

REVIEW

Electrodeposition of binary alloys: an account of recent developments

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Recent literature on the electrodeposition of binary alloys is critically reviewed. Activity in alloy deposition from aqueous media has shown a continuing expansion in recent years. Alloy plating for protective and decorative purposes continues to be prominent. The principal newer areas of interest for practical usage are magnetic alloys for various applications in computer technology, from electroforming diverse articles for intricate electronics hardware to large parts for use in aircraft and aerospace industries and for electroplating on plastics. In addition to the plating of alloys from aqueous solutions, the binary alloys plated from fused salt electrolytes are also mentioned. It is apparent that alloy plating will continue to fill a need in technology for special items which cannot be produced in any other way.

1. Introduction

The present article cannot fully cover the scope of alloy plating, but it may indicate the important role that alloy plating can assume. An extensive survey of the field up to 1960 was made by Brenner [1] in his comprehensive monograph. It is the purpose of this paper to update the information on the recent developments in alloy plating.

The problem of the production of alloys by electrolysis, especially as coatings on other metals, is of great interest. The use of binary or higher alloys appreciably increases the possibility of satisfying a wide variety of practical requirements not met by the applications of pure metals. Relative to the single metals involved, alloy plates can be denser and harder, more corrosion resistant in certain composition ranges, more protective of the basis metal, tougher and stronger, more wear resistant, more suitable for subsequent electroplate overlays and conversion chemical treatments and superior in antifriction service. Alloy plates also continue their applications as decorative coatings, but have found additional usefulness in magnetic alloy films for the computer industry, in electroforming, in the electronics industry and for the plating of plastics.

The first alloy electroplates were made in the

decade 1835–1845, the same era when the electrodeposition of individual metals began. These were alloys of the noble metals and brass. Because alloy deposition is governed by the same variables and parameters as individual metal plating, progress in both has depended on the same advances: purity of commercial chemicals, commercially available electric power and reliable conversion equipment to supply direct current, instrumentation, and a change from the practice of an 'art' to one of technology. Advances in these areas were considerable in the last decade of the nineteenth and the first decade of the twentieth century. In the field of the electrodeposition of alloys in recent years, the maximum number of publications has been reported by the Russians. The United States stands second. Japanese, German, French, Indian and Czechoslovakian sources have also contributed in this field.

At present, more than two hundred systems of binary alloys have been investigated to a greater or less extent to help solve the problem of alloy production by electrolysis. At the same time, only a few of these alloys have been used industrially. This is because the electrodeposition of alloys is a much more complex process than the electrodeposition of pure metals and often needs continuous control and regulation.

