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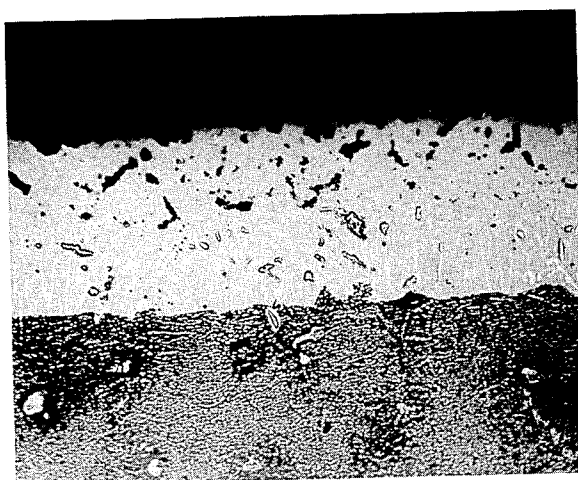
R. B. PUYEAR ET AL

3,079,276

VAPOR DIFFUSION COATING PROCESS

Filed Oct. 14, 1960

Fig. 1.



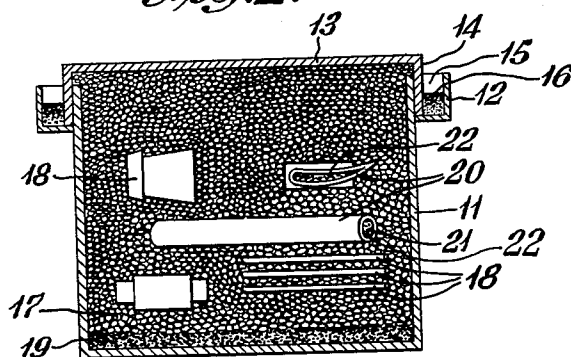
Area A

Area B

Area C

Area D

Fig. 2.



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1

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VAPOR DIFFUSION COATING PROCESS

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This invention relates to the application of diffused metallic coatings to metal articles.

Engineering parts subjected to high temperatures and corrosive atmospheres are very often required to bear a protective surface coating. The protective coating composition is selected to give increased resistance to those forces which attack the surfaces of an article, namely oxidation, corrosion and erosion, just as proper metallurgical design selects the alloy base of the article on the basis of its tensile and creep strengths. Chromizing or diffusion coating with chromium provides satisfactory protection where chromizing is applicable. Other processes apply coatings of metals other than chromium.

Protective coatings composed of alloyed metals are very desirable, in view of the superior properties possessed by many alloys over the elemental metals. Of exceptional utility as a protective coating would be a diffusion coating of certain nickel-aluminum intermetallic compounds which are reported to possess exceptional corrosion and oxidation resistance. These compounds further possess a high temperature resistance greater than that of the individual constituents of the compounds.

Present methods for producing alloy coatings on metallic articles generally require a two-step operation with a coating of one metal followed by the coating with the other metal, and then a heating to achieve the alloying. Such processes are complicated and unwieldy, and do not consistently yield the desired coating composition or thickness. It is especially difficult to coat articles having complex shapes by electrodeposition. The part of the article nearest the sacrificial electrode receives the heaviest coating and the crevices, internal cavities and corners receive little or no coating.

It is the primary object of this invention, therefore, to provide a process for diffusion coating metal articles with nickel-aluminum alloys.

It is another object of this invention to provide a method for applying nickel-aluminum diffusion coatings on metallic articles which process is simple and requires a minimum of steps.

It is also an object of this invention to provide a nickel-aluminum diffusion coating for metallic articles giving maximum oxidation, corrosion, and erosion resistance to the coated article, thereby allowing its use in more stringent applications than formerly possible.

Other aims and advantages of the invention will be apparent from the following description and the appended claims.

In accordance with these objects a process for diffusion coating metallic articles is provided comprising embedding an article to be coated in particulated prealloyed charge material consisting essentially of from 15 percent to 37 percent by weight aluminum and the balance substantially all nickel and incidental impurities, and heating said charge and contained articles in the presence of a carrier material selected from the group consisting of ammonium halides, nickel halides, and aluminum halides, said heating being conducted at a temperature from about 1400° to 1650° F. for at least four hours followed by a heating at a temperature from about 1850° to 2050° F. for at least two hours.

