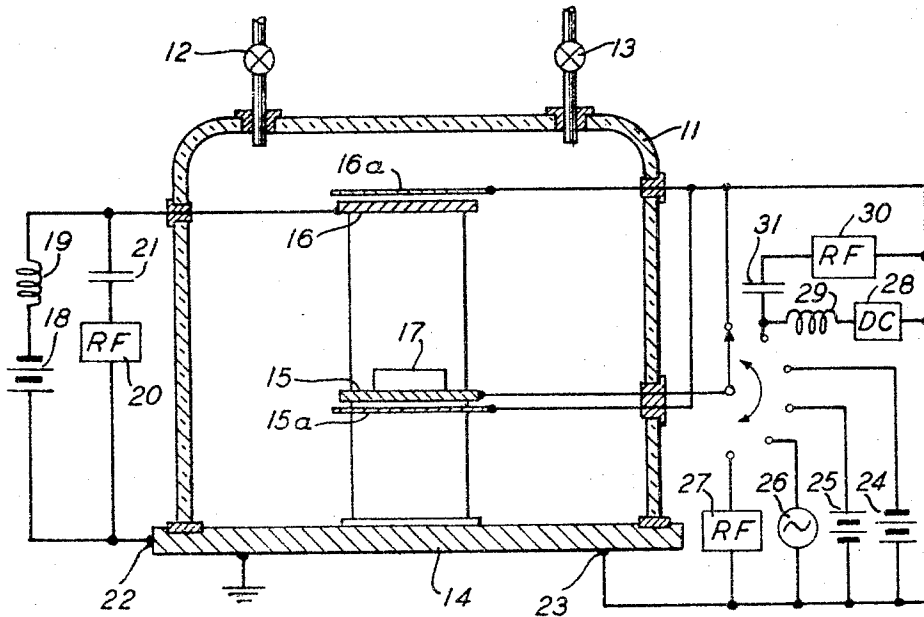


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CATHODIC SPUTTERING FROM A CATHODICALLY BIASED TARGET
ELECTRODE HAVING AN RF POTENTIAL SUPERIMPOSED
ON THE CATHODIC BIAS
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CATHODIC SPUTTERING FROM A CATHODICALLY BIASED TARGET ELECTRODE HAVING AN RF POTENTIAL SUPERIMPOSED ON THE CATHODIC BIAS

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ABSTRACT OF THE DISCLOSURE

Enhanced deposition rates are attained during conventional cathodic sputtering or dielectric oxide reactive sputtering in a system wherein a d-c potential and RF excitation are simultaneously applied at the cathode. The resultant increased deposition rates permit operation of the process at substantially lower sputtering pressures than employed heretofore, so avoiding an apparent source of impurities.

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 the electronics industry in thin film components and circuits prepared by cathodic sputtering procedures. Although widely practiced, certain limitations have precluded total exploitation of the sputtering technique. Thus, limited deposition rates during conventional and reactive sputtering, control of thickness uniformity, pressure limitations, et cetera, have adversely affected electrical and structural parameters.

In accordance with the present invention, a technique for enhancing the deposition rate during conventional or dielectric reactive sputtering is described wherein the aforementioned drawbacks are appreciably lessened. The inventive technique involves sputtering in a system in which there are three electrodes, an anode, a cathode and a third electrode, and in which the cathode member is simultaneously biased with a direct-current potential and RF excitation, the anode member being maintained at ground potential or biased at least ± 1 volt with respect to the third electrode. The described technique departs from conventional diode sputtering and bias sputtering as described in copending application Ser. No. 372,537, filed June 4, 1964, now abandoned, in one major aspect, namely, the simultaneous application of a direct-current potential and RF excitation to the cathode.

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

The figure is a schematic representation of an exemplary apparatus suitable for the practice of the present invention.

