

July 20, 1971

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3,594,301

SPUTTER COATING APPARATUS

Filed Nov. 22, 1968

2 Sheets-Sheet 1

Fig. 1.

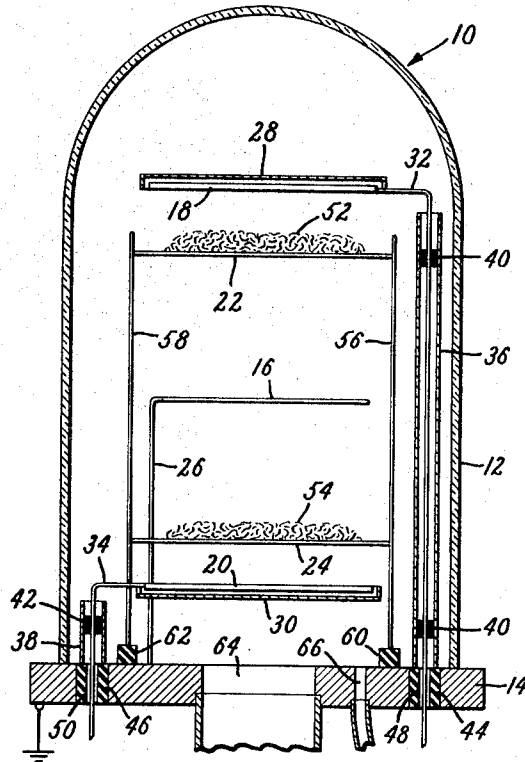


Fig. 1A.

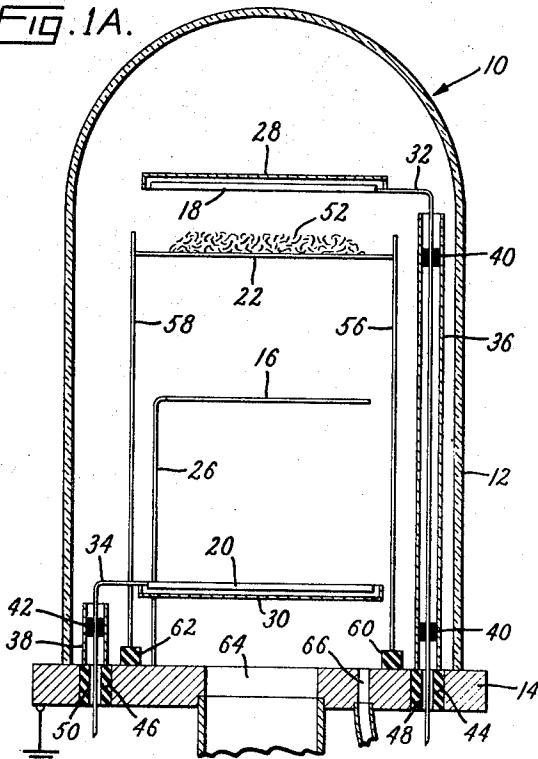
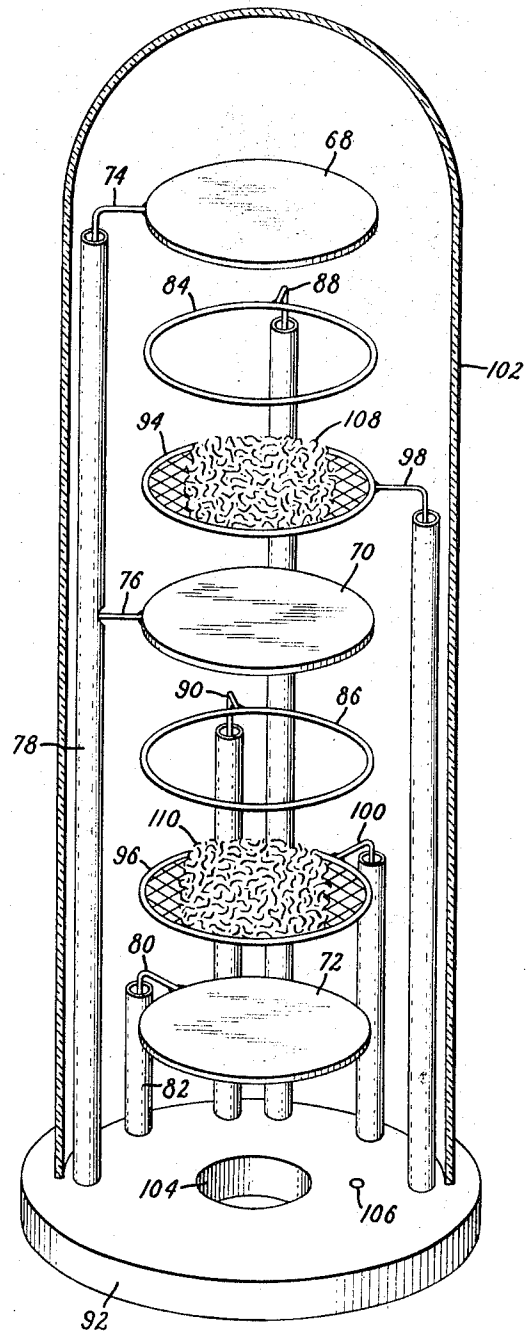


