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ELECTROSTATIC DIRECTION OF EXPLODED VAPORS

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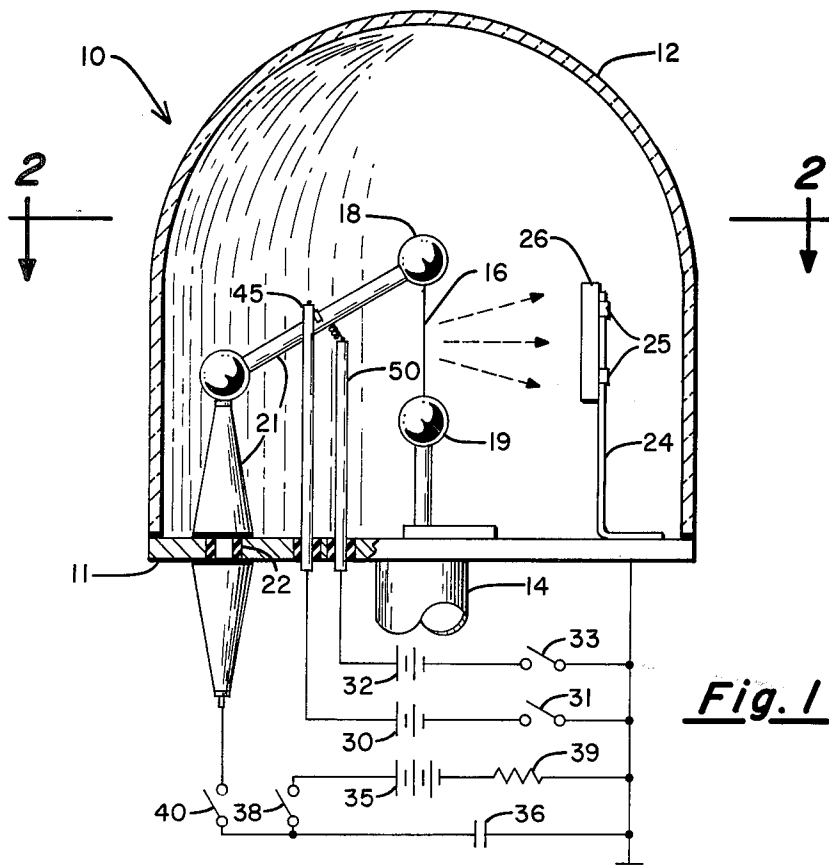


Fig. 1

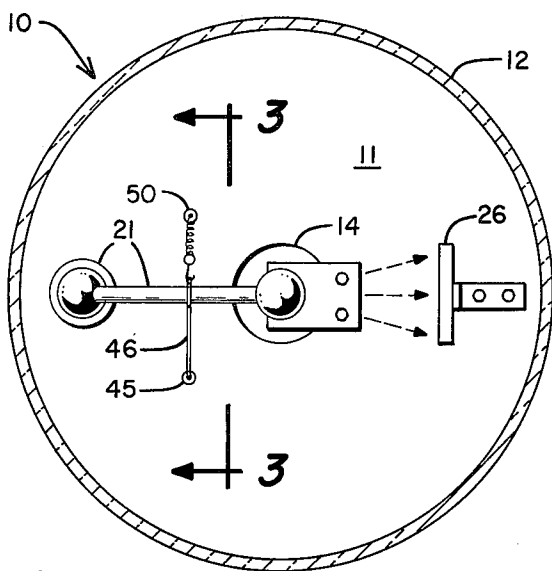


Fig. 2

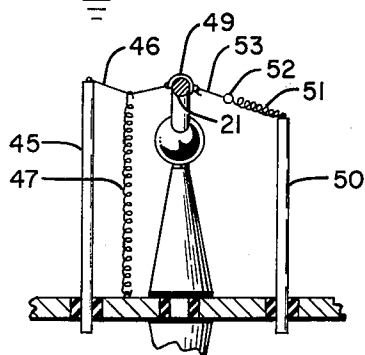


Fig. 3

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ELECTROSTATIC DIRECTION OF
EXPLODED VAPORS

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The present invention relates generally to the deposition of thin films, and more particularly to the explosive evaporative and controlled deposition of thin films of a magnetic metallic material onto a suitable substrate or base member.

