

[54] METHOD OF FABRICATION OF A  
COMPOSITE PRODUCT MADE UP OF AT  
LEAST TWO COMPONENTS HAVING  
DIFFERENT COMPOSITIONS

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287/189.365

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[57] ABSTRACT

At least two components having different compositions, such as niobium and alumina, and mating contact surfaces are brought together in edgewise relation in order to be diffusion-bonded, the contact surface adopted for each component or the line of extension of said surface being such as to pass through an axis which is common to both components.

Each surface is polished to an optical finish and the components are assembled so that said surfaces are in mating relation with zero clearance. A moderate holding pressure is applied to the components and these latter undergo a heat treatment at high temperature for a limited period of time and at low pressure in an inert atmosphere or in a vacuum.

5 Claims, 4 Drawing Figures

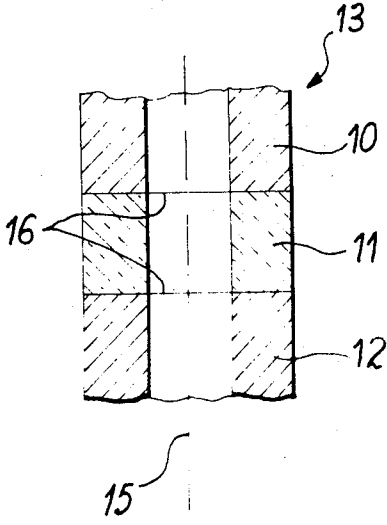


FIG. 1

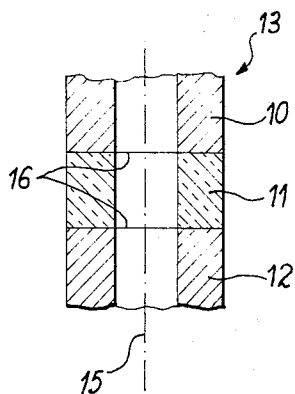


FIG. 2

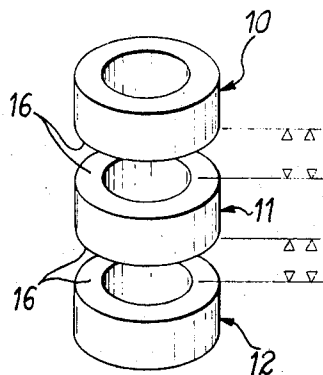


FIG. 3

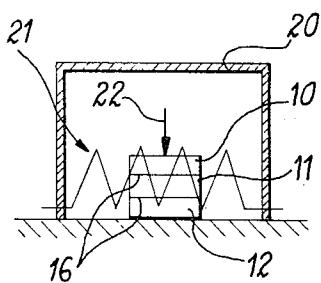
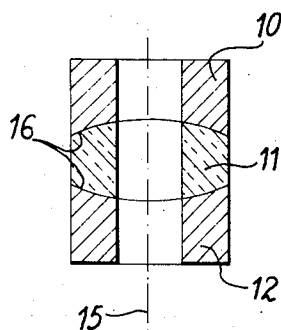


FIG. 4



# **METHOD OF FABRICATION OF A COMPOSITE PRODUCT MADE UP OF AT LEAST TWO COMPONENTS HAVING DIFFERENT COMPOSITIONS**

This invention is generally concerned with the formation of a composite product made up of at least two elements having different compositions and joined together by diffusion bonding along mating surfaces which are brought together in edgewise relation. By way of example, consideration will be given more particularly to the application of a product of this type to the construction of a thermionic conversion diode.

Thermionic conversion diodes are designed for high-temperature operation and are usually made up of two metallic sheaths between which is interposed an insulating spacer element. In general, the metal employed for the fabrication of the sheaths aforesaid is pure or alloyed niobium and the associated insulating spacer element is of alumina.

Accordingly, one of the problems involved in the construction of diodes of this type lies in the need for an intimate bond between two tubular components having different compositions, one component being formed of either pure or alloyed niobium and the other component being formed of alumina.

It has already been proposed to form a bond of this type by brazing but it proves very difficult to select a suitable brazing compound by reason of the high temperature which said compound must subsequently be capable of withstanding at the time of operation of the diode under conditions of service, and also by reason of the fact that diodes usually contain vapors of an alkali metal (caesium) which is chemically very active in the case of many metals employed as brazing agents.

In consequence, it has also been proposed to form the bond aforesaid by means of the diffusion bonding technique.

In this known technique, the parts to be bonded are abuttingly applied against each other at high temperature and along two complementary contact surfaces.

However, since it is necessary to form a composite product which is made up in practice of at least two successive tubular components to be joined together in pairs for the construction of a thermionic conversion diode, the diffusion bonding operation has been performed up to the present time by adopting contact surfaces which are developed cylindrically about the axis of said tubular components.

