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OXIDATION RESISTANT HIGH TEMPERATURE STRUCTURES

Filed March 25, 1969

2 Sheets-Sheet 1

FIG. 1

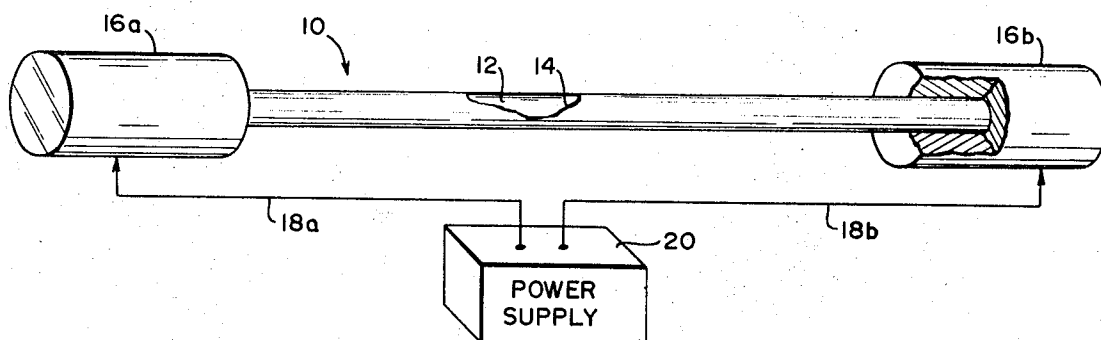
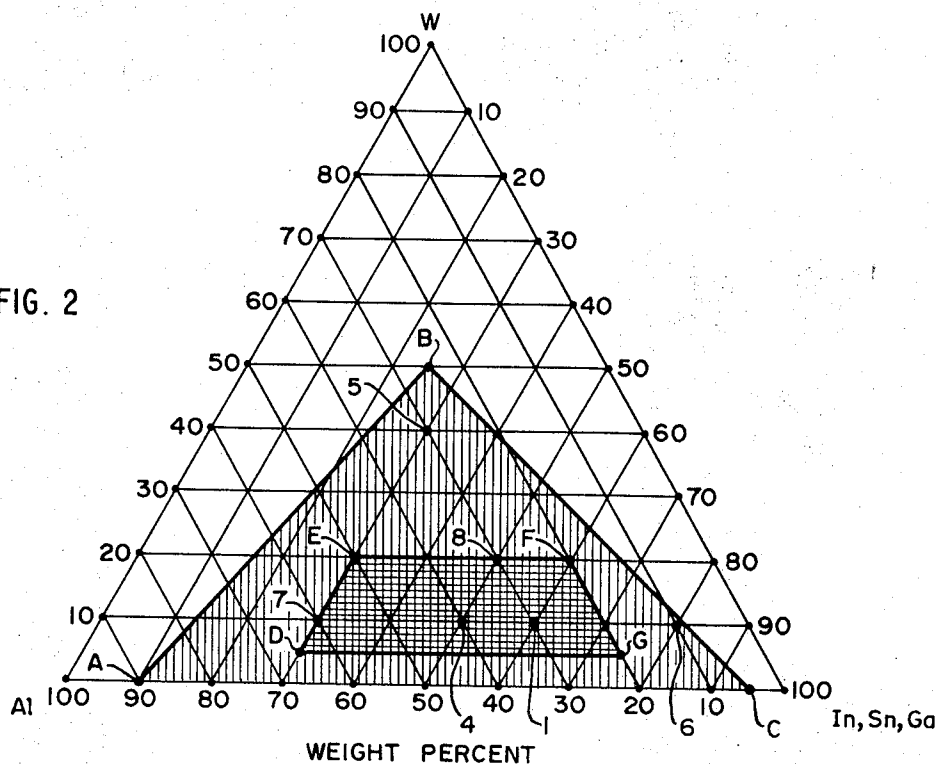


