NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
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REPLY TO
ATTN OF: GP

APR 28 1971

TO: USI/Scientific & Technical Information Division
Attention: Miss Winnie M. Morgan

FROM: GP/Office of Assistant General Counsel for
Patent Matters

SUBJECT: Announcement of NASA-Owned U. S. Patents in STAR

In accordance with the procedures agreed upon by Code GP and Code USI, the attached NASA-owned U. S. Patent is being forwarded for abstracting and announcement in NASA STAR.

The following information is provided:

U. S. Patent No. : 3,472,742

Government or Corporate Employee : General Dynamics/ astronautics San Diego, Calif.

Supplementary Corporate Source (if applicable) : n/a

NASA Patent Case No. : XNP-04148

NOTE - If this patent covers an invention made by a corporate employee of a NASA Contractor, the following is applicable:
Yes ☒ No ☐
Pursuant to Section 305(a) of the National Aeronautics and Space Act, the name of the Administrator of NASA appears on the first page of the patent; however, the name of the actual inventor (author) appears at the heading of Column No. 1 of the Specification, following the words ". . . with respect to an invention of . . ."

Dorothy J. Jackson

Dorothy J. Jackson
Enclosure
Copy of Patent cited above

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PLATING NICKEL ON ALUMINUM CASTINGS

James E. Webb, Administrator of the National Aeronautics and Space Administration, with respect to an invention of Bernard J. Brinks, Escondido, Calif.

No Drawing. Filed Mar. 15, 1966, Ser. No. 536,210
Int. Cl. C23b *5/50, 17/00, 5/08*
U.S. Cl. 204—38 **1 Claim**

ABSTRACT OF THE DISCLOSURE

A process of nickel plating aluminum which comprises etching the aluminum, applying an alkali metal silicate to the etched aluminum surface to fill irregularities, and baking this silicate onto the aluminum surface, treating the aluminum with a hot electroless nickel plating solution, exposing the plated aluminum to an acidic bath to remove residual particles, treating the aluminum with a relatively warm electrolytic nickel plating solution to electrodeposit nickel thereon and fill all surface irregularities.

The invention described herein was made in the performance of work under a NASA contract and is subject to the provisions of section 305 of the National Aeronautics and Space Act of 1958, Public Law 85–568 (72 Stat. 435; 42 U.S.C. 4257).

This invention relates to a process for applying nickel to an aluminum casting.

Heretofore, plated aluminum contained surface pits and voids and when certain plated aluminum components were used in cryogenic pumps it has been difficult, if not impossible, to prevent or restrict the leakage of the cryogenic material to an acceptable minimum. For instance, heretofore many, if not most cryogenic pumps have included components which were chrome plated, but even with the plating, surface pits and voids were present. These surface irregularities, although small, permitted excessive leakage of cryogenic materials such as liquid hydrogen, nitrogen, helium and the like. The lack of a well defined surface tension of the cryogenic materials is such as to defeat the efforts heretofore attempted to control the leakage, and this inability to control leakage results in several adverse effects, among which is the possible damage to materials in adjoining regions which are unable to tolerate cryogenic temperatures, and the obvious loss of a relatively expensive material.

A solution to the aforementioned problem of surface irregularities comprises applying nickel to an aluminum casting, the first step or phase being to treat the casting with an acidic pickling or etching solution. Thereafter the treated casting has a coating of material baked thereon to fill the voids and pits as thoroughly as possible to thereby provide a relatively uniform surface. Then the casting is treated with an electroless nickel plating solution so a thin, relatively uniform layer of nickel is deposited thereon. Following the nickel plating step, the casting is again treated with an acidic solution to remove residual particles remaining from the immediately preceding step. Finally, the casting is treated with an electrolytic nickel plating solution to achieve filling of all remaining pits and voids which had not heretofore been filled. Between certain of the steps the casting is rinsed in a suitable solution so as not to unnecessarily contaminate the solution used in the next succeeding step and also to stop the chemical reaction of the immediate step.

