_2O_7$ 5 gms/l. The solution is held in the chlorination tank at least 5 minutes.

Chemical Reactions in the electrolytic cell for manufacturing of chlorate IV: Imagava, H., *J. Electrochem. Soc.* Japan 20, 571-4 (1952); *C. A.* 46, 4930; *C. A.* 47, 3720.

The rate of the reaction $2HClO + ClO^- = ClO_3^- + 2HCl$ was determined in the presence of $NaClO_3$ in various concentrations. The velocity constants, K_s are expressed by the equation $K_S = K (P_S/P)^2$, where K is constant for the reaction in the absence of $NaClO_3$, P, the vapour pressure of HClO in the absence of $NaClO_3$ and P_S, the vapour pressure of HClO in the presence of $NaClO_3$.

Electrolytic manufacturing of chlorate with magnetite anodes II: Perchlorate formation: Nagai T., *Proc. Fac. Eng.*, Keiogijuku Univ. 3, No. 8,10-15 (1950).

N investigated the mechanism of perchlorate formation in the electrolysis of $NaClO_3$ with magnetite and platinum anodes. Various current densities were used and at high c.ds, the potential was higher for magnetite than platinum. The potential had no direct effect on the oxidation.

Water soluble chlorates by electrolysis: Farbenfabriken Bayer, Brit. 679,339, Sept. 17, 1952; (*C. A.* 47, 3730i).

Improved graphite anodes for electrolysis of NaCl are hollow, internally cooled and water-proofed with xylene - formaldehyde or polyvinyl chloride resins. The cathodes are made of stainless steel. Such cells operated at a c.d. of 0.02-0.5 amp/sq.cm. and an acidity of 0.02-0.04 mole HCl/l deliver 100 kg. $NaClO_3$ per 570-80 Kw. hrs. with a current yield of 83—5%.

Electrolytic preparation of Iodine from underground brine: Takeuchi, K. and others, *Japan* 6460 (1951) Oct. 19; *C. A.* 47, 3732b.

During the first stage of the process, the brine, rich in iodine contents is treated with mineral acids to the desired pH and electrolysed by circulating through anode chamber in a divided cell, the catholyte being brine containing chlorides or sulphates. The iodine liberated is absorbed by activated carbon and is recovered in the form of NaI soln. by heating it with alkali from catholyte.

This is then electrolysed in a diaphragmless cell to get iodate from which free iodine is obtained by the treatment of the product with mineral acids.

Electrolytic Manganese dioxide: Abraham L. Fox. U. S. 2,608,531, Aug. 26, 1952, *C. A.* 47, 982i.

Manganese dioxide used in primary cells is prepared by the electrolysis of $MnSO_4$, H_2SO_4 in water by using a special type of porous titanium anode.

Production of Sodium Manganate and Permanganate by anodic solution of Manganese: R. I. Agladze and G. M. Domanskaya, *Zhur. Priklad, Khim.*, 1951, 24 915-24; *C. A.* 47, 5277d.

A thorough investigation of the production of sodium manganate and permanganate by anodic dissolution of manganese in alkaline solution of various concentrations and under different conditions of c.d., temperature etc., has been carried out and data are also given for optimum yield of the product.

Electrolytic production of chromium hydride: Snavely C. A., U. S. 2,635,993,, Apr. 21, 1953; M. F. Quaely, *C. A.* 47, Aug. 10, 1953, 7351-52.