FIG. 2



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FIG. 3

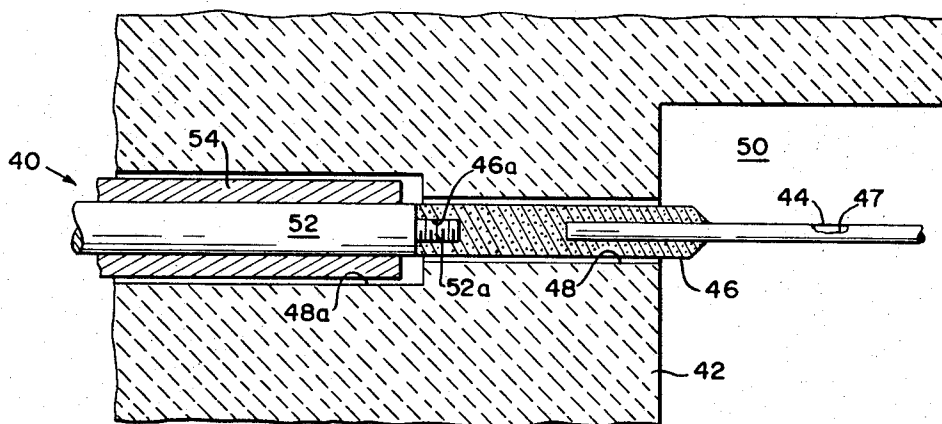
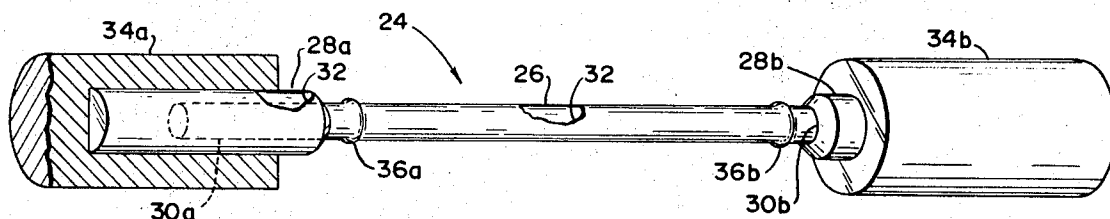


FIG. 4

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## OXIDATION RESISTANT HIGH TEMPERATURE STRUCTURES

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24 Claims

### ABSTRACT OF THE DISCLOSURE

A structure such as a crucible or heating element which operates at very high temperatures in air without atmospheric corrosion has a core member made of tungsten, graphite, carbon or molybdenum. A protective coating covers the core member and is composed of aluminum, preferably also tungsten and a low-melting, nonreactive metal from the group consisting of indium, tin, and gallium.

At the operating temperatures of the structure, the coating remains in a partially liquid state forming a continuous protective film on the core member for an extended period.

### BACKGROUND OF THE INVENTION

This invention relates to structures which are capable of operating at very high temperatures without degradation due to oxidation. While we are concerned here specifically with high temperature electrical heating elements, the invention has application also in connection with crucibles, heat shields and linings, furnace components, heated filaments and other such structures which encounter very high temperatures in normal use.

Conventionally, heating elements are made of tungsten, carbon, graphite, molybdenum and other such materials which are able to withstand relatively high temperatures. However, at very high temperatures, e.g. over 1300° C., these materials deteriorate very rapidly due to oxidation. As a result, the prior elements burn out almost immediately. Even at lower temperatures, these elements deteriorate, although at a slower pace, so that their heat outputs vary and they still have a relatively short life.

In order to minimize the degradation due to atmospheric corrosion of structures made of these refractory materials, the elements are usually operated in an inert atmosphere. However, this requires that they be housed in an airtight enclosure which has to be purged of air or charged with an inert gas each time work is placed in the enclosure. This, of course, makes the overall unit relatively large and heavy, as well as expensive.

