

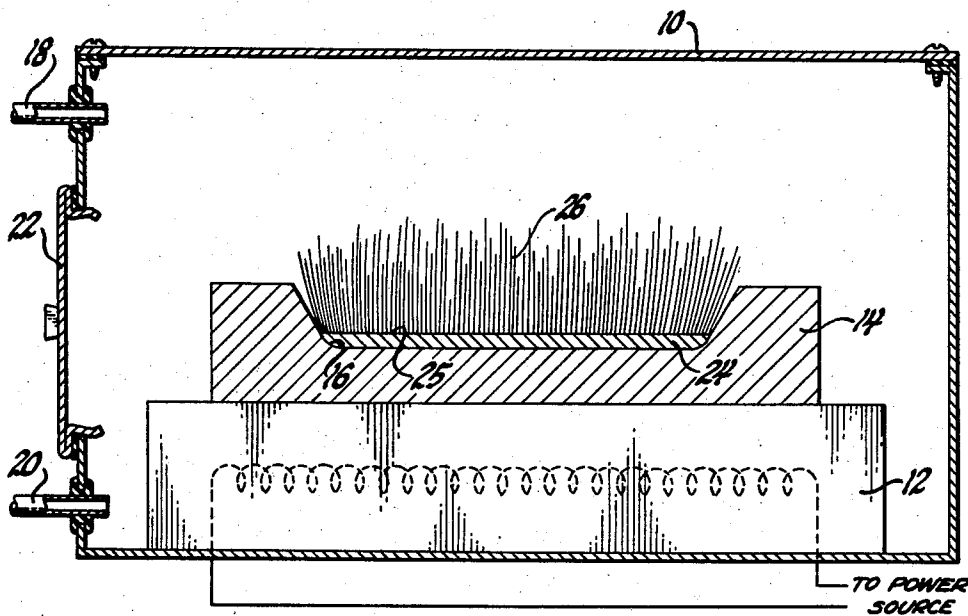
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METHOD OF GROWING COPPER FILAMENTS

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METHOD OF GROWING COPPER FILAMENTS
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This invention relates to a method of preparing filaments and more particularly to a process by which filaments of copper may be prepared.

It is known that fine filaments—commonly called whiskers—have been demonstrated to have the highest strengths known in materials and to have other desirable properties which make them useful as structural materials. The values of mechanical properties such as tensile strength, elastic limit and the like of known metals such as copper are much lower than their theoretical properties. It has been demonstrated that the fine crystal filaments or whiskers in some instances are capable of approaching these theoretical limits. The copper filaments of the present invention have particular application in the manufacture of cables and the like.

It is the basic object of the present invention to provide a method of producing fine copper filaments. It is a further object of this invention to provide a method for making copper filaments which consists essentially of heating copper metal in contact with a layer of lead alloy selected from the class consisting of lead-sulfur alloys, lead-tellurium alloys and lead-selenium alloys, in an inert environment such as helium or a vacuum at a temperature sufficient to soften or melt the surface of the lead alloy adjacent the copper and under conditions such that a temperature differential is established between the copper surface and the lead alloy surface exposed to the environment.

More specifically, the method of growing copper filaments of the present invention consists of depositing a layer of a lead alloy taken from the group consisting of lead-sulfur alloys, lead-tellurium alloys and lead-selenium alloys, on a surface of a copper metal member, heating the copper member and the adjacent lead alloy in a vacuum or an inert environment to a temperature such that the lead alloy adjacent the copper surface becomes molten or in a plastic state and under conditions such that a temperature differential is established between the copper surface supporting the lead alloy and the lead alloy surface exposed to the surrounding environment. Preferably the copper element is heated to temperatures within the range of about 500° F. to about 1500° F. It has been found that under these conditions, copper filaments will grow from the lead alloy surface to virtually any length singly and in bundles and in diameters of about .025 millimeters and smaller.

These and other objects of the invention will be more apparent from the following detailed description of the invention made with reference to the accompanying drawing which is a schematic illustration of apparatus by which the process of the present invention may be carried out.

