United States Patent

Corth et al.

[15] **3,650,850**

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[54] METHOD OF MAKING AN UNDISTORTED COILED-COIL TANTALUM CARBIDE FILAMENT

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[52] **U.S. Cl.** .....**148/13.1,** 23/208 A, 29/25.14, 29/25.17, 29/25.18, 148/20.3, 313/218

[51] **Int. Cl.** **C23c 9/00,** C23c 11/10, C22c 29/00

[58] **Field of Search** .....148/13, 13.1, 19, 20.3; 29/25.14, 25.17, 25.18; 313/218; 23/208 A

[56] **References Cited**

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3,411,959	11/1968	Corth	148/20.3 X
3,523,044	8/1970	Johansen	148/20.3 X

Primary Examiner—Charles N. Lovell

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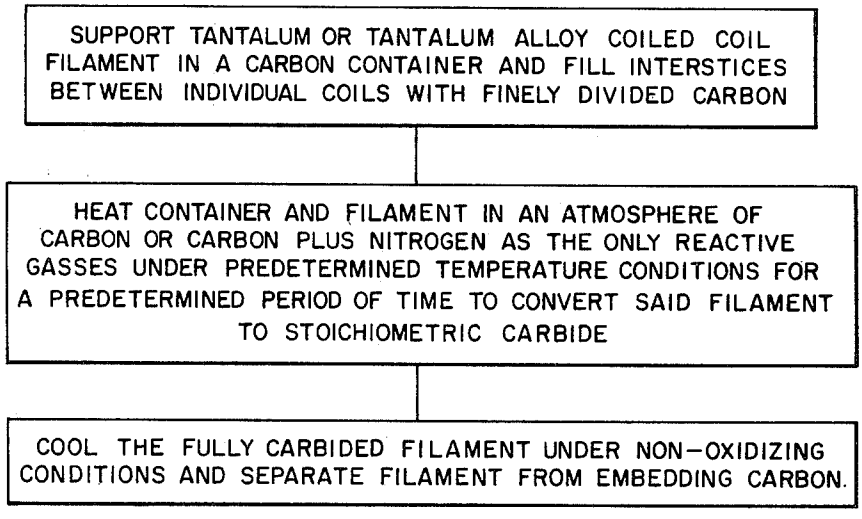
[57] **ABSTRACT**

To carbide coiled-coil tantalum or tantalum alloy filament without causing it to distort, the coiled-coil metal filament is supported in a firing container consisting essentially of carbon as the only container component which will react with tantalum. The interstices between individual coils of the coiled-coil filament are filled with finely divided carbon, preferably graphite. The container and filament are then heated in an atmosphere of carbon or carbon plus nitrogen as the only reactive gases under predetermined temperature conditions and for a predetermined period of time sufficient to convert the filament to stoichiometric carbide.

8 Claims, 4 Drawing Figures



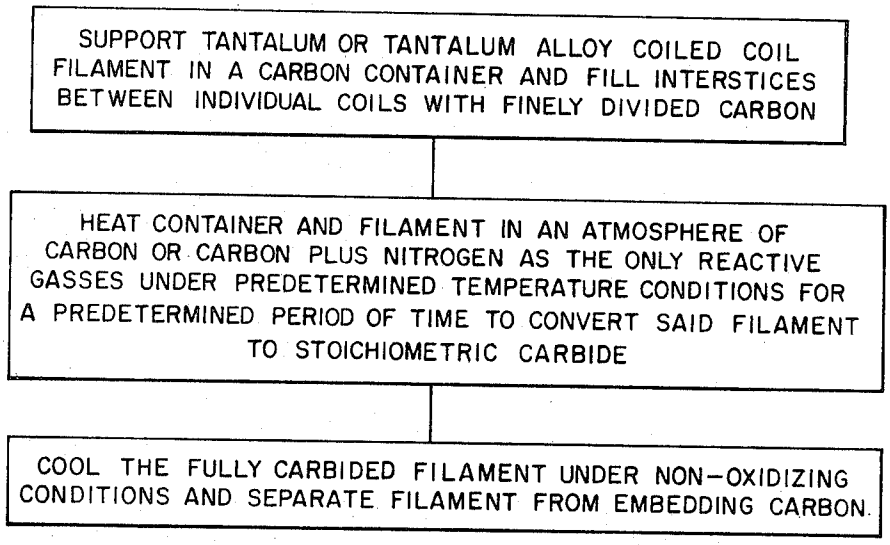


FIG. 1.

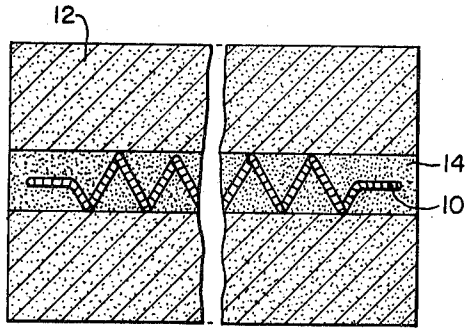


FIG. 2.