The articles to be coated according to this process may be alloys having a base of a metal selected from the group

2

consisting of nickel alloys and cobalt alloys. Most of the common commercial nickel-base alloys and cobalt-base alloys will accept the nickel-aluminum coating of this invention. The coatings are produced on nickel-base alloys in a preferred embodiment of this invention.

Preferably the articles to be coated are selected from those nickel-base alloys containing a predominant amount of nickel. In this regard, predominant means possessing that minimum amount of nickel which will result in the superior coating thickness and characteristics shown in the drawing and the examples following. Specifically nickel-base alloys containing a minimum of about 45 percent by weight nickel are selected.

The charge material is a crushed or shotted pre-alloyed nickel-aluminum compound of the indicated proportions and preferably in the proportion of 30 percent by weight of aluminum, and 70 percent by weight nickel. The charge material may be of a size passing a No. 2 mesh sieve and held at a No. 100 mesh sieve and is preferably that material passing a No. 4 mesh sieve and held on a No. 20 mesh sieve—the sieve sizes being those of the United States standard screen series.

The carrier materials, which serve to bring the coating material to the surface of the article to be coated, may be any of the halides of ammonium, nickel, or aluminum. Preferred carriers are ammonium fluoride, ammonium chloride, ammonium bromide, ammonium iodide, and aluminum chloride. Ammonium bromide is the carrier used in the preferred embodiment of the invention. The proportion of carrier material to charge material is from about 0.25 percent to 3 percent by weight carrier material and from 97 percent to about 99.75 percent by weight nickel-aluminum alloy.

The two-step heat treatment consisting of a heating at from 1400° F. to 1650° F. followed by heating at from 1850° F. to 2050° F. is preferably carried out at about 1600° F. for about 16 hours after which the temperature is raised to about 2000° F. for about 6 hours.

In the drawings:

FIG. 1 is a vertical cross-section, magnified 400 times, of the diffused coating produced on a metallic article by the process of this invention;

FIG. 2 is a schematic representation of a reaction chamber or retort for containing the charge material, articles to be coated, and carrier material.

Heat-resistant retort 11 is fitted with an encircling channel-shaped flange 12. Cover 13 fits over the retort with its sides 14 in the channel leaving space 15. A suitable sealing material 16 such as a fusible silicate or glass is placed in this space so that on heating of the retort, the glass melts to form a liquid seal allowing the escape of air from the retort. Contained in the retort is the charge material 17 of particulated nickel-aluminum alloy in which are embedded several parts 18 to be coated. Under the charge material is a layer 19 of the selected carrier material. Those parts 20 which have internal cavities 21 to be coated are given an addition of carrier material 22 inside the cavity. After the retort and its contents have been heated and the reaction is complete, the retort is allowed to cool. The liquid seal material hardens forming a solid seal preventing the influx of contaminating air into the retort while the contents cool.

In operation when the retort and its contents are heated, the halogen carrier volatilizers driving out the air from the retort through the liquid seal. The gaseous carrier reacts with the nickel-aluminum alloy charge material to form volatile metal halides. These compounds decompose at the surface to be coated depositing the coating elements and liberating the halogen which is then free to again enter into the coating process by reacting with the charge material.

The composition and characteristics of the diffused

coating vary with the composition of the nickel-aluminum alloy charge material. In Table I the coatings produced by different charge alloy compositions are given.

TABLE I

Effect of Charge Alloy Composition on Coating

Composition of charge		Coating thickness in mils	Coating characteristics
Ni	Al		
40 60	60 40	11	Poor, mainly NiAl_3 . Thick coating of Ni_3Al having good oxidation resistance but very brittle.
70	30	2	Ductile coating having excellent oxidation resistance. Cross-section shown in Fig. 1.
87	13	0.9	Thin coating of Ni_3Al having marginal oxidation resistance.

As seen in Table I the composition of the alloy charge material affects the composition and characteristics of the coating. The various intermetallic compounds of aluminum and nickel have different properties—some being ductile, others very brittle, etc. In Table II the comparative properties of some of these nickel-aluminum compounds are given.

