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(73) Proprietor: **HENKEL CORPORATION (a Delaware corp.)**
300 Brookside Avenue
Ambler, PA 19002 (US)

(72) Inventor: **McMillen, Mark W.**
4120 Albert Road
Oxford
Michigan 48051 (US)

(74) Representative: **von Kreisler, Alek,**
Dipl.-Chem. et al
Patentanwälte
von Kreisler-Selting-Werner
Postfach 10 22 41
D-50462 Köln (DE)

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Description

This invention relates to a novel process for cleaning and deoxidizing aluminum prior to conversion treatment. The process is based on an acid or alkaline cleaning system and a chromate-free deoxidizer system.

Traditionally, chromated deoxidizers have been used to enable aluminum samples to pass designated corrosion tests, as for example, MIL-C-5541C. The aluminum is first-cleaned in a relatively non-etching alkaline soak cleaner, deoxidized in an acidic chromate solution and subsequently chromated. Alternatively, if etching was desired, an alkaline etch or alkaline chemical milling solution was used. This step was followed by a deoxidation step which usually employed a chromated deoxidizer, to remove "smut" produced by etching. The aluminum was then chromated. In the past, attempts to replace chromate in the deoxidizing step have generally involved the use of iron salts such as ferric sulfate. However, iron based deoxidizers have never provided results equal to the chromate containing deoxidizer systems.

U.S. Patent 4,451,304 to Batiuk provides a treatment for aluminum which employs an alkaline cleaning step and a deoxidizer step. The deoxidizer step employs a non-chromate deoxidizer which is an aqueous solution of sodium or potassium nitrite. This patent, includes a detailed discussion of the prior art in this area.

US-A-4 459 216 discloses an aqueous acidic dissolving solution for metals: e.g. aluminum and aluminum alloys comprising hydrogen peroxide, an inorganic acid, and at least one aromatic compound, said solution can be used at a temperature of from 10 to 80 °C and for a treating time of from 5 sec. to 15 min.

GB-A-2 186 292 describes a process for cleaning aluminum surfaces employing:

- a prewash with an aqueous solution preferably an acidic prewash solution,
- a primary alkaline cleaner stage to remove residual aluminum from the surface of the treated container without undesirable etching,
- a acidulated water rinse stage with an aqueous rinse solution having a pH of not more than 6, said rinse solution may contain fluoride and/or phosphate ions as an accelerator for the removal of the oxide coating and
- an optional conversion treatment.

GB-A-2100 757 is related to a fluoride-free, low-temperature solution for cleaning aluminum comprising sulfuric acid, phosphoric acid and surfactants.

The present invention is defined by the wording of the independent claims. Further embodiments can be found in the dependent claims.

The present invention is advantageous in that it eliminates the use of hexavalent chromium in the cleaning and deoxidizing step and further provides enhanced performance over previously disclosed non-chromium processes for the cleaning and deoxidizing of aluminum prior to chemical conversion treatments.

In accordance with the present invention, aluminum is cleaned in an aqueous acidic or alkaline solution. The preferred acidulants are acids such as sulfuric acid, mitric acid, hydrofluoric acid, phosphoric acid, citric acid, oxalic acid, acetic acid, gluconic acid, hydroxyacetic acid and the like or mixtures of two or more such acids. Preferably the acidic solution is a dilute solution which provides low etching.

The alkaline cleaning solutions useful in the present invention generally contain alkali metal hydroxides or other water soluble alkaline materials such as trisodium phosphate, alkali silicates, tetrasodium pyrophosphate and the like.

The etchants can also comprise acidic solutions of fluoride compositions such as hydrogen fluoride and fluoride salts, fluoride complexes and the like, such as ammonium bifluoride, fluorosilicic acid, fluorophosphonic acid, its salts, and the like.

Following the cleaning step, the aluminum is then immersed in an aqueous acidic deoxidizer solution containing at least one of the following deoxidizer compositions hydrogen peroxide or heteropoly vanadic acids or its salts. Optionally, the aluminum can be rinsed before contact with the deoxidizer solution. The use of a rinse is preferred.

In another embodiment of the invention the aluminum article can be cleaned and deoxidized in a single step by including a deoxidizer compound such as hydrogen peroxide in the acid cleaning solution.

In the description of the invention, the term "aluminum" used in connection with the substrate metal to be treated, includes aluminum metal per se and also those aluminum alloys which are generally subjected to a cleaning and deoxidizing treatment prior to chromating or other chemical or electrochemical conversion treatment.

