United States Patent

[11] 3,598,957

[72] Inventors Nobuaki Yasuda
Zushi-shi;
Iwao Higashinakagawa, Kanagawa-ken;
Hiroaki Okamoto, Tokyo, all of, Japan

[21] Appl. No. 856,695
[22] Filed Sept. 10, 1969
[45] Patented Aug. 10, 1971

[73] Assignee Tokyo Shibaura Electric Co., Ltd.
Kawasaki-shi, Japan

[32] Priority Sept. 13, 1968
[33] Japan
[31] 43/66518

[54] VACUUM DEPOSITION APPARATUS

11 Claims, 8 Drawing Figs.

[52] U.S. Cl. 219/271, 118/49.1
[51] Int. Cl. C23c 13/00
[50] Field of Search ..... 219/271, 275; 118/621, 623, 48, 49, 49.1; 117/106 R, 107, 93.4 R, 93.4 A, 93.4 NC

[56] References Cited

UNITED STATES PATENTS

2,604,870	7/1952	Blood et al.	118/48 X
3,131,078	4/1964	Fuller et al.	117/107 X
3,142,587	7/1964	Weber	118/49.1
3,213,826	10/1965	Lins et al.	118/49.1

Primary Examiner—J. V. Truhe
Assistant Examiner—C. L. Albritton
Attorney—Flynn & Frishauf

ABSTRACT: A vacuum deposition apparatus comprising a rodlike vapor deposition source vertically mounted substantially in the center of a vacuum vessel, a cylindrical member inside the vacuum vessel and surrounding the vapor deposition source which is located substantially at the central axis of the cylindrical member, a substrate being mounted on the inner surface of the cylindrical member, a shutter for selectively shutting off a deposition metal supplied from said rodlike vapor deposition source from being deposited on the substrate, and a coil for generating a magnetic field positioned outside said cylindrical member. A heater is optionally included to heat the substrate.