TABLE II

Properties of Compounds of Nickel and Aluminum

Compound and its composition	NiAl (70Ni—30Al)	$\text{NiAl}+\text{Ni}_3\text{Al}$ (83Ni—17Al)	Ni_3Al (87Ni—13Al)
Tensile Strength, p.s.i.			
At Room Temp.	20,000	83,800	50,600
At 1,500° F.		52,900	
Hardness: Rockwell "A" scale.	71	69	62
Density: g./ml.	6.1	6.99	7.39
Elongation, Percent: Room Temp.	0	0	6.3
Oxidation Resistance.	Excellent	Good	Fair.
Wear and Erosion Resistance.	do.	do.	Good.

As seen in Table II the intermetallic compound NiAl has the greatest oxidation and wear resistance but possesses little ductility which makes it less satisfactory as a diffusion coating on a normally ductile article. The compound Ni_3Al , on the other hand, has excellent ductility, about 6.3 percent, which would allow for excellent bonding to nickel-base alloys which usually possess the same degree of ductility. However Ni_3Al does not have as high an oxidation resistance as NiAl . Obviously the best coating would combine the superior ductility of Ni_3Al and the high oxidation resistance of NiAl . A coating having these properties is produced when the alloy charge material has a composition consisting of from 15 percent to 37 percent aluminum and from 63 percent to 85 percent nickel. In FIG. 1 a cross-sectional view is shown of the diffusion coating produced using a 30 percent aluminum-70 percent nickel alloy charge material on a nickel-base alloy containing 5 percent molybdenum, 12 percent chromium, 6 percent aluminum, 70 percent nickel plus small amounts of other materials.

By X-ray diffraction methods, it is shown that the coating illustrated in the drawing changes in composition from its surface to the base metal. The entire cross section is mainly nickel with the aluminum content concentrated more at the surface, area A, and gradually diminishing to only traces at area D.

The surface layer, area A, is rich in the compound NiAl . In the intermediate zone, area B, the compounds NiAl plus Ni_3Al is found. In the boundary zone, area

C, NiAl plus Ni_3Al are also found but there is a heavier concentration of Ni_3Al . In area D small amounts of aluminum have diffused deep into the base metal.

The surface layer, area A, which is actually exposed to the corrosive atmospheres is made up of NiAl which has the highest hardness, oxidation, and corrosion resistance. The intermediate zone, area B, contains NiAl plus Ni_3Al , the strongest nickel-aluminum compound, which provides the required tenacity to the entire coating. The boundary zone, area C, is richer in Ni_3Al which, because of its strength and ductility, gives optimum bonding with the base metal. This compound Ni_3Al with a room temperature elongation of 6.3 percent is highly compatible with nickel alloy bases for most nickel-base alloys exhibit an elongation of from 4.5 to 10 percent.

It is to be noted that the use of the terms "layers" or "zones" does not here mean separated and distinct layers or laminations, but rather means areas or zones of the coating as a whole where one nickel-aluminum compound is more plentiful than another. For example, areas B and C both contain the compound NiAl plus Ni_3Al but area C is richer in the Ni_3Al end of the compound. Additionally throughout the entire coating minor amounts of other intermetallic compounds, e.g., Ni_2Al_3 and NiAl_3 , may be present. Furthermore it is to be noted that the coating is continuous through the entire cross section and is thoroughly diffused into and integral with the base metal.

The formation of the NiAl surface layer is the result of the use of a charge material having the composition 15 to 37 percent aluminum and 63 to 85 percent nickel. It can be seen from a phase diagram of the aluminum-nickel system that this composition range is also the composition range of the compound NiAl at the temperatures at which the heating steps of this invention are carried out—about 2000° F. Under equilibrium conditions the surface coating on the article forms as NiAl because of the presence of that material in the charge. At room temperature the composition range of 15 to 37 percent aluminum-balance nickel will also contain other nickel-aluminum compounds but when raised to the reaction temperature the mixture will be mainly NiAl . The NiAl compound exists alone between the composition range 20 to 38 percent aluminum-balance nickel and this is a preferred range. The specific composition used—30 percent aluminum, 70 percent nickel—approximates the stoichiometric relationship of the compound NiAl .