Aluminum metal per se invariably contains trace impurities of other metals. Exemplary of such impurity metals are copper, manganese, nickel, zinc, titanium, vanadium, sodium and gallium.

Aluminum alloys generally contain larger amounts of other metals. Such alloying metals include silicon, chromium, lead, iron, copper, magnesium, manganese, and zinc.

Such metals in the form of ions as well as aluminum ions invariably are present in the deoxidizer solutions by virtue of the action of such solutions on the metal and smut. It is one aspect of the present invention that the deoxidizer solutions containing stabilizers can continue to function in the presence of such other metal ions, particularly iron, copper and manganese. The multivalent metal ions tend to catalyze the decomposition of the hydrogen peroxide component. This is especially true with copper and manganese.

In respect to the first step of this invention the aluminum substrate is subjected to cleaning and/or etching preferably with a dilute aqueous acidic solution at a pH of less than 2.

Preferably the pH is below 2 and most preferably below 1.5. The cleaning solutions generally contain from 0.005% to 5% by volume or 10% by weight of the acids but can contain up to 50% by weight, and preferably from 0.2 to 8% by weight and more preferably from 0.4% to 2.5% by volume. It is also preferred to employ a mixture of acids such as sulfuric acid and hydrofluoric acid.

While optional, suitable surfactants can be included in the cleaning solutions. They can be used in amounts of from 0% to 5% by weight, and preferably up to 2% and most preferably up to 0.5% by weight. Any suitable surfactants which are compatible with the cleaning solution and do not leave an undesirable film on the aluminum's surface can be used. Such surfactants include the cationic, anionic, amphoteric and nonionic surfactants.

The cleaning and/or etching step can be done in an alkaline cleaner etching solution. Known alkaline cleaning solutions which contain alkali metal hydroxide, alkali metal carbonate, trialkali metal phosphate (such as trisodium phosphate) tetralkali metal pyrophosphate (tetrasodium pyrophosphate) alkali metal silicates, alone or in combination can also be used in the cleaning step of the present invention. Alkaline cleaning compositious such as Ridoline® 53, P3 Almeco® 18 and Ridoline® 322 can be used in the cleaning step.

In an alternate embodiment of the invention, an acid cleaning-etching step can be combined with the deoxidizer step by including hydrogen peroxide and a hydrogen peroxide stabilizer with an acid cleaning solution. The combined cleaning and deoxidizing steps can be carried out at a temperature of from ambient to 76,6 °C (170 °F) and preferably from 32,2 °C (90 °F) to 65,5 °C (150 °F). The pH of the solution is preferably below 3 and more preferably below 1.5 and most preferably below 0.5.

It will be appreciated that the cleaning-etching step can be conducted in a short period of time with substantially little etching or can be continued for a longer period of time if a deeply etched surface is desired.

It will also be appreciated that the cleaning-etching step can be conducted at ambient temperatures but it is preferred to conduct the cleaning step at elevated solution temperatures. Higher temperatures increase the rate of cleaning and/or etching action of the cleaner solution and a certain degree of care must be exercised to ensure that the desired degree of cleaning or etch is obtained. The temperature of the cleaning/etching solution is preferably from 32,2 °C (90 °F) to 79,4 °C (175 °F) and most preferably from 37,7 °C (100 °F) to 65,5 °C (150 °F).

In respect to the deoxidizing step, it is preferred that the deoxidizer bath be acidic and preferably be below a pH of 4, and more preferably at or below 2. When the heteroploy ions are employed in the deoxidizer bath, they can be added to the bath as such or can be generated in situ from their components.

When heteropoly vanadic acid or its salts are used, the deoxidizer comprises from 0.1 gram/liter to the solubility limit of the material and an acid to form a composition having a pH below 3. Generally, mineral acids are used and nitric acid is preferred.

When the deoxidizer is hydrogen peroxide, it is preferred that the solutions contain from 0.03 to 30% by weight hydrogen peroxide and it is further preferred that one or more stabilizers for hydrogen peroxide also be employed in the solution. With respect to hydrogen peroxide deoxidizer solutions, it is also preferable to adjust the pH to 3 or less and preferably with nitric acid. The pH is preferably not greater than 1.5 and most preferably 0.5 or below. The deoxidizer solution is preferably maintained at a temperature from ambient to 65,5 °C (150 °F) depending on the time the aluminum article is in contact with the solution.

The aluminum article preferably is in contact with the deoxidizer solution from 1 to 25 minutes. The contact time is generally shorter at higher temperatures.