Fig. 2.



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Fig. 3.

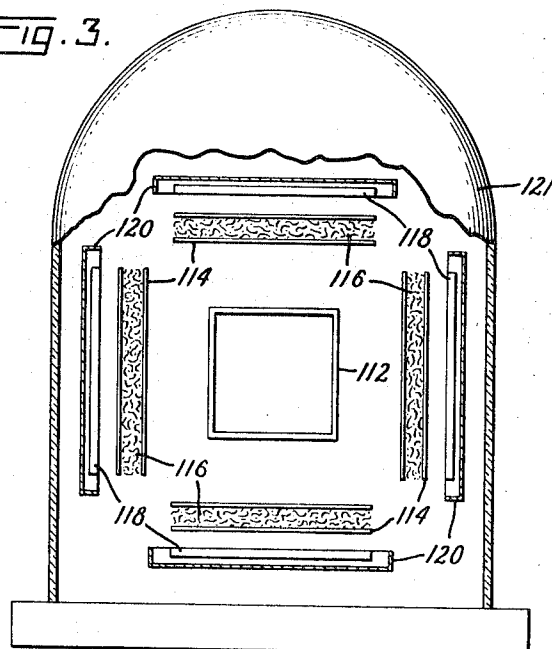


Fig. 4.

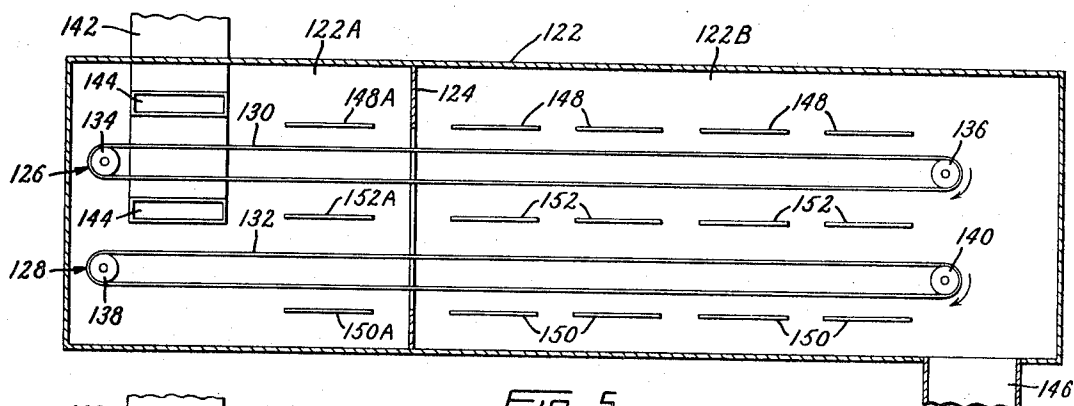
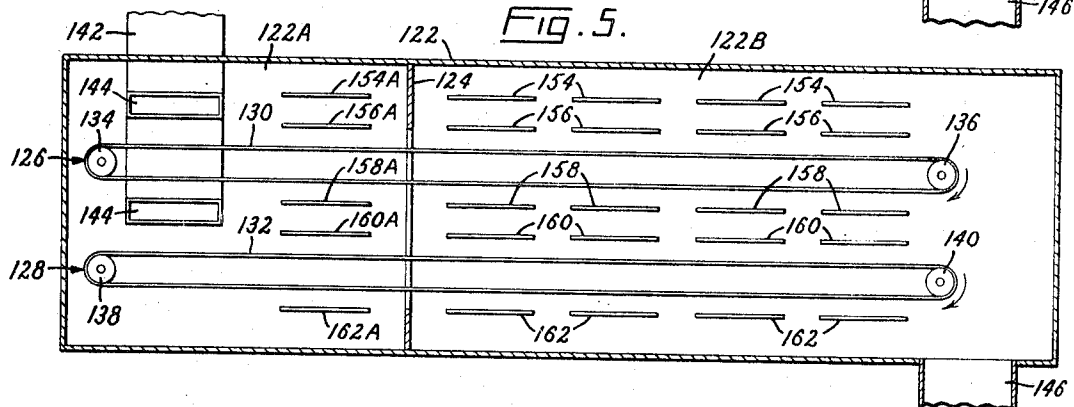


