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2,711,364

POLISHING METALS AND COMPOSITION THEREFOR

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This invention deals with a method of polishing metal articles which predominantly consist of metals of Group IV of the Periodic System of the Elements, such as zirconium, hafnium, titanium, lead, tin, or alloys containing at least one of these metals as the predominant ingredient. More particularly, the invention deals with a process of polishing the aforementioned metals by chemical means, for instance as a preparatory step for electroplating. The compositions used in this novel method form also part of this invention.

Hydrofluoric acid has been used for polishing the Group IV-metals; however, this method was found unsatisfactory because the reaction was too violent and too difficult to control. A mixture of hydrofluoric acid and ammonium fluoride has also been employed and this mixture has given very satisfactory results. This method using hydrofluoric acid and ammonium fluoride forms the subject matter of copending application, Serial No. 355,858, filed by John G. Beach et al. on May 15, 1953. However, in some instances, for instance when zirconium contains occlusions of zirconium carbide, a smooth surface cannot be obtained with a solution of ammonium bifluoride or a mixture of ammonium fluoride and hydrofluoric acid; the surface then shows a relief pattern formed by zirconium carbide granules.

A mixture of hydrofluoric acid, ammonium fluoride and nitric acid was also examined for polishing Group VI-metals. A mixture containing 60 grams of ammonium bifluoride per liter in a 50% (by volume) nitric acid (70%) was used for polishing zirconium containing some zirconium carbide; it was found that both the zirconium metal and the zirconium carbide dissolved at an equal rate so that no relief pattern was noticeable after treatment and the surface obtained was smooth and bright. However, a temperature of 130° F. was necessary for this kind of polishing solution. The necessity of using this relatively high temperature is a disadvantage, since the reaction is exothermic and the temperature is liable to go above 130° F. and the mixture is very unstable at temperatures above 140° F. and starts to decompose. It is very difficult to control the operating temperature within a range as narrow as that from 130 to 140° F. Another disadvantage frequently encountered with the bifluoride-nitric acid mixture is the formation of noxious fumes at these high temperatures.

It is an object of this invention to provide a method and a solution for polishing Group IV-metals by which the above-described disadvantages are overcome.

It is another object of this invention to provide a method and a solution for polishing Group IV-metals whereby the metals are made suitable for plating, and in particular for electroplating, so that the coating obtained thereby is bonded firmly to the polished article.

It is another object of this invention to provide a method and a solution for polishing Group IV-metals whereby the reaction can be controlled easily.

It is still another object of this invention to provide a method and a solution for polishing Group IV-metals whereby no noxious fumes are developed.

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It is finally also an object of this invention to provide a method and a solution for polishing Group IV-metals whereby temperatures as low as 60° F. may be satisfactorily employed.

These and other objects are accomplished by immersing the article to be polished in an aqueous solution containing a water-soluble fluoride, nitric acid and fluosilicic acid. The optional operating temperature is about 60° F.; however, it may vary widely.

While no treatment is necessary prior to immersion into the polishing solution, it has been found advantageous to remove any scale adhering to the surface by sand- or vapor-blasting.

The concentrations of the various components of the polishing solution may vary widely. However, ranges of from 350 to 600 cc. of nitric acid (70%), 175 to 400 cc. of fluosilicic acid (30%) and at least 50 grams of a water-soluble fluoride, such as hydrogen fluoride, ammonium bifluoride or ammonium fluoride, all per 1 liter of solution have been found preferable. The content of ammonium fluoride or bifluoride may reach saturation. The best results were obtained with a solution containing 400 cc. of 70% nitric acid, 200 cc. of 30% fluosilicic acid and 50 to 100 grams of ammonium fluoride per liter of solution.

In the following example one embodiment of the invention is given in detail.

Example

Zirconium metal was immersed in a solution containing 100 grams of ammonium bifluoride, 400 cc. of a 70% nitric acid and 200 cc. of a 30% fluosilicic acid per 1 liter of solution. Immersion was carried out at various temperatures, and the thicknesses of zirconium removed thereby in each instance from each surface per minute were measured. The results are summarized below.

	removed/min./surface
70° to 80° F.-----	0.0006"
80° to 85° F.-----	0.0008"
90° to 95° F.-----	0.0018"
100° to 110° F.-----	0.0025"
110° to 120° F.-----	0.004"

All surfaces of the zirconium after the treatments at the various temperatures were smooth, bright and uniform.

Similar results were obtained by treating titanium, tin and lead with a solution identically composed.

Satisfactory results were obtained with Group IV-metals using solutions containing 480 cc. of 70% nitric acid, 320 cc. of a 30% fluosilicic acid and 195 cc. of a 50% hydrofluoric acid, or a solution containing 600 cc. of a 70% nitric acid, 400 cc. of a 30% fluosilicic acid and saturated in ammonium fluoride or ammonium bifluoride, or a solution containing 356 cc. of a 70% nitric acid, 178 cc. of a 30% fluosilicic acid and 109 cc. of a 50% hydrofluoric acid.

The invention is applicable for all purposes where a smooth surface is desired, and in particular for electroplating, as has been mentioned before. For instance, the invention is suitable par excellence as a pretreatment of zirconium prior to electroplating with a metal, e. g. with nickel.

It will be understood that this invention is not to be limited to the details given herein but that it may be modified within the scope of the appended claims.

What is claimed is:

1. A process of polishing the surface of zirconium metal containing zirconium carbide occlusions comprising immersing said surface into an aqueous solution of about 60 to 85° F. consisting, per one liter of solution, of water, at least 50 grams of a water-soluble fluoride, 350 to 600 cc. of concentrated nitric acid and from 175 to 400 cc. of a 30% fluosilicic acid.

2. The process of claim 1 in which the solution contains from 50 to 100 grams of ammonium bifluoride, 400 cc. of a 70% nitric acid, and 200 cc. of a 30% fluosilicic acid per one liter of solution.

3. An aqueous solution for chemically polishing the surfaces of zirconium containing zirconium carbide occlusions consisting, per one liter of solution, of 175 to 400 cc. of a 30% fluosilicic acid, 350 to 600 cc. of a 70% nitric acid, and at least 50 grams of water-soluble fluoride.

4. The solution of claim 3 in which the contents are

from 50 to 100 grams of ammonium bifluoride, 400 cc. of a 70% nitric acid, and 200 cc. of a 30% fluosilicic acid.

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