May 5, 1942. J. W. JIRA 2,281,843

METAL FILM RESISTOR

Filed Feb. 3, 1940

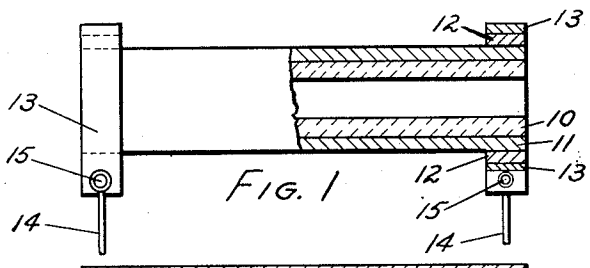


FIG. 1

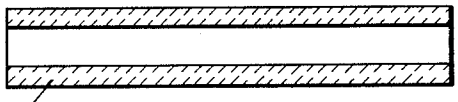


FIG. 2

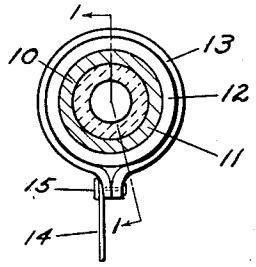


FIG. 8

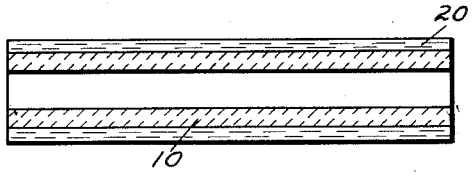


FIG. 3

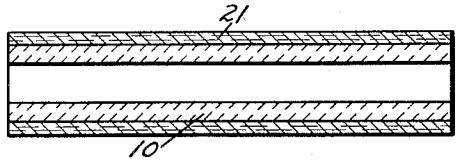


FIG. 4

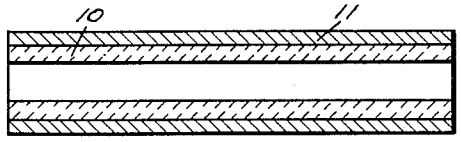


FIG. 5

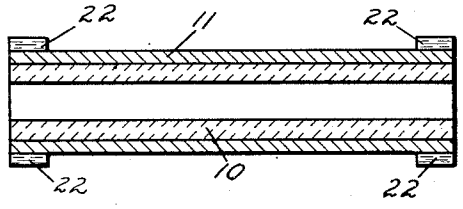


FIG. 6

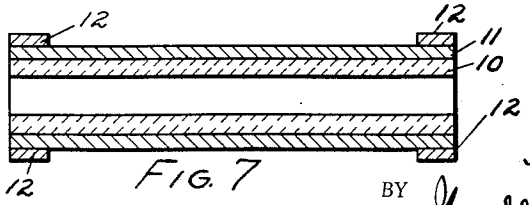


FIG. 7

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METAL FILM RESISTOR

Joseph W. Jira, Newburgh Heights, Ohio, assignor to Continental Carbon, Inc.

Application February 3, 1940, Serial No. 317,154

11 Claims. (Cl. 201—76)

My invention relates in general to resistance units and more particularly to precision resistance units and the process for making the same.

A precision resistance unit must possess the following requirements:

(1) Stable shelf life. This term implies that the resistor must not change more than 1.0% from its original value when subject for 10,000 hours to normal atmospheric conditions without any current flowing through the unit.

(2) Minimum voltage characteristic. When the voltage is changed from that value required to give 2% of rated wattage to that value required to give 200% of rated wattage, the resistance shall not change more than 0.1% for values below 100,000; 0.2% for values below 500,000 ohms and 0.3% for values above 500,000 ohms.

(3) Minimum temperature coefficient of resistance. The average change in resistance due to temperature must not exceed .02% per degree centigrade.

(4) Low, no load humidity characteristic. Resistors must not deviate more than 1.0% from their initial value when conditioned 100 hours at a relative humidity of 90% at 40° C.

(5) Stability at rated and double rated wattage. Resistors subjected to continuous operation for 5,000 hours at rated wattage shall show no permanent change in resistance exceeding 2.5% when measured by the bridge method at 25° C. At double rated wattage the change in resistance shall not exceed 5% from the original when measured at 25° C.