Fig. 5.



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SPUTTER COATING APPARATUS
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U.S. Cl. 204—298

14 Claims

ABSTRACT OF THE DISCLOSURE

Apparatus for efficiently providing ionic cleaning and sputter coating of materials. The sputtering apparatus basically includes at least two targets (cathodes) with a porous anode located therebetween with the material to be coated positioned between at least one of the targets and the anode. The use of a porous anode allows sputtering atoms to pass therethrough to provide substantially uniform coating of the material.

BACKGROUND OF THE INVENTION

The subject invention generally relates to the field of coating and, in particular, to apparatus for cathodic sputter coating of materials.

It is often desirable to apply a thin coating of a desired substance to many types of materials. Particularly, in the field of fiber reinforced composites it is often necessary to coat fibers with elements or compounds prior to the manufacture of the fiber reinforced composite. The coatings may act, for example, as a diffusion barrier to prevent reaction between the fiber and matrix or to promote wetting and bonding between the fiber and matrix. It is necessary in these cases to assure a strong bond between the coating and the fiber so as to achieve high strength in the composites.

Coating by cathodic sputtering in a glow discharge is generally considered one of the best methods of coating certain types of materials. As part of this coating process, it is generally desirable to have an ionic cleaning step prior to the actual coating. While prior art devices do generally show apparatus for cathodic sputtering, some of which include the ionic cleaning step, these have not been able to efficiently provide a uniform coating on a significant amount of freshly ionically cleaned surfaces of material, particularly where a mat of fibers is to be coated.

SUMMARY OF THE INVENTION

Therefore, it is an object of the subject invention to provide a sputtering apparatus which can simultaneously coat two sides of a given material.

A further object is to provide apparatus for continuously sputter coating materials.

In order to fulfill the above-mentioned objects, the subject invention provides a sputtering apparatus utilizing a porous anode which allows sputtering atoms to pass therethrough. At least two cathodes are provided with the porous anode located therebetween. A mat of material to be coated is positioned on a support between at least one of the cathodes and the anode. The side of the mat of material facing the cathode is directly coated by cathodic atoms and the opposite side is coated by cathodic atoms which have passed through the porous anode. Another mat of material may be located between the other cathode and anode in which case the side of the mat not facing the cathode is coated by cathodic atoms which have passed through the opposite mat of material and the porous anode. Additional cathodes and/or anodes may be added to the structure to insure uniform coating when the material to be coated, such as a mat of fibers, is such that it does not allow cathodic atoms to readily pass there-

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through or when more than two mats of material are to be simultaneously coated.

The subject matter which is regarded as the present invention is particularly pointed out and distinctly claimed in the concluding portion of this specification.

