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3,594,288 **PROCESS FOR ELECTROPLATING NICKEL ONTO METAL SURFACES** David H. Reinert, Wayne, Pa., assignor to The Boeing Company, Seattle, Wash. No Drawing. Filed July 31, 1968, Ser. No. 748,965 Int. Cl. C23b 1/00, 5/46, 5/08 U.S. Cl. 204-34

6 Claims

ABSTRACT OF THE DISCLOSURE

An adherent, wearable nickel coating is applied to a metal surface by a multistep process in which the metal surface is (a) subjected to an anodic current density in an aqueous acid treatment bath, preferably containing sulfuric acid in an amount between about 20 and 30% by volume, at a bath temperature of 40 to 85° F. and a current density of 75 to 250 a.s.f.; (b) coated with a thin nickel base coat by means of a nickel strike applied from 20an electroplating bath preferably containing 30 to 34 oz./gal. of nickel chloride and 9 to 12% by volume of hydrochloric acid, and utilizing a 99% minimum purity nickel anode and a cathodic current density between about 50 and 100 a.s.f., and (c) then applying a nickel 25coating of the desired thickness to the metal surface in a nickel sulfamate plating bath maintained at a temperature of 110 to 125° F. and at a pH of 3.5 to 4.2. The nickel sulfamate plating bath preferably contains 62 to 66 oz./ gal. of nickel sulfamate (27 to 31° Bé.), 0.7 to 2.0 oz./gal. 30 nickel chloride, 4 to 5 oz./gal. boric acid, 1 to 3 oz./gal. naphthylene trisulfonic acid, and a sufficient quantity of an organic wetting agent to maintain the bath surface tension at between about 20 and 60 dynes/cm.². A 99% minimum purity nickel anode is used in the nickel sulfamate plating bath and plating is carried out at a current density preferably between 60 and 100 a.s.f.

This process can be applied to various metal surfaces including copper, brass and both high and low carbon steels. The resultant article has an adherent, we arable $_{40}$ nickel coating having an excellent combination of hardness, ductility and compressive stress properties.

Many attempts have been made in the past to provide an adherent and "wearable" coating on metals such as 50steel. The provision of such nickel coatings has been of particular interest to those dealing with air foil sections such as propeller blades for airplanes, rotor blades for helicopters, and the like.

These parts, which are normally made of stainless steel, 55 are subjected to severe corrosion, abrasion, and fatigue in operation. Thus, take-offs and landings of aircraft such as helicopters from dusty areas, contact of rotor and propeller blades with water and salt water spray, and other adverse operating conditions cause extensive corro-60 sion and erosion problems for the blades. Further, the blades are often subjected to severe dynamic stresses and vibrations which produce fatigue failures.

Protective coatings for such blades must have outstanding combinations of hardness, ductility, and compressive 65 stress. Blades having these properties and, therefore, achieving resistance to the elements and shocks encountered in use, are said to have good "wearability."

It has been found, for example, that helicopter rotor blades having a combination of a hardness between about 70 450 and 550 d.p.h., a ductility exhibited by passing of the ¹/₄-inch mandrel test (ASTM #B-456), and a compres-

sive stress between about -3,000 and -15,000 p.s.i., achieve outstanding endurance and resistance to erosion, corrosion, and fatigue.

It has been heretofore recognized that the provision of nickel coatings on rotor or propeller blades provide such blades with desirable endurance properties. However, great difficulties have been encountered in prior art attempts to produce adherent nickel coatings on metal blades of this type. The provision, on a reproducible basis, of coat-10 ings which are both adherent and provide good wearability properties is a goal which has long existed for experts in the electroplating field.

It is accordingly a primary object of this invention to provide an adherent nickel coating on a metal surface 15 having good wearability properties.

It is a further object of this invention to provide a nickel coating for various metal surfaces, including both low-carbon and high-carbon steels, which is highly adherent to the base metal and provides the base metal with a coating having a good combination of hardness, ductility, and compressive stress.

An object of a specific embodiment of the present invention is to provide improved nickel erosion strips for aircraft rotor blades and to thereby provide blades with outstanding corrosion, erosion, and fatigue resistance.

Additional objects and advantages of this invention will be set forth in part in the description that follows, and in part will be obvious from the description, or may be learned by practice of the invention, the objects and advantages being realized and attained by means of the methods, processes, articles, and improvements particularly pointed out in the appended claims.