Referring to the drawing, the apparatus consists of a gas-tight chamber 10 having disposed therein an electrical heater 12. Positioned on the heater 12 is a copper vessel or container 14 of substantial thickness having a relatively shallow and flat depression 16 therein. Conduit means 18 and 20 are provided for either evacuating the vessel 10 or filling it with an inert gas in an obvious manner. An access opening 22 is provided whereby the copper vessel 14 may be inserted and removed from the chamber 10. Means (not shown) is provided for

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supplying electrical energy to the electrical resistance heating element 12.

A specific and preferred embodiment of the method of the present invention and its mode of operation is as follows. A relatively thin layer 24, preferably about 3 millimeters in thickness, of lead sulfide (PbS), is placed in the depression 16 of the copper vessel 14. The chamber 10 is then evacuated. The copper vessel 14 is then heated to a temperature of about 1200° F. by means of the heating element 12. As a result, the lead sulfide layer 24 adjacent the copper of the depression 16 becomes plastic and molten while the surface 25 of the lead-sulfur alloy layer will have a temperature of approximately 700° F. whereby a temperature differential of about 500° F. is established between the surface 25 of the lead sulfide and the surface 16 of the copper vessel. Under these conditions copper filaments or whiskers 26, about 0.02 millimeter in diameter, will grow from the lead sulfide surface 25 at a rate of about 3 millimeters per minute.

In the practice of the present invention the lead alloy layer 24 may be taken from the group consisting of lead-sulfur alloys, lead-tellurium alloys and lead-selenium alloys. Of these alloys, it is preferred to employ lead-sulfur alloys ranging from an alloy consisting of about 0.04% by weight sulfur and the balance substantially lead. It is preferred, however, to employ the molecular compound lead sulfide. When lead-tellurium and lead-selenium alloys are used, the molecular compounds lead telluride (PbTe) and lead selenide (PbSe) are preferably used although lead-selenium and lead-tellurium alloys containing as little as about 0.04% by weight tellurium and about 0.04% by weight selenium, respectively, will produce a growth of copper filaments.

Although the lead alloy layer 24 is preferably of a thickness in the order of 2 to 4 millimeters, the thickness is not particularly critical since filaments may be grown from a lead alloy surface of the type described having a thickness greater and smaller than 2 to 4 millimeters. Best results are obtained, however, when the lead alloy layer is between 2 to 4 millimeters. Excellent results have been obtained where the copper retaining member 14 is in the form of a copper strip having a lead sulfide layer of about 2 millimeters thick. Excellent results have also been obtained where the retaining member 14 is in the form of a relatively thick cup as shown in the drawing and a lead sulfide layer of 3 to 4 millimeters is used.

In the practice of the process of the present invention, the copper retaining member 14 may be heated in a vacuum as described above or in an inert atmosphere such as helium. The term "inert environment" as used herein is intended to refer to both a vacuum or an inert gas such as helium. Although it is preferred to heat the copper member 14 to a temperature of approximately 1200° F. as described above in connection with utilizing a lead sulfide layer 24, it has been found that temperatures between 500° F. and 1500° F. may be utilized provided that a temperature gradient exists between the copper surface 16 and the lead alloy layer 25. In general the rate of growth increases as the temperature gradient is increased. To this end, the vessel 10 is constructed of a material which has sufficient heat transfer capacity to establish a suitable differential. In the instant illustration, a container 10 formed of a ferrous sheet metal is found to operate satisfactorily to establish a desired temperature differential.

Although theoretically an infinitesimal temperature gradient will produce copper filament growth, a temperature gradient of at least about 25° F. is necessary to produce copper filament growth at a reasonable rate and a temperature differential of about 400° F. to 500° F. is preferred.

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While the present invention has been described by means of certain specific examples, it will be understood that the scope of the invention is not to be limited thereby except as defined in the following claims.

I claim:

1. A method for producing copper filaments comprising the steps of placing a layer of copper having a surface in contact with a lead alloy layer having opposed first and second surfaces whereby said copper surface is in contact with said first lead alloy surface, said lead alloy layer being selected from the group consisting of lead-sulfur alloys, lead-tellurium alloys and lead-selenium alloys, heating said copper and lead alloy layers in an inert environment to a temperature below the melting point of copper and sufficient to melt the said first lead alloy surface and to establish a temperature differential between the said copper layer surface and said second lead alloy surface which is exposed to said environment.

