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ALUMINUM OXIDATION RESISTANT COATING FOR NICKEL AND COBALT BASE ALLOY PARTS

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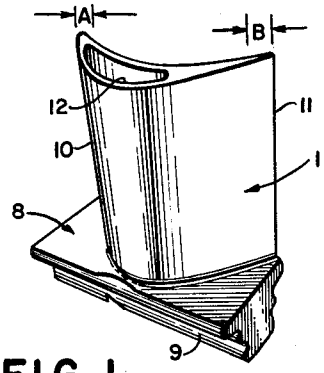


FIG. 1.

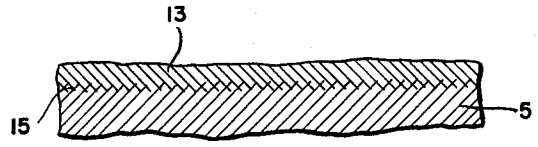


FIG. 2.

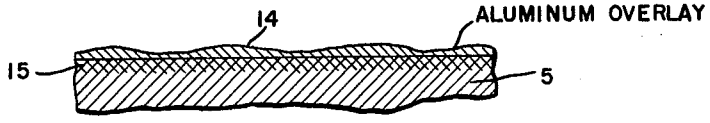


FIG. 3.

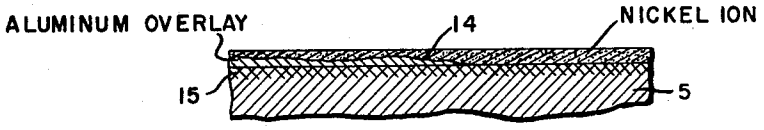


FIG. 4.

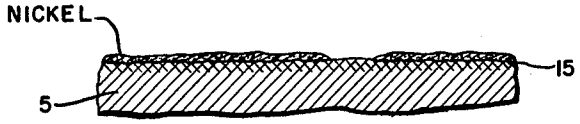


FIG. 5.

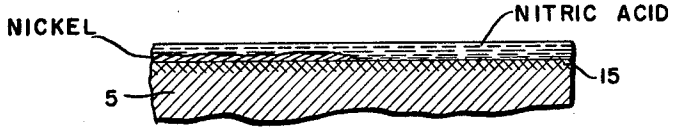


FIG. 6.

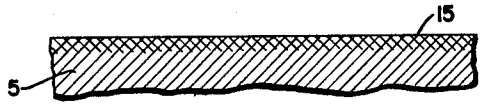


FIG. 7.

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ALUMINUM OXIDATION RESISTANT COATING FOR NICKEL AND COBALT BASE ALLOY PARTS

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This invention relates to a method for the surface preparation of various types of nickel and cobalt base alloy parts. The process is particularly useful as a surface treatment for vanes or buckets made of these materials which are commonly used in conjunction with rotors of turbines in the gas turbine type of engine. The invention is especially directed to a method of coating the exposed surfaces of such parts with aluminum or an aluminum alloy, which method inherently provides precise control over the thickness of such coating material by accurate removal of any excess of aluminum, the result being an even, uniform and protective coating of the desired thickness. Upon completion of the procedures herein set forth the coated part thus emerges as one having a thin but even aluminum-alloy surface, the latter being in reality aluminum which has been molecularly bonded, fused or alloyed into the surface of the base material, here generally nickel or cobalt base alloys of the type mentioned.

It is of course well known in the art that various parts used in the turbine section of jet and turbo shaft engines are subjected to extremely high temperatures where oxidation of such parts under these arduous conditions is an ever present hazard, resulting in deterioration, fracture and consequent imbalance, and other destructive influences. Thus it has heretofore been proposed that alloys of the described type, which are commonly used for the fabrication of such elements as turbine vanes or buckets, be coated with aluminum or an aluminum alloy, and various processes have been directed to this end. However, the difficulty, and a serious one, has been that of control. The ever present desire has been to alloy such a coating with the base material with an exact evenness of application, and without any excess of the coating material being retained upon the surface. Precise control in this respect has heretofore been unobtainable.