The present invention is an improvement over that certain invention disclosed and claimed in Patent No. 3,142,587 of Erwin G. Weber, Serial No. 80,301, filed January 3, 1961, and entitled "Apparatus for Producing Electrical Conductor Films by Explosive Evaporation." In that patent, the explosive evaporation and consequent deposition of magnetic thin films is disclosed. However, in connection with the technique of the present invention means are provided for steering, guiding, and controlling the flow of the evaporant from the source or explosion locus to the ultimate deposition of the evaporant upon a suitable substrate member.

In the past, various ways have been utilized to prepare magnetic thin films, particularly thin films for use in connection with binary magnetic memory devices. For example, certain of these techniques being set out in detail in Rubens Patent No. 2,900,282. In that patent, reference is made to a vacuum deposition of thin magnetic films wherein the evaporation operation is accomplished by inductively or otherwise heating a source of ferromagnetic material and causing the vapors to condense onto a substrate. In the present invention, magnetic or non-magnetic films may be prepared by deposition in vacuum or in a non-contaminating atmosphere onto a suitable substrate by explosively evaporating a conductive body which is preferably resistively preheated, means being provided to steer, guide, or otherwise control the transmission of the material from the locus of the explosive operation onto the surface of the substrate.

Explosive evaporation offers the advantages of: (1) a short deposition time of less than 1 μ sec., which reduces the interaction between the metal vapor and the residual atmosphere in the vacuum to a minimum; (2) absence of contact between the material to be evaporated and other materials all of which may be held at high temperatures (for example, crucibles, boats, etc.) prevents alterations of the material or of the vaporization process; (3) rapid heat generation prevents fractional distillation; (4) short deposition time minimizes interaction of the condensing vapor with the substrate (diffusion, chemical reaction, etc.); and (5) exact calibration of the evaporated total mass necessary is readily carried out by a simple length measurement of the conductor exploded. In addition, explosive evaporation offers the substantial advantage of making it possible to evaporate binary, ternary, or higher order alloys without regard to the individual vapor pressures at the evaporation temperatures which are employed. Conventional evaporative deposition of these higher order alloys is difficult to achieve due to the differences in vapor pressure at the evaporating temperatures, since the composition of the individual films will vary from the initial evaporant reaction to the composition of the later evaporated materials. When magnetic thin films are being prepared for use in connection with memory elements, certain compositions are undesirable inasmuch as magnetostriuctive effects may develop in the material causing differing magnetic switching characteristics to occur, both

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of which have a deleterious effect upon the use of these materials as magnetic memory elements.

In utilizing explosive evaporative techniques in the preparation of thin films, a greater degree of uniformity with regard to deposition across a relatively large cross-sectional area along with other features may be achieved when a mechanism for steering or otherwise controlling the flow of material from the locus of the evaporation to the substrate is employed. The steering means or mechanism is the arrangement of a source of an electrostatic field or electromagnetic field or combination thereof at a point which is remote from the location of the substrate. The material which is to be explosively evaporated is preferably disposed at a location which is along a line between the source of the steering means and the substrate surface.

It is, therefore, the object of the present invention to provide a method and apparatus for controllably effecting explosive evaporation of an electrical conductor.

It is a further object of the present invention to provide means for preheating the conductor to be evaporated and to provide at least a portion of the energy required to vaporize the conductor by the preheating operation.

It is yet a further object of the present invention to provide a steering means for the evaporant, the steering means being provided by the presence of an electrostatic and electromagnetic field due to the charging of the energy transmission portion of the explosive evaporating system and the flow of energy through the system and the conductor being evaporated, the energy transmission means being disposed adjacent to the conductor being evaporated and in sufficiently close proximity thereto to have an influence upon the evaporant medium.

