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[54] **MASKING METHOD FOR USE IN ALUMINIZING SELECTED PORTIONS OF METAL SUBSTRATES**

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[51] **Int. Cl.²** **C23C 9/02;** B05D 1/32

[58] **Field of Search** ..... 117/5.5, 107.2 P; 29/196.2; 427/253, 252, 282, 287

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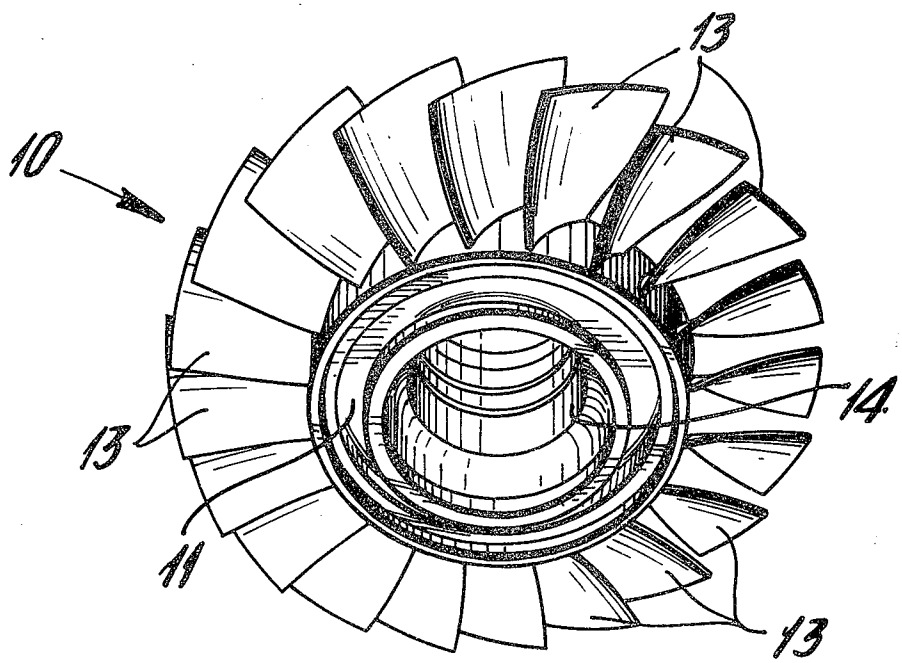
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[57] **ABSTRACT**

Selected areas of steel parts are aluminized by masking the surface of a steel part except for the area to be aluminized by coating the surface of said part outside said selected portion with a masking metal. The part is then aluminized by embedding it in an aluminizing cementation pack and heated to an aluminizing temperature, following which the part is removed from the pack and the metal mask removed by dissolution in an aqueous solution selective to said metal.

