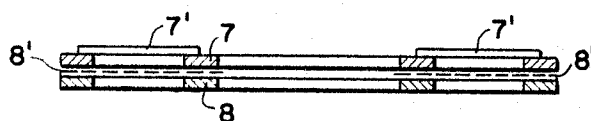
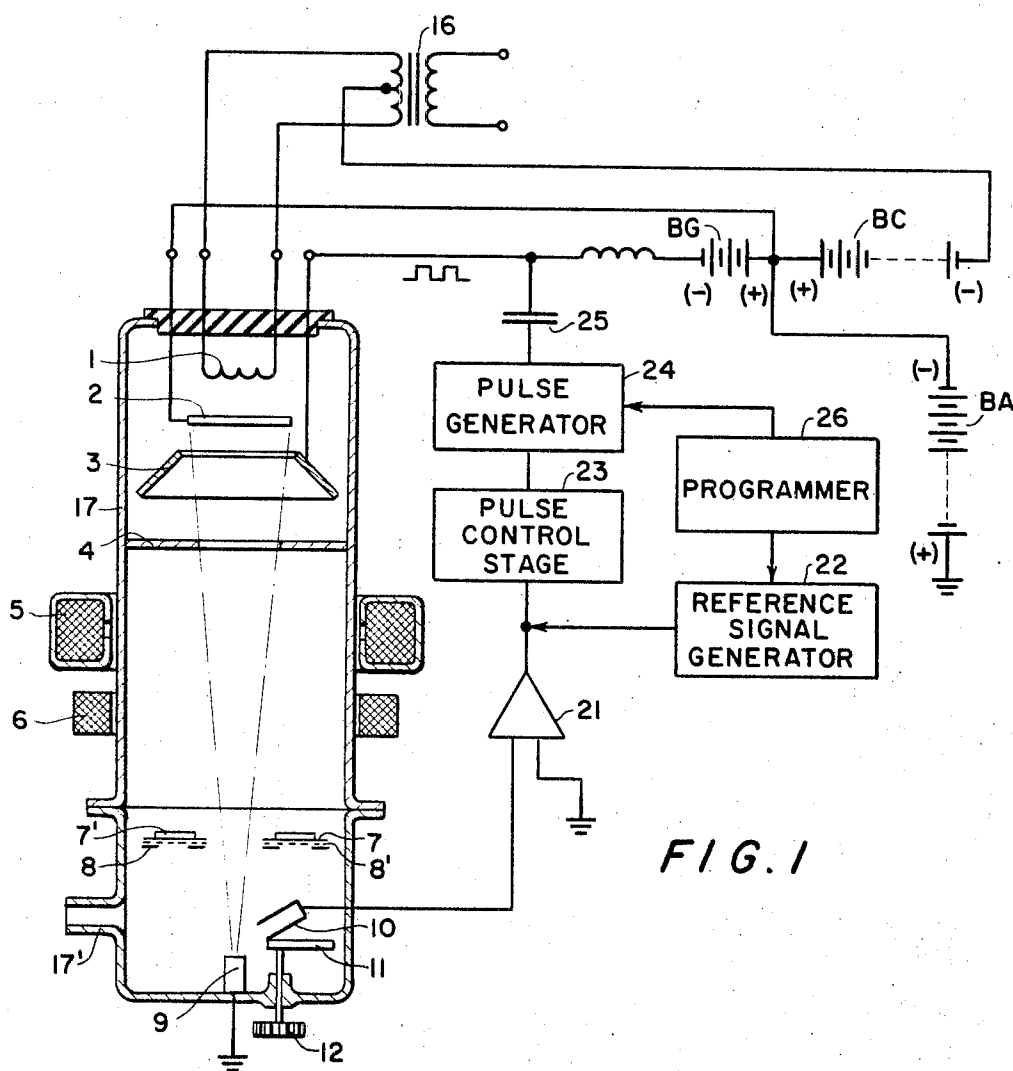


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METHOD AND APPARATUS FOR VAPOR DEPOSITION EMPLOYING AN ELECTRON BEAM

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Our invention relates to the production of thin layers or films of solid material by vaporizing the material and causing the vapor to condense on a substrate. More particularly, our invention concerns the vapor-deposition of material from a vaporous phase produced by bombarding a body of material with electrons from an electron gun.