Heating elements made of other materials do exist which can be operated in air for varying periods of time at very high temperatures. However, they have other disadvantages which limit their application. For example, structures made of some platinum alloys can operate at temperatures on the order of 1600–1700° C. However, they are expensive, and their useful lives at these temperatures are quite short. For example, a 0.20 inch diameter platinum rhodium alloy element operated at a furnace temperature of 1650° C. will burn out after only 5 or 6 hours. If the temperature is reduced to 1600° C., its life span is still only a day or two. Moreover, this type of element must be very small and be operated in a small furnace, i.e. a 3/4 inch diameter tube, in order to develop these high temperatures without prohibitive cost. Therefore, the amount of work that can be heated by it at any one time is very small.

Silicon carbide rods are capable of operating at temperatures as high as 1700–1750° C. Here again, however, their

useful lives are quite short. In addition, the silicon carbide material is quite brittle and has a low tensile strength. Therefore, the element as a whole is quite fragile. Another disadvantage of the silicon carbide rods is that, at high temperatures, they have a very low watt density with the result that it takes a relatively long time for the heating elements to reach their operating temperatures.

Still other conventional high temperature heating elements have been made of molybdenum disilicide. These, like the silicon carbide rods, are quite fragile. Moreover, when the elements are oriented horizontally, they tend to sag. Therefore, they need support all along their lengths. Also, deterioration tends to occur at these supported regions and the supports themselves must be built to withstand the very high operating temperatures.

Heating elements constructed of other materials have been proposed. However, invariably they are deficient in one or more of the respects of high cost, fragility and short life.

### SUMMARY OF THE INVENTION

Accordingly, this invention aims to provide a structure which is capable of operating at very high temperatures in air with minimal atmospheric corrosion.

Another object of the invention is to provide an improved electrical heating element which can operate at these high temperatures for a relatively long period without failure.

A further object of the invention is to provide an improved electrical heating element which can withstand repeated temperature cyclings over a relatively wide temperature range.

Another object is to provide a high temperature heating element having selected reproducible heating characteristics.

Yet another object of the invention is to provide a high temperature heating element which is supported only at its ends, yet which does not sag in use.

Still another object of the invention is to provide an improved high temperature electric furnace using one or more of these heating elements.

Other objects of the invention will in part be obvious and will in part appear hereinafter.

The invention accordingly comprises the features of construction, combination of elements and arrangement of parts which will be exemplified in the constructions hereinafter set forth, and the scope of the invention will be indicated in the claims.

Briefly, our improved high temperature structure comprises a core member made of tungsten, graphite, carbon, molybdenum or combinations of these refractory materials. A protective alloy coating covers the core member and is comprised of aluminum, preferably also tungsten, and a low-melting, nonreactive metal from the group consisting of indium, tin, and gallium. The preferred compositions of the alloy coatings will be described in more detail later.

At room temperature, the alloy coating forms a solid sheath around the core member. However, at the normal operating temperature range of the heating element or other structure, the coating is maintained in a partially liquid state so that it forms a continuous protective film on the core member which isolates the latter from the air and thereby protects it from oxidation and other atmospheric corrosion. Also, for this reason, the coating is able to tolerate the expansion and contraction of the core member during temperature changes.

The aluminum component of the coating provides a protective oxide film on the surface of the element. The indium, tin or gallium form a low melting fluid at the operating temperature which is essentially nonreactive with the underlying core material and which enables the

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aluminum oxide to remain flowable on the surface of the core member so as to preserve the continuity of the coating. The tungsten is desirable because it lowers the solubility of the coating for the core material and minimizes attack on it. It also increases the viscosity of the alloy so that a coating of optimum thickness can be applied to the core member.