BRIEF DESCRIPTION OF THE DRAWINGS

The subject invention, however, together with further objects and advantages thereof, may best be understood by reference to the following description taken in connection with the accompanying drawings in which:

FIG. 1 is a side sectional view of one embodiment of sputtering apparatus in accordance with the subject invention;

FIG. 1A is a view of the apparatus shown in FIG. 1 used to coat only one mat of fibers;

FIG. 2 is a perspective view of a second embodiment of sputtering apparatus in accordance with the subject invention;

FIG. 3 is a side schematic view of a third and preferred embodiment of the subject invention;

FIG. 4 is a simplified side view of the basic apparatus shown in FIG. 1 adapted for continuous processing; and

FIG. 5 is a simplified side view of the embodiment shown in FIG. 2 adapted for continuous processing.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

In FIG. 1 a first embodiment of cathodic sputtering apparatus is shown.

The apparatus basically consists of a bell jar 12 which is sealed at its open end to a base plate 14; a centrally located "porous" anode 16; upper and lower cathodes 18, 20; and two work holders 22, 24, respectively located between one of the cathodes 18, 20 and the anode 16.

The porous anode 16 can be any anode having a substantial portion of the area within its perimeter open. Such an anode could have a grid configuration, be of a perforated material or be ring shaped. The anode 16 should be made of a material having good electrical conductivity, such as aluminum, steel or copper. The anode 16 is connected to the base plate 14 by an anode support member 26, the base member in turn being connected to ground.

The cathodes 18, 20 are each formed of plates of the material to be sputtered. The cathodes 18, 20 shown in FIG. 1 are of a planar shape, but may be of any desirable configuration. Each of the cathodes 18, 20 preferably has a shield 28, 30 adjacent the portions thereof not facing the work holders 22, 24 to confine most of the glow discharge to the region between the two cathodes. The cathodes 18, 20 are connected to a suitable electrical power supply by electrically conductive support members 32, 34. The vertical portion of each cathode support member 32, 34 is spaced within a vertically extending tube 36, 38 by electrically insulating spacers 40, 42, respectively. The lower end of each of the tubes 36, 38 is attached to an electrically insulating, vacuum feedthrough 44, 46 which extends through the base member 14. A central bore 48, 50 is located in each feedthrough 44, 46 to allow the cathode support members 32, 34 to extend therethrough.

The work holders 22, 24, which are used to support mats 52, 54 of fibers to be coated are very porous so that they cover no more than a small percentage of the surface of the material to be coated, preferably being comprised of a fine wire grid. Work holder support members 56, 58 are connected to the work holders 22, 24 to provide support therefor. These support members 56, 58 are attached to the base plate 14 via insulators 60, 62 which serve to electrically insulate these members from the base member.

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The base member 14 has a large hole 64 therethrough which allows the interior of the bell jar to be connected with a suitable means for retaining a desirable vacuum therein such as a vacuum pump. Also, a small passageway 66 extends through a base member 14 which serves as an inlet for the gas to be used within the bell jar 12.

In order to obtain a proper bond between the coating material and a substrate, which in this case is the fibers of the fibrous mat, it is generally desirable to have a plurality of cleaning steps before the sputtering process takes place. Before the fiber mats are inserted into the sputtering chamber they may be cleaned by such methods as washing in organic solvents or agents, vacuum heat treatment, or any other cleaning method well known in the art.

The second cleaning step occurs after the fibrous mats are inserted in the sputtering chamber. This type of cleaning is known as ion cleaning which is achieved primarily by regulating the gas pressure at which the glow discharge within the chamber is maintained. For this step, many types of gases may be used, but an inert gas such as argon or krypton is preferred.

The pressure within the bell jar 12 is maintained very low, in the range of 1-10 microns of pressure so that a dark space known as the Crooke's or cathode dark space is present near the cathodes 18, 20 and extending toward the anode 16. The smaller the pressure, the greater the thickness of the dark space so that with pressures within the above-mentioned range the dark space extends a substantial distance toward the anode, including the space wherein the fibrous mats 52, 54 are located. Ion cleaning is effected in the dark space because it is a region in which gas ions, formed by collisions with electrons from the cathode, are accelerated toward the cathode and attain maximum energy just before they reach the cathode. The primary method of surface cleaning achieved by these ions involves billiard-ball type collisions in which the ions hit the surface atoms with sufficient energy to dislodge these atoms into the gas. Ionic cleaning is also effected by simple evaporation due to heating of the substrate (fibers) by the ion bombardment.

