

[54] PROCEDURES FOR COATING
SUBSTRATES WITH SILICON CARBIDE

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[63] Continuation of Ser. No. 566,098, July 18, 1966, abandoned.

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[51] Int. Cl. B32b 15/02, B44d 1/42, C03c 25/02

[58] Field of Search.... 117/93.1, 229, 107.2, 106 C, 117/126 AF, 126 GF, 169 R, 135.1, 128, 121, 93, 115, 231; 65/3

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[57] **ABSTRACT**

Thin, adherent oxidation and corrosion resistant silicon carbide coatings are provided on filamentary base materials by continuously passing a hot surface of the filamentary material through a liquid organo silicon halide, wherein the temperature of the hot surface is high enough to cause the organo silicon halide immediately surrounding the hot surface to film boil, decompose, and deposit the silicon carbide coating. The filamentary base material has a melting point which exceeds the formation temperature of silicon carbide. The base material may be tungsten, tantalum, columbium, or alloys thereof. The base material may also be glass or alumina. When the base material is glass, the glass filaments may be coated directly after forming. All of the substrates may be preheated to the required temperature and the substrates which are electrically conductive may be heated by electrical resistance heating.

15 Claims, 4 Drawing Figures

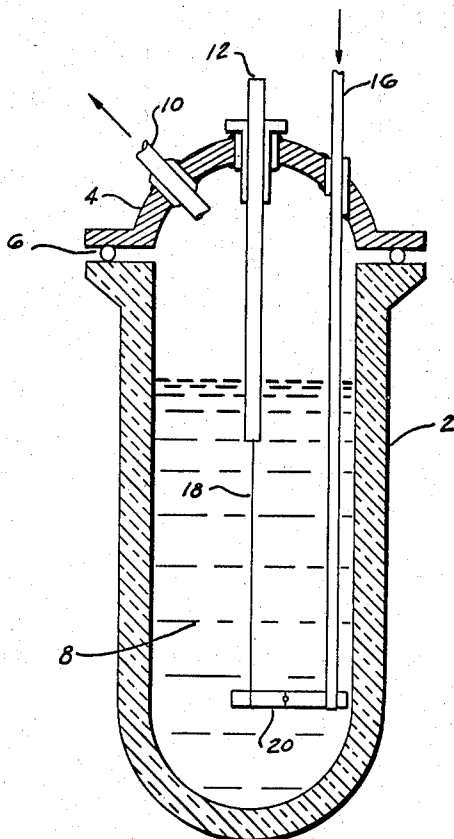
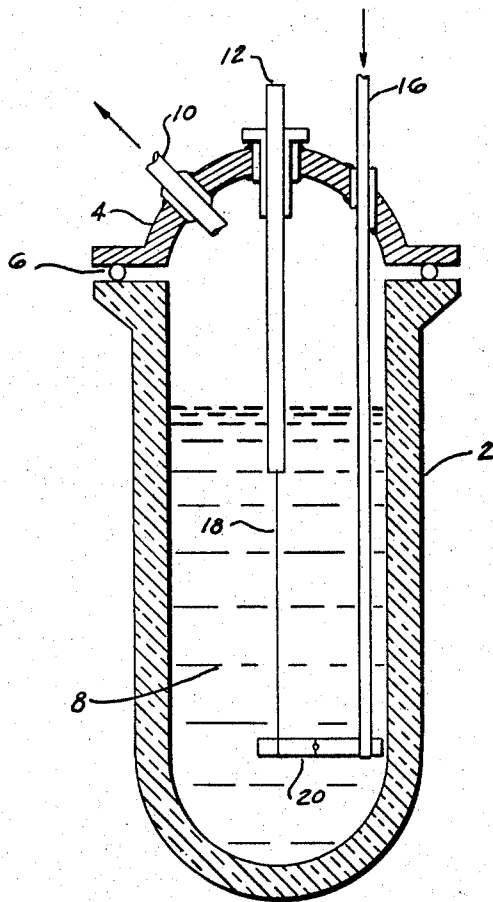