7 Claims, 2 Drawing Figures



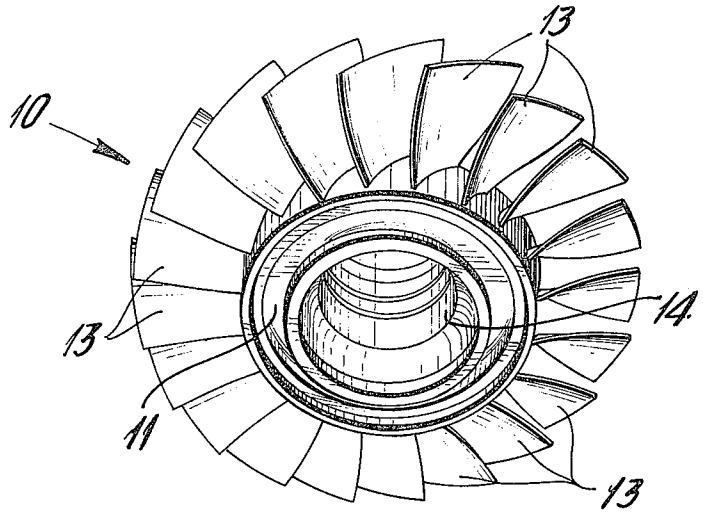


FIG.1

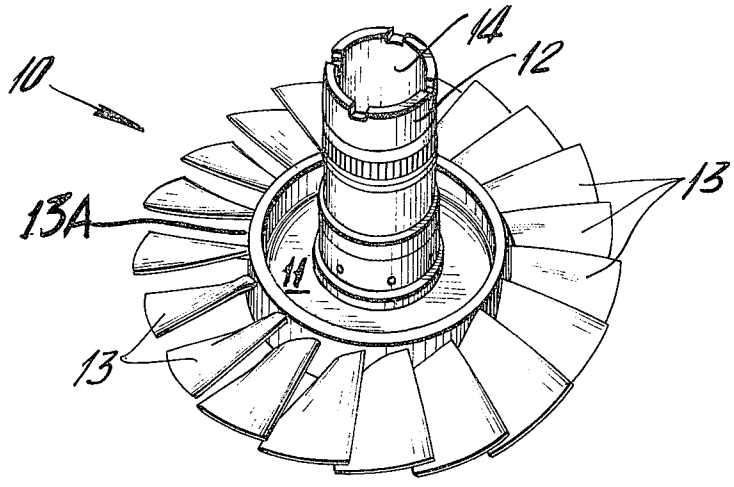


FIG.2

MASKING METHOD FOR USE IN ALUMINIZING SELECTED PORTIONS OF METAL SUBSTRATES

This invention relates to a method for aluminizing a selected portion of a steel part while preventing other portions of the part from being aluminized.

STATE OF THE ART

It is known to diffusion coat metal parts or articles with aluminum to protect said parts against corrosion. Examples of such parts include turbine vanes, shrouds, and other jet engine components. One method is to embed the part, which is usually made of a chromium-containing steel, in a powder pack which contains the coating metal (e.g. aluminum powder) mixed with an inert filler (powdered alumina, magnesia, silica, zirconia, calcia, or the like) and an energizer component (such as a halide, e.g. NH_4I, $AlCl_3$ and alkali metal halides for aiding the transport or transfer of the coating metal from the powdered pack to the surface of the part to be coated).

The pack with the embedded part is heated in a closed retort (usually in the partial absence of oxygen) to relatively high temperatures for a sufficient time to produce the diffusion of the coating metal into the surface of the articles to a desired depth. Such processes are employed to enhance the oxidation resistance of the articles at elevated temperatures and to enhance erosion and corrosion resistance for a variety of purposes and uses. The conventional pack cementation diffusion coating techniques may require that the coating step be prolonged for many hours, or for even more than a day, at relatively high temperatures (ranging anywhere from about 1200°F to as high as 1800°F, depending upon the particular metal substrate, the thickness of coating desired and other characteristics). With an aluminum pack, aluminide coatings containing largely iron aluminide are formed on chromium-containing steel substrates.

The application of aluminide coatings to 400 series type of stainless steels has met with widespread acceptance as a method of enhancing the corrosion resistance of aerospace components. The coating thickness generally obtained is normally a maximum of 0.8 mil, with about one-half forming as a diffusion layer and one-half causing a build-up or an increase in the dimensions of the part.

PROBLEM CONFRONTING THE ART

It is important that critical dimensional tolerances measured in ten-thousandths of an inch be maintained when building components into assemblies. Thus, any change in dimensions in certain portions of a part attributed to the aluminide coating may not be acceptable under certain military and other specifications.

In some situations, it may not be desirable to coat certain areas of a part, e.g. bearing areas, journals, shafts, platforms, and the like, due to the surface characteristics of the resulting coating. For example, the fretting resistance or bearing resistance of the coating on bearing areas, shafts, and the like may not be adequate. Thus, it may be prudent not to apply a coating to such areas.

