Jan. 23, 1962 J. T. KRAUSE 3,017,851

VAPOR DEPOSITION APPARATUS

Filed Dec. 21, 1959

FIG. 1

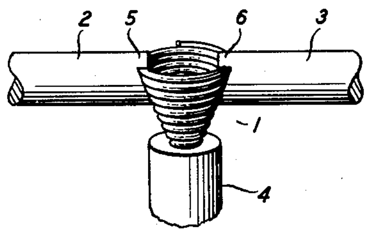


FIG. 2

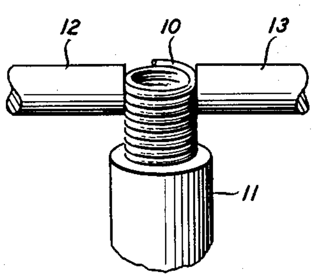


FIG. 3

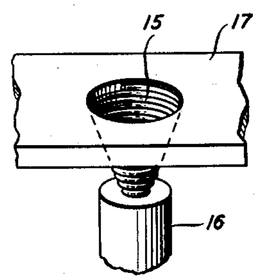
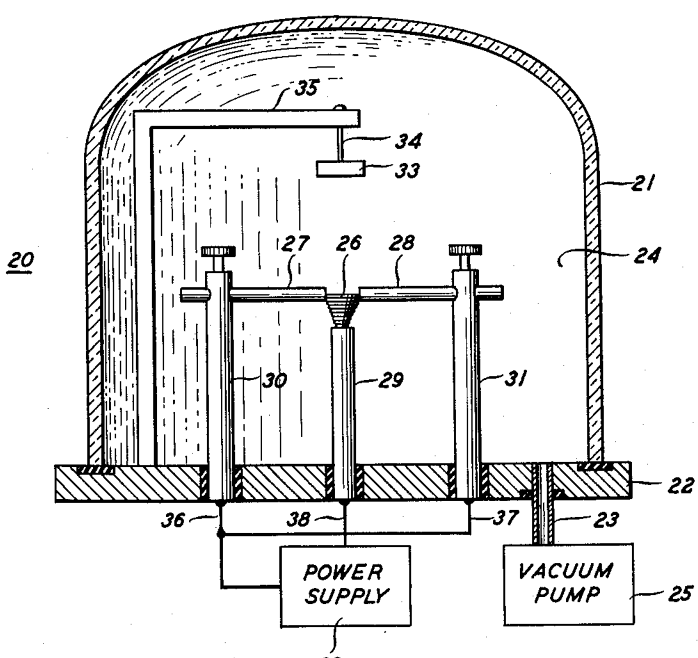


FIG. 4



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VAPOR DEPOSITION APPARATUS

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This invention relates to vapor deposition apparatus and, more particularly, to vapor deposition apparatus utilizing an improved resistance heated container.

Resistance heated containers of the filament type are extensively used in vapor deposition apparatus to hold source materials. Joule heating of these containers causes evaporation of the source material onto a substrate placed at a distance from the container. Typically, films in the order of 200 angstroms to 10,000 angstroms are formed on the substrate in this manner. Filament containers take the form of an open wound wire spiral having the configuration of a helix or conical basket. Current is applied to the container by attaching each end of the wire to an electrode. By using a wire made of a material that is relatively unreactive with the source material at elevated temperatures and has a higher melting point than the volatilization temperature source material, a large number of source materials can be evaporized. Typically, the wires are made of a refractory metal such as tungsten, tantalum, molybdenum, columbium, platinum, iron, nickel and "Chromel," an alloy of 90 percent nickel and 10 percent chromium. Tables found on pages 110 through 114 and 512 through 518 of L. Holland's book "Vacuum Deposition of Thin Films," 1956 edition, are exemplary of the many source materials that can be evaporated from filament containers. The electrodes, due to their resistivity and size, undergo negligible joule heating, thereby sharply confining the elevated temperatures to the region of the container itself. Radiant heating of the substrate material is minimized. As noted on page 2 of the above-mentioned book by Holland, this technique permits the deposition of films on both metallic and non-metallic substrates.

Filament type containers suffer several disadvantages, however, which limit their potential use. Since the container is in the form of an open wound spiral, only those source materials having a particle size greater than the spacing of the convolutions can be evaporated. If the particle size is smaller, the material must wet or be physically attached to the convolutions. Further, the danger of short circuiting of the individual windings by the source material is implicit in this design. Such short circuiting is undesirable since it causes the container to become more conductive than the wire leads on contact with the electrodes. Excessive heating of the leads coupled with a temperature drop in the container results. Normally, radiant heating of the substrate is minimized when filament containers are utilized since the area of joule heating is sharply confined to the container and wire leads. However, excessive heating of the wire leads due to the short circuiting of the container causes excessive radiation which can be a major problem when the substrate material is heat sensitive. Excessive heating of the wire leads may also cause these wires to melt before the source material in the container has volatilized.

