

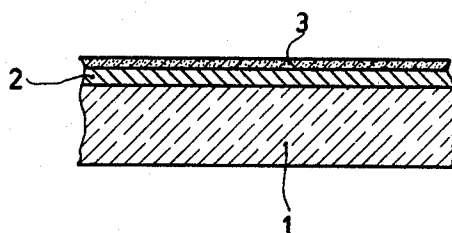
Oct. 14, 1969

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3,472,691

STABLE RESISTANCE FILM OF Ni-CR

Filed March 17, 1967



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3,472,691

## STABLE RESISTANCE FILMS OF Ni-Cr

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Filed Mar. 17, 1967, Ser. No. 623,924

Claims priority, application Netherlands, Mar. 23, 1966, 6603768

Int. Cl. H01b 1/02; C23c 13/02

U.S. Cl. 117-217

2 Claims 10

## ABSTRACT OF THE DISCLOSURE

Produce nickel-chromium thin film resistors by deposition in oxygen atmosphere of  $10^{-5}$  mm. Hg and then in oxygen atmosphere of  $10^{-4}$  mm. Hg.

This invention relates to methods of manufacturing resistors consisting of thin films of nickel-chromium, by evaporation-deposition in vacuo.

Such resistors may be used in miniature circuits. When used for this purpose, it is desirable that the resistors should have a low temperature coefficient and a high stability even if the ambient temperature is comparatively high. Further it is desirable that, if subsequent to the application of the resistance material to the carrier, variations in resistivity of irreversible nature occur, for example, during a thermal treatment for thermal stabilisation, this variation is reproducible.

Nickel-chromium resistors are generally manufactured by evaporating a nickel-chromium alloy (80-20) in a vacuum having a residual gas pressure of  $10^{-5}$  mm. Hg and depositing the vapour evolved onto a carrier which has a temperature of approximately  $300^{\circ}$  C. After a certain resistivity is reached, the evaporation-deposition is stopped. The films thus obtained are generally found to have still an inadequate thermal stability. Consequently, in practice, such resistance films are also heated for a short time in air, for example, at approximately  $300^{\circ}$  C. During this heating treatment oxidation and recrystallization processes presumably take place in the resistance film, resulting in a permanent variation in resistivity which progressively decreases, so that a desired stability is ultimately obtained. For this variation in resistivity upon stabilisation it applies in general that the higher the film resistance, that is to say the thinner the resistance film, the greater is the variation in resistivity upon stabilisation by heating. In practice, this has been found to happen in a manner which is poorly reproducible and hence not well predictable.

It is thus difficult on a large scale to obtain miniature circuits in which the resistances satisfy comparatively narrow tolerances such as, for example, between  $\pm 3\%$  of the desired value.

The variation in the resistance of the film upon thermal stabilisation in air, in practice also sets a limit to the value of this resistance. From literature it appears, for example, that for this reason also the resistance is chosen to be not higher than  $200\Omega$  to  $300\Omega$  per square.

An object of the invention is to manufacture resistors of the type consisting of a thin film of nickel-chromium on a carrier by evaporation-deposition in vacuo, the resistivity of which resistors varies to a comparatively small extent upon thermal treatment in air for stabilisation.

This object is attained in a process in which in a first step a film of nickel-chromium is evaporation-deposited on a carrier in an atmosphere of oxygen at a pressure of the order of  $10^{-5}$  mm. Hg until the desired resistance of the film is obtained, whereafter in a second step oxygen is admitted up to a pressure of the order of  $10^{-4}$  mm. Hg

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and the deposition of nickel-chromium is continued for some further time, whereby the resistivity of the deposited film does not vary.

The atmosphere of oxygen at a pressure of the order of  $10^{-5}$  mm. Hg may be obtained, for example, by reducing the residual gas pressure in the space in which the resistance films are provided to a pressure of the order of  $10^{-6}$  mm. Hg or less and then admitting oxygen to the space until a pressure of the order of  $10^{-5}$  mm. Hg is reached. During the evaporation-deposition of the nickel-chromium, a certain oxidation already takes place. It is therefore preferable during this process to admit oxygen steadily in order to maintain the pressure of the order of  $10^{-5}$  mm. Hg. A substantially constant content of oxygen for the resistance film is thus ensured. After the desired resistance is reached, more oxygen is admitted to an extent such that, with continued deposition of nickel-chromium, variation in the resistivity of the film deposited at a pressure of oxygen in the order of  $10^{-5}$  mm. Hg does not occur any more.

In practice, this pressure is found to be of the order of  $10^{-4}$  mm. Hg. It appears to be preferable also in the second step to maintain the pressure constant, by steadily admitting oxygen to the evaporation space.

The thickness of the oxidic covering layer on the resistance film depends upon the extent of protection and thermal stability which it is desired to obtain and upon the possibility of contacting and etching.