When applying such vapor-deposition methods in the production of semiconducting, conducting, resistive or dielectric layers on various substrates in the manufacture of electrical or electronic components, such as resistors, capacitors, diodes or transistors, as well as in the vapor-deposition of materials for the purpose of producing electrical solid-state networks of active and inactive components known as microcircuits or microelectronic circuits, it is desirable to obtain highly uniform depositions together with accurate values of layer thickness and electrical properties.

It is, therefore, an object of our invention to devise a vaporization method and apparatus that reliably and readily afford the vapor deposition of solid layers or films upon substrates with the desired high degree of uniformity or constancy as regards electrical properties throughout the layer, and also permit obtaining a correspondingly high accuracy in layer thickness.

The use of an electron beam as a source of heat for vaporization of material is well known. However, the above-mentioned purposes require accurately controlling the amount of material being evaporated so as to maintain it constant during the processing period, and the evaporation process must be performed a definite length of time if the resulting layer is to be uniform in quality and of the desired, predetermined thickness. These conditions are difficult to meet when the vaporization of the material is effected by bombardment with electrons. This is because the amount of vaporization fluctuates with any change in position and size of the electron-beam spot, as may be caused by the rising or changing temperature of the electron gun and a resulting positional change of the electrodes.

It is, therefore, another, more specific object of our invention to obviate such difficulties and to afford producing a vapor-deposited layer or film of accurate thickness and uniform qualities by effecting a control and regulation of the heating and vaporizing action caused by an electron beam, without change in electron-gun temperature and without appreciable change in beam-spot size.

To achieve the above-mentioned objects, and in accordance with our invention, we direct an electron beam from a grid-controlled electron gun within a processing vessel onto a body of material, thus producing the necessary vaporous phase from which the formation of a solid film or layer by condensation on a substrate surface is to take place; and during such vaporization we apply to the beam-forming electrode or grid of the electron gun a pulsating bias voltage so that the electron beam has a correspondingly pulsating beam-current intensity. We further sense a given vaporization quantity in the vessel near the location of the substrates, and control the voltage pulses in dependence upon the sensed quantity to thereby main-

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tain a substantially uniform median rate of vaporization and deposition.

According to another, more specific feature of our invention, the grid bias voltage is essentially a squarewave voltage of substantially constant pulse amplitude, and the control imposed upon the pulse voltage by the sensor signal is caused to vary either the pulse width or the pulse repetition frequency or both. With such a control, the beam current is intermittent and, for a constant gun plate voltage, maintains a substantially constant spot size during the active pulse intervals while being virtually switched off during the inactive intervals.

The significance of these features, as well as further objects, advantages and features of the invention will be apparent from, and will be described in the following with reference to the accompanying drawing in which:

FIG. 1 shows schematically an apparatus according to the invention by a sectional view, in combination with a diagram of the appertaining electric control component; and

FIG. 2 shows details of the same processing vessel on a larger scale.

The illustrated apparatus is equipped with an electron gun which comprises a filament 1 to generate the electron current for bombarding the gun cathode 2. An electron-beam forming electrode 3 is disposed between the cathode 2 and the anode 4 of the electron gun. The electrode 3, often called "Wehnelt electrode" or "grid" performs a function comparable to that of the control grid in a triode tube in principle. Electron sources of this type are known as such (for example: Chapter 2 in the book, "Electron Optics and the Electron Microscope," by Zworykin et al., John Wiley & Sons, New York). While in the illustrated embodiment a Pierce-type electron gun is shown, any other controllable source of electrons, such as a kinescope electron gun, gradient electron gun or toroidal electron gun, may be used.

