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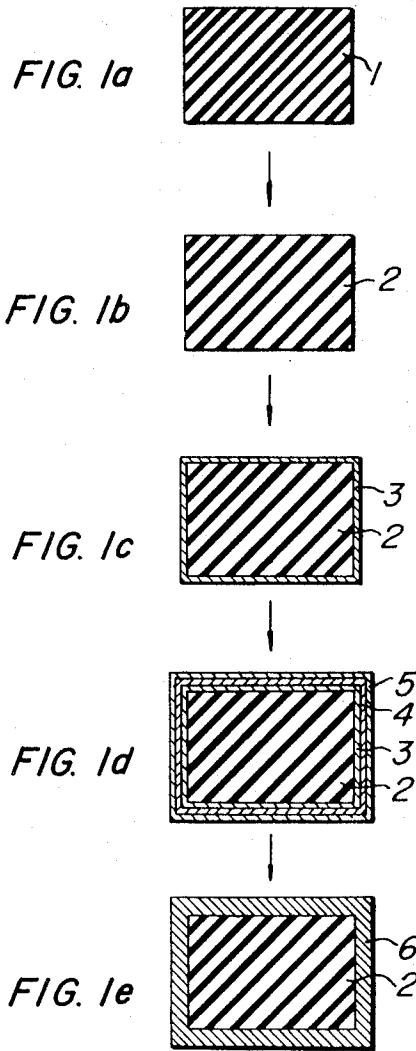
TAKESHI HASEGAWA ET AL

3,621,567

PROCESS FOR PRODUCING METALLIC FILM RESISTORS

Filed Dec. 24, 1968

3 Sheets-Sheet 1



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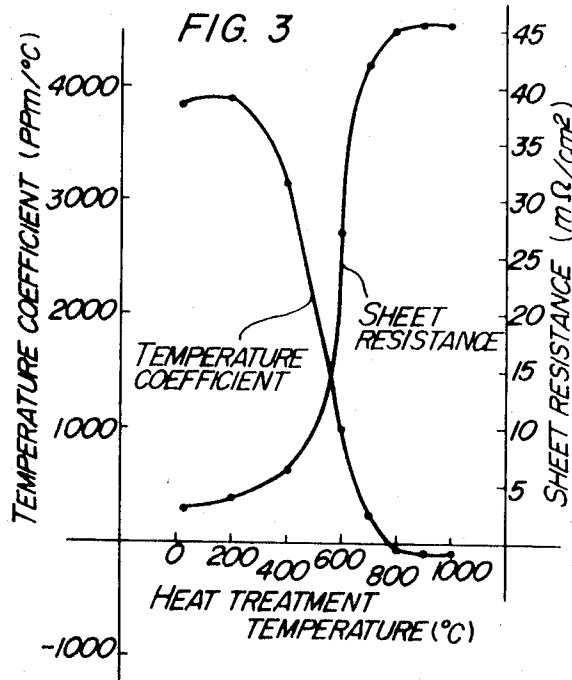
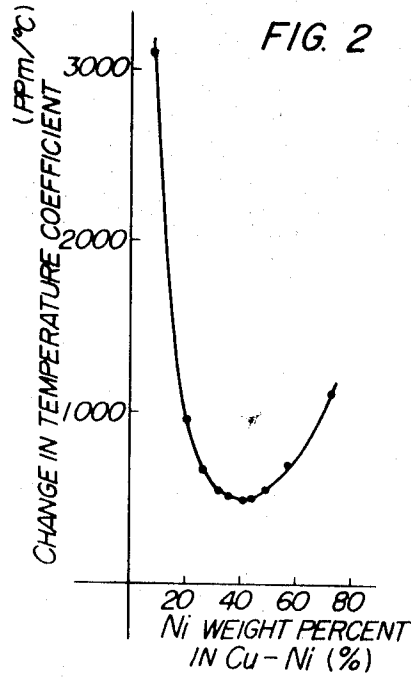
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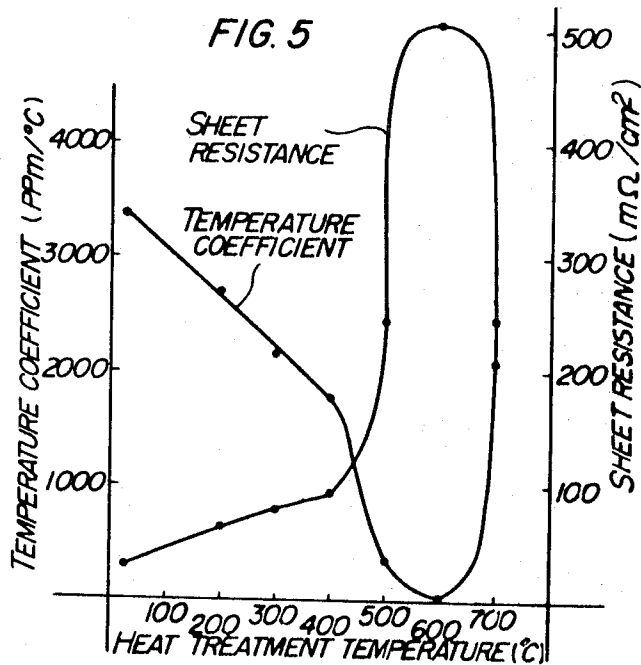
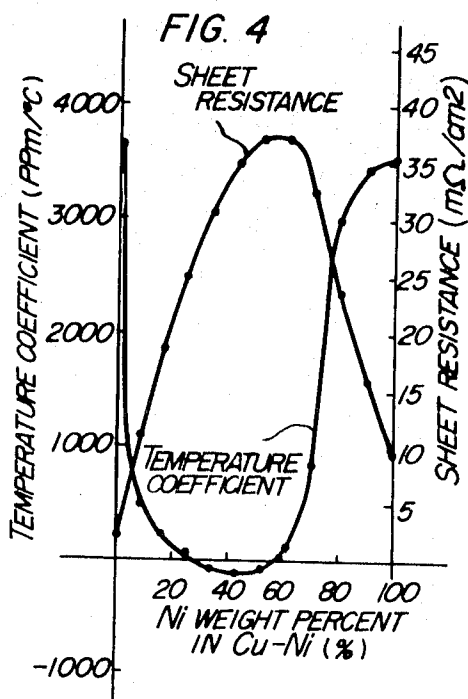
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3,621,567

