

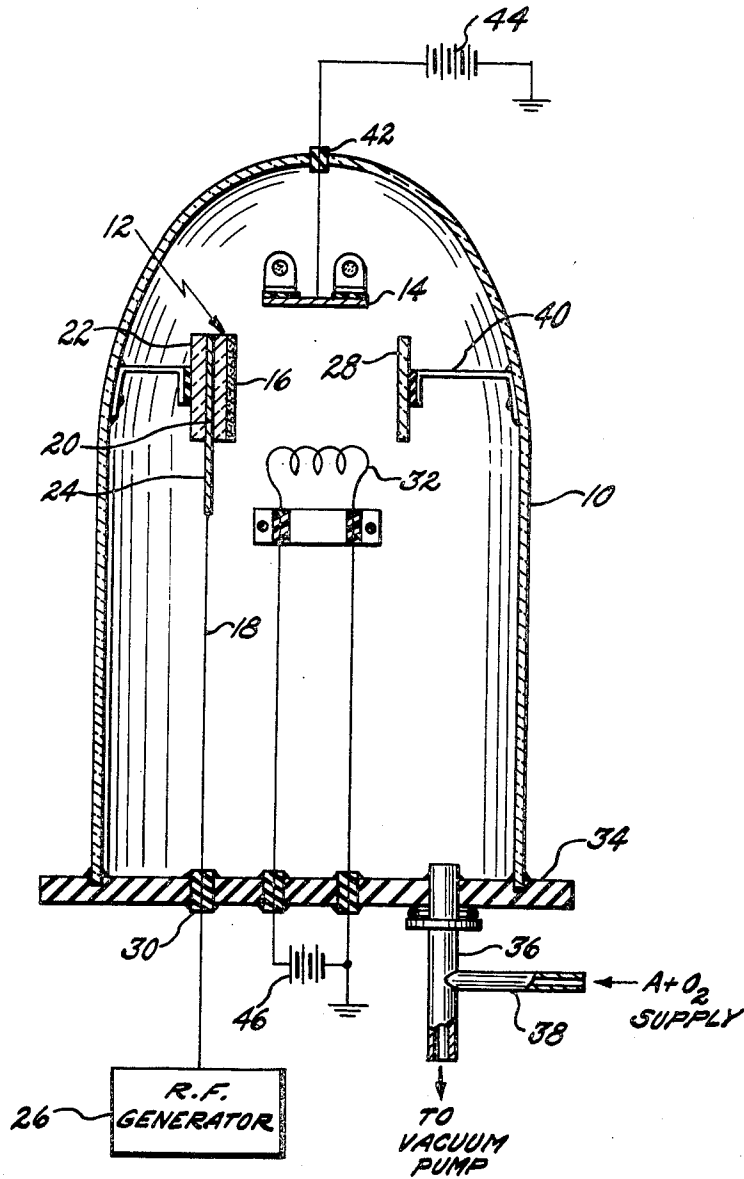
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PREPARATION OF THIN CERMET FILMS BY RADIO FREQUENCY SPUTTERING

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PREPARATION OF THIN CERMET FILMS BY RADIO FREQUENCY SPUTTERING

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ABSTRACT OF THE DISCLOSURE

Cermet films are prepared by forming a target for sputtering by coating an electrically nonconductive refractory base with a mixture including a ceramic component and a metallic component and then firing the coated base to form a layer of cermet superimposed on the base. A radio-frequency potential is then applied to the target while contacting a sputtering discharge supported by an anode and a thermionic cathode, causing the cermet layer to sputter-deposit on a desired substrate.

The invention described herein may be manufactured and used by or for the United States Government for governmental purposes without payment to me of any royalty thereon.

This invention relates to cathodic sputtering of metal and dielectric films. In a more particular aspect, this invention relates to an improved method for the cathodic sputtering of cermet type films within a plasma generated by a radio frequency field. Cermets are composite materials consisting of an intimate mixture of ceramic and metallic components, usually in the form of powders.

The ceramic component may consist of metallic oxides, carbides, borides, silicides, nitrides or mixtures of these compounds; while the metallic component may include a wide variety of metals such as aluminum, cobalt, iron, chromium, silicon, silver, gold, platinum, palladium and others. The selection of the particular ceramic and metallic component depends on the contemplated application of the respective cermet. Friction parts, cutting and drilling tools, as well as nuclear reactor fuel elements have been made from cermets. Generally, cermets possess corrosion and high temperature resistance, together with high strength and high wear resistance. Heretofore, commercial cermet type glazes have been coated onto various substrates by using conventional silk screen techniques. Such techniques produce a relatively thick film which does not find utility, for example, in the production of silicon based micro-circuits.