Removal of excess coating material is an important and significant factor, for retention of same may result in a poorly adherent surface which will be subject to spalling during engine operation. Furthermore, excess aluminum may penetrate, or diffuse, completely through thin airfoil surfaces causing loss of strength and consequent premature component failure.

Hense the control feature mentioned in the foregoing, and having reference to an accurate removal of excess, becomes most important. Such control, to be satisfactory, must be one which provides for accurate removal despite any errors which might be introduced as a result of manipulative handling of the involved procedures. In other words, the excess must be removed accurately, evenly, to the desired degree and by methods so controlled that the alloyed surface or desired protective layer is not only evenly and thoroughly applied, but not attacked or damaged in any way during any of the procedural steps. It is thus important that control be effectuated irrespective of the part geometry or variable conditions subject to personal error, or inability to effectuate the involved conditions, on the part of the operator.

As mentioned, the procedures heretofore employed for this purpose have not attained the desideratum herein

set forth—application of an oxidation resistant coating representing an aluminum alloy surface, and representing also a surface completely free of excess aluminum coating within accurate and controlled limits.

The instant invention resolves these difficulties, and particularly those revolving about a controlled procedure wherein, by chemical replacement after dipping, the excess of the coating material is replaced by a metal lower in the electromotive series. The replaced metal is then representative of the excess, free or unalloyed aluminum and when such is removed, and as accomplished by this invention without attack upon the alloyed portion of the aluminum, the latter remains as the true coating with such excess completely eliminated. Inter alia, the salient steps of the process of this invention are briefly summarized as follows:

(*a*) Cleaning, or fluxing of the base material to assure a surface suitable for adhesion of the aluminum or aluminum alloy coating.

(*b*) Dipping in molten aluminum to achieve a coating of the aluminum or aluminum alloy, with subsequent centrifuging action, vibration or equivalent forms of agitation to remove a substantial portion of the excess, or overlay, of the coating material.

(*c*) Replacement of the remaining excess, i.e., the unalloyed portion of same by a chemical replacement reaction wherein a nickel salt substitutes the nickel thereof for the excess aluminum, the result being that the aluminum rich alloyed portion of the surface is covered with a thin coating of nickel representative of the excess, or overlay, which results after dipping.

(*d*) Finally, removing the nickel coating, which as stated, represents the overlay covering the alloyed aluminum coating, by reaction with an acid of sufficient concentration to achieve this objective, which acid is reactive with the replaced nickel but not the aluminum or aluminum alloy. The result is complete removal of firstly, the aluminum overlay by reaction with the nickel salt to substitute the nickel thereof and secondly, removal of the replaced nickel by its dissolution in a suitable acid such as nitric acid, with which is readily reacts.

It is accordingly a primary purpose of the instant invention to provide a process for the application of an oxidation resistant aluminum or aluminum alloy coating which assures accurate deposition of the required amount of said coating material by precise control over the removal of the overlay of such material.

It is a further objective of the invention to provide a method wherein the control mentioned in the foregoing is attained by a novel intermediate step wherein the overlay or excess of coating material which must be removed is replaced by a metal lower in the electromotive series, such as nickel. Such represents the essence of the herein described control system, for this intermediate step represents a chemical replacement reaction which assures complete and accurate removal of the overlay without penetration into, or removal of any of the desired coating, that is, the bonded, fused or alloyed aluminum.

A further objective of the invention is to provide such a replacement procedure as herein identified which includes an additional and accurate control system by the use of a specified inorganic acid which will react only with the replaced nickel (representing the overlay) but which, by its very nature, and particularly under the conditions herein involved, will not react with the aluminum or aluminum alloy with which the base material is coated.

And finally, an additional object of the invention is to provide a process, and with reference to the relative complexities of the art, which is simplified to the extreme, efficient in the sense that it eliminates possibility of human

error, and permits an effective oxidation and corrosion resistant coating to be obtained at minimum cost.

