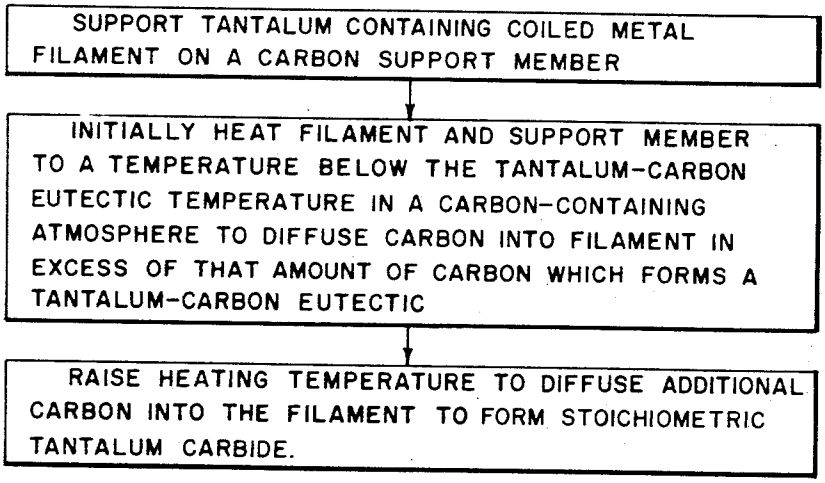
Nov. 19, 1968 R. CORTH 3,411,959

METHOD FOR PRODUCING TANTALUM CARBIDE AND TANTALUM-ALLOY CARBIDE FILAMENTS

Filed March 21, 1966

FIG. 1.



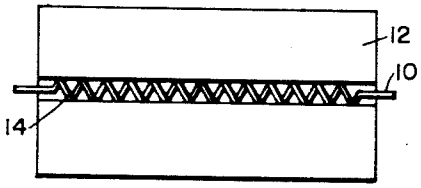


FIG. 2.

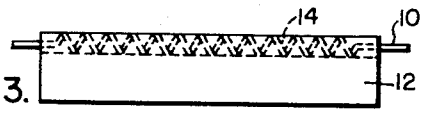


FIG. 3.

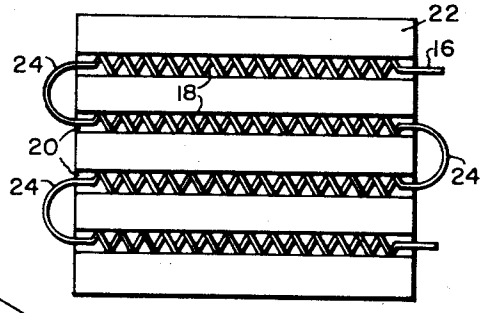


FIG. 4.

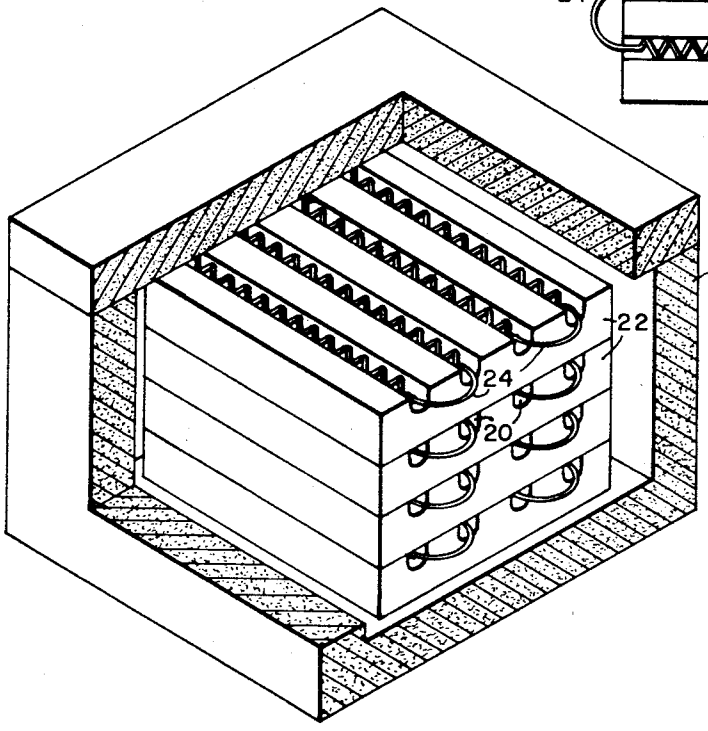


FIG. 5.