FIG-1



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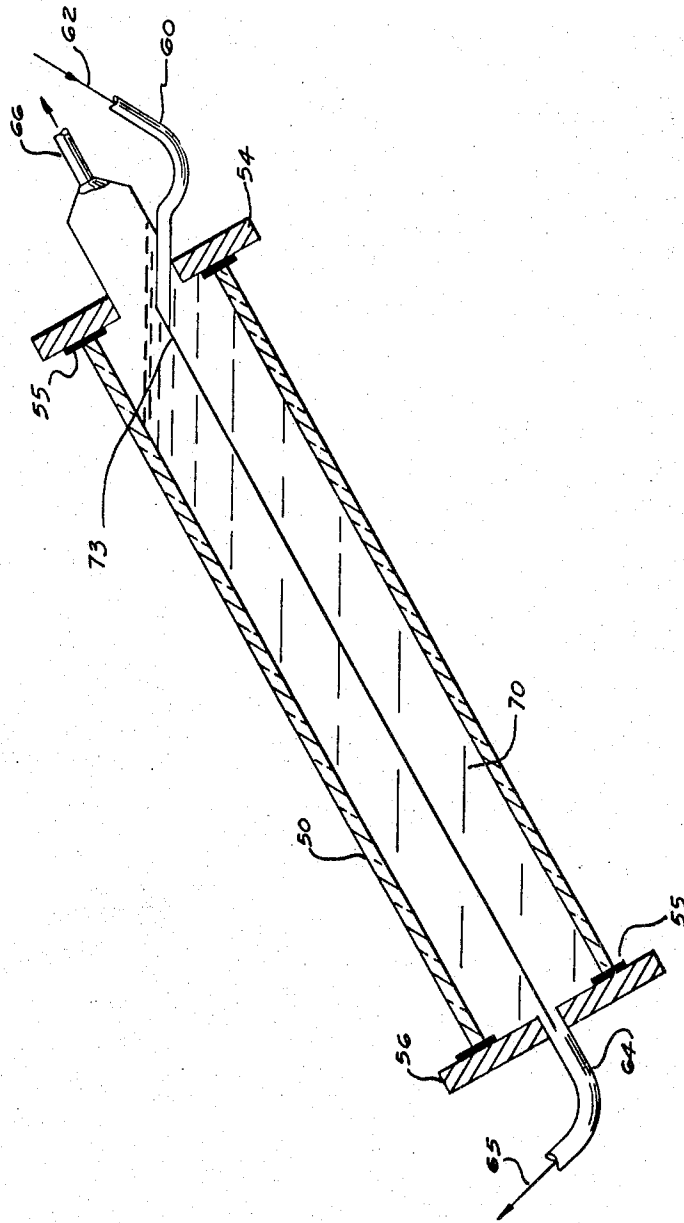


FIG-2

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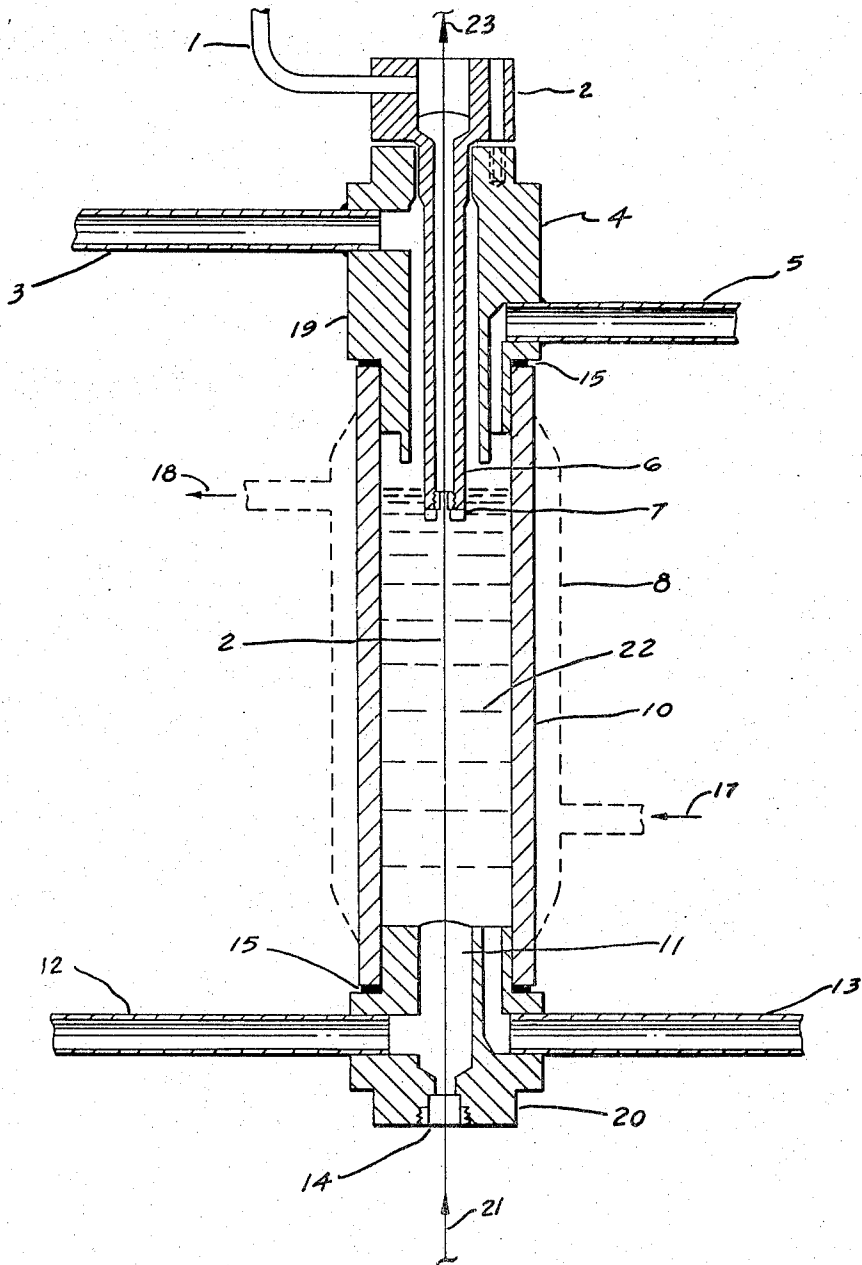