The process of the invention is illustrated with respect to the several accompanying figures, which are more or less diagrammatic, but which do clearly depict the fundamental sequence of steps generally set forth in the foregoing. Reference in the following description will be made to these figures, wherein:

FIGURE 1 is a perspective view of a vane or blade of a type generally utilized in the industry in gas turbine engines of the type generally found in the aircraft industry;

FIGURE 2 is a diagrammatic illustration, greatly enlarged, indicating the manner by which the coating material, aluminum or aluminum alloy, forms a metal band or surface alloy with the base material as a result of the dipping procedure;

FIGURE 3 shows the non-uniform surface of such aluminum coating, and particularly the excess thereof, induced either as an inherent result of dipping or as a resultant material after centrifuging or other agitation method to remove the great proportion of overlay;

FIGURES 4 and 5 graphically demonstrate the manner by which a nickel salt solution enters into a replacement reaction with such overlay to substitute nickel for the excess aluminum;

FIGURE 6 graphically depicts the coating of replaced nickel as being attacked by an inorganic acid which will effectuate its removal down to the alloyed aluminum rich coating material; and

FIGURE 7 is a graphic illustration of the finalized product—the bonded aluminum or aluminum alloy coating representing one of even, uniform depth, and one with the flat even surface from which the excess of the coating material, or overlay, has been completely removed.

The type of unit, of a nickel or cobalt alloy base material, which is visualized as a representative metal part susceptible of treatment by the process of this invention, is illustrated in FIGURE 1. It is made of nickel or cobalt alloy base material **5**. Such a base alloy may vary somewhat in its specific characteristics. However, certain typical alloys are preferred for manufacture of the structures here under consideration. The constituents thereof, in exemplified form, are represented in the two following tables. Table I sets fourth four examples of the metal components of four rather specifically different nickel base alloys. On the other hand, Table II identifies preferred examples, four in number, of cobalt base alloys which are likewise suitable as base alloys for the vanes or blades subject to the coating procedures of the invention.

In both tables the percentages of the component materials are stated in percentages by weight.

TABLE I

Nickel Base Alloys

[Percent by weight]

Element	Alloy 1	Alloy 2	Alloy 3	Alloy 4
Co	1.00 max		16.00–20.00	13.00–17.00.
Cr	11.00–14.00	14.00–17.00	16.00–20.00	8.00–11.00.
Mo	3.50–5.50	4.50–6.00	3.00–5.00	2.00–4.00.
Fe	2.50 max	8.00–12.00	2.00 max	1.00 max.
C	0.08–0.20	0.10–0.20	0.10 max	0.15–0.20.
Si	0.50 max	0.30 max	0.30 max	
B	0.005–0.020	0.05–0.10	0.003–0.010	0.008–0.020.
S	0.015 max		0.015 max	
Mn	0.20 max	0.10 max	0.20 max	
Zr	0.050–0.20			0.03–0.09.
Al	5.50–6.50	2.50–3.50	2.75–3.25	5.00–6.00.
Al+Ti				10.0–11.5.
P				
Ti	0.35–0.90	1.50–2.50	2.75–3.25	4.50–5.50.
V				0.70–1.20.
Cb				
Cb+Ta	1.50–2.50			
W				
Ni	Balance	Balance	Balance	Balance.
Cu			0.10 max	

TABLE II

Cobalt Base Alloys

[Percent by weight]

Element	Alloy 1	Alloy 2	Alloy 3	Alloy 4
Co	Balance	Balance	Balance	40.00–44.00.
Cr	24.5–26.5	20.00 [1]	20.0–22.0	19.00–21.00.
Mo				3.50–4.50.
Fe	2.00 max		1.50–2.50	5.0 max.
C	0.45–0.55	0.48 [1]	0.40–0.50	0.32–0.42
Si	1.00 max	0.60 [1]	0.50 max	0.30–0.90.
B		0.05 [1]		
S			0.040 max	
Mn	1.00 max	0.60 [1]	0.50 max	0.60–1.80.
Zr				
Al				
Al+Ti				
P			0.040 max	
Ti				
V				
Cb				3.00–4.50.
Cb+Ta			1.50–2.00	
W	7.00–8.00	12.50 [1]	10.0–12.0	3.50–5.00.
Ni	9.50–11.50		1.0 max	19.00–21.00.
Cu				

[1] Nominal.