Briefly, in accordance with the present invention there is described an improved resistance heated container in which source materials are evaporated. The container consists of successive convolutions of a wire formed of a refractory metallic material. Provision is made for applying a current to the container and supporting the container in a fixed geometrical position by at least two electrodes, one electrode contacting the base of the container and the other electrode contacting the periphery

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of the container at at least one point spaced from the base of the container. The convolutions are tightly wound so that successive convolutions either initially or because of the pressure exerted by the electrodes touch each other. The container is susceptible of many embodiments. For example, it can take the form of a cone or a cylindrical helix.

The configuration of the resistance heated container of the instant invention prevents loss of the source material. Therefore the particle size of the source material is immaterial and it is not necessary for the material to adhere to the container. Due to its geometry the container is deliberately short circuited. However, such short circuiting is immaterial since by eliminating the lead-in wires the container is made a unit in itself entirely separate from the electrodes. The area of electrical resistance, and therefore joule heating, is as a result sharply confined to the container. The geometry permits the attainment of high temperatures with a minimum of heat radiation. High melting point source materials can therefore be evaporated and deposited on heat sensitive substrate materials. Reproducibility of a given evaporation is possible due to the rigid geometric position in which the container is held by the electrodes.

The parameters governing evaporation from filament containers have been thoroughly investigated and are well understood by the art. In general, vapor deposition is done under a vacuum which is obtained by pumping. In some instances the apparatus is first flushed with an inert gas and then pumped. Generally, atmospheres of 10^{-2} and 10^{-6} millimeters of mercury are maintained in the deposition apparatus which may be a bell jar 18 inches to 10 feet in height. In accordance with accepted procedure, the maximum distance between the substrate and the container approximates the mean free path of the evaporant molecules as determined by the partial pressure in the apparatus. The substrate area that can be coated is dependent upon the particular processing conditions utilized, such as distance between the substrate and the container, and the container angle. This area can be calculated from various design equations, for example those discussed in the beforementioned book by L. Holland, pages 144 through 148, and also found in "Procedure in Experimental Physics," by John Strong, 1938 edition, pages 177 through 187. As an example, when a 90 degree conical container and a point source are utilized, the diameter of the covered area, defined as that portion over which the thinnest layer is 20 percent of the center layer, is twice the distance of the substrate from the container. Smaller evaporation angles result in more uniform layers. Larger angles are limited by the structural rigidity of the container and the criticality of edge thickness. Based on these considerations, a practical conical container angle range would be from five degrees to 90 degrees.

Utilizing these considerations, and knowing the area and thickness of the desired layer, the amount of source material required can be readily calculated. This source material will in turn dictate the container size. The wire material from which the container is fashioned is determined by the source material. The wire material should relatively unreactive with the source material and exhibit joule heating sufficient to vaporize the source material without undergoing melting itself. The electrodes contacting the container, due to their resistivity and size, exhibit a minimum of joule heating thereby sharply confining the elevated temperatures to the region of the container itself. Typically, a 0.01 to one ratio of joule heating of one leg of the electrical circuit, delineated for example by the bottom electrode, to the joule heating of the container has been found satisfactory in confining joule heating to the container. This ratio may, of course, be varied without causing excessive heating of the electrode or melting of the container, both of which can be determined visually. It is apparent that the joule heating exhibited by the various electrodes may vary in relation to each other while still minimizing overall joule heating. Preferably the electrodes exhibit a one to one ratio of the joule heating of the bottom electrode, which forms one leg of the electrical circuit, to the combined joule heating of the electrode or electrodes contacting the periphery of the container, which forms the other leg of the electrical circuit. The ratio may also be varied without causing excessive joule heating of one leg of the electrical circuit.

A more complete understanding of the features of this invention together with additional objects thereof may be gained from the reading of the following description in conjunction with the accompanying drawing, in which:

FIG. 1 is a perspective view of one container of the present invention;

FIG. 2 is a perspective view of another container of the present invention;

FIG. 3 is a perspective view of another container of the present invention; and

FIG. 4 is a schematic front elevation view partly in section of a typical deposition apparatus including one container of the present invention.

Referring now more particularly to FIG. 1, there is shown a conical container **1** having an angle of, for example, five to 90 degrees formed of successive convolutions of a tightly wound wire of tungsten or other refractory metallic material. Container **1** is supported and held in a fixed geometrical position by electrodes **2**, **3** and **4**. Electrode **4** is machined to receive the apex of container **1**. Although not shown, electrode **4** may be mounted on a bias spring to insure intimate contact of electrodes **2**, **3** and **4** with container **1**. Electrodes **2** and **3** are machined to receive the periphery of convolutions spaced from the apex of container **1**. Preferably, a point contact between these electrodes and the container is to be avoided since the tips of the electrodes might excessively heat thereby creating a hot spot on the container which could cause that region of the container to melt. Electrodes **2** and **3** contact the periphery of container **1** above that portion of the container holding the source material to insure that the source material is uniformly heated. Preferably electrodes **2** and **3** contact the upper portion of container **1** to insure that substantially all of the container is heated. This lessens the chance of the evaporated source material condensing on the upper portions of the container. To avoid short circuiting of container **1** electrodes **2** and **3** are spaced from the apex of the container. In the embodiment shown in FIG. 1, electrodes **2** and **3** have protruding lips **5** and **6** which engage the top convolution of container **1** and prevent the container from being pushed out of position.