Heating elements made in accordance with this invention are able to operate in air at temperatures as high as 1900° C. and more for relatively long periods without failure. This is in sharp contrast to uncoated elements having the same core material which fail substantially immediately (i.e. within 1-3 seconds). Moreover, the elements can be recycled many times between these high temperatures and room temperature without any material adverse affect. Also, the elements are structurally relatively strong and durable and have a minimum tendency to sag. Therefore, they can be supported solely at their ends. Yet, with all of these advantages, the elements are still relatively easy and inexpensive to make compared to those prior conventional ones which are able to withstand these very high temperatures. Consequently, a relatively large volume, high temperature furnace can be constructed using a number of these elements without the cost of the furnace becoming excessively high.

#### BRIEF DESCRIPTION OF THE DRAWINGS

For a fuller understanding of the nature and objects of the invention, reference should be had to the following detailed description taken in connection with the accompanying drawings, in which:

FIG. 1 is an isometric view of a high temperature electric heating unit made in accordance with this invention;

FIG. 2 is a triangular compositional diagram of the coating utilized in the practice of the present invention;

FIG. 3 is an isometric view of another embodiment of our heating element; and

FIG. 4 is a sectional view with parts in side elevation showing another heating element embodiment used in an electric furnace.

#### DESCRIPTION OF THE PREFERRED EMBODIMENTS

Referring to FIG. 1 of the drawings, our heating element 10 comprises a solid core member 12 made of tungsten, graphite, carbon or molybdenum. Member 12 may be a straight rod or wire, as shown, or it may be formed into a loop or coil depending upon the particular application. A coating 14, to be described in more detail later, covers core member 12.

In use, element 10 is heated by passing an electric current through it. For this, a pair of sleeve-like electrodes 16a and 16b are engaged over the ends of the element and these are connected by electrical leads 18a and 18b to a standard power supply 20. Electrodes 16a and 16b are made of a highly thermally conductive metal such as brass and they are relatively massive so that they can also function as heat sinks to cool the ends of the heating element in the vicinity of its electrical connections to power supply 20. With this arrangement, element 10 can operate in air at precisely controlled very high temperatures, in excess of 1300° C., for a relatively long period without failure. Moreover, it can be recycled between these high temperatures and room temperature many times without appreciably shortening its operating life.

Coating 14 is an alloy composed of aluminum and a metal selected from the group consisting of indium, tin and gallium; and a preferred coating also includes tungsten.

Referring now to FIG. 2, all possible coating compositions are illustrated by that triangular diagram. How-

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ever, all compositions (represented by the diagram as a whole) do not make a satisfactory coating 14. When alloy coating 14 is composed of the ingredients mixed in the proportions shown by the shaded area designated ABC in FIG. 2, then the operating temperature and life of element 10 are substantially increased, with optimum results being obtained with compositions within the range indicated by the shaded area DEFG.

Turning again to FIG. 1, when applying coating 14 to core member 12, the coating ingredients are melted and thoroughly mixed. Then the core member is dipped into the molten alloy. Alternatively, the coating may be sprayed or sputtered directly onto the surface of member 12. At room temperature, the alloy coating 14 forms a solid sheath which clads member 12. Over most of the operating range of the heating element, however, coating 14 is partially liquid so that it forms a flowable, oxidation-resistant film on the surface of member 12 which protects the latter from atmospheric corrosion. The fact that the alloy coating is flowable when element 10 is in use insures that the protective film is continuous over the entire surface of core member 12. Thus, no part of that member is exposed to the atmosphere. Also, the alloy coating is able to follow readily the expansions and contractions of core member 12 as that changes temperature so that there is no breach in the alloy coating at any point in the operating cycle.

The precise reason why the coating ingredients coact as they do to enable element 10 to withstand such high temperatures in air is not altogether understood. It is believed, however, that the aluminum forms a protective oxide film on the surface of the coating. The indium, tin or gallium, on the other hand, provide a low melting fluid medium which is essentially nonreactive and, when used in conjunction with the other coating components, remain in equilibrium with the underlying core material so that the oxide film tends continuously to heal itself. Aluminum, without indium, tin or gallium, does not possess this "healing" capability and does not appreciably improve the operating life of the heating element.