The anode 4, as a rule and as shown, is conductively connected with the metallic evaporator vessel 17 and is thus maintained at ground or zero potential. During operation, the cathode 2 is kept at a negative potential with respect to the anode 4 and evaporator vessel 17.

In the illustrated embodiment, the beam path, extending from the electron gun in the axial direction of the tubular vessel 17 toward the lower end thereof, is surrounded by electron-optical focusing means consisting of an electromagnetic lens 5, although it will be understood that an electrostatic lens, or a combination of suitable lenses, may be used. The vessel is further equipped with a deflection coil 6 which permits deflecting the electron beam away from the material to be bombarded. Mounted across the vessel is a substrate holder 7 which, as exemplified, may consist of a ring-shaped plate whose large central opening permits a free passage of the electron beam and which has smaller openings covered by respective substrates 7' when the device is in operation. A second holder structure 8 of similar design is mounted beneath the substrate holder for supporting respective evaporation masks 8'. The material to be evaporated is mounted in form of a solid body 9 at a location near the lower end of the tube so that the top surface of the body 9 is exposed by the electron beam concentrated upon the surface by means of the lens 5.

During operation of the device, the electron beam causes material from body 9 to vaporize. The vapor condenses on the exposed bottom surfaces of the substrates 7 and coats them with a layer whose shape is determined by the masks 8'. The amount or density of evaporation caused by the electron beam is sensed by a sensor or monitor head 10 mounted between the location of the body 9

and the mask holder 8 at a locality near the beam path but sufficiently spaced therefrom laterally to prevent interfering with the electron bombardment. A shutter 11 mounted on a shaft can be rotated from the outside of the vessel by means of a knob or gear 12. When the shutter 11 is turned into the path of the beam in front of the top surface of body 9, the beam is completely prevented from reaching the body 9.

The sensor 10 for detection and measurement of the evaporation pressure or density of the material may consist of any device known for such purposes, particularly for measuring vapor pressure. These devices operate on one of the following principles:

(A) Measuring the ion current by regularly ionizing the vapor of the material being evaporated.

(B) Using a mass spectrometer or mass filter to furnish a signal voltage indicative of the mass density.

(C) Detecting and measuring the changing resonance conditions of a quartz crystal due to material being vapor-deposited upon the surface of the crystal, the latter forming part of a quartz-crystal oscillator.

(D) Employing a transparent material, preferably a Mylar film or other plastic material, and measuring the transparency temperature of light passing through the transparent structure. This can be done by means of a photoelectric cell whose voltage output furnishes the desired signal.

(E) Using a microscopic micro-balancer for measuring the amount of vaporization.

Any of these sensing means, or generally any transducer capable of converting the measuring vaporization quantity into an electric quantity, is applicable for the purposes of the invention.

The illustrated sensor 10 is a vapor-pressure transducer and has its housing and thereby one of its output-voltage terminals connected with the grounded vessel 17. The other output terminal is connected by a lead with an amplifier 21. The amplified signal voltage from the sensor is compared with a reference voltage from a standard signal generator 22, the reference signal being set in accordance with a desired datum value of the evaporation density. The differential result of the comparison serves to maintain the evaporation density at the desired constant value. For this purpose the difference or error voltage is applied to a pulse-controlling converter stage 23 which issues a pulse controlled in dependence upon the error signal, with respect to the pulse frequency or pulse width (duration). The output pulse from converter stage 23 controls a pulse generator 24 which generates the necessary voltage amplitude required for controlling the power of the electron beam. The voltage pulses from pulse generator 24 are applied to the grid electrode 3 of the electron gun through a coupling capacitor 25. The capacitor 25 may be substituted by a coupling pulse transformer or other coupling means, if desired.

Before continuing the description of the control system exemplified on the drawing, it appears helpful to understand that there are several ways of applying the signal from the sensor 10 for controlling the gun-grid voltage pulses in dependence upon the evaporation pressure or density according to the invention.