(6) Low noise characteristic. When precision resistors are tested for noise at rated wattage by connecting unit and battery in series with a balancing network connected to the input of a 120 DB resistance coupled amplifier, the measured noise must not exceed 0.2 microvolt per volt for any given value of resistance.

(7) Good high frequency characteristic. When precision resistance units are subjected to high frequency, they must have a low inductive and capacitative reactance.

The applicant finds from years of experience in the development, testing and manufacture of resistance units that carbon composition and wire wound resistance units do not possess all of the requirements of a precision resistor.

In comparing the characteristic of carbon composition resistors with the above mentioned requirements of a precision resistance unit, it is noted that the carbon composition resistors are deficient due to the following properties:

(1) Carbon resistors, as a whole, do not possess stable shelf life or permanency of resistance over an extended period. Tests performed on hundreds of units of various makes show that the shelf life may vary anywhere from 1.0% to 30.0% in 250 hours.

(2) The minimum voltage characteristic may vary anywhere from 0.1% to 1.0% for values below 100,000 ohms; 1.0% to 5.0% for values below 500,000 ohms and from 2.0% to 10.0% for values above 500,000 ohms.

This variation of resistance with applied voltage could not possibly be tolerated in precision electrical equipment.

(3) The minimum temperature coefficient of resistance may vary anywhere from —0.07 to +0.05% per degree centigrade. In other words, the resistance in ohms of any given value may change from —7.0% to +5.0% for a temperature variation of 100° centigrade.

(4) The low, no load humidity characteristic was found to vary anywhere from —15.0% to +20.0% in 100 hours.

(5) At normal rated wattage the load characteristic varied anywhere from —10.0% to +10.0% in 100 hours, while at twice rated wattage the load characteristic varied from —15.0 to +15.0%.

(6) Noise, inherent within all carbon composition type resistors, varied anywhere from 1.0 to 4.0 microvolts per volt for any given value.

The wire wound resistor is deficient due to the following characteristics:

(1) For any given wattage rating wire wound resistors increase in size with resistance value, so that resistors varying in value from 200,000 to 1,000,000 ohms are not only excessively large, but prohibitive in price.

(2) If the wire wound resistors are expected to remain reasonably stable under varying atmospheric conditions, the wire must be protected with vitreous enamel fused upon the surface of the unit at very high temperatures. High vitrificating temperatures cause alterations in the metal-crystal structure within the wire comprising the resistance unit and consequently hot spots appear which seriously impair the operating characteristics of the resistor.

(3) Alterations in the metal crystal structure due to high vitrificating temperatures also cause a great reduction in strength and elasticity of the resistance wire thus initiating open circuits prior to the application of voltage.

(4) Due to the extremely reduced diameter of the wire (.001") employed in fabricating wire wound resistors of high value (15,000 to 100,000

ohms), breaks often appear at the contact due to thermal expansion of the unit while under the influence of heat.

(5) Noise may vary between the limits of 0.2 to 1.0 microvolt per volt brought about by oxidation between the resistance wire and the external terminals.

(6) Wire wound resistors are not particularly well adapted to high frequency circuits due to their inherently high inductive and capacitative effects. At high frequencies (5 to 60 megacycles) the inductive and capacitative reactance exerts a pronounced influence upon the initial direct current value of resistance. The shift in resistance may be in either a positive or negative direction depending upon the electrical parameters of the wire wound unit. This shift in high frequency resistance may be as much as 60% from the original measured value.

Apart from some of the physical and electrical deficiencies affixed to wire wound resistors, they present many noteworthy features, when properly fabricated.

1. A very stable shelf life.
2. Minimum voltage characteristic.
3. Minimum temperature coefficient of resistance.
4. Low humidity characteristic.
5. Stability of resistance while subject to either normal or twice rated wattage.
6. Low noise level.

An object of my invention is to construct a resistance unit possessing the good qualities of each of the carbon composition resistors and the wire wound resistors.

Another object of my invention is to construct a resistance unit possessing all of the qualifications of a precision resistor.

Another object of my invention is to construct the resistance element part of the resistor by atomically depositing a thin film of a metal upon the surface of a non-conductive carrier.

Another object of my invention is to construct the resistance element part of the resistor by coating a non-conductive carrier with an organo-compound of a metal and heating same to atomically deposit a thin film of the metal upon the non-conductive carrier and to oxidize the remaining portion of the organo-compound of the metal.