Stabilizers for peroxides are well known and are exemplified in U.S. Patent 4,059,678. Other suitable stabilizers for hydrogen peroxide are well known in the art and any of these can be conveniently used provided they are compatible with acidic solutions. A preferred group of stabilizers is disclosed in U.S. Patent Application Serial No. 07/221,063 filed on July 19, 1988, in the name of Mark McMillen and entitled "Stabilization of Peroxide Solutions corresponding to EP 0351 772 A1.

After the deoxidation treatment, the treated aluminum substrate is then chromated by chromating treatments for aluminum, which are well known in the art. Chromating with chromating compositions such Alodine® 1200S or Alodine® 600 product of Parker + Amchem a division of Henkel Corporation are useful. The aluminum substrates are preferably rinsed before being treated with the chromating compositions. Rinsing is preferred to reduce contamination of the chromating composition with the components of the deoxidizing treatment composition.

The examples which follow are intended to illustrate this invention and are not to be considered limiting.

EXAMPLE 1

A cleaning solution was prepared as follows:

A. 70 ml of a solution prepared by diluting 323 ml of 96% sulfuric acid with water to one liter.

B: 70 ml of hydrofluoric acid concentrate solution prepared by diluting 214 grams of 70% hydrofluoric acid to one liter. 70 ml A and 70 ml B were mixed and diluted to 7 liters with a mixture of 25% Ambler Borough (PA.) tap water and 75% deionized water. The mixture of tap water and deionized water had a conductance of 250 micromhos.

A deoxidizer solution was prepared as follows:

A concentrate was first prepared by blending 3800 ml of 35% hydrogen peroxide and 200 ml of Dowfax® 241 (sodium dodecyl diphenyl oxide disulfonate).

1610 ml of the hydrogen peroxide concentrate as prepared above and 475 ml of 70% nitric acid were diluted to 18.9 liters.

Panels of aluminum alloy 2024-T3 were immersed in the cleaning solution at a temperature of 48,8 °C (120 °F) for a period of ten minutes. The panels were then removed, rinsed with deionized water and immersed in the deoxidizer solution at ambient temperature for 15 minutes. The panels were rinsed and subsequently chromated. The chromated panels successfully passed MIL-C-5541C test for neutral salt spray specifications. The MIL-C-5541C specification and test methods are incorporated herein by reference.

EXAMPLE 2

A cleaner solution concentrate was prepared by blending 100 ml of 96% sulfuric acid and 416 ml of 75% phosphoric acid and diluting to one liter.

A cleaner solution was prepared by mixing 140 ml of cleaner solution and 70 ml of hydrofluoric acid solution concentrate as prepared in Example 1 and diluting to 7 liters. The deoxidizer solution used in this example was identical to that used in Example 1.

Panels of aluminum alloy, 2024-T3, were immersed in the above cleaning solution at a temperature of 48,8 °C (120 °F) for a period of 10 minutes. Thereafter the panels were removed, rinsed with water and immersed in the deoxidizer solution at ambient temperatures for a period of 15 minutes. Thereafter, the panels were rinsed and chromated. These panels passed the MIL-C-5541C neutral salt spray specifications.

EXAMPLE 3

A cleaner concentrate solution was prepared by blending 256 ml of 96% sulfuric acid, 90 grams of Mirawet® B (amphoteric surfactant), 90 grams of Surfonic® LF-17 (ethoxylated alcohol) and diluting to one liter with water.

4 liter cleaning baths were prepared containing 50 mls of the cleaner concentrate to which varying amounts of hydrofluoric acid were added as shown in Tables I and II.

The amount of hydrofluoric acid added in the baths set forth in Tables I and II and based on the hydrofluoric acid concentrate as prepared in Example 1. In respect to the .17% solutions, 40 milliliters of the hydrofluoric concentrate were added. In respect to the .09% solutions, 21 milliliters of the hydrofluoric concentrate were added, and in respect to the 0.009%, 2 milliliters of the hydrofluoric acid concentrate were added.

A 4 liter deoxidizer bath was prepared in a manner similar to that of Examples 1 above. The ingredients employed in the deoxidizer baths are set forth in Tables I and II below.

In respect to the baths exemplified in Table 1, it should be noted that reagent grade 70% nitric acid was used and the stabilizer of Table I was Dowfax® 2A1 (sodium dodecyl diphenyl oxide disulfonate). Further in respect to the baths of Table I, 10.2 milliliters of the stabilizer were added to obtain the .255% stabilizer concentration and 0.4 milliliters of the stabilizer were added to get the 0.01% stabilizer concentration.

In respect to the peroxide, 400 milliliters of 35% hydrogen peroxide diluted to one liter with water, were used for the 10% concentrations and 240 milliliters were used for the 6% concentrations.