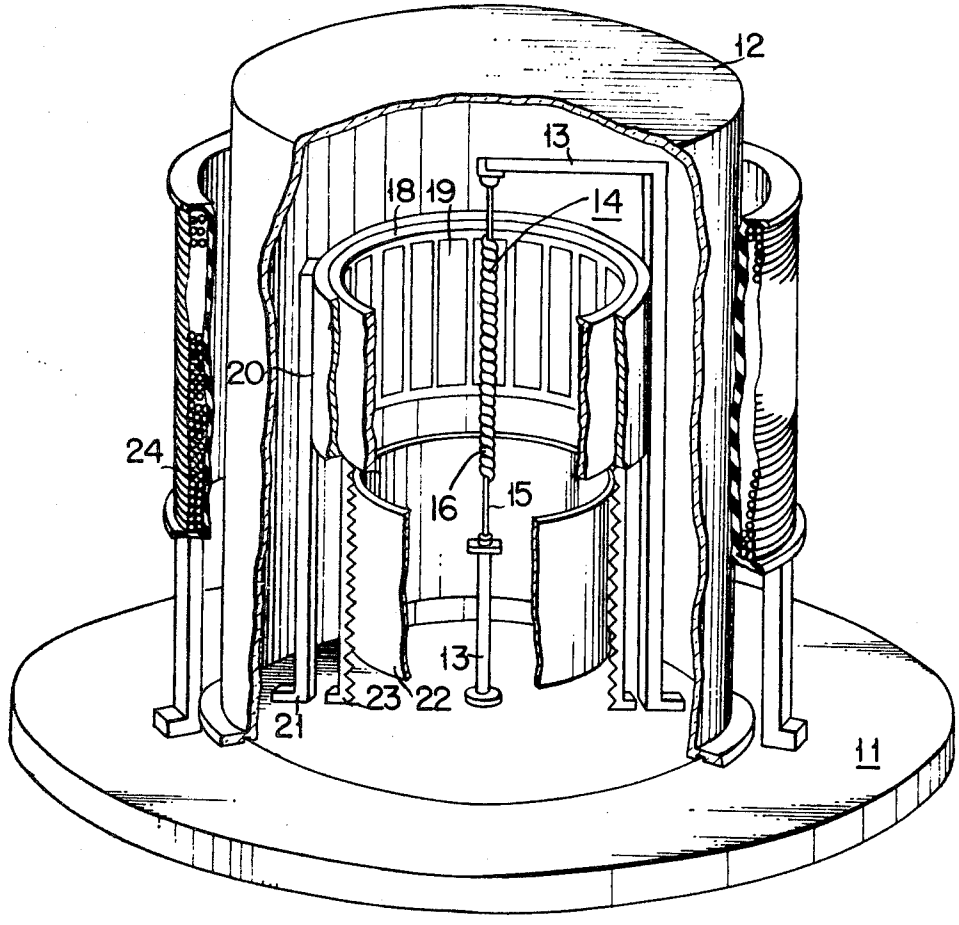


FIG. 1

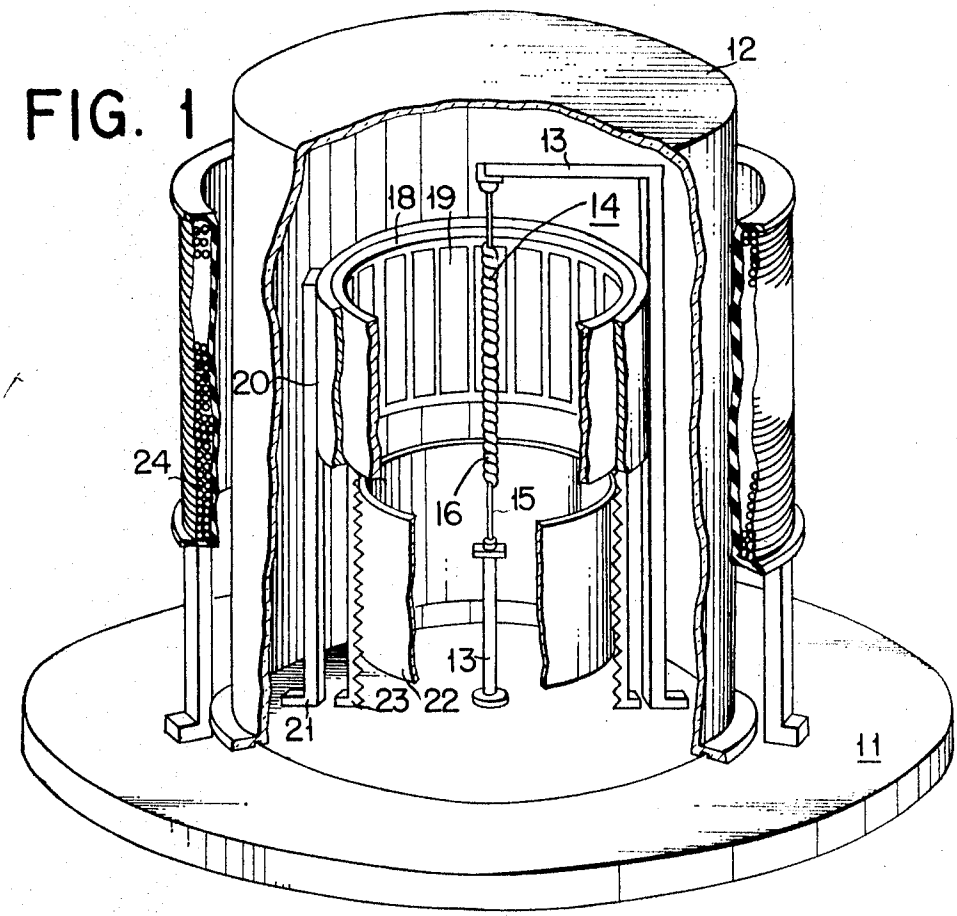
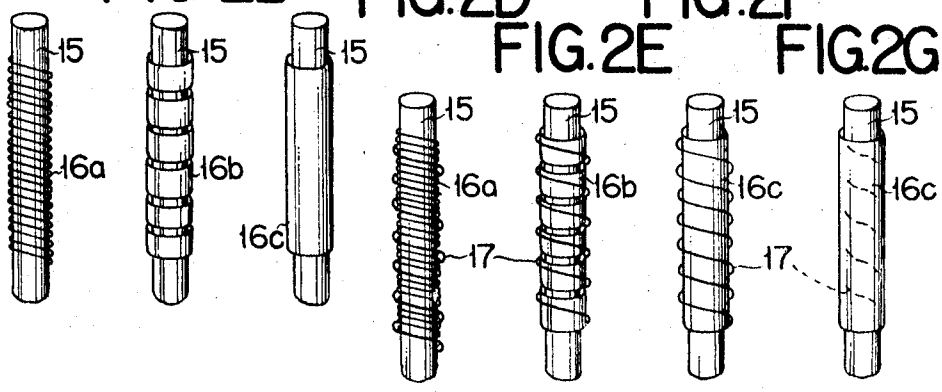


