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E. J. ROEHL

2,504,239

NICKEL PLATING

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Fig. 1.

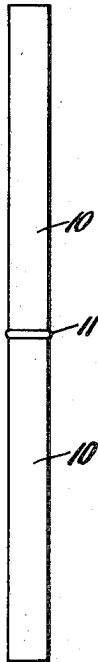


Fig. 3.

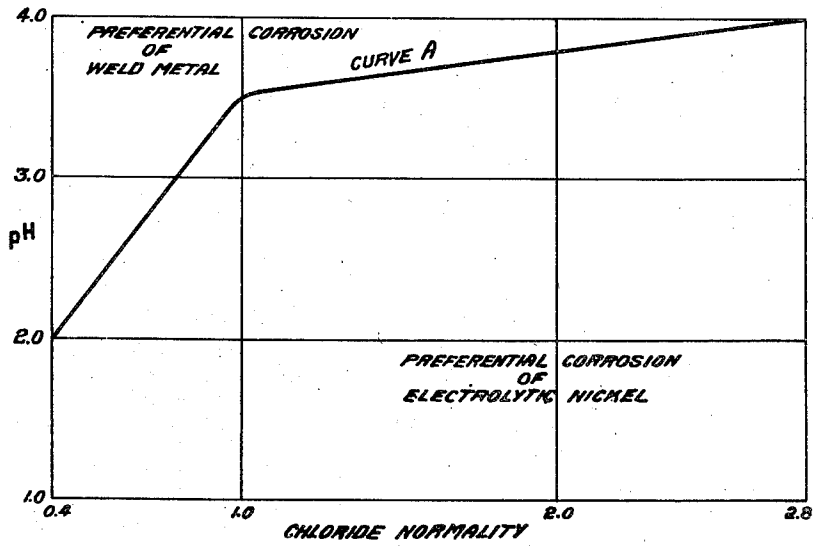
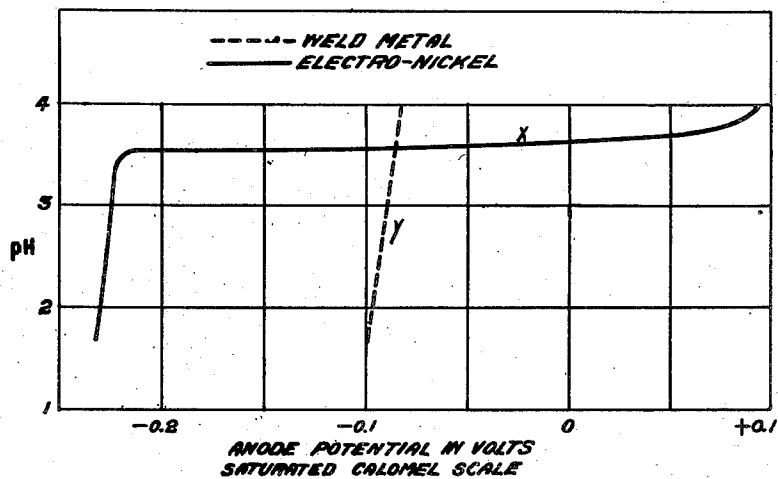
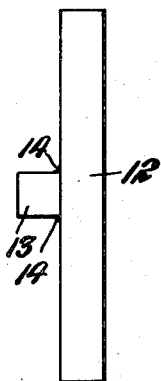


Fig. 4.

BATH — 1.0 NORMAL CHLORIDE
TEMPERATURE — 130°F
CURRENT DENSITY — 30 AMPERES PER SQUARE FOOT

Fig. 2.



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NICKEL PLATING

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2 Claims. (Cl. 204-49)

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The present invention relates to electrolytic nickel anodes and a method for electrodepositing nickel employing electrolytic nickel anodes. More particularly, the present invention relates to welded electrolytic nickel anodes and a method for electrodepositing nickel employing such welded anodes.

