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2,746,129

METHOD OF PRODUCING A TAUT THIN MEMBER

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FIG. 1

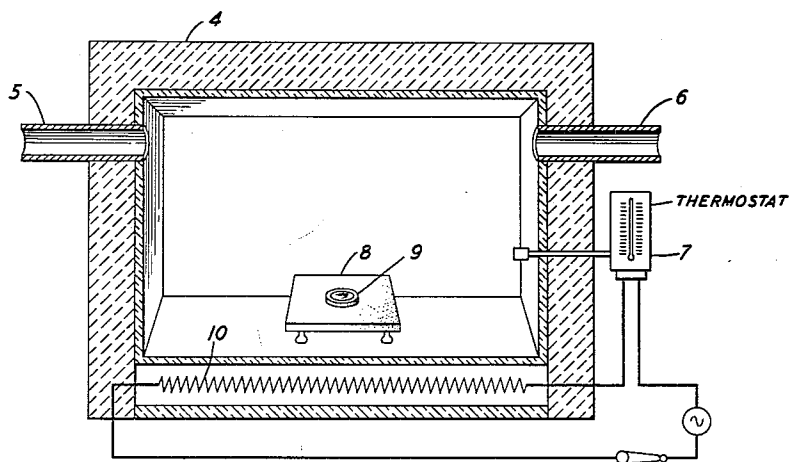
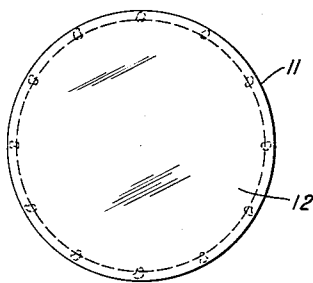


FIG. 2



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METHOD OF PRODUCING A TAUT THIN MEMBER 5

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4 Claims. (Cl. 29—155.5)

This invention relates generally to thin metal foils and, more specifically, to a method for making thin, taut metal foils particularly suitable for use in thermally sensitive conductive devices.

Recent developments in certain applications of infrared devices have brought out the necessity for thermally sensitive resistor elements which in some cases might be as large as one or more square inches in area and as thin as a micron (10^{-4} cm.). They must also be flat and uniform. Requirements of this sort have not been met by thermally sensitive conductive foils fabricated by the standard sinter powder method or by any other method that is used at present. This invention provides a method for producing micron thin, taut foils which can be made into flakes having high negative coefficients of resistance.

The method in accordance with this invention comprises the steps of depositing, as by electroplating or evaporating, a metal, such as nickel, upon another metal, such as stainless steel. The thin foil of electroplated nickel may then be removed from the stainless steel and welded on a frame having an area up to several square inches. This assembly is then inserted in a furnace and given a heat treatment which draws the foil taut by eliminating porosity and altering the atomic structure of the foil so as to produce a uniformly flat metal film which can later be oxidized to form a thermally sensitive conductive device.

One of the objects of this invention is to produce a uniformly flat metal foil which can be oxidized and used as a thermally sensitive conductive device.

Another object is to enable the production of metal foils having an area up to several square inches and a thickness range from a fraction of a micron to several microns.

A further object is to facilitate the forming of flat metal foils which are very frail.

Still another object is the improvement of methods for making thin metal foils generally.

A further object of the invention is to produce a uniformly flat metal oxide foil having a negative temperature coefficient of resistance.

A feature of this invention involves the phenomena that when the foil which has been spot welded onto a frame is placed in a furnace at a given temperature, the structure of the foil will be atomically changed so that it will grow taut and have a uniform thickness.

The above-mentioned and other objects and features will be more fully understood from the drawings and the following description.

In the drawing:

Fig. 1 illustrates a typical furnace which can be used to carry out the process; and

Fig. 2 illustrates a typical foil and frame.

In Fig. 1, the furnace 4 has an evacuator lead 5, an input lead 6, a thermostat 7, and a heater element 10. A reduction or oxidation atmosphere may be piped into the furnace through lead 6. The frame-held foil 9 is

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placed in the furnace on plate 8 at a time and in a manner to be described later.