FIG-3

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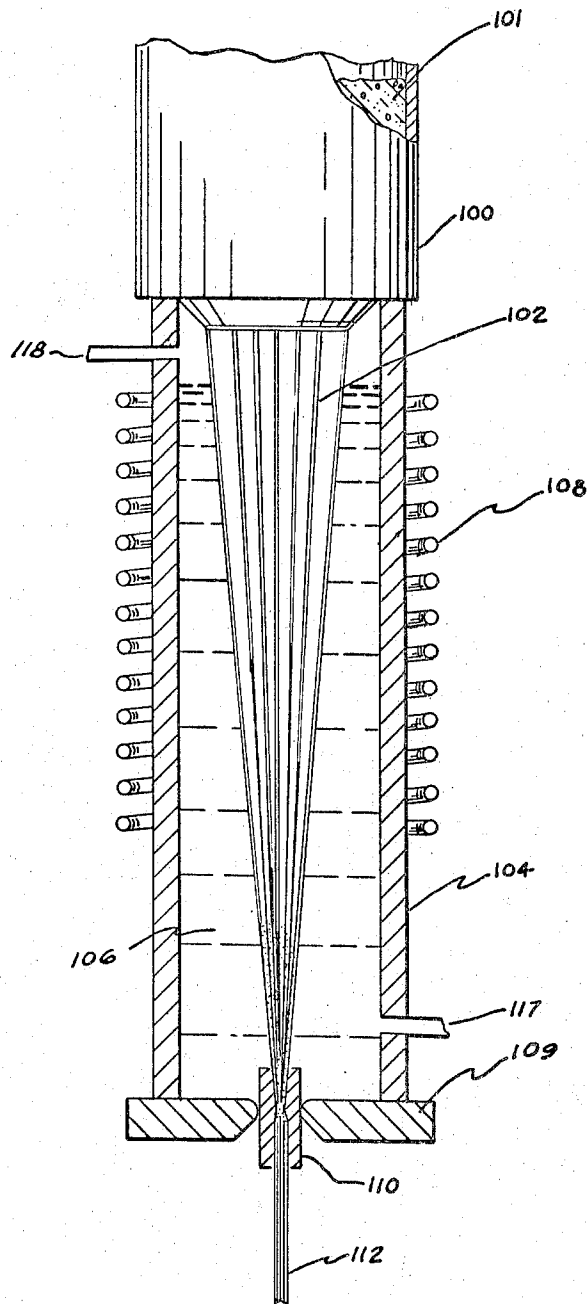


FIG-4

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PROCEDURES FOR COATING SUBSTRATES WITH SILICON CARBIDE

The present application is a continuation of U.S. application Ser. No. 566,098, filed July 18, 1966, now abandoned, and relates to methods for plating or coating insulating as well as electrically conducting substrata with silicon carbide and also to the plating or coating of filamentary forms of such substrata with silicon carbide.

One object of the present invention is to provide improved procedures for imposing a controlled coating of silicon carbide on both insulating and conducting substrata.

Another object of this invention is to continuously coat substrata of the type described with silicon carbide at high rates, so as to render such substrata resistant to oxidation, contamination and wear.