The superior coating described above is produced with the use of the 30 percent aluminum-70 percent nickel charge material. However of equal importance with the composition of the charge material in the process is the heating cycle given the materials. The first heating at the lower range of temperatures, 1400° to 1650° F., has the effect of producing a thin, smooth and uniform coat; while the second heating at the higher range of temperatures, 1850° to 2050° F., produces the necessary thickness in the coating. The distribution of intermetallic compounds is also a product of this specific heating cycle.

The first heating is conducted at a minimum temperature of 1400° F. because at temperatures below 1400° F., the diffusion rate is too low. Actually the minimum temperature is determined by the decomposition temperatures of the carrier compounds; and while the ammonium chloride decomposes at 662° F. and the compound aluminum chloride sublimes at 352° F., the lower temperature limit is set at 1400° F. for effective coating and diffusion. The time of heating may vary from about 4 hours to about 20 hours and 16 hours at 1600° F. is preferred as producing the required coating in a shorter time. The first heating should not be conducted at temperatures appreciably higher than 1600° F. for sintering of particles of the nickel-aluminum charge material to the coating will occur. At 1800° F. after 24 hours

5

particles will sinter onto the coatings. Therefore an upper limit for this first heating is set at about 1650° F. After the first thin coating is deposited at these temperatures there is no danger of sintering at the higher temperatures of the next heating step.

The second heating at from 1850° to 2050° F. gives the smooth thick coating with the proper zoning of the nickel-aluminum compounds. This heating may be for from 2 hours to 8 to 10 hours depending on the size, configuration, and composition of the article to be coated, thickness of coating desired, the size of the retort, carrier used, etc. In the preferred embodiment the heating is at 2000° F. for about 6 hours.

Under the above-described heating conditions, there will be no sintering of particles to the coating and the coating will possess a lustrous appearance. It is to be understood that the two heat treatments need not be separate and distinct. The temperature may be gradually increased from 1400° F. to 2050° F. as a continuous, properly-timed, production-line operation—the exact times and temperatures being determined for the specific process.

As an example of the practice of the invention, the following description of the preparation of the prealloyed charge material is given. This material may be prepared by several methods, such as powder metallurgical methods, but alloying by fusion is preferred. In an induction furnace melting operation commercially available electrolytic nickel (about 99.8 percent nickel) and commercially available pure aluminum shot (about 99 percent aluminum) were used as raw materials in the proportion of 70 percent by weight nickel and 30 percent by weight aluminum. The nickel was melted first in a magnesia-lined crucible at a temperature of about 2950° F. Aluminum was added to the molten nickel in batches of about 10 percent of the total amount to avoid the undesirable effects of an overly exothermic reaction. After all of the aluminum was added and alloyed with the nickel, the melt was covered with a lime slag to prevent oxidation and volatilization of the aluminum. An inert atmosphere blanket may also be used for this purpose. The bath temperature was increased to 3150° F. and the alloy was poured through a shotting tower to produce alloy shot. The washed and dried shot had the following actual and intended composition. The incidental impurities contained in the alloy do not interfere with the coating process.

COMPOSITION IN WEIGHT PERCENT

	Desired	Actual
Aluminum	30.0	29.28
Nickel	Balance	Balance
Carbon	Low	.04
Manganese	do	.02
Magnesium	do	.16
Silicon	do	Trace
Calcium	do	Do.

The shot was then crushed in a jaw crusher to a size passing a No. 4 mesh sieve and held on a No. 20 mesh sieve. In other diffusion coating processes a much finer material is used with a resulting dust problem and the possible inclusion of fine particles in the coating.

There are no catalysts, fillers or absorbents used with the charge material. A getter to remove oxygen, such as titanium, may be used.

The articles to be coated are first cleaned by any of the usual methods, such as grit blasting. The articles are then packed within the charge material in the retort with at least enough space between each article to allow for a layer of charge material. Cavities which are to be coated are charged with carrier material and, if desired, with charge material. The remainder of the carrier material need not be mixed with the charge material (but may be so mixed) and can be placed at the bottom of

6

the retort. This separate layer of carrier material on the floor of the retort heats more quickly and volatilizes to purge the system of air and begin the coating action earlier than when the carrier agent is intermixed with the charge material. From 0.5 to 1 percent by weight carrier material to 99 to 99.5 percent charge materials is generally used.