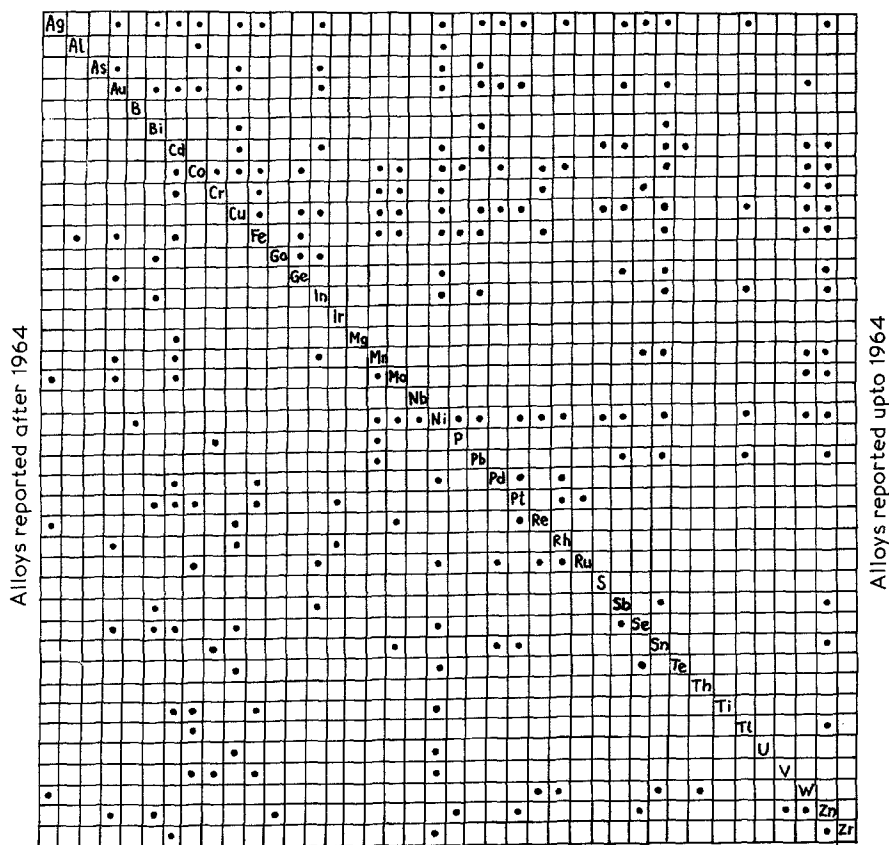


Fig. 1. Electrodeposited binary alloys represented by 'dots' from aqueous solutions reported in the literature up to 1974.

2. New electrolytic binary alloys

Under this heading, only the alloys, which are reported to have been electrodeposited from aqueous media for the first time between 1964 and 1974, are discussed in the light of their recent developments. The alloys reported in the literature up to 1974 have been summarized in Fig. 1.

Because the durability of machine parts often depends on the surface, electrochemical treatments are a common consideration for increasing the wear resistance and hardness of metals. Cadmium is highly effective in the protection of base metals from corrosion. The addition of cadmium to the chromium electroplating bath produced a coating with high strength, hardness and wear resistance [2]. Japanese investigators claim the electro-deposition of a cadmium-titanium alloy coating by rotary barrel plating which is several times as anticorrosive as the usual cadmium coating [3]. A few other alloys containing cadmium have been electroplated viz., cadmium-palladium and

cadmium-manganese [4, 5] alloys from sulphate baths and cadmium-molybdenum alloys from pyrophosphate baths [6]. The electrolytic deposition of cadmium-magnesium alloys has also been reported from a bath containing cadmium sulphate, magnesium sulphate, trilon B, trisubstituted ammonium citrate, hydroxylamine sulphate and joiners' glue [7].

Adherent uranium-containing nickel or copper coatings, which are useful in nuclear physics and chemistry, were made by electroplating from baths containing nickel or copper salts and $\text{UO}_2(\text{NO}_3)_2$ or suspended U_3O_8 [8]. The kinetics of electro-deposition of nickel-boron alloys from an alkaline nickel plating electrolyte containing NaBH_4 was studied by taking polarization curves and potential versus time curves using the oscilloscopic method [9]. A nickel-vanadium alloy deposited from a sulphate bath has also been reported [10]. A study was made of the electroplating of a nickel-zirconium alloy [11, 12]. The effect of various bath parameters on the quality of the nickel-

zirconium plates, cathode current efficiency and on the zirconium content in the alloy plates was examined. The high quality nickel-zirconium electroplates with zirconium contents $\leq 5\%$ (cathode current efficiency 95–100%) were obtained at the cathodic current density $2\text{--}5\text{ A dm}^{-2}$. This alloy also showed an increased chemical resistance in iron-, copper- and ammonium chloride solutions.

Platinum alloys with copper, mercury, iron, tin, cadmium, cobalt or nickel electrodeposited from HCl solutions on titanium or titanium alloy cathodes can be used in the electrolysis of aqueous alkali metal chloride electrolytes for the preparation of chlorine, hypochlorites or chlorates. These were found to have low and constant chlorine overvoltage characteristics [13].