In respect to the deoxidizer baths of Table II, commerical grade nitric acid at 67.5% was employed and the stabilizer was Monofax® 1214 (alkyl phosphate ester). In respect to the nitric acidic 400 milliliters of the nitric acid were used for the 10% solution, 240 milliliters ofr 6% solutions and 80 milliliters for the 2% solutions. Furthermore in respect to the peroxide, 400 milliliters were used for the 10% solution, 240 milliliters for the 6% solution and 80 milliliters for the 2% solution were employed.

TABLE I

CLEANER			DEOXIDIZER			
96% Sulfuric Acid	70% HF	Surfactant	35% Peroxide	Stabilizer	pH Adj. w/HNO	Time
0.3%	0.17%	0.2%	6%	0.255%	0.5	10 Min
0.3%	0.09%	0.2%	6%	0.01%	1.0	5 Min
0.3%	0.09%	0.2%	6%	0.255%	0.5	15 Min
0.3%	0.09%	0.2%	10%	0.255%	0.5	10 Min

TABLE II

CLEANER			DEOXIDIZER			
96% Sulfuric Acid	70% HF	Surfactant	35% Peroxide	Stabilizer	42 ° Be HNO	Time
0.3%	0.17%	0.2%	6%	0.1g/l	6%	15 Min
0.3%	0.09%	0.2%	10%	2.55g/l	6%	15 Min
0.3%	0.09%	0.2%	10%	2.55g/l	6%	15 Min
0.3%	0.09%	0.2%	10%	2.55g/l	6%	5 Min
0.3%	0.09%	0.2%	6%	2.55g/l	6%	10 Min
0.3%	0.009%	0.2%	2%	5g/l	6%	10 Min
0.3%	0.17%	0.2%	6%	2.55g/l	10%	10 Min
0.3%	0.17%	0.2%	6%	2.55g/l	2%	10 Min

Aluminum panels of alloy, 2024-T3, were cleaned in a specified cleaner bath set forth in Table I and Table II above. The panels were then rinsed and immersed in the deoxidizer bath as indicated in Tables I and II above, each for a specified period of time listed. After the deoxidizing step, the panels were removed, rinsed and chromated. The chromated panels were then tested in neutral salt spray for 336 hours (twice as long as MIL-C-5541C requires). All panels showed no pitting and no corrosion after testing.

It has been found that preferred systems are those wherein the cleaning solution contains 0.3% by volume sulfuric acid (96%), 0.17% by volume hydrofluoric acid (70%), and up to 0.2% by volume sodium 2-ethyl hexyl sulfate surfactant for a strong etch cleaner. This cleaner should be used for 5-10 minutes at temperatures of from about 43,3 ° C (110 ° F) to about 54,4 ° C (130 ° F).

It is preferred to then subject the aluminum panels to a deoxidiziang step for about 10-15 minutes by immersion in a deoxidizing bath of preferrably 8% by volume hydrogen peroxide (35%), 3% by volume (42 ° Bé) nitric acid and up to 0.5% of a stabilizer for the peroxide (preferrably MIRATAINE® CBS (cocoamidopropyl hydroxy sultaine).

When it is desired to have an etching cleaner with lower etching properties, it is preferred to employ a cleaning solution of 0.5% by volume sulfuric acid (96%), 1.7% phosphoric acid by volume (75%), and up to 0.2% of a surfactant combination which is a mixture of Triton® N-100 and Surfonic® LF-17. This is used at a temperature of from about 24,1 ° C (110 ° F) to about 60 ° C (140 ° F) for 5 to 10 minutes followed by the same deoxidizer step as established above for from about 5 to 15 minutes.

EXAMPLE 4

A cleaner-etching-deoxidizer bath was prepared containing 8% H_2O_2, 2.5% HNO_3, 1.5% phosphoric acid and 2 ml/liter of Triton® X-102 surfactant (octylphenoxy polyethoxy ethanol). Aluminum panels were

immersed in the cleaning-etching deoxidizer solution for 10 minutes at 48,8 °C (120 °F). The panels were rinsed and then chromated in an Alodine® 1200S chromating solution according to manufacturer's recommendations. The chromated panels were then tested in neutral salt spray for 336 hours. There was no pitting or corrosion of the aluminum panels.