A typical blade, made of the type of alloy falling within the above examples, is illustrated at **1**. It is provided with a base portion generally indicated at **S**, the latter having suitable flanges **9** of usual design which permit its affixation to the periphery of a turbine disk by known methods. This blade, as here more or less graphically depicted, represents an airfoil type of unit having the usual more or less concavo-convex cross sectional configuration. The leading edge is represented at **10** and the trailing edge at **11**. As sometimes made the airfoil portion of the unit is formed with a hollow core **12**.

The critical portions are generally indicated at A and B. These represent parts of the blade that are most susceptible to oxidation and thermal shock, and accordingly these critical portions of a typical blade or vane are the ones where it is desired that the aluminum and antioxidant layer be most efficiently applied. Areas A and B represent distances of about $\frac{3}{32}$ of an inch or 0.090 inch from the leading and trailing edges respectively, in the type of blade unit that is here referred to for exemplary purposes. At any rate, the significant factor with respect to any such type of unit as here referred to, is that the coating must be accurately and evenly applied, else, under the extreme conditions normally encountered during turbine operation, faulty performance results as a consequence of oxidation, cracking, premature failure, etc.

As an initial step in the process of this invention it is necessary that the blades or vanes be thoroughly cleaned prior to subsequent treating stages. To this end the same are vapor blasted to remove soils, light scales, ink stamps, etc. Such wet abrasive blast preferably comprises the use of aluminum oxide as the abrasive, the particle size of same being about 625 mesh. Such alumina is placed in suspension in water in relative proportions of about three pounds of the said abrasive per gallon of water. The surfaces are vapor blasted with this type of suspension at a pressure of about 30 p.s.i.

After such preliminary treatment to remove surface scale or other types of imperfection and discoloration, the parts to be coated are placed in suitable racks, fabricated of stainless steel. Where vanes of the type of FIGURE 1 are being treated, these racks are so formed as to be capable of positioning such parts in a vertical position with the core opening downwardly for all subsequent processing operations. The racks are further so designed as to not prevent the coating of any of the critical areas or parts of the vanes requiring the coating treatment. It is of course desirable that subsequent to the vapor blasting referred to, utmost care be exercised to avoid excessive handling or contamination of the surfaces after the cleaning step, and the referred to racks also achieve this purpose.

The cleaning phase of this procedure is followed by

fluxing of the parts to prepare the surfaces for ready alloying or acceptance of the aluminum or aluminum nickel alloy coating to be applied. Such fluxing involves immersion of the racked parts in a crucible containing a fused salt solution. This fluxing and preheating salt solution is preferably comprised of the halide salts of the alkali metals and aluminum. A preferred specific composition of the flux is a molten neutral salt containing 45% to 55% each of potassium chloride (KCl) and sodium chloride (NaCl) with sufficient additions of aluminum fluoride (AlF_3) and cryolite (Na_3AlF_6) to both reduce oxidation of the salt and dissolve aluminum oxide (Al_2O_3).

The crucible in which the fluxing step is conducted is preferably made of silicon nitride bonded silicon carbide. It is capable of being loaded into a vertical loading type of furnace and of maintaining the contents, here the molten fluxing salt, at a preferred temperature of about 1340° F. Depending upon certain other variable factors the temperature of this fluxing bath may vary from about 1250° F. to about 1500° F.

It is to be understood that this fluxing bath is maintained at the aforesaid temperatures, or preferred temperature of about 1340° F., prior to immersion of the parts therein. The racked parts are dipped in and permitted to remain in such fluxing bath until they have attained the temperature of such salt bath, as stated, about 1340° F.

After reaching the stated temperature the rack parts are quickly transferred to a molten aluminum or aluminum alloy bath, the molten coating material being contained in a similar type of crucible heated by a similar type of furnace. The molten aluminum is preferably of a commercial purity equivalent to Grade 9930A as set forth in ASTM specification No. B24.