2. A method for producing copper filaments comprising the steps of placing a layer of copper having a surface in contact with a lead alloy layer having opposed first and second surfaces whereby said copper surface is in contact with said first lead alloy surface, said lead alloy being selected from the group consisting of lead-sulfur alloys, lead-tellurium alloys and lead-selenium alloys, heating said copper and lead alloy layers in an inert atmosphere within a temperature range from about 500° F. to 1500° F., said heating being performed under heat transfer conditions such that a temperature gradient is established between said copper surface and the said second lead alloy surface which is exposed to said environment.

3. A method for producing copper filaments comprising depositing a layer of a lead-sulfur alloy having opposed first and second surfaces and containing about 0.04% to about 7.7% sulfur by weight and the balance essentially lead on a surface of a copper metal layer whereby said first lead alloy surface is in contact with said copper surface, and heating said copper layer to a temperature in the range of from about 500° F. to 1500° F. in an inert atmosphere, said heating being performed under heat transfer conditions such that a temperature differential is established between said copper surface and the second lead-sulfur alloy surface exposed to said atmosphere.

4. A method for producing filaments of copper comprising the steps of applying a layer of lead sulfide of a thickness of about 2 to 4 millimeters having first and second opposed surfaces to a surface of a copper layer whereby said copper surface is in contact with said first lead sulfide surface, and heating said copper layer and said lead sulfide layer to a temperature between 500° F. and 1500° F. while surrounded by an inert environment, said heating being accomplished under conditions such that a temperature gradient exists between said copper surface and the said second lead sulfide surface exposed to said environment.

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5. A method for producing filaments of copper comprising the steps of applying a lead sulfide layer of a thickness of about 2 to 4 millimeters having first and second opposed surfaces onto the surface of a copper plate whereby said first lead sulfide layer is in contact with said copper surface, surrounding the said copper surface and lead sulfide layer with an inert environment, and heating the copper plate to a temperature of about 1200° F., said heating being accomplished under conditions such that a temperature gradient of about 400° F. to 500° F. exists between the said surface of said copper plate surface and the second surface of said lead sulfide.

6. A method for producing filaments of copper comprising the steps of applying a layer of a lead base alloy about 2 to 4 millimeters in thickness having first and second opposed surfaces to a surface of a copper layer whereby said copper surface is in contact with said first lead base alloy surface, said lead base alloy selected from the group consisting of lead-sulfur alloys containing at least 0.04% by weight sulfur, lead-tellurium alloys containing at least 0.04% by weight tellurium and lead-selenium alloys containing at least 0.04% by weight selenium on the surface of a copper plate, surrounding the copper surface and lead base alloy layer with an inert environment, and heating the copper layer to a temperature between about 500° F. and 1500° F., said heating being accomplished under conditions such that a temperature gradient of from about 400° F. to 500° F. exists between said surface of said copper layer and the said second surface of said lead alloy layer.

7. A method for producing filaments of copper comprising the steps of applying a layer having opposed first and second surfaces of a lead base alloy selected from the group consisting of lead-sulfur alloys, lead-tellurium alloys and lead-selenium alloys on the surface of a copper layer whereby said first lead alloy surface is in contact with said surface of said copper layer, surrounding the copper surface and the said lead base alloy layer with an inert environment, and heating the copper layer to a temperature between 500° F. and 1500° F., said heating being accomplished under conditions such that a temperature gradient of about 400° F. to 500° F. exists between the said surface of said copper layer and the said second surface of said lead alloy.

References Cited in the file of this patent

- Handbook of Chemistry and Physics, 28th edition, 1944, Chemical Rubber Publishing Co., Cleveland, Ohio, pp. 402-403.
- Hofman: Metallurgy of Lead, McGraw-Hill Book Co., New York City, N.Y., 1918, pp. 44-45.
- Progress in Metal Physics, vol. 6, Pergamon Press Ltd., London, 1956, p. 47.
- Bell Telephone System Technical Publications, New York, 1957, Monograph 2635, Growth and Properties of Metal Whiskers, p. 3.