Other and further objects of present invention will become apparent to those skilled in the art upon a study of the following specification, appended claims, and accompanying drawings in which,

FIG. 1 is a front plan view of apparatus specifically adapted for carrying out the technique of the present invention, the enclosure being shown in section, and further showing a schematic illustration of typical circuitry which may be employed in connection with the explosive evaporative operation;

FIG. 2 is a horizontal sectional view taken along the lines and in the direction of the arrows 2—2 of FIG. 1; and

FIG. 3 is a partial plan view of the preheat portion of the system utilized in connection with the operating cycle, certain components being shown in section.

Referring now to a preferred embodiment of the present invention as is illustrated in the accompanying drawings, the evaporating system generally designated 10 includes a base platform 11 which is surrounded by a suitable bell-jar enclosure or the like 12. The platform 11 together with bell-jar 12 is preferably fabricated from a non-magnetic material in order that any tendency toward generation of stray magnetic fields which may have an adverse effect upon the preparation of the film, may be completely if not substantially entirely eliminated. Within the base 11, an evacuating tube 14 is provided, the tube 14 communicating with the interior of the bell-jar 12 and permitting proper evacuation of the jar. In the alternative, tube 14 provides access for introducing a desired inert atmosphere to the enclosure.

Referring now to the apparatus which is adapted to accomplish the explosive evaporation of the desired material, a suitable wire, band strip, or the like such as the wire 16 fabricated from a nickel-iron alloy comprising 82% nickel, balance iron, is maintained between the retaining members 18 and 19, these retainers being adapted to maintain the conductor 16 in a properly taut relation-

ship. Conductive leads and steering field generating means consisting of the rods 21—21 are provided in order to transmit electrical energy at the desired level to the conductor 16. The leads 21—21 are insulatingly supported within the base 11 through the insulating grommet 22. The substrate supporting bracket 24, is secured by any desired means to the base plate 11, the bracket 24 including a pair of brackets 25—25 thereon which are adapted to hold or retain the substrate member 26 in a predetermined position relative to the conductor 16 and to the steering field generating means 21—21.

Referring now to the schematic illustration of the circuitry employed in the system, two phases of the operation are indicated, the first being the preheat mechanism which includes a heater power source 30 together with controlling switch 31, and a fusing power source 32 together with controlling switch 33. The main explosive power is provided by the high voltage D.C. source 35, this source being connected across capacitor 36. Charging switch 38 is provided in the capacitor charging circuit along with the charging resistor 39. Upon completion of each of the preliminary steps prior to the explosion operation, per se, switch 40 is adapted to complete the circuit from the power source through main leads 21—21, upper ball 18, the conductor to be exploded 16, the support ball and rod 19 and thence through the base plate 11 to ground.

The equipment and circuitry which is adapted to operate the preheat cycle is arranged to be disconnected from the remaining phases of the circuitry at the completion of the heating cycle, thereby being isolated therefrom. Suitable hold-down schemes are utilized in order to provide the appropriate disconnect for this operation. With particular attention directed to FIG. 3 of the drawings, it will be seen that preheat post 45 is provided adjacent upper rod 21, this post being insulatingly disposed within the base plate 11 and adapted to conduct preheat current into the conductor to be exploded 16. Spring biased conductor or line 46, held in biased position by the resilient member 47, is adapted to carry current to the half-round connector 49, which is held in electrical contact with the upper portion of conductor rod 21. Half-round connector 49 is arranged to be physically separated and disconnected from the rod 21 when desired, this separation being accomplished by means of the fusing arrangement disposed to the right of the preheat arrangement. The fusing portion includes the post 50, the resilient conductor 51, the fuse or fusible element 52 which is attached or secured to the clamp 49 by means of the conductor element 53. Upon completion of the preheat cycle, switch 33 will be closed thereby energizing the fusing portion of the mechanism, the current being sufficient to rupture fuse element 52 and thereby permit the connector 49 to be displaced from the remaining portions of the mechanism. Resilient member 47 will carry the connector 49 therealong and will accordingly perform the disconnect operation around the rod 21.