With reference now more particularly to the figure, there is shown a vacuum chamber 11 provided with an outlet 12 for connection to a vacuum pump (not shown), an inlet 13 for the introduction of either an inert or reactive gas or mixtures thereof during the sputtering process, and a base plate 14 which serves the purpose of a third or ground electrode. Shown disposed within chamber 11 is a substrate holder or anode member 15 and sputtering shield 15a and a cathode member 16 and sputtering shield 16a, member 16 being comprised of a material described hereinafter. Cathode member 16 is connected to the negative pole 18 or a direct-current high potential supply by means of inductor 19 and to an RF supply 20 by means of

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ponents as needed), the positive pole of the direct-current supply and one end of RF supply 20 being connected to base plate 14, as at 22. Anode member 15 may be connected to (a) base plate 14, as at 23, (b) the negative pole 24 of a direct-current source, the positive pole of which is connected to base plate 14 (as at 23), (c) the positive pole 25 of a direct-current source the negative pole of which is connected to base plate 14 (as at 23), (d) a source of alternating current 26, one side of which is connected to base plate 14 (as at 23), (e) an RF supply 27, one side of which is connected to base plate 14 (as at 23) or (f) a direct-current high potential supply 28 by means of inductor 29 and an RF supply 30 by means of capacitor 31, one end of said direct-current supply and said RF supply being connected to base plate 14 (as at 23).

The present invention may conveniently be described by reference to an illustrative example wherein it is desired to cathodically sputter manganese or 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 the figure. Further, the inventive technique may be utilized in the preparation of any of the oxides, nitrides and carbides thereof.

Substrate 17 is first vigorously cleaned. Conventional cleaning agents are suitable for this purpose, the choice of a particular one being dependent upon the composition of the substrate itself. Substrate 17 is then placed upon substrate holder 15, as shown in the figure, the latter being composed of a suitable conductor, typically, the material it is desired to deposit or any material compatible therewith. 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). In accordance with such procedures, the vacuum chamber is first evacuated, flushed with an inert gas, as for example, any of the members of the rare gas family such as helium, argon or neon and the chamber re-evacuated. The extent of the vacuum 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 initial pressure range is 10^{-5} to 10^{-7} torr, while suitable inert or reactive gas pressure during sputtering ranges from 10^{-4} to 10^{-1} torr.

After the requisite pressure is attained, cathode 16 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 base plate 14 and a source of RF excitation simultaneously impressed thereon.

The minimum voltage necessary to produce sputtering is dependent upon the particular film-forming metal employed. For example, a direct-current potential of approximately 1,000 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.

With regard to the RF excitation, it has been found that in order to produce the desired effect the frequency employed must be at least 0.1 megacycle and may range up to the plasma frequency which is defined by the following equation:

wherein

n =electron density

e =electron charge

ϵ_0 =dielectric constant of material sputtering, and

m =effective electron mass.

The use of frequencies less than 0.1 megacycle fail to significantly enhance the operation of the process since the plasma density is not appreciably increased whereas the plasma frequency, as defined above constitutes the absolute maximum beyond which the system shuts down. The potential of the RF source may range from 1 volt to 10 kilovolts, the limits being dictated by practical considerations.

The next step in the invention procedure involves applying a potential to substrate holder 15 and substrate 17 whereby they are maintained at ground potential or made electrically negative or positive with respect to base plate 14, as described above. This end may be attained by applying (a) a ground potential, (b) a positive or negative direct-current potential, (c) an alternating-current potential, (d) an RF potential or (e) an RF and a direct-current potential to holder 15 by means described above.

For the purposes of the present invention, it has been found that if holder 15 is to be maintained at a potential (with respect to base plate 14) other than ground it may be at least ± 1 volt direct-current and range up to $\pm 1,000$

grounded, the cathode was biased at 4 kilovolts negative with respect to ground and an RF supply of 7.05 megacycles was impressed thereon.

A glass microscope slide was used as the substrate. The slide was washed in a nonionic detergent followed by a sequential rinse in water, hydrogen peroxide and distilled-deionized water. The tantalum cathode was employed in the form of an arc melted ingot slab.

The vacuum chamber was initially evacuated to a pressure of the order of 10^{-6} torr, flushed with argon and re-evacuated to a partial argon pressure of 20 millitorr.

The substrate holder and cathode were spaced 3 inches apart, the substrate being placed upon the former. A direct-current voltage of 4,000 volts was impressed between the cathode and base plate 14 and an RF supply of 7.05 megacycles at 1 kilovolt impressed upon the cathode.