In other words, the tubular components aforesaid are first engaged in interfitting relation and joined together in pairs at their respective lateral contact surfaces.

This mode of operation calls in practice for extremely costly and complex installations. In fact, it is necessary in the first place to make provision for a predetermined clearance between the tubular components to be bonded in order that these latter may be fitted one inside the other without forcible engagement. In the second place, it is also necessary to apply a very high pressure of the order of 700 bars, for example, between the tubular components to be assembled in order to ensure an intimate contact between these latter in spite of the clearance aforesaid.

In point of fact, it is only by means of a pressure of this order, combined with the plasticity exhibited by materials constituting the metallic components employed and caused by the high temperature at which

the operation is performed, that the necessary contact for good execution of the desired diffusion bonding process can be achieved at all points of these components.

Furthermore, it is necessary to maintain the heat treatment for a period of several hours.

This method of operation is even more costly by reason of the fact that, once the diffusion bonding has been completed over all the cylindrical mating surfaces of the tubular components, the central portions of said components are removed by machining in order to achieve the desired fabrication of a composite product consisting of three tubular components disposed successively in the axial direction, the first component being formed of metal, the second of alumina and the third of metal.

A method has also been proposed for bonding a ceramic element of alumina and an element of metal such as niobium without addition of any other material between the two elements to be bonded. In accordance with this method, a niobium disc is applied against the extremity of a tube at a sufficient pressure to ensure that they are maintained in position. The entire assembly is heated to 1,800° C and the disc is heated to 2,050° C for a few seconds in order to facilitate penetration of the disc at the surface of the alumina.

Diffusion bonding of two ceramic components formed of alumina, for example, by interposition of a metallic component has already been carried out with aluminum, titanium and vanadium but this type of bond has not been formed with niobium.

However, an alumina-niobium bond has been formed by placing the niobium in a mold, by pouring particles of alumina into said mold and then heating to a temperature within the range of 1,500° to 1,800° C between 150 and 250 kg/cm<sup>2</sup>.

Leak-tight joints have also been formed between ceramic and metal components within vacuum tubes or evacuated enclosures. To this end, the surfaces to be placed in contact with each other are polished and coated with a thin layer of silver which is polished until an optical surface finish is obtained, diffusion being carried out in the hot state and under pressure. The metals employed are silver, gold or platinum.

The present invention is directed to a method of obtaining a composite product of this type in the most economical manner.

The invention is also directed to the composite product which is thus obtained.

The method according to the invention, which is generally intended for the fabrication of a composite product made up of at least two components having different compositions and joined together by diffusion bonding along mating contact surfaces brought together in edgewise relation, is characterized in that the contact surface adopted for each component aforesaid or the line of extension of said surface intersects with an axis which is common to both components, said surface is polished to an optical finish, the components are assembled so that said surfaces are in mating relation with zero clearance, a moderate holding pressure is applied thereto and said components are subjected to a heat treatment at high temperature for a limited period of time and at low pressure in an inert atmosphere or in a vacuum.

The contact surface of the components to be joined together is a flat, conical or spherical surface. As a re-

sult, it is possible to assemble these components with zero clearance; this absence of clearance combined with the high surface polish given to the mating surfaces of said components permits diffusion bonding of these latter without any need to apply a high pressure thereto and without making it necessary to extend the heat treatment over a long period of time.

It should be pointed out that, under these conditions, the bonding process in accordance with the invention does not simply consist in forming a bond by diffusion of the components into each other at the level of their contacting surface. In point of fact, there takes place a reduction of the alumina by the pure or alloyed niobium. The aluminum which is formed penetrates into the metal to a depth which can attain 100 microns whilst the metal penetrates into the alumina to a depth which can attain 20 microns.

In practice, the pressure applied to these components must be just sufficient to ensure relative position-maintenance of these latter and is of the order of 1 bar.

At the same time, the heat treatment in accordance with the invention is advantageously applied only for a period of less than one hour; since the diffusion bonding operation should preferably be carried out in a protective atmosphere, this operation can be performed in accordance with the invention either in a vacuum or in an inert atmosphere at ordinary pressure or at a slight overpressure.

Be that as it may, the possibility of dispensing with any further need to apply high pressures for the bonding operation results in economic simplification of the installations required, which in turn advantageously reduces the cost price of the composite product thus formed.

Tests have shown that the composite product obtained in accordance with the invention exhibits good mechanical strength which is equal to or higher than the mechanical strength of aluminum, namely of the order of 7 kg/mm<sup>2</sup>, and permits in particular any remachining operations which may prove necessary. Leak-tightness tests performed with a helium mass spectrometer have shown that the diffusion bonds of said product are of excellent quality. In fact, when performing these tests at temperatures which can attain 1,800° C, the observed leakage flow rate is of the order of 10<sup>-10</sup> torr/liter/second.