Methods which have been proposed to overcome the foregoing problems are to mask selectively the surface of the steel part not to be coated so as to obstruct physically the penetration of the aluminum in the areas outside of the areas to be coated. Such masks may be formed of coatings produced from inert chemical slurries. However, these proposed methods have not been adequate for the reason that in the aluminum pack process, a highly reactive halide compound is employed, for example, aluminum chloride, which produces large volumes of hydrogen chloride which, under pressure, penetrates the cracks and crevices of the mask and causes small quantities of aluminum coating to form under the mask. Removal of this coating produces a change in dimensions of the part. In some cases, it may be necessary to hone the masked surfaces lightly to clean off surface residues formed by the interactions of the mask with coating vapors.

An important aerospace assembly that requires precise areas of aluminum coating and substantially perfect masking to avoid dimensional changes is the compressor blisk. The blisk is an integral blade and disk assembly machined from forged stock of AM 355 steel comprising about 15.5% Cr, 4.25% Ni, 2.7% Mo, 0.15% C, 0.10% N and the balance iron. The airfoils and airfoil platforms require the aluminum coating but the other areas, such as shafts, mating surfaces and the bores, should preferably be entirely of dimensional changes. Since these areas do not take the brunt of heat and corrosion, they need not be coated.

As far as we are aware, existing masking techniques have not been satisfactory because of the foregoing problems. We now provide a masking technique which enables the production of precision aluminized parts, while maintaining controlled dimensional tolerances in non-aluminized areas.

OBJECTS OF THE INVENTION

It is thus the object of the present invention to provide a method for masking steel parts preparatory to aluminizing said part by pack cementation process.

Another object is to provide a method for aluminizing selected areas of steel parts by the pack cementation diffusion process while avoiding the aluminizing of other selected areas in which the dimensions thereof should be held to controlled tolerances.

These and other objects will be clearly apparent from the following disclosure and the appended drawing, wherein:

FIGS. 1 and 2 depict a compressor blisk comprising an integral blade and disk assembly machined from forged stock.

STATEMENT OF THE INVENTION

Stating it broadly, the invention is directed to a method for aluminizing a selected area of a steel part which comprises, masking the surface of said part except for the area to be aluminized by applying a masking coat or plating of a metal selected from the group consisting of copper, nickel and chromium, to the selected surface area or portion, subjecting the steel part to aluminizing by embedding it in an aluminizing cementation pack and heating said part and pack to an aluminizing temperature, removing the aluminized part from the pack, and then selectively dissolving the metal mask in an aqueous solution selective to said metal, e.g. copper.

The foregoing method has been found to be markedly superior to other methods utilizing, for example, coating slurries of aluminum oxide mixed with iron powder and a binder of lacquer, or silicon dioxide

mixed with aluminum and a binder of a gelling agent, such as an organic derivation of hydrous magnesium silicate, and others. For example, a mask made of copper compared to the latter techniques did not leave substantially any residue when it was dissolved away following aluminizing of the part. The masking metal coating is generally dense so that the aluminum halide vapor is prevented from penetrating it and coating the steel substrate with aluminum.

Copper is preferred as the masking metal. The copper is selectively applied by electroplating to the surface to be coated with a layer of finite thickness. For best results, the finite coating layer should be in excess of 1 mil (0.001 inch), as thicknesses below 1 mil, for example 0.1, 0.2 and even up to 0.4 mil, do not produce the kind of masking coat that can be removed easily. When thicknesses below 1 mil were used, the copper aluminide formed with the copper penetrated the whole cross section of the copper plate which made its removal difficult.

On the other hand, when copper coatings in excess of 1 mil are used, for example, 2 mils, and up to 5 mils or higher, only a surface coating of copper aluminide is formed, with the remainder of the thickness being rich in copper. Thus, when the aluminized part is subjected to dissolution in a nitric acid solution, the acid selectively penetrates the cracks or fissures in the copper aluminide layer and preferentially dissolves the underlying copper coat, thus enabling the removal of the masked portion of the surface.

For example, when the dense copper coat was 5 mils thick, prior to aluminizing, the resulting coat after aluminizing was 6 mils, thus showing a growth in thickness of about 1 mil due to the formation of copper aluminide. Analysis has shown the outer surface to contain by weight about 43.8% aluminum and 56.2% copper which approximates the intermetallic compound $CuAl_2$. Under the $CuAl_2$ layer, an enriched copper phase corresponding to 19.4% of aluminum and 80.5% copper was detected, while underneath the two layers is the remaining layer of the dense unreacted copper plate.