EXAMPLE 5

A cleaner-etching-deoxidizer bath was prepared containing 8% H_2O_2, 2.5% HNO_3, 1 ml/liter of 79% HBF_4 and 2 ml/liter Triton® X-102 surfactant. Aluminum panels were immersed in the cleaner-etching-deoxidizer solution for 10 minutes at 48,8 °C (120 °F). The panels were rinsed and then chromated in an Alodine® 1200S solution according to manufacturer's recommendations. The chromated panels were then tested in neutral salt spray for 336 hours. There was no pitting or corrosion of the aluminum panels.

The combining of the acid cleaning-etch step with the deoxidizer step into one treating step is an advance in the art. The combining of two steps into a single step reduces the equipment necessary to provide a commercial process and in addition reduces the processing time for each aluminum piece.

Claims

1. A process for treating an aluminum article which comprises: cleaning, deoxidizing and conversion coating of the aluminum article characterized by deoxidizing the aluminium article by contacting the aluminium article with an acidic, stabilized hydrogen peroxide solution, an acidic heteropoly vanadic acid solution or an acidic heteropoly vanadic acid salt solution.

2. A process of Claim 1 wherein the pH of the acidic stabilized hydrogen peroxide solution is at a pH below 3.

3. A process of Claim 1 wherein the aluminum article is cleaned by contact with an alkaline cleaning solution.

4. A process of Claim 3 wherein the cleaning solution is at a temperature of from 32 °C (90 °F) to 80 °C (175 °F).

5. A process of Claim 3 wherein the deoxidizing solution is a stabilized hydrogen peroxide solution at a temperature of from ambient to 65 °C (150 °F) and at a pH below 3.

6. A process of Claim 5 wherein the pH is below 0,5.

7. A process of Claim 1 wherein the aluminum article is cleaned by contact with an aqueaus acidic solution at a pH below 2.

8. A process of Claim 7 wherein the aluminum article, after contact with the acidic cleaning solution, is contacted with a stabilized hydrogen peroxide solution at a pH below 1.5.

9. A process of Claim 8 wherein the pH of the stabilized hydrogen peroxide deoxidizing solution is below 0.5.

10. A process of Claim 7 which comprises:

a. Contacting an aluminum article with an acidic solution at a temperature of from 32 °C (90 °F) to 80 °C(175 °F), at a pH below 2 for from 1 to 15 minutes to clean the aluminum article; and

b. Contacting the cleaned aluminum article with an acidic stabilized hydrogen peroxide solution at a temperature from about ambient to 65 °C(150 °F) for from 1 to 25 minutes to deoxidize the aluminum article.

11. A two step process of Claim 1 wherein the combined cleanning and deoxidizing step comprises: Contacting an aluminum article with a cleaning-etching-deoxidizing acid stabilized hydrogen peroxide solution at a temperature of from 32 °C (90 °F) to 80 °C (175 °F) for from 2 to 25 minutes, wherein the pH of the solution is below 2.

12. A process of Claim 11 wherein the pH is below 0,5.

13. A process of Claim 11 wherein the cleaning-etching-deoxidizing solution comprises:
a) hydrogen peroxide in an amount of 0,03 to 30% by weight.
b) 0.1 to 5 g/l stabilizer for the hydrogen peroxide
c) nitric acid in an amount of 0.5 to 10% by weight of 42° Baumé
d) a source of fluoride ion resulting in 0.009 to 0,17% by weight of HF.

14. A Process of Claim 11 wherein the cleaning-etching-deoxidizing solution comprises:
a) hydrogen peroxide in an amount of 0.003% to 30% by weight
b) a 0.1 to 5g/l stabilizer for the hydrogen peroxide
c) phosphoric acid
d) nitric acid in an amount of 0.5 to 10% by weight of 42° Baumé.

15. A process of Claim 11 wherein the deoxidized article is conversion coated by contact with a chromate containing conversion coating composition.

16. A process of Claim 1 wherein the aluminum is cleaned by contact with a cleaning solution comprising 0.3% sulfuric acid (96%), 0.17% hydrofluoric acid (70%), and 0.2% sodium 2-ethyl hexyl sulfate and wherein the aluminum substrate is cleaned in this solution for a period of 5 to 10 minutes at a temperature of from 43°C (110°F) to 54°C (130°F).

17. A process of Claim 1 wherein the deoxidizing solution comprises 8% by volume hydrogen peroxide (35%), 3% by volume 42°Bé nitric acid and wherein the aluminum is immersed in the deoxidizing solution for a period of from 10 to 15 minutes at ambient temperatures.

18. A process of Claim 1 wherein the aluminum is cleaned by contact with a cleaning solution which comprises about 0.5% sulfuric acid (96%), 1.7% phosphoric acid (75%), and up to 0.2% of a surfactant and the aluminum is treated in this solution for a period of from 5 to 10 minutes at a temperature of 43°C (110 F) to 60°C (140°F).