FIG.2A FIG.2C

FIG.2B FIG.2D FIG.2F

FIG.2E FIG.2G



VACUUM DEPOSITION APPARATUS

The present invention relates to a vacuum deposition apparatus and more particularly to a vacuum deposition apparatus capable of vapor-depositing a magnetic thin film of uniaxial anisotropy over a broad area.

Rapid progress is being made in the method of preparing active and passive elements in the form of a magnetic thin film as a means for promoting the quantity production of compact and lightweight electronic devices. Above all, a vapor-deposited thin film used as a memory element for a computer is known to have excellent high-speed and high-density properties. Such films are prepared by various processes, for example, vacuum deposition, sputtering, impingement of electron beams and electrodeposition. They are selectively employed according to the object for which the film element is to be used.

The vacuum deposition method heretofore adopted, for example, consists in providing in a vacuum vessel a source of vapor deposition material at an opposite position to a substrate of vapor deposition, simply winding said material about a resistance heat element, or mounting it thereon to form said source, charging said material in a heat-resistant crucible provided with a heater or directly impinging electron beams on said material.

When large amounts of vapor-deposited film are to be manufactured by these vacuum deposition methods of the prior art, there is used a process of providing in a vacuum vessel a means for allowing a substrate to move along, continuing vapor deposition until the substrate is coated with a deposition material to a desired thickness, removing said substrate to a separate place upon completion of vapor deposition, introducing a fresh substrate to be vapor-deposited opposite to a source of vapor deposition, repeating the same cycle of operation, and continuing vapor deposition until all the vapor deposition material initially charged is consumed. However, such conventional vacuum deposition methods have the drawbacks as listed below.

1. An area of vapor deposition where the film formed can have a uniform thickness is extremely limited, for example, to about 6 cm. × 6 cm. at most.

2. There is imposed limitation on the size of a source of vapor deposition material and in consequence the amount of said material to be mounted thereon, so that vapor deposition can be carried out on only several substrates per charge of said material, thus requiring a separate means for its supply.

3. The material of vapor deposition varies in quality between the initial stage where it is fully charged in the vacuum vessel and the stage where a little amount of it is left. This defect become prominent when vapor deposition material is alloys or is ready to react with the heat element of said source, with the result that the properties of a vapor deposited film have poor reproducibility.

4. Installation of a substrate-driving means in a vacuum vessel presents difficulties in keeping said vessel in a fully evacuated state. Namely, it requires a longer period of time in obtaining the desired degree of vacuum, unavoidably leading to the limited effective volume of said vessel and the high cost of a vapor deposition apparatus as a whole.

5. Particularly where it is desired to prepare a magnetic thin film, there is applied a magnetic field parallel to the plane of a substrate, and there is vapor deposited a film of magnetic material on the substrate heated to a temperature of about 300° C., thereby generating uniaxial anisotropy in said film utilizing magnetic anisotropy. After said vapor deposition, however, it is sometimes required to perform heat treatment in the magnetic field. In such case, therefore, it is not desirable to remove a substrate immediately after it is coated with a vapor-deposited material.

6. When there is prepared a magnetic film, it is preferred that the area of vapor deposition be as broad as possible. It is required, however, that a magnetic field applied to a substrate

be entirely parallel all over the substrate. With the prior art apparatus, however, there is imposed limitation on the area of a substrate which permits a magnetic field in parallel, necessarily reducing the area of vapor deposition. In consideration, therefore, of the above-mentioned requirements in addition to the allowable range of uniform film thickness, there can be obtained a vapor-deposited film only having an area of about 15 cm. × 15 cm. at most.