Another object of this invention is to coat continuous filaments of conducting as well as non-conducting material with silicon carbide.

A further object of the present invention is to produce composite silicon carbide coated filaments having high strength and modulus to weight ratios for use in the fabrication of structural members, including filament wound structures and fibre reinforced structures.

Additional objects and advantages of the invention will be set forth in part as part of the description and drawings which follow and in part will be obvious from the description, or may be learned by practice of the invention, the objects and advantages being realized and attained by means of the compositions, methods and apparatus particularly pointed out in the appended claims.

According to the present invention, a new and improved method has been developed to achieve dense, uniform, adherent, non-porous coatings of silicon carbide under controlled conditions at extremely high rates on a wide variety of insulating and conducting substrata.

According to one embodiment, a suitable substratum is contacted with an organosilicon halide, the temperature of the substratum being sufficiently high to cause decomposition of the organosilicon halide, to thereby deposit silicon carbide on the surface thereof. Thus, according to a preferred embodiment of the invention, a suitable substratum in contact with or prior to contact with an organosilicon halide, is heated to a temperature above the decomposition temperature of the organosilicon halide, i.e., above about 500°C. and preferably above about 800°C., but below the melting point of the substratum, to thereby cause on contact decomposition of the organosilicon halide and simultaneous deposition of silicon carbide on the substratum.

FIG. 1 is a schematic illustration of a suitable bath reactor for carrying out one embodiment of the invention;

FIG. 2 is a schematic illustration of a suitable inclined reactor for carrying out a continuous coating procedure in accordance with another embodiment of the invention;

FIG. 3 shows a schematic illustration of a vertical embodiment of an apparatus which may be used to alleviate the hot spot problem of the FIG. 2 apparatus; and

FIG. 4 shows a schematic illustration of an apparatus suitable for coating glass fibers directly after their formation.

FIG. 1 is a schematic illustration of a suitable bath reactor for carrying out such an embodiment of the invention. A suitable reaction vessel 2, which may for example be made of glass, is suitably sealed with head plate 4 by means of gasket 6. The reaction vessel is filled with a liquid organosilicon halide 8 to the desired level. The head 4 is provided with an exhaust 10 for venting gases released by the decomposition of the organosilicon halide. The reaction vessel 2 is also provided with insulated electrodes 12 and 16 and is submerged in the liquid reactant material 8. The hinged lower grip 20 provides tension to keep the wire 18 straight. The exhaust opening 10 prevents pressure build-up in the reaction vessel and is vented to atmosphere. When the substrata is conducting, voltage, either A-C or D-C, is applied to the electrodes 12 and 16 to heat the substrate above the decomposition temperature of the organosilicon halide. Alternatively, when the substrata is nonconducting, a suitable auxiliary pre-heating device, such as an inductance coil, may be used to heat the substratum to the required temperature. In operation with a conducting substratum, the apparatus of FIG. 1 is filled with a liquid organosilicon compound, for example, methylene dichloride silane ($\text{CH}_2\text{SiHCl}_2$). As sufficient voltage is applied to the wire to heat it to a temperature high enough to cause the liquid reactant to decompose, silicon carbide will deposit as a solid on the hot substrate while gaseous hydrogen chloride will bubble off through port 10. At the end of the run, argon or another inert gas may be bubbled through the hollow electrode 16 to purge the gaseous reaction products from the liquid reactant.

The process is adaptable for coating a wide variety of substrate material, as will be clear from the following examples,

EXAMPLE 1

Using the apparatus of FIG. 1, a ½ mil tungsten substrate was coated with silicon carbide. The tungsten filament was attached to electrodes 12 and 16 as shown in FIG. 1 and heated by resistance heater to a temperature of between 900° and 1,500°C. The liquid reactant material was methylene dichloride silane. The plating times and resulting thickness of the deposit were as follows:

½ Mil Tungsten Substrate		Final Diameter of Wire in Inches
Coating Time		
5	Seconds	.005
16	do.	.018
20	do.	.022
22	do.	.025

X-ray analysis of the silicon carbide coated tungsten wire of Example 1 showed the deposit to be beta silicon carbide, with undetectable amounts of anything else present.