Heretofore, one of the factors preventing the more extensive use of electrolytic nickel for anodes, when employed for electrodepositing nickel from chloride-bearing nickel plating baths, has been that of preferential corrosion of weld metals employed in electrolytic nickel anodes. Thus, difficulty has been encountered with weld metals used in welding together lengths or parts of electrolytic nickel to provide anodes that exceed in length the generally available electrolytic nickel stock. Similarly, difficulty has been encountered with weld metals employed in welding together pieces of electrolytic nickel to provide anodes having some particular or desired configuration, as the weld metals hereinbefore employed have been found to corrode preferentially often resulting in severance of the anodes along the weld joint or joints.

Many proposals and attempts have been made to solve the problem of preferential corrosion of welds in electrolytic nickel anodes. For example, in Canadian Patent No. 392,783, it is proposed to prepare an anode by connecting together several pieces of electrolytic nickel by means of a dove-tailed joint protected against electrolytic attack by a rubber band covering the joint. This method has not proved to be entirely satisfactory because the anode corrodes preferentially due to an increase in current density at the edges of the rubber band thereby eventually causing the anode to be severed along one edge of the rubber band. When severance of the anode occurs, such as by preferential corrosion of the weld joint or joints, the portion of the anode below the line of severance drops to the bottom of the bath thereby greatly diminishing the amount of anode remaining for further plating. The portion of the anode that drops results in a substantial economic loss and possible damage to the work in progress. Furthermore, if an anode bag is employed, the dropping of the severed portion of the anode often ruptures the bag releasing particles enclosed therein into the plating bath thereby adversely affecting the deposit on the cathode due to the physical inclusion of the particles in the deposit.

As will be readily appreciated from the foregoing, there is a well recognized need in the art of nickel electroplating for a welded electrolytic nickel anode and a method for electrodepositing nickel employing a welded electrolytic nickel anode, whereby the welded electrolytic nickel anode does not exhibit preferential corrosion of

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the weld joint or joints when the weld metal is in contact with the electrolytic nickel of the anode proper and the anode is subjected to the electrolytic and/or corrosive effects of a chloride-bearing nickel electro-plating bath. As far as I am aware, the proposals and attempts that have been made heretofore in an endeavor to overcome the foregoing and other difficulties and problems have not been completely satisfactory, when carried into practice, in overcoming the obstacle of preferential corrosion.

I have discovered that undesirable preferential corrosion of a dissimilar metal composition, as for example, a welded joint or joints in an electrolytic nickel anode, when employed in electrodepositing nickel from a chloride-bearing bath, is overcome when the dissimilar metal composition comprises a nickel weld such as the compositions hereinafter described, and the welded electrolytic nickel anode is employed in electrodepositing nickel from an aqueous bath operated under correlated conditions of pH and chloride normality.

It is an object of the present invention to provide a method whereby welded electrolytic nickel anodes may be employed without exhibiting preferential corrosion of the weld joint or joints in electrodepositing nickel from a chloride-bearing plating bath.

It is another object of the present invention to provide a method for electrodepositing nickel employing a welded electrolytic nickel anode and a bath operated under correlated conditions of pH and chloride normality whereby the weld metal composition in contact with the electrolytic nickel of the anode proper does not exhibit preferential corrosion with respect to the electrolytic nickel.

It is a further object of the present invention to provide a welded electrolytic nickel anode in which the weld metal does not preferentially corrode when subjected in contact with the electrolytic nickel of the anode proper to the electrolytic, corrosive and/or other detrimental effects of a chloride-bearing nickel electro-plating bath, and to provide a method for producing such welded electrolytic nickel anodes.

It is another object of the present invention to provide an elongated nickel anode by welding together members of electrolytic nickel whereby the anode does not exhibit preferential corrosion of the weld joint or joints when the anode is subjected to the electrolytic, corrosive and/or other detrimental action of a chloride-bearing nickel electroplating bath and to provide a method for producing such an elongated electrolytic nickel anode.

It is also within the purview of the present invention to provide welded electrolytic nickel anodes shaped to conform approximately with

the cathode, which anodes do not exhibit preferential corrosion at the weld joint or joints when subjected to the electrolytic, corrosive and/or other detrimental action of a chloride-bearing nickel electro-plating bath and to provide a method for producing such electrolytic nickel anodes.