Preferably, coating 14 also includes tungsten. This material increases the viscosity of the coating alloy so that a coating of optimum thickness can be applied (i.e. 8-20 mils). In addition, when the underlying core member 12 is itself made of tungsten, the tungsten component in coating 14 minimizes attack on the core member. On the other hand, when member 12 is made of graphite, the tungsten in the coating serves to enhance the adherence of the coating to the graphite. For these reasons, tungsten improves the oxidation protection afforded member 12 and thereby raises the operating temperature of element 10 and prolongs its useful life.

It is also found that oxidation protection is maximized if a small amount of the tungsten in the coating is replaced by boron. Although the boron content of coating 14 can be as high as 10% by weight, the preferred amount of boron is found to be between 0.5% and 2%, with 1% being the optimum. In any event, the tungsten content of the coating should still be at least 5% by weight as shown by area DEFG in FIG. 2.

In some applications, for reasons of cost or availability, gold, silver or copper may be substituted for a minor part of the indium, tin or gallium. Also, for the same reasons, a minor amount of aluminum (i.e. up to 30 wt. percent) may be replaced by chromium.

Tests were run on a number of heating elements with the results noted below. In each case, the coating 14 was applied by dipping core member 12 in the molten alloy and the coating materials used were of very high purity. Each element was connected in the arrangement shown in FIG. 1 and the temperature of the heating element was raised immediately to the indicated temperature in air as measured by a Leeds and Northrup "Ray O Tube" brand radiation pyrometer focused on the center of the element.

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## EXAMPLE 1

A coating was applied to a 0.125 inch diameter, 4 inch long, tungsten rod to a thickness of approximately 8 mils. The coating composition by weight percent was as follows (see point 1 in FIG. 2):

	Percent
Tin -----	60
Aluminum -----	30
Tungsten -----	9
Boron -----	1

The temperature of the heating element was raised immediately to 1820° C. by passing a current through it. The heating element was maintained at this temperature for 23½ hours in open air before failure. Failure was indicated, as in all the other tests, by a trail of core material oxide vapor originating from a point on the heating element.

Further, this construction was found to be quite resistant to thermal cycling. For example, a heating element made in accordance with Example 1 was cycled in 10 second intervals from room temperature to 1800° C. and down again for 50 cycles without failure or flaking of the coating.

## EXAMPLE 2

A heating element consisting of a 0.100 diameter tungsten rod core member was coated with the Example 1 coating material. This element was heated to 1900° C. and maintained at that temperature for four hours in open air before failure.

## EXAMPLE 3

The heating element having a tungsten rod core was coated with the following alloy (see point 1 in FIG. 2):

	Percent
Tin -----	60
Aluminum -----	20
Chromium -----	10
Tungsten -----	9
Boron -----	1

This element was heated gradually while monitoring it with the pyrometer. Failure of the coating did not occur until a temperature of 2085° C. was attained.

## EXAMPLE 4

A heating element having a 0.175 inch diameter tungsten rod as a core member had a coating of the following composition (see point 4 in FIG. 2):

	Percent
Tin -----	50
Aluminum -----	40
Tungsten -----	9
Boron -----	1

This element was operated at 1820° C. for 33 hours in air without failure at the time the test was terminated.

The following examples show results obtained at various extremes of the FIG. 2 coating composition range.

## EXAMPLE 5

A heating element having a 0.100 inch diameter tungsten rod as a core member was coated with an alloy of the following composition (see point 5 in FIG. 2):

	Percent
Tungsten -----	39
Aluminum -----	30
Tin -----	30
Boron -----	1

The heating element was raised immediately to 1800° C. and lasted 5½ hours before failure. A comparable uncoated element failed in 1-2 seconds.

## EXAMPLE 6

A heating element having a tungsten rod core member

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had an alloy coating of the following composition (see point 6 in FIG. 2):

	Percent
Tin -----	80
Aluminum -----	10
Tungsten -----	9
Boron -----	1

This element lasted for 2½ hours at 1800° C. A comparable uncoated element failed immediately.