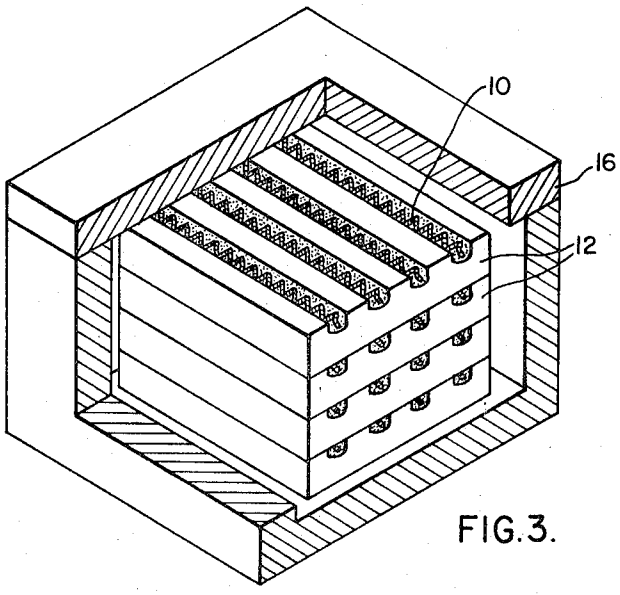


FIG. 3.

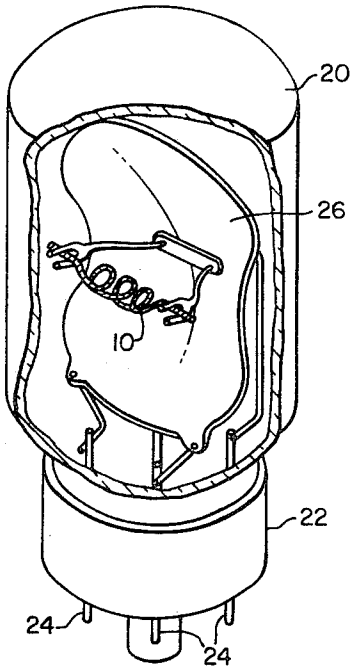


FIG. 4.

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METHOD OF MAKING AN UNDISTORTED COILED-COIL TANTALUM CARBIDE FILAMENT

BACKGROUND OF THE INVENTION

This invention relates to tantalum carbide filaments such as can be used in incandescent lamps and, more particularly, to a method for carbiding a coiled-coil tantalum carbide filament without causing the filament to distort.

In recent years considerable effort has been expended upon the development of tantalum carbide as a filmentary material because of its favorable spectral emission properties and its high melting point (about 4,200° K.) which is the highest known for any substance. Such filaments have been investigated in considerable detail for possible use in projection lamps.

In U.S. Pat. No. 3,411,959 dated Nov. 19, 1968 to R. Corth, one of the inventors herein, is disclosed a method for producing tantalum carbide and tantalum-alloy carbide filaments wherein the filament is heated in an atmosphere consisting essentially of carbon as the only reactive constituent at a temperature below the eutectic melting temperature of the filament as partially carbided. This initial heating is continued for a sufficient time to cause carbon to diffuse into the filament in an amount sufficient to raise the melting temperature to substantially more than the tantalum-carbon eutectic melting temperature. Thereafter the heating temperature is raised to greater than the eutectic melting temperature in order to complete the carbiding of the filament.

In copending application Ser. No. 698,962, filed Jan. 18, 1968 by Herman A. Johansen, now U.S. Pat. No. 3,523,044 dated Aug. 4, 1970, and owned by the present assignee, is disclosed a method for carbiding a tantalum or tantalum alloy filament wherein the carbiding atmosphere is nitrogen and carbon. It has been found that the presence of the nitrogen accelerates the carbiding process and it can be carried out at a lower temperature, such as 2,100° to 2,300° C.

Both of these processes can be used to make excellent single coil filaments. If a very compact light source is desired, however, it is desirable to fabricate the filament as a coiled-coil member, wherein a coil is wrapped into a coil to form the coiled-coil. By way of further explanation, when the tantalum is carbided there results a volume increase of approximately ten-percent. Apparently this volume increase results in distortions of the carbided filament and it is theorized that the distortions are due to a nonuniform rate of carbiding which causes the filament to act in the manner of a bimetal strip.