Bright ruthenium-indium alloys may be deposited from sulphate or sulphamate baths [14, 15]. Acid chloride baths have been used to produce wear-resistant nickel-ruthenium and cobalt-ruthenium alloys [16–18]. Electroplated platinum-ruthenium alloys are reported to have very high chemical resistance as they are almost insoluble in acids and bases during anodic polarization or in aqua regia on boiling [19]. To obtain a rhenium-ruthenium alloy, NH_4 nitropentachlorourethane and sulphamic acid were added to an electrolyte containing NH_4ReO_4 [20]. Ruthenium has also been codeposited with palladium.

Considerable interest has been shown in various silver alloys. The preparation of silver-rhenium alloys, first reported in 1965 [21] has been developed into an industrial process [22]. Applications of these alloys, plated from solutions containing silver cyanide, sodium perrhenate, potassium carbonate and potassium cyanide, range from solid film lubrication, grease retention-secondary lubrications, swaged in situ bearings, electrical rotary switches and fretting corrosion-resistant coatings. A silver-molybdenum deposit can be obtained by substituting sodium molybdate for the perrhenate, but the silver-rhenium alloy was found to be superior to other silver or gold alloys in lubrication properties and wear life [23].

The importance of various electroplated selenium alloys is based on various of its properties. Thin layers of cadmium-selenium deposits [24] and selenium-tellurium layers were found to have

semiconductor properties. Nitric acid solutions yielded uniform coatings of a selenium-bismuth alloy [25], while selenium-antimony alloys were obtained from tartrate-citrate bath [26]. Alloys of selenium with chromium, nickel and zinc were produced from acid electrolytes, and alkaline-cyanide solutions yielded alloys with copper or gold. Nickel-selenium alloys are used for composite nickel coatings with improved corrosion resistance [27].

Besides these new binary alloys mentioned above, a number of other alloys are also reported to have been electrodeposited. A gallium-bismuth alloy was electrodeposited on platinum, tantalum and copper from a glycerol bath [28]. The alloy electrodeposit obtained on copper was more compact, fine-grained and had better adherence to the substrate as compared with those on platinum or tantalum. Rhenium and molybdenum can be simultaneously electrodeposited from aqueous solution [29]. X-ray structural analysis shows the deposit to be a solid solution of molybdenum in rhenium. A high quality indium-manganese alloy [30] produced electrolytically was reported, and the baths for the production of phosphorous-zinc, phosphorous-chromium [31] and phosphorous-manganese [32] electrolytic coatings have also been reported and investigated. Recently a gold-germanium electroplate of $5\text{ }\mu\text{m}$ thickness was obtained on a stainless steel cathode coated with gold of $0.05\text{ }\mu\text{m}$ thickness from a bath containing KCN, Na Au (CN)₂, GeO_2 and KOH (to regulate bath pH) [33]. Tungsten-thorium alloys films $\sim 5\text{ }\mu\text{m}$ thick, useful for corrosion-resistant incandescent lamp filaments or coatings on printed circuits, were made by electrodeposition from a bath containing WO_3 , Na_3PO_4 and a 10% Th (SO_4)₂ solution, added drop by drop into the bath during electrolysis [34]. Electrolytic baths to deposit zinc-bismuth [35] and palladium-tin [36] alloys have also appeared in the literature.

3. Prior binary alloys from aqueous solutions

A survey of electrolytic binary alloys of commercial importance, reported up to 1964, has been made. Among the frequently mentioned alloys are: iron-nickel, cobalt-nickel, nickel-tin, chromium-nickel, lead-tin, copper-tin, copper-zinc, copper-tungsten, nickel-zinc, gold-copper, gold-nickel,

cadmium-tin, cadmium-zinc, tin-zinc, iron-zinc and nickel-tungsten. Apart from these, many other alloys also reported in the literature were not of much industrial importance. Recent developments of those of potential practical value have been discussed here with their fields of application.

