STABILIZED GRAIN-SIZE TANTALUM ALLOY

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2 Claims

ABSTRACT OF THE DISCLOSURE

A method of producing a cold worked annealed tantalum alloy having a fine equiaxed grain structure and the product so produced. The method consists of cold working the tantalum alloy, heating the alloy to a temperature above its recrystallization, cold working to the desired shape and finally annealing the alloy at a temperature sufficient to provide an equiaxed grain structure.

The present invention is directed to tantalum alloys and more particularly to stabilized grain-size tantalum alloys having improved drawing characteristics.

In a copending application, Ser. No. 408,684, filed Nov. 3, 1964 by Maurice L. Torti, Jr., now U.S. Patent No. 3,268,326, and assigned to the assignee of the present invention, there is described a new stabilized grain tantalum alloy. The present invention is directed to improvements of such alloys, particularly with respect to an alloy which has a stabilized grain size at elevated temperatures as well as having excellent deep drawing characteristics.

Accordingly, it is a principal object of the present invention to provide an improved alloy containing grain-size-stabilizing amounts of yttrium and which has other physical properties such as strength, elongation and the like substantially equivalent to pure annealed tantalum.

These objects are accomplished in the present invention by providing for the addition of grain-stabilizing amounts of yttrium, as taught in the above-mentioned copending application, with the provision of intermediate heat treatments which, for some reason which is not completely understood, provide a final product having some of the attributes of the yttrium-containing alloys and some of the properties of pure tantalum.

The invention accordingly comprises the process involving the several steps in relation to the order of one or more of such steps with respect to each of the others, and the product resulting therefrom, which are exemplified in the following detailed disclosure and the scope of the application of which will be indicated in the claims.

For a fuller understanding of the nature and objects of the invention, reference should be had to the following detailed description.

In a preferred embodiment of the invention, the tantalum alloy is prepared by arc melting tantalum with the addition of a small amount of yttrium (on the order of .2 percent), the melting being accomplished so that a tantalum ingot is obtained containing about 10 to 1000 p.p.m. yttrium and preferably about 30 to 1000 p.p.m. yttrium. This ingot is then reduced to plate stock about 1/4 inch thick and then subjected to an anneal of at least 3000°F. and preferably 3400°F. to 3600°F. to achieve substantially complete recrystallization of the tantalum. Thereafter, the tantalum alloy is rolled to a sheet, preferably with an intermediate anneal at about .050 inch thick. The sheet is then preferably rolled to .020 and given a final anneal at 2200°F. The final anneal is generally conducted at a temperature of about 2000°F. to 2400°F. All of the annealing times may be on the order of one hour.

The product of the treatment outlined above is found to have rather unusual characteristics. It has the fine grain structure and resistance to grain growth characteristic of tantalum alloys containing yttrium in excess of 10 p.p.m. as described in the above-mentioned copending application of Torti. However, for some reason which is not completely understood, the recrystallization temperature of the final sheet is much lower than would normally be expected, this recrystallization temperature being somewhat less than 3000°F. and approaching that of pure tantalum. Additionally, the product of the present invention is very ductile, has an equiaxed grain structure, and does show directional grain structures which give "earing" during drawing operations.

In order that the invention may be more fully understood, reference should be had to the following nonlimiting examples:

Example 1

A tantalum ingot containing 60 p.p.m. yttrium of 41/2 inches in diameter was forged to 3/4 inches and rolled to 1/4 inch plate stock, 24 inches by 30 inches. This plate stock was then annealed in a vacuum furnace to a pressure of 10⁻⁴ torr for 1 hour at 3600°F. Thereafter the plate stock was rolled on a 2 high mill to sheet .030" thick. This sheet was then annealed for 1 hour in a vacuum furnace at 10⁻⁴ torr at a temperature of 2200°F. The material was then tested and found to have ultimate tensile strength of about 46,000 p.s.i. which compares to 48,000 p.s.i. for pure tantalum. The .2% yield strength was 32,000 p.s.i. This compared to about 35,000 p.s.i. for pure tantalum. Percent elongation was about 30% as compared to about 35% for pure tantalum. This then subjected to a standard physical test and the results are shown in Table I below, where the product of Example 1 is compared to pure tantalum.