Another object of my invention is to deposit metal upon the end portions or upon spaced surfaces of the thin film of metal to serve as a connecting area for the terminals.

Another object of my invention is to provide a method for controlling the thickness of the thin metal film deposited upon the non-conductive carrier.

Other objects and a fuller understanding of my invention may be had by referring to the following description and claims taken in conjunction with the accompanying drawing, in which:

Figure 1 shows a longitudinal view of a resistance unit embodying the features of my invention, partly in section along the line 1—1 of Figure 8; and

Figures 2 to 8, inclusive, show the steps by which my invention is constructed, Figure 8 being a section taken transversely through the terminal connection.

With reference to Figure 1, my resistance unit comprises a non-conductive carrier 10, a thin metal film 11 deposited upon the outer surface of the non-conductive carrier 10, a body of thin metal deposit 12 upon each end portion of the thin metal film 11, and a terminal member 13 having a lead 14 connected to the body of the thin metal deposits 12 upon each end of the resistor.

The non-conductive carrier 10 may be constructed of any suitable material and may comprise a rod or a hollow tube as shown in Figure 2 and be made of ceramic material which will withstand thermal shock and which possesses a very low moisture absorbing characteristic. In actual practice, I find that a ceramic material like Isolantite or its equivalent is suitable and preferable.

The thin metal film 11 is atomically deposited upon the outer surface of the non-conductive carrier 10 by coating the outer surface with an organo-compound of a substantially stable and substantially non-oxidizable metal and heating the same. Before applying the organo-compound to the ceramic tube 10, the outer surface is subjected to a degreasing or cleaning operation. This may be done by suspending or immersing the ceramic tube into a boiling solution of trisodium phosphate for about 10 minutes. It has also been found through careful research that numerous other chemical cleaning agents are adapted for use in the cleaning operation. Outstanding among these are ethylene di-chloride and tetra-chloro-ethane. The ceramic tube 10, after it is removed from the cleaning solution, is washed five or six times in clear running water and then thoroughly dried.

Upon the drying of the ceramic tube, a thin coating of the organo-compound comprising an organic resinate of a stable and non-oxidizable metal is applied to the ceramic tube by either dipping, brushing or spraying. The term substantially stable and substantially non-oxidizable metal comprises those metals principally of the noble group although not limited thereto. The metal must remain substantially stable and be substantially non-oxidizable under high temperatures sufficient to burn out the residue remaining after deposition of the metal upon the ceramic tube. Under the general class of compounds known as the resinates are included the constituents of natural occurring resinates, resins, exudations from trees and synthetic preparations. In preparing my metallic organo-compound, the metal is substituted into or added to the organo-resinate. Of the metals, I find that palladium or platinum may be substituted into or added to the resinate, giving substantially palladium or platinum resinate. My invention will be described with palladium resinate but it will be understood that it includes platinum resinate or its equivalent.

To obtain a thin metal film upon the ceramic carrier to give a high resistance value, I find that it is difficult to do so by applying a substantially saturated compound of palladium resinate, without an appropriate solvent, to the ceramic carrier. Even though one attempts to apply by brushing or dipping a substantially saturated compound of palladium resinate, in the absence of a solvent, as thinly as possible upon the carrier, yet the chemically deposited metal film results in too great a thickness to give a high ohmic value of resistance. The solvent functions as a medium for carrying the palladium resinate, so that the palladium resinate is evenly distributed upon the ceramic carrier 10. That is to say, the palladium resinate is evenly dispersed in the solvent, and when applied to the ceramic carrier gives in effect a reduced amount of palladium. Of the

suitable solvents, I find that a high boiling ketone works satisfactorily. In the practice of my invention, a good thin metal film is obtained by adding a sufficient amount of solvent to the palladium resinate so that the amount of palladium is substantially one percent.

