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PROCESS FOR MAKING A THERMISTOR BY OXIDATION
OF A NICKEL MANGANESE ALLOY
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FIG. 1

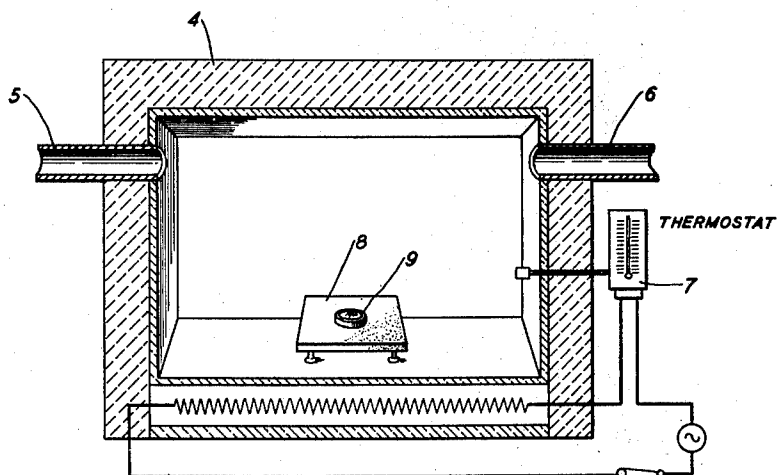
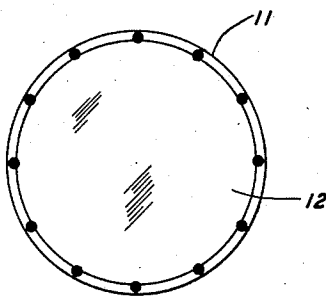


FIG. 2



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PROCESS FOR MAKING A THERMISTOR BY
OXIDATION OF A NICKEL MANGANESE
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5 Claims. (Cl. 252-490)

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This invention relates to thermistors generally and, more particularly, it relates to a method for the production of thin thermally sensitive conductive flakes.

It is to be noted that the term "thermistor" will be used in this specification to designate a thermally sensitive resistive element.

In the prior art, there are several methods for producing thermally sensitive conductive flakes. Copper oxide has been produced having a thickness of 25 microns and a resistance of from 10^9 ohm centimeters at 25° C. to 10^3 ohm centimeters, depending on the type oxide used. However, copper oxide and other single metal oxide thermistor materials do not have very stable electrical properties and consequently are unsuitable as articles of commerce. It is known also that certain bimetal oxides make better thermistors than do the single metal oxides. The electrical properties of the bimetal oxide thermistors are much more stable which makes them commercially useful. One of the principal present methods for manufacturing bimetal oxide thermistor elements comprises the sintering and pressing together into a thin foil the preformed oxides of the metals used. This method has been found not entirely satisfactory in that the surfaces of the thermistor foils are not uniformly smooth, which results in a degradation of electrical properties; also the thickness of the foils is in the order of several decades of microns. This order of thickness is undesirable in many cases where thermistor flakes are utilized.

It has also been attempted to produce bimetal thermistor elements by oxidizing an alloy of the two metals. Up to the present time, however, this has resulted in a division of the two component oxides into two separate pieces, which is, of course, unsatisfactory.

This invention pertains to a method for making bimetal oxide thermistor flakes of the proper crystalline structure having a smooth surface and having a thickness of, for example, from one-half micron to twenty-five microns.

One of the objects of this invention is to produce a thin thermistor flake having stable electrical properties.

Another object of this invention is the production of a thin thermistor flake which is relatively free from mechanical irregularities.

A further object is to produce thermistor flakes comprising two metal oxides, having a thickness less than twenty-five microns and a relatively large area.

Still another object is the improvement of

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methods for making thermistor flakes generally.

The invention comprises a method for making a bimetal thermistor material in thin sheets of from approximately one-half micron to about twenty-five microns thickness having a spinel type crystalline structure. For example, in one embodiment a rolled sample of 80-20 atomic per cent manganese-nickel of the order of one-third to eighteen microns thick is placed in an oxidizing atmosphere furnace at a temperature between 600° C. and 700° C. and the furnace is then allowed to cool to a temperature in the range from about 450° C. to about 550° C. in not less than sixteen hours to produce oxidation. The furnace is then readjusted to come up to a temperature of about 875° C. to 1150° C. in about two hours to produce the spinel type crystalline structure in the sample.

A feature of this invention lies in the smoothness of the surfaces of the thermistor flake. This is of particular moment in bolometer and similar devices involving translation of heat waves or of an electron beam which is used to scan the thermistor flake.

A further feature of the invention is the oxidation of the bimetal alloy while in the foil form.

The above-mentioned and other objects and features will be more fully understood from the drawing 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 held in a 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 atmosphere may be piped into the furnace 4 through lead 6. The frame-held foil 9 is placed in the furnace on tray 8 at a time and in a manner to be described later.