## EXAMPLE 7

A similar tungsten core element had an alloy coating of the following composition (see point 7 in FIG. 2):

	Percent
Aluminum -----	60
Tin -----	30
Tungsten -----	9
Boron -----	1

This element operated for four hours at temperatures ranging from 1840° C. to 1950° C.

## EXAMPLE 8

A heating element consisting of 0.100 inch diameter W-shaped tungsten rod was coated with an alloy of the following composition (see point 8 in FIG. 2):

	Percent
Tin -----	50
Aluminum -----	30
Tungsten -----	10
Boron -----	10

This element was heated to 1750° C. in air. However, when the temperature was raised to 1800° C., the coating began to fail.

We should emphasize again at this point that heating elements consisting of equivalent uncoated core members burn out in seconds when operated in air at these high temperatures. Failure is immediately apparent by the presence of smoke issuing from a point on the heating element indicating the formation of an oxide of the core material.

Referring now to FIG. 3, we have found that the operating characteristics of our heating element are enhanced when each end of the element's core member is encased in a graphite end piece before applying the protective coating so that the entire unit, including the graphite end pieces, is coated.

A heating element of this type is indicated generally at 24 in FIG. 3. The element has a solid core member 26 similar to member 12 in FIG. 1. A pair of larger diameter cylindrical graphite end pieces 28a and 28b have axial bores 30a and 30b which are arranged to snugly receive the ends of rod 26. Then an alloy coating 32 having a composition defined by the shaded areas in FIG. 2 is applied to member 26 as well as to the graphite end pieces 28a and 28b.

Element 24 is heated by passing an electrical current through it by way of electrodes 34a and 34b which engage over end pieces 28a and 28b. These electrodes also act as heat sinks. The electrodes are normally positioned so that the portions of end pieces 28a and 28b adjacent rod 26 project out from the electrodes about ½ to ¾ inch. Actually, only these projecting portions need be coated because the remainders of the end pieces within the electrodes (heat sinks) are kept sufficiently cool that they suffer no oxidation.

We have found that if end pieces 28a and 28b project out by this amount, after element 24 has been in operation for only a short time, a "doughnut" shaped annular protuberance tends to form in coating 32 near each end piece. These formations are indicated at 36a and 36b in FIG. 3 and are exaggerated for clarity.

At the operating temperatures of the element, these formations 36a and 36b remain at yellow heat. On the

other hand, the projecting portions of graphite end pieces 28a and 28b, as well as the portions of the coated rod 26 immediately adjacent these formations, remain black. This indicates that these portions stay relatively cool and we have found that this has a beneficial effect on the operating life of element 24 as a whole.

It is believed that these formations are due initially to the differential expansion of the molten alloy coating 32 and tungsten core member 26, together with the restrictive effect on the coating of the graphite end pieces 28a and 28b. Further, tests indicate that there may be a chemical as well as a physical effect produced by this construction. That is, these formations 36a and 36b have been found after testing to be black to metallic grey in color and very hard to grind. While their composition is not known exactly, they have the appearance and characteristics of a metallic-bonded, mixed oxide-carbide material.

The following examples will illustrate the efficacy of the FIG. 3 construction:

#### EXAMPLE 9

A heating element consisting of a 0.175 inch diameter tungsten rod and graphite end pieces approximately 1¼ inch long and ⅝ inch in diameter were coated with the alloy of Example 1. The element was mounted in brass heat sinks with the graphite ends protruding as described above in connection with FIG. 3. Electric current was passed through the element to bring it rapidly to 1820° C. This element remained operating at this temperature in air for 44 hours before failure. An element made of a similarly coated tungsten rod, but without the graphite end pieces failed after 23½ hours at this temperature.

#### EXAMPLE 10

Another heating element having a similar tungsten rod core and graphite end pieces was coated with the Example 4 alloy. This element operated in air at 1820° C. for 47½ hours without failure at the time the test was terminated.

