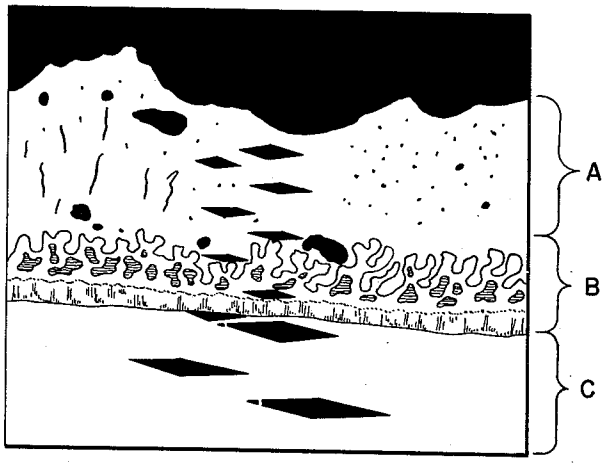
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COMPOSITE METAL BODY FOR HIGH TEMPERATURE USE

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COMPOSITE METAL BODY FOR HIGH TEMPERATURE USE

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This invention relates to the production of refractory metal bodies, and particularly to those in which a base of niobium metal or of an alloy in which niobium is a major ingredient is coated with an oxidation-resistant material. More specifically, the invention relates to metal bodies comprising a niobium or niobium-alloy base to which has been applied an adherent, protective coating of an alloy of iron and aluminum.

The valuable properties of nobium are well known. Its high-melting and softening points, its resistance to attack by acids and other corrosive chemicals, its high-temperature strength, and its ease of working make it a particularly useful metal for many applications. In combination with these desirable properties, the low-neutron absorption cross-section exhibited by niobium gives it a unique place among metals useful for atomic energy applications.

Disadvantageously, however, niobium in the unalloyed state becomes "sticky" in working, exhibiting a tendency to seize, tear and gall. In addition, niobium has a marked affinity for certain gaseous elements, and, particularly at high temperatures, will rapidly combine with oxygen. The niobium oxide formed does not protect the underlying metal; instead, the oxidation progresses rapidly to the inner part of the metal, thus making the otherwise valuable metal useless for applications in which it would be exposed to an oxidizing atmosphere. In addition to this progressive inward oxidation of the niobium metal, the oxides which form on the surface tend to spall, continuously exposing fresh surface to the action of the corrosive atmosphere.

To overcome these disadvantageous properties of niobium metal, and particularly its affinity for oxygen, it has been proposed to alloy the niobium with other metals. Some niobium-base alloys have been produced which are proving useful in overcoming this undesirable property of niobium metal. However, in many applications, an advantage may be gained by coating the alloy in the manner described in this specification, since the application of the coatings described provides a surface which is highly resistant to oxidation, thus making it possible for structural components of the alloy to be in use for relatively long periods of time before the niobium alloy itself is actually exposed to the deteriorating influence of oxygen.

It is the object of this invention to produce composite metal bodies comprising a niobium or niobium-alloy base to which there has been applied a firmly bonded coating which protects the base metal or alloy against corrosion, particularly atmospheric corrosion at high temperatures.

The objects of this invention are accomplished by coating niobium or niobium alloys with an alloy composition comprising aluminum and iron in the proportions of between 5% and 25% by weight of aluminum and between 95% and 75% by weight of iron. The coating may be applied by immersing the metal to be coated in a molten alloy mix; or the alloy may be applied to the base metal by flame spraying; or, alternatively, the object to be coated may be packed in a powdered coating material and heat applied to obtain a temperature such that the alloy coating is firmly bonded to the surface of the base metal object. All of these methods are well known, and a preferred procedure in using them is to carry out the coating operation in vacuum or an inert gas atmosphere. The iron-aluminum metal coating may be in the form of a prealloyed powder mix, or it may be an unalloyed mixture of the two metals in powder form in the correct proportion to give the desired alloy coating when heat treatment is applied.