In the apparatus shown in FIG. 1, both fibrous mats 52, 54 are cleaned simultaneously. The sides of the fibrous mats nearest the anode 16 receive the preponderance of the ion bombardment, while the sides nearest the cathodes 18, 20 receive the preponderance of the bombardment by the gas atoms which have become de-ionized by collision with the cathodes 18, 20 and have been subsequently reflected back into the gas. The fibers in the mats 52, 54 are generally small enough so that heat produced by ion and atom bombardment is uniformly distributed therethrough so that thermal cleaning (by evaporation) occurs uniformly on both sides. In this manner the fibrous mats 52, 54 are uniformly and effectively cleaned.

For the sputter coating process, the gas pressure within the bell jar 12 is increased to the range of about 20-100 microns and the electrode potential is modified to obtain optimum conditions. With this increase in pressure the dark space decreases to a point where it is not discernible and the sputtering of atoms from the cathodes 18, 20, proceeds at much higher rates. The surface atoms which receive sufficient energy in collisions with the gas ions enter the gas and diffuse radially outward. Many of them collide with and adhere to the fibers nearest the source cathodes 18, 20, thereby coating them. Some do not hit the fibers and proceed toward the anode 16; and others become ionized due to the collisions with electrons, gas atoms, and gas ions. The porous anode 16 acts in a two-fold manner on these sputtered atoms and sputtered atom ions. First, any sputtered atom ions are charge-repelled back toward the fibers and cathodes due to the polarity of the anode 16. Secondly, because the anode 16 has a substantial portion of open space, most of the sputtered atoms reaching it are allowed to pass therethrough toward the fibers on the other side of the opposite fibrous mat, thereby increasing the number of sputtered atoms striking

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the surface of the fibers facing the anode. Because of the porous configuration of anode 16, there is achieved uniformity of coating of the fibrous mat.

In FIG. 1A the basic apparatus of FIG. 1 is shown in its simplest form with only one work holder 22 and mat 52 of material to be coated. The side of the mat 52 facing away from the upper cathode 18 is coated by cathodic atoms from the lower cathode 20 which passes through the porous anode 16. The one mat 52 is uniformly coated on both sides regardless of the density of the mat, hence a piece of solid material could be coated.

The embodiment shown in FIG. 1, which is of the cathode-work holder-anode-work holder-cathode construction, allows adequately uniform coating only of multiple fibrous mats of relatively low density, i.e. those being quite porous. In order to provide uniform coating of two or more fibrous mats of relatively high density or of solid materials, a second embodiment of the subject invention (as shown in FIG. 2) may be used.

The apparatus shown in FIG. 2 is of a cathode-anode-work holder-cathode-anode-work holder-cathode configuration. Three cathodes 68, 70, 72 being located at the top, center and bottom of the apparatus, respectively, are used. The upper and center cathodes 68, 70 are supported by members 74, 76 which are joined together and housed within a hollow tube 78 in a similar manner to that described in FIG. 1. Similarly, the lower cathode 72 is supported by a member 80, the vertical portion of which is located within a hollow tube 82.

Two anodes 84, 86 are located, respectively, below the upper and central cathodes 68, 70. Porous anodes 84, 86 similar to those described above are each connected via their support members 88, 90 to the base plate 92 of the apparatus which is electrically grounded.

The two work holders 94, 96 are each located directly below the two anodes 84, 86, respectively. The work holders 94, 96 are electrically connected via their respective support members 98, 100 and a large series resistance to their adjacent cathodes.

As in FIG. 1, a bell jar 102 is located about the apparatus of FIG. 2 and suitably sealed to the base plate 92. Also, a hole 104 is located through the base plate 92 which is connected to a suitable vacuum pump, for maintaining the desired vacuum, as discussed above, within the bell jar. A small gas inlet aperture 106 is also provided for allowing a desired gas to enter the interior of the bell jar.

Cathodes 68 and 70 are preferably connected to a common DC electric power supply, and cathode 72 is connected to a separate DC electric power supply (not shown).