**PROCESS FOR PRODUCING METALLIC
FILM RESISTORS**

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2 Claims

ABSTRACT OF THE DISCLOSURE

A process for producing metallic film resistors, comprising forming two or more layers of different metals on the surface of heat-resistant insulating substrates one on the other by non-electrolytic plating or electroplating and thereafter subjecting the coated substrates to heat treatment, whereby the different metals are alloyed by interlayer diffusion and thus a metal alloy coating is formed on the surface of the insulating substrates.

This invention relates in general to a process for producing metallic film resistors, and more particularly to a process for producing metallic film resistors which comprises forming layers of different metals on the surface of heat-resistant insulating substrates one on the other by plating and subjecting the coated substrates to heat treatment to effect interlayer diffusion of the metals, whereby an alloy coating is formed on the surface of the substrates.

In the production of metallic film resistors, two methods are being employed at present, i.e. vacuum evaporation coating and sputtering. Vacuum evaporation coating is a method of depositing a metal on the surface of an insulating substrate by placing said substrate in a vacuum of the order of 10^{-4} to 10^{-5} mm. Hg wherein the metal is evaporated at an elevated temperature. However, since the operation has to be carried out in vacuum, this method has the disadvantages of requiring an apparatus which is complicated in structure and difficult to handle, being low in productivity and raising the cost of the product. Furthermore, according to this method it is difficult to control the composition ratio of component metals in the vacuum evaporation of an alloy of said metals. On the other hand, sputtering is a method of depositing a metal on the surface of an insulating substrate in vacuum by placing the substrate in front of a positive electrode and impressing a voltage of the order of 1000 to 1500 v. across said metal, which constitutes a negative electrode, and said positive electrode. This method like vacuum evaporation coating has also to be carried out in vacuum, and therefore has the drawbacks of requiring a complicated apparatus, being low in productivity and raising the cost of the product resistor.

The present invention has for its object the provision of a process for producing metallic film resistors, which comprises coating the surface of a heat-resistant insulating substrate with a layer of chemical plating, forming layers of electroplatings on said layer of chemical plating in superposed relation and thereafter subjecting the coated substrate to heat treatment in a non-oxidizing atmosphere, whereby an alloy coating is formed on said substrate upon interlayer diffusion.