One way is to control the mean power of the electron beam by normally applying a negative cut-off bias voltage to the grid electrode, and changing the repetition frequency of the positive voltage pulses having a constant amplitude and a constant pulse width, in response to the vaporization quantity supplied by the sensor. This particular mode of operation is embodied in the illustrated system, still to be more fully described.

Another way of vapor-deposition control according to the invention is to proceed in the same manner as described above, except that the pulse width instead of the repetition frequency is changed in response to the sensor signal.

A third way of performing the method of the invention

is to control the mean value of the electron beam power by normally applying to the electron-gun grid a positive constant bias voltage, and changing the repetition frequency of negative control pulses having a constant amplitude and a constant width, rated for cut-off of the electron beam current.

A fourth method of control according to the invention is to proceed in the manner last described but to change the pulse width instead of the repetition frequency.

With the first and second modes of operation, the electron beam issues from the electron gun onto the material to be vaporized only during those intervals in which the voltage pulses are applied to the grid electrode. With the third and fourth modes of operation described above, the electron beam is normally active but is interrupted during the interval of time in which the voltage pulses are being applied to the grid. In each case, the potential of the gun-grid electrode is constant when the electron-beam current is being issued. Consequently the size of the electron-beam spot stays constant regardless of the change in median electron-beam power thus effected.

As mentioned, the illustrated embodiment embodies the first mode of operation requiring a negative cut-off bias to be normally applied to the grid electrode 3 of the electron gun. This negative bias is supplied from voltage source BG.

The system further comprises a programmer 26 which sets the reference signal generator 22 to the desired datum value of reference voltage and also prescribes a desired evaporation period. The programmer is essentially a conventional monitor device operating with a rotating contactor, a punchcard or other carrier of controlling information that determines the course of the processing program to be performed by the vapor-deposition apparatus. As explained, the control system operates to maintain uniform vaporizing conditions and consequently a uniform quality condition in the material being precipitated upon the substrates. After elapse of a given timing period set by the programmer 26, the electron gun is switched off immediately, and the vapor-deposited film has now reached the predetermined thickness. If necessary or desired, the evaporation shutter 11 may then be placed in closed position. This may also be done under control by the programmer then acting upon a drive (not shown) for the gear 12. In the embodiment shown, the electron gun is simply cut off as soon as the transmission of pulses from the pulse generator 24 is terminated by action of the programmer 26.

The filament is energized from a alternating supply line through a filament transformer 16. A source of electron-generating voltage, denoted by BC, is connected between the filament 1 and the electron-emitting cathode 2. In principle, the filament itself may serve as the cathode so that a separate cathode 2 is not always required. However, it is preferable to provide a separate cathode structure, as shown, in order to produce a well-defined electron beam.

A source BA of plate voltage has its positive pole grounded and the negative pole connected to the cathode 2 in the manner shown.

The interior of the evaporator vessel 17 is maintained under negative pressure, preferably between 10^{-2} and 10^{-6} mm. Hg. For this purpose the vessel has an exhaust duct 17' for connection to a vacuum pump.

The method and apparatus described above with reference to the drawing is applicable for precipitating a great variety of metallic and non-metallic substances upon insulating or conducting substrates of different materials. For example, silicon can thus be precipitated upon substrates of silicon for producing p-n junction devices and microelectronic components or modules. Germanium can be precipitated upon substrates of germanium. Germanium or silicon can further be precipitated in the same manner and by the same equipment upon substrates of

