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SURFACE TREATMENT OF METALLIC URANIUM

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This invention relates to the preparation of uranium metal surfaces for the application of coatings. It is particularly concerned with the treatment of metallic uranium to provide a surface to which adherent electroplates can be applied.

Attempts to prepare metallic uranium surfaces for the application of adherent electroplates by the ordinary pickling reagents used in the steel industry have in general proven unsatisfactory. Thus treatment of the metal in dilute sulfuric acid does not improve the surface. Anodic treatment in concentrated sulfuric acid provides a surface to which uniform electroplates may be applied but they are not adherent. Anodic treatment in dilute sulfuric acid etches the surface severely and produces a dark brown coating which is rapidly attacked by atmospheric oxygen and by electroplating baths.

Likewise, the use of alkaline cleaning agents has proven unsatisfactory. Thus, a 5% sodium cyanide solution, although it removes a part of the oxide film, leaves a sufficient film to provide a distinctly gold-colored surface. Neither anodic nor cathodic treatment with this solution further reduces the thickness of the oxide film. Cathodic treatment with dilute solutions of alkalies and sodium cyanide produces results similar to the results obtained with a 5% sodium cyanide cleaner. Anodic treatment with such solutions effects some removal of the film but produces deep pits in the surface of the metal. All of these treatments leave the metal surface in a condition such that it is rapidly oxidized by electroplating baths.

An object of the present invention is to provide metallic uranium surfaces which can be electroplated to produce smooth, uniform, firmly adherent electroplates.

Another object is to remove oxide films from metallic uranium effectively without excessively pitting or otherwise injuring the metal surface for the application of smooth uniform coatings thereon.

Another object is to remove distorted crystal structures from metallic uranium surfaces so that electroplates may be applied directly upon and interlock with the basic crystal structure of the metal.

A further object is to render the metal surface resistant to attack by air during transfer of the metal from the treatment bath to the coating bath and also to render the surface resistant to attack by electroplating solutions.

In accordance with the present invention, metallic uranium is subjected to an etchant treatment in aqueous concentrated hydrochloric acid, and the etched metal is then treated to dissolve off the resulting black oxide and/or chloride film without destroying the etched metal surface.

It is desirable that in each step the treatment be limited to the minimum effective for accomplishing the desired purposes. The treatment with concentrated hydrochloric acid should be sufficiently extensive to form a black coating on the metal surface. The time required may be from 20 to 45 seconds, depending upon the condition of the metal surface and the temperature and concentration of the hydrochloric acid. The oxide or chloride removal

may be effected by means of moderately concentrated nitric acid in 3 to 20 seconds. Preferably a very short nitric acid treatment is used so that the coating on the surface of the metal is removed without sufficiently passivating the metal surface to hinder subsequent electroplating. If a nitric acid treatment long enough to effect substantial passivation is employed, the metal may be given a subsequent very short depassivating treatment with concentrated hydrochloric acid.

While the process is applicable to surfaces which have been worked by machining, extruding or other processes, or highly polished surfaces, it is advantageous to give such surfaces a preliminary treatment to remove foreign matter and assist in removing worked metal or polished metal surfaces. This preliminary treatment may be effected by sand-blasting the surface or by scrubbing it with powdered pumice. Chemical pretreatments also may be used for partially preparing the metal surface. A preliminary treatment with concentrated nitric acid to remove oxides is especially advantageous and contributes substantially to the uniformity of action of the hydrochloric acid etch.

The process of the invention is useful for preparing metallic uranium surfaces for the application of electroplates by means of aqueous sulfate electroplating baths. It is also useful for preparing metallic uranium surfaces for application of coatings by dipping in molten metal baths.

The improvement obtained by the process of the invention is especially noteworthy in the case of electroplates because such coatings are more critically affected by the condition of the metal surface prior to application. Whether the subsequent treatment of the metallic uranium is the application of an electroplate or a hot dip coating, the treatment should follow the preparation of the surface promptly.

Suitable hydrochloric acid solutions for the etchant treatment contain from about 30% to about 40% HCl by weight. The temperature of the hydrochloric acid and the metal may be between 25° and 35° C.

The nitric acid treatment for removing oxide and/or chloride from the metal surface may be carried out with aqueous nitric acid of a concentration between 40% and 55% HNO_3 by weight at temperatures between 25° and 35° C.

The following examples in which quantities are expressed in terms of weight unless otherwise indicated, illustrate the invention.