To achieve the foregoing objects and in accordance with its purpose, this invention provides a process for 35 applying an adherent, wearable nickel coating to a metal surface by a multistage electroplating process. In accordance with this process, the metal surface is first treated by electrolysis in an aqueous acidic electrolyte, and then coated with nickel in two stages of electroplating, the first being a brief nickel strike and the second applying an adherent nickel coating of the desired thickness to the thin, nickel coated base metal surface.

In this process the metal surface to be coated is inserted into an aqueous acid bath as an anode and an anodic current density of about 75 to 250 amperes per square foot (a.s.f.) is applied to the metal surface at a bath temperature of 40 to 85° F. Following this treatment, a small amount of nickel is deposited on the metal surface by inserting the metal surface to be plated into an electroplating bath as a cathode and passing a cathodic current density of about 50 to 100 a.s.f. from a nickel containing anode to the metal surface. The electroplating bath used in this nickel strike step comprises an aqueous acid solution containing about 20 to 40 oz./gal. of an ionizable nickel salt, and is maintained at a temperature between about 40 and 85° F. during electroplating.

The metal surface, containing a thin coating of nickel from the first electroplating bath, is then inserted in a nickel sulfamate plating bath as a cathode. A direct electric current of at least about 40 a.s.f. is passed from a nickel containing anode to the metal surface in the nickel sulfamate bath for a time sufficient to build up the desired thickness of nickel on the metal surface.

The nickel sulfamate plating bath is maintained in a critical temperature range of 110 to 125° C. and a critical pH range of 3.5 to 4.2 during electroplating. The sulfamate plating bath is an aqueous based bath containing:

40 to 80 oz./gal. nickel sulfamate

0.5 to 3.0 oz./gal. of an ionizable metal salt

4 to 5 oz./gal. boric acid, and

1 to 5 oz./gal. naphthylene trisulfonic acid

This invention relates to electroplating processes. More $_{45}$ particularly, it relates to a process for providing an adherent, wearable, nickel coating on a metal surface by a multistep electroplating process.

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It is to be understood that both the foregoing general description and the following detailed description are exemplary and explanatory but are not restrictive of the invention.

The nickel plating process of this invention has its primary utility in the application of adherent and wearable nickel coatings to steel, and the adherent nickel coatings can be applied in accordance with this invention to both low-carbon and high-carbon steels. The application of such coatings to high-carbon stainless steels used in pro- 10 peller and rotor blades for aircraft are of particular commercial importance.

It is to be understood, however, that the process of the present invention is not limited to the application of nickel coatings to steel substrates. This process can also 15 be utilized to apply adherent, wearable nickel coatings to other metal surfaces, such as copper and brass. Thus, while the present invention will be described primarily herein with regard to the application of nickel coatings to steel substrates, these teachings are also intended to 20 apply to the application of such coatings to surfaces constructed of these other metals.

The first required step of the present process comprises the treatment of the metal surface to be coated in an electrolytic, acidic treatment bath. In accordance with 25preferred practice, however, the metal surface is precleaned before being treated in the acid bath.

This precleaning is carried out in an electrolytic, alkaline cleaning bath in a conventional manner, to remove oils and other contamination from the metal surface to be coated. Conventional alkaline degreasing agents based on sodium hydroxide or other strongly alkaline materials can be used in this precleaning step. A commercially available cleaner which can be satisfactorily used is Clepo 42s produced by Gumm Co., Kerny, N.J. 35

The alkaline cleaner is used in a conventional manner, such as at a bath temperature of about 150 to 180° F. and a cathodic current density of about 50 to 150 a.s.f. or as is common practice, 6 to 12 volts potential for a period of about 2 to 4 minutes. None of these conditions 40 are critical and the alkaline cleaning bath is merely an optional step in the present process.

If the alkaline cleaning step is used, the metal surface should be water-rinsed following its removal from the alkaline treatment bath. The metal is preferably rinsed 45with cold water to achieve thorough removal of alkaline material from its surface.

The acid bath used in the first required step of the present process comprises an aqueous solution of sulfuric or hydrochloric acid. If hydrochloric acid is used, the 50 electrolyte solution comprises about 5 to 40% by volume hydrochloric acid, while if sulfuric acid is used, the solution comprises about 10 to 50% by volume of sulfuric acid.