3.1. *Magnetic alloys*

Thin ferromagnetic films are of great interest from the practical and theoretical points of view. This is because the properties of thin films (up to 10^{-3} cm) are different from those of compact ferromagnetic materials. In recent years the number of papers on the electrodeposition of magnetic coatings has increased, which indicates a promising use of this thin-preparation method. Hard, i.e., with a coercive force > 100 oersted, and soft magnetic coatings can be obtained by electroplating. The former include coatings based on cobalt alloys and the latter those based on nickel-iron alloys.

The deposition of magnetic coatings is one of the most important problems of electroplating because of the ever-increasing use of thin ferromagnetic films in microelectronics, in computers [37, 38] and data-handling technology, in the aircraft and aerospace industries [39–41] and in electroplating on plastics [42].

A large number of papers and patents related to iron-nickel alloys mainly because of their use in producing magnetic coatings for various computers and recording devices have appeared. Single crystal electrolytically deposited nickel-iron films have been reported [43]. More uniform films with constant composition were prepared by discharging a capacitor across the electrolytic cell at the beginning of the electrodeposition. Patents have been granted for the plating of magnetic alloys on wires [44–47] and on non-conductive substrates [48]. Electrodeposited permalloy (80% Ni–20% Fe) films with a thickness of less than 200 nm [49–51] were found to possess excellent magnetic properties.

The magnetic properties of electroless and electroplated cobalt and its alloys have been reviewed [52]. Cobalt codeposited with nickel, tungsten and chromium has been found to have magnetic properties [53–57]. Conditions were studied for obtaining constant quality deposits of a cobalt-platinum alloy whose composition and

magnetic parameters depended on the concentration of platinum ions in the bath and the potential at which the alloy was deposited [58]. Nickel-rhenium films are also reported to have an electromagnetic effect [59]. A more controllable permanent magnetization/coercivity ratio in the electroplate was obtained [60] when a soluble rhenium salt was added to the conventional baths for the electroplating of cobalt-nickel magnetic films.

3.2. *Decorative and protective coatings*

Among the various binary alloys of decorative and protective value, electrolytic brass coatings, in the field of commercial alloy plating, have been widely used. One of the early functional uses of brass, during World War II, was as protective and lubricating coatings on steel shell castings. Brass plating of aircraft engine parts was used to reduce friction. Copper conductors have been plated with brass as a protection against specific types of corrosion [61]. Brass finishes are usually coated with clear transparent organic coatings to prevent tarnishings [62].

At present, most brass is plated for decorative purposes and it is used quite extensively for furniture and builders' hardware. Electroplated brass over aluminium and zinc die castings, steel tubings, and stampings, because of its decorative effect, has been widely used as a substitute for solid brass where weight and cost are factors. Much of the decorative brass is a 'Flash' deposit over bright nickel. A number of changes in plating conditions and the use of addition agents have been investigated to improve or modify the deposition of brass from the cyanide bath [63–66]. Brighteners viz., tetraethylenepentaamine, diethylamine and ethylenediamine were very recently used to produce electrolytic brass coatings from the cyanide bath [67]. Baths using triphosphate [68], pyrophosphate [69] and tripolyphosphate ions [70] as complexing agents have been formulated. Organic complexing agents such as polyethylene-polyamine [71], ethylenediamine [72–74], cyclohexanone [75] and seignette salt [76] have been reported. Bright, adherent, gold-coloured brass electroplates were obtained on zinc and zinc alloys by barrel plating in a pyrophosphate bath [77].

Some interest has also been shown in white brass deposits. Since 1955, the Ford Motor Company has used white brass between a layer of copper and bright nickel which is followed by the conventional chromium as a means for protecting bumpers [78], bumperettes and other exterior parts of their motor cars [79]. The baths to produce decorative and protective white brass have been investigated by several workers [80–83].