respectively different materials, including glass, ceramics, oxide-coated metals and other insulators. Analogously, silver or copper can be precipitated from metallic bodies or compounds of these metals upon substrates of silicon or any of the other above-mentioned materials. It will be understood that the operating conditions are preferably adapted to each particular application. Particularly the pulse width and the pulse repetition frequency are preferably chosen in accordance with the evaporation material and the particularities of the control system used. For example, when operating with a constant pulse width of 10 μ s. (microseconds), the pulse-repetition frequency may be sensor-controlled to vary in the range of 100 c.p.s. to 75 kc.p.s. (kilocycles per second). These pulse data have been found applicable, for example, for the vapor deposition of silicon layers upon silicon substrates. Also applicable, for example, is a constant pulse width of 50 μ s. with a controlled repetition frequency in the range of 100 c.p.s. to 15 kc.p.s. The apparatus has further been operated with a constant pulse width of 100 μ s. and a controlled repetition frequency in the range of 100 c.p.s. to 75 kc.p.s., also with a constant pulse width of 500 μ s. and a controlled repetition frequency in the range of 1 c.p.s. to 15 kc.p.s. A pulse width of 1 ms. (millisecond) and a controlled repetition frequency of 1 c.p.s. to 750 c.p.s. has likewise been found applicable. Consequently the particular pulse data are not critical. It should be understood, however, and will be explained in the following, that the invention is analogously applicable with a fixed pulse repetition frequency and a sensor-controlled variable pulse width. As mentioned, the pulse voltage applied to the control grid of the electron gun has a substantially square wave shape with steep leading and trailing edges.

By virtue of the invention, as explained above, the evaporation quantity per unit time can be controlled with extreme accuracy with the aid of control system whose response time is short and precise. This affords maintaining any irregularities in evaporation at an extreme minimum, due to the fact that the electron beam power is controlled by virtue of the invention without changing the size of the electron beam spot. As a consequence, the qualities and thickness of the vapor-deposited film can thus be controlled with an accuracy of 1%, for instance.

The significance of the improvement achieved by the invention will be further apparent from the following comparison with the methods heretofore used or contemplated for controlling the beam current of an electron gun in vapor deposition equipment generally of the type here involved. These other methods are based upon the principles of

(1) Changing the temperature of the electron-emitting cathode. According to this method, the electron gun is operated at temperatures varying over a limited range.

(2) Applying a positive or negative voltage to the grid (Wehnelt or beam-forming) electrode positioned between cathode and anode of the gun, and changing the applied voltage amplitude for controlling the beam current.

(3) Changing the anode voltage of the electron gun for controlling the electron-beam power.

The method (1) of variable temperature operation causes the electron path to change in accordance with the varying beam-current density. Therefore, the focal distance and the size of the electron-beam spot vary accordingly. Furthermore, a change in cathode temperature requires some amount of time so that the response is accordingly delayed.

The method (2) of changing the voltage applied to the grid electrode also results in changing the beam path and the beam spot in accordance with the varying voltage.

The method (3) of changing the gun-anode voltage does not lend itself for use with electron lenses for concentrating the beam onto the material to be vaporized. If, for example, an electromagnetic focusing lens is used,

the relationship between the ampere turns (NI), anode voltage (Va) of the lens and the focal distance (f) of the lens is as follows:

$$(NI)^2 = KV a \frac{2r}{f}$$

where r is the radius of the lens aperture, and K is a proportionality constant.

It will be recognized that the focal length (f) of the lens varies with changes in anode voltage (V_a) since the ampere-turn value (NI) of the lens is constant. This also applies to the use of electrostatic focusing lenses.

Since with each of these methods, the size of the beam spot changes, they leave much to be desired as regards the purposes of the present invention. That is, the varying size of the beam spot on the material being vaporized greatly aggravates or defeats the aim of attaining constant quality in the layer being deposited, since the characteristics of the vapor-deposited material differ with changes in composition and distribution of the evaporation pressure resulting from a change in size of the beam spot. This is particularly pronounced when the material being vaporized by electron bombardment is an alloy or a chemical compound.

In contrast thereto, if the power of the electron beam is controlled according to the invention by applying a pulse voltage to the grid electrode of the electron gun in the manner described above, the size of the electron-beam spot remains substantially constant so that the deficiencies of the above-mentioned other methods are obviated.

Upon a study of this disclosure, it will be obvious to those skilled in the art that our invention permits of many variations and modifications, and hence can be given embodiments other than particularly illustrated and described herein, without departing from the essential features of the invention, and within the scope of the claims annexed hereto.