Prior techniques for applying nickel onto aluminum employed the usual etching bath. Immediately thereafter, the

aluminum was placed in a nickel plating solution wherein a current was applied to achieve the plating of the nickel onto the aluminum. Then, in order to achieve satisfactory adhesion of the nickel to aluminum, the plated aluminum component was subjected to baking at temperatures as high as 1,000° F. Although the foregoing process may obtain satisfactory results for limited purposes, it is quite expensive and inefficient. For instance, the plating bath, which contains as many as four chemical compounds, is expensive, and the expense and inefficiency is increased by the requirement of having an electrical current pass therethrough. The expense and inefficiency is further compounded by the baking step when it is realized it is costly to produce the aforementioned elevated temperature and hold it for a relatively extended period of time. After all this, there is no assurance the pits and voids have been entirely filled since the object of the prior technique is merely to plate aluminum with nickel, not fill the pits and voids.

Other processes follow much the same procedures as above defined, but with minor variations; the variation in one instance occurring in the formulation of the etching solution or pickling bath. Another variation occurs in the plating bath, per se. Otherwise, the processes employ electrical currents to achieve plating, together with costly chemical compounds, yet none of the processes are directed to entirely filling the pits and voids present in an aluminum casting.

The hereinafter specifically described process achieves a result not heretofore accomplished, i.e., substantially entirely fill irregularities in an aluminum port or casting, and provide a relatively hard surface which can be subsequently subjected to grinding, lapping and polishing to provide a fine finish.

Initially, the aluminum casting, identified as 355T6 and containing about 5% silicon, is treated to a pickling or etching bath whereby a unform etching of the casting is obtained so subsequent baked-on components and the plating of nickel thereon will tenaciously adhere and withstand the environment to which the finished casting is exposed. Compounds which produce nitrate and fluoride ions are considered to be the most applicable components for the bath, and it is preferred and it has been found an acidic bath producing the aforementioned ions is most acceptable. Such a bath consists of nitric and hydrofluoric acid, the concentrations being reagent grade and about 48% respectively, the ratio of nitric to hydrofluoric acid being about 3:1 respectively. Because the casting contains silicon, the bath includes hydrofluoric acid for reaction therewith, but in the absence of silicon, the fluoride ion producing component may be eliminated. On the other hand, if the casting contains other alloying components, a substitute for the fluoride ion component may be introduced into the etching bath. It is known acid concentration has a considerable influence on grain structure and stress and should be carefully controlled within the limits heretofore and hereinafter defined where acid components are employed.

The exposure time of the casting in the acidic bath should range between 4–10 seconds, the optimum time for accomplishing the most satisfactory results being about 5 seconds.

Following the etching treatment, the casting is thoroughly rinsed, using, initially, ordinary tap water followed by a thorough rinsing with distilled water.

The next step or phase of the process is designed to fill as many of the surface irregularities in the casting as is possible, i.e., pits and voids.

To achieve this result, the casting is treated with an alkaline metal silicate such as sodium or potassium silicate, preferably sodium silicate, known also as "water glass." With the sodium silicate distributed thoroughly over the surface of the casting, it is dried, and the drying is hastened by baking over a hot plate or in an oven held at a temperature of about 190° F. for 3–8 minutes, preferably about 5 minutes.

The silicate solution comprises about 15 grams of sodium silicate in about 1,200 ml. of distilled water. This ratio provides a solution which will have a suitable surface tension to provide ease of flow to achieve a thorough distribution of the silicate over the casting surface to accomplish filling of the pits and voids, yet the solution is not so thick as to provide an undesirable or unacceptable sodium silicate layer to defeat depositing nickel on the casting with such adherence as to result in the nickel being removed from the casting when exposed to cryogenic materials such as liquid hydrogen (LH_2).

Following the immediately preceding step of the process, a light or flash nickel plating is applied to the casting in lieu of a nickel strike. Application of the nickel plate requires no electrical current as is generally the case with the processes heretofore employed. The casting is immersed into a relatively hot electroless nickel plating solution, the temperature being about 190° F., and the variation therefrom not to exceed ±5%. The plating rate in this solution is approximately 0.001 inch per hour and the time the casting remains immersed in the solution ranges between 10–15 minutes, the preferred length of time being about 10 minutes.

There are several electroless plating solutions available, all of which may be considered satisfactory. However, one solution to be given special attention comprises about 30 grams per liter of nickel sulfate, 50 grams per liter sodium glycollate, 10 grams per liter sodium hypophosphite, all of which is dissolved in about 1 gallon of distilled water. The quantities mentioned may obviously be adjusted so as to conform to gallon volume as opposed to the liter volume. It is submitted the best results will be obtained if the pH of the solution is adjusted so as to range between about 4–6.