In Fig. 2, a circular frame 11 is shown holding a foil 12. Any shape frame upon which it is possible to secure a foil may be used, such as a square frame, a rectangular frame, a rhombic frame, etc. Three dimensional frames may also be used if it is desired to produce taut foils having three dimensions (disregarding thickness as a dimension). It has been found advantageous to perforate the foil adjacent to where it is fastened to the frame to facilitate removal of the foil from the frame upon completion of the oxidation process and while the frame and foil are still heated, thus mitigating the chances of the foil cracking upon cooling.

Speaking generally, the procedure for making these foils is to:

(1) Obtain a foil of the thickness desired by any known method such as electroplating, evaporation, or rolling process. Foils may be produced of uniform thicknesses of from a fraction of a micron and thicker by the proper choice of the aforementioned methods;

(2) Weld this foil to a frame;

(3) Rapidly heat the assembly in a reducing atmosphere furnace 4 to a proper minimum temperature and maintain this temperature until the foil becomes flat.

Steps 1 and 2 above, namely the production of the foil and the welding onto the frame, need no further explanation since they are old in the art. Step 3 will later be discussed in more detail.

Since nickel oxide has advantageous properties for use in certain large area unbacked bolometer applications, the foil material selected herein for discussion is metallic nickel, the metallic form being chosen to permit the welding of the foil to the frame. Some of the advantageous electrical properties of NiO are that it has a negative temperature coefficient of resistance, a specific resistance of about 10^9 ohm centimeters at 25° C. with about a six to eight per cent variation in resistance per degree centigrade. These characteristics make NiO well suited to designs where the current flows thickness wise through the bolometer. It is to be noted, however, that this process of producing taut metal foils is applicable to metals other than nickel. For example, all of the iron group can be formed into taut, thin foils by the process. In the case of metals other than nickel though, it is necessary to heat the metal rapidly to temperature ranges different from those given in the present discussion for nickel, depending upon what other metal is being used. Furthermore, the length of time a particular metal foil must be held at a certain temperature will also vary.

In the case of nickel, a reducing atmosphere furnace 4 is heated, as by a resistance heater 10, to a temperature in the approximate range of 950° C. to 1000° C. When this temperature is attained, the frame-held nickel foil 9 is placed therein. The foil must be left in the furnace from thirty minutes to twenty-four hours approximately. During this period the metal foil 12 will grow taut in the frame. The reduction atmosphere in the furnace serves the dual purpose of removing any oxygen from the foil as well as insuring that no additional oxygen is allowed to combine with the metal. Removal of oxygen from the foil causes a porous condition which results in greater contraction of the foil during the heating process.

The choice of material selected for the frame is made on the basis of the amount of tension desired in the heat-treated specimen. A frame material 11 of lower thermal expansion than the foil 12 will place the foil in tension because it contracts less upon cooling from the heat treatment temperature. When the frame 11 and film 12 are of the same material, the specimen becomes flat without any appreciable tension. This is a

property unique to this process. Therefore, if the only tension requirement for a bolometer specimen is that the surface be flat, and since nickel would produce no alloying problems with a nickel oxide flake, the material selected for the hollow frame can be nickel when the thermistor flake is to be of nickel oxide, although other metals can be used as frame material which will produce comparable results.

The requirement allowing the foil to stay in the reducing atmosphere furnace 4 at a temperature between 950° C. to 1000° C. for a period of time greater than thirty minutes allows any transient phenomena to be substantially completed since the thirty minutes heat treatment time is long compared with the heating interval for the sample, thus lessening the chance of undue stresses or strains being set up in the foil. At the end of the heating interval of time, the foil is allowed to slowly cool within the furnace to room temperature.