SUMMARY OF THE INVENTION

In accordance with the present invention, a coiled-coil filamentary member formed of tantalum or an alloy principally comprising tantalum is carbided without any distortion by a method which comprises first supporting the coiled-coil member in a firing container consisting essentially of carbon as the only container component which will react with tantalum. Thereafter, the interstices between the individual coils of the filamentary member are filled with finely divided carbon, preferably high purity graphite. The container and the coiled-coil filamentary member are then heated in an atmosphere of carbon or carbon plus nitrogen as the only reactive gases with the heating temperatures being predetermined and maintained for a sufficient period of time to cause the filamentary member to convert to stoichiometric tantalum carbide. As a final step, the fully carbided member is cooled in a nonoxidizing atmosphere.

BRIEF DESCRIPTION OF THE DRAWINGS

For a better understanding of the invention, reference may be had to the exemplary embodiment shown in the accompanying drawings, in which:

FIG. **1** is a flow chart setting forth the steps of the present method;

FIG. **2** is an enlarged view of a coiled-coil tantalum filamentary member as retained in a firing container with the interstices between the coils filled with high purity graphite powder;

FIG. **3** is an isometric view, shown partly in section, illustrating a plurality of stacked containers as may be used in the carbiding process; and

FIG. **4** is an isometric view, shown partly in section, of a projection lamp which incorporates a coiled-coil filament which was previously carbided in accordance with the present invention.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

The basic steps of the present method are outlined in the flow chart shown in FIG. **1**. As described therein, a tantalum or tantalum alloy wire is formed into a coiled-coil by conventional techniques and such a coil **10** is shown in enlarged form in FIG. **2**. The coil **10** is supported in a firing container **12** which consists essentially of carbon as the only container component which will react with tantalum. Preferably the container **12** is formed of graphite. The interstices between the individual coils of the filament **10** are filled with finely divided carbon, which preferably is finely divided, high purity graphite **14**.

A preferred arrangement for conducting the carbiding is to stack a plurality of individual containers **12** in a carbon crucible **16**, as is shown in FIG. **3**. The actual carbiding of the coil **10** can follow the process steps as outlined in the aforementioned U.S. Pat. No. 3,411,959 or the aforementioned copending application Ser. No. 698,962, filed Jan. 18, 1968. When following the method steps of U.S. Pat. No. 3,411,959, the crucible **16** is placed into an electrically heated or induction-heated furnace which has a controlled inert gas atmosphere such as argon, although vacuum could be used if desired. In accordance with the steps as outlined in this patent, the supported filament **10** and firing crucible **16** are heated to a predetermined temperature which is below the eutectic melting temperature of tantalum-carbon (2,800° C.), 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. More specifically, the filament is heated in an argon atmosphere at a temperature of from 2,600° to 2,650° C. for a period of 30 minutes. In the next step, the crucible **16** and filament **10** are rapidly heated to a final heating temperature which is 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 and form stoichiometric carbide. Preferably the final heating temperature is at least 3,000° C. and as a specific example, a temperature of about 3,100° C. maintained for a period of 30 minutes is normally sufficient. As a final processing step, the partially carbided filament is cooled under non-reactive conditions to a temperature at which the filament will not oxidize. The carbided coiled coil **10** is then removed from the embedding carbon **14**.

When carbiding in accordance with the teachings of copending application Ser. No. 698,962, filed Jan. 18, 1968, nitrogen is introduced into the furnace in a controlled amount and this has a marked effect in promoting the carbiding, thus allowing a lower carbiding temperature to be used. More specifically, the crucible **16** and thus the filament **10** are heated to a temperature of at least about 1,800° C., with the heating atmosphere consisting essentially of carbon and nitrogen as the only reactive gases, and any remaining gaseous component being inert gas, with the volume ratio of nitrogen to inert gas being at least about 10/90 and desirably from 10/90 to 60/40. This heating is continued until the filament is converted to stoichiometric tantalum carbide, and the carbide

member is thereafter cooled in a nonoxidizing atmosphere. More specifically, the filament is heated to a temperature of from about 1,800° C. to about 2,500° C., and preferably from 2,100° to 2,300° C., with the preferred time being about one hour. In practicing this method, the preferred volume ratio of nitrogen to inert gas is about 30/70.

It is desirable to initially heat the filament to a temperature of about 1,800° C. to about 2,100° C. for at least about 10 minutes, and preferably at about 1,800° C. for about 20 minutes, with only the inert gas atmosphere, and thereafter complete the carbiding with the nitrogen present.

After the carbiding is completed and the filament is cooled, it is removed from the fine powder of graphite. Preferably, a relatively heavy tantalum rod is inserted in each end section of the filament prior to the carbiding and during the carbiding process, the overwound filament turns are diffusion bonded to the tantalum rod and both are completely carbided. This facilitates mounting the filament in a projection lamp and such a member is disclosed in copending application Ser. No. 535,835, filed Mar. 21, 1966, by R. Corth, one of the coinventors herein, and owned by the present assignee.