The total time needed for evaporation-deposition of the resistance film and the covering layer depends upon a number of geometrical factors, such as the distance between source of evaporation and substrate to be deposited on; this distance may be determined in a simple manner in each individual case.

In order that the invention may be readily carried into effect, one embodiment of the method according thereto will now be explained in detail, with reference to the example following hereinafter and the accompanying drawing, the sole figure of which shows diagrammatically the structure of a resistor according to the invention.

## EXAMPLE

Nickel-chromium is deposited by evaporation on a plurality of glass plates in a vacuum recipient containing a holder for the said plates, a source of evaporation consisting of a foil of nickel-chromium alloy (80-20) which may be heated by the passage of electric current, an aperture through which the space may be exhausted and an inlet for the oxygen to be supplied, and means for heating the substrates to be deposited on. One glass plate has previously been provided with metal contacts, included in an electric circuit so that the resistivity of the film can be observed during the depositing process. After reaching a pressure of  $10^{-6}$  mm. Hg, oxygen is admitted to the vacuum space up to a pressure of 1 to  $2 \cdot 10^{-5}$  mm. Hg. The glass plates are heated to a temperature of approximately  $300^{\circ}$  C. by radiation heating and nickel-chromium is evaporation-deposited until the desired resistivity is reached. Subsequently, more oxygen is admitted, without interrupting the depositing process, until the pressure is, for example, from 0.6 to  $0.7 \times 10^{-4}$  mm. Hg. This pressure has to be such that the resistivity of the film does not vary any more with continued deposition of the nickel-chromium.

The depositing process is stopped after some time, in this example after 2 minutes.

As a measure of the increased stability of such resistance film according to the invention, the table following hereinafter shows several representative values of variations in resistance if the resistance films are heated at  $200^{\circ}$  C., for example for 24 hours.

The first column shows the resistivities per square of the resistance films, the next column shows whether an oxidic covering layer as described has been applied, and the last column shows the variation in resistance after heating at 200° C. in air for 24 hours.

	Rt in $\Omega/\square$	Oxidic layer provided	R/R in percent after 24 hours heating at 200° C. in air
1.	300	Yes	+0.3
2.	300	No	+20
3.	300	No	+38
4.	500	Yes	+5
5.	500	No	+56
6.	1,200	Yes	12.5
7.	1,200	No	+230

From literature it is known that, upon thermal treatment of resistance films having resistivities of 100 $\Omega$  to 150 $\Omega$  per square, variations in resistivity between -5% and +10% may occur upon thermal after-treatment. Said resistivities were obtained by evaporation- deposition at a residual gas pressure lower than 10<sup>-4</sup> mm. Hg.

From the table it may be seen that a considerable improvement in this respect is obtained by combination of the two steps.

The temperature coefficient of the resistors according to the invention is low and usually of the order of -50 p.p.m. per ° C.

It is also known to protect nickel-chromium resistors from atmospheric influences by covering the resistance film by evaporation with a layer of SiO<sub>2</sub> or MgF<sub>2</sub>. The effect thus envisaged is obtained in a considerably simpler manner by using the method in accordance with the invention.

The first step of the method according to the invention has more particularly for its purpose to obtain higher resistivities at comparatively thicker layers in a reproducible manner.

The sole figure in the drawing shows, on an enlarged scale, the structure of a resistor obtained in accordance with the invention. A substrate 1, for example of glass, is covered with a first nickel-chromium layer 2 of a low content of oxygen and the desired resistivity and with a second nickel-chromium layer 3 which is rich in a oxygen. The last-mentioned layer, which has no electric conductivity, is of a composition which is not known but possibly consists of a mixture of oxides of nickel and chromium.

What is claimed is:

1. A method of manufacturing a thin film resistor, said method comprising the steps depositing on a carrier, in an oxygen atmosphere of about 10<sup>-5</sup> mm. Hg, a first evaporation layer of chromium-nickel until a desired resistivity is achieved and then time stabilizing the resistivity of said layer by depositing thereon, in an oxygen atmosphere of about 10<sup>-4</sup> mm. Hg, a second evaporation layer of chromium-nickel having a thickness at which the resistivity of the composite layer is no longer substantially effected by time.

2. A resistor produced by the method of claim 1.

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U.S. Cl. X.R.

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UNITED STATES PATENT OFFICE  
CERTIFICATE OF CORRECTION

Patent No. 3,472,691 Dated October 14, 1969

Inventor(s) CORNELIS KOOY ET AL

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

Column 1, line 5, "Company" should read -- Corporation  
Column 1, line 46, "is" should read -- in --.  
Column 2, line 50, "supplier" should read -- supplied -

Signed and sealed this 28th day of July 1970.

(SEAL)

Attest:

Edward M. Fletcher, Jr.  
Attesting Officer

WILLIAM E. SCHUYLER, JR.  
Commissioner of Patents