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METHOD FOR PRODUCING TANTALUM CARBIDE AND TANTALUM-ALLOY CARBIDE FILAMENTS

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10 Claims. (Cl. 148—13.1)

This invention relates generally to the production of tantalum carbide filaments and, more particularly, to a method for producing improved tantalum carbide and tantalum-alloy carbide filaments which are strong, distortion free and relatively elastic.

Tantalum carbide and tantalum-alloy carbide filaments for incandescent lamps are known in the art. When operated in a gaseous atmosphere comprising halogen, hydrogen and carbon, for example, such filaments can be operated at a very high true temperature, such as from 3300° K. to 3700° K. This operating temperature range approaches and actually exceeds the melting point of tungsten per se (3643° K.) and is far above the normal operating temperature of tungsten filaments. This makes a tantalum carbide or tantalum-alloy carbide filament particularly useful in applications such as projection lamps where it is desirable to operate the lamp in as bright a manner as possible commensurate with even a relatively short life.

One of the major drawbacks which prevents commercial use of tantalum carbide filaments is the difficulty encountered in fabricating the filament. Tantalum carbide and tantalum-alloy carbide filaments are extremely brittle and cannot be formed into a coil or otherwise bent. In addition, the filaments are subject to distortion during operation, which coupled with their usual brittleness results in excessive breakage. The brittleness also results in breakage when mounting the filament as well as breakage due to the usual shocks encountered during shipment of the lamps and handling of same after they are put into use. For these reasons, tantalum carbide or tantalum-alloy carbide filaments have not become any appreciable factor in the field of illumination such as projection lamps, in spite of the tremendous gains which can be achieved in brightness of operation because of the high operating temperatures which can be used. In the practices of the prior art, the filament has been mounted in the completed lamp as a metallic member, and thereafter converted to tantalum carbide. This is very impractical from a commercial standpoint.

It is the general object of this invention to provide a method for producing strong, distortion free, elastic tantalum carbide and tantalum-alloy carbide filaments.

It is another object to produce a method for mass producing tantalum carbide and tantalum-alloy carbide filaments which are strong, distortion free and elastic.

It is a further object to provide a method for completely forming a tantalum-carbide or tantalum-alloy-carbide filament prior to incorporation into a lamp.

The foregoing objects of the invention, and other objects which will become apparent as the description proceeds, are achieved by supporting on a carbon member a filament of tantalum metal or a refractory tantalum-containing alloy. The metal filament has previously been coiled into its final, desired form and preferably is stress relieved after coiling. The supported coiled filament is initially heated on the support member in an atmosphere consisting essentially of carbon as the only reactive constituent at a predetermined temperature below the eutectic melting point of the filament as partially carbided, but which heating temperature is sufficient to cause carbon to readily diffuse into the filament. This initial heating temperature is maintained for a time sufficient to permit carbon to be diffused into the filament in an amount which is in excess of that carbon content required to form a tantalum-carbon eutectic. This increases the filament melting temperature. Thereafter, the support member and supported filament are heated in a similar atmosphere to a final temperature which is greater than the melting temperature of the tantalum-carbon eutectic, but less than the melting temperature of the now partially carbided filament. This final heating temperature is maintained for a sufficient time to diffuse additional carbon into the filament to form stoichiometric tantalum carbide or tantalum-alloy carbide. The completely carbided filament is then cooled under non-reactive conditions.

For a better understanding of the invention, reference should be had to the accompanying drawings, wherein:

FIGURE 1 is a flow diagram illustrating the basic steps of the present method;

FIG. 2 is a plan view of a graphite block such as is used as a filament support member, with a tantalum or tantalum-alloy metallic coiled filament supported therein;

FIG. 3 is an elevational view of FIG. 1;

FIG. 4 is a plan view of an alternative graphite support member wherein plural grooves are provided in a surface thereof to support a plural-coil filament; and

FIG. 5 is an isometric view, partly broken away, showing a plurality of support members in heating position in a carbon crucible, wherein the support members are stacked for heating in accordance with the present method.

With specific reference to the form of the invention as illustrated in the drawings, the basic steps of the present method are shown in the flow diagram of FIG. 1. The filament of the present invention will normally have a coiled configuration since the best applications for high brighteners light sources are those which require the light to be concentrated. There is first formed from either tantalum metal or a refractory tantalum-containing alloy a filament coil **10** such as shown in FIGS. 2 and 3. As a specific example, the coil **10** is preferably formed of an alloy of 90% tantalum and 10% tungsten by weight. The coil **10** could be formed of tantalum metal or other refractory tantalum-containing alloys which contain a major percentage of tantalum. In this regard, see the alloys referred to in U.S. Pattent 3,219,493, dated Nov. 23, 1965.