The parts are permitted to remain in the molten aluminum bath for a period of about five seconds, during which time they are agitated to assure adequate access of the aluminum into all crevices and openings, etc., in each of the parts.

The molten aluminum, during this dipping step, is maintained at a preferred temperature of from about 1340° F. to 1350° F. Dipping its not conducted whenever the temperature falls out of the limits of the range of from about 1330° F. to 1360° F., although the operation is workable in this latter range.

Immersion in such a molten aluminum or molten aluminum alloy bath results in combining, by molecular affinity, of a portion of aluminum with the base material, i.e., the nickel or cobalt base alloy of the units being treated. This represented in FIGURE 2 where such base material **5** has, as a result of such dipping step, become alloyed near its surface with the aluminum. The aluminum so alloyed is represented at **15** of FIGURE 2. It will also be noted that in this figure, for explanatory purposes, the aluminum layer **13** is represented as temporarily having a smooth and even surface.

At any rate subsequent to this dipping step in the molten coating material, the coated parts are withdrawn from the aluminum melt and by means of a whip action, centrifuging or similar agitation of known type, a substantial proportion of excess aluminum, and exclusive of that actually alloyed to the base material, is removed. The coated parts are now removed from the crucible containing molten aluminum and permitted to cool to room temperature. When cooled, the parts are washed in hot water of from 180°–200° F. to remove any residual flux salts.

At this juncture of the procedure it will be noted the aluminum coating is not one of even application, at least for the purposes for which this type of vane or turbine blade here represented is designed. As graphically illustrated in FIGURE 3, the surface of the aluminum, here indicated at **14**, is represented as uneven. This may occur as an inherent result of the dipping procedure, or whip action or agitation step which removes the substantial

part of excess aluminum. In any event, it is desired, as a primary function of the process that only the alloyed portion **15** of the aluminum-nickel (or cobalt) base alloy component remain as a coating. The instant method provides for accurate removal of this excess of coating material, represented by free, or essentially unalloyed aluminum. The latter constitutes those uneven portions of the coating material as represented at **14** of this figure (FIGURE 3).

When the units have reached room temperature following the molten aluminum coating step and agitation step referred to above, they are then washed in hot water to remove flux and placed in wire mesh baskets of suitable design for immersion in an aqueous solution which will effectuate chemical replacement to replace such excess aluminum, as the excess shown at **14**, with a coating of nickel. In other words by chemical replacement, a nickel coating or nickel plate is substituted on the parts, which nickel plate is chemically equivalent to the amount of the excess aluminum coating **14**.

It is to be here observed that a replacement reaction is desired which replaces the referred to excess aluminum with a metal lower in the electrochemical series than aluminum, said metal being suitable for ultimate removal after replacement through the use of nitric acid. In the instant case, the salts of nickel and cobalt are utilized to effectuate this replacement reaction. Nickel salts other than nickel chloride are adaptable. Thus, nickel formate, nickel fluoborate, nickel perchlorate, nickel sulfate and nickel sulfamate will all effectuate such replacement reaction.

On the other hand, a cobalt chloride salt solution may be used to also effect a replacement reaction wherein cobalt is substituted for excess aluminum and such replaced cobalt is subsequently removed by nitric acid. The end result is similar to the reaction utilizing a nickel salt solution.

Modification of any of these nickel salt solutions by the addition of excess ammonium hydroxide until distinctly basic or by the addition of small mounts of acid to increase the activity of the aluminum in nickel salt solution will result in the desired replacement of aluminum by a nickel plate. As to the small amounts of acid just mentioned, hydrochloric, sulfuric, and perchloric are useful in increasing this aluminum activity.

The preferred leach solution for the described replacement reaction is nickel chloride. In the preferred process of the invention a suitable nickel chloride solution is obtained by admixing 48 ounces of nickel chloride salt per gallon of water, representing about 34.2% solution (by weight) of nickel chloride. The concentration of the solution may, however, vary from between about 10% to 65%, by weight, of nickel chloride.