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The present invention will now be described in detail in conjunction with the accompanying drawings, in which: FIGS. 1a to 1e are views illustrating in sequence the steps of the process for producing metallic film resistors according to the present invention;

FIG. 2 is a characteristic diagram according to the first embodiment of the invention;

FIG. 3 is a characteristic diagram according to the second embodiment of the invention;

FIG. 4 is a characteristic diagram according to the third embodiment of the invention; and

FIG. 5 is a characteristic diagram according to the fourth embodiment of the invention.

The process of the instant invention will be described with reference to FIGS. 1a to 1e in sequence. FIG. 1a shows a heat-resistant insulating substrate and FIG. 1b shows the substrate 1 of FIG. 1 after the surface thereof has been subjected to etching with hydrogen fluoride to facilitate bonding of a coating thereto. Numeral 2 designates the heat-resistant insulating substrate thus treated. FIG. 1c is a chemical plating step in which a layer of chemical plating 3 of copper or nickel is formed on the surface of the heat-resistant insulating substrate 2 of FIG. 2 in a thickness of 0.4 to 0.5μ . FIG. 1d is an electroplating step in which a layer of electroplating 4 of copper or nickel is formed on the surface of the chemical plating 3, formed on the substrate 2 in the step of FIG. 1c, in a thickness not greater than 2μ and further a layer of electroplating 5 of a metal different from that of the layer of electroplating 4, e.g. nickel, copper or zinc, is formed on top of said electroplating layer 4 in a thickness not greater than 2μ . Where a coating of greater thickness or a coating of an alloy of three or more different metals is required, this may be attained by forming a multiplicity of plating layers, each having a thickness of 2μ or smaller, alternately one on top of another. FIG. 1e is a heat-treatment step in which the coated substrate of FIG. 1d is subjected to heat treatment in a non-oxidizing atmosphere for a predetermined period at a temperature of 400° C. or higher to effect interlayer diffusion and thereby to form an alloy coating. The product thus obtained is used as an electric resistor or a heating element.

EXAMPLE 1

A 3.5μ thick chemical plating layer of nickel was formed on the surface of porcelain rods having a diameter of 3 mm. and a length of 11 mm. and on top of the nickel plating layer was further formed an electroplating layer of copper. The thickness of the copper plating layers on the respective substrate porcelain rods was varied by adjusting the current conducted, such that the percentage by weight of the nickel varies in the range from 7 to 70%. The material resistors thus produced were subjected to heat treatment in a 10% hydrogen-containing nitrogen atmosphere for 2 hours at 700° C., whereby a copper-nickel alloy coating was produced on the respective substrates. The temperature coefficients of the respective finished resistors were measured, the results of which are shown in FIG. 2.

EXAMPLE 2

A 0.4 to 0.5μ thick chemical plating layer of copper was formed on the surface of porcelain rods having a diameter of 3 mm. and a length of 11 mm. and on top of the copper plating layer were formed 1μ thick electro-

plating layers of copper and 1μ thick electroplating layers of nickel alternately, the total thickness of the copper electroplating layers and the total thickness of the nickel electroplating layers being 5μ respectively and the total thickness of the electroplating layers being 10μ . The resistors thus produced were subjected to heat treatment in a 10% hydrogen-containing nitrogen atmosphere each for 2 hours at 200° to 1000° C. The temperature coefficients and the sheet resistances of the respective finished resistors were measured with the results as shown in FIG. 3. From the electron microscopic photos and the analysis by X-ray diffraction, it was confirmed that diffusion begins abruptly at 400° to 600° C. and the metals are alloyed completely at 800° C.

EXAMPLE 3

A 0.4 to 0.5μ thick chemical plating layer of copper was formed on the surface of porcelain rods having a diameter of 3 mm. and a length of 11 mm. and on top of the copper plating layer were formed electroplating layers of copper and nickel alternately such that the total thickness of said electroplating layers becomes 11μ and the percentage by weight of the nickel varies in the range from 0 to 100% (a coating of 100% by weight of nickel was obtained by using nickel for the chemical plating layer and forming an electroplating layer of nickel on top of the chemical plating layer or nickel). The respective material resistors thus produced were subjected to a heat treatment in a 10% hydrogen-containing nitrogen atmosphere for 2 hours at 800° C., whereby a copper-nickel alloy coating was formed on each substrate porcelain rod. The temperature coefficients and the sheet resistances of the finished resistors were measured with the results as shown in FIG. 4.