Due to the large amount of heat generated near the upper limits of the temperature capability of our heating element, the electrical contacts to the element may become overheated unless steps are taken to prevent this. This may be done, for example, by cooling electrodes 34a and 34b by circulating water through them.

Actually, in the FIG. 3 element, the graphite end pieces 28a and 28b, being of relatively large diameter, do not generate appreciable heat during the passage of current through the element as compared with the coated core member 26. Therefore, they are often cool enough to serve as the electrical leads for the element where they extend through the thermal insulation of a furnace wall.

FIG. 4 illustrates another heating element embodiment indicated generally at 40 mounted in a refractory insulating furnace wall 42. Only one side of the furnace wall and one end of the element are shown.

Heating element 40 comprises a tungsten rod core member 44 having a larger diameter graphite end piece 46 similar to end piece 28a in FIG. 3. A coating 47 of the type described above covers core member 44 and end piece 46 as described above in connection with FIG. 3.

The graphite end piece 46 is snugly received in a passage 48 in furnace wall 42. The element is positioned in this passage so that the coated core member 44, as well as an inch or so of the coated end piece 46, project out of passage 48 into the furnace cavity 50. End piece 46 thus supports the coated rod 44 and serves as an electrical lead within the intermediate temperature zone inside the furnace wall.

The end piece may extend entirely through the wall. Alternatively, as shown in FIG. 4, a rod 52 of thermally conductive metal such as aluminum can substitute for the graphite in the end piece midway through wall 42. In this event, the aluminum rod has a reduced diameter

threaded end portion 52a which screws into a correspondingly threaded axial bore 46a in end piece 46. The utilization of the aluminum rod 52 is desirable in some cases because the aluminum is less expensive and stronger than the coated graphite.

A rather massive thermally conductive electrode-heat sink 54 snugly engages over aluminum rod 52 within wall 42, the passage 48 through the wall being enlarged at 48a to accommodate it. Thus, the graphite and aluminum members 46 and 52 serve as conductors for the element 40 in the cooler regions of the furnace, connecting the element both electrically and thermally to the heat sink 54.

The same arrangement is, of course, used at the other end of heating element 40.

It is important to note that our heating elements are all self-supporting and, unlike some conventional units which operate at much lower temperatures, they do not sag appreciably even at their maximum service temperature, i.e. 1900° C. As a result, our elements can be suspended within a furnace chamber solely by their end leads. This is highly desirable because any contact with the hot midportion of the element can result in contamination and have a deleterious effect on the element's performance. However, even in those applications where contact with the element is essential, this can be accomplished using a high purity alumina ceramic (e.g. 99% Al<sub>2</sub>O<sub>3</sub>), provided that there is no appreciable abrasion of the coated surface when the element is in use.

It will be seen from the foregoing then that our coated structures are able to function effectively at very high temperatures in air for relatively long periods without degradation due to oxidation and other atmospheric corrosion. The high temperature electric heating elements specifically described above function effectively in air at work temperatures on the order of 1900° C. These elements may be used singly or in groups to maintain a very high operating temperature within a relatively large refractory enclosure. Moreover, since these elements are capable of withstanding oxidation, there is no need to maintain an oxygen-free atmosphere within the furnace.

The elements described herein are advantaged also in that they are rugged, nonbrittle, durable and have no tendency to buckle or sag in use. Moreover, they can withstand repeated temperature cyclings over their operating ranges without seriously shortening their operating lives.

It will thus be seen that the objects set forth above, among those made apparent from the preceding description, are efficiently attained and, since certain changes may be made in the above constructions without departing from the scope of the invention, it is intended that all matter contained in the above description or shown in the accompanying drawings shall be interpreted as illustrative and not in a limiting sense.

It is also to be understood that the following claims are intended to cover all of the generic and specific features of the invention herein described, and all statements of the scope of the invention which, as a matter of language, might be said to fall therebetween.