After the coating comprising the palladium resinate and the solvent, which is represented by the reference character 20 in Figure 3, is applied to the ceramic carrier 10, it is then permitted to dry in air for about 30 minutes at a temperature of approximately 20 to 25 degrees centigrade. The thickness of the coating is exaggerated in the drawing. Upon drying, the ceramic carrier and coating is given its first stage of heat treatment to chemically deposit the palladium upon the carrier. The precipitation of pure metallic palladium from the palladium resinate starts at temperatures ranging anywhere from approximately 200° to 400° centigrade. Careful study shows that a very fine cubical crystal formation of the precipitated palladium occurs at 300° centigrade and that the precipitation progresses with time until approximately a 100% metallic deposit results. The time was found to vary anywhere from 15 to 30 minutes, depending upon the surface area of the ceramic carrier and the thickness of the applied coating. Accompanying the precipitation of palladium is the formation of ash on the carrier mixed with the pure palladium which comprises essentially carbon as the residue of the applied coating and is represented by the reference character 21 in Figure 4.

The second stage heating is to completely oxidize the ash or residue and to insure a thorough precipitation of the palladium. The second stage heating may range from 400° to 750° centigrade for about one hour. Figure 5 shows the ceramic tube after the residue is thoroughly oxidized leaving the thin palladium film 11 which may be characterized as the basic resistance. The metallic film possesses unusually good bonding properties. In fact, the metal bonding characteristic is such that the only means of removal from the ceramic carrier is by grinding. The ceramic and the deposited metal are virtually one.

The next general series of steps in my process is to connect the terminal members 13 having leads 14 to the end portions of the thin metal film 11. To do this, I first deposit a body of thin metal 12 upon the end portions of the palladium film 11 as shown in Figures 1 and 7. The body of thin metal 12 may be physically deposited upon the end portions of the palladium film 11 by coating the end portions with a band of colloidal silver as indicated by the reference character 22 in Figure 6 and heating the same at approximately 500° to 600° centigrade for 30 minutes or thereabouts.

After the band of silver 12 is deposited about the end portions, the terminal members 13 comprising preferably thin strips of copper metal are clamped thereon as shown in Figures 1 and 8, making a good electrical contact with the silver. The terminal members 13 may be securely clamped about the silver deposit by fastening the free ends together by means of a rivet 15. The leads 14 may be connected to the terminal members 13 by the same rivets that hold the free end of the terminal members 13 together. The connection at the rivet may also be soldered.

The silver is ideally suited in this part of the process since it possesses a low thermo-electric effect against either the palladium or the copper metal of the terminal members. This type of contact is of extremely low resistance, thus reflecting its advantages in producing a resistor possessing a minimum noise and voltage characteristic.

To complete the resistance unit, it may or may not be spiralled depending upon the resistance value desired. The resistor is then given a coat of moisture proof lacquer which when dried completes the process. In my invention, the heating to burn out the ash or residue is below the melting point of the precipitated metal and also below the oxidizing temperature of the metal, giving a good stable and continuous film.

The initial basic resistance value of the palladium film 11 may be controlled by the number of coats applied to the ceramic carrier or by varying the amount of the palladium resinate in the applied coating. In the event an additional number of coatings are applied, they may be added at different stages in the process. As a first example, the additional coatings may be applied on top of the coating 20 in Figure 3, after each applied coating is allowed to dry. After the several coatings are dried, the process is then carried out as above explained. As a second example, an additional coating may be applied on top of the coating 21 in Figure 4, which means that two first stage and one second stage firings are required. Under the second example, any number of coatings may be applied by repeating the procedure. After the additional coatings have been added under the second example, the general process is then completed as previously described. As a third example, an additional coating may be applied on top of the metal film 11 in Figure 5, which means that two first stage and two second stage firings are required. Under the third example, any number of coatings may be applied by repeating the procedure. After the additional coatings have been added under the third example, the general process is then completed as previously described.

The resistance may also be varied by spiralling, that is, by grinding a very narrow helical groove along the periphery of the carrier, giving a helical path for the current to flow.

This spiralling procedure has the effect of increasing the total effective resistive path and by doing so increases the resistance value of the unit manifold. As an example, a resistance having a basic value of 1000, 5000 and 10,000 ohms when ground with a helical path equally along the periphery of the carrier for a period of ten revolutions will produce 100,000, 500,000 and 1,000,000 ohms respectively. Therefore, by utilizing five or six basic values a great number of various resistance values are possible.