<table>
<thead>
<tr>
<th>Product of</th>
<th>Pure Tantalum</th>
</tr>
</thead>
<tbody>
<tr>
<td>Example 1</td>
<td></td>
</tr>
<tr>
<td>Room temperature, annealed (° C.)</td>
<td>1,200</td>
</tr>
<tr>
<td>Ultimate (p.s.i.)</td>
<td>45,000</td>
</tr>
<tr>
<td>2% yield (p.s.i.)</td>
<td>32,000</td>
</tr>
<tr>
<td>Percent elongation</td>
<td>30</td>
</tr>
<tr>
<td>Elevated temperature ultimate strengths (p.s.i.):</td>
<td></td>
</tr>
<tr>
<td>1,000°F</td>
<td>4,600</td>
</tr>
<tr>
<td>2,000°F</td>
<td>2,500</td>
</tr>
<tr>
<td>ASTM number after annealing at 3,700°F</td>
<td>6.0</td>
</tr>
<tr>
<td>.005&quot; thick</td>
<td>.350</td>
</tr>
<tr>
<td>.002&quot; thick</td>
<td>.200</td>
</tr>
</tbody>
</table>

* For the Olsen Cup values the annealed .005" thick stock was rolled to .005" and .002" and reannealed at 2,200°F. for 1 hour. All the other tests were on the .003" stock.

Example 2

In this case, the initial annealing temperature at the 1/4" plate was 3600°F. for one hour. The sheet was annealed at .050" for 1 hour at 2200°F. and the final anneal at .020" was 2200°F. for 1 hour. The properties of this product were essentially the same as the product obtained in Example 1.

Example 3

In this example, the initial anneal at the 1/4" plate size was 3 hours at only 2200°F., while the other treatments were the same as in Example 1. In this case the final product had much higher strength and lower elongation with a recrystallization temperature of about 4000°F. When formed into a cup, it had very poor characteristics with extreme "earing." While several preferred embodiments of the invention have been described above, numerous modifications thereof may be made without departing from the spirit of the invention. The yttrium concentrations and annealing times and temperatures may be varied from the specific examples given as defined in the specification.
Since certain changes can be made in the above process and product without departing from the scope of the invention herein involved, it is intended that all matter contained in the above description shall be interpreted as illustrative and not in a limiting sense.

What is claimed is:

1. The process of producing a cold worked annealed tantalum alloy having a fine grain structure and being resistant to grain coarsening upon annealing, said alloy having an ASTM number greater than 3 upon heating to 3700°F. for one hour, said alloy having yield strength and percent elongation properties equivalent to pure annealed tantalum and showing Olsen cup ductility properties essentially equivalent to pure annealed tantalum, said alloy having an ultimate strength at elevated temperatures essentially in excess of pure tantalum, said alloy consisting of between about 10 and 1000 p.p.m. yttrium the balance being tantalum, said process comprising the steps of:
   (a) partially cold working said alloy to reduce its thickness;
   (b) annealing said alloy at a temperature in excess of 3000°F. prior to final cold working of said alloy to achieve complete recrystallization of said alloy;
   (c) finally cold working said alloy to final dimension; and then
   (d) annealing said final cold worked alloy to a temperature of about 2000°F. to 2400°F. to provide an equiaxed grain structure.

2. A cold worked annealed tantalum alloy having a fine grain structure and being resistant to grain coarsening upon annealing, said alloy having an ASTM number greater than 3 upon heating to 3700°F. for one hour, said alloy having yield strength and percent elongation properties equivalent to pure annealed tantalum and showing Olsen cup ductility properties essentially equivalent to pure annealed tantalum, said alloy having an ultimate strength at elevated temperatures substantially in excess of pure tantalum, and further characterized by having a recrystallization temperature less than 3000°F. and having an equiaxed grain structure; and in line 10, said alloy having been produced by the process of claim 1, said alloy consisting of between about 10 and 1000 p.p.m. yttrium the balance being tantalum.

No references cited.

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