With the present invention, however, it has been found that thin uniform cermet films can be deposited upon various substrates using the radio frequency sputtering technique of this invention. The films produced by the method of this invention find special utility in the production of micro-circuits for low power circuits and analog circuits.

The primary object of this invention is the provision of an improved method for effecting the deposition of cermet materials onto a substrate.

A further object of this invention is the provision of an improved method for effecting the deposition of thin uniform layers of cermet materials upon a substrate.

Another object of this invention is the provision of an improved method for effecting the deposition of cermet films onto a substrate by means of a sputtering technique.

Other objects, advantages and features of the invention will be pointed out in the following detailed description, taken in conjunction with the accompanying drawing which depicts an apparatus suitable for use in the practice of the method of this invention.

Referring to the drawing:

The figure is a schematic representation of a cathodic sputtering apparatus suitable for depositing thin films of cermet materials.

In its broad aspects the invention disclosed herein contemplates the production of thin uniform films of cermet materials onto a substrate base by maintaining the target substrate surface under controlled ionic bombardment during sputtering so as to effect removal from said surface a quantity of sputtered material which will condense on a substrate to a given thickness.

More particularly, in the practice of this invention, deposition is accomplished by placing the source for the material to be deposited as a separate electrode in a low pressure, high density gas discharge plasma with low background impurity pressure between the cathode and an anode. The electrode, which provides the cermet material to be deposited, and the substrate are positioned opposite to each other and are subjected to ionic bombardment to effect deposition of the cermet material onto the substrate surface. The method consists of maintaining a desired vacuum within a vacuum chamber which has positioned therein an anode and a cathode. The target consists of a ceramic substrate upon which a cermet glaze has been fired. A tungsten filament is also disposed within the vacuum chamber and sufficient bias is supplied to the filament to cause electron emission. The filament is then biased cathodically so as to function as a thermionic cathode. The cermet coated target electrode is connected to an RF generator. A desired frequency, subject to FCC control, is applied to the target electrode while immersed in the plasma, thereby causing sputtering of the cermet material. A substrate placed within the discharge will then pick up the sputtered cermet material effecting deposition of the cermet onto the substrate surface.

Referring more particularly to the drawing, there is shown in the figure an apparatus suitable for depositing a thin uniform film of cermet material through the method contemplated by this invention. Shown in the figure is a vacuum chamber 10 in which are disposed a ceramic target electrode 12 and an anode 14. The target electrode 12 may be composed of an electrically non-conductive base such as alumina, Vycor or quartz and is coated with a cermet glaze 16 which has been either sprayed, screened or painted thereon and fired at the recommended temperature, usually about 800° C. The cermet target electrode 12 is connected to a metal electrode 18. A sheet of aluminum foil 20 is placed on the back of the ceramic cathode and is held there by a backer plate 22 of the same material that is used as the substrate of the target electrode 12. A tab of aluminum 24 is allowed to extend from this sandwich. Through this tab the radio frequency energy from an RF generator 26 is fed to the target electrode 12, thereby causing sputtering of the cermet glaze 16 onto a desired substrate 28. Suitable substrates for use in this invention are glazed alumina or passivated silicon. The RF generator 26 is positioned outside the vacuum chamber 10 but is connected thereto in a sealed relationship by rubber gasket 30. Opposite the anode 14 is a thermionic cathode comprising a tungsten filament 32. A platform 34 of non-conductive material is disposed in a sealed relationship for supporting the vacuum chamber 10. The vacuum chamber 10 may be a bell jar or some other suitable container. Provision is made for evacuating the chamber by means of a conduit 36 which is connected to a suitable vacuum pump, not shown. Provision is also made for admitting to the chamber 10 a mixture of argon and oxygen or other gases through conduit 38 during the sputtering process. The anode 14 which is electrically insulated by rubber gaskets 42 is biased by an electrical source 44. The substrate 28 which is to receive the cermet

glaze is supported adjacent the side of the bell jar 10 by conventional supporting means as shown at 40. The tungsten filament 32 is connected to a source of electrical energy 46.