Essentially pure, face-centred, cubic Cr hydride is produced by electrolysis of an aq. CrO_3 soln. containing an appreciable amount of Cr^{+++} ion and a high acid ratio. The bath contains 1000 g. CrO_3 and 10-20 g. sugar per l, and sufficient H_2SO_4 to give a CrO_3:SO_4 ratio of 300 : 1. It is operated at 2—8°. The anode is preferably antimonial Pb. The cathode may be copper, brass, steel, etc., Deposition of hydride takes place at a c d.of 115 amp./sq.ft. Periodic additions of CrO_3 to the bath are necessary during prolonged use of the electrolyte. Baths having concentrations varying from 500 g of CrO_3 per l to that of a satd. soln and operated at 28—430 amp./sq.ft., will produce the hydride. If operated at 28 amp./sq.ft., the solution should be saturated with CrO_3. At a c. d. of 430 amp./sq.ft., the bath should contain 500g. l. of CrO_3 The hydride deposits on the cathode. The lattice parameter of the hydride structure was found to be 3. 85 Å and the distance between centres of adjacent Cr. atoms 2. 712 Å. H atoms are believed to occupy the second largest openings in the lattice. The hydride is deposited as CrH and CrH_2. The sugar is added in order to reduce some of the Cr to the trivalent state, which is necessary for proper operation of the bath.

Electrolytic preparation of chromium borides: Andrieux J. L. and Marion, S., *Compt. Rend.* 236, 805-7 (1953): Newcombe H., *C. A.* 47, July 10, 1953 p. 6277.

By electrolysis of molten mixturss of B_2O_3, CaO, CaF_2, and Cr_2O_3 in graphite crucibles with graphite electrodes for 30 min. at 1000-1100° with a current of 40 amps., B_4Cr_3, BCr, B_3Cr_5, BCr_2, and BCr_4 were prepared and identified by chemical and X-ray analyses. B_3Cr_5 and BCr_4 are new compounds. The chemical properties of all the compounds were similar.

Preparation of Potassium Permanganate and Manganate by anodic solution of metallic manganese: Agladze R. I., and Domanskaya G. M., *J. Appl. Chem.* U, S. S. R. 24 887-907 (Engl. Translation; *Zhur. Priklad. Khim* 24, 787-97 (1951); C. M. M., *C. A.* 47, July 10. 1953 p. 6277.

Electrolytic Mn, 99.9% pure, was used as anode with a Ni cathode to study the solution of Mn in alkaline (KOH or K_2CO_3) solutions of Mn. The effect of temperature, c. d., concn., interelectrode distance, and time were determined upon the quantity of Mn dissolved. Passivation was not observed. Fe was harmful.

Electrolytic preparation of beryllium hydroxide-aqueous sodium beryllium fluoride as cathode liquor: Parikh R. K., and Kammermeyer K., *Ind. Eng. Chem.* 45, 1583-5 (1953); *C. A.* 47 Sept. 10, 1953, p. 8554.

A suspension of $Be(OH)_2$ was produced by electrolysis using graphite electrodes separated by a diaphragm. Best results were obtained with a solution containing 1.77 g. Na_2BeF_4/l with a cathode c. d. of 0. 0345 amp. per sq. cm. Time of electrolysis was 1. 25 hrs. with continuous stirring. Current efficiency was 96.0%, recovery 94.6%. The process required 9-19 V, the lower value being in the presence of NaCl.

Electrolytic decomposition of potassium alum: Gallo A., *Chimica* (Milan) 8, 48-51 (1953); Bravo G. A., *C. A.* 47 Sept. 25, 1953, p. 9186.

Electrolysis of a solution of K alum in a cell containing a porcelain diaphragm with a Pb screen anode and a Fe—or Cu—screen cathode, yields K aluminate at the cathode and H_2SO_4 at the anode. The impurities (Fe, Ca, Mg etc.) are precipitated as hydroxides, and H and O are collected. K aluminate is decomposed with CO_2, giving $Al(OH)_3$ and K_2CO_3. Addition of K_2SO_4 to the solution of alum to raise the ratio Al_2O_3/K_2O to the value of 1/1.8 increases yields.

Electrolytic process for recovering by-products from industrial waste liquors: Horvitz.G.J.,U.S.2,618,592, Nov. 18, 1952; London E.L., *C.A.* 47, Aug. 10.1953, p. 7351.