Various other objects, advantages and features of the present invention will become apparent from the following detailed description when taken in conjunction with the accompanying drawings in which:

Fig. 1 illustrates an elongated, welded, electrolytic nickel anode prepared in accordance with the present invention;

Fig. 2 is a welded electrolytic nickel anode having an irregular shape prepared in accordance with the present invention;

Fig. 3 represents a chart showing chloride normality as abscissa plotted against pH as ordinate, showing the area for correlation of pH and chloride normality for plating baths whereby the weld metal joint or joints of the electrolytic nickel anodes prepared in accordance with the present invention do not exhibit preferential corrosion; and

Fig. 4 depicts curves showing anode potentials for electrolytic nickel and the weld metal compositions employed in preparing an anode such as employed in practicing the present invention.

Generally speaking, the present invention provides a method for electrodepositing nickel, employing an electrolytic nickel anode having at least one nickel weld joint, from a chloride-bearing nickel electro-plating bath operated under correlated conditions of pH and chloride normality whereby the weld metal does not exhibit preferential corrosion with respect to the electrolytic nickel of the anode proper. The present invention also provides a welded electrolytic nickel anode, comprising a weld metal composition dissimilar to the electrolytic nickel of the anode proper, and in contact therewith, which dissimilar composition does not exhibit preferential corrosion when subjected to the electrolytic, corrosive and/or other detrimental action of a chloride-bearing nickel plating bath in electroplating therefrom.

For the purpose of obtaining satisfactory results by practicing the process of the present invention in electrodepositing nickel from chloride-bearing baths employing welded electrolytic nickel anodes, a nickel weld metal is employed for welding the electrolytic nickel anode. I have found that the generally available type of nickel weld metals, particularly nickel weld metals containing at least about 90% nickel and preferably at least about 97% nickel are suitable when employed in welding electrolytic nickel anodes whereby the weld joint or joints do not preferentially corrode when subjected in contact with the electrolytic nickel of the anode proper to the electrolytic and/or corrosive effects of a chloride-bearing plating bath correlated in pH and chloride normality as hereinafter described. As is well known to those skilled in the art, nickel weld metals contain modifying ingredients, in addition to their nickel content, in order that the weld metal composition will perform satisfactorily in weld operations. Examples of such ingredients are aluminum and silicon as deoxidizers to promote weld soundness, titanium as a deoxidizer to promote weld soundness and to prevent "hot shortness" and as a stabilizer for arc welds, magnesium to promote hot ductility by "fixing"

sulfur, carbon to promote weld soundness and as an arc stabilizer in arc welds and manganese as an auxiliary or buffer element, and, with magnesium, to prevent "hot shortness."

I have found, in practicing the present invention, that a nickel weld containing about 90% nickel and having a composition such as the following provides satisfactory results such as contemplated by the present invention, i. e., the weld metal does not preferentially corrode when in contact with the electrolytic nickel of the anode proper and is subjected to the electrolytic and/or corrosive effects of a nickel plating bath correlated in pH and chloride normality as hereinafter described.

Element	Percent
Silicon.....	0.1 to 2.0
Carbon.....	0.03 to 1.3
Titanium.....	0.05 to 2.0
Aluminum.....	0.0 to 2.0
Manganese.....	0.0 to 2.0
Magnesium.....	0.04 to 0.12
Nickel.....	Balance

While the composition given hereinbefore has been found satisfactory for welding the electrolytic nickel anodes, in carrying the present invention into practice, I prefer to employ a weld metal composition such as the following containing about 97% nickel:

Element	Percent
Silicon.....	0.4
Carbon.....	0.4
Titanium.....	1.5
Aluminum.....	0.2
Manganese.....	0.3
Magnesium.....	0.1
Nickel.....	Balance

When reference is made herein that nickel constitutes the "balance" of the weld metal composition, it is to be understood that smaller amounts, i. e., up to about 2% and preferably not more than about 0.2% of minor constituents, impurities and incidental elements may be present, replacing an equivalent amount of the nickel content. Although the presence of these minor constituents is not essential for obtaining the improved results of the present invention, these minor constituents are generally found to be present as unavoidable impurities in the production of available nickel welds. Examples of such unavoidable impurities are iron, copper, sulfur and cobalt.