19. A process of Claim 1 wherein the aluminium article is cleaned by contact with an acidic cleaning solution containing up to 10% by weight of at least one acid and from 0 to 5% by weight of a surfactant wherein the pH of the acidic cleaning solution is less than 2.

20. A process according to Claim 1, wherein the stabilized hydrogen peroxide solution contains from 0.03 to 30% by weight hydrogen peroxide and is at a pH not greater than 1.5.

21. A process of Claim 1 wherein the conversion coating step comprises contact with a chromate containing conversion coating composition.

Patentansprüche

1. Verfahren zur Behandlung eines Aluminium-Gegenstandes, umfassend das Desoxidieren und Umwandlungsbeschichten des Aluminium-Gegenstandes, dadurch gekennzeichnet, daß der Aluminium-Gegenstand durch In-Berührung-Bringen des Aluminium-Gegenstandes mit einer sauren, stabilisierten Wasserstoffperoxid-Lösung, einer sauren Heteropolyvanadinsäure-Lösung oder einer sauren Lösung eines Salzes einer Heteropolyvanadinsäure desoxidiert wird.

2. Verfahren nach Anspruch 1, worin der pH-wert der sauren stabilisierten Wasserstoffperoxid-Lösung kleiner als 3 ist.

3. Verfahren nach Anspruch 1, worin der Aluminium-Gegenstand durch Berührung mit einer alkalischen Reinigungs-Lösung gereinigt wird.

4. Verfahren nach Anspruch 3, worin die Reinigungs-Lösung eine Temperatur von 32 °C (90 °F) bis 80 °C (175 °F) hat.

5. Verfahren nach Anspruch 3, worin die desoxidierende Lösung eine stabilisierte Wasserstoffperoxid-Lösung bei einer Temperatur von Umgebungs-Temperatur bis 65 °C (150 °F) und einem pH-wert unterhalb von 3 ist.

6. Verfahren nach Anspruch 5, worin der pH-Wert unterhalb von 0,5 liegt.

7. Verfahren nach Anspruch 1, worin der Aluminium-Gegenstand durch Berührung mit einer sauren wäßrigen Lösung bei einem pH-Wert unterhalb von 2 gereinigt wird.

8. Verfahren nach Anspruch 7, worin der Aluminium-Gegenstand nach der Berührung mit der sauren Reinigungs-Lösung mit einer sauren, stabilisierten Wasserstoffperoxid-Lösung bei einem pH-Wert unterhalb von 1,5 in Berührung gebracht wird.

9. Verfahren nach Anspruch 8, worin der pH-Wert der desoxidierenden stabilisierten Wasserstoffperoxid-Lösung unterhalb von 0,5 liegt.

10. Verfahren nach Anspruch 7, umfassend
    a. das In-Berührung-Bringen eines Aluminium-Gegenstandes mit einer sauren Lösung bei einer Temperatur von 32 °C (90 °F) bis 80 °C (175 °F) bei einem pH-Wert unterhalb von 2 während 1 bis 15 min, um den Aluminium-Gegenstand zu reinigen; und
    b. das In-Berührung-Bringen des gereinigten Aluminium-Gegenstandes mit einer sauren stabilisierten Wasserstoffperoxid-Lösung bei einer Temperatur von Umgebungs-Temperatur bis 65 °C (150 °F) während 1 bis 25 min, um den Aluminium-Gegenstand zu desoxidieren.

11. Zweistufiges Verfahren nach Anspruch 1, worin der kombinierte Schritt des Reinigens und Desoxidierens umfaßt: In-Berührung-Bringen eines Aluminium-Gegenstandes mit einer durch Säure stabilisierten Wasserstoffperoxid-Reinigungs-Ätz-Desoxidierungs-Lösung bei einer Temperatur von 32 °C (90 °F) bis 80 °C (175 °F) während 2 bis 25 min, wobei der pH-Wert der Lösung unterhalb von 2 liegt.

12. Verfahren nach Anspruch 11, worin der pH-Wert der Lösung unterhalb von 0,5 liegt.

13. Verfahren nach Anspruch 11, worin die Reinigungs-Ätz-Desoxidierungs-Lösung umfaßt:
    a) Wasserstoffperoxid in einer Menge von 0,03 bis 30 Gew.-%,
    b) 0,1 bis 5 g/l Stabilisator für das Wasserstoffperoxid,
    c) Salpetersäure in einer Menge von 0,5 bis 10 Gew.-% 42° Baumé,
    d) eine Quelle für Fluorid-Ionen, die in 0,009 bis 0,17 Gew.-% HF resultiert.