Due to the fact that the conducting medium of my invention is metal, many of the difficulties arising from the use of carbon were completely eliminated. The first of these deficiencies was temperature coefficient of resistance. It is well known that carbon possesses a negative temperature coefficient, a factor partly responsible for resistor failures when subject to normal or twice rated wattage.

Since gas carbon is now chiefly employed in carbon composition type resistors, it exerts a certain amount of influence upon the water repellency of the manufactured unit. Since gas carbon is hygroscopic it prevents the manufacture of a resistor possessing a stable shelf life or a low humidity characteristic, even if thoroughly impregnated in wax under reduced pressure.

Furthermore, since the conventional carbon

composition resistor is an admixture of carbon and a bonding agent, usually a polymerizable resin, it is more or less prone to carbonization and therefore deterioration while under the influence of heat, a factor chiefly responsible for resistor failure.

By virtue of the bonding medium and countless particles of carbon that do not contribute towards the resistance value (undispersible conductor) hundreds of minute capacities are formed which exert a pronounced influence upon the high frequency characteristic of the unit. This distributed capacity is usually more evidenced in units of high resistance value, say 100,000 ohms and above.

All the above shortcomings of the carbon type resistor were completely rectified by the metal film resistor.

The new resistor not only possessed all the desirable electrical characteristics of a wire wound unit, but for a given value and wattage rating was one-tenth the size, a factor of commercial importance.

The most difficult steps encountered in fabricating a resistor of the type were the following:

(1) The applied metallic-organo film had to be evenly distributed over the surface of the carrier, otherwise areas of uneven density appeared over the periphery of the unit and caused erratic resistance fluctuations during the spiralling operation.

(2) Temperatures had to be carefully controlled otherwise nonadhering or only partly adhering metallic films resulted.

(3) The deposited metallic film 11 had to be of sufficient thickness to prevent discontinuous electrical paths resulting from a difference in the linear coefficients of expansion of the metal film and the ceramic carrier.

Although I have described my invention with a certain degree of particularity, it is understood that the present disclosure has been made only by way of example and that numerous changes in the details of the combination and arrangement of substances may be resorted to without departing from the spirit and the scope of the invention as hereinafter claimed.

I claim as my invention:

1. The process of constructing a resistance unit comprising the steps of providing a non-conductive carrier, providing a metallic organo-compound comprising an organic resinate of a substantially stable and substantially non-oxidizable metal, coating the non-conductive carrier with the metallic organo-compound, heating the coated non-conductive carrier to first atomically deposit a thin continuous film of the metal upon the non-conductive carrier and to secondly oxidize the carbonaceous residue remaining after the deposition of the thin film of metal, providing terminals for the resistance unit, and connecting the terminals to spaced surfaces of the thin film of the metal.

2. The process of constructing a resistance unit comprising the steps of providing a non-conductive carrier, providing a compound of palladium resinate, coating the non-conductive carrier with said compound, heating the coated non-conductive carrier to first atomically deposit a thin film of the palladium upon the non-conductive carrier and to secondly oxidize the remaining portion of the said compound, providing terminals for the resistance unit, and connecting the terminals to spaced surfaces of the thin film of the palladium.

3. The process of constructing a resistance unit comprising the steps of providing a non-conductive carrier, providing a compound of platinum resinate, coating the non-conductive carrier with said compound, heating the coated non-conductive carrier to first atomically deposit a thin film of the platinum upon the non-conductive carrier and to secondly oxidize the remaining portion of the said compound, providing terminals for the resistance unit, and connecting the terminals to spaced surfaces of the thin film of the platinum.

4. The process of constructing a resistance unit comprising the steps of providing a non-conductive carrier, providing a palladium resinate, cleaning the surface of the carrier, coating the carrier with said palladium resinate, first heating the coated carrier in a temperature range from 200 degrees to 400 degrees centigrade to atomically deposit a thin film of the palladium upon the carrier, secondly heating the coated carrier in a temperature range from 400 degrees to 750 degrees centigrade to oxidize the remaining portion of said palladium resinate, providing terminals for the resistance unit, and connecting the terminals to spaced surfaces of the thin film of the palladium.