In producing a welded electrolytic nickel anode, I have found that when the weld metal employed comprises the compositions hereinbefore described, the welded anode does not exhibit preferential corrosion of the weld joint or joints when the weld metal is in contact with the electrolytic nickel of the anode proper and the anode is subjected to the electrolytic and/or corrosive effects of a chloride-bearing nickel plating bath having the pH and chloride normality correlated as hereinafter described.

Referring to the drawings, Fig. 1 illustrates an elongated nickel anode, such as embodied by the present invention, prepared by welding together members of electrolytic nickel with a nickel weld of the compositions hereinbefore set forth. As shown in Fig. 1, two members of electrolytic nickel, designated by reference character 10, are welded together at weld 11 with the nickel weld metal composition employed by the present

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invention. A welded electrolytic nickel anode, such as shown by Fig. 1, has been found to perform satisfactorily in the process of the present invention hereinafter described, in that the weld metal does not preferentially corrode when subjected in contact with the electrolytic nickel of the anode to the effects of a chloride-bearing, nickel electroplating bath maintained under correlated conditions of pH and chloride normality.

Furthermore, as shown in Fig. 2, the present invention is applicable to provide electrolytic nickel anodes having an irregular shape; for example, a shape conforming approximately to that of the cathode. Such anodes are desired for use in the plating of articles which themselves have an irregular shape whereby the effect of current density upon the surface of the article to be plated is equalized and the throwing power increased. As illustrated in Fig. 2, the electrolytic nickel anode may comprise several differently shaped members of electrolytic nickel designated by reference numerals 12 and 13. As shown, members 12 and 13 are jointed together at welds 14 with the hereinbefore described weld metal compositions. In this manner, electrolytic nickel anodes are provided which do not exhibit preferential corrosion at the weld joint or joints when employed in contact with the electrolytic nickel of the anode to the electrolytic and/or corrosive effects of a chloride-bearing nickel electro-plating bath maintained under correlated conditions of pH and chloride normality.

I have found that in electrodepositing nickel from a chloride-bearing bath, electrolytic nickel anodes prepared as hereinbefore described do not exhibit preferential corrosion at the weld joint or joints when the electrodeposition is carried out from a bath maintained under correlated conditions of pH and chloride normality. The conditions for correlation of pH and chloride normality of the electro-plating bath in order to obtain the results contemplated by the present invention, i. e., whereby the weld metal does not preferentially corrode, are clearly shown by reference to Fig. 3 in which chloride normality as abscissa is plotted against pH as ordinate. I have found that in electrodepositing nickel from a chloride-bearing bath, and employing welded electrolytic nickel anodes prepared as hereinbefore described, the weld metal does not preferentially corrode when the plating bath is maintained under conditions of pH and chloride normality falling in the area under curve A of Fig. 3. Curve A of Fig. 3 shows the approximate maximum pH which should be employed for the bath, correlated with the chloride normality of the bath, in order that the weld joint or joints will not preferentially corrode. Thus, as will be observed by reference to Fig. 3, when a nickel electro-plating bath having a chloride normality of about 0.4 is employed, the maximum pH for preventing preferential corrosion of the weld is about pH 2.0. Similarly, for a bath having a chloride normality of about 1.0, the maximum pH is about 3.5, and for a bath having a chloride normality of about 2.8, the maximum pH is about 4. Hence, as will be appreciated by those skilled in the art, it is a simple matter when the pH or the chloride normality of a bath is known, to refer to Fig. 3 and correlate pH and chloride normality of the nickel electroplating bath to fall under the area defined by curve A and thereby obtain the satisfactory results embodied by the present invention, i. e., that the weld metal of the electrolytic nickel anodes prepared as here-

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inbefore described does not preferentially corrode with respect to the electrolytic nickel of the anode proper.