The use of a sulfuric acid electrolyte is preferred, since problems of surface smutting are more readily avoided when sulfuric acid is used. An aqueous acid bath comprising about 20 to 30% by volume sulfuric acid is considered optimum in the present process.

The metal surface to be coated is inserted in the acid 60 treatment bath as an anode and then subjected to an anodic current density of about 75 to 250 a.s.f., preferably about 150 to 200 a.s.f. The time utilized for this electrolysis is not critical and can be varied widely depending largely on the thickness of the metal part being treated. Generally, electrolysis times of about 2 minutes should be used to secure optimum results, with time periods of about 1 to 3 minutes being advantageous.

The acid bath etches the metal surface, removing sur-70face oxides to prepare the surface for subsequent electroplating. The action of this bath is not limited to oxide removal, however, and the adherence of the nickel plating of this invention to the metal substrate is greatly enhanced by the treatment of the surface in the acid bath 75 is initiated.

in accordance with the process conditions described herein.

The metal surface functions as the anode in the acid electrolysis bath and anodic electrolysis is preferably used exclusively. It is possible, however, to initiate electrolysis cathodically and then reverse the electrolysis to anodic. It is to be understood, therefore, that when it is stated herein, and in the appended claims, that anodic electrolysis is used, this expression is intended to include procedures where cathodic electrolysis is used initially and then the electrolysis is reversed.

In accordance with the invention, electrolysis of the metal surface in the acid bath is carried out in a critical temperature range between 40 and 85° F., and preferably at bath temperatures between 40 and 74° F. The use of bath temperatures in this range is necessary to achieve the desired later adhesion of the nickel coating to the steel or other metal substrate, and the use of temperatures in this range affords one of the bases for the improved results achieved by the present process.

When the metal surface to be coated is removed from the acid electrolysis treatment bath, it is preferably waterrinsed to remove any residual amounts of alkaline or acid material present on the surface and thereby prevent contamination of the electroplating bath used in the next step of the process. The water used in this rinse step may be cold or warm water, and is preferably cold water.

The second required step of the present process is the deposit of a thin film of nickel on the metal surface by a nickel strike from an electroplating bath. In this step, the metal surface to be plated is inserted in an electroplating bath containing an ionizable nickel salt as a cathode and a cathodic current density of about 50 to 100 a.s.f. is passed to the metal surface from either a nickel-containing consumable anode or a nonconsumable, carbon anode.

Only a small amount of nickel is deposited on the metal surface during this step. The provision of this nickel strike is important to the present process, however, in that it protects the oxide-free metal surface and forms a base for the nickel coating applied in the final step of the process. The use of this nickel strike step is highly important in promoting adhesion of the nickel coating to the steel or other metal substrate in accordance with the present process.

The nickel strike is carried out in an electroplating bath which comprises an aqueous acid solution containing an ionizable nickel salt. The acid used in the bath is preferably hydrochloric acid, and the optimum concentration of HCl is about 9 to 12% by volume.

The ionizable nickel salt is present in the bath in an amount between about 20 and 40 ounces per gallon, preferably about 30 to 34 ounces per gallon. Nickel chloride is the preferred ionizable nickel salt for use in this first electroplating bath.

The electroplating bath is maintained at a temperature of 40 to 85° F. during the nickel strike, and is preferably maintained at about room temperature, i.e., 68 to 74° F. The use of temperatures above 85° F. substantially decreases the adhesion of the nickel coating of this invention to the metal substrate and thus must be avoided.

The nickel strike is carried out using cathodic electrolysis, although the electrolysis may be initiated anodically and then reversed. The latter procedure complicates the process and the use of cathodic electrolysis exclusively in the nickel strike step is preferred.

To achieve optimum performance, the metal surface should be initially inserted in the hydrochloric acid-nickel chloride bath in an absence of current. This allows the hydrochloric acid in the bath to exercise a washing and neutralizing action on the surface, providing optimum results. After the surface has been allowed to stand in the HCl-NiCl bath for a short time, cathodic electrolysis

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A current density of about 50 to 100 a.s.f. is preferably applied to the metal surface through the HCl-NiCl electrolyte from a nickel containing anode. A current density of about 74 to 76 a.s.f. achieves particularly good results.