EXAMPLE 4

A 0.4 to 0.5μ thick chemical plating layer of copper was formed on the surface of porcelain rods having a diameter of 4.5 mm. and a length of 14 mm. and on top of the copper plating layer were formed a 1μ thick electroplating layer of nickel and a 1μ thick electroplating layer of zinc in the order mentioned. The resistors thus produced were subjected to heat treatment in a 10% hydrogen-containing nitrogen atmosphere for 1 hour at prescribed temperatures ranging from normal temperature to 700° C. The temperature coefficients and the sheet resistances of the respective finished resistors were measured with the results as shown in FIG. 5.

EXAMPLE 5

Resistors were produced by forming a copper-nickel system coating on each of bath a porcelain rod of 4.5 mm. in diameter and 14 mm. in length (2-watt type) and a porcelain rod of 7 mm. in diameter and 39 mm. in length (5 watt type) and subjecting the coating to a heat treatment at 800° C. for 1 hour. Using the resistors thus formed, resistor units of the 2 w. type, 51 ohms and 5 w. type, 160 ohms were produced respectively, on which various characteristic testings were conducted with the results shown in Table 1.

TABLE 1.—TEST RESULTS

Tests	Type	
	2 w., 51 ohms	5 w., 160 ohms
Temperature coefficient (p.p.m./ $^\circ$ C.)	+60	+40
Short-time overload (percent)	+0.03	-0.017
Humidity (percent)	+0.18	-0.029
Load life (percent)	+0.74	+0.007
Life in humidity (percent)	+0.23	+0.42
Soldering effect (percent)	+0.02	-0.016
Shelf life (percent)	+0.001	+0

Testing conditions

(1) Temperature coefficient.—The temperature coefficients were measured in a temperature range from -30° to $+180^\circ$ C.

(2) Short-time overload.—A voltage 2.5 times the rated voltage was impressed for 5 seconds.

(3) Humidity.—The resistor units were left to stand in a space at an ambient temperature of 40° C. and a humidity of 90 to 95% (90 to 95 R.H.) for 240 hours with no load connected thereto.

(4) Load life.—A cycle of impressing the rated voltage for 1.5 hours and interrupting the voltage for 0.5 hour was repeated for 1000 hours at an ambient temperature of 40° C.

(5) Life in humidity.—A cycle of impressing the rated voltage for 1.5 hours and interrupting the voltage for 0.5 hour was repeated for 1000 hours in a space at an ambient temperature of 40° C. and a humidity of 90 to 95%.

(6) Soldering effect.—A lead wire (resistor terminal) was immersed in a solder at 350° C. for 3 seconds.

(7) Shelf life.—The resistor units were left to stand in a space at room temperature and normal humidity for 1 year with no load connected thereto.

According to the vacuum evaporation coating and the sputtering which have been employed heretofore in the production of metallic film resistors, the operations must be carried out in vacuum and, therefore, the apparatus used becomes complicated and the composition ratio of an alloy coating to be formed can hardly be controlled, with the accompanying results that the productivity of the methods is low and that the cost of the finished resistors becomes high, as stated previously.

On the contrary, the process of this invention wherein use is made of the chemical plating and electroplating techniques, can be performed at normal temperature and normal pressure. Therefore, the apparatus used can be simplified; the thickness and the composition of the coating can be easily controlled; the productivity can be enhanced and the cost of the product can be reduced. Furthermore, although wire wound resistors are predominantly being used at present as resistors of low resistance value, it is possible according to the process of this invention to produce film resistors whose resistance value is in the same range as the wire wound resistors. It is also to be noted that since the plating techniques are used in the process of this invention, the shape of a resistor produced can be selected freely and the temperature coefficient of the resistor can also be selected freely in the range from -100 to $+3500$ p.p.m./ $^\circ$ C. by changing the composition ratio of the metals used. Therefore, this invention is of great industrial advantage over the prior art.

We claim:

1. A process for producing metallic film resistors comprising: forming a layer of copper chemical plating on a heat-resistant insulating substrate; forming alternating layers of copper and nickel electroplating, starting with the copper layer on top of said layer of copper chemical plating; and thereafter heating the plated substrate in a non-oxidizing atmosphere to cause diffusion between discrete layers of plating, thereby obtaining an alloy coating resistor film.

2. A process for producing metallic film resistors comprising: forming a layer of copper chemical plating on a heat-resistant insulating substrate; forming alternating layers of nickel and copper electroplating, starting with the nickel layer on top of said layer of copper chemical plating; and thereafter heating the plated substrate in a non-oxidizing atmosphere to cause diffusion between discrete layers of plating, thereby obtaining an alloy coating resistor film.

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