In a typical explosive operation adapted to be carried out in connection with the apparatus shown in the drawings will now be described.

The atmosphere within the bell-jar 12 is either evacuated or filled with a suitable inert gas. The pressure is preferably between about 1×10^{-4} mm. Hg and 1×10^{-6} mm. Hg and there is no minimum pressure limit. In the initial step, the operator closes switch 31 which initiates the preheat cycle. The preheat proceeds for a predetermined time, that is, the conductor to be exploded is held at a sufficiently high temperature for a sufficiently long period of time as determined either by practice or through observation with suitable temperature measuring device such as an optical pyrometer or the like. After the preheat cycle has been carried out for a suitable length of time, switch 33 is closed which provides for a surge of current through fusible element 52, the element 52 separating and permitting the contact element 49 to be removed from physical contact with the conducting rod 21.

Switch 38 has previously been closed, this permitting charging of the capacitor 36 from source 35. Upon completion of the charging operation, as determined from the requirements of the explosive operation, switch 40 will be closed so as to enable a full surge of electrical energy to be passed through the system the quantity of energy being adequate and available at such a rate so as to explosively evaporate conductor 16. Switch 38 is preferably closed just prior to closing of switch 40.

The high voltage and energy surge from the discharge of capacitor 36 (which may be a bank of condensers totaling six microfarads of capacity, for example) may be effected by previously charging in any desired manner, such as, for example, by a 110 volt source, which is operatively associated with a suitable high voltage charging source. The capacitor 36 is charged by the high voltage charging source through the high resistance (9 megohms, for example) resistor 39.

When conductor 16 explodes upon the application of the high voltage together with the high electrical energy, the vapor which is generated as a result of this explosion, as steered, guided or the like along the direction of the arrows, where indicated upon the surface of the substrate member 26. If desired, the substrate 26 may be masked, as required.

In connection with certain materials being evaporated and deposited, it is desirable that the preheating temperature of the conductor to be evaporated be held below the melting point thereof, although the temperature is preferably sufficiently high so as to be immediately below the melting point. The purpose of the preheating cycle is to supply a portion of the energy which is required to accomplish the explosive operation. For an 80–20 Permalloy layer having the length of four inches, 20 mils diameter, the optimum preheating temperature is about 1000° C. At a much lower temperature the amount of heat generated in the wire by the discharge energy of the system is too small to completely evaporate the wire, and with higher temperatures, the evaporation is not uniform because of apparent weak spots in the surface tension along the element. It has been found that greater uniformity results with a temperature held at about 1000° C. with this wire. With a temperature of about 1000° C. the four inch wire as indicated herein above forms films of about 500 Angstroms thickness on a substrate which is located one inch from the wire source.

The preheating temperature may be maintained for a period of about 10 minutes, or longer if desired, in order to permit outgassing to occur. The elevated temperature should not be maintained for such a period of time so as to enable oxidation to occur along the surface of the wire. As between a plurality of 8 mm. diameter circular Permalloy films deposited onto ordinary glass microscope slides, a remarkable uniformity with regard to the type of hysteresis loop, coercive force and anisotropy field was noted. For most films the coercive force was in the range of from 2–10 oe. and the anisotropy field was always greater than 100 oe. Uniformity from one evaporating wire charge to the next can be expected if the wires have uniform characteristics, if the preheat temperature is maintained substantially the same, and if the operation cycle is carried out in a uniform manner. For example, a recommended time lapse between cessation of the preheating cycle and the commencement of the explosion cycle may be about 0.1 second. Times in excess of this period are not recommended since the degree of uniformity between products may suffer. However, it should be pointed out, that longer periods may be utilized provided they are uniform and provided the rate of cooling of the wire is maintained substantially uniform. In this regard it will be appreciated that the thermal gradients along the lengths of the wire are much greater when the preheat cycle is not continued to the extent that a certain constant temperature is achieved at the contact balls 18 and 19. These gradients along the

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wire are preferably avoided inasmuch as they may encourage splattering of the wire onto the substrate due to certain portions of the wire not having reached a sufficiently high temperature to become completely vaporized during explosion.