14. Verfahren nach Anspruch 11, worin die Reinigungs-Ätz-Desoxidierungs-Lösung umfaßt:
    a) Wasserstoffperoxid in einer Menge von 0,003 bis 30 Gew.-%,
    b) 0,1 bis 5 g/l Stabilisator für das Wasserstoffperoxid,
    c) Phosphorsäure,
    d) Salpetersäure in einer Menge von 0,5 bis 10 Gew.-% 42° Baumé.

15. Verfahren nach Anspruch 11, worin der desoxidierte Gegenstand durch Berührung mit einer Chromat enthaltenden Umwandlungsbeschichtungs-Zusammensetzung umwandlungsbeschichtet wird.

16. Verfahren nach Anspruch 1, worin das Aluminium durch Berührung mit einer Reinigungs-Lösung gereinigt wird, die 0,3 % Schwefelsäure (96 %), 0,17 % Flußsäure (70 %) und 0,2 % Natrium-2-ethylhexylsulfat umfaßt, und worin das Aluminium-Substrat während einer Zeitspanne von 5 bis 10 min bei einer Temperatur von 43 °C (110 °F) bis 54 °C (130 °F) gereinigt wird.

17. Verfahren nach Anspruch 1, worin die desoxidierende Lösung 8 Vol.-% Wasserstoffperoxid (35 %), 3 % Salpetersäure (42° Bé) umfaßt und worin das Aluminium eine Zeitspanne von 10 bis 15 min bei Umgebungs-Temperatur in die desoxidierende Lösung eingetaucht wird.

18. Verfahren nach Anspruch 1, worin das Aluminium durch Berührung mit einer Reinigungs-Lösung gereinigt wird, die etwa 0,5 % Schwefelsäure (96 %), 1,7 % Phosphorsäure (75 %) und bis zu 0,2 % eines Tensids umfaßt, und worin das Aluminium in dieser Lösung während einer Zeitspanne von 5 bis

10 min bei einer Temperatur von 43 °C (110 °F) bis 60 °C (140 °F) behandelt wird.

19. Verfahren nach Anspruch 1, worin der Aluminium-Gegenstand durch Berührung mit einer sauren Reinigungs-Lösung gereinigt wird, die bis zu 10 Gew.-% wenigstens einer Säure und 0 bis 5 Gew.-% eines Tensids enthält, worin der pH-Wert der sauren Reinigungs-Lösung kleiner als 2 ist.

20. Verfahren nach Anspruch 1, worin die stabilisierte Wasserstoffperoxid-Lösung 0,03 bis 30 Gew.-% Wasserstoffperoxid enthält und einen pH-Wert hat, der nicht höher als 1,5 ist.

21. Verfahren nach Anspruch 1, worin der Schritt des Umwandlungsbeschichtens die Berührung mit einer Chromat enthaltenden Umwandlungsbeschichtungs-Zusammensetzung umfaßt.

Revendications

1. Procédé pour le traitement d'un article en aluminium qui comprend les étapes consistant à : nettoyer, désoxyder et revêtir par conversion l'article en aluminium, caractérisé par le fait de désoxyder l'article en aluminium par mise en contact de celui-ci avec une solution de peroxyde d'hydrogène acide stabilisée, une solution d'acide vanadique hétéropolaire acide ou une solution de sel d'acide vanadique hétéropolaire acide.

2. Procédé selon la revendication 1, dans lequel la valeur de pH de la solution de peroxyde d'hydrogène stabilisée acide est inférieure à 3.

3. Procédé selon la revendication 1, dans lequel l'article en aluminium est nettoyé par mise en contact avec une solution de nettoyage alcaline.

4. Procédé selon la revendication 3, dans lequel la solution de nettoyage est à une température allant de 32 °C (90 °F) jusqu'à 80 °C (175 °F).

5. Procédé selon la revendication 3, dans lequel la solution désoxydante est une solution de peroxyde d'hydrogène stabilisée à une température allant de la température ambiante jusqu'à 65 °C (150 °F) et à une valeur de pH inférieure à 3.

6. Procédé selon la revendication 5, dans lequel la valeur de pH est inférieure à 0,5.

7. Procédé selon la revendication 1, dans lequel l'article en aluminium est nettoyé par mise en contact avec une solution acide aqueuse à une valeur de pH inférieure à 2.