FIG. 2, another embodiment of the present invention, shows a cylindrical helix **10** formed of successive convolutions of a tightly wound wire formed of a refractory metallic material. Electrode **11** contacts and seals one end of the helix while electrodes **12** and **13** contact the periphery of convolutions spaced from the sealed end.

FIG. 3, another embodiment of the present invention, shows a conical container **15** formed of successive convolutions of a tightly wound wire of a refractory metallic material. Electrode **16** supports the apex of container **15** and electrode **17** contacts the entire periphery of convolutions spaced from the apex of container **15**.

FIG. 4 shows a typical vacuum deposition apparatus **20** defined by bell jar **21** and base member **22** which is typically made of steel. Port **23** communicates with vacuum chamber **24** and is attached to vacuum pump **25**. Positioned within vacuum chamber **24** is container **26** constructed in accordance with the embodiment shown in FIG. 1. Container **26** is supported and held by electrodes **27**, **28** and **29**. Electrode **29** contacts the apex of container **26**. Electrodes **27** and **28** contact the periphery of convolutions spaced from the apex. Electrodes **27** and **28** which form one leg of the electrical circuit are held by supports **30** and **31**, respectively. These supports, typically made of copper, extend through and are insulated from base member **22** and make electrical contact with power supply **32** through leads **36** and **37**, respectively. Electrode **29** which forms the other leg of the electrical circuit extends through and is insulated from base member **22** and makes electrical contact with power supply **32** through lead **38**. Power supply **32** may be any type known to the art. For example, it is common practice to obtain stepless control of the container voltage by connecting the primary of a fixed ratio low voltage transformer to a variable ratio autotransformer. Substrate **33** is positioned and held above container **26** by arm **34** which in turn is attached to support **35**.

As illustrative of procedures used in the formation of vapor deposited films on substrate materials, specific examples are given below. In all cases a vapor deposition apparatus utilizing the container configuration of FIG. 2 was used. The container consisted of eight turns of 0.20 inch diameter tungsten wire closely wound to form an angle of 40 degrees. The resulting conical crucible which was 0.19 inch in maximum diameter and 0.26 inch high was mounted between two tungsten electrodes 0.156 inch in diameter which formed one leg of the electrical circuit. The apex of the crucible rested on and was sealed by a vertical electrode 0.180 inch in diameter which formed the other leg of the electrical circuit. A one by three inch glass microscope slide was mounted 3.55 inches above the crucible at right angles to the axis of the crucible and symmetrical with respect to the center of the vapor stream. The slide was first cleaned in accordance with accepted practice, for example by washing with a detergent, rinsing first with distilled water and then acetone to dry it.

In each experiment a new crucible and slide were used. The evaporation took place in a residual atmosphere of air at a pressure of 8×10^{-5} millimeters of mercury. The procedure was to load the filament with 10 milligrams of titanium dioxide and heat the crucible during a 15 to 20 second interval to a temperature of 2300° C. This temperature was maintained until a total of 40 seconds had elapsed. Thickness of the deposited layer was measured on an interferometer. Such measurements made in three separate experiments have resulted in a reproducibility of ±7.5 percent about a mean value thickness of 1074 angstroms.

What is claimed is:

1. A vapor deposition apparatus comprising a vacuum chamber defined by a bell jar and a base member, and a wire wound resistance heated container consisting of successive convolutions of a wire refractory metallic material forming a cone positioned within said vacuum chamber together with provision for supporting and applying a current to said container including three metallic members, one of said metallic members contacting the apex of said cone and the other said metallic members contacting said cone at at least two points on the periphery of a convolution spaced from the apex of said cone.

2. A vapor deposition apparatus comprising a vacuum chamber and a wire wound resistance heated container consisting of successive convolutions of a wire refractory metallic material forming a cone positioned within said vacuum chamber together with provision for supporting and applying a current to said container including two metallic members, one of said metallic members contacting the apex of said cone and the other metallic member contacting substantially the entire periphery of at least one convolution spaced from the apex of said cone.

3. A vapor deposition apparatus comprising a vacuum chamber and a wire wound resistance heated container consisting of successive convolutions of a wire refractory metallic material forming a cone positioned within said vacuum chamber together with provision for supporting

and applying a current to said container including a first metallic member and at least one additional metallic member, said first metallic member contacting the apex of said cone and the at least one additional metallic member contacting said cone on at least two substantially diametrically opposed points on the periphery of a convolution spaced from the apex of said cone.

References Cited in the file of this patent

UNITED STATES PATENTS

2,164,332	Macksoud	July 4, 1939
2,530,946	Cheesman et al.	Nov. 21, 1950
2,633,427	Dimmick et al.	Mar. 31, 1953