Because a layer of copper plate in the unreacted condition is present, the entire layer of copper aluminide and copper can be easily removed without damage to the regular layer of iron aluminide in the selected areas of the part. By using, for example, a 50% solution of fresh nitric acid at room temperature, the residual copper undercoat is selectively dissolved through defects in the copper aluminide layer which undercuts the overlying layer of copper aluminide which is easily removed. The temperature of the nitric acid solution is preferably maintained below 100°F (38°C). Under normal conditions, about 3 mils of copper plate can be removed after the aluminizing step by acid stripping in about 20 minutes.

DETAILS OF THE INVENTION

As stated hereinbefore, the invention is particularly applicable to the engine component referred to as a blisk depicted by way of illustration in FIGS. **1** and **2** which are different perspective views of the component.

The component is an integral blade and disk assembly machined from forged stock. Referring to the perspective view of FIG. **2**, the blisk is generally indicated by the numeral **10**, the blisk comprising a disk **11** with indented platform having a central opening (note FIG. **1**) having extending coaxially and integrally therefrom a hollow hub or shaft **12**. The outer shaft surface has critical dimensional tolerances measured in ten-thousandths of an inch and, therefore, no dimensional changes due to coating can be tolerated. FIG. **1** is a perspective view of blisk of FIG. **2** as viewed from the bottom.

Extending radially from the periphery or airfoil platform **13A** of the disk are a plurality of spaced airfoils **13**. The airfoils and the airfoil platform surface are aluminized while the axially extending hollow hub, including the bore **14** thereof and the indented platform of the disk are not aluminized since the outer and inner surfaces of the hub would be subject to fretting corrosion if provided with an aluminum coating. In addition, the inner and outer surface of the hub must have precise dimensions for mounting purposes which otherwise would be lost if the hub is permitted to be coated with aluminum by the pack cementation process.

Broadly speaking, in preparing the assembly to receive the masking metal, for example, copper plating, the surfaces not to be copper plated, that is, the platform faces of the disk and the airfoils, are coated with an easily removable chemically resistant material, e.g. wax, plastic, and the like material, and the uncoated areas then provided with a copper plate by electroplating from a copper solution. Following the copper plating step, the resist is removed and the selectively copper-coated assembly then prepared for pack cementation in an aluminizing pack. The detail aspects of the overall process are as follows.

The steel assembly or part prior to copper plating is hot vapor degreased in trichloroethylene. After vapor degreasing the part, masking tape (an adhesive coated rubberized tape) is applied to all of the surfaces where it is intended to apply the copper plating. The tape is trimmed to produce the desired edge definition between the area to be coated and the area to be masked.

The taped part is then placed in the plating fixture and the part and fixture immersed in a container of molten wax (resist material) to coat at least the untaped surfaces.

Thereafter, the tape is removed to expose the areas to be copper plated and edge definition re-established by trimming the wax edges with a wooden spatula wherever necessary and the part now has wax masking over all the surfaces not to be plated.

In the case of the blisk of FIGS. **1** and **2**, the inner and outer surfaces of the follow hub or shaft and the disk surfaces are plated with copper, while the radially extending airfoils and the airfoil platform are provided with a wax coating to prevent the copper plating thereof.

Prior to plating, the part is preferably cleaned in a cold soap solution and then rinsed in deionized water.

The copper plating bath may be any of the well known copper plating baths, for example, a copper sulfate-sulfuric acid bath. A typical bath in grams per liter (gpl) is one containing about 56 gpl of copper (7.5 oz/-gallon) or 207 gpl (29 oz/gallon) of copper sulfate ($CuSO_4 \cdot 5H_2O$), 60 gpl sulfuric acid (8 oz/gal) and 50 ppm of chlorine ion. The bath has a specific gravity of approximately 1.17 at 25°C and a resistivity at 21°C of about 4.2 to 4.3 ohms/cm^3. The composition of the electroplating solution may range from about 150 to 250 gpl copper sulfate ($CuSO_4 \cdot 5H_2O$), about 45 to 110

gpl H_2SO_4, with the specific gravity ranging from about 1.115 to 1.21.