The nickel anode should have a minimum purity of 99% by weight nickel. It may contain minor amounts of sulfur, silicon, copper and organic impurities in aggregate amounts up to about one percent. A non-consumable, carbon anode can be used in place of the nickel anode. 10

The time of electrolysis in the nickel strike step is not critical, so long as the purpose of providing a thin nickel film of from about 0.0001 inch to 0.0005 inch on the metal surface is achieved. Electrolysis must be continued for a time sufficient to provide the desired pro- 15 tective nickel base on the metal substrate, but the major thickness of nickel coating is applied from the nickel sulfamate bath of the third required step of the present process, and hence it is not desirable to apply too great a thickness of nickel in the nickel strike step. Gener- 20 ally, electrolysis times on the order of about 1 to 4 minutes are satisfactory in the nickel strike step, with a time of about two minutes being optimum.

Following the nickel strike step, the coated metal surface is preferably again washed with cold or warm water 25 to prevent the transport of contaminants from the nickel strike plating bath into the sulfamate plating bath of the final step of the process.

A nickel sulfamate electroplating bath is used to apply the desired thickness of adherent nickel coating to the 30 steel or other metal surface in accordance with this invention. The sulfamate bath is an aqueous bath, preferably prepared with deionized water.

The basic ingredient of this bath is nickel sulfamate, which supplies ionized nickel for plating on the metal 35 surface. The plating bath should contain nickel in an amount between 8 and 14 ounces per gallon, by analysis, and preferably about 8 to 12 ounces per gallon. To supply this nickel, nickel sulfamate advantageously is present in the plating bath in amounts between 40 and 40 80 ounces per gallon, and an optimum plating bath contains about 62 to 66 ounces per gallon of nickel sulfamate (27 to 31° Bé.).

Satisfactory nickel sulfamate compositions are available commercially, for example, as M & T sulfamate from $_{45}$ Metals & Thermit Corporation of Matawan, N.J., or as Barrett sulfamate from Allied Research Company of Baltimore, Md. Any commercially available nickel sulfamate which is controlled in the amounts of contaminants present by prior substantial removal of iron and similar 50metals, can be satisfactorily used in the sulfamate plating bath of this invention.

The sulfamate plating bath also contains an ionizable metal salt in an amount between about 0.5 and 3.0 oz./ gal. Ionizable metal salts such as one or more of sodium 55 chloride, sodium fluoride, sodium bromide, potassium chloride, potassium bromide, and the like, can be used to supply this necessary ingredient. Nickel chloride is a particularly preferred ionizable metal salt for use in the sulfamate bath of the present process. 60

The function of the ionizable salt in the bath is to promote dissolution of the consumable, nickel anode used in the electroplating bath, and nickel chloride has been found to be a particularly efficient anode dissolution agent. 65

The concentration of the ionizable metal salt in the sulfamate bath must be closely controlled, and the presence of 0.5 to 3.0 oz./gal. of this salt is necessary to the proper function of the present process. The use of about 0.7 to 2.0 ounces per gallon of nickel chloride 70 or other ionizable metal salt is preferred.

Boric acid is also present in the sulfamate plating bath of this invention, in amounts between about 4 and 5 oz./gal. Boric acid acts as a buffer in the plating tive in providing a smooth, uniform, and non-grainy plated surface.

Another important ingredient of the sulfamate plating bath of this invention is naphthylene trisulfonic acid. This ingredient is particularly important in that it controls the compressive strength of the plated surface produced by the present process, and allows the production of plated objects having the wearability properties desired in this invention. To achieve this function, naphthylene trisulfonic acid should be present in an amount between about 1 and 5 oz./gal. of the sulfamate solution, preferably, about 1 to 3 oz./gal. A naphthylene trisulfonic acid content of about 2 oz./gal. is considered optimum.

In accordance with the preferred practice of the present process, a sufficient amount of a wetting agent is added to the sulfamate plating solution to provide a plating bath having a surface tension between about 20 and 60 dynes/cm.². Baths having a surface tension in this range have been found to be more amenable to proper agitation during electroplating, and to therefore allow the production of superior plated products. Wetting agents such as sodium lauryl sulfate and commercially available equivalents thereof can be used to achieve this purpose.

It has been found that the pH of the nickel sulfamate plating bath is critical to the operation of the present process in the desired manner. The pH of the sulfamate plating bath is therefore maintained between 3.5 and 4.2, with an optimum pH being about 3.7. A pH controlling agent such as sulfamic acid, boric acid, or nickel carbonate is added to the plating bath, as needed, to maintain the pH of the bath into this operating range.