In any event the cooled parts are permitted to remain in the nickel chloride solution until such parts are completely nickel plated. This is determined by the appearance of the units and also, by observation as to whether or not there is any apparent or continued chemical activity between the surface of the parts and the nickel chloride solution. It is here obvious that the reaction will continue until the replacement procedure is completed; for this reason it is unnecessary that additional manipulative conditions be observed such as the length of time the parts remain in the nickel chloride solution. The reason for this is simply that, as stated, the reaction will continue until all of the excess aluminum **14**, or that portion of the coating which is unalloyed with the base material, is replaced by nickel. It is also to be observed that the nickel substitution step can be repeated as many times as practical to achieve full replacement of the excess aluminum.

This chemical reaction, in its ionic aspects, operates on the theory that active or electro positive materials such as aluminum will replace a less active or more noble metal from a solution of its ions. Nickel, in the electro-

motive series, represents a metal lower by some seven members in the electromotive series from the metal aluminum. Therefore, it will replace aluminum in the aluminum-nickel chloride reaction herein referred to. In its ionic aspects, therefore, aluminum plus nickel ion react to produce nickel and aluminum ions, as follows:

$$Al+Ni^{++}\rightarrow Ni+Al^{+++}$$

The aqueous nickel chloride solution is quite obviously a highly ionized solution. The chemical replacement reaction is of simplified type, represented by the following characteristic equation:

$$2Al+3NiCl_2\rightarrow 3Ni+2AlCl_3$$

The replacement reaction is graphically illustrated in FIGURES 5 and 6 where the nickel chloride is seen as reacting with the excess aluminum **14**. The specified salt solution is unreactive with the alloyed aluminum **15**, and hence, the replacement reaction proceeds only with respect to the referred to excess **14**.

The result is a substantially even coating of nickel which extends from the surface thereof to the alloyed material **15**, as seen in FIGURE 5.

It is now necessary to remove the nickel plating or coating resulting from the replacement action just described, so that only the alloyed aluminum coating remains. This is done by removal of the nickel coated parts from the nickel chloride solution and, after thorough rinsing of the same in cold water, immersion of the parts in an acid solution of a type that will react with nickel, but not aluminum or aluminum alloy, and of a concentration to effectuate this reaction. Nitric acid, which is readily active with nickel, is preferred. Such acid is also unreactive with aluminum, at least under the conditions and concentrations under which this inorganic acid is utilized in this procedure. Hence the nitric acid solution effectively dissolves the nickel from the base material, leaving only such base material **5** coated with an alloyed covering of aluminum alloy **15** as the same is diagrammatically shown in FIGURE 6.

To obtain a nitric acid solution of required concentration for the purpose of this stripping action, it has been found that when two quarts of a concentrated commercial grade of nitric acid is mixed with an equal quantity of water, the proper and optimum concentration is reached.

Here again, the reaction between the nitric acid and nickel coating effectuating removal of the latter takes the following ionic form:

$$Ni+HNO_3\rightarrow Ni^{++}+NO+H_2O$$

As a balanced equation the same may be expressed as follows:

$$3Ni+8HNO_3\rightarrow 3Ni(NO_3)_2+4H_2O+2NO\uparrow$$

By this reaction the nickel metal is dissolved in the nitric acid solution, and thus removed from the surface of the metal parts. Hence only the alloyed portion **15** of aluminum remains as a surface coating over the base material.

As stated, aluminum and aluminum alloys do not dissolve in nitric acid. This is because the surface of such metal or alloy thereof becomes passivated by the relatively strong oxidizing acid of the stated concentration. Hence the instant procedure provides its own end point or control of the coating. The stripping is simply permitted to continue until the reaction ceases, at which time, all nickel has been removed.

This nickel replacement procedure, followed by the subsequent step of nickel stripping results in an aluminum rich alloy layer, graphically represented in FIGURE 6 at **15** which yields a preferred thickness of coating after diffusion heat treatment as later described. An excess coating of aluminum is considered to be any amount exceeding 0.0015 in thickness; less than 0.0005 is insufficient to achieve the desired results.