The surface is plated for about 3 hours under the foregoing conditions to provide a coating thickness of about 0.75 mil/hour or a total thickness of approximately 2.25 to 3 mils, since the current efficiency is generally less than 100%.

The plating is performed at a current density of about 0.2 amps/in^2 or about 29 amps/ft. As is known by those skilled in the art, addition agents, e.g. 0.005 gpl thiourea, or 0.2 glue, or similar agents, are generally added to the baths to provide a bright dense deposit substantially free from trees. Copper plating techniques which may be employed including addition agents are disclosed on pages 172 to 200 in the book entitled *Modern Electroplating*, (2nd Ed. 1963, John Wiley and Sons, Inc.).

In plating the part, a conforming anode structure is preferably provided which is configurated to simulate as closely as possible the contour of the part to be plated so as to assure uniform throwing power to the surface or area to be plated, the part being the cathode. The part is plated until a metal thickness of at least about 1 mil is obtained, the thickness generally ranging from about 2 to 6 or 7 mils. A preferred thickness is about 2 to 5 mils.

After plating the part, it is removed from the copper plating tank and rinsed in deionized water. The wax is removed by melting in a hot water solution and the part is then hot vapor degreased. This removes the residual traces of wax from the surfaces to be coated.

The part is preferably prepared for coating by a 400 mesh honing treatment that cleans the surface to be coated, although, in some instances, this may not be required, or other methods of cleaning used.

The copper plated part is then placed in a retort and surrounded by a power pack comprising a dry mixture of 80% particulate aluminum and 20% aluminum oxide as the inert material, to which mixture has been added about 3% aluminum chloride ($AlCl_3$). As will be apparent to those skilled in the art, other inert materials may be used in place of alumina, e.g. silica, calcia, zirconia, magnesia and other refractory oxides. To minimize the absorption of moisture and hence the formation of hydrochloric acid, the packing is performed in an enclosed area where the relative humidity level is maintained at less than 60%, e.g. 50% or lower.

The retort is sealed and placed in a cold oven and a slow heating rate used for the coating cycle. This allows for an endothermic arrest to occur at substantially the sublimation temperature of $AlCl_3$ (356.4°F) which results in the generation of copious amounts of chloride gases that both clean the surface of the part to be aluminized and that promote the diffusion of aluminum into the steel surface at higher temperatures (e.g. 875°F). Aluminum diffuses both into the surface to be coated and, to some extent, into the copper plate. On the completion of the coating cycle for 42 hours at 875°F, the retort is removed and allowed to cool.

The part is removed from the retort and residual powder blown off the surfaces. The part is placed in a neutralizing solution to eliminate any acidic residue that might remain after the coating operation. This is followed by a rinsing in water. The assembly is then ready for removal of the masking material. This is achieved by placing the part in a fresh 50% nitric acid solution maintained at room temperature. The copper aluminide has limited or no solubility in the nitric acid solution. However, defects in the copper aluminide coating (e.g. cracks, pores, etc.) allow the nitric acid to react rapidly with the underlying layer of copper. The dissolution of the copper is normally completed within twenty minutes. The regular aluminide coating on the part (e.g. iron aluminide) is not substantially soluble in nitric acid at room temperature. Frequent water rinses, however, during the copper stripping step maintain the reaction at a low enough temperature to completely preserve the final aluminide coating.

A visual check will generally ensure that all copper or copper aluminide has been removed from the part. After a final rinse in deionized water, the process is complete. The aluminide is formed only in the areas required and the critical dimensions and the original surface condition of the previously masked areas maintained.