The desired thickness of nickel coating is applied to the metal surface from the sulfamate bath by passing a direct electric current of at least 40 a.s.f. from a nickelcontaining anode to the metal surface, which is maintained in the bath as the cathode. The cathodic current density in the sulfamate plating bath is generally maintained in the range of 40 to 200 a.s.f., preferably 60 to 100 a.s.f., optimumly about 80 a.s.f.

The particular current density selected is a function of the agitation maintained in the plating bath, and applicant does not wish to be limited to the use of any particular current density. Through the use of relatively new speed plating techniques, it may be possible to use extremely high current densities in excess of 1,000 a.s.f. if proper circulation techniques are used. However, for present operating techniques, the current density levels set forth above are generally considered optimum.

Cathodic electrolysis is utilized exclusively in the sulfamate plating bath of this invention, and no reversing of the electrolysis is permitted in this step of the process.

The anode utilized in the sulfamate plating bath is a high purity nickel anode, preferably having a minimum purity of 99% nickel and preferably depolarized. The remaining one percent, or less, of the anode may be made up of such impurities as sulfur, silicon, carbon, cobalt, and organic materials. Anode bar stock or chips may be used.

The nickel sulfamate bath is maintained in a narrow and critical temperature range during electroplating. The temperature of the bath must be maintained in the range of 115 to 125° F. in order to produce the adherent, wearable coatings desired from the present process. If the temperature of the sulfamate plating bath is increased above 125° F., the coating tends to be streaky and nodular, giving a bad plating appearance and a less adherent and less fatigue resistant plating. On the other hand, use of temperatures below 110° F. causes the plating to be brittle and to have poor fatigue resistance.

The electroplating from the nickel sulfamate plating bath is carried out for a period of time sufficient to apply a nickel coating of the desired thickness on the metal surface. The time required is normally predetermined solution and its presence in the required amounts is effec- 75 through a check of the rate of nickel build-up on the

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test sample before the surface to be plated is inserted into the sulfamate bath.

By way of illustration, a nickel coating thickness on the order of 0.015 inch can usually be produced in a plating time of about 2 to 3 hours.

After the plated part is removed from the nickel sulfamate bath, it is again water rinsed to remove residual salts from the surface. Either cold, warm, or hot water can be used in this rinse step, but the use of hot water is preferred since it facilitates and speeds removal of these 10 residual salts.

The plating process of this invention can be carried out either by masking the surface to be plated to produce the desired plating configuration, such as an erosion strip on a helicopter blade, or, in some instances, the entire 15 piece can be plated and plating removed from certain areas by machining or the like, if desired. The plating can be of any desired thickness, and this thickness can be varied across the area plated through proper masking and positioning of the metal surface in the plating bath 20 or by the use of conforming anodes.

Practice of the present process produces metal surfaces provided with adherent and wearable nickel coatings having excellent hardness, ductility and compressive stress properties.

The process therefore is highly useful in the provision of corrosion, erosion, and fatigue resistant nickel coatings on metal parts.

For a clearer understanding of the invention, specific examples of it are set forth below. These examples are 30 merely illustrative, and are not to be understood as limiting the scope and underlying principles of the invention in any way.

EXAMPLES

In each of the following examples various metal surfaces are treated in accordance with the process of the present invention. In each example, the metal surface is first precleaned with an alkaline electrolytic cleaning bath. The metal surface is inserted as a cathode in an aqueous solution containing about 10 oz./gal. of Clepo 42s alkaline cleaner. A non-consumable anode or the steel cleaning tank itself is used in the bath as an anode and a current density of about 84 a.s.f. is passed from the anode to the metal surface cathode for a period of about 2 to 4 minutes at a bath temperature of about 180° F.

The cleaned and degreased sample is then cold-water rinsed using tap water to a waterbreak time of 60 seconds.

The rinsed metal surface is then inserted in a sulfuric acid etching bath in accordance with the first required $_{50}$ step of the present process. The acid concentration of this bath, and the bath temperature, the current density used in electrolysis, and the time of electrolysis are all set forth for each example in Table 1 below. In each case the metal surface is inserted in the bath as an anode and $_{55}$ the electrolysis is anodic. The sulfuric acid used in the acid etching bath in each example is 66° Bé acid.