After coil formation, which is conventional, the coil **10** is placed into a carbon supporting member **12** which preferably is formed of graphite. In the preferred form of the invention, the filament is positioned within a groove **14** which is provided in the top surface of the graphite supporting member. Preferably, the supported filament **10** and graphite support member **12** are placed into a carbon crucible and the filament heated under non-reactive conditions to a relatively low temperature in order to relieve the winding stresses which are introduced into the filament during coiling. The stress-relieving temperature may be varied considerably depending upon the wire dimensions, but for a 10-mil diameter wire, which wire size will be considered in detail hereinafter, the filament is stress relieved by heating to a temperature of about 1500° C. for about 15 minutes in either a vacuum or an inert gas atmosphere, such as argon.

Immediately after stress relief, the supported filament **10** and the supporting memer **12** are heated in a carbon crucible in an atmosphere consisting essentially of carbon as the only reactive constitutent at a predetermined temperature which is below the eutectic melting temperature of tantalum-carbon (2800° C.), but which is sufficient to cause carbon to diffuse into the filament in total amount less than that required to form stoichiometric tantalum carbide. Sufficient carbon is diffused into the filament during the initial heating, however, to exceed that carbon

content required to form a tantalum-carbon eutectic and also to raise the filament melting temperature to substantially more than the eutectic melting temperature. As a specific example, the 10-mil wire is heated in an argon atmosphere to a temperature of from 2600° C. to 2650° C. for a period of about 30 minutes. It should be understood that these initial heating conditions can be varied considerably depending upon the wire dimensions and the time available to diffuse carbon into the coil, which of course is a commercial consideration.

In explanation of the tantalum-carbon eutectic, when carbon is diffused into tantalum, a small amount of diffused carbon will cause the resulting material to exhibit a eutectic melting point at approximately 2800° C. When a preformed tantalum filament is being carbided, gross distortion will occur if incipient melting or any melting occurs. Apparently the melting creates a structure which makes the resulting tantalum carbide extremely brittle. In accordance with the present method, carbon is initially diffused into the tantalum or tantalum-alloy filament at a temperature which is slightly below this eutectic melting temperature and the initial heating and resulting carbon diffusion into the filament is continued until carbon is present in the filament in sufficient amount that the melting point of the resulting tantalum-carbon filament is substantially more than the eutectic melting temperature. Preferably the initially heated and partially carbided filament has a melting temperature greater than 3,100° C.

In the next step, the support member **12** and supported filament **10** are rapidly heated to a final heating temperature in excess of the melting temperature of the eutectic, but less than the melting temperature of the now partially carbided filament, and this final heating temperature is maintained for a predetermined period of time which is sufficient to cause additional carbon to diffuse into the filament to form a stoichiometric tantalum carbide. Preferably the final heating temperature is at least 3,000° C. As a specific example, a 10-mil partially carbided filament is heated in the final heating step at a temperature of 3100° C. for a period of 30 minutes, which is sufficient to form a stoichiometric tantalum-tungsten carbide. The initial and final heating steps are conducted in the atmosphere consisting essentially of carbon as the only reactive element, preferably with an inert gas surrounding atmosphere, argon being preferred.

As a final processing step, the completely carbided filament is cooled under non-reactive conditions, such as an argon atmosphere or vacuum, to a temperature at which the filament will not oxidize. As a specific example, the foregoing 10 mil filament is cooled under the non-reactive conditions to a temperature less than about 100° C.

In order to form a multi-sectional filament which comprises a plurality of filament coils connected by uncoiled portions, it is highly desirable to carbide the tantalum or tantalum-alloy coil in its final multi-sectional form, since any bending of the completely carbided filament is apt to result in fracture. In FIG. 4, there is shown a four-barrel coiled filament **16** wherein the individual filamentary coils **18** are placed into plural grooves **20** provided in a graphite supporting member **22**. The uncoiled connecting sections **24** of the filament **18** project slightly from the edge portions of the graphite support **22**. The support member **22** is particularly adapted to be nestled or stacked for production runs so that many filaments can be heat treated at the same time. A plurality of such stacked crucibles are shown in FIG. 5. There is also shown in FIG. 5 the carbon crucible **26**, partly broken away, in which the support members **22** are stacked. Preferably, the crucible **26** is placed into a conventional, electrically or induction-heated furnace which has a controlled argon atmosphere and the carbiding steps can be completed in one sequential operation. Any other inert atmosphere or even a vacuum could be maintained in the furnace during the heating steps.

Preferably, the stress relief, initial heating step and final heating step are sequentially performed with no cooling between each step. It should be understood that the specific schedule as outlined hereinbefore can be varied considerably depending on the size of the wire being carbided. While the stress relief step is very desirable, it can be dispensed with if desired.