As a result of the heat teratment of the niobium or niobium-alloy base material with alloys of iron and aluminum, or with an unalloyed mixture of these two metals in the correct proportions to give the desired alloyed coating, an alloy layer of ternary or higher composition is formed between the coating alloy and the base metal or base alloy. This alloy layer is of such composition that it forms a firm bond between the metal base and the coating alloy and affords protection to the base metal. I have found that a particularly useful alloyed coating is obtained when the proportions of metals used are in the range of 10–20% by weight of aluminum and 90–80% by weight of iron.

The drawing is a cross-sectional view at 500 magnifications of a piece of niobium metal which has been coated with an alloy consisting of 16% by weight of aluminum and 84% by weight of iron.

Referring now to the drawing, it will be seen that ternary or higher-order alloys designated by reference letter *b* in the drawing form between the iron-aluminum coating *a* and the base metal or alloy *c*. It is this formation of an intermediate alloy of the base and the iron-aluminum coating that makes the coating strongly adherent to the base. Moreover, this intermediate layer also serves as a protective coating for the base material. The diamond-shaped markings on the metal are Knoop Hardness indentations. These indentations show the distinct difference in the hardness of the unalloyed niobium as compared with the alloy composition of the intermediate and coating alloy layers.

For a clearer understanding of the invention, the following specific examples are given. These examples are intended to be merely illustrative of the invention and not in limitation thereof.

Example I

A piece of niobium sheet 0.02" x 0.5" x 1.0" was coated by packing it in an alloy powder of 16% by weight aluminum, balance iron, and heating at 1450° C. for 24 hours in vacuum. The sample was furnace cooled, and subsequently subjected to a flow of air of 1 liter/min. at 1000° C. for 67 hours. At 67 hours, the oxidation rate of this coated metal sample was found to be 0.083 mg./cm.²/hr. The overall weight gain on this coated sample was found to be 2.14%.

A photomicrograph of a cross section of this coated sample is shown in the drawing. The formation of an alloy layer between the base and the iron-aluminum alloy coating and the niobium is visible. This alloy layer firmly bonded the coating to the base metal.

A piece of niobium sheet heated under somewhat similar conditions of temperature and exposure to air was completely oxidized after two hours. The niobium metal, under these conditions, forms oxides which spall away from the main portion of the metal, exposing fresh surface to the atmosphere. The result is the complete conversion of the metal to broken-up pieces of oxide.

Example II

Niobium sheet 0.020" x 0.5" x 1.0" was coated with an alloy comprising 20% by weight aluminum and 80% by weight iron, by dipping the piece into the molten iron-aluminum alloy at a temperature of 1600° C. This treatment was carried out in vacuo. After cooling, the cross-section of a portion of the coated sheet was examined

and the microstructure showed several distinct layers, indicating the formation of alloys of several compositions in the coated sheet. No attempt has been made up to the present time to identify these alloys, but the oxidation-resistance of the coated niobium piece was tested by subjecting it to a flow of air of 1000 cc./min. at 1000° C. At 88 hours, the oxidation rate was found to be 0.0893 mg./cm.^{2}/hr. at 1000° C. The over-all weight gain resulting from this oxidation test was 1.12% on the weight of the coated niobium sample exposed to the air.

Example III

Sheet niobium 0.020" x 1" x 0.5" was packed in a powder mixture comprising 75% iron powder and 25% aluminum powder. The packed sheet was heated in vacuum for 16 hours at a temperature of 1400° C. After furnace cooling, the sample was examined and found to be completely and evenly coated with an iron-aluminum alloy which was firmly bonded to the niobium base metal. An examination of the cross-section of a portion of the coated sheet showed several structurally different layers, indicating the formation of alloys between the coating and the base metal.

A piece of niobium thus coated was exposed to a flow of air of 1 liter/min. in a tube 2.5" in diameter, 30" in length for a period of 118 hours at 1000° C. At this time a weight gain of 0.083 mg./cm.^{2}/hr. was observed. The absolute over-all weight gain on the sample of coated sheet was 17.2%.