Cadmium is highly effective in the protection of base metals from corrosion. But it has been noted that cadmium coatings tend to deteriorate rapidly when steel is exposed to environmental conditions of many different types and these coatings are not satisfactory in high temperature synthetic oils, jet fuels or organic acid vapours. Moreover, because of its cost, it is frequently combined with a cheaper metal to obtain an alloy which has essentially the same properties. Cadmium alloy with 20% zinc was found to have the same anti-corrosive properties as those of pure cadmium [84]. Cadmium-zinc alloys were deposited using various surface active agents such as EDTA [85, 86], Trilon B [87], cetyl-trimethyl ammonium bromide [88], etc. Deposits from these baths are dense, smooth and fine-grained, having good adhesion and covering power and polish to a high lustre. The interest in the electrodeposition of cadmium-tin alloys is of rather recent origin. These have potentialities in the near future because of the high nobility of the electrodeposited alloys as compared to the cast alloy. The coatings of cadmium-tin alloy will effectively prevent corrosion of engine parts under adverse operating and storage conditions. The electrodeposition of the cadmium-tin alloy was made from various baths. It has been stated that the deposit of tin-cadmium alloy from the fluosilicate bath is better than that from the sulphate bath [89–93]. The use of cetyl-trimethyl ammonium bromide, as addition agent, is known to have resulted in good cadmium-tin deposits [94].

The preferred bath for plating bright bronze alloy containing 10% tin includes copper(I) cyanide, sodium or potassium cyanide, stannous sulphate or chloride, sodium or potassium pyrophosphate and Rochell salts [95]. The mechanism of electrodeposition and the properties of the bronzes obtained from the pyrophosphate electrolyte have been studied [96, 97]. The bronzing of

steel strips from a phenolsulphonic electrolyte [98] and the production of corrosion-resistant white bronze coatings [99] have also been done.

Gabe [100, 191] reviewed the service requirements and properties of protective and decorative coatings including solution composition and constitution and the electrochemistry of the cathodic processes. The selection of appropriate electrolytes was discussed in terms of the important deposit properties. The behaviour of coatings in corrosive environments and the role of post-treatments were examined and the importance of standard specifications for the control of processing and performance emphasized. Recent developments highlighted, included nickel-chromium systems, tin-free steel and chromated steel.

Cast and wrought nickel-chromium alloys give excellent wear resistance, corrosion resistance, electrical resistance and high hot strength. Smooth, bright and adherent nickel-chromium coatings containing 2–2.5% nickel were obtained from an electrolyte containing CrO_3 , $\text{NiSO}_4 \cdot 7\text{H}_2\text{O}$ and $(\text{NH}_4)_2\text{SO}_4$. Mineral acids of strength 0.1–2.0 N did not attack the coating which is recommended for coating brass, copper and iron objects [102]. Electrodeposited alloy coatings probably have similar properties and allow a cheap substrate to be used, thus eliminating the need for more expensive cast or wrought alloys. Numerous attempts have been made to develop a suitable commercial plating process. Reviews by Arrowsmith [103] and Chisholm [104] indicate that no such process has been perfected. However, good quality deposits were recently reported from a bath containing potassium chromium sulphate, nickel formate, trisodium citrate, boric acid, sodium fluoride and glycine [105]. Studies on the outdoor corrosion resistance [106, 107] and the behaviour of nickel-chromium electrodeposits in industrial, marine and urban environments [108] indicated the value of such plates when used for decorative purposes.

Good results for the protection of steel by electrolytic zinc-nickel coatings have been reported [109–111]. Coatings containing 10–20% nickel have the most favourable protective and mechanical characteristics. Their resistance to sea water and humidity is claimed to be 6–7 times higher than that of zinc plate [112, 113]. Alloys containing higher nickel contents tend to become brittle. But those with 7–9% nickel have internal stresses

small enough to permit the plated steel to be formed, rolled or drawn without rupturing the plate [114]. Periodic reversal of the current increased the adherence of the deposit and improved the decorative value and uniformity of distribution of the deposit [115]. Both nickel or chromium can be plated on nickel-rich alloy surfaces, but not on zinc-rich alloy surfaces [116]. The chrome plated coatings are useful for automobile bumpers and trim.