It is accordingly the object of the present invention to provide a vacuum deposition apparatus capable of preparing by vacuum deposition a vacuum-deposited film, particularly a broad magnetic thin film useful as a memory element of a computer in large amounts and at low cost.

SUMMARY OF THE INVENTION

In accordance with the present invention, a vacuum deposition apparatus comprises a vacuum vessel including a base member, a vapor deposition source including a rodlike heater element vertically fitted on the base substantially in the center of the vacuum vessel and vapor deposition material on the surface of the rodlike heater a cylindrical member supported on the base inside the vessel and surrounding the vapor deposition source which is located substantially at the central axis of the cylindrical member, the inner surface of the cylindrical member being adapted to receive a substrate to deposit said deposition material on the substrate heat element, a movable shutter positioned between the source and substrate, for selectively covering the substrate and a coil for generating a magnetic field, the coil surrounding and being in concentric relation with the cylindrical member.

This invention can be more fully understood from the following detailed description when taken in connection with reference to the accompanying drawing, in which:

FIG. 1 is a perspective view, with part broken away, of a vacuum deposition apparatus according to an embodiment of the present invention; and

FIGS. 2A to 2G represent in perspective view various modifications of vapor deposition material used in the present invention.

There will now be described by reference to the drawing a vapor deposition apparatus according to an embodiment of the present invention. Numeral **11** denotes the base of the apparatus, which is connected to an evacuator (not shown). On the base **11** is mounted an upright cylindrical bell jar **12**, the interior of which is evacuated by said evacuator to a degree of less than 10^{16} Torr. Substantially in the center of the bell jar **12** is provided a vapor deposition source **14** comprising a rodlike heat element **15** and vapor deposition material **16**, for example, ferromagnetic material such as Permalloy which is mounted on the surface of the heat element **15**. Said source **14** is held upright by a support member **13** formed of insulating material. The source **14** may be prepared by winding, as shown in FIG. 2A, a wire **16***a* of vapor deposition material about the prescribed portion of a rodlike resistance heat element **15** made of molybdenum, tungsten, tantalum or the like, or as shown in FIG. 2B, ribbon-shaped vapor deposition material **16***b* about said heat element **15**, or by depositing, as shown in FIG. 2C, vapor deposition material **16***c* over the entire surface of said heat element by means of, for example, plating. Where vapor deposition is to be continued for a long period of time, molten vapor deposition material tends to flow downward over the surface of the heat element **15**. To prevent the occurrence of such event, it is preferable to wind, as shown in FIGS. 2D to 2G, a high-melting heat-resistant wire **17** made of, for example, molybdenum, tantalum, tungsten or the like at a suitable space over the surface of vapor deposition materials **16***a*, **16***b* and **16***c* or insert such winding into the interior thereof.

On the inner surface of cylindrical member **18** supported on the base **11** and centered about the source **14** there are mounted a plurality of substrates **19** of vapor deposition by means of a suitable holder or heat-resistant adhesive agent. On

the outer surface of said cylindrical member is attached, if necessary a heater **20** for heating said substrate **19**. These substrates **19** and heater **20** are held in place by a support stand **21**. Between the source **14** and the cylindrical member **18** supporting substrates **19** is a cylindrical shutter **22** concentric with the cylindrical member **18** so as to surround the vapor deposition source **14** as a central axis. The shutter **22** is supported by a support stand **23** comprising a vertically driving mechanism (not shown). Outside of the heater **20** is positioned a coil **24** for generating magnetic field which has a fully greater width than the substrates **19** and a larger diameter than the heater **20**, in a concentric relationship to the cylindrical member **18** and in such a manner that the central zone of said coil **24** is disposed at a level substantially corresponding to the assembled substrates **19**. Said coil **24** may be located inside of the bell jar **12** instead of outside thereof, as shown in FIG. **1**.