Sputtering was conducted for 30 minutes, so resulting in a tantalum film 2,830 angstroms thick, the deposition rate being approximately 94.2 angstroms/minute. The resultant film evidenced a specific resistivity of 51 microhm-cm.

For comparative purposes the procedure described above was repeated with and without RF and with and without direct current varying the parameters slightly. The results are set forth in the table below.

TABLE

Example	Cathode	Gas	Pressure (millitorr)	RF Freq. (mc.)	D.C. at cathode	Deposition rate, A./min.
1.....	Ta	Ar	20	7.05	4,000	94.2
2.....	Ta	Ar	20	0	4,000	80
3.....	Ta	Ar	20	7.05	0	0
4.....	Ta	O ₂	20	0	4,000	0.5
5.....	Ta	O ₂	20	7.05	0	0
6.....	Ta	O ₂	20	7.05	4,000	57.9
7.....	Ta	Ar	1.5	0	4,000	(¹)
8.....	Ta	Ar	1.5	29.7	4,000	76
9.....	Mn	O ₂	20	0	4,000	5
10.....	Mn	O ₂	20	7.05	4,000	100

¹ No Discharge.

volts on the positive side and approximately $-5,000$ volts on the negative side. Alternatively, an alternating-current potential ranging up to 5,000 volts may be applied to the ungrounded substrate holder, so attaining similar results. The RF requirements in the remaining alternatives are as previously discussed.

The spacing between the substrate holder (anode) and cathode is not critical. However, the minimum separation is that required to produce a glow discharge. For the best efficiency during the sputtering process, the substrate should be positioned immediately without the well known Crooke's dark space.

The balancing of the various factors of voltage, pressure and relative positions of the cathode and substrate holder to obtain a high quality deposit is well known in the sputtering art.

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 substrate 17 or a dielectric oxide layer is deposited thereon depending upon the sputtering gas employed. Sputtering is conducted for a period of time calculated to produce the desired thickness.

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

From the data, it was established that metal films cannot be deposited by the sole application of either low frequency or RF excitation at the cathode nor can dielectric oxide films be deposited by the sole application of low frequency excitation at the cathode using an oxygen reactive atmosphere. On the other hand, the simultaneous use of a negative potential and RF excitation at the cathode produces higher deposition rates of metal films than conventional direct-current cathodic sputtering and higher deposition rates of dielectric oxide films than by conventional reactive sputtering. As a result of the higher deposition rate at conventional sputtering pressures (20 millitorr) it is possible to decrease sputtering pressures to a magnitude of 0.1 millitorr and obtain conventional deposition rates.

While the invention has been described in detail in the foregoing specification, it will be appreciated by those skilled in the art that a screen plate or ring assembly and magnetic field may be employed for further increasing the plasma density and sputtering rate.

What is claimed is:

1. A method for the deposition of thin films upon a substrate by cathodic sputtering in a vacuum chamber in which are disposed a first electrode which is designated a target electrode and serves as a source of material to be sputtered which is conductive in nature, a second electrode having a substrate positioned thereon and a third electrode which serves as a reference electrode which comprises the steps of evacuating the said vacuum chamber, admitting a sputtering gas thereto and simultaneously biasing said first electrode with RF excitation having a potential of at least one volt at a frequency of at least 0.1 megacycle and a negative direct current, said first electrode being negative with respect to said third

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2. A method in accordance with claim 1 wherein said second electrode and said third electrode are maintained at the same potential.

3. A method in accordance with claim 1 wherein said second electrode is biased positive with respect to said third electrode.

4. A method in accordance with claim 1 wherein said second electrode is biased negative with respect to said third electrode.

5. A method in accordance with claim 1 wherein said second electrode is biased with RF excitation with respect to said third electrode.

6. A method in accordance with claim 1 wherein said second electrode is simultaneously biased positive with a direct-current potential and RF excitation with respect to said third electrode.

7. A method in accordance with claim 1 wherein said second electrode is simultaneously biased negative with a direct-current potential and RF excitation with respect to said third electrode.

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8. A method in accordance with claim 1 wherein said second electrode is biased with an alternating current ranging up to 5,000 volts with respect to said third electrode.

9. A method in accordance with claim 1 wherein said third electrode is a structural member defining the inner surface of said vacuum chamber.

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