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3,690,949

ALKALINE BATH FOR NONETCHING REMOVAL OF SCALE FROM TITANIUM WORKPIECES

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16 Claims

ABSTRACT OF THE DISCLOSURE

Titanium scale, generally comprising oxides and nitrides of titanium, may be removed from titanium workpieces without simultaneously rapidly etching the workpiece by subjecting the workpiece to a solution in water of from 2 to 10 pounds per gallon of the mixture consisting essentially of sodium or potassium nitrite, 2–25 weight percent, sodium or potassium gluconate 2–25 weight percent, sodium or potassium nitrate, 2–25 weight percent, and sodium hydroxide, balance to 100 weight percent, e.g., by immersing the workpiece in an elevated temperature bath at temperatures between about 200° F. and 300° F.

BACKGROUND OF THE INVENTION

(1) Field of the invention

This invention has to do with the removal of scale, such as oxides, nitrides, carbides and the like from the surfaces of titanium workpieces. Titanium scale and particularly oxides of titanium constitutes virtually the most refractory of all known materials. Titanium oxides have very high melting points and are resistant to almost all types of chemical attack. Many uses of titanium require the removal of the natural oxide coating or an oxide coating induced by metal treating processes such as heat treating, anodizing or oxides produced in the use of a part.

(2) Prior art

The heretofore accepted method for descaling titanium and removing the oxides from workpiece surfaces has included an alkaline precleaning step, sometimes followed by an alkaline conditioning step and a post treatment in a bath of nitric-hydrofluoric acid. Titanium oxide has been thought to be impervious to alkaline solutions and accordingly, efforts at improving the cleaning of titanium workpieces have been directed primarily at the development of more vigorous and more effective acids.

In copending application Ser. No. 837,950, assigned to the same assignee as this application, now U.S. Pat. 3,553,015, a highly effective alkaline descaling system is disclosed. However, that system etches titanium workpieces and where etching is not wanted, its use is not desirable. The present system is as effective in descaling but without etching the workpiece.

SUMMARY OF THE INVENTION

It has now been discovered that a highly alkaline solution such as an aqueous solution of sodium hydroxide is effective to remove scale from titanium workpieces where the scale removing solution contains certain amounts of sodium or potassium nitrate, nitrite and gluconate salts. Sodium gluconate may be formed in situ in a descaling bath comprising the solution of reaction product of gluconic acid and sodium hydroxide.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

In carrying out the present invention, a treating solution is prepared by dissolving in water from 2 to 10 pounds and preferably about 8 pounds of a mixture of sodium or potassium nitrite, sodium or potassium nitrate, sodium

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or potassium gluconate, and sodium hydroxide. The solution is heated to elevated temperature, generally about 175° F. up to just below the boiling point of the solution and preferably between about 200°–300° F.

The mixture dissolved in the solution consists essentially of sodium or potassium nitrite at 2–25 weight percent and preferably 5 weight percent sodium nitrite, sodium or potassium nitrate at 2–25 weight percent and preferably 5 weight percent sodium nitrate, sodium or potassium gluconate at 2 to 25 weight percent and preferably 5 weight percent sodium gluconate and the balance to 100 percent of sodium hydroxide.

The workpiece which may be degreased by conventional procedures in advance of scale removal, is subjected to the action of the treating solution having the composition indicated above, by applying the solution to the workpiece, e.g. by spraying, brushing, flow coating and, particularly by immersion in a bath comprising the solution. When immersed in a bath this may range from five minutes up to thirty minutes or more, e.g. up to 8 hours for badly scaled forgings and castings. Multiple immersions in successive or the same aqueous bath can be used where desired.

The titanium workpiece following treatment is free of scale particularly of heat scale and welding discoloration and the workpiece surface is left suitable for use subsequently in welding, bonding, plating or painting. Thus, the descaling solution herein disclosed serves as a cleaner and a pickle for titanium.

Titanium alloys can be similarly treated as titanium workpieces. Titanium alloys with such materials as aluminum, vanadium, molybdenum, iron and nickel, e.g. up to 10 percent by weight of the alloy, may be cleaned by the indicated procedures, as can niobium and tantalum metals.

EXAMPLE 1

All parts and percentages are by weight

A titanium scale removal bath was prepared as follows: A mixture of 5 percent sodium nitrite, 5 percent sodium nitrate, 5 percent sodium gluconate, and correspondingly 85 percent sodium hydroxide was mixed dry. An exothermic reaction took place with introduction of water. The mixture was dissolved at the rate of 8 pounds per gallon of bath in water. The water solution was heated to 260° F. Titanium workpieces having oxides and nitride scale on their surfaces resulting from heat treating were immersed in the bath for periods ranging from 5 minutes to thirty minutes. The workpieces were removed from the bath, when the scale thereon had been removed, rinsed and dried. The workpiece surfaces were noted to be clean and suitable for welding, bonding, plating or painting operations.