We claim:

1. A structure capable of withstanding very high temperatures in air, said structure comprising

(A) a core member made of at least one refractory material selected from the group consisting of tungsten, graphite, carbon and molybdenum, and

(B) a coating covering said core member, said coating comprising an alloy of

(1) aluminum, and

(2) a low-melting, relatively nonreactive metal selected from the group consisting of indium, tin and gallium.

2. The structure defined in claim 1 wherein said coating is defined by and included within area A, B, C, of the FIG. 2 diagram.

3. The structure defined in claim 2 wherein a minor amount of said aluminum is substituted for by chromium.

4. The structure defined in claim 2 wherein said alloy coating also contains a minor amount of at least one metal selected from the group consisting of gold, silver and copper.

5. The structure defined in claim 2 wherein said alloy coating contains tungsten.

6. The structure defined in claim 5 wherein up to 10% by weight of said tungsten is substituted for by boron.

7. The structure defined in claim 2 wherein said coating is an alloy selected from those defined by and included within area D, E, F, G of the FIG. 2 diagram.

8. The structure defined in claim 7 wherein

(A) up to 2% by weight of said tungsten is substituted for by boron, and

(B) said alloy contains a minimum of 5% by weight of tungsten.

9. An improved oxidation-resistant electrical heating element, said element comprising

(A) an electrically conducting core member composed of at least one material selected from the group consisting of tungsten, graphite, carbon and molybdenum, and

(B) a protective alloy coating covering said core member, said coating being composed of an alloy selected from those defined by and included within area A, B, C of the FIG. 2 diagram.

10. The heating element defined in claim 9

(A) and further including a pair of refractory end pieces engaging over the ends of said core member, and

(B) wherein said coating is applied to said core member and at least the surface portions of said end pieces adjacent said core member.

11. The heating element defined in claim 10 wherein said end pieces are made of graphite.

12. The heating element defined in claim 10 and further including thermally conductive electrodes in intimate contact with said end pieces for conducting current to the coated core member and conducting heat therefrom.

13. The heating element defined in claim 12 wherein said electrodes are spaced an appreciable distance from the ends of the end pieces adjacent the coated core member.

14. The heating element defined in claim 12 wherein said end pieces have thermally and electrically conductive metallic extensions.

15. An improved heating element as defined in claim 9 wherein said alloy coating contains tungsten.

16. An improved heating element as defined in claim 15 wherein up to 10% by weight of said tungsten is substituted for by boron.

17. The heating element defined in claim 9 wherein up to 30% by weight of said aluminum is substituted for by chromium.

18. The heating element defined in claim 9 wherein said alloy coating also contains a minor amount of at least one metal selected from the group consisting of gold, silver and copper.

19. The heating element defined in claim 9 wherein said coating is an alloy selected from those defined by and included within area D, E, F, G of the FIG. 2 diagram.

20. The heating element defined in claim 19 wherein (A) up to 2% by weight of said tungsten is substituted for by boron, and

(B) said coating contains at least 5% by weight of tungsten.

21. An improved high temperature electric furnace comprising

(A) thermally insulated container,

(B) one or more electric heating elements mounted in said container for heating its interior, each said element comprising

(1) a refractory core member, and

(2) a coating covering said core member, said coating being composed of an alloy selected from those defined by and included within the area A, B, C of the FIG. 2 diagram, and

(C) electrically and thermally conductive means supporting the ends of the coated core member in the container.

22. The electric furnace defined in claim 21 wherein said coating is an alloy selected from those defined by and included within the area D, E, F, G of the FIG. 2 diagram.

23. The electric furnace defined in claim 22 wherein a minor amount of the tungsten in said coating is substituted for by boron.

24. The electric furnace defined in claim 23 wherein said end supporting means comprise

(A) graphite end pieces engaging over the ends of the coated core member, the portions of said end pieces adjacent the core member also having the same coating, and

(B) thermally conductive electrodes engaging around said end pieces at points thereon spaced appreciably from the coated core member.

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