In determining the necessary correlation of pH and chloride normality for operation of the chloride-bearing baths, whereby the weld metal of the electrolytic nickel anodes is not preferentially corroded, nickel plating baths of varying compositions were employed. Among the aqueous plating baths used were those having the following compositions:

Grams per Liter			Chloride, Normality
$\text{NiSO}_4 \cdot 7\text{H}_2\text{O}$	$\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$	H_2BO_3	
300	45	30	about 0.4
285	112	40	about 1.0
29	336	40	about 2.8

In practicing the present invention, the temperature of the electro-plating bath may be varied over a wide range of temperature, such as from about 100° F. to the boiling point of the bath. However, for best results, I prefer to employ a bath temperature of 120° F. to 140° F., i. e. about 130° F.

With reference to current density, a wide range of current density may be employed in practicing the present invention and the desired results contemplated obtained; i. e., that the weld joint or joints of the electrolytic nickel anodes do not preferentially corrode when employed in electrodepositing nickel from a bath correlated in pH and chloride normality to fall in the area under curve A of Fig. 3. Thus, for example, anode current densities of 30 and 100 amperes per square foot have been found satisfactory for electrodepositing nickel from baths having chloride normalities and pH correlated to fall in the area under curve A of Fig. 3. I have found that current densities lower than 30 amperes per square foot and higher than about 100 amperes per square foot may also be employed with satisfactory results in electrodepositing nickel from baths correlated in pH and chloride normality as hereinbefore set forth. The current densities of 30 and 100 amperes per square foot are set forth merely to show that the present invention is applicable to a wide range of current density and particularly for current densities generally employed in large scale commercial nickel electro-plating operations.

For the purpose of giving those skilled in the art a better understanding of the present invention and the improved results obtained thereby, the following illustrative examples are given:

Example I

An electrolytic nickel anode was prepared by welding together pieces of electrolytic nickel with a weld metal having the following composition:

Element	Percent
Silicon.....	0.1 to 2.0
Carbon.....	0.03 to 1.3
Titanium.....	0.05 to 2.0
Aluminum.....	0 to 2.0
Manganese.....	0 to 2.0
Magnesium.....	0.04 to 0.12
Nickel.....	Balance

The welded electrolytic nickel anode was used as an anode in an aqueous nickel electroplating bath containing 300 grams per liter of nickel sul-

fate, 45 grams per liter of nickel chloride and 30 grams per liter of boric acid. The chloride normality of the bath was about 0.4. At a bath temperature of about 130° F. and employing an anode current density of about 30 amperes per square foot, it was found that the weld did not exhibit preferential corrosion with respect to the electrolytic nickel of the anode proper when the pH of the bath did not exceed about 2.0, but that the weld preferentially corroded at a pH exceeding about 2.0.

Example II

An electrolytic nickel anode was prepared by welding together pieces of electrolytic nickel with a weld metal having the following composition:

Element	Percent
Silicon.....	0.4
Carbon.....	0.4
Titanium.....	1.5
Aluminum.....	0.2
Manganese.....	0.3
Magnesium.....	0.1
Nickel.....	Balance

The welded electrolytic nickel anode was used as an anode in an aqueous nickel electroplating bath containing 285 grams per liter of nickel sulfate, 112 grams per liter of nickel chloride and 40 grams per liter of boric acid. The chloride normality of the bath was about 1.0. At a bath temperature of about 130° F. and by employing an anode current density of about 30 amperes per square foot, it was found that the weld metal did not corrode preferentially with respect to the electrolytic nickel of the anode when the pH of the bath did not exceed about 3.5, but that the weld metal corroded preferentially when a pH of about 3.5 was exceeded.