The completely carbided filaments display no distortion even though they are of a coiled-coil configuration. Apparently the presence of the graphite throughout the interstices of the filament provides a constant vapor pressure of carbon, thereby eliminating non-uniformities in the rate of carbiding and thus distortions. A specification for a completed filament is as follows: the wire diameter is 9.0 mils (229 microns), the primary mandrel diameter is 10 mils (254 microns), the primary coil has 79.5 turns per inch (31.3 turns per cm.), the secondary mandrel which is used in forming the coiled-coil and which determines the diameter of the bore of the outer coil is 31 mils (787 microns), there are 23.33 turns per inch (9.2 turns per cm.), in the secondary coil, with the total turns being 5, and the primary coil diameter is 28 mils (712 microns).

In FIG. **4** is shown a projection lamp which incorporates a filament member **10** which has been fully carbided in accordance with the present invention. The lamp **18** comprises a radiation-transmitting envelope **20** which encloses an atmosphere such as nitrogen and has a base **22** affixed thereto with conventional contact pins **24**. A reflector **26** is supported within the envelope and the coiled-coil filament is supported proximate the focal point of the reflector. Preferably, the end sections of the filament **10** incorporates relatively heavy tantalum carbide rod members **28** which are diffusion bonded to the overlying coil portions to facilitate filament mounting, and the filament is mounted between bifurcated end portions **30** of the lead-in and support members **32**.

While the coiled-coil filament **10** is preferably formed of an alloy of 90 percent tantalym and 10 percent tungsten by weight, other refractory-containing alloys which contain a major percentage of tantalum can also be used, as described in U.S. Pat. No. 3,219,493, dated Nov. 23, 1965. The filament prior to carbiding can also be 100 percent tantalum.

We claim as our invention:

1. The method of carbiding a coiled-coil filamentary member formed of tantalum or an alloy principally comprising tantalum, which method comprises:

a. supporting said coiled-coil filamentary member in a firing container consisting essentially of carbon as the only container component which will react with tantalum;

b. filling the interstices between individual coils of said filamentary member with finely divided carbon;

c. heating said container to heat said filamentary member to a temperature of at least 1,800° C. with the heating atmosphere consisting essentially of carbon and nitrogen as the only reactive gases, and any remaining gaseous component being inert gas with the volume ratio of nitrogen to inert gas being at least 10/90, for a sufficient period of time to cause said filamentary member to display the gold color of substantially stoichiometric tantalum carbide; and

d. cooling said carbided filamentary member in a nonoxidizing atmosphere and separating said carbided filamentary member from the embedding carbon.

2. The method as specified in claim **1**, wherein said finely divided carbon is high purity graphite.

3. The method as specified in claim **1**, wherein said container and filamentary member are heated to from about 1,800° C. to about 2,500° C. with the volume ratio of nitrogen to inert gas being from about 10/90 to 60/40.

4. The method as specified in claim **3**, wherein prior to heating said container wherein the container atmosphere consists essentially of carbon, nitrogen and inert gas, said container is initially heated to heat said filamentary member to a temperature of from about 1,800° C. to about 2,100° C. for at least about 10 minutes, with the container atmosphere during this initial heating consisting essentially of carbon and inert gas.

5. The method as specified in claim **4**, wherein said container is initially heated to heat said filamentary member in an atmosphere consisting essentially of carbon and inert gas to a temperature of about 1,800° C. for about 20 minutes, and thereafter said container is heated to heat said filamentary member in an atmosphere consisting essentially of carbon, nitrogen and inert gas to a temperature of from about 2,100° to 2,300° C. for about one hour.

6. The method of carbiding a coiled-coil filamentary member formed of tantalum or an alloy principally comprising tantalum, which method comprises:

a. supporting said coiled-coil filamentary member in a firing container consisting essentially of carbon as the only container component which will react with tantalum;

b. filling the interstices between individual coils of said filamentary member with finely divided carbon;

c. initially heating said container and said filamentary member in an atmosphere consisting of carbon as the only reactive constituent 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 filamentary member;

d. maintaining said initial heating for a predetermined period of time to cause carbon to diffuse into said filamentary member in an amount less than that required to form stoichiometric tantalum carbide but in amount sufficient to raise the melting temperature of said partially carbided filamentary member to substantially more than said eutectic temperature;

e. heating said filamentary member 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 filamentary member;

f. maintaining the final heating temperature for a predetermined period of time to cause additional carbon to diffuse into said filamentary member to form stoichiometric tantalum carbide; and

g. cooling said carbided filamentary member in a nonoxidizing atmosphere and separating the carbided filamentary member from the embedding carbon.

7. The method as specified in claim **6**, wherein said initial heating temperature is less than 2,800° C., and said final heating temperature is more than 3,000° C.

8. The method as specified in claim **7**, wherein said initial heating is conducted at a temperature of from 2,600° to 2,650° C. for a period of about 30 minutes, and said final heating is conducted at a temperature of about 3,100° C. for a period of about 30 minutes.

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