EXAMPLE 2

Example 1 was repeated except that a 1.8 mil tantalum wire was used as the substratum. Plating time and resulting thicknesses of deposit were as follows:

1.8 Mil Tantalum Substrate		Final Diameter of Wire in Inches
Coating Time		
20	Seconds	.026
40	do.	.041

EXAMPLE 3

Example 1 was repeated except that an 18 mil stainless steel wire was used as a substratum. Plating times and resulting thicknesses of deposit were as follows:

18 Mil Stainless Substrate		Final Diameter of Wire in Inches
Coating Time		
3	Seconds	.020
5	do.	.024

EXAMPLE 4

A 2 mm glass rod was heated to a dull red temperature and plunged into liquid methyl dichlorosilane. A 1 mil thick coating of silicon carbide was formed on the glass rod.

Although the procedure using the apparatus of FIG. 1 has been described with reference to the coating of substratum in filament form, it should be understood that using the apparatus described, a silicon carbide coating may be imposed on a suitable substratum of any desired shape to provide protection against wear, oxidation or erosion.

Heating of the object to be coated may be accomplished by self-resistance heating of a substratum in contact with the liquid reactant, or by induction or other suitable pre-heat means, followed by exposure to the liquid reactant. Self-resistance heating is most suitable for wires or ribbons. Large objects could be heated for example by induction coils placed outside the reactor. When large pieces or smaller objects, e.g., wire, are to be coated continuously, it is desirable to circulate the reactant liquid to a cooling chamber, in order to avoid over heating of the reactant liquid.

In any event, when the reactant is liquid, care should be used to maintain the bulk of the liquid below its boiling point to avoid excessive bubbling. It is recognized, however, that at the preferred temperatures, the liquid immediately surrounding the immersed object will boil vigorously and film boiling will be established.

The procedure of the embodiment described has many advantages over conventional pyrolytic processes. Thus, as compared with such conventional techniques, the present procedure requires no metering of any kind of gas and is therefore susceptible of good control. Since extremely high concentrations of reactant material are possible, i.e., a pure or substantially pure organosilicon halide may be used as the reactant fluid, a rapid rate of deposition is assured. Reaction by-products are removed immediately as they form and therefore do not have an opportunity to foul up the system. A further advantage is the fact that stoichiometry is achieved without any metering or control being necessary, and a uniform distribution of reactant material is therefore quite easy to obtain. Thus, parts of any shape can be coated uniformly.

The invention may be used to coat a wide variety of objects, including engine components having improved high temperature capability, e.g., improved oxidation and erosion resistance. Parts subject to severe wear may also be coated in accordance with the present invention to provide high hardness even at elevated temperatures.

The invention is particularly suitable for protecting tungsten, columbium, and tantalum components from decomposition due to oxidizing atmospheres. When applied to the coating of glass, the mechanical properties of the glass are greatly improved.

FIG. 2 is a schematic illustration of a suitable inclined reactor for carrying out a continuous coating procedure in accordance with a further embodiment of the invention. A suitable reaction vessel 50, which may, for example, be made of glass, is suitably sealed with head plates 54 and 56, by means of gaskets 55. The reactor is filled with a liquid organosilicon halide 70. The head 55 is provided with an exhaust 66 for venting reaction gases. The head plates 54 and 56 are also provided with insulated electrodes 60 and 64. Electrodes 60 and 64 are preferably hollow and may be filled with a liquid, e.g., mercury, for sealing and/or electrical contact purposes. The substrate to be coated is a continuous wire 62 which passes through electrode 60, the liquid reactant 70 and electrode 64 from which it emerges as a silicon carbide coated wire 65. The apparatus of FIG. 2 is especially suitable for coating substrates in the form of wires which are electrically conducting, since such wires can readily be heated by self-resistance. Appropriate feed and take-up reels or other devices may be used to feed and take-up the filament substrate 62 and the coated wire 65, respectively. When the substrate is conducting, voltage, either A-C or D-C, may be applied to the electrodes 60 and 64 to heat the substrate. Alternatively, when the substrate is non-conducting, a suitable pre-heater may be used to heat the substrate prior to passage through hollow electrode 60 which, in the case of non-conducting substrates, would function merely as a seal and guide for the wire. In operation with a conducting substratum, the apparatus of FIG. 2 is filled with a liquid reactant of the type described, for example, methyl dichlorosilane ($\text{CH}_3\text{SiHCl}_2$). As sufficient voltage is applied to the wire to heat it to a temperature high enough to cause the liquid reactant to decompose, silicon carbide will deposit as a solid on the hot substrate while gaseous hydrogen chloride will bubble off through port 66.

