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METHOD OF ETCHING TANTALUM AND NIOBIUM FOR ELECTROPLATING

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The invention described herein was made in the course of, or under, a contract with the United States Atomic Energy Commission.

The invention relates to a method of etching tantalum and niobium metal and, in particular, it relates to a method for forming an adherent electrodeposit of a metal such as nickel or copper on a tantalum or niobium base metal.

The present invention is based on the discovery that tantalum and niobium metals can be successfully etched electrolytically in a bath composed of a solution of absolute methyl alcohol and a small but approximately equal amount of concentrated hydrofluoric (48 weight percent hydrogen fluoride in aqueous solution) and hydrochloric (38 weight percent hydrogen chloride in aqueous solution) acids. Using this etching solution it is possible to obtain a uniform pattern of small, deep pits in both niobium and tantalum with near vertical and in some cases undercut sides in the pits. The desired metal is then electrodeposited on the etched surface with the deposit entering these pits which provide a multitude of tiny "anchors" to secure the said deposited metal with a high degree of adhesion or bonding to the niobium or tantalum base metal.

This method has been useful in obtaining a strongly adherent copper nickel metallic film on a tantalum tube heating element and on a tantalum cyclotron target assembly. In the development of the plasma thermocouple, this etching process is presently being used to plate a metallic film of nickel on the niobium metal base so as to prevent the formation of niobium oxide.

It is therefore an object of this invention to provide a method of etching tantalum and niobium base metal surface so that any desired metal can be electrodeposited on said surface to form an adherent metallic film.

Further objects of this invention will become apparent from the following description of the preferred embodiment of this invention.

The tantalum or niobium metal surface is scrubbed vigorously with any suitable fine abrasive paste such as pumice or powdered magnesium oxide. This surface is then dried and made anodic at 0.125 amp per square inch for 40 minutes in an absolute methyl alcohol solution containing 2.5 volume percent concentrated hydrofluoric acid and 2.5 volume percent concentrated hydrochloric acid at a temperature of 35° C. This treatment produces a uniform pattern of small, deep pits on the surface of the tantalum and/or niobium metal surface. The etched surface is then electroplated with the desired metal, and the deposit which enters these pits forms "anchors" which provide a high degree of mechanical bonding of the film to the said base metal.

The following are listed as specific examples of the invention:

Example I

The tantalum metal surface is cleaned using any suitable abrasive. The cleaned metal is placed in an absolute

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methyl alcohol bath containing 5 volume percent concentrated hydrofluoric acid and 5 volume percent concentrated hydrochloric acid. The tantalum is made anodic at a current density of 0.05 amp per square inch and the bath is maintained at a temperature of about 15° C. for a period of about 60 minutes.

Example II

The niobium metal surface is cleaned and placed in a methyl alcohol bath containing 1 volume percent concentrated hydrofluoric and 1 volume percent concentrated hydrochloric acid. The niobium is made anodic at a current density of 0.50 amp per square inch and the bath is maintained at a temperature of about 65° C. for about 10 minutes.

It is understood that in all of the above examples that either niobium or tantalum can be substituted as the base metal.

Critical parameters in this etching process are the following: time, temperature, current density, and the concentration of acid in the methyl alcohol solution. The time period necessary to obtain a satisfactory etched surface can vary between 10 and 60 minutes dependent on the temperature, current density, and acid concentration. The higher the temperature, current density, and acid concentration, the shorter the time will be.

The temperature of etching solution must be maintained at not less than 15° C. and not more than 65° C. Current density of the tantalum or niobium base metal must be regulated between 0.05 and 0.5 amp per square inch. At higher temperatures or higher current density the tantalum or niobium surface is too severely etched leaving an uneven, partially polished surface not productive of adhesion. At lower current densities or temperatures (below the aforementioned limits) uniformity of attack on tantalum or niobium metal to produce an etched surface becomes a serious problem. The acid concentration of the hydrofluoric and hydrochloric acids must be adjusted so that the acids are present in nearly equal amounts and their concentration cannot individually exceed 10 volume percent and each must be at least 1.0 volume percent in order to obtain a uniform etch of the tantalum or niobium surface. The use of either hydrofluoric or hydrochloric acids alone in the methyl alcohol bath tend to produce an uneven attack.

While the present invention has been described with reference to a particular embodiment thereof, it will be understood that numerous modifications may be made by those skilled in the art by adjusting the aforementioned parameters within the ranges started without departing from the scope of the invention. Therefore, the appended claims are intended to cover all such equivalent variations as come within the true spirit and scope of the foregoing disclosure.

What is claimed is:

1. A method for electrodepositing an adherent metallic film on a metal selected from the class consisting of tantalum and niobium comprising the steps of making the said metal anodic in a methyl alcohol bath consisting essentially of approximately equal amounts of concentrated hydrofluoric and concentrated hydrochloric acids, the concentration of each of said acids ranging between 1.0–10.0 volume percent, the temperature of said bath ranging between 15–65° C., and the current density of the said metal ranging between 0.05–0.50 amp per square inch and electrodepositing any desired metal as a film on the etched surface of the said metal.

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2. The method of claim 1 in which said metal is tantalum and the methyl alcohol solution contains 2.5 volume percent concentrated hydrofluoric acid and 2.5 volume percent concentrated hydrochloric acid, the current density is 0.125 amp per square inch, making said metal anodic for a period of about 40 minutes and maintaining said bath at a temperature of about 35° C.

3. The method of claim 1 in which the surface of the tantalum and niobium metal is cleaned with an abrasive selected from the class consisting of pumice and powdered magnesium oxide prior to placing said metal in the methyl alcohol bath.

4. The method of claim 1 in which said metal is niobium and the methyl alcohol solution contains 2.5 volume percent hydrofluoric acid and 2.5 volume percent hydrochloric acid, the current density is 0.125 amp per square

inch, the temperature is 35° C., and said metal is made anodic for a period of about 40 minutes.

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