July 30, 1968 E. H. MAYER ETAL 3,395,089

METHOD OF DEPOSITING FILMS OF CONTROLLED SPECIFIC RESISTIVITY
AND TEMPERATURE COEFFICIENT OF RESISTANCE
USING CATHODE SPUTTERING

Filed Dec. 14, 1964

FIG. 1

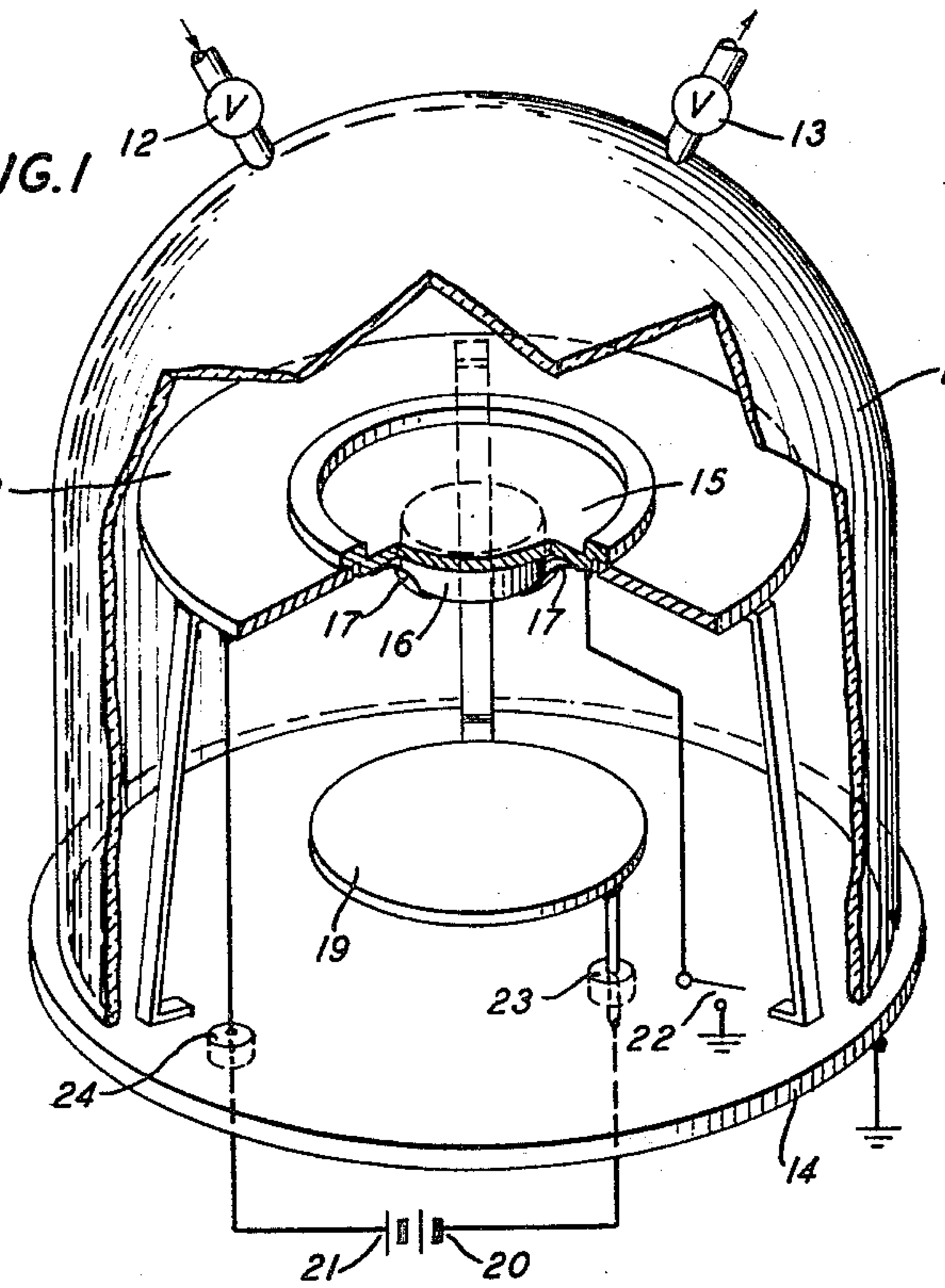
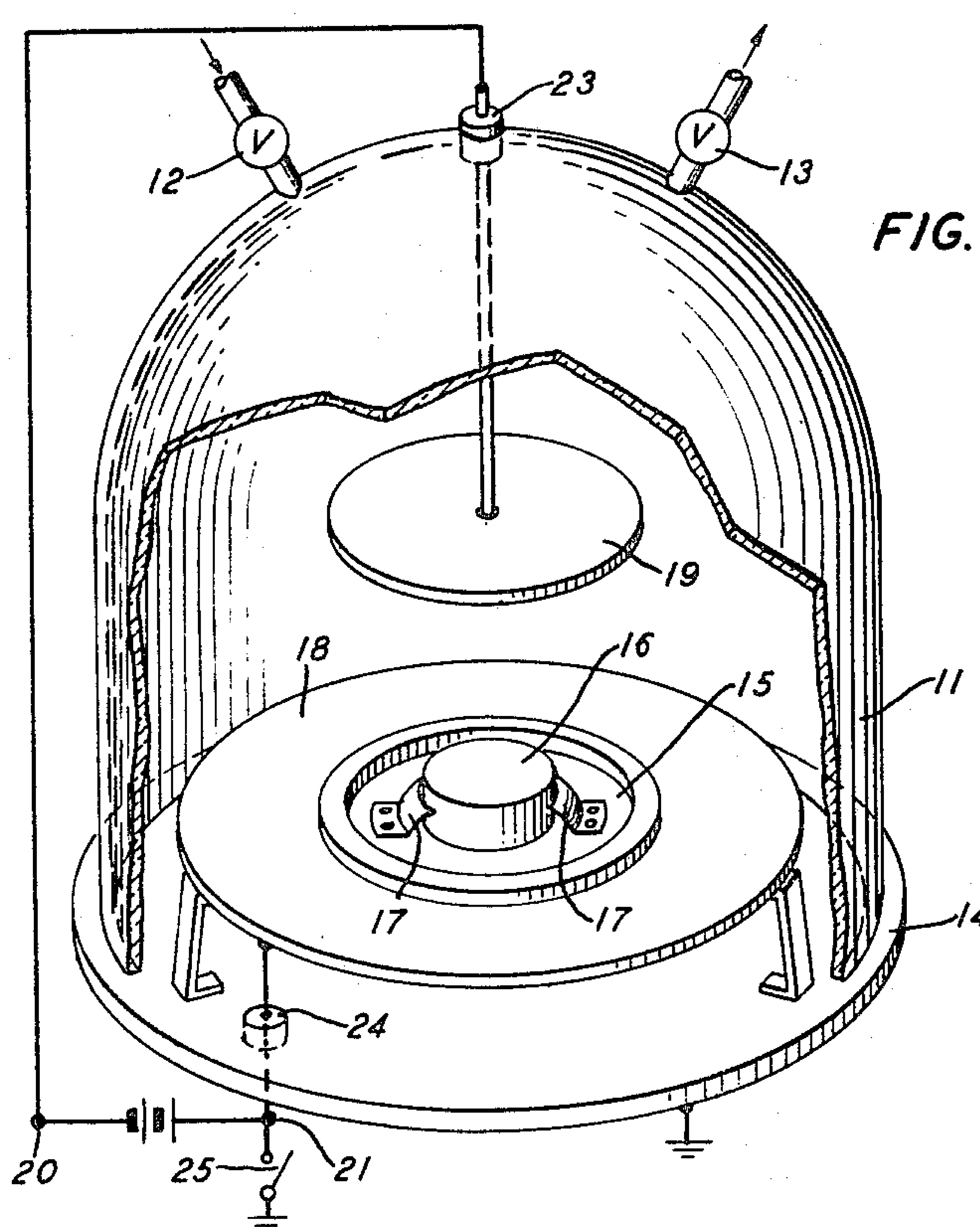