Filaments which have been carbided in accordance with the present method are extremely strong and distortion free, as well as relatively elastic. Such filaments have been operated in projection lamps for a period of 30 hours at a true temperature of 3700° K. This is in contrast to the usual projection lamp which operates at a true temperature of about 3225° K. The preferred mode for mounting the filament in a projection lamp or other incandescent source is disclosed in copending application Ser. No. 535,835, filed concurrently herewith, entitled "Tantalum Carbide or Tantalum-Alloy Carbide Filament Mounting and Method" by the present inventor and owned by the present assignee.

It will be recognized that the objects of the invention have been achieved by producing a tantalum carbide or tantalum-alloy carbide filament which is strong, distortion free and relatively elastic. There has also been provided a method whereby such filaments can be produced on a production basis. The filament is completely carbided prior to incorporation into a lamp.

Although the preferred application for the present filaments is in conjunction with projection lamps, it should be understood that the present filaments can be used in conjunction with any type of high-intensity light source, such as photoflood lamps or sealed-beam headlamps.

While preferred embodiments of the present invention have been illustrated and described in detail, it is to be particularly understood that the invention is not limited thereto or thereby.

I claim as my invention:

1. The method of forming a filamentary material which at least principally comprises tantalum carbide, which method comprises:

(a) supporting on a carbon member a filament of tantalum metal or a refractory alloy principally comprising tantalum metal;

(b) initially heating said support member and supported filament in an atmosphere consisting essentially of carbon as the only reactive constitutent at a predetermined temperature below the eutectic melting temperature of said filament as partially carbided, but sufficient to cause carbon to readily diffuse into said filament;

(c) maintaining the initial heating for a predetermined period of time to cause carbon to diffuse into said filament in amount less than that required to form stoichiometric tantalum carbide, but in amount sufficent to raise the melting temperature of said partially carbided filament to sustantially more than said eutectic melting temperature;

(d) heating said support member and supported filament in said atmosphere consisting essentially of carbon to a final heating temperature greater than said eutectic melting temperature, but less than the melting temperature of said initially heated filament;

(e) maintaining the final heating temperature for a predetermined period of time to cause additional carbon to diffuse into said filament to form stoichiometric tantalum carbide; and

(f) cooling said filament under non-reactive conditions to a temperature below which said filament will not oxidize.

2. The method as specified in claim **1**, wherein said atmosphere under which said filament is heated includes an inert gas such as argon.

3. The method as specified in claim **1**, wherein said filament before initial heating is formed of an alloy of 90% by weight tantalum and 10% by weight tungsten,

said initial heating temperature is less than 2800° C., and said final heating temperature is more than 3000° C.

4. The method as specified in claim 1, wherein said filament before initial heating is formed into its desired final configuration which is that of a coil.

5. The method as specified in claim 1, wherein said carbon support member is a graphite block having an open groove therein to receive said filament, and said filament is heated in a carbon crucible.

6. The method as specified in claim 1, wherein said initial heating is conducted at a temperature of from 2600° C. to 2650° C. for a period of about 30 minutes, and said final heating is conducted at a temperature of about 3100° C. for a period of about 30 minutes.

7. The method as specified in claim 1, wherein said filament is formed prior to initial heating as a plurality of coils each connected by an uncoiled filamentary member, and said carbon support member is a graphite block having plural open grooves provided in a surface thereof to receive said plural coil of said filament.

8. The method as specified in claim 7, wherein a plurality of said filaments are simultaneously heated, and prior to such simultaneous heating a plurality of said filament-supporting graphite members are nestled on top of one another with the bottom of one of said members forming the cover portion for the said member immediately therebelow.

9. The method of forming a tantalum carbide-tungsten carbide filament wherein the ratio by weight of tantalum to tungsten is about 90:10, which method comprises:

(a) supporting on a graphite member a filament of tantalum-tungsten alloy wherein the ratio by weight of tantalum to tungsten is about 90:10;

(b) initially heating said support member and said supported filament in carbon crucible containing an inert gas at a temperature of from about 2600° C. to 2650° C. for a period of about 30 minutes;

(c) rapidly raising the heating temperature to about 3100° C. and maintaining this temperature for about 30 minutes; and

(d) cooling said filament in the inert gas atmosphere to a temperature of at least about 100° C.

10. The method as specified in claim 1, wherein said filament prior to said initial heating is formed into its desired final configuration which is that of a coil, said coil is placed into an open groove provided in a surface of a graphite member, and said graphite member and said coil are then heated under non-reactive conditions to a temperature of about 1500° C. for about fifteen minutes to stress relieve said filament coil.

References Cited

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