The loaded retort is heated according to the preferred process. After the heating cycle is completed, the retort is cooled to a safe handling temperature. The coated objects are then removed and washed. They have a smooth finish and require no further processing steps.

The crushed charge material may be reused. In a series of tests a single batch of crushed 70 percent nickel-30 percent aluminum charge material was used six times and was still usable. Only a washing with water and a screening to remove fine, exhausted particles is required before each reuse.

A number of alloy articles were diffusion coated according to this process. The nominal compositions of these alloys are listed in Table III. The compositions do not include the carbon, or other minor constituents.

TABLE III

Nominal Compositions of Articles To Be Coated

Alloy	Composition in weight percent							Condition
	Ni	Co	Fe	Mo	Cr	W	Al	
Pure Nickel	99+							Wrought.
1	74			4.5	12.5		6	Cast.
2	65		9	5.3	15.5		3	Do.
3	64		5	25	5			Wrought.
4	62	15		3	10		5.5	Cast.
5	61		5	28				Wrought.
6	54		5.5	16	15.5			Do.
7	47		18	9	22			Cast.
Pure Cobalt		99+						Wrought.
8		61	1.5		21	11		Cast.
9	10	53	2		20	15		Wrought.
10	10.5	56			25.5	7.5		Cast.
1010 Steel			99					Wrought.

These articles were diffusion coated according to the preferred process of the invention and had the coating characteristics listed in Table IV.

TABLE IV

Coatings Deposited by the Process of This Invention

Alloy	Weight change during coating process, Mg./Cm. ²	Coating thickness, mils	Surface appearance
Pure Nickel	+10.61	2.5	Good.
1	+4.42	3.1	Do.
2	+2.81	2.2	Do.
3	+6.57	2.5	Do.
4	+4.38	2.9	Do.
5	+7.32	2.7	Do.
6	+3.54	2.0	Do.
7	+2.89	2.0	Do.
Pure Cobalt	-5.17	0.7	Fair.
8	-2.14	0.4	Do.
9	-1.79	0.9	Do.
10	-0.90	1.1	Do.
1010	-24.25	(¹)	Poor.

¹ Up to 3.7—non-uniform.

The nickel-base articles formed better diffusion coatings than the cobalt-base articles. The coating on the cobalt-base articles as will be seen below is sufficient to enable their use in many applications which the uncoated article would be unsuited for. The iron-base article did not accept the nickel-aluminum coating. Instead of nickel aluminum being uniformly deposited on the iron, the iron itself was carried off by the halides. Iron articles, therefore, are not suited to the process of this invention and must be plated by the conventional electrodeposition methods. However the nickel-base alloys coated by the pro-

7

ess of this invention may contain relatively large amounts of iron provided there is present in the nickel-base alloy a predominant amount of nickel.

Several of these coated articles were tested for oxidation resistance by exposure in a moving stream of dry air at 2000° F. for 216 hours. The weight loss of each article, in a coated and uncoated condition, is given in Table V. The weight gain is due to the formation of protective oxides on the surface of the article. The weight loss is due to spalling of oxides from the surface of the article. A surface coating which does not spall off is superior in oxidation resistance to one that does spall off leaving the base metal unprotected.

TABLE V
Oxidation Test Results

Alloy	Mg./Cm. ² weight change, 216 hours at 2,000° F.	
	Uncoated alloy	Coated alloy
1.....	-12.5	+1.78
2.....	-35.6	+1.68
3.....	-59.6	+1.70
5.....	-195	-7.86

The alloys all exhibited exceptional improvements in oxidation resistance.

Nickel-base alloy No. 1 was further tested for tensile strength and impact strength as shown in Tables VI and VII. Alloy specimens, coated and uncoated, were subjected to standard tensile and impact tests to determine whether the coating process affected their strength properties.