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TABLE 1.-ACID ETCH CONDITIONS

Ex. No.	Metal of substrate	Acid conc., vol. percent	Bath temp., ° F.	Current density, a.s.f.	Time of electrol- ysis, minutes
1	301 stainless steel 1	10	60	180	2
2	do.1	10	78	180	2
3	do.1	25	72	120	2
4	do.1	25	70	120	2
5	do.1	25	70	180	2
	do.1	50	48	180	2
	316 stainless steel 1	30	74	180	2
	do.1	30	74	180	2
9	4130 steel 2	10	40	180	1
10	do.2	10	40	180	1

¹ High chrome-nickel S. S. ² Chrome-molybdenum high tensile steel.

15 After this acid electrolysis is completed, the metal surface is again cold-water rinsed to a waterbreak of 60 seconds. Each metal sample is then inserted in a nickel-strike electroplating bath containing the amounts of nickel chloride and hydrochloric acid indicated for each 20 example in Table 2. The metal surface is inserted in this electroplating bath as a cathode using a consumable 99% minimum purity nickel anode. Cathodic electrolysis is then carried out at the current density and bath temperature set forth in Table 2 for each example, for the period 25 of time also shown in Table 2.

TABLE 2.- NICKEL STRIKE CONDITIONS

Example number	Acid conc., oz./gal.	Nickel salt conc., oz./gal.	Bath temp., °F.	Current density, a.s.f.	Time of electrol- ysis, min.
1	16	32	70	75	2
2	16	32	70	75	2
3	16	32	74	75	2
4	16	32	74	75	2
5	16	32	68	75	2
6	16	32	68	75	2
7	16	32	48	72	2
8	16	32	48	72	2
9	16	32	68	72	2
10	16	32	68	72	2

The metal surface containing a thin, nickel-strike film, is removed from the nickel-strike electroplating bath and again cold-water rinsed to waterbreak of 60 seconds in each example.

Each sample is then inserted in a nickel sulfamate plating bath having the composition set forth in Table 3. With the metal surface as the cathode, and using a 99% minimum purity nickel anode, cathodic electrolysis is carried out in the nickel sulfamate electroplating bath at the current density and for the time period set forth in Table 3. During electrolysis, the bath temperature and pH are maintained at the values also set forth in Table 3.

In each example, a sufficient amount of Anti-Pit, organic wetting agents (produced by Metals and Thermit Corporation of Matawan, N.J.), is added to the nickel sulfamate bath prior to electrolysis to provide a bath having the surface tension set forth in Table 3.

TABLE 3.- NI SULFAMATE BATH OPERATION

	Bath composition, oz/.gal.				Bath		Bath surface	Current	Time of	
– Example number	Ni sul- famate	Ni chlorido	Total Ni	Boric acid	N.T.A.1	Temp., F.	$_{\rm pH}^{\rm Bath}$	tension, dynes/cm.2	density, a.s.f.	electrol- ysis, min.
1 2 3 4 5 6 7 8 9 10 10	$\begin{array}{c} 62\\ 63\\ 62\\ 62\\ 62\\ 62\\ 63\\ 63\\ 62\\ 64\\ 62\\ 62\\ 62\\ \end{array}$	$\begin{array}{c} 0.80\\ 0.86\\ 0.89\\ 0.89\\ 0.896\\ 1.0\\ 1.2\\ 0.88\\ 1.2\\ 1.2\\ 1.2 \end{array}$	$\begin{array}{c} 9,70\\ 10,07\\ 10,0\\ 10,02\\ 10,02\\ 10,02\\ 9,70\\ 10,45\\ 10,10\\ 10,10\end{array}$	$\begin{array}{r} 4.7\\ 4.5\\ 4.71\\ 4.71\\ 4.71\\ 4.76\\ 5.0\\ 5.0\\ 4.96\\ 5.0\\ 5.0\\ 5.0\end{array}$	$1.3 \\ 1.5 \\ 1.3 \\ 1.3 \\ 1.58 \\ 1.50 \\ 1.50 \\ 1.8 \\ 1.0 \\ 1$	$125 \\ 125 \\ 118 \\ 116 \\ 118 \\ 118 \\ 118 \\ 118 \\ 118 \\ 118 \\ 1120 \\ 120$	3. 70 3. 65 3. 70 3. 70 3. 70 3. 70 3. 70 3. 70 3. 70 3. 70 3. 70	38 42 28 26 38 38 32 38 38 38	80 80 80 80 54 60 80 80 80 80	135 135 135 135 135 150 360 135 120 120

¹ Naphthylene trisulfonic acid.