The replacement step and nitric acid stripping step can be repeated where indicated, respectively accomplishing recoating with nickel for any remaining excess aluminum, and removal of that nickel by subsequent acid treatment. When all of the excess aluminum has been thus replaced by the nickel, whether this be in one or more steps, and the nickel stripped off, further immersion of the parts in the nickel chloride solution produces no further apparent nickel plate. This demonstrates that the initially diffused coating has reached the point of alloy chemical activity or potential where further replacement does not take place.

The replacement and acid stripping steps are followed, as stated, by a cold water rinse procedure.

The units are now prepared for the subsequent diffusion heat treatment phase. To this end, they are charged into a suitable retort containing an inert gas, here preferably argon with a maximum dew point of −40° F. This inert gas is used as the atmosphere in the retort to protect the thinly coated parts from unwanted oxidation during such diffusion treatment. The latter comprises subjection of the parts while in the retort to a temperature of about 2050° F. for a period of about two and one-half hours. Such parts are supported on stainless steel mesh racks in such fashion as to prevent the parts from touching. The temperature may, under certain circumstances, vary from about 1875° F. to about 2200° F. After the stated period of time the parts are cooled to about 1300° F. maximum prior to removal from the protective argon atmosphere.

After this diffusion heat treatment procedure, the base materials are given a dry silicon carbide or aluminum oxide abrasive blast to finish the surface and to remove any minute amounts of overlay. This strong abrasive blast also acts as a test of coating durability or adhesion propensity. Such dry blast involves the use of silicon carbide of preferably 220 mesh, applied at an air pressure of from about 90 to about 100 p.s.i.

Suitable visual tests have been devised to determine, after such abrasive blasting, whether the coated parts have received a sufficient coating of aluminum or aluminum alloy. A heat tint test at about 1125° F. for a period of preferably about ten minutes, and not in excess of fifteen minutes, will result in producing a surface color on the parts which will indicate whether the parts are properly coated. This preferred temperature may be varied from about 1110° F. to about 1140° F. It has been found that a resultant "gold" or bronze color indicates a proper coating; a "silvery" color demonstrates that some excess of the coating has been permitted to remain or crazing has occurred, in which case the parts are reprocessed as necessary as per the foregoing; a "brownish" tint indicates too thin a coating; a "bluish" tint indicates either no coating at all or significant base metal oxidation. In any event by following the procedure outlined in the foregoing it will be found that a proper aluminum rich alloy layer has been formed upon the base material in most effective fashion and with attainment, as indicated by the gold surface of the tint test, of a proper and effective coating. It will also be found that crazing has been reduced to a minimum with practically none resulting from the described procedure.

The instant process represents a procedure which is in sharp contrast to methods heretofore considered wherein, after an aluminum dip for coating purposes, excess coating is attempted to be removed by application of an acid or other reagent directly reactive with the excess aluminum. These procedures, wherein excess aluminum removal is achieved by using acids which react with the aluminum, depends on solution of the aluminum and hydrogen ion replacement. Such a reaction will take place on all metals except those below hydrogen in the electrochemical series, e.g., the noble metals.

In such situation the aluminum and the hydrogen ions of the acid (such as hydrochloric acid) will react to given

aluminum ions and hydrogen gas as per the following ionic representation:

$$Al + H^{+} \rightarrow Al^{+++} + H_2$$

Although this type of reaction will dissolve any metal used as a coating if that metal be above hydrogen in the electrochemical series, the control of an aluminum coating by such acidic reaction is extremely difficult, if at all possible. This is because the end point of such a reaction can only be determined by guesswork and hence is dependent solely upon intervention by the operator. Hence the inherent non-uniformity of a dipped aluminum coating cannot be effectively nor practically rectified by such a procedure involving direct acidic attack.

