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**METHOD FOR ELECTRO-NICKEL PLATING
WOLFRAM CARBIDE**

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No Drawing. Application May 10, 1951
Serial No. 225,680

2 Claims. (Cl. 204—32)

This invention relates to electroplating methods and more particularly, to methods for treating refractory carbides, such as wolfram carbide preparatory to electroplating a metal coating thereon. Specifically, this invention is directed to a method for the electrochemical etching of wolfram carbide articles on which a strongly adherent metal coating can be deposited suitable for attaching another metal body thereto.

In certain nuclear apparatus, such as neutron multiplying devices, experimental piles, reactors and the like, a need has long been felt for a method of rigidly attaching some fissionable materials in close proximity to certain dense refractory neutron reflector materials, such as wolfram carbide or cemented wolfram carbide materials. Prior methods for attaching uranium and other fissionable materials to the refractory metal carbides involved brazing and have been largely unsuccessful, because (1) the brazing materials utilized have reacted undesirably with the fissionable materials at the temperature of brazing, and (2) because those brazing materials do not adhere to the refractory metal carbides, particularly, wolfram carbide. While the first-mentioned difficulty can be surmounted by the use of pure nickel brazing materials the presence of which are not detrimental from a nuclear standpoint, the problem of coating or plating the wolfram carbide piece is more complicated. No simple efficient procedure was known prior to the time of this invention for obtaining a deep, uniform etch on wolfram carbide compositions to which there could be placed a firm, adherent coat of a metal suitable for the subsequent non-reactive attaching of the uranium. It was believed that if a firm adherent metal coating could be placed both on the wolfram carbide and on the fissionable material, the materials could be readily and effectively welded into an integral unit.

It is therefore a primary object of this invention to provide a method for obtaining a deep, uniform etch on articles of wolfram carbide.

It is another object of this invention to provide an electrochemical method for treating wolfram carbide articles to accept a thin, adherent coating of nickel.

It is a further object of this invention to provide a simple, efficient method for etching wolfram carbide articles whereby said articles may be electroplated with a firm, adherent nickel coating.

It is a still further object of this invention to provide a method for treating wolfram carbide articles by electrochemical methods to place thereon a firm, adherent nickel coating which coating is mutually adherent to uranium.

Further objects and advantages will become apparent to one skilled in the art from the description and example which follow.

The objects of this invention are achieved by a process which comprises making a wolfram carbide body anodic in an aqueous solution of sodium pyrophosphate.

During the course of the experimentation with wolfram carbide materials which preceded applicant's dis-

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covery it was noted that electrochemical acid etching solutions were very unsatisfactory. For all practical purposes these solutions produce no discernible etch, or at best, an extremely light etch, entirely unsuited for the purpose of obtaining a firm, adherent nickel coating. Other data indicated that a better etch on the wolfram carbide materials could be obtained with electrolytes comprising alkaline media. While sodium hydroxide and potassium hydroxide solutions up to 25 percent concentration gave improved results, no deep etch was obtained suitable for the firm, adherent plating of nickel necessary for the attachment of uranium thereon.

Similarly, phosphate baths, such as tri-sodium phosphate and orthophosphoric acid baths were tried without success. In every case an unsatisfactory etch was obtained on the wolfram carbide article to which electroplated metals, particularly nickel, would not adhere.

A method for obtaining a deep and satisfactory etch was discovered using an etching solution of sodium pyrophosphate, which method is illustrated in the following example.

Example I

A wolfram carbide disk 3 inches in diameter and 1/4 inch thick is degreased in trichloroethylene, dipped in a 1:1 nitric acid solution for 30 seconds and dried. The disk is then made anodic in an aqueous solution containing 200 grams per liter of sodium pyrophosphate ($\text{Na}_4\text{P}_2\text{O}_7$) using a lead rod inserted in the solution as the cathode. A current of about 20 amperes, corresponding to a current density of about 2.0 amperes per square inch, is passed through the solution for 10 minutes. The temperature of the solution is maintained at about 60° C. The current is turned off and the disk withdrawn from the solution, washed in water and air dried. A clean, smooth, uniformly etched surface is found to have formed on the disk. When the etched article is electroplated in a nickel chloride solution by one skilled in the art a uniformly adherent deposit of nickel from 0.0001 to 0.0005 inch thick may be deposited thereon.

The coating as applied in the above example gives excellent bonding for any welding operation and does not chip, flake, peel or otherwise become non-adherent under the conditions of welding and heat treatment.

Other embodiments of the invention are possible. For example, the salt content of the electrolyte may vary from 100 to 250 grams per liter, the current density may vary from 1 to 5 amperes per square inch, the temperature of the solution may vary from room temperature, that is, about 20° C., to as high as 60° C., while the etching time may vary from 10 to 30 minutes, depending on the other variables. Also, the pretreatment of the piece, that is, the degreasing and cleansing steps, may be varied to apply to the condition of the material undergoing treatment. Solvents other than trichloroethylene may be used for degreasing; and the dilute nitric acid dip may comprise a solution having a composition of from 10 to 40 percent nitric acid.

The step of making the wolfram carbide body anodic in an aqueous solution composed of sodium pyrophosphate is a forward step in the improvement for obtaining an electroplate directly on the wolfram article and is particularly useful in a process for welding or brazing another metal to the wolfram carbide article. As stated hereinbefore, electroplating techniques used in the past have been unable to provide a satisfactory method for attaching uranium to said wolfram carbide bodies. The successful application of a nickel plate to wolfram carbide articles through the use of the sodium pyrophosphate etching method, has made possible a sim-

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ple, efficient process for attaching uranium to wolfram carbide articles without the deleterious effects associated with previous welding or brazing methods, because of the more favorable nuclear properties of nickel when used in nuclear reactors. By the method of the present invention the nickel plating is attached so firmly to both the uranium and the carbide that the attached strips of uranium metal are themselves ruptured before separating from the nickel plate.

While many advantages of the present invention will become obvious to those skilled in the art, it is to be understood that the invention is not to be limited except as indicated in the appended claims.

What is claimed is:

1. The method of electro-nickel plating articles comprising wolfram carbide which comprises making the article anodic at a current density from 1 to 5 amperes per square inch in an aqueous solution containing from 100 to 250 grams per liter of sodium pyrophosphate for a time of from 10 to 30 minutes within a temperature range of from 20° to 60° C., and then electrolytically depositing a nickel coating on the resultant etched surface from an aqueous nickel salt solution.

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2. The method of electro-nickel plating articles comprising wolfram carbide which comprises making the article anodic at a current density of about 2 amperes per square inch in an aqueous solution containing about 200 grams per liter of sodium pyrophosphate at a temperature of from 20° to 60° C. for a time of about 20 minutes and then electrolytically depositing a nickel coating on the resultant etched surface from an aqueous nickel salt solution.

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