Wastes containing 5—25% H_2SO_4 and 5—35% $FeSo_4$ are inexpensively concentrated in successive ion-migration chambers separated by diaphragms containing glass fibre or asbestos precoated with silicates. The cathode stream in a nascent O electrolyzer is caused to react with the H_2SO_4 and $FeSO_4$ to form $Fe(OH)SO_4$, S, and H_2O, while the anodic cells processed in a nascent H electrolyzer with $Fe_2(SO_4)_3$ and SO_2 probably form $Fe(OH)SO_4$, H_2SO_4 and S.

Separator plates for Lead storage battery: Dutch Pat. No. 72089, April 1953.

Microporous plates are formed by mixing synthetic resin powder with wood powder (having smaller particle size) and bringing the mixture in contact with the vapours of a solvent until the particles of the resin have become tacky and moulding the plates by pressure rolls. Wood powder is impregnated with a 1% solution of the resin. (polystyrene or p. v. chloride in benzene.)

Primary Cells· U. S. Pat. No. 2,638,489 (1953).

A dry cell is prepared with an Al cup as anode. A paper layer separates it from the electrolyte which is a gel composed of an aqueous solution of $MnCl_2$ to which 5% of a flour—corn starch mixture is added. A depolariser bobbin is composed of a mixture of 90% MnO_2 and 10% acetylene carbon with enough $MnCl_2$ to permit it to be moulded. A carbon rod acts as a cathode.

Primary battery: U. S. Patents. 2, 640, 090 and 2, 640, 091, May 1953.

In the water activated batteries sheet or other forms of magnesium are used as negative electrodes, copper wire grids pasted with cuprous chloride to which is added a small quantity of silver chloride, ammonium chloride or potassium chloride to strengthen the paste are used as positive electrodes. These cells have a voltage of about 1.5 and operate at temperatures of —58° to 140°F.

Cuprous chloride for use as a paste can be replaced by $CuCl_2, 3\,CuO.3H_2O$, prepared by treating cuprous chloride contained in a shallow tray in a steam chamber for about 6 hours till its colour changes to lemon. It is then dried. This has higher mechanical strength and is easier to handle than a paste of anhydrous cuprous chloride.

Spongy cadmium electrode· U. S. 2,616,939, (1952).

A paste made up of equal weights of cadmium oxide and water is applied to a cadmium plated copper screen, dried and then converted to spongy cadmium in potassium hydroxide of 1.29 specific gravity with a current density of 1—4.5 amp. per square foot. It is then washed, dried and used. The cadmium sponge thus obtained can be instantly activated and operated in a primary battery system with lead dioxide in sulphuric acid or in a secondary system with nickel oxide in potassium hydroxide. This electrode has a high capacity per unit of weight and volume and can be stored indefinitely.

Amalgamation method for the electrolysis of alkali salts: Dutch patent 71,709 Feb.16, 1953.

In the electrolysis of sodium sulphate in a cell with an inclined anode and diaphragm, a following mercury cathode is used and to both parallel electrodes an inclination of 1:10 is maintained. The inclined smooth bottom of the cell over which the cathode flows is made of a metal like iron (a noncoated metal forming an amalgam insoluble in mercury). By this arrangement a very thin layer of mercury is formed and the mercury content of the cell is reduced considerably .

Manufacture of conductive rubber and conductive rubber bodies: Indian Pat. No. 46,962. *J. Sci. Ind. Res.* 12, A. 492 (1953).

A dispersion of carbon black is mixed with rubber solution and the coagulum obtained by adding a suitable substance to the mixture is dried.

Terylene polyester Fibre: *Brit. Rayon & Silk. J.* 29 No. 342, 44-52 (1952); *C. A.* 47, 6662a.

Is claimed to be resistant to hot acid solutions and oxidising agents and to be useful as anode bags in the electroplating industry.