8. Procédé selon la revendication 7, dans lequel l'article en aluminium, après mise en contact avec la solution de nettoyage acide, est mis en contact avec une solution de peroxyde d'hydrogène stabilisée à une valeur de pH inférieure à 1,5.

9. Procédé selon la revendication 8, dans lequel la valeur de pH de la solution désoxydante de peroxyde d'hydrogène stabilisée est inférieure à 0,5.

10. Procédé selon la revendication 7, qui comprend les étapes consistant à :

a. mettre en contact un article en aluminium avec une solution acide à une température allant de 32 °C (90 °F) jusqu'à 80 °C (175 °F) à une valeur de pH inférieure à 2 pendant 1 à 15 minutes pour nettoyer l'article en aluminium ; et

b. mettre en contact l'article en aluminium nettoyé avec une solution de peroxyde d'hydrogène stabilisée acide à une température allant d'environ la température ambiante jusqu'à 65 °C (150 °F) pendant 1 à 25 minutes pour désoxyder l'article en aluminium.

11. Procédé en deux étapes selon la revendication 1, dans lequel l'étape combinée de nettoyage et de désoxydation comprend : la mise en contact d'un article en aluminium avec une solution de peroxyde d'hydrogène stabilisée acide nettoyante-décapante-désoxydante à une température allant de 32 °C (90 °F) jusqu'à 80 °C (175 °F) pendant de 2 à 25 minutes, dans lequel la valeur de pH de la solution est inférieure à 2.

12. Procédé selon la revendication 11, dans lequel la valeur du pH est inférieure à 0,5.

13. Procédé selon la revendication 11, dans lequel la solution nettoyante-décapante-désoxydante comprend :

a) du peroxyde d'hydrogène dans une quantité de 0,03 à 30 % en poids
b) 0,1 à 5 g/l de stabilisant pour le peroxyde d'hydrogène
c) de l'acide nitrique dans une quantité de 0,5 à 10 % en poids de 42° Baumé
d) une source d'ions fluorure donnant 0,009 jusqu'à 0,17 % en poids de HF.

14. Procédé selon la revendication 11, dans lequel la solution nettoyante-décapante-désoxydante comprend :

a) du peroxyde d'hydrogène dans une quantité de 0,003 jusqu'à 30 % en poids
b) 0,1 à 5 g/l de stabilisant pour le peroxyde d'hydrogène
c) de l'acide phosphorique
d) de l'acide nitrique dans une quantité de 0,5 à 10 % en poids de 42° Baumé.

15. Procédé selon la revendication 11, dans lequel l'article désoxydé est revêtu par conversion par mise en contact avec un chromate contenant une composition de revêtement par conversion.

16. Procédé selon la revendication 1, dans lequel l'aluminium est nettoyé par mise en contact avec une solution nettoyante comprenant 0,3 % d'acide sulfurique (96 %), 0,17 % d'acide fluorhydrique (70 %) et 0,2 % de 2-éthyl hexylsulfate de sodium et dans lequel le substrat d'aluminium est nettoyé dans cette solution pendant une durée de 5 à 10 minutes à une température allant de 43°C (110°F) jusqu'à 54°C (130°F).

17. Procédé selon la revendication 1, dans lequel la solution désoxydante comprend 8 % en volume de peroxyde d'hydrogène (35 %), 3 % en volume d'acide nitrique 42° Baumé et dans lequel l'aluminium est immergé dans la solution désoxydante pendant une durée allant de 10 à 15 minutes à température ambiante.

18. Procédé selon la revendication 1, dans lequel l'aluminium est nettoyé par mise en contact avec une solution de nettoyage qui comprend environ 0,5 % d'acide sulfurique (96 %), 1,7 % d'acide phosphorique (75 %) et jusqu'à 0,2 % d'un agent tensioactif et l'aluminium est traité dans cette solution pendant une durée allant de 5 à 10 minutes à une température de 43°C (110°F) jusqu'à 60°C (140°F).

19. Procédé selon la revendication 1, dans lequel l'article en aluminium est nettoyé par mise en contact avec une solution de nettoyage acide contenant jusqu'à 10 % en poids d'au moins un acide et de 0 à 5 % en poids d'un agent tensioactif dans lequel la valeur de pH de la solution nettoyante acide est inférieure à 2.

20. Procédé selon la revendication 1, dans lequel la solution de peroxyde d'hydrogène stabilisée contient de 0,03 à 30 % en poids de peroxyde d'hydrogène et la valeur de pH n'est pas supérieure à 1,5.

21. Procédé selon la revendication 1, dans lequel l'étape de revêtement par conversion comprend la mise en contact avec un chromate contenant une composition de revêtement par conversion.