TABLE VI

Tensile Test—Standard 1/4-Inch Test Specimens—Alloy 1 Coated by Preferred Method

Uncoated		Coated	
Test temperature, ° F.	Ultimate tensile strength, p.s.i.	Ultimate tensile strength, p.s.i.	Elongation, percent
80.....	123,000	123,080	8.8
1,200.....	121,000	134,000	7.3
1,600.....	111,000	108,000	16.6
1,700.....	80,000	87,270	20.8

TABLE VII

Charpy Impact Test—Standard Specimens—Unnotched Bars—Alloy 1 Coated by Preferred Method

Test temperature, ° F.	Average strength, in foot pounds	
	Coated	Uncoated
1,600.....	26	29
1,800.....	28	29

It is seen from the results of Table VI that the coating process does not significantly affect the tensile strength of the coated article. It is to be noted that even those specimens which suffered a 20.8 percent elongation did not exhibit a coating failure due to spalling, cracking, or chipping.

The impact test results of Table VII further show that the coating process does not affect the strength of the base article. The difference of a few pounds is within the expected experimental error by Charpy impact testing. The fractured specimens showed no spalling or cracking of the coating.

A series of hot and cold gas erosion tests were also conducted in a special testing device. Specimens of

8

coated and uncoated parts were rotated automatically to alternately hot and cold stations. At the hot stations, the specimens were heated by the blast of an oxy-acetylene torch for nine seconds to a temperature of about 2000° F. Then at the cold stations the specimens were rapidly cooled in a blast of compressed air to about 300° F. in nine seconds. The direct impingement of the torch flame and compressed air on the specimens constitutes a hot and cold gas erosion test. The results of these tests are shown in Table VIII for specimens having the coating of this invention as well as for other coated and uncoated specimens. The base article was alloy No. 1 of Table III.

TABLE VIII
Thermal Shock Test

Specimen No.	Condition of specimen	Erosion resistance
1.....	Uncoated.....	Failed 50 cycles.
2.....	Coated according to this invention.....	OK to 100 cycles.
3.....	do.....	OK to 250 cycles.
4.....	do.....	OK to 450 cycles.
5.....	Chromized.....	Failed 50 cycles.

It is seen from the results of Table VIII that the articles coated according to the invention were superior in erosion resistance to the uncoated (specimen No. 1) and chromium coated (specimen No. 5) articles. Failure of the articles was due to spalling of the oxide coating.

It is seen, therefore, that this invention constitutes a valuable addition to the high temperature metallurgy field. The application of the diffusion coating of this invention to nickel- and cobalt-base alloy articles so protects them from oxidation and erosion at high temperatures that the alloys may be selected on the basis of their strength properties and not their oxidation resistance. This gives the designer a much greater flexibility in his choice of engineering materials and ensures maximum performance by each part.

What is claimed is:

1. A method for diffusion coating metallic articles composed of a material selected from the group consisting of nickel-base alloys and cobalt-base alloys comprising embedding the article to be coated in particulated prealloyed charge material consisting essentially of from 15 percent to 37 percent by weight aluminum and the balance substantially all nickel and incidental impurities and heating said charge material and contained articles in the presence of a carrier material selected from the group consisting of ammonium halides, aluminum halides, and nickel halides, said heating being conducted with the exclusion of air at a temperature from about 1400° F. to 1650° F. for at least 4 hours followed by a heating at a temperature from about 1850° F. to 2050° F. for at least two hours.

2. A method for diffusion coating metallic articles composed of a material selected from the group consisting of nickel-base alloys and cobalt-base alloys comprising embedding the article to be coated in particulated prealloyed charge material consisting essentially of from 15 percent to 37 percent by weight aluminum and the balance substantially all nickel and incidental impurities, and heating said charge material and contained articles in the presence of a carrier material selected from the group consisting of ammonium halides and aluminum chloride, the carrier material being present with the charge material in the relationship of from 0.25 percent to 3 percent by weight carrier material to from 97 percent to 99.75 percent charge material, said heating being conducted with the exclusion of air at a temperature from about 1400° F. to 1650° F. for at least 4 hours followed by a heating at a temperature from about 1850° F. to 2050° F. for at least two hours.