It has been found that some specimens of metal foil will not grow taut when subjected to the heat treatment discussed above. This is due to a lack of porosity in the foil and can be cured by subjecting the foil to a preliminary heat treatment, involving the oxidation of the surfaces of the foil. Then when the foil is heat treated in a reducing atmosphere, the oxygen will be removed from the surface of the foil 12 leaving a porous condition which will cause the foil to contract and grow taut. The procedure used to produce this surface oxidation of a one micron thick foil for example is to place the foil in an air atmosphere furnace at a temperature of approximately 850° C. for a period of ten to twenty seconds. This is insufficient time to permit the oxidation process to penetrate to the center of the foil but will allow oxidation of the surface to a depth of from ten to twenty per cent of the thickness of the foil. This oxidizing process produces a porous film by a mechanism wherein atoms are drawn out of the crystal structure of the material of the foil by oxygen on its surface leaving vacant lattice sites. An energy transformation takes place in this manner and energy is stored in the foil principally near the surface since the vacant lattice sites tend to migrate toward the surface. From a macroscopic standpoint this porosity or energy storage manifests itself as a surface tension. When the porous foil is heated, the atoms in the crystal structure become more mobile and the energy stored in the surface by virtue of the presence of the vacant lattice sites is released by the atoms flowing into the vacant sites to produce a contraction of the foil.

In the case of the nickel, it is to be noted that the frame-held foil 9 is placed in the furnace 4 at a time when the furnace has already attained a temperature of 950° C. to 1000° C. This insures an extremely rapid heating of the foil. If the foil is heated slowly, it has been found that it will not become taut.

The frame-held foil which is now taut and uniform can now be placed in an air atmosphere furnace at a temperature sufficiently high and for a long enough time to completely oxidize it. The temperature of the furnace and the length of time required to oxidize the foil is determined by the kind of foil material used.

In the preferred embodiment herein described, the frame-held nickel foil is placed in a cold oven which is then heated slowly enough so that the ring and the foil expand thermally at an equal rate. For a foil one micron thick welded to a frame ten mils thick, the furnace should be allowed to heat up to 450° C. in one hour. The oxidizing atmosphere is then forced into the furnace and the heating up of the furnace is continued at a rate of about 400° C. per hour until the furnace attains a temperature of 750° C. at which time the foil will be completely oxidized. A probe can then be used to sever the foil from the frame along the perforations.

It is to be noted that the invention has been described merely with reference to a preferred embodiment and that various changes may be made therein without departing from the spirit or scope of the invention.

What is claimed is:

1. The method of producing a taut, thin, metallic member comprising securing a member having a thickness of the order of a micron to a frame, oxidizing the surface portions only of said member, rapidly heating the oxidized member in a reducing atmosphere to reduce the oxidized surface portions of the member and draw the member taut, and then slowly cooling the frame-held member.

2. The method in accordance with claim 1 wherein the oxidation of the surface of said member extends into said surface a depth of 10 to 20 per cent of the thickness of said member.

3. The method of producing a taut, thin foil which comprises preparing a foil of a metal of the iron group having a thickness of about a micron, securing said foil to a frame, oxidizing the surface portions only of said member, then rapidly heating the oxidized, frame-held foil in a reducing atmosphere to reduce the oxidized surface portions and to draw the foil taut, and then slowly cooling the frame-held foil.

4. The method of producing a taut, nickel foil which comprises securing a nickel foil having a thickness of about one micron to a nickel frame, heating said frame-held foil in air to about 850° C. for about 10 to 20 seconds to oxidize the surface of the foil, rapidly heating the oxidized foil to about 950° C. to 1000° C. in a reducing atmosphere for from 30 minutes to 24 hours to reduce the oxidized surface portions and to draw the foil taut, and then slowly cooling the frame-held foil.

References Cited in the file of this patent

UNITED STATES PATENTS

1,275,022	Harrison	Aug. 6, 1918
1,747,847	Schlenker	Feb. 18, 1930
1,863,073	Smythe	June 14, 1932
2,162,808	Gallup	June 20, 1939
2,373,661	DePhillips	Apr. 17, 1945
2,431,113	Glyptis et al.	Nov. 18, 1947
2,449,872	Brasch et al.	Sept. 21, 1948
2,451,360	Skehan	Oct. 12, 1948
2,654,940	Law	Oct. 13, 1953