The characteristic features and advantages of the invention will in any case become apparent from the following description which is given by way of example, reference being made to the accompanying diagrammatic drawings, in which:

FIG. 1 is a partial axial sectional view of the composite product to be fabricated;

FIG. 2 is a view in perspective showing said composite product prior to assembly;

FIG. 3 is a view in sectional elevation which illustrates the bonding of said composite product;

FIG. 4 is a view which is similar to FIG. 1 and relates to an alternative form of construction.

As illustrated diagrammatically in FIG. 1, the invention is applied to the bonding of three tubular components placed successively in end-to-end relation, namely a metallic component 10, an insulating component 11 and a metallic component 12.

The resultant composite product 13 finds an application especially in the construction of thermionic conversion diodes, in which case the metallic component

10 is made integral with a first metallic sheath (not shown) whilst the metallic component 12 is made integral with a second metallic sheath (not shown), said metallic sheath being separated from each other by the insulating distance-piece or spacer element 11.

In practice, the metallic components 10, 12 are frequently formed of pure or alloyed niobium and the spacer element 11 is formed of alumina.

However, it is apparent that the method according to the invention is very broad in scope and can apply to a wide range of different metals and ceramic materials having substantially matched coefficients of expansion.

The invention proposes to effect diffusion bonding of the components aforesaid along contact surfaces such that either the surfaces themselves or their lines of extension intersect with their axis 15.

In the embodiment which is illustrated in FIG. 1, said contact surfaces are plane surfaces 16 located at right angles to the axis 15.

In accordance with the invention, said plane surfaces 16 are carefully machined as illustrated diagrammatically in FIG. 2 in such a manner as to give them a highly polished surface finish.

The thickness of this polished surface is at least of the order of one-tenth of a micron and preferably one-hundredth of a micron.

The components 10, 11 and 12 are applied against each other in end-to-end relation at their contact surfaces 16, then placed within a sealed enclosure 20 which is equipped with heating means 21. The components 10, 11 and 12 are held in position by subjecting these latter to a moderate pressure of the order of 1 bar as indicated diagrammatically by the arrow 22 of FIG. 3 and a plurality of units to be bonded can be placed on top of each other.

A vacuum is then created within the enclosure 20 and the components 10, 11 and 12 are brought by heating means 21 to a high temperature of the order of 1,500°-1,800° C, for example, depending on the nature of the materials to be bonded.

This temperature is maintained for a period of less than 1 hour.

The components 10, 11 and 12 are then joined together by diffusion bonding and allowed to cool naturally either in a vacuum or in a neutral atmosphere; this cooling process can last a few hours.

As described in the foregoing, tests made on the composite product 13 obtained have shown the good properties of mechanical strength and leak-tightness of this latter.

In accordance with the embodiment which is illustrated diagrammatically in FIG. 4, the contact surfaces 16 of the tubular components 10, 11 and 12 are spherical, the centers of said surfaces being preferably located on the axis of revolution of said components.

In the particular case in which the metal employed is either pure or alloyed niobium and in view of the fact that the coefficient of thermal expansion of niobium is lower than that of alumina, the arrangement adopted in this embodiment is such that the alumina component 11 is put in compression at the time of high-temperature service of the composite product which is thus formed; this putting in compression is conducive to long service life of the component since the compressive strength of alumina is higher than its tensile strength.

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It is readily understood that this invention is not limited to the embodiments which have been described with reference to the accompanying drawings but extends to any alternative form of execution, both in regard to the geometry of components and in regard to the choice of metallic and ceramic materials to be bonded.

Finally, the invention does not apply solely to the construction of thermionic diodes but extends more generally to all devices which embody the principles of electron physics, e.g., electron tubes, particle accelerators, vacuum devices, semiconductor devices and the like.

I claim:

1. A method of diffusion bonding components of alumina to niobium or niobium alloy components comprising the steps of polishing to an optical finish the contact surfaces of said components, assembling the components so that the contact surfaces are in mating relation with zero clearance, bonding said surfaces to-

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gether by diffusion bonding by applying a moderate holding pressure of about one bar to the components while subjecting the components to a heat treatment at a temperature within the range of 1,500° to 1,800°C. for a period of time on the order of one hour in an inert atmosphere or vacuum, and then naturally cooling the components.

2. A method according to claim 1, wherein the optical polish applied to the contact surfaces is at least of the order of one-tenth of a micron and preferably of the order of one-hundredth of a micron.

3. A method according to claim 1, wherein the contact surfaces are flat.

4. A method according to claim 1, wherein the contact surfaces are spherical or conical.

5. A method according to claim 1, the components being tubular shaped and being mated end to end along their common axis.

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