United States Patent Office.

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PROCESS OF ELECTROLYTIC PRODUCTION OF NICKEL OR ALLIED METALS.

SPECIFICATION forming part of Letters Patent No. 665,915, dated January 15, 1901.

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To all whom it may concern:

Be it known that I, MORITZ KUGEL, a subject of the King of Prussia, Emperor of Germany, residing at Berlin, Germany, have in-5 vented certain new and useful Improvements in Processes for the Electrolytic Production of Nickel or Allied Metals; and I do hereby declare the following to be a full, clear, and exact description of the invention, such as will 10 enable others skilled in the art to which it ap-

pertains to make and use the same.

The usual methods of electrolytically separating nickel from aqueous solutions of its salts do not permit of the production of de-15 posits of more than about one one-hundredths of a millimeter in thickness, since with a longer continuance of "nickeling" the layer formed comes off in thin scales. This disadvantage can be avoided by using a heated 20 electrolyte, as has been long known; but a thick nickel plate thus produced shows a crystalline structure as compared with the rolled nickel of commerce, is very brittle, and is therefore not suited for direct manufac-25 ture or technical utilization without being

first melted over again.

The object of the present invention is to produce electrolytic nickel in layers of desired thickness in such manner that the prod-30 uct obtained is fully equal to rolled nickel as regards its mechanical properties, toughness, firmness, and ductility, and that, therefore, the otherwise necessary process of remelting, rolling, &c., can be omitted. For this pur-35 pose any desired nickel-salt solution which by its composition is suited to the electrolytic separation of nickel is provided with an addition of a strong mineral acid which will not be changed in its chemical composition 40 by the current—that is to say, the products of decomposition recombine immediately to reconstitute the original material, and a qualitative change of the chemical composition of the bath does not take place. Such an addi-45 tion of acid ordinarily makes the nickel-bath useless, as it effects an immediate scaling off of the deposited metal; but if the electrolyte has been preliminarily heated and is kept at a temperature of over 30° centigrade the scal-50 ing off of the nickel deposit is prevented and an absolutely tough, flexible, and ductile nickel of homogeneous non-crystalline struc- | to maintain a uniform acidity of the bath

ture of any desired thickness can be obtained. The most suitable acids for producing this effect are perchloric acid, perbromic 55 acid, and sulfuric acid. The relative quantity of the addition to be advantageously employed depends upon the temperature and concentration of the bath and also on the desired hardness of the deposit. The proport 60 tion ranges, say, between two per cent. and twenty percent. of that quantity of acid which is contained in the like volume of the simple normal solution. All those acids which are chemically changed by the current and the 65 products of decomposition of which, secondarily, by chemical action make the deposit useless are not suited to this process. This is especially true of nitric acid, the halogen

acids, and all organic acids.

In carrying out the process a difficulty is encountered in that with the high temperature of the bath the acidity of the solution is soon diminished if refuge is not taken in the use of very small anodes or insoluble acces- 75 sory anodes; but both means have the disadvantage, among others, of causing a loss of energy in consequence of the higher current tension which becomes necessary in the bath. The difficulty may be avoided wholly or for 80 the greater part if there be chosen for the electrolyte a highly-concentrated solution of a salt which is formed of the above-named acids and a light metal, such as magnesium, aluminium, ammonium, sodium, and potas- 85 sium. In choosing the salt it is also a condition that it undergoes no chemical change other than that which results in the immediate recombining of its elements during the regular course of the electrolytic process. 90 Combinations of sulfuric acid with potassium or sodium are therefore not well adapted for the purpose, as during the electrolysis the various possible stages of combination of these salts are formed and reformed in the bath, 95 so that, on the one hand, control of the chemical composition of the bath for the time being is impossible, and, on the other hand, also a steady loss of energy appears in consequence of the reactions. On the other hand, 100 the magnesia salts have proved especially suitable, for even when relatively large anodesurfaces are used these salts make it possible

without adding new acids or the like. For instance, in order to make a nickel cliché (which, as is known, possesses the advantage of greater hardness and durability than the usual copper cliché) according to the process described a solution of nickel sulfate and

magnesium sulfate can be used.

The temperature of the solution employed is as high as possible—say 90° to 100° centi-10 grade—because the conductivity increases with the heat, so long as the mold on which the metal is to be deposited is not changed thereby. In molds of easily-fusible materials this heating is naturally not permissible, and 15 a temperature of 30° to 40° centigrade is then sufficient to attain the same effect on the deposit. The concentration of the solution has no essential influence on the constitution of deposit. Here, too, therefore, one may be 20 guided by a regard for the greatest possible diminution of the bath resistance, and, for example, at 90° centigrade and for one liter of water use eight hundred grams nickel sulfate and eight hundred grams magnesium 25 sulfate.

The practical acidity of the solution depends in the first place on the quantity of the current. The action of the free acid is shown first by the appearance of a brisk de-30 velopment of the hydrogen at the cathode. If the quantity of the current be too high, with a high acidity, the development of gas may become so violent that the purely mechanical effect of the same becomes detri-35 mental to the deposit. It is practical, therefore, when there is great acidity to choose a somewhat low current. The current may, however, without harm to the deposit be used essentially stronger than is usual in nickel-40 ing-that is, up to ten to twenty amperes per square decimeter—a correspondingly lively movement of the electrolyte in order to mix well being of course necessary.

The process may be used not only for nickel,

45 but also for allied metals—cobalt, for instance. Similarly it is possible without difficulties to separate other metals also, as copper and zinc, together with the nickel, and thus to produce white-metal by the galvanic method, which is also thoroughly tough and flexible. Thus what is true for nickel and allied metals is true also for the alloys of these metals, so that the expression "nickel and allied metals" is here to be understood to cover also alloys of these metals. For this purpose the solution is made from a mixture of salts of these metals. As anodes I use

either plates of white-metal or strips of the metals in question, to which individually the current is supplied in such strength as is nec- 60 essary for the constant composition of the bath.

Finally, the process is suited also to the electrolytic production of nickel or allied metals from ores or nickel regulus in combi- 65 nation with one of the familiar electrometallurgical processes. For this purpose the nickelous solution is acidulated, heated, and then electrolyzed, and it is of no consequence here whether the nickel regulus serves di- 70 rectly as are anode or the solution is produced by the chemical method and electrolyzed between insoluble electrodes. In any case the process serves to essentially improve the physical properties of the electrolytic nickel 75 produced. Furthermore, the fact is especially noteworthy that the acidulating alone, especially with sulfuric acid, already at times brings about a chemical purification of the solution. Electrolytic nickel frequently con- 80 tains lead, so that even after the remelting it is useless for many purposes. Free sulfuric acid precipitates the lead already in the solution and therefore prevents the separation of the same at the cathode, and thus improves 85 the electrolytic nickel also in this regard.

I claim as my invention—

1. Process for the electrolytic production of tough, rollable nickel and allied metals by subjecting the same to the action of an electric current in an electrolyte, the temperature of which is over 30° centigrade and keeping the electrolyte acid by the addition of a strong mineral acid not changeable by the current.

2. Process for the electrolytic production of tough, rollable nickel and allied metals, consisting in subjecting the same to the action of an electric current, in an electrolyte, the temperature of which is over 30° centinos grade, keeping the electrolyte acid by the addition of a strong mineral acid not chemically changeable by the current, and adding a highly-concentrated solution of a light metal salt not changeable by the current, to facilizate the maintenance of a uniform acidity without the addition of more acid, substantially as described.

In testimony whereof I affix my signature in presence of two witnesses.

MORITZ KUGEL.

Witnesses:

MAX. C. STAEHLER, HENRY HASPER.