The embodiment shown in FIG. 2 operates in a similar manner to that of FIG. 1, regarding both the ion cleaning sputter coating operations except that the center cathode 70 is utilized as a common cathode to coat the sides of fibrous mats 108, 110 on the work holders 94, 96 facing this cathode rather than relying upon sputtering atoms to pass through the mats.

In this apparatus sputtering atoms from the upper cathode 68 pass through the upper porous anode 84 and are deposited on the upper side of the fiber mat 108. Sputtering atoms from the upper side of the central cathode 70 are deposited on the lower side of the upper fiber mat 108 so as to provide uniform coating of this mat. The upper side of the lower fiber mat 110 is coated by sputtering atoms coming from the lower side of the central cathode 70 which pass through the lower porous anode 86. The lower side of the lower mat 110 is coated by the deposition of sputtering atoms from the lower cathode 72 so as to provide uniform coating of the lower fiber mat 110.

If desired, one or more of the cathodes 68, 72 may be shielded, as shown in FIG. 1, so as to concentrate the sputtering atoms emanating therefrom into the desired region.

To increase the amount of material that can be sputter-coated in a single apparatus many modifications can be

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made, within the subject invention, to the embodiments shown in FIGS. 1 and 2. By stacking additional elements one or more extra work holders can be added. For example, the apparatus of FIG. 1 may be modified by removing the shield from the upper cathode 18 and installing additional layers of work holder, anode, cathode (if one extra work holder is desired) or work holder, anode, work holder cathode (if two extra work holders are desired). An example of this is shown in FIG. 3 to be described hereinafter.

Similarly, the apparatus of FIG. 2 can be modified by adding above the top cathode 68 layers of work holder, anode, cathode (if one extra layer is desired); or work holder, anode, cathode, work holder, anode, cathode (if two extra work holders are desired). The apparatus of FIG. 1 can also be modified by utilizing a porous anode of cylindrical, spherical or polygonal shape and arranging one or more work holders to be disposed about the anode in a polygonal cylindrical, or spherical shape with one or more cathodes disposed about the work holders in a substantially similar configuration.

In FIG. 3, the apparatus of FIG. 1 is modified so that four work holders may be utilized with a single porous anode 112. The porous anode 112 is centrally located and is preferably of a rectangular shape. Four work holders 114 are spaced therefrom in a substantially rectangular configuration with fibrous mats 116 fixedly attached thereto. Spaced outwardly from each work holder 114 is one of four cathodes 118 each with its respective shield 120. These members are all encased in a suitably sealed bell jar 121, as in the apparatus of FIG. 1. In this simplified view, the support and electrical connections for each of the members are not shown. The electrical connection for each member is the same as that of FIG. 1, i.e. each cathode being connected to an appropriate DC electric power supply (not shown), each work holder being connected in series with a very large resistance which is in turn connected to the cathodes, and the anode being connected to ground.

It can readily be seen that in this embodiment utilizing one porous anode and four cathodes, four mats of fibrous material can be coated simultaneously.

Another way of coating a relatively large amount of material by sputtering is by use of a continuous process apparatus as shown in FIGS. 4 and 5. The embodiments shown in FIG. 4 utilizes the electrode arrangement shown in FIG. 1, and the apparatus of FIG. 5 utilizes the electrode arrangement shown in FIG. 2. The continuous sputter coating embodiments shown in FIGS. 3 and 4 differ only in their electrode configurations.

The basic parts of the continuous sputter coating embodiments, as shown in FIGS. 4 and 5 include a substantially elongated vacuum chamber 122 which is divided off by partial partition members 124 into an ion cleaning subchamber 122A and a sputtering subchamber 122B. Running substantially lengthwise within the subchambers 122A, 122B are two conveying systems 126, 128; each including a continuous, porous belt 130, 132 covering no more than a small percentage of the surface of the material to be coated acting as a work holder which is substantially supported and driven by rotatable members 134, 136 and 138, 140 located at the ends of the respective belts 130, 132. One or both of the rotatable members for each belt may be driven in the direction shown by arrows by suitable means for providing rotational motion such as an electric motor. If only one member is driven, the other member will act as an idler. Additional idler or power driven members may be positioned at suitable locations along the conveyor belt, if needed. Any other suitable conveying system could be employed. Of course, if desired, only one conveying system may be used.