A preferred process for aluminizing the part by pack cementation is disclosed in U.S. Pat. No. 3,762,885 which is owned by the same assignee. This process is preferred as it enables the use of aluminizing temperatures low enough to avoid overheating of the steel part which would normally cause crystallographic changes in the part. Thus, the diffusion temperature is advantageously below (but not limited to) that temperature which would be detrimental to the physical properties of the steel. The process employs a cementation pack having uniformly mixed therewith an amount of dry aluminum chloride powder as a cleansing and transport-inducing agent effective to promote the deposition and diffusion of aluminum onto the steel article at the desirable metal coating and diffusion temperature. The aluminum chloride is characterized by substantial vapor pressure when heated to above ambient temperature and below the metal coating and diffusion temperature. The steel article is completely embedded into the cementation pack confined in a closed retort and the assembly subjectd to a time-temperature heating cycle in which the pack is brought up from ambient temperature to the desired metal coating and diffusion temperature, preferably below the temperature which adversely affects the physical properties of steel, at a rate characterized by an endothermic arrest at the sublimation temperature of aluminum trichloride below the metal coating and diffusion temperature, whereby substantial outgassing occurs during the period of endothermic arrest, the heating being continued to and at the preferred temperature until the desired aluminum coating thickness has been obtained. The part is held at substantially the sublimation temperature (about 355°F to 375°F) for at least about 10 or 15 minutes.

The aforementioned preferred process utilizing the endothermic arrest provides markedly improved activity at substantially lower temperatures. In essence, the process greatly decreases the incubation period which normally prevails in prior processes due to contamination of the metal substrate by surface oxides, residues, and the like, and due also to the surface contamination (e.g. oxides) of the particulate coating metal itself. The disclosure of the foregoing patent is incorporated herein by reference.

In carrying out the process, the aluminum content of the pack may be varied from 100 to 60 percent and, when less than 100 percent metal content is employed in the pack, an inert refractory oxide filler, e.g. aluminum oxide, is selected as the inert material. Preferably,

the oxide filler has a mesh size which is similar to the metal particles, for example, −60 to +140 but it has been found that the −325 mesh refractory oxide, e.g. aluminum oxide, can also be used. The aluminum chloride is added to the pack in the range of about 1 to 3 percent, although up to about 5 percent can be employed. Processing of the coating compound is preferably carried out under humidity control conditions with a relative humidity less than 60 percent or, more advantageously, 45 percent or lower employed to prevent undesirable reaction with moisture. Freshly blended powder is preferably burnt out prior to being used for the coating cycle.

The invention is particularly applicable to chromium-containing steel parts. Such composition may include from about 5 to 25 percent chromium, up to about 5 percent tungsten, up to about 5 or 10 percent nickel, up to about 4 percent copper, up to about 3 percent aluminum, up to about 2 percent titanium, and the balance essentially iron, and including, in particular, precipitation hardenable chromium-containing stainless steels.

As has been pointed out hereinbefore, a particular advantage of the aluminizing process is the relatively low temperature range over which high quality coatings can be achieved. Such low coating temperatures can range from about 750°F to 900°F, and, more advantageously, from about 775°F to 850°F. However, the diffusion temperature may be higher, if desired.

As regards the masking metal, nickel may be used in place of copper as the plating metal. A typical nickel plating bath that may be used is a bath referred to as the Watts bath comprising 330 gpl nickel sulfate ($NiSO_4 \cdot 7H_2O$), 45 gpl nickel chloride ($NiCl_2 \cdot 6H_2O$) and 38 gpl boric acid (H_3BO_3). The bath is generally used at a temperature of about 45°C to 65°C at current densities ranging from about 2.5 to 10 amps/dm^2 (or 23 to 93 amps/sq.ft.). Other details regarding nickel plating are given in the aforementioned book entitled *Modern Electroplating*, pages 260 to 309.

Similarly, chromium may be employed as a masking coat. A typical chromium plating bath is one containing about 250 gpl chromic acid and about 2.5 gpl of free sulfate ion (e.g. as H_2SO_4). The temperature is generally about 40°C with the cathode current density varying from 3.1 to 15.5 amps/dm^2 (29 to 145 amps/ft^2). Details concerning chromium plating are given on pages 80 to 140 of the aforementioned book, *Modern Electroplating*. Chromium similarly reacts with aluminum during pack cementation to form an aluminide at the surface thereof.