CONTROL A

Example 1 is duplicated but omitting the sodium gluconate. There was no appreciable removal of scale in an hour and a half.

CONTROL B

Example 1 is duplicated employing a mixture of sodium hydroxide and sodium gluconate. The resulting bath attacked the titanium oxide, but at a slow rate, requiring at least five times as long for removal. In addition, smut was produced on the titanium surface which was removable only with a dip in 5–30 percent HNO₃.

CONTROLS C, D AND E

Example 1 is duplicated but omitting the sodium hydroxide, sodium nitrite or sodium nitrate. The resulting baths were ineffective for removing titanium scale from titanium workpieces.

Accordingly, combination of any two of the cleaning

bath components is not effective as a descaler due to slowness of attack on the substrate and oxide and uneconomically long processing times.

CONTROL F

A titanium scale removal bath was prepared, according to the teachings of U.S. Ser. No. 837,950, as follows: A mixture of 18.5 percent sodium gluconate, 13.5 percent triethanolamine, and correspondingly 68 percent sodium hydroxide was dissolved at the rate of 3.1 to 5 pounds per gallon of bath in water. The water solution was heated to 200° F. Scaled titanium workpieces were removed from the bath, rinsed and dried. The workpieces were noted to be clean and visibly etched to a dull matte finish to break up the sheen on ground areas and obliterate the grinding marks.

In the typical practice of the present invention, contact times between the solution and the workpiece will range between five and ninety minutes with longer or shorter periods, depending on scale conditions, temperature of the solution and concentration of the solution.

EXAMPLE 2

A solution having the composition given for the bath in Example 1 and at 260° F. is sprayed onto a scaled titanium workpiece in a closed chamber for 5 minutes. The workpiece is thereafter rinsed and dried. The workpiece surfaces are clean and free of scale. It is estimated the workpiece would have required 10 to 15 minutes to be cleaned in an immersion bath.

I claim:

1. Method of descaling titanium comprising subjecting a scaled titanium workpiece to a solution, heated to above about 175° F., of from 2 to 10 pounds per gallon of a mixture free of triethanolamine consisting essentially of

	Weight percent
Sodium or potassium nitrite	2-25
Sodium or potassium nitrate	2-25
Sodium or potassium gluconate	2-25
Sodium hydroxide, balance to 100.	

made up to a gallon with water and removing the workpiece from the bath following scale removal.

2. Method according to claim 1 in which said mixture contains 5 percent by weight of sodium nitrite.

3. Method according to claim 1 in which said mixture contains 5 percent by weight of sodium nitrate.

4. Method according to claim 1 in which said mixture contains 5 percent by weight sodium gluconate.

5. Method according to claim 1 in which each salt is present in an amount of 5 percent by weight in the mixture.

6. Method according to claim 1 in which said bath has a temperature between 200° and 300° F.

7. Method of descaling titanium comprising immersing a scaled titanium workpiece in a bath heated above 175° F., said bath being a solution of from 2 to 10 pounds per gallon of a mixture consisting essentially of

Weight percent

Sodium or potassium nitrite	2-25
Sodium or potassium nitrate	2-25
Sodium or potassium gluconate	2-25
Sodium hydroxide, balance to 100.	

made up to a gallon with water and removing the workpiece from the bath following scale removal.

8. Method according to claim 7 in which said mixture contains 5 percent by weight of sodium nitrite.

9. Method according to claim 7 in which said mixture contains 5 percent by weight of sodium nitrate.

10. Method according to claim 7 in which said mixture contains 5 percent by weight sodium gluconate.

11. Method according to claim 7 in which each salt is present in an amount of 5 percent by weight in the mixture.

12. Method according to claim 7 in which said bath has a temperature between 200° and 300° F.

13. Method according to claim 7 in which said workpiece is immersed in the bath for a period of 5 to 90 minutes.

14. Method according to claim 7 in which said bath contains 8 pounds of said per gallon.

15. Method of descaling titanium comprising immersing a scaled titanium workpiece for about 15 minutes in a bath heated to about 260° F., said bath being a solution of about 8 pounds per gallon of a mixture consisting essentially of

	Weight percent
Sodium nitrite	5
Sodium nitrate	5
Sodium gluconate	5
Sodium hydroxide, balance to 100.	

made up to a gallon with water and removing the workpiece from the bath.

16. Method according to claim 15 in which said bath is free of triethanolamine.

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