After removal of the casting from the initial nickel plating solution, it is rinsed in the manner heretofore defined, i.e., tap water is first used and then a thorough rinsing with distilled water follows.

In the event any residual electroless nickel particles remain after the water rinsing is completed, the now thinly nickel plated casting is treated with an acidic solution which may be considered an acidic rinse. The acidic rinse preferred consists of about 25% by volume of hydrochloric acid, but it is to be understood other acids may be substituted for the one specifically identified, and the concentration thereof may vary as much as ±25%.

The duration of the acidic rinse ranges between about 1–2 minutes, the optimum length of time being about one minute. Again the casting is rinsed with water in the aforementioned manner.

The casting now is in condition to receive the principal and final nickel plating of nickel. In this step all the pits, voids and surface irregularities not previously filled are filled. An electrolytic nickel plating bath is employed using the aforementioned electroless nickel plating solutions with one exception. Instead of using about 1 gallon of distilled water an equal amount of sulfamic acid is used. With this solution, nickel deposits on the casting at a rate of 0.002–0.003 inch per hour, and the casting remains in the solution between 2–10 hours, the optimum length of time being about 5 hours. The solution is continuously filtered and agitated in any suitable manner, the preferred form of agitation being with a cathode rocker arm, although the agitation occurring with the filtering may in some circumstances be adequate. The filtering is a standard procedure to eliminate precipitated particles from the solution, because such particles could deposit on the casting to thereby cause imperfections in the plating.

The temperature of the plating bath may range ±5% of the preferred temperature bath of about 115° F. and the pH is to be adjusted to provide the aforementioned acidic condition.

Attention is directed to the importance of temperature control. Wide temperature changes cause large variations in the mechanical properties and in the internal stress in electroformed nickel. Therefore, maintaining the temperature within the ranges specified in all the aforementioned steps is important in order to achieve the desired final result.

Another matter which is often neglected or overlooked is the importance of achieving proper agitation which has a considerable influence on the mechanical properties of electroformed nickel. In circumstances where the mechanical properties of the electroformed nickel is not critical, any of several agitation techniques may be employed, but in the present instance, the mechanical properties are of considerable concern and the use of the defined agitation method is preferred.

Prior to the immediately preceding step the casting was held throughout the process with stainless steel wire. This assures no local imperfections in the plating will occur. As an example, if copper wire was used in the preceding steps, some of the copper would dissolve and then redeposit itself on the casting. This would alter the surface structure locally and affect the quality of the plating. However, in the last developed step of the process or the final nickel deposition step, a good heavy conductor is required as an aid. It has been found copper is, of course, one of the best. Therefore, copper for holding the casting is substituted for the stainless steel. Since the casting now has a flash coating of nickel, the copper does not present the aforementioned problem.

After the final deposition of nickel is applied to the casting it is thoroughly rinsed with distilled water and dried. Grinding, lapping, and polishing may now be employed to provide a final surface finish.

The quantities defined throughout the process are a function of part size and tank size. These quantities may by varied by one skilled in the art in acccordance with practice of tank volume to part shape.

With the exception of the two plating steps, the quantities of chemical compounds used may vary as much as ±25%. The quantity of the chemical components used in the two plating steps should not vary more than ±5%. Additionally, the temperature of baths in the two plating steps should not vary more than ±5%.

What is claimed is:

1. A process for nickel plating aluminum which comprises:

(a) etching the aluminum in a bath which produces at least nitrate ions;

(b) applying and then baking on said aluminum a solution having a concentration of 15 grams of sodium silicate dissolved in 1,200 ml. of water, wherein the amount of sodium silicate can vary ±25%, said baked solution substantially filling all surface irregularities;

(c) treating said aluminum with a relatively hot electroless nickel plating solution to thereby apply a flash plate of nickel thereto;

(d) then exposing said flash plated aluminum to an acidic bath to remove residual particles of the solution employed in step (c);

(e) again treating said aluminum with a relatively warm electrolytic nickel plating solution to thereby apply an electrodeposition of nickel thereto, and to fill substantially all surface irregularities not previously filled; and

(f) rinsing said aluminum following steps (a), (c), (d) and (e) to remove the chemical components therefrom.

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