At the upper left portion of the chamber 122 a vacuum-lock entrance port 142 is shown which allows entry of uncoated fibers into the chamber without destroying the vacuum in the chamber. This may be accomplished by hav-

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ing an air lock chamber at the entrance to the ionic cleaning subchamber 122A. Fibers could be deposited in the air lock chamber, then this chamber could be evacuated and opened to the interior of the cleaning subchamber. The fibers then could be moved through the entrance port and directly deposited by gravity on the upper conveyor belt 130. Means such as a chute 144 is used to deposit uncoated fibers on the lower conveyor belt 132. In the lower right hand corner of the chamber 122B a vacuum-lock exit port 146 (similar in operation to the entrance port 142) is located for receiving the coated fibers falling off the ends of the conveyor belt 130, 132.

In the embodiment shown in FIG. 4 an upper set of cathodes 148 is located above the upper conveyor 130 and a lower set of cathodes 150 is located below the lower conveyor 132. A plurality of porous anodes 152 are located between the two conveyors 130, 132. All three sets of electrodes 148, 150, 152 include portions 148A, 150A, 152A extending into the ion cleaning chamber 122A.

In operation, one or more vacuum pumps are suitably connected to the chamber 122 so as to maintain a pressure distribution such that a substantial portion of the ionic cleaning chamber 122A is maintained within pressure range (1-10 microns of pressure) needed for effective cleaning and a substantial portion of the sputtering chamber 122B is maintained within the pressure range (20-100 microns) to assure effective coating. The cathodes 148, 150 are connected to a suitable electrical power supply; the cathodes 148A, 150A are connected to a separate electric power supply to provide the optimum potential; and the anodes 152, 152A are connected to ground.

Uncoated fibers enter the chamber 122 via the entrance port 142 and are deposited on the upper surfaces of the moving conveyor belts 130, 132. The fibers are then moved through the ionic cleaning chamber 122A so as to be suitably cleaned by the method which has been previously described. Then the cleaned fibers enter the sputtering chamber 122B wherein cathodic material from the cathodes 148, 150 is substantially uniformly deposited on the fibers, as the conveyors 130, 132 and the anodes 152 all have a substantial percentage of open space. Coating action is similar to that described in regard to the embodiment shown in FIG. 1. The coated fibers then drop off the ends of the moving conveyors 130, 132 into the exit port 146 which allows withdrawal of the fibers from the apparatus with little effect on the pressure within the apparatus.

The apparatus of FIG. 5 is substantially the same as that shown in FIG. 4 except for the electrode structure which is basically of the same configuration as that shown in FIG. 2. In FIG. 5, the electrode structure consists of an upper set of cathodes 154, 154A above the upper conveyor 130 and an upper set of anodes 156, 156A located therebetween. Between the two conveyors 130, 132 are located a center set of cathodes 158, 158A and a lower set of anodes 160, 160A. Below the lower conveyor 132 is located a lower set of cathodes 162, 162A. The basic continuous coating process in regard to the embodiment shown in FIG. 5 is similar to that described in regard to the embodiment shown in FIG. 4 except that the sputter coating portion of the process works in the manner described in regard to the embodiment shown in FIG. 2.

If desired, the basic continuous process apparatus shown in FIGS. 4 and 5 can be modified to provide two or more different types of coatings on a continuous basis by using cathodes of different cathodic substances. Also, of course, the electrode structure may be modified in accordance with some of the suggested modifications discussed above with regard to FIGS. 1, 2 and 3.

To improve the effectiveness of the coating, a number of modifications may be made to any of the embodiments disclosed herein. In order to reduce the amount of gas that may become trapped in the coating, the gas pressure

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required for both ion cleaning and sputtering should be reduced. This may be accomplished by using one or more auxiliary hot cathodes to supply the electrons required to sustain a glow discharge at reduced pressure.