FIG. 2



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United States Patent Office 3,395,089
Patented July 30, 1968

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3,395,089

METHOD OF DEPOSITING FILMS OF CONTROLLED SPECIFIC RESISTIVITY AND TEMPERATURE COEFFICIENT OF RESISTANCE USING CATHODE SPUTTERING

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Filed Dec. 14, 1964, Ser. No. 418,142
2 Claims. (Cl. 204—192)

ABSTRACT OF THE DISCLOSURE

The electrical properties of thin films deposited by cathodic sputtering techniques may be controlled either by varying electrode configurations or by adjustment of the boundary of the dark space, the described configurations including a substrate holder which is maintained either in a floating state or at ground potential.

The present invention relates to a technique for the deposition of thin films by cathodic sputtering.

In recent years, considerable interest has been generated in thin films and the preparation of such films by cathodic sputtering techniques. Unfortunately, it has frequently been noted that the specific resistivity and temperature coefficients of resistance of certain of these films are prone to variability and nonuniformity. These difficulties have been controlled to a limited extent by painstaking control of background pressures, leak rates and deposition parameters; however, substantial variability is still found in the quality of the deposited films.

In accordance with the present invention, a technique for the deposition of thin films by cathodic sputtering is described wherein the electrical properties of deposited films are controlled by the use of apparatus having varied electrode configurations. More specifically, it has been determined that reproducibly low specific resistivities and high temperature coefficients of resistance are obtained by sputtering in a system including an anode member, a cathode member and a substrate holder wherein the substrate holder is either electrically isolated or maintained at ground potential, the anode and high voltage return being electrically isolated from ground.

In an alternative embodiment, a technique is described for the deposition of thin films manifesting a high degree of uniformity by suitable adjustment of Crooke's dark space in a system including a cathode member, an anode member electrically connected to the vacuum chamber and an electrically isolated substrate holder, the elements of said system being parallel to each other, so permitting the boundary of the dark space to be more apparent. More specifically, this end is attained by adjusting the vacuum until the outline of the dark space becomes flat and parallel to the substrate holder.

The invention will be more readily understood by reference to the following detailed description taken in conjunction with the accompanying drawing wherein:

FIG. 1 is a front elevational view, partly in section, of an exemplary apparatus suitable for the practice of the present invention; and

FIG. 2 is a front elevational view, partly in section, of another apparatus utilized in the practice of the invention.

With reference now more particularly to FIG. 1, there is shown a vacuum chamber **11** provided with an outlet **13** for connection to a vacuum pump (not shown), an inlet **12** for the introduction of a suitable sputtering gas, and a base plate **14** which is maintained at ground potential. Shown disposed within chamber **11** is a substrate holder **15**, to which a substrate member **16** is attached by means of clips **17**, an anode ring member **18** and a cathode member **19**, the latter being comprised of the material which is required to be deposited upon substrate member **16**. Cathode member **19** is connected to the negative pole **20** of a direct current high potential supply, the positive pole of which is connected to anode **18**, as at **21**. Substrate holder **15** may be connected to ground bias by closing switch **22** or may be permitted to float by leaving switch **22** in the open position. Cathode **19** and anode **18** are electrically isolated from base plate **14** by means of insulators **23** and **24**, respectively.

In an alternative configuration, shown in FIG. 2, anode **18** is connected to the positive pole **21** of a direct current high potential supply and to ground, substrate holder **15** being maintained at a floating potential.

The present invention may conveniently be described by reference to an illustrative example wherein it is desired to cathodically sputter any of the well known film-forming metals, for example, tantalum, niobium, titanium, zirconium, aluminum, et cetera, in an apparatus of the type shown in FIG. 1

The substrate member is first vigorously cleaned and then placed upon substrate holder **15**, the latter being composed of, for example, nickel, stainless steel, et cetera.

The vacuum techniques utilized in the practice of the present invention are known (see "Vacuum Deposition of Thin Films," L. Holland, J. Wylie & Sons, Inc., New York 1956). By this process, the vacuum chamber is first evacuated to as low a pressure as the system is capable of attaining, typically less than 1×10^{-6} torr, the substrate being heated during the pumpdown. Following pumpdown, an inert gas, for example, argon, helium, neon, et cetera, is admitted into the chamber, the inert gas input being controlled so as to dynamically stabilize the chamber pressure at the required sputtering value. The pressure required is dependent upon consideration of several factors which are well known to those skilled in the art. However, for the purposes of the present invention, a practical pressure would be within the range of 5×10^{-3} to 15×10^{-3} torr.