In Fig. 2 a circular metal frame 11 is shown holding a foil 12. It is to be understood that it is not necessary to use a frame to hold the foil but as one alternative, the foil can be treated without a support. In the instance where a frame is used, however, as when a very thin foil is used, the foil may be welded to the frame to hold the foil securely so that it will remain flat during the process. Any shape frame may be used.

In a preferred embodiment of this invention, a foil of 80-20 atomic per cent manganese-nickel for example, is formed by one of the known means, such as evaporation or rolling, depending upon the thickness of the thermistor flake desired. It is to be noted, however, that the per-

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centage of manganese need not be exactly 80 per cent. It may vary from 40 per cent to 90 per cent. This thin sheet can be cut into samples having the cross-sectional area desired. The samples can then be welded to frames, if desired, placed in a tray 3, which can be made of nickel chromium alloy, and then placed in an air atmosphere furnace 4 which has a temperature of from 600° C. approximately, to 700° C. approximately. It is left in the furnace a minimum of sixteen hours, during which time the temperature of the furnace is allowed to come down to a range of 450° C. approximately, to 500° C. approximately. This will produce complete oxidation of the nickel and the manganese without a separation of the two oxides. The thermistor flake, however, is not yet completed, since the aforementioned heat treatment has not produced the proper crystalline structure in the foil. The desired crystalline structure is known as a spinel formation.

To produce this crystalline formation, the sample is left or again placed in an air atmosphere furnace 4 having a temperature of about 450° C. and the furnace controls are so adjusted that the temperature will come up to about 875° C. to 1150° C. in approximately two hours. The resultant product is a smooth surfaced thin thermistor flake comprised of a relatively uniform mixture of nickel and manganese oxides and having a uniform thickness in a range extending from one-half micron to twenty-five microns. The crystalline structure is of the spinel type which gives the flake a resistivity of the same order of magnitude as 80-20 atomic per cent manganese-nickel oxides thermistor material when produced by the sintered oxide powder method.

It is understood that only a preferred embodiment of the invention is described herein and that the method can be utilized to encompass many other types of bimetal thermistors without departing from the spirit or scope of the invention.

What is claimed is:

1. A process for making thin flakes of uniformly mixed oxides of 40-60 to 90-10 atomic per cent manganese-nickel alloy having a thickness of from one-half micron to twenty-five microns which comprises forming a uniformly thin foil of approximately 40-60 to 90-10 atomic per cent manganese-nickel of from one-half to twenty-five microns thickness, heating the manganese-nickel alloy foil in an oxidizing atmosphere at a temperature of from 600° C. to 700° C., gradually cooling the foil to a temperature of from 450° C. to 550° C., keeping said foil at a temperature of at least 450° C. for not less than sixteen hours, and heating the resultant product at a temperature between about 875° C. and 1150° C. to produce a spinel type crystalline structure.

2. A process for making thin flakes of uniformly mixed oxides of approximately 40-60 to

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90-10 atomic per cent manganese-nickel alloy having a thickness of from one-half micron to twenty-five microns comprising the steps of forming a uniformly thin foil of approximately 40-60 to 90-10 atomic per cent manganese-nickel of from one-third micron to 18 microns thickness, placing said foil in an oxygen atmosphere temperature of from 600° C. to 700° C., allowing the foil to gradually cool to a temperature of from 450° C. to 550° C., keeping said foil at a temperature of at least 450° C. for not less than sixteen hours, and then placing the resultant product in a furnace which is allowed to come up to a temperature of from 875° C. to 1150° C. in an approximate two-hour interval to produce a spinel type crystalline structure.

3. A process for making flakes of oxidized 80-20 atomic per cent manganese-nickel having a uniform thickness of from one-half micron to twenty-five microns comprising the steps of forming a uniformly thin foil of 80-20 atomic per cent manganese-nickel alloy of from one-half to twenty-five microns thickness, heating the manganese-nickel alloy foil in an oxygen atmosphere temperature of from 600° C. to 700° C., allowing the foil temperature to gradually drop to from 450° C. to 550° C., and keeping the foil at a temperature between 450° C. and 550° C. for at least sixteen hours.

4. A process for making flakes of mixed oxides of 40-60 to 90-10 atomic per cent manganese-nickel alloy having a thickness of from one-half micron to 25 microns which comprises forming a uniformly thin foil of from one-half to 25 microns thickness of alloy, heating the foil in an oxidizing atmosphere at a temperature of from 600° C. to 700° C., gradually cooling the foil to from 450° C. to 550° C., and maintaining the foil at a temperature of at least 450° C. for of the order of sixteen hours.

5. The process for making thin flakes of uniformly mixed oxides of nickel and manganese wherein the atomic ratio of the metallic constituents of the oxides is from 40 to 90 per cent manganese and 60 to 10 per cent nickel, which comprises forming a uniformly thin foil of an alloy consisting of from 40 to 90 atomic per cent manganese and 60 to 10 atomic per cent nickel and of from one-half to 25 microns thick, heating the manganese-nickel alloy foil in an oxidizing atmosphere at a temperature from 600 to 700° C., and gradually cooling the foil to a temperature of from 450 to 550° C. over a period of not less than 16 hours.

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