We claim:

1. The method of depositing solid layers from vapor of the layer-forming material, which comprises directing an electron beam from a grid-controlled electron gun within a processing vessel onto a body of material to produce the vapor, subjecting a substrate to the vapor in the vessel for deposition of condensing material on the substrate, impressing during vaporization a pulsating grid voltage upon the electron gun to make the electron-beam current pulsate accordingly, sensing a given vaporization quantity in the vessel, and controlling the pulse voltage in dependence upon the sensed quantity to maintain substantially uniform quality of the precipitating material.

2. The method of depositing solid layers from vapor of the layer-forming material, which comprises directing an electron beam from a grid-controlled electron gun within a processing vessel onto a body of material to produce the vapor, subjecting a substrate to the vapor in the vessel for deposition of condensing material on the substrate, impressing during vaporization a pulsating grid voltage upon the electron gun so that the electron beam is also pulsating, translating a given vaporization quantity in the vessel into a signal voltage, comparing the signal voltage with a predetermined reference voltage and controlling the pulse voltage in accordance with the difference between signal and reference voltages to maintain the median vaporization rate at a constant value dependent upon said reference voltage.

3. The method of depositing solid layers from vapor of the layer-forming material, which comprises directing an electron beam from a grid-controlled electron gun within a processing vessel onto a body of material to produce the vapor, subjecting a substrate to the vapor in the vessel for deposition of material on the substrate, applying to the electron gun during vaporization a pulsating grid voltage of substantially rectangular wave shape and substantially constant pulse amplitude to cause a flow

of beam current only during a given one of each two successive pulse and interpulse intervals, sensing the resulting vaporization quantity in the vessel and translating the sensed quantity into signal voltage, and controlling the ratio of said two grid-voltage intervals in dependence upon said signal voltage.

4. The vapor-deposition method of claim 3, wherein said grid voltage has constant pulse width, and said signal voltage is applied for varying the pulse repetition frequency of said grid voltage.

5. The vapor-deposition method of claim 3, which comprises continuously applying to the electron a negative cut-off bias and superimposing thereupon said pulsating grid voltage, said grid voltage having a positive pulse amplitude to cause flow of the beam current during said pulse intervals.

6. The vapor-deposition method of claim 3, wherein said grid voltage has a constant pulse repetition frequency and said signal voltage is applied for varying the pulse width of said grid voltage.

7. The vapor-deposition method of claim 3, which comprises applying to the electron a continuous positive bias potential and superimposing thereupon said pulsating grid voltage, said grid voltage having a negative pulse amplitude for cut-off control of the gun, whereby the beam current flows only during the inter-pulse intervals of the grid voltage.

8. Apparatus for depositing solid layers from vapor of the layer-forming material, comprising a sealable processing vessel having means for maintaining it at negative pressure, an electron gun in said vessel, said gun having a beam-forming grid electrode, said vessel having a mounting location for material to be vaporized, said location being spaced from said gun in the beam direction, electron optical means for concentrating the beam onto said location, substrate holder means mounted in said vessel near said location for accommodating a substrate to be exposed to the vapor produced by the electron beam

from the material, sensor means responsive to the vapor density in said vessel, pulse-voltage supply means electrically connected to said grid electrode for controlling the electron beam current, and control circuit means connected between said pulse-voltage supply means and said sensor means for varying the voltage pulse in dependence upon the sensed quantity to maintain a substantially uniform median rate of vapor-deposition.

9. In vapor-deposition apparatus according to claim 8, said vessel having the shape of an elongated tube, said electron gun being mounted at one end of the tube and having a beam direction extending coaxially toward the other end of said tube, said material location being situated at said other end, said substrate holder means being mounted between said gun and said location and extending about said tube axis for free passage of the electron beam, and said sensor being mounted between said substrate holder and said locality in eccentric relation to the tube axis.

10. Vapor-deposition apparatus according to claim 8, comprising a source of bias voltage connected to said grid electrode for normally maintaining the beam current in one of its on-off conditions, said voltage pulse having substantially rectangular wave shape and a constant pulse amplitude of the polarity required to pass the beam current to said other condition.

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