EXAMPLE 5

The run of this example was made in the inclined reactor shown in FIG. 2. The reactor was first purged with oxygen to remove the air and water vapor and then filled with methyl dichlorosilane so that both electrodes (mercury filled) were submerged. The wire take-up was started and then power was applied and the wire heated to approximately 1,100°C. The hottest spot on the wire was at the entrance (upper end) as shown at 73. The reactor was 6 inches long, wire pulling rate was 200 feet per hour, with a current of 250 milliamps. One-half mil tungsten wire was the substrate and 2 mil silicon carbide coated filament was continuously produced. The temperature was limited by the hot spot because the gas that formed rose to the top and soon the upper electrode was surrounded by gas. This section got much hotter than the wire in the liquid

and the current was therefore limited to prevent burn-out in this section. The silicon carbide coated tungsten wire product tested 180,000 psi tensile.

In FIG. 3 is shown a vertical embodiment of an apparatus which may be used to alleviate the hot spot problem encountered with the FIG. 2 apparatus as explained in Example 5.

As shown in FIG. 3, 10 is a cylindrical reactor which may be provided with a suitable cooling jacket 8 having suitable coolant inlet and outlet ports 17 and 18, respectively. Reactor 10 is sealed by upper header assembly 19 and lower heating assembly 20, respectively. The header assemblies are sealed to reactor tube 10 by means of gaskets 15.

Upper header assembly 19 includes an upper mercury inlet tube 1 and an upper electrode and seal jewel assembly 2. Upper liquid or gas port 3 and upper gas exhaust port 5 extend horizontally into upper electrode and seal liquid and gas port assembly 4. Tube 6 is a continuation of upper mercury inlet tube 1 and extends into the chamber within reactor 10. Tube 6 is fitted at its lower end with a removable jewel holding cap 7 which serves to guide the wire 21 being coated. Lower header 20 is provided with a lower mercury electrode 11, and a lower mercury fill tube 12. Also provided is a gas or liquid inlet tube 13 and a removable lower jewel holder 14. The reactor 10 is filled to a suitable level with liquid reactant material 22.

The operation of the FIG. 3 apparatus will be clear from the following examples:

EXAMPLE 6

The trouble with the hot spot which developed in the vertical apparatus of FIG. 2 as described in Example 5 was eliminated by using the vertical apparatus of FIG. 3, in which the wire feed-in was at the bottom, thus keeping the hot spot under the liquid at all times. In FIG. 3, the gas port 5 was used to exhaust the produced gas, both HCl, H₂, etc., as well as vaporized silane. When the water jacket 8 was used (0°C. water circulated in the jacket), the decrease in liquid level was very slow. Additional silane was added through inlet tube 13 from a reservoir and valve system. The methyl dichlorosilane was also directly cooled by circulating it by means of ports 3 and 13 through a copper coil immersed in an ice bath. A small circulatory pump was used for this purpose.

In a 6-inch long reactor tube 10 at 1,125 feet per hour, 1.4 mil silicon carbide filament was produced on a 0.5 mil tungsten substrate with a current of 280 milliamps. The coated material had a tensile strength of 331,000 psi.

EXAMPLE 7

In another modification of processing procedure, the vertical reactor of FIG. 3 was run in a combined liquid-gas phase operation. That is to say the liquid methyl dichlorosilane level was kept somewhere below the top jewel 7. This procedure essentially produced a two-stage reactor with a coating being put on in the liquid and another put on in the fully saturated gas above the liquid. The temperature was controlled by adjusting the liquid level and gas input to the top section. At half liquid, half gas, the wire was hotter in the gas section but due to the small amount of SiC deposited from the liquid section, the hot spot problem was eliminated and a very uniform elevated temperature of about 1,500°C.

was maintained in the upper section. In such a combined reactor the exhaust was through port 5 and a stream of H₂ was added at port 3 to keep the jewel 7 clean and cool. Starting with one-half mil tungsten wire, a SiC coated filament 4.2 mils in diameter was produced in a 10 inch long reactor filled with about 4 inches of liquid methyl dichlorosilane at a rate of 103 feet per hour. This filament had a tensile strength of 250,000 to 350,000 psi. At 206 feet per hour a 3.35—3.40 mil SiC coated tungsten fibre was produced that had a tensile strength of 312,000 – 345,000 psi.

The apparatus of FIGS. 1–3 could readily be adopted for plating glass filament. Since glass is not electrically conducting, it cannot be heated by self-resistance. Accordingly, a suitable pre-heater, such as a resistance, would be required to heat the glass to a temperature to cause decomposition of the liquid reactant upon contact.

The silicon carbide coated filaments of the type described could be used to satisfy the current need for lightweight, high strength and high modulus fibres for use in wound structures as well as in fibre reinforced structures.