According to the vapor deposition apparatus of the present invention having the aforementioned arrangement, the bell jar **12** is evacuated to a degree of less than 10^{16} Torr, the assembled substrates **19** are heated to a desired temperature by the heater **20**, electric current is introduced to the source **14** for preliminary heating so as to remove gases and volatile impurities from the source **14**, with the assembled substrates still enclosed by the cylindrical shutter **22**. After the vacuum deposition material **16** is heated to a desired melting temperature, the cylindrical shutter **22** is shifted in position to allow said material **16** to be vapor deposited on all the substrates at the same time. The coil **24** is used to generate a magnetic field in which there is vapor deposited a magnetic material. Said field can be applied on the surface of the substrates **19** in parallel relationship with the axial direction of the source **14**.

As mentioned above, the vacuum deposition apparatus of the present invention enables a substrate to be vapor deposited with good reproducibility along the periphery of a circle centered about the source **14** and spaced therefrom by a given distance. For example, with said distance set at 25 cm., and the width of each of the substrates **19** set at 30 cm., then there will be obtained an area of vapor deposition as large as about 150 cm. × 30 cm. Namely, it is possible to carry out vapor deposition at the same time on 120 substrates 6 cm. × 6 cm. in size or 30 substrates 5 cm. × 30 cm. This offers a great advantage in elevating productivity and reducing product cost.

When a magnetic thin film is to be used for a memory element, the applying direction of a magnetic field must be in the "easy" direction of magnetization of ferromagnetic material having uniaxial anisotropy. While it is desired that the length in said direction be as great as possible, the prior art apparatus only permitted a length of about 10 to 15 cm. at most. In contrast, the present invention can extend said length to 30 to 50 cm. or over, thus helping to reduce the cost of the entire memory system. Also where there is to be applied a parallel magnetic field over a long distance, the present invention only requires a single coil having a still larger width, so that the magnetic field applied can have an excellent degree of parallelism.

Further, the present invention offers a great advantage in that there is vertically set up a rodlike vapor deposition source. Heretofore, it has been common practice to dispose said source horizontally. In such case, the vapor deposition material melted on the resistance heat element assumes the form of water drops due to its surface tension and gravity, thus causing itself to be evaporated in different amounts between above and below the vapor deposition source. With the prior art apparatus, it is also common that the degree of reaction of vapor deposition material with the heat element and the velocity of evaporation of said material vary between the upper and lower positions of said source, and the properties of a film vapor deposited above the source are different from those of a film formed below it. However, the vertical placement of the vapor deposition source as adopted by the present invention eliminates all such drawbacks and allows said material to be evaporated right and left in the same manner. Where evaporation is continued for a long period of time, however, there will appear the aforesaid phenomenon that the molten vapor deposition material tends to run downward. To prevent such occurrence, it is effective to use such a type of source as shown in FIGS. **2D** to **2G**. Namely, a high-melting heat-resistant wire **17** wound about a rodlike heat element at a suitable space serves to prevent the downward flow of molten vapor deposition material and permits said material to be evaporated in situ.

There will now be described an embodiment of the present invention associated with preparation of a magnetic thin film. The vapor deposition source comprises a rodlike resistance heat element of tungsten 2.4 mm. in diameter and 50 cm. long. This tungsten rod is wound with a wire 0.2 mm. in diameter of Permalloy having a composition of approximately 80 percent Ni-20 percent Fe. Both ends of the wire are fixed in place by welding. Further, there is wound a tungsten wire 0.2 mm. in diameter in such a manner that one turn of said tungsten wire is interposed between every three turns of the aforesaid Permalloy wire with all these turns spaced about 0.3 mm. from each other. Both ends of said tungsten wire are fixed in place similarly by welding. In this case, all that is required is to wind at the same time each group of three turns of the 0.2-mm.-diameter Permalloy wire and one turn of the 0.2mm.-diameter tungsten wire about the 2.4-mm.-diameter tungsten rod with care taken not to allow them to be superposed on each other. Further there are assembled 30 glass substrates each having the aforementioned 6 cm. × 30 cm. area of vapor deposition. The vessel is evacuated to below 10^{16} Torr and the substrates are heated to 300° C. Across the substrates is applied by a coil a magnetic field of about 50 Oersted to vapor deposit a film of Permalloy at the rate of 100 to 200 A./sec. This process is capable of forming over the entire area of vapor deposition a magnetic film in large amounts, at low cost and with good reproducibility which has a thickness of 1,000 to 1,200 A. and in which there is uniformly distributed uniaxial anisotropy retaining a coercive force of 3.0 Oersted and an anisotropic field of 4.5 Oersted.