Improvements in the properties of nickel-molybdenum and nickel-tungsten electroplates from sulphamate baths were related to a judicious choice of operating parameters resulting from a study of the internal stresses in the deposits [117]. Internal stresses in nickel-tungsten plates increased rapidly with increasing molybdenum content. Thicker electroplates without cracking are obtained by multilayer deposition of alternate layers rich and less rich in molybdenum content. Suitably chromized nickel-molybdenum, and nickel-tungsten electroplates have excellent resistance in oxidizing and reducing acids and show, in certain cases, an increased tendency toward passivation.

Nickel-copper and nickel-tin alloys were also reported to have good corrosion resistance [118, 119]. The recent severe shortage of nickel has promoted the development and commercial use of processes for depositing bright cobalt and cobalt-nickel alloys in all proportions [120]. These processes were perfected for broad industrial use and accepted by numerous firms within a period of months. However, the durability of such coatings leaves some doubt about their acceptance after a long period owing to some impairment of their decorative function, although from purely protective considerations, many of high cobalt content alloys appear to be superior to comparable nickel coatings.

Electrodeposited lead-tin alloys have three major applications, viz., the protection of steel from corrosion, as running surfaces for sleeve bearings (both in the range of 4–10% tin) and as solderable coatings (50–70% tin) [121]. Various bath compositions were given for corrosion resistant, super-bright lead-tin solder [122, 123]. Fluoroborate baths are usually employed with peptone [124, 125] or animal glue [126] as addition agents and a process was described for obtaining microcrystalline, non-porous,

homogeneous and dense lead-tin electrodeposits [127]. Still in a preliminary stage is a method for bright lead-tin solder plating for printed circuit boards [128]. Low-cost, corrosion resistant, very bright and easily soldered tin-lead coatings may be deposited in both rack- and barrel-plating directly on steel, copper wire [129], brass or other copper alloys [130].

The alloys of rhodium and palladium [131] are used by the jewelery industry and patents have been obtained for the electrochemical production of bright, high-carat alloys of gold with cobalt [132], nickel [133–135] and silver [136]. A heat-resisting coating of rhodium-palladium alloy is also reported to have been electrodeposited from a complex bath [137].

Electrodeposited platinum-palladium alloy was obtained from a sulphamic acid bath containing platinum and palladium amine nitro salts [138, 139]. Monoethanolamine was introduced in the sulphamate bath to obtain dense, corrosion-resisting, shiny and hard electroplates based on platinum-palladium alloys with increased palladium content [140].

3.3. *Miscellaneous uses*

Tin-lead alloys, as previously mentioned, are widely used in the manufacture of printed circuits [141–144]. The use of gold-nickel or gold-cobalt instead of gold plate for terminals has produced excellent results from a corrosion and solderability standpoint [145, 146]. Pressure resistors can be made by first coating an insulating substrate with a sensitizing layer and then by a nickel-phosphorous solid solution deposited by electroless plating [147]. Nickel-rhenium films have been studied for such applications as film resistors [148] and electrical point contacts [149], and the effect of atmospheric exposure on the contact resistances of plated tin alloys has also been investigated [150]. An electrodeposited alloy of silver with 3% antimony is recommended as being suitable for contact components [151], and a silver-tungsten alloy is reported to meet the requirements of pressure resistance [152]. Corrosion-resistant, high-frequency conducting coatings of gold containing 5–20% silver can be applied to electrical components from cyanide baths containing EDTA [153].