Also, the shape of the cathodes may be modified to improve the uniformity of coating within the mat. When a flat plate cathode is used the flux of sputtered atoms evaporating from the cathode is highest in the direction perpendicular to the center of the cathode plate and becomes progressively lower with increasing distance from the center of the cathode. However, if the cathode plates are concave (i.e. the concave surfaces face the anode), for example parabolic or hemispheric, the flux of sputtered atoms will increase with increasing distance from the center, thereby providing more uniform coating of the entire fibrous mat.

Thus, the subject invention provides an efficient apparatus for providing substantially uniform cathodic sputter coating of substrate materials, particularly fibrous materials.

The subject invention may be modified in many ways without departing from the scope thereof. Therefore, it is intended the scope of the present invention be considered only in regard to the appended claims.

What I desire to secure by Letters Patent of the United States is:

1. In a vacuum sputtering apparatus for coating materials comprised of a vacuum chamber having therein electrodes including at least one anode and one target, support means within said vacuum chamber for supporting the material, the improvement comprising:

- (a) upper and lower targets,
- (b) said anode being located between said targets and having a substantial portion of the area within its perimeter open; and
- (c) said support means including at least one work holder covering no more than a small percentage of the surface of said material to be coated and being located between said anode and one of said targets, said work holder permitting deposition on the upper and lower surfaces of said material to be coated from said upper and lower targets, respectively.

2. Sputtering apparatus as in claim 1 wherein said support means further includes a second work holder covering no more than a small percentage of the surface of said material to be coated and being located between said anode and the other of said targets.

3. Sputtering apparatus as in claim 1 wherein said anode is in the form of an annular ring.

4. Sputtering apparatus as in claim 2 further including target shielding means for substantially confining any glow discharge to the region between said two targets.

5. A sputtering apparatus as in claim 2, further including

- a second anode located between said upper target and the adjacent work holder; and
- a third target located between said first mentioned anode and said work holder adjacent said upper target.

6. A sputtering apparatus as in claim 5, wherein said anodes are each in the form of an annular ring.

7. A sputtering apparatus as in claim 1, wherein at least three targets are included which are polygonally spaced about said anode; and one of said work holders is located substantially parallel to each one of said targets and between said respective target and said anode.

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8. A sputtering apparatus as in claim 1, including an upper and lower set of targets, each set including a plurality of substantially coplanar, aligned targets; a set of substantially coplanar, aligned anodes located between said sets of targets, each of said anodes having a substantial portion of the area within its perimeter open; and

said support means includes means for translating said work holder between one of said sets of targets and said set of anodes in a plane substantially parallel to the planes of said sets of targets.

9. A sputtering apparatus as in claim 8, further including means for depositing uncoated material on said work holder and means for collecting said material after it has been coated.

10. A sputtering apparatus as in claim 9, wherein said support means further includes a second work holder covering no more than a small percentage of the surface of said material to be coated and being located between said set of anodes and the other of said sets of targets, said second workholder permitting deposition on the upper and lower surfaces of the material to be coated from said upper and lower targets and means for translating said second work holder between said other set of targets and said set of anodes in a plane substantially parallel to the planes of said sets of targets;

a second set of substantially coplanar, aligned anodes are included which are located between said upper set of targets and the adjacent work holder; and

a third set of substantially coplanar aligned targets are included which are located between said first mentioned set of anodes and said work holder adjacent said upper set of targets.

11. A sputtering apparatus as in claim 10, wherein said anodes are substantially ring-shaped.

12. A sputtering apparatus as in claim 8, wherein said vacuum chamber is divided into an ion cleaning subchamber and a sputter coating subchamber, said ion cleaning subchamber being maintained at a substantially lower pressure than said sputter coating subchamber;

at least one of said targets and anodes in each of said sets of targets and anodes is within said ion cleaning subchamber; and

wherein said work holder is adapted to move first through said ion cleaning subchamber then through said sputter coating subchamber.

13. A sputtering apparatus as in claim 12, further including means for depositing uncoated material on the portion of said work holder which is located within said ion cleaning subchamber; and means for collecting said materials after coating which is located within said sputter coating subchamber.

14. A sputtering apparatus as in claim 13, wherein said means for translating said work holder includes a motor driven conveyor belt.

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