After the requisite pressure is attained, cathode **19** which may be composed of any of the above-noted film-forming metals, or, alternatively, may be covered with any of the film-forming metals, for example, in the form of a foil, is made electrically negative with respect to anode **18** which is isolated from the base plate **14**, substrate holder **15** being maintained either electrically isolated or in a grounded state.

The minimum voltage necessary to produce sputtering is dependent upon the particular film-forming metal employed. For example, a direct current potential of approximately 5000–6500 volts may be employed to produce a sputtered layer of tantalum suitable for the purposes of this invention, minimum voltages for other film-forming metals being well known to those skilled in the art. However, in certain instances it may be desirable to sputter at voltages greater than or less than the noted voltage.

The spacing between the substrate holder, anode and cathode is not critical. However, the minimum separation is that required to produce a glow discharge.

The balancing of the various factors of voltage, pressure and relative positions of the cathode, anode and substrate holder to obtain a high quality deposit is well known in the sputtering art. However, it will be appreciated that the main impact of the present invention lies in the discovery that the use of specific electrode electrical configurations during sputtering permits control of film parameters.

With reference now more particularly to the example under discussion, by employing a proper voltage, pressure and spacing of the various elements within the vacuum chamber, a layer of a film-forming metal is deposited upon the substrate member, sputtering being conducted for a period of time calculated to produce a film having the desired thickness.

In an alternative embodiment, alluded to hereinabove, deposited films manifesting a high degree of uniformity over a broad area may be obtained utilizing a configuration of the type shown in FIG. 2. The configuration of FIG. 2 differs from that of FIG. 1 in that the former presents the option of having the anode and high voltage return connected to ground through switch **25**.

In the operation of a process utilizing this configuration, pumpdown proceeds as described above. However, the pressure is adjusted so that the outline of the dark space is flat and parallel to the substrate holder after which time sputtering is continued while maintaining the above-described flat outline. Again, it will be understood that the pressures required are dependent upon consideration of several factors well known to those skilled in the art.

Several examples of the present invention are described in detail below. These examples are included merely to aid in the understanding of the invention and variations may be made by one skilled in the art without departing from the spirit and scope of the invention.

EXAMPLE I

This example describes the preparation of a sputtered tantalum film.

A cathodic sputtering apparatus similar to that shown in FIG. 1 was used to produce the tantalum layer. In the apparatus employed, anode **18** was an 8 inch stainless steel circular member designed with a 4½ inch by 4½ inch hole in which substrate holder **15** was mounted, holder **15** being electrically isolated from anode **18** and from ground. Cathode **19** was an 8 inch disc of 0.040 inch thick capacitor grade tantalum, the cathode being spaced approximately 2½ inches from the anode. Substrate **16** was a glass microscope slide previously cleaned by conventional cleansing procedures. The anode and high voltage return were maintained in an electrically isolated state, base plate **14** being grounded.

The vacuum chamber was initially evacuated to a pressure of the order of 10^{-6} torr. Argon was admitted until a dynamic pressure (in the bell jar) of 8×10^{-3} torr was obtained.

Following, a direct current voltage of 5000 volts was impressed between the cathode and anode and sputtering initiated. Sputtering was conducted at a deposition rate of 50 Angstroms per minute until a film of 1030 Angstroms thickness was produced. The resultant tantalum film evidenced a specific resistivity of 66 μohm-centimeters and a temperature coefficient of resistance of +960 p.p.m./° C.

EXAMPLE II

The procedure of Example I was repeated with the exception that a voltage of 5100 volts was impressed between the cathode and anode and sputtering continued until a film having a thickness of 1025 Angstroms was produced. The resultant film evidenced a specific resistivity of 50 μohm-centimeters and a temperature coefficient of resistance of 1100 p.p.m./° C.

EXAMPLE III

This example describes the preparation of tantalum sputtered films in an apparatus of the type shown in FIG. 1 wherein the substrate holder **15** was connected to ground. The configuration employed was identical to that described in FIG. 1.

The vacuum chamber was initially evacuated to a pressure of the order of 10^{-6} torr, argon having been admitted until a dynamic pressure (in the bell jar) of 9×10^{-3} torr was obtained.

Following, a direct current voltage of 5000 volts was impressed between cathode and anode and sputtering initiated. Sputtering was conducted at a deposition rate of 57 Angstroms per minute until a film of 1140 Angstroms thickness was produced. The resultant tantalum film evidenced a specific resistivity of 52 μohm-centimeters and a temperature coefficient of resistance of 1225 p.p.m./° C.

