Patented Sept. 6, 1949

2,481,306

UNITED STATES PATENT OFFICE

2,481,306

ELECTROCHEMICAL POLISHING OF TANTALUM

John F. Gall, Narberth, and Henry C. Miller, Wyndmoor, Pa., assignors to The Pennsylvania Salt Manufacturing Company, Philadelphia, Pa., a corporation of Pennsylvania

No Drawing. Original application March 15, 1944, Serial No. 526,656. Divided and this application September 17, 1948, Serial No. 49,853

2 Claims. (Cl. 204—140)

1

This invention relates to electropolishing of metals and electrolytes employed in that operation. More particularly the invention comprises a method of electropolishing tantalum and an electrolyte comprising hydrofluoric acid and a strong acid such as hydrochloric or sulfuric acid.

It is known that tantalum metal exhibits "valve action." That is, when a current is passed through the metal, as an electrode submerged in an electrolyte, and in such a direction as to make the metal anodic, a resistance is created causing the flow of current to cease. When this occurs, even the slight degree of anodic solution necessary for electropolishing cannot take place, and it is this phenomenon which has heretofore prevented the electropolishing of tantalum.

An object of this invention is to provide a method of polishing tantalum metal wherein the "valve action" of the metal is overcome.

Another object of the invention is to provide an electrolyte enabling the contemplated method to be carried out.

Certain other objects and advantages are apparent from a consideration of this specification and claims.

According to the invention, the tantalum metal to be polished is made anodic in an aqueous solution, containing hydrofluoric acid together with hydrochloric acid and/or sulfuric acid of a certain, predetermined, critical composition and concentration, thereby obtaining, upon passage of current at a certain critical anodic current density, a dissolution of the surface irregularities of the metal giving it a fine high polish.

The composition and concentration of the electrolytic bath employed will preferably be:

	Percent
Hydrofluoric acid	2–7
Hydrochloric acid	32–38

the total combined acid concentration being between 34% and 45%, the balance being substantially water, in the case hydrochloric acid is employed; and

	Percent
Hydrofluoric acid	2–7
Sulfuric acid	75–98

the total combined acid concentration being between 77% and 100%, the balance being substantially water, when sulfuric acid is employed. The percentage figures are by weight.

A current density of from 40 to 160 milliamperes per square centimeter has been found to avoid "valve action," employing an electrolytic

2

bath having a composition and concentration as above stated.

A substantial departure from the conditions of the invention will result in unsatisfactory polishing. The metal may dissolve too rapidly with excessive loss of material and production of an etched surface or it may "valve," that is, the passage of current may cease and the surface of the metal remain unchanged or the metal may behave erratically and a periodic pulsation of current, accompanied by the formation of flaky material dropping from the surface, may result.

As specific examples of the invention yielding good results, the following are cited:

Example 1

Tantalum is made anodic at a current density of 67 milliamperes per square centimeter in an aqueous solution of hydrofluoric and hydrochloric acids, which is 4%, by weight, hydrogen fluoride and 34%, by weight, hydrogen chloride. The operation is conducted at room temperature. In about 10 minutes the surface has developed a high mirror-like finish.

Example 2

Tantalum is made anodic with a current density of 40 milliamperes per square centimeter in an aqueous solution of hydrofluoric and sulfuric acids, which is 4%, by weight, hydrogen fluoride and 89%, by weight, sulfuric acid. The operation is conducted at room temperature. In this case polishing will result after about 20 minutes of electrolysis.

The cited examples are in no way restrictive of the invention, its essence being the discovery that tantalum metal can be electropolished employing the electrolytes, set forth, at the indicated ranges of concentrations, and the current densities, also as indicated.

This application is a division of our co-pending application Serial No. 526,656 of the same title as this application filed March 15, 1944 (now Patent Number 2,466,095).

We claim:

1. The electropolishing of metallic tantalum by making it anodic in an electrolyte, containing 2%–7% by weight of hydrogen fluoride and 32%–38% by weight of hydrochloric acid, the combined acid concentration being between 34% and 45%, the balance being substantially water, and passing current at an anodic current density of from 40 to 160 milliamperes per square centimeter until the metal surface has developed a high, mirror-like polish.

2. The electropolishing of metallic tantalum by making it anodic at a current density of 67 milliamperes per square centimeter in an aqueous solution of hydrofluoric and hydrochloric acids containing 4% by weight of hydrogen fluoride and 34% by weight of hydrogen chloride, the balance being substantially water, until the metal surface has developed a high, mirror-like polish.

JOHN F. GALL.
HENRY C. MILLER.

REFERENCES CITED

The following references are of record in the file of this patent:

UNITED STATES PATENTS

Number	Name	Date
2,115,005	Blaut et al.	Apr. 26, 1938

FOREIGN PATENTS

Number	Country	Date
530,041	Great Britain	Dec. 4, 1940

OTHER REFERENCES

"Comprehensive Treatise On Inorganic and Theoretical Chemistry," by Mellor, vol. 9 (1929), page 891.