3. A method for diffusion coating metallic articles composed of nickel-base alloys comprising embedding the article to be coated in particulated prealloyed charge material consisting essentially of from 15 percent to 37 percent by weight aluminum and the balance substantially all nickel and incidental impurities, and heating said charge materials and contained articles in the presence of a carrier material selected from the group consisting of ammonium halides and aluminum chloride, the amount of carrier material present with the charge material being in the relationship of from 0.25 percent to 3 percent by weight carrier material to from 97 percent to 99.75 percent charge material, said heating being conducted with the exclusion of air at a temperature from about 1400° F. to 1650° F. for at least 4 hours followed by heating at a temperature from about 1850° F. to 2050° F. for at least two hours.

4. A method for diffusion coating metallic articles composed of nickel-base alloys wherein nickel is the predominant constituent comprising embedding the article to be coated in particulated prealloyed charge material consisting essentially of from 15 percent to 37 percent by weight aluminum and the balance substantially all nickel and incidental impurities, and heating said charge and contained articles in the presence of ammonium bromide carrier material, the amount of carrier material present with the charge material being in the relationship of from 0.25 percent to 3 percent by weight ammonium bromide to from 97 percent to 99.75 percent by weight charge material, said heating being conducted with the exclusion of air at a temperature of about 1600° F. for about 16 hours followed by a heating at a temperature of about 2000° F. for about 6 hours.

5. A method for diffusion coating metallic articles composed of nickel-base alloys wherein nickel is present in a minimum amount of 45 percent by weight comprising embedding the article to be coated in particulated prealloyed charge material consisting essentially of from 20 percent to 37 percent by weight aluminum and the balance substantially all nickel and incidental impurities, and heating said charge material and contained articles in the presence of ammonium bromide, the amount of carrier material to charge material being in the relationship of from 0.25 to 3 percent by weight ammonium bromide to from 97 percent to 99.75 percent by weight charge material, said heating being conducted with the exclusion of air at a temperature of about 1600° F. for about 16 hours followed by a heating at a temperature of about 2000° F. for about 6 hours.

6. A method for diffusion coating metallic articles composed of nickel-base alloys wherein nickel is present in a minimum amount of 45 percent by weight comprising embedding the article to be coated in particulated prealloyed charge material consisting essentially of about 30 percent by weight aluminum and the balance substantially all nickel and incidental impurities and heating the charge

material and contained articles in the presence of ammonium bromide carrier material, the amount of ammonium bromide present with the charge material being in the relationship of from 0.25 percent to 3 percent by weight ammonium bromide to 97 percent to 99.75 percent charge material, said heating being conducted with the exclusion of air at a temperature of about 1600° F. for 16 hours followed by a heating at a temperature of about 2000° F. for about 6 hours.

7. A method for diffusion coating metallic articles composed of nickel-base alloys wherein nickel is present in a minimum amount of 45 percent by weight comprising embedding the articles to be coated in particulated prealloyed charge material passing a No. 2 mesh sieve but held on a No. 100 mesh sieve, said charge material consisting essentially of about 30 percent by weight aluminum and the balance substantially all nickel and incidental impurities, and heating the charge material and contained articles in the presence of ammonium bromide carrier material, the amount of ammonium bromide present with the charge material being in the relationship of from 0.25 percent to 3 percent by weight ammonium bromide to from 97 percent to 99.75 percent by weight charge material, said heating being conducted with the exclusion of air at a temperature of about 1600° F. for 16 hours followed by a heating at a temperature of about 2000° F. for about 6 hours.

8. A method for diffusion coating metallic articles composed of nickel-base alloys wherein nickel is present in a minimum amount of 45 percent by weight comprising embedding the article to be coated in particulated prealloyed charge material passing a No. 4 mesh sieve and held on a No. 20 mesh sieve, said charge material consisting essentially of about 30 percent by weight aluminum and the balance substantially all nickel and incidental impurities, and heating said charge material and contained articles in the presence of ammonium bromide carrier material, the amount of ammonium bromide to charge material being in the relationship of from about 0.5 percent to 1 percent by weight carrier material to from about 99 percent to 99.5 percent by weight charge material, some of the carrier material being placed before the heating in the cavities of the articles to be coated, said heating being conducted with the exclusion of air at a temperature of about 1600° F. for about 16 hours followed by a heating at about 2000° F. for about 6 hours.

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