Example III

An electrolytic nickel anode was prepared by welding together pieces of electrolytic nickel with a weld metal having the following composition:

Element	Percent
Silicon.....	0.1 to 2.0
Carbon.....	0.03 to 1.3
Titanium.....	0.05 to 2.0
Aluminum.....	0 to 2.0
Manganese.....	0 to 2.0
Magnesium.....	0.04 to 0.12
Nickel.....	Balance

The welded electrolytic nickel anode was employed as an anode in an aqueous nickel electroplating bath containing 29 grams per liter of nickel sulfate, 330 grams per liter of nickel chloride and 40 grams per liter of boric acid. The chloride normality of the bath was about 2.8. At a bath temperature of 130° F. and employing an anode current density of about 30 amperes per square foot, it was found that the weld metal did not corrode preferentially when the bath pH did not exceed about 4.0 whereas at a pH exceeding about 4.0, the weld metal corroded preferentially.

Although the exact nature of the phenomena, explaining the improved resistance to preferential corrosion exhibited by the weld metal employed in accordance with the present invention, has not been definitely ascertained, it is believed that the following theory discussed in connection with the welding of electrolytic nickel anodes will be helpful in understanding the principles underlying the present invention.

In the welding of nickel anodes, as proposed

by the prior art, it is believed that preferential corrosion of the weld metals employed occurs because the anode potential of the weld metal is more active or less noble than that of the electrolytic anode material proper. Therefore, as the anode potential of the weld metal is more active, the current density effective thereon is higher than on the electrolytic nickel of the anode and preferential corrosion is exhibited by the weld metal when in contact with electrolytic nickel of the anode and subjected to the electrolytic, corrosive and/or other detrimental effects of the plating bath. By employing a welded electrolytic nickel anode, prepared in accordance with the present invention, in a chloride-bearing bath correlated in pH and chloride normality to fall in the area under curve A of Fig. 3, the weld metal has a less active or more noble anode potential than the electro-nickel of the anode proper. Thus, the current density effective upon the weld metal is less than upon the electrolytic nickel of the anode proper and preferential corrosion of the weld metal does not occur. In this connection, Fig. 4 is a chart showing anode potential curves by plotting anode potential in volts versus bath pH for the electrolytic nickel and the weld metal compositions employed by the present invention. The values depicted in Fig. 4, i. e., anode potential values on the saturated calomel scale, were obtained by employing a saturated calomel electrode fitted with a saturated potassium chloride salt bridge in series with a plating bath bridge. In Fig. 4, curve X designates the anode potential curve for the electrolytic nickel of the anode whereas curve Y shows the anode potential curve for the weld metal employed by the present invention. As will be noted from these curves, the anode potential of the weld metal employed by the present invention is more noble than that for electrolytic nickel. Because the weld metal employed by the present invention does not corrode preferentially when in contact with electrolytic nickel and subjected to the electrolytic and/or corrosive effects of a nickel plating bath correlated in pH and chloride normality as hereinbefore set forth, it is believed that the resistance to preferential corrosion of the weld metal is due to its more noble anode potential. By reference to Fig. 4 it will be readily observed that the weld metal has a more noble anode potential than electrolytic nickel at a bath pH not exceeding about 3.5 whereas with a bath pH exceeding about 3.5, the electrolytic nickel has a more noble anode potential than the weld metal. Although the believed reasons for the improved resistance to preferential corrosion exhibited by the weld metal of the present invention are recited hereinbefore, it is to be understood that the theory underlying the improved performance obtained by practicing the present invention may be different than that recited hereinbefore. However, regardless of the reasons underlying the improved results obtained with the present invention, it has been found that preferential corrosion of the weld metal employed by the present invention does not occur when the weld metal is in contact with electrolytic nickel of the anode proper and subjected to the electrolytic, corrosive and/or other detrimental effects of an aqueous nickel plating bath having a chloride normality and pH correlated to fall under the area defined by curve A in Fig. 3.