In the operation of the method of this invention, the vacuum chamber 10 is first evacuated, flushed with an inert gas as, for example, any of the rare gas family, such as helium, argon or neon, and the chamber then re-evacuated. The system is now pumped down to 1×10^{-6} torr and backfilled with the sputtering ambient. An argon-oxygen gas mixture is used if the cermet has a metal oxide basis to a pressure of about 15.0×10^{-2} torr. Again, the system is pumped down but only to 5×10^{-2} torr if a glow discharge is to be used or to 5×10^{-3} if a low energy electron discharge is used. A small positive bias of about 40 volts is placed on the anode 14. The tungsten filament 32 is positioned opposite the anode 14 and sufficient bias is applied to cause electron emission. The target electrode 12, consisting of the ceramic substrate to which the cermet glaze has been fired on, is placed within the plasma generated by the slow electrons at 1-10 microns of mercury pressure. The target electrode 12 is backed by the metal electrode 18 which is shielded from the plasma and is connected to an RF generator 26 of about 1 kilowatt. A frequency of about 40 mc. (subject to FCC control) is applied to the cathode 12 while immersed in the plasma, thereby causing sputtering of the cermet material. A substrate 28 placed within the discharge will then pick up this sputtered material.

For the method of this invention, it is preferred to employ a sputtering source anode bias of about 40 volts, a pressure of from 5×10^{-2} torr to 5×10^{-3} torr and a frequency energy of about 40 mc. The spacing between the anode and the cathode is not critical.

For effective operation the tungsten filament should be shielded from the target to some extent in a low energy electron discharge which must be present for sputtering to occur. Balancing of various factors such as voltage, pressure and relative positions of the target, anode and substrate to obtain a high quality deposit is well known in the sputtering art.

An example of the present invention is described below. By employing proper voltage, pressure and spacing of the various elements within the vacuum chamber, a thin uniform film of an oxidized palladium-gold cermet with a ratio of 45 percent palladium and 55 percent gold can be deposited upon glazed alumina substrate. Sputtering is conducted for a specified time at a voltage of 40 volts and a pressure of 5×10^{-3} torr using an oxygen-argon gas mixture with the substrate positioned about four inches from the target.

The thickness of the palladium-gold cermet deposited on the substrate was within the range of from 500 to 1000 angstroms.

The sputtering target electrode of the example was composed of an alumina substrate on which had been fired the palladium-gold glaze referred to above. The palladium-gold glaze is either sprayed, screened or painted onto the alumina electrode and fired at a temperature of about

800° C. These cermets possess a resistivity of from 1 to 20,000 ohms per square per mil. The palladium-gold cermet used in the example was Glaze No. 6910. A frequency of about 40 mc. was applied to the sputtering cathode by means of the RF generator. Sputtering was conducted for a period of about one-half to one hour producing the thin uniform cermet film referred to above with a thickness of approximately 500 angstroms.

Circuitry for the operation of the low pressure plasma discharge system of the invention, as shown in the figure, and referred to in the example is well known in the art. The example and the apparatus described in the figure are included merely to aid in the understanding of the invention. Obviously, variations may be made by those skilled in the art without departing from the spirit of the invention, the scope of which is defined by the appended claims.

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 restricted in character. The several modifications which will readily show themselves to persons skilled in the art are also considered in the scope of this invention with reference being made to the appended claims.

What is claimed is:

1. A method for the deposition of a film of cermet material onto a work-piece surface comprising the steps of forming a cermet coated target electrode, including providing an electrically non-conductive refractory base, coating said base with a mixture of a ceramic component and a metallic component and firing said mixture at a temperature sufficiently high to form a coating of said cermet material positioning an anode, a thermionic cathode, and said target electrode within a vacuum chamber in a spaced relationship to each other, supporting said cermet coated target electrode and a work-piece within the vacuum chamber oppositely disposed to each other in a spaced relationship, introducing a low pressure ionizable gas atmosphere into the vacuum chamber, applying a potential to the said thermionic electrode to cause slow flow of electrons from said thermionic cathode to said anode thereby creating a low pressure gas discharge plasma within said vacuum chamber, and applying radio frequency energy to the cermet coated target electrode to effect sputtering of the cermet material onto the surface of the work-piece.

2. A method in accordance with claim 1 wherein said radio frequency energy is applied at about 40 megacycles.

3. A method in accordance with claim 1 wherein said atmosphere possesses a pressure of between one to ten microns of mercury.

No references cited.

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