Example 1

An extruded uranium rod about ⅜ inch in diameter was first dipped for one minute in aqueous 50% HNO_3 solution at 25° C., and then rinsed with water. It was immediately dipped in aqueous 36% HCl solution until the surface was completely black (about 40 seconds). It was then removed from the HCl solution, rinsed with water and dipped for 5 seconds in 50% HNO_3 solution.

The rod, which now had a gray uniformly etched appearance, was again rinsed briefly with water and then immediately electroplated by placing it in an aqueous electroplating bath, containing, per liter, 240 grams of $NiSO_4.7H_2O$, 45 grams of $NiCl_2.6H_2O$, and 30 grams of H_3BO_3 and passing a current through the solution with the rod as cathode at an initial current density of 75 amperes per square foot for one minute and at a current density of 20 amperes per square foot for 3 minutes longer.

A smooth, uniform, firmly adherent nickel electroplate was formed on the surface of the metal.

Example 2

A metallic uranium rod similar to that used in Ex-

ample 1 was cleaned in the same way and electroplated by placing it in a ferrous ammonium sulfate solution containing 300 grams of ferrous ammonium sulfate hexahydrate $FeSO_4(NH_4)_2SO_4.6H_2O$, per liter of solution. The temperature of the solution was 24° C. and the pH was 5.5.

With the uranium rod as cathode, current was applied at a cathodic current density of 20 amperes per square foot for 10 minutes. A firmly adherent iron electroplate 0.0003 inch thick was produced on the rod.

Example 3

A metallic uranium rod was cleaned in the same manner as described in Example 1 and electroplated by placing it in a zinc sulfate bath containing 360 grams of $ZnSO_4$ per liter of solution. The temperature of the solution was 30° C. and its pH was between 1 and 2.

A cathodic current density of 45 amperes per square foot was applied. In this manner a firmly adherent smooth uniform electroplate of zinc was obtained.

Example 4

A metallic uranium rod of the type used in Example 1 was dipped for 20 seconds in 36% HCl solution, removed from the solution, rinsed quickly in water and dipped for 15 seconds in 50% HNO_3 solution, again rinsed quickly in water and dipped in 36% HCl solution until a flash of purple was observed on the metal (about 2 seconds).

After the final hydrochloric acid dip, the rod was rinsed quickly in water and promptly electroplated in a nickel sulfate solution of the same composition as employed in Example 1 at a current density of 25 amperes per square foot for 15 minutes.

An adherent, smooth, uniform nickel electroplate about 0.0003 inch thick was formed on the metal surface.

It will be understood that we intend to include variations and modifications of the invention and that the preceding examples are illustrations only and in no wise to be construed as limitations upon the invention, the scope of which is defined in the appended claims, wherein we claim:

1. The method of preparing metallic uranium for electroplating, which comprises treating the surface of the uranium for about one minute in aqueous 40% to 55% nitric acid solution to remove foreign material and expose a clean metal surface, treating the cleaned surface with aqueous 30% to 40% hydrochloric acid until it is etched black, promptly withdrawing it from the hydrochloric acid and treating it with aqueous 40% to 55% nitric acid for a few seconds to remove the black coloration and provide a gray etched metal surface.

2. In the application of an electroplate to metallic uranium, the improvement which comprises removing foreign matter from the surface of the uranium to expose a clean metal surface, treating the cleaned metal surface with aqueous 30% to 40% hydrochloric acid to etch the metal surface, and treating the etched metal surface with aqueous 40% to 55% nitric acid to dissolve the resulting coating therefrom, and immediately electroplating it in an acid electroplating bath.

3. In the application of an electroplate to metallic uranium, the improvement which comprises treating the surface of the uranium for about one minute in aqueous 40% to 55% nitric acid solution to remove foreign material and expose a clean metal surface, treating the cleaned surface with aqueous 30% to 49% hydrochloric acid until it is etched black, promptly withdrawing it from the hydrochloric acid and treating it with aqueous 40% to 55% nitric acid for a few seconds to remove the black coloration and provide a gray etched metal surface, and immediately electroplating it in an acid electroplating bath.

4. In the application of an electroplate to metallic uranium, the improvement which comprises treating the surface of the uranium with aqueous 30% to 40% hydrochloric acid so as to etch the metal surface, then treating the etched surface with aqueous 40% to 55% nitric acid to dissolve the resulting coating therefrom and passivate the metal, and finally treating the surface with aqueous 30% to 40% hydrochloric acid for a period sufficient to depassivate the metal but insufficient to form a substantial coating thereon, and immediately electroplating it in an acid electroplating bath.

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