Example IV

As previously pointed out, niobium base alloys, that is alloys containing at least 50% by weight of niobium, can be protected in the same manner.

For example, a piece of niobium base alloy of 20% titanium-80% niobium (0.05" x 0.5" x 0.75") was coated by packing it in an alloy-powder of 16% by weight aluminum, balance iron, and heating at 1400° C. for 24 hours in vacuum. The sample was furnace cooled and subsequently subjected to a flow of air of one liter per minute at 1000° C. for 24 hours. There was found to be only slight oxidation of the sample after this exposure, and the coating was found to adhere firmly to the base alloy after heating and cooling.

From these examples it will be seen that niobium metal or alloys of niobium metal can be coated with alloys consisting of iron and aluminum to produce a firmly bonded protective coating on the base metals. This treatment improves the resistance of the niobium base to oxidation at high temperatures without impairing the valuable properties which are characteristic of niobium. Another advantage of the coating is that it has less tendency to seize, tear or gall at elevated temperatures. Thus, the coated niobium particles are much easier to hot work than untreated niobium base.

Although temperatures for treating niobium metal with the iron-aluminum alloy have been given in the above examples as ranging between 1400° C. and 1600° C., the temperatures for this treatment may vary from about 1200° C. upward, depending upon the method of applying the coating and upon the composition of the iron-aluminum alloy chosen. It is evident that if the method of dipping the base metal into the alloy is chosen as the coating technique, the temperature used will be higher than if the base metal is coated by the method of packing the piece in the alloy powder and heat-treating it.

If the dipping technique is used, the preferred temperature of the alloy bath will be from about 1500° C. to about 1600° C., the lower temperature being used when the higher ratios of aluminum to iron are used. If the niobium metal or alloy base is to be coated by packing it in the iron-aluminum powder mix, the preferred temperature range will be about 1200° C. to about 1450° C., depending upon the composition chosen for the iron-aluminum alloy.

In using the method of flame-spraying the alloy coating on the base metal, an oxyhydrogen flame was used, and the alloy particles caused to impinge and bond onto the metal base surface. The conditions for the flame spraying will necessarily have to be decided experimentally, since the flame temperature, distance from flame to base metal, the kind and condition of base metal surface, the composition of the alloy coating material used, and other factors will vary from one time to another. The application and bonding of the iron-aluminum alloys to niobium metal or its alloys as disclosed in this invention has, however, been easily accomplished using the standard techniques of flame-spraying well known to those versed in this art.

The oxidation-resistant products of this invention will be found useful where the properties of strength at high temperature, and high softening and melting points are particularly desirable. One valuable application, for example, is in the production of high-temperature radiation shields for heat-treating furnaces. Such shields may be fabricated according to the disclosures of my invention using cold-worked niobium metal and coating the cold-worked pieces by packing in iron-aluminum powdered alloy mix followed by heating to effect bonding of alloy to base metal.

Since it is obvious that many changes and modifications can be made in the above-described details without departing from the nature and spirit of the invention, it is to be understood that the invention is not to be limited to said details except as set forth in the appended claims.

I claim:

1. A high-temperature corrosion-resistant composite metallic body of non-uniform chemical composition, comprising at least three integrally bonded distinct layers, said body consisting of a base metal selected from the group consisting of niobium and alloys in which the niobium content is at least 50% by weight; a surface layer consisting essentially of an applied, adherent alloy of iron and aluminum in which the aluminum constitutes about 5% to 25% by weight of the alloy used and the balance being essentially iron; and at least one intermediate layer in said composite body consisting of aluminum, iron, and the ingredients of the niobium-containing base metal, the said several layers of the composite body being firmly bonded to each other.

2. A composite body according to claim 1 in which the base metal is niobium.

3. A composite body according to claim 1 in which the base metal comprises an alloy having a niobium content of at least 50% by weight.

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