5. The process of constructing a resistance unit comprising the steps of providing a non-conductive carrier, providing a metallic organo-compound comprising an organic resinate of a substantially stable and substantially non-oxidizable metal, cleaning the surface of the carrier, coating the non-conductive carrier with the metallic organo-compound, first heating the coated carrier in a relatively low temperature range to atomically deposit a thin continuous film of the metal upon the carrier, secondly heating the coated carrier in a relatively high temperature range to oxidize the carbonaceous residue remaining after the deposition of the thin film of metal, providing terminals for the resistance unit, and connecting the terminals to spaced surfaces of the thin film of the metal.

6. The process of constructing a resistance unit comprising the steps of providing a non-conductive carrier, providing a metallic organo-compound comprising an organic resinate of a substantially stable and substantially non-oxidizable metal, adding a solvent to the metallic organo-compound to reduce the concentration of the metal, cleaning the surface of the carrier, coating the non-conductive carrier with the metallic organo-compound and the solvent, first heating the coated carrier in a relatively low temperature range to atomically deposit a thin continuous film of the metal upon the carrier, secondly heating the coated carrier in a relatively high temperature range to oxidize the carbonaceous residue remaining after the deposition of the thin film of metal, providing terminals for the resistance unit, and connecting the terminals to spaced surfaces of the thin film of the metal.

7. The process of constructing a resistance unit comprising the steps of providing a non-conductive carrier, providing a palladium resinate, adding a solvent to the palladium resinate to reduce the concentration of the palladium, cleaning the surface of the carrier, coating the carrier with the palladium resinate and the solvent, first heating the coated carrier in a relatively low temperature range to atomically deposit a thin film of the palladium upon the carrier, secondly heating the coated carrier in a relatively high tem-

perature range to oxidize the remaining portion of the palladium resinate, providing terminals for the resistance unit, and connecting the terminals to spaced surfaces of the thin film of the palladium.

8. The process of constructing a resistance unit comprising the steps of providing a non-conductive carrier, providing a platinum-resinate, adding a solvent to the platinum resinate to reduce the concentration of the platinum, cleaning the surface of the carrier, coating the carrier with the platinum resinate and the solvent, first heating the coated carrier in a relatively low temperature range to atomically deposit a thin film of the platinum upon the carrier, secondly heating the coated carrier in a relatively high temperature range to oxidize the remaining portion of the platinum resinate, providing terminals for the resistance unit, and connecting the terminals to spaced surfaces of the thin film of the platinum.

9. The process of constructing a resistance unit comprising the steps of providing a non-conductive carrier, providing a palladium resinate, adding a high boiling ketone to the palladium resinate to reduce the concentration of the palladium therein to substantially one percent, cleaning the surface of the carrier, coating the carrier with the palladium resinate and the ketone, first heating the coated carrier in a relatively low temperature range to atomically deposit a thin film of the palladium upon the carrier, secondly heating the coated carrier in a relatively high temperature range to oxidize the remaining portion of the palladium resinate, providing a silver colloid, applying the silver colloid to spaced surfaces of the thin film of palladium, thirdly heating the carrier, the thin film of palladium and the silver colloid to physically deposit the silver upon the said spaced surfaces, providing terminals for the resistance unit, and connecting the terminals to the deposited silver.

10. The process of constructing a resistance unit comprising the steps of providing a non-conductive carrier, providing a metal resinate such as palladium resinate, coating the non-conductive carrier with the palladium resinate, drying the said coated carrier for approximately 30 minutes at room temperature, heating the coated non-conductive carrier for approximately 30 minutes in a temperature range of 200° to 400° centigrade effecting a thin film of atomic deposit of palladium upon the non-conductive carrier, oxidizing the remaining portion of the palladium resinate in a temperature range of 400° to 750° centigrade for substantially one hour, providing terminals for the resistance unit, and connecting the terminals to spaced surfaces of the thin film of the palladium.

11. The process of constructing a resistance unit comprising the steps of providing a non-conductive carrier, providing a metallic organo-compound comprising an organic resinate of a substantially stable and substantially non-oxidizable metal, coating the non-conductive carrier with the metallic organo-compound, heating the coated non-conductive carrier to a temperature below the melting point and the oxidizing point of the metal to first atomically deposit a thin continuous film of the metal upon the non-conductive carrier and to secondly oxidize the carbonaceous residue remaining after the deposition of the thin film of metal, providing terminals for the resistance unit, and connecting the terminals to spaced surfaces of the thin film of the metal.

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