The use of extremely fine glass as a substrate in the practice of this invention results in a thin filament, e.g., one having a total diameter between one or two mils with properties far superior to those of the uncoated glass. Glass is especially useful because it is possible to make extremely fine filaments of this material, which intrinsically has a low density. Fine diameter fibres of silicon coated glass filaments produced in accordance with this invention are easy to handle in wrapping or winding applications, and have extremely high strength and modulus to weight ratios. Especially suitable as a substratum for the preparation of fine silicon carbide coated glass filaments in accordance with this invention is "E" glass yarn. The glass should be pre-heated well above the decomposition temperature of liquid reactants described herein so as to make it possible to maintain it for a sufficient period of time at decomposition temperature in the deposition chamber.

This technique can be extended to provide a high speed, efficient system for coating glass fibre or yarn with silicon carbide. Thus, instead of providing a separate furnace for preheating the already formed glass, the deposition chamber may be placed below the exit orifice of a glass filament producing furnace or spinnerette. The glass emerging from either the furnace or spinnerette is thereby already hot enough for deposition to take place. Such a procedure greatly simplifies the entire system and allows the silicon carbide to deposit as part of the glass forming process. In the case of yarn, placing the furnace below the spinnerette but in front of any gathering device, allows for coating of the filaments before gathering, thereby insuring complete and uniform coating of each filament in the yarn.

An apparatus suitable for carrying out such an embodiment of the invention is shown in FIG. 4. In FIG. 4, 100 represents a suitable spinnerette into which is fed molten glass 101 at temperature and pressure conditions well known to the art. The hot glass filaments 102 emerge from the spinnerette directly into reaction chamber 104 which is filled to a suitable level with a silicon carbide source compound 106 of the type described herein. A suitable heat exchanger mechanism 108 surrounds the chamber 104 to keep the reactant liquid at desirable temperature. The filaments 102 pass

through the fluid reactant 106 and are coated with a silicon carbide in accordance with the teachings contained hereinabove. If desired, a suitable gathering device 109 may be provided to gather the glass filaments 102 into a yarn. The yarn gatherer 109 is provided with a suitable sealing device 110, e.g., a mercury seal, to prevent loss of source compound 106 from the reaction chamber. The gathered glass yarn 112 may be stored on a suitable storage device, for example, reels. If desired, the source compound used with the apparatus of FIG. 3 could be gas, although it should be understood that in the preferred embodiment, liquid source compound would be used. In the embodiment wherein gas is used as the reactant, the heat exchanger 108 would be utilized to maintain the reactant in a gaseous state and to prevent loss of heat from filaments 102.

It should be pointed out that the coating rates of this invention are very high when liquid source compound is used, so that even though the substratum, e.g., glass filament, is rapidly cooled below the deposition temperature upon contact with the liquid source compound, it will be coated with a thin layer of silicon carbide.

The silicon carbide source compound for use herein may be any suitable organosilicon halide corresponding to the formulae:



wherein R is an alkyl, aryl or alkyl aryl radical, and X is halogen, i.e., chloride, bromide, fluoride or iodide, including mixtures of such compounds.

Preferred for use are the lower alkyl (i.e., less than 8 carbon atoms) dichloro and trichloro silanes, such as methyl dichlorosilane, methyl trichlorosilane, ethyl dichlorosilane, ethyl trichlorosilane, and the like. Also may be mentioned phenyl trichlorosilane and phenyl dichlorosilane. When the lower alkyl silicon halides referred to are employed, the surface in contact therewith will be heated to between about 500° and 1,500°C., preferably between about 800° and 1,500°C.

The reaction chambers suitable for use in carrying out the processes of the invention can be constructed entirely of glass or quartz, or metal, or partly of each of these materials. Ordinarily, the reaction will be carried out under atmospheric pressure, although operation under pressure is also contemplated.

The invention is particularly suitable for imposing a thin, adherent, non-porous oxidation and corrosion resistant silicon carbide coating on base metals such as columbium, tantalum and tungsten, which are vulnerable to oxidation at high temperature and which will fail rapidly if not protected by a suitable coating. Such metals or alloys thereof, and particular alloys containing one or more of the foregoing, are useful as vanes for gas turbines, especially jet aircraft engines. Other substrates which may be advantageously coated with silicon carbide in accordance with this invention are those which have a melting point exceeding the formation temperature of silicon carbide and include molybdenum, rhenium, silicon, carbon, glass, alumina, graphite and the like.