After the nickel-plated metal surface is removed from the nickel sulfamate plating bath, it is subjected to a hot-water rinse to remove residual salts present on the plated surface.

As illustrated by Table 4, nickel plating runs carried out following the process of this invention produce adherent nickel coatings having good ductility, hardness and compressive stress properties on various metal substrates.

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TABLE 4.-PRODUCT PROPERTIES

Example number	Metal of substrate	Adherence of Ni plating	Ductility of Ni plating 1	Hardness of Ni plat- ing, DPH	Compressive stress of Ni plating, p.s.i.
1					9,800
2	do				-10,100
3	do	do	do	- 539	-15,600
	do				-10.100
5	do	do	do	- 539	-10,800
6	do	do	do	- 503	-10,100
7	. 316 stainless steel	do	do	- 515	-10,100
8	do	do_	oh	464	-14,200
9	4130 steel	do	do	474	-10,100
10	do	do	do	474	-10,100

¹ A rating of "good" indicates that the sample passed the ¹/₄-inch mandrel test, ASTM No. B-456.

This invention in its broader aspects is not limited to the specific details shown and described, but departures 25may be made from such details without departing from the principles of the invention and without sacrificing its chief advantages.

What is claimed is:

 $\mathbf{30}$ 1. A process for applying an adherent, wearable nickel coating to a stainless steel surface, which consists essentially of:

- (a) inserting a stainless steel surface as an anode into an aqueous acid bath containing 10 to 50% by volume of sulfuric acid or 5 to 40% by volume of hy- 35 drochloric acid, and applying an anodic current density of about 75 to 250 a.s.f. to said surface at a bath temperature between about 40 and 85° F.;
- (b) depositing a film of nickel about 0.0001 to 0.0005 40 inch thick on the stainless steel surface by inserting said surface in an electroplating bath as a cathode and passing a cathodic current density of about 50 to 100 a.s.f. to said stainless steel surface from a nickel anode containing at least about 99% by weight of nickel, said electroplating bath comprising an aqueous solution containing 9 to 12% by volume of hydrochloric acid and 20 to 40 oz./gal. of nickel chloride, and being maintained at a temperature between about 40 and 85° F. during electrolysis; and
- 50(c) applying an Ni coating of the desired thickness to said stainless steel surface by passing a direct electric current of about 40 to 200 a.s.f. from an anode containing at least about 99% by weight of Ni to said surface as a cathode in an aqueous nickel sul-55 famate plating bath containing:

0.5 to 3.0 oz./gal. of nickel chloride

4 to 5 oz./gal. boric acid

- 1 to 5 oz./gal. naphthylene trisulfonic acid, and
- an amount of an organic wetting agent sufficient to provide a bath having a surface tension between 20 to 60 dynes/cm.2;

said nickel sulfamate plating bath being maintained at a temperature of 110 to 125° F. and a pH of 3.5 to 4.2 during electroplating.

2. The process of claim 1 in which:

- (a) a current density of about 150 to 200 a.s.f. is applied to the stainless steel surface in the acid bath of step (a) at a temperature of about 40 to 74° F.;
- (b) a current density of about 74 to 76 a.s.f. is applied

to the stainless steel surface in the nickel strike of step (b) at a temperature of about 68 to 74° F.; and (c) a cathoic current density of about 60 to 100 a.s.f. is used in the nickel sulfamate bath of step (c).

3. The process of claim 2 in which the stainless steel surface is rinsed with cold water following both the acid treatment of step (a) and the electroplating of step (b), and is rinsed with hot water following the electroplating of step (c).

4. The process of claim 2 in which the pH of the nickel sulfamate plating bath is about 3.7.

5. The process of claim 2 in which the metal surface is precleaned in an electrolytic alkaline cleaning bath and then water rinsed before said metal surface is inserted in the sulfuric acid bath of step (a).

6. The process of claim 1 in which the metal surface is a low carbon steel rotor blade for an aircraft.

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⁴⁰ to 80 oz./gal. nickel sulfamate