The voltage to which the capacitor 36 is charged is preferably in the range of 20 kv., however, this value may be varied considerably depending upon the conductive characteristics of the particular wire or conductor being exploded. It has been found that 20 kv. is sufficient to explode 81-19 Permalloy wire which is 6" long and 25 mils in diameter or which is 4" long and 30 mils in diameter. There is no apparent maximum value for the high voltage other than the limitations imposed by the equipment being utilized, and the minimum voltage requirement is that determined by the characteristics of the material being evaporated.

The steering or guiding field is set up by the vertical and diagonal conductors or rods 21-21. The fields which are set up must be, of course, disposed adjacent from the substrate with the conductor to be exploded being arranged between the substrate and the feed-through lines. The field is believed to be both an electromagnetic and electrostatic, the electromagnetic field being generated during the period of time that current is flowing along the conductors. Indications are, however, that the greatest influence on the plasma or evaporant is due to an electrostatic field rather than electromagnetic, however, the composite influence is believed to be a combination of the two types of fields.

In accordance with the present invention, it has been found that the use of a guiding, deflecting or steering field provides a greater degree of uniformity between the magnetic films so prepared. In this regard, films having a greater degree of uniformity, one to another, as well as a greater degree of magnetic uniformity, one to another, are prepared with the use of the steering field as compared to those prepared without the use of the steering field. When an explosive evaporation of a metallic substance occurs, and when there is no electrostatic field or the like present to direct the film forming substance in any one direction, the distribution of evaporant will be substantially random. When a steering field such as an electrostatic or combined electrostatic or electromagnetic field is provided the evaporant may be carefully and controllably directed onto the surface of a substrate member. In this connection, it is possible to explosively evaporate electrical conductors having a mass which is substantially closely related or correlated to the mass of the film desired. It is accordingly not necessary to evaporate large quantities of superfluous evaporant when a steering field is available to control the flow of evaporant from the locus of the explosion to the surface of the substrate. Therefore, the minimum requirements of the mass of the

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film being prepared may be compatible with the maximum requirements of the means for providing explosive evaporations. Inasmuch as relatively large quantities of energy must be available to perform explosive evaporation operation of electrical conductors, the presence of a steering field renders the energy requirements more modest, and hence compatible with the equipment available for performing the task.

It will be appreciated that the apparatus and techniques of the present invention as set forth in this application, are exemplary only and are not to be construed as any limitation upon the scope of the invention disclosed in claims herein. It will accordingly be understood that those skilled in the art may depart from the examples illustrated here and without actually departing from the spirit and scope of the present invention. Having now, therefore, fully illustrated and described our invention, what we claim to be new and desire to protect by Letters Patent is:

1. Apparatus for depositing a film on a substrate member comprising:
 - an evacuable enclosure, said substrate member being disposed within said enclosure,
 - an electrical conductor disposed within said enclosure adjacent said substrate member,
 - evaporating means operatively associated with said conductor for providing an evaporant by explosively evaporating said conductor, and
 - means for generating a steering field with resultant force components sufficient to direct substantially all of said evaporant generally toward said substrate.
2. Apparatus as in claim 1 wherein said evaporating means includes said generating means.
3. Apparatus as in claim 2 wherein said electrical conductor is disposed at a location which is along a line between said evaporating means and said substrate member.

References Cited by the Examiner

UNITED STATES PATENTS

2,357,415	9/44	McManus	118-48	X
2,382,432	8/45	McManus	118-48	X
2,975,332	3/61	Starr	315-169	
2,976,174	3/61	Howard	117-93.2	
2,996,418	8/61	Bleil	117-93.2	
3,142,587	7/64	Weber	117-107	

FOREIGN PATENTS

702,937	1/41	Germany.
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