EXAMPLE IV

The procedure of Example III was repeated at a pressure of 11×10^{-3} torr of mercury with a 5100 volt difference of potential impressed between cathode and anode. Sputtering was conducted at a rate of 100 Angstroms per minute until a film of 1000 Angstroms in thickness was produced. The resultant film evidenced a specific resistivity of 51 μohm-centimeters and a temperature coefficient of resistance of 1196 p.p.m./° C.

EXAMPLE V

The procedure of Example IV was repeated at a pressure of 12.5×10^{-3} torr of mercury. Sputtering was conducted at a rate of 127 Angstroms per minute until a film 1270 Angstroms in thickness was produced. The resultant film evidenced a specific resistivity of 45 μohm-centimeters and a temperature coefficient of resistance of 1270 p.p.m./° C.

It will be noted that in each case the specific resistivity of the resultant sputtered films is approximately 50 μohm-centimeters as compared with resistivities of approximately 150 μohm-centimeters and higher for films sputtered in accordance with conventional sputtering techniques, such low resistivities being the basis for use in certain device applications.

EXAMPLE VI

This example describes the preparation of tantalum films having a uniformity of sheet resistivity within tolerances of ±1 percent, in an apparatus of the type shown in FIG. 2.

In the apparatus employed, anode **18** was a 16" nickel circular member designed with a 7½" x 7½" hole in which substrate holder **15** was mounted, holder **15** being electrically isolated from anode **18** and from ground. Cathode **19** was a 14" planar element of .050" thick capacitor grade tantalum, the cathode being spaced approximately 3½" from the anode. Substrate **16** was a 1 x 3" glass microscope slide previously cleaned by conventional cleansing procedures. The high voltage return from the anode was connected to ground.

The vacuum chamber was initially evacuated to a pressure of the order of 10^{-7} torr, argon was admitted until a dynamic pressure (in the bell jar) of 16×10^{-3} torr was obtained.

Following, a direct current voltage of 6500 volts was impressed between the cathode and the anode, so resulting in the formation of the well-known Crooke's dark space. The pressure was then adjusted by throttling or a change of flow rate to a pressure of 20×10^{-3} torr, so resulting in a dark space approximately one-half the inner electrode spacing. Next, the pressure was rapidly decreased to 13×10^{-1} torr at which time a dome shape was observed over the substrate holder. Next, the pressure was increased to 16×10^{-3} torr, thereby causing the dark space to become flat and parallel to the substrate holder. Sputtering was then continued at a deposition rate of 200 Angstroms per minute until a film of 1200 Angstroms in thickness was produced. The resultant tantalum film evidenced a sheet resistance of 15.00±0.1 ohms per square throughout the 6" x 6" surface area of the substrate material.

EXAMPLE VII

The procedure of Example VI was repeated nine times. The resultant tantalum films evidenced an average sheet resistance distribution of less than ±0.8 percent, respectively.

While the invention has been described in detail in the foregoing specification and the drawing similarly illustrates the same, the aforesaid is by way of illustration only and is not restrictive in character. The several modifications which will readily suggest themselves to persons skilled in the art are all considered within the scope of this invention, reference being had to the appended claims.

What is claimed is:

1. A method for controlling specific resistivity and temperature coefficient of resistance of a resistor in the deposition of thin films of a metal selected from the group consisting of tantalum, niobium, titanium, zirconium and aluminum by cathodic sputtering in a vacuum chamber having a grounded electrically conductive portion, which comprises the steps of evacuating said vacuum chamber in which there is disposed a cathode member, an anode member and a substrate holder, the said substrate holder being electrically isolated from said anode and said cathode members and electrically connected to the conductive portion of said vacuum chamber, and applying an electric potential across the said anode and cathode members, so resulting in the formation of said glow discharge with said substrate holder, said members and said conductive portion being substantially axially aligned with said discharge and the initiation of sputtering, the anode and high voltage return being electrically isolated from ground and adjusting the electrical characteristics of the discharge to control the specific resistivity and temperature coefficient of resistance in said film.

2. A method in accordance with the procedure of claim 1 wherein said thin film comprises tantalum.

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