As with copper plate, the thickness of the other masking metals may be at least about 1 mil and generally fall within the range of about 2 to 7 mils, e.g. 2 to 5 mils.

As will be obvious to those skilled in the art, other metals may be employed as masking metals, which metals are deemed to be equivalent to the foregoing metals.

Nitric acid is preferably employed as the stipping acid and may range from about 15% to 75% by volume of concentrated nitric acid, for example, preferably from about 20% to 60% by volume of concentrated acid. Thus, the 50% solution of nitric acid mentioned hereinbefore means 50% by volume of concentrated nitric acid.

Other stripping solutions may be employed to selectively remove the masking material following pack cementation of the aluminum coating. For example, hot concentrated sulfuric acid may be used to dissolve copper as well as concentrated acetic acid. Hydrochloric acid may be used but it has certain hazards. Nitric acid is more preferred. Where the stripping solution employed might attack the iron aluminide coating formed on the areas of the steel part to be coated, then it is advisable to apply a resist coating to such areas to protect such areas from dissolution during the removal of the masking metal. Such resist materials may be the same as those applied earlier in the process and may include wax, plastics and the like.

Although the present invention has been described in conjunction with preferred embodiments, it is to be understood that modifications and variations may be resorted to without departing from the spirit and scope of the invention as those skilled in the art will readily understand. Such modifications and variations are considered to be within the purview and scope of the invention and the appended claims.

What is claimed is:

1. A method for aluminizing a selected area of a steel part which comprises,

masking the surface of said part except for the area to be aluminized by electroplating a metal coating of a masking metal selected from the group consisting of copper, nickel and chromium of thickness at least about 1 mil to said surface,

subjecting said steel part to aluminizing by embedding said part with the masked surface in an aluminizing cementation pack and heating said part and pack to an aluminizing temperature,

removing said part from said pack,

said selectively removing the masking metal while preserving the area with the aluminized coating.

2. A method for aluminizing a selected area of a steel part which comprises,

masking the surface of said part except for the area to be aluminized by electropolating a metal coating of at least about 1 mil thick of a masking metal selected from the group consisting of copper, nickel and chromium to said surface,

subjecting said steel part to aluminizing by embedding said part in an aluminizing cementation pack comprising particulate aluminum and containing a small but effective amount of an energizer of aluminum trichloride and heating said part and pack to an aluminizing temperature,

removing said part from said pack,

and selectively dissolving the masking metal in an aqueous acid solution of sufficient dissolution strength while preserving the area with the aluminized coating.

3. A method of aluminizing a selected area of a chromiun-containing steel part which comprises,

applying a coating of a resist material to said selected area, while leaving the remaining area exposed,

electroplating a metal coating of a masking metal selected from the group consisting of copper, nickel and chromium of thickness of at least about 1 mil to said exposed area,

removing the resist coating from said steel part and cleaning said part,

subjecting said steel part to aluminizing by embedding said part in a cementation pack comprising

particulate aluminum powder and containing a small but effective amount ranging up to about 5% by weight of said pack of aluminum trichloride and heating said pack to an elevated aluminizing temperature,

removing said steel part from said pack,

and selectively dissolving the masking metal in an aqueous nitric acid solution of sufficient dissolution strength while preserving the area with the aluminized coating.

4. The method of claim **3,** wherein the resist material is wax, and wherein the masking metal is copper.

5. The method of claim **4,** wherein the thickness of the copper coating ranges from about 2 to 7 mils.

6. The method of claim **5,** wherein the area with the copper masking metal is selectively removed following aluminizing by dissolution with aqueous nitric acid solution of strength ranging from about 15% to 75% by volume of concentrated nitric acid.

7. The method of claim **6,** wherein the strength of the aqueous nitric acid solution ranges from about 20% to 60% by volume of concentrated nitric acid.

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