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Baxter

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[45] **Dec. 23, 1975**

[54] **METHODS OF AND MEANS FOR VACUUM DEPOSITION**

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[21] Appl. No.: **323,424**

Related U.S. Application Data

[63] Continuation-in-part of Ser. No. 113,313, Feb. 8, 1971, abandoned.

[30] **Foreign Application Priority Data**

Feb. 12, 1970 United Kingdom..... 6789/70

[52] **U.S. Cl.** **427/132;** 118/49; 118/49.1; 219/271; 219/275; 427/248; 427/250; 427/251

[51] **Int. Cl.**[2]..... **C23C 13/02;** C23C 13/04

[58] **Field of Search**..... 117/106 R, 107, 107.1, 117/107.2 R; 118/49, 49.1; 219/271, 275; 427/248, 250, 251, 132

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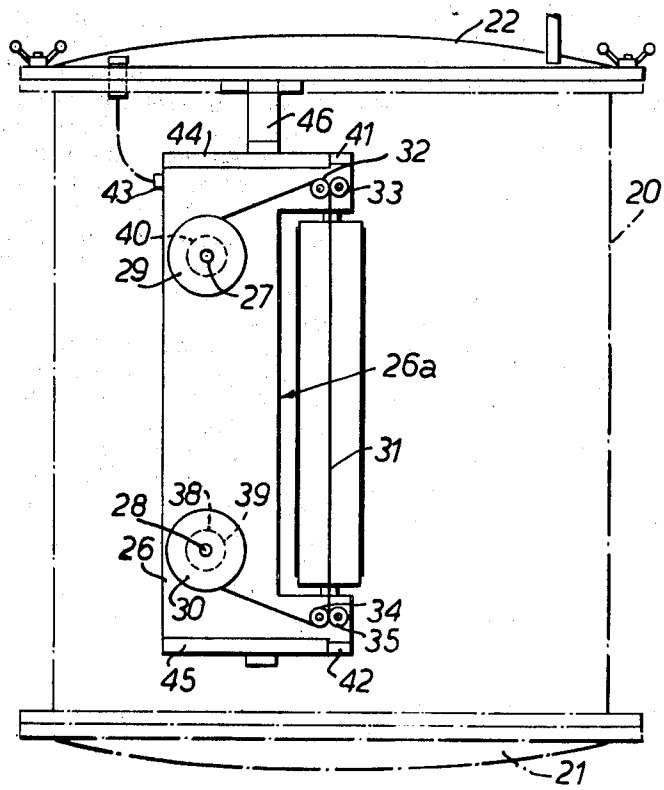
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Attorney, Agent, or Firm—Brisebois & Kruger

[57] **ABSTRACT**

Method of coating a surface of an article comprises steps of carbonizing organic material to provide a flexible elongated electrically conductive heater element, forming a solid coating of a vaporizable coating material on said element, positioning the said article and heater element in a vacuum chamber with said heater element stretched between two spaced electrodes and heating said element by passing electrical current through said electrodes and heating element to vaporize the vaporizable coating thereon.