It should be understood, however, that using the teachings contained herein it is possible to coat liquid substrata, e.g., liquid drops or droplets or globules, by contacting them with the liquid organosilanes described. In FIG. 4, outlets 117 and 118 may be used to

feed or circulate reactant 106 to the reaction chamber 104.

The invention in its broader aspects is not limited to the specific details shown and described, but departures may be made from such details within the scope of the accompanying claims without departing from the process of the invention and without sacrificing its chief advantages.

What is claimed is:

1. A method for depositing a thin, adherent oxidation and corrosion resistant silicon carbide coating on a filamentary base material to produce a structural composite filament which comprises:

selecting a filamentary base material which has a melting point which exceeds the formation temperature of silicon carbide; and

continuously passing a hot surface of the filamentary base material through liquid organosilicon halide, the temperature of said hot surface being sufficiently high to cause the organosilicon halide immediately surrounding said hot surface to film boil, decompose, and deposit on said filamentary base material an adherent coating of oxidation and corrosion resistant silicon carbide.

2. The method of claim 1 wherein the base material is an electrical conducting material which is heated by electrical resistance heating above the decomposition temperature of the organosilicon halide while in contact with the liquid organosilicon halide.

3. The method of claim 1 wherein the base material is an electrical insulating material, which is pre-heated to a temperature above the decomposition temperature of the organosilicon halide prior to contact therewith.

4. The method of claim 1 wherein the organosilicon halide is a lower alkyl silicon chloride.

5. The method of claim 4 wherein the temperature of the base material in contact with the liquid organosilicon halide is between about 800° and 1,500°C., and wherein the organosilicon halide is a member selected from the group consisting of methyl dichlorosilane, methyl trichlorosilane, ethyldichlorosilane and ethyl trichlorosilane.

6. The method of Claim 1 wherein the base material is a member selected from the group consisting of tungsten, tantalum, columbium, alloys containing one of the foregoing, and alloys of the foregoing.

7. The method of Claim 1 wherein the base material is an insulating material selected from the group consisting of glass and alumina.

8. The method of Claim 1 wherein the base material is a glass filament having a diameter of less than about 2 mils.

9. A method of depositing silicon carbide on a wire comprising the steps of immersing a wire in an organic liquid chemically including silicon and carbon, heating said wire until said liquid boils vigorously and film boiling is achieved, and maintaining said film boiling for a period of time thereafter, said wire being an electrically conductive wire and having a melting point such that said film boiling can be maintained for a period of time sufficient to deposit a desired coating of said silicon carbide on said wire, said organic liquid containing a compound selected from the group consisting of methyl trichlorosilane, methyl dichlorosilane, ethyl trichlorosilane and ethyl dichlorosilane.

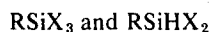
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10. The method of claim 9 wherein said heating is effected by passing an electrical current through said wire.

11. The method of claim 10 wherein said wire is metal, metal alloy or graphite.

12. The method of claim 11 wherein said wire is tungsten.

13. A method of depositing silicon carbide on a wire comprising the steps of immersing a wire in an organic liquid chemically including silicon and carbon, heating said wire until said liquid boils vigorously and film boiling is achieved, and maintaining said film boiling for a period of time thereafter, said wire being an electrically conductive wire and having a melting point such that film boiling can be maintained for a period of time sufficient to deposit a desired coating of said silicon carbide on said wire, said organic liquid containing a compound selected from the group consisting of organosilicon halides defined by the formulae



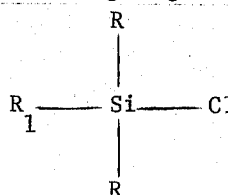
wherein R is a lower alkyl radical and X is chlorine.

14. The method of coating an object with silicon carbide which consists of:

heating said object to at least 1,150°C; and then quenching said object in a liquid bath consisting essentially of one or more members chosen from the

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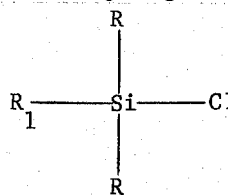
group of silanes having the general formula



wherein each R is a member of the group consisting of chlorine and hydrogen and R₁ is a member of the group consisting of methyl, ethyl and propyl groups.

15. The method of coating an object with silicon carbide which comprises:

heating said object to at least 1,150°C; and then immersing said object in a liquid bath consisting essentially of one or more members chosen from the group of silanes having the general formula



wherein each R is a member of the group consisting of chlorine and hydrogen and R₁ is a member of the group consisting of methyl, ethyl and propyl groups.

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