The foregoing embodiment relates to the case where, as shown in FIG. **2D**, there are wound about a heat element a wire of vapor deposition material and a wire of high-melting heat-resistant material in such a manner that the number of turns of the former winding and that of the latter winding bear a ratio of 3 to 1. Even where the vapor deposition material assumes the form of, for example, foils or platings other than wires, the winding of the aforesaid high-melting heat-resistant wire will display the same effect of vapor deposition as described above. It will be apparent that winding ratio of vapor deposition material may be freely chosen to obtain a vapor-deposited film of varying thickness. It will be further understood that the present invention is not limited to the aforementioned embodiment but may be practices in various modifications without changing the spirit and scope of the invention. For example, to form a film of uniform thickness all over a broad area of vapor deposition, it is advisable to wind more turns of a wire of vapor disposition material about the portion of the heat element facing the upper and lower portions of a substrate where the deposition is usually comparatively thin, than about the portion of said heat element facing the central portion of the substrate.

As for the vapor deposition material, it is also possible, besides above-mentioned ferromagnetic material, to employ nonmagnetic materials e.g. copper, aluminum titanium or gold. When it is desired to superpose two or more of the same or different kind of materials, e.g. three layers composition of Permally-copper-Permalloy, on a substrate, this can be easily conducted by putting necessary vapor deposition materials in vessel individually, positioning each vessel one after another at the prescribed place, and vapor depositioning each material in sequence on a substrate without breaking the vacuum in the apparatus. Vapor deposition materials to be vaporized or already vaporized may be kept in a suitable place that does not

hinder the deposition operation e.g. near the shutter 22. In this case vapor deposition materials can be carried by any suitable conveying means.

What we claim is:

1. A vacuum deposition apparatus comprising: a vacuum vessel which includes a base member;

a vapor deposition source including a rodlike heater element vertically supported on said base in the center of said vacuum vessel, and vapor deposition material on the surface of said rodlike heater element;

a cylindrical member supported on said base inside said vessel and surrounding said vapor deposition source which is located substantially at the central axis of said cylindrical member, the inner surface of said cylindrical member being adapted to receive a substrate to deposit said deposition material on said substrate;

a movable shutter disposed between said vapor deposition source and said cylindrical member and for selectively covering said substrate where it is desired not to deposit; and

a coil having the same axis as said cylindrical member and surrounding said cylindrical member to generate and apply a magnetic field to said substrate.

2. Apparatus according to claim 1 further comprising a heater which is attached on the outer surface of said cylindrical member for heating said substrate mounted on the inner surface of said cylindrical member.

3. Apparatus according to claim 1 wherein said coil is supported on said base and outside said vacuum vessel.

4. Apparatus according to claim 1 wherein said coil is supported on said base and inside said vacuum vessel.

5. Apparatus according to claim 1 wherein said vapor deposition material is in the form of wire wound about said rodlike heater element.

6. Apparatus according to claim 5 wherein said vapor deposition source further includes a high-melting-point heat-resistant wire wound about said wire-shaped vapor deposition material.

7. Apparatus according to claim 1 wherein said vapor deposition material is in the form of a ribbon wound about said rodlike heater element.

8. Apparatus according to claim 7 wherein said vapor deposition source further includes a high-melting-point heat-resistant wire wound about said ribbon-shaped vapor deposition material.

9. Apparatus according to claim 1 wherein said vapor deposition material is in the form of a cylinder surrounding said rodlike heater element.

10. Apparatus according to claim 9 wherein said vapor deposition source further includes a high-melting-point heat-resistant wire wound about said cylindrical vapor deposition material.

11. Apparatus according to claim 1 wherein said vacuum vessel includes a bell jar mounted on said base member.