It is by the use of the intermediate replacement step of this invention that a full and accurate control over removal of excess of the coating material, or overlay, is obtained. The result is achieved accurately with total elimination of any guesswork as to how much or how little of the overlay has been removed. This is the necessary result of (*a*) the replacement reaction which proceeds until all aluminum except the aluminum rich alloy layer is replaced and substituted by nickel, and (*b*) the acid stripping step which effectively removes substituted nickel. Once such overlay has been removed by the replacement or substitution reaction the latter reaction ceases for the specified acid is of that type which, being readily reactable with the previously deposited nickel coating, is totally unreactive with the aluminum or aluminum alloy rich layers previously alloyed with the base material.

While other obvious expedients and alternates may be substituted for those herein set forth, it is to be understood that the invention is not to be limited except as defined in following claims:

We claim:

1. In a process for coating an alloy base material selected from the group consisting of nickel and cobalt base alloys with a layer of oxidation resistant aluminum, the method comprising dipping said material in a molten aluminum bath whereby said layer is alloyed to said material and an excess of aluminum is deposited over said layer, reacting nickel chloride with said excess to replace said excess with nickel metal, and then reacting said nickel metal with nitric acid, whereby said nickel metal is removed.

2. In a process for coating nickel and cobalt alloy base materials with an alloyed layer of oxidation resistant coating containing aluminum, the method comprising dipping said materials in a bath containing molten aluminum whereby said layer is alloyed with said materials and an excess of aluminum is deposited over said layer, reacting a solution of nickel ion with said excess to chemically replace said excess with nickel metal, and then reacting said nickel metal with nitric acid which is reactable with said nickel metal but not reactable with said alloyed layer, whereby said nickel plate is removed.

3. The process as defined in claim 2 wherein said molten aluminum dipping step is followed by removal of said materials from said bath, and said materials are agitated to remove the major portion of said excess.

4. In a process for coating an alloy base material with an alloyed protective layer of oxidation resistant aluminum, said material being selected from the group consisting of nickel base alloys and cobalt base alloys, the method comprising fluxing said material by dipping said material in a fused fluxing salt bath, dipping said material in a bath containing molten aluminum whereby said layer is alloyed to said material and an excess of aluminum is deposited over said layer, cooling said material to ambient temperature, reacting in aqueous solution with said excess an ionizable salt of a metal selected from the group consisting of nickel and cobalt to replace said excess with said metal, and then reacting said metal with nitric acid, whereby said metal is removed and said protective layer of aluminum, with said excess removed, remains alloyed to said material.

5. The process as defined in claim 4 wherein said metal is nickel.

6. The process as defined in claim 4 wherein said metal is cobalt.

7. The process as defined in claim 4 wherein said ionizable salt is selected from the group consisting of nickel chloride, nickel fluoborate, nickel perchlorate, nickel sulfate and nickel sulfamate.

8. The process as defined in claim 4 wherein said ionizable salt is cobalt chloride.

9. The process as defined in claim 4 wherein said salt bath comprises a mixture of sodium and potassium chloride, aluminum fluoride and cryolite.

10. The process as defined in claim 4 wherein said salt bath is maintained at a temperature of from about 1250° F. to about 1500° F., and said molten aluminum bath is maintained at a temperature of from about 1330° F. to about 1360° F.

11. The process as defined in claim 4 wherein said metal is present in an amount chemically equivalent to said excess of aluminum.

12. In a process for coating turbine parts with a protective layer comprised essentially of aluminum, the steps of:

(*a*) fluxing said parts by dipping said parts in a fused halide and metal salt bath having from about 45% to 55% each of sodium chloride and potassium chloride;

(*b*) maintaining said fused salt bath at a temperature of from about 1250° F. to about 1500° F. during said dipping;

(*c*) removing said parts from said salt bath and immersing said parts in molten aluminum while maintaining said molten aluminum at a temperature of from between 1330° F. to 1360° F. whereby a portion of said aluminum is alloyed to the surface of said parts;

(*d*) removing a portion of the excess and unalloyed of said aluminum by agitation;

(*e*) removing the remaining portion of said excess by firstly, reacting said excess with an ionizable nickel salt to substitute a chemically equivalent amount of nickel plate for said remaining portion, and secondly, reacting said nickel plate with nitric acid;

whereby said excess of said aluminum is completely removed leaving only said alloyed aluminum as said protective layer.

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