The hardness and wear resistance of gold is increased by alloying it with graphite [154], antimony [155], palladium [156] and nickel, cobalt or tin [157]. Electrodeposited silver-tin [158] and silver-antimony [159] alloys also possess these properties. The cobalt-rhenium alloys needed to be heat-treated at 315° C. The nickel-rhenium system was found to be less satisfactory. However, heat-treated nickel-tungsten alloy plates exhibited abrasion resistances which were nearly the same as those of hard chromium plating [160]. Electrolytic tin-manganese [161] and lead-manganese alloys [162] possess good antifriction properties and are resistant to corrosion by sulphuric acid or seawater. The abrasion resistance of chromium-molybdenum alloys is reported to be 2–3 times that of hard chrome plating [163], while metal surfaces with good sliding characteristics are obtained by electrodepositing gold-copper, indium-copper or cadmium-copper alloys on steel or aluminium and then heat treating to obtain phase segregation in the electrodeposited layer [164].

Dispersion strengthened alloys have been formed by the codeposition of metals with fine particulate matter such as alumina, titania, silica, zirconia, barium sulphate, silicon carbide, titanium carbide, Kaolin, glass powder or graphite [165–169]. They are widely applied in printed circuitry and electroforming. Gold-copper composite surfaces are used for plated wire memory substrates [170] and a composite electromagnetic shielding material has been described for making printed circuits [171].

4. Fused salt electrolysis

Literature on recent progress in the electrolysis of fused salts has been reported [172]. One of the most promising applications is a highly corrosion resistant aluminium-manganese alloy plated on a base metal from a chloride melt at a comparatively low temperature [173]. Aluminium-manganese alloys were successfully electroplated from a fused salt bath containing AlCl_3 , NaCl , KCl and MnCl_2 . The bath was normally operated at 163–177° C. The electroplating technique and properties of the alloy deposit are also mentioned [174]. The aluminium-manganese electroplating of steel fasteners for aluminium aircraft structures is proposed to prevent corrosion. The process produces a very attractive alternative to cadmium plating in

terms of corrosion resistance, cost and absence of pollution due to cyanide wastes. Chloride melts have also been used to produce an iron-aluminium alloy [175], a lead-bismuth alloy [176] and alloys of refractory metals such as titanium [177–179] and tantalum [180]. Molten alkali fluoride mixtures [181] can be used for the deposition of most refractory metals and their alloys in a coherent and compact form.

Less conventional procedures produced molybdenum-based alloys by carrying out the plating process in molten sodium containing powdered molybdenum-zirconium (1:1) in an argon atmosphere [182]. Molybdenum and rhenium can be simultaneously electrodeposited as oxides from a molten bath containing molybdenum anhydride and potassium perrhenate [29]. The preparation of aluminium-silicon, copper-silicon and copper-aluminium-silicon alloys by the electrolysis of SiO_2 -cryolite melts using aluminium or copper cathodes was studied [183, 184]. Large single crystals of tungsten oxide bronzes can be formed in $\text{Na}_2\text{WO}_4/\text{WO}_3$ melts [185]. Molten electrolytes also yielded alloys of samarium [186] and yttrium [187]. The electrowinning of yttrium-aluminium and yttrium-magnesium alloys from molten fluorides are mentioned [188]. The preparation of high purity yttrium-magnesium alloys by the electroreduction of Y_2O_3 dissolved in a $\text{YF}_3 \cdot \text{LiF}$ bath on a molten magnesium cathode was investigated in laboratory-scale apparatus. [189].

A protective layer of aluminium alloy containing chromium (preferably 2–11% chromium) was deposited on a metal surface such as steel or titanium from a molten salt bath to which chromium ions were added preferably by means of chromium granules or by means of a chromium or chromium-alloy anode [190].

A lead-potassium alloy was produced by electrodeposition from a molten salt electrolyte on a liquid lead cathode [191]. The process for the manufacture of lead-sodium alloys by melt electrolysis has also been investigated [192].

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