The electrolytic nickel employed in preparing the anodes used in the process of the present in-

vention was prepared in accordance with the method set forth in United States Patent No. 2,394,874, L. S. Renzoni, filed November 5, 1942, setting forth a process for electrorefining nickel employing a chloride-containing electrolyte. A typical composition for the electrolytic nickel obtained from the chloride-containing electrolyte is as follows:

Element	Percent
Nickel	99.89 to 99.98
Cobalt	0.10 to 0.01
Copper	0.005 to 0.003
Iron	0.001
Arsenic	0.001
Lead	0.001

Although the present invention has been described in conjunction with certain preferred embodiments, such as the electrolytic nickel anodes depicted in Figs. 1 and 2, those skilled in the art will understand that various modifications thereof may be made. Thus, generally speaking, it should be understood that the present invention is applicable to the welding of component parts of electrolytic nickel anodes that are employed in electrodepositing nickel from a chloride-bearing nickel plating bath maintained under correlated conditions of pH and chloride normality falling in the area defined by curve A in Fig. 3 of the drawings. Furthermore, although a typical composition of electrolytic nickel, such as obtained from chloride-containing electrolytes, has been set forth hereinbefore for preparing the anodes, it should be understood it is not intended to limit the present invention to the composition set forth as, in practicing the present invention, the electrolytic nickel compositions generally available to the nickel electro-plating industry for use as anodes may be satisfactorily employed.

I claim:

1. The method of electrodepositing nickel with a welded electrolytic nickel anode which comprises establishing an aqueous nickel electroplating bath having a pH up to about 4.0, a chloride normality of about 0.4 to about 2.8, and a nickel sulfate content corresponding to 300 to 29 grams per liter of $\text{NiSO}_4 \cdot 7\text{H}_2\text{O}$, said pH and chloride normality being correlated to fall under curve A in the accompanying Fig. 3; immersing in said nickel electroplating bath a welded electrolytic nickel anode comprised of a plurality of portions of electrolytic nickel welded together with at least one nickel weld metal joint, said welded anode being so immersed in said bath that said nickel weld metal joint is exposed in contact with said electrolytic nickel to said nickel electroplating bath, said nickel weld metal containing about 0.1 to 2% silicon, about 0.03 to 1.3% carbon, about 0.05 to 2% titanium, up to about 2% aluminum, up to about 2% manganese, about 0.04 to 0.12% magnesium, with the balance essentially nickel constituting at least 97% of the weld metal, said electrolytic nickel being the type

produced from a sulfate-chloride electrolyte and containing about 0.001% lead, about 0.001% arsenic, about 0.001% iron, about 0.003% to about 0.005% copper, about 0.01% to about 0.1% cobalt, with the balance essentially nickel; and passing a plating current from said welded anode to a cathode immersed in said nickel electroplating bath to anodically corrode said welded anode without producing preferential corrosion of said nickel weld metal with respect to said electrolytic nickel of said welded electrolytic nickel anode.

2. The method of electrodepositing nickel with a welded electrolytic nickel anode which comprises establishing an aqueous nickel electroplating bath having a pH up to about 4.0, a chloride normality of about 0.4 to about 2.8, and a nickel sulfate content corresponding to 300 to 29 grams per liter of $\text{NiSO}_4 \cdot 7\text{H}_2\text{O}$, said pH and chloride normality being correlated to fall under curve A in the accompanying Fig. 3; immersing in said nickel electroplating bath a welded electrolytic nickel anode comprised of a plurality of portions of electrolytic nickel welded together with at least one nickel weld metal joint, said welded anode being so immersed in said bath that said nickel weld metal joint is exposed in contact with said electrolytic nickel to said nickel electroplating bath, said nickel weld metal containing about 0.1 to 2% silicon, about 0.03 to 1.3% carbon, about 0.05 to 2% titanium, up to about 2% aluminum, up to about 2% manganese, about 0.04 to 0.12% magnesium, with the balance essentially nickel, said electrolytic nickel being the type produced from a sulfate-chloride electrolyte and containing about 0.001% lead, about 0.001% arsenic, about 0.001% iron, about 0.003% to about 0.005% copper, about 0.01% to about 0.1% cobalt, with the balance essentially nickel; and passing a plating current from said welded anode to a cathode immersed in said nickel electroplating bath to anodically corrode said welded anode without producing preferential corrosion of said nickel weld metal with respect to said electrolytic nickel of said welded electrolytic nickel anode.

EDWARD JUDSON ROEHL.

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