Metallization of non-conducting materials: C. S. I. R. Indian. Pat. No. 45,696. Feb. 25, 1953; *C. A.* 47, 6283a.

The method employs a reducing bath of cuprammonium sulphate and Na dithionate. The article can be plated with copper in an acid $CuSO_4$ bath after metallization.

Diaphragm for electrolysis: Japan Pat. No. 3216 (1952); C. A. 47, 6800c.

By moulding the polymer or copolymer of methyl or ethyl methacrylate with or without other film forming material.

Electrophoretic insulating coatings: U. S. 2,635,995: April 21, 1953; *C. A.* 47, 7350 i.

The bath contains Al_2O_3 Al $(NO_3)_3$, Mg $(No_3)_2$, $Al(OH)_3$, water and MeOH. After the deposition, the coating is baked at 1600° to make it free from cracks.

Electrophoretic borating of copper wire: U. S. 2, 640,024, May 26, 1953, *C. A.* 47, 7921c).

A coating of vitreous material like borate or fritted glass can be given to metallic wires by passing the wires through a non-aqueous bath in which the metal oxide and the vitreous materials are suspended.

New compounding ingredient for rubber latex: Le. Foll. J., *Rev. gen. Caoutchouc.* 30, 388-92 (1953): Fr. patent 630, 727; *C. A.* 47, 9654i.

Obtained by extraction from cork, Subelat is a good stabilizing agent against mechanical coagulation, a heat sensitizing agent and helps the penetration of latex into fabrics.

New stabilizer for latex: J. Gossot, *Rev. gen. Caoutchouc* 3, 0, 399-400 (1953;) *C. A.* 47, 9655a. 1953.

These are stabilizers L, OC, and N of the Societe, Doittau Produits Chimiques, suitable for normal latex, heat sensitized latex and latex foam respectively.

Bonding rubber to metal: U. S. 2,643,273, June 23, 1953; *C. A.* 47, 9659f.

Incorporation of a concentrated aqueous solution of a cobalt salt in the rubber mix will effect an improvement in the adhesion of the coating on vulcanization.

Rubber impregnated fabrics for diaphragms: Dutch Pat. No.71,710 Feb. 16, 1953; *C. A.* 47, 9659g.

Latex is impregnated into an acid-resistant fabric which has been treated with a latex stabilizing agent. The latex is afterwards coagulated on the fabric under suitable conditions. The diaphragm is suitable for use in electrolytic cells, in particular those that form acids at the anode.

A simple, compact and inexpensive ozoniser: Bonner, W. A., *J. Chem. Edn.*, 30, 452-3 (1953).

A complete description of the apparatus together with dimensions and constructional details is given.

Organic Synthesis by electric discharge: Editor, *Chemical Age*, 591,(1951), Sugino, K,. and Inoue, E., *Bull. Chem. Soc.* Japan, June 1951.

Several reactions occurring in silent discharge are reported viz. (i) formation of phenol from benzene, (ii)benzyl alcohol, benzaldehyde, benzoic acid and cresols from toluene and air (iii) cyclohexanol and cyclo hexanone from cyclohaxane and air (iv) aniline and other basic compounds from benzene and ammonia.

Rapid Determination of free Formaldehyde: Haslam J., and Soppet, W. W., *J. Appl. Chem.* 3, 328-329 (1953).

A neutralised alcoholic solution of HCHOin phenol-formaldehyde syrup is treated with hydroxylamine hydrochloride solution andthe liberated hydrochloric acid is then titrated with standard alkali to pH 3.4 by means of a direct-reading pH meter.

An electron pump for producing high vacuum: Foster, J. S., Jr., Lawrence E. O. and Lofgren E. J. *Rev. Sci. Inst.* 24, 388 (1953)

Magnetically collimated electric discharge is used to produce very high vacuum. The pumping speed is in the range of 3000 to 7000 litres/second with a base pressure of 10^6 m. m. of Hg. A complete description and construction details are given.