4 Claims, 6 Drawing Figures



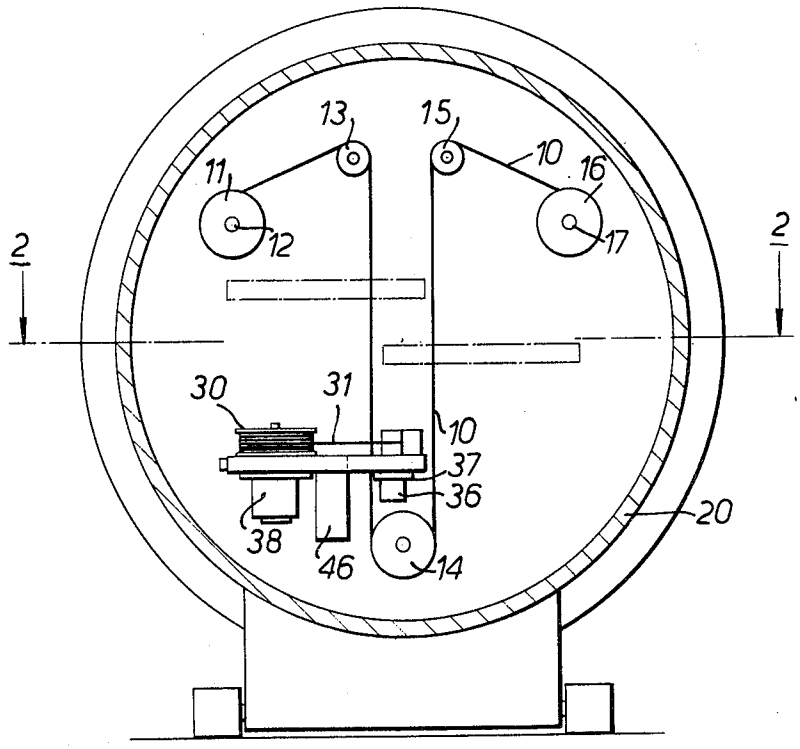


FIG. 1

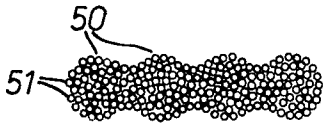


FIG. 3.

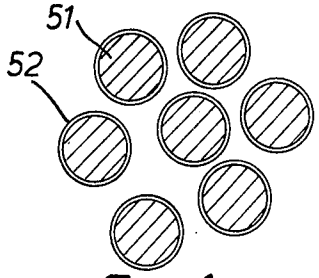


FIG 4.

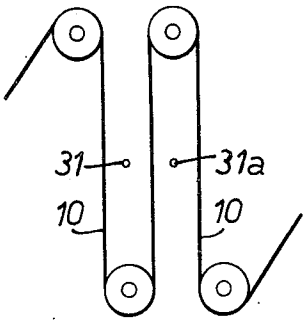


FIG. 6.

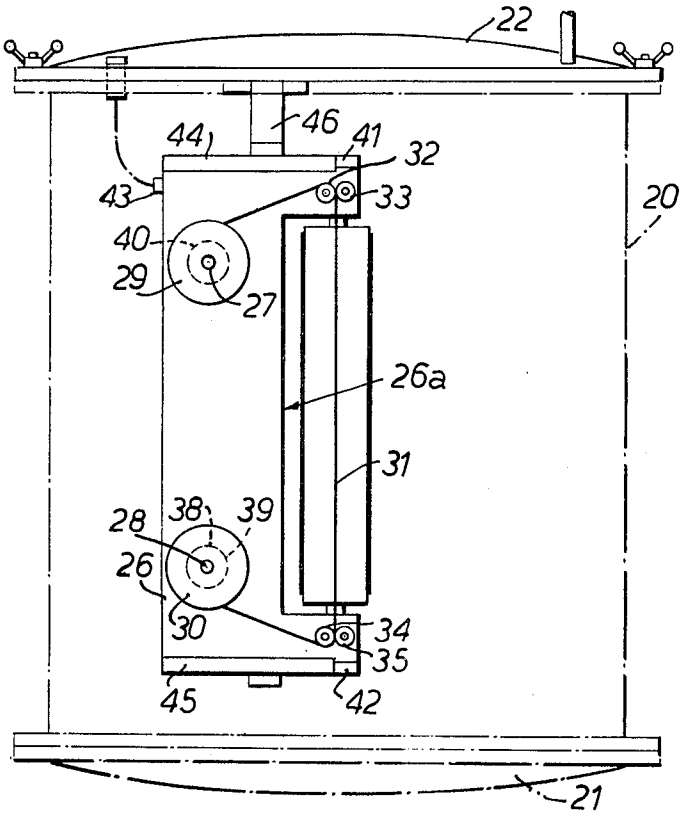

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26a
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21

FIG. 2

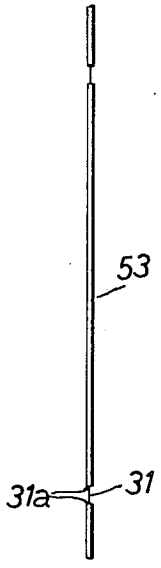

53
31a
31

FIG. 5.

METHODS OF AND MEANS FOR VACUUM DEPOSITION

This invention relates to a vacuum deposition and is concerned with improved methods of and means for vacuum deposition, and is a continuation in part of my application Ser. No. 113,313 Filed Feb. 8, 1971 and now abandoned.

In vacuum deposition, the article to be coated is loaded into a vacuum chamber, and the material to be deposited is brought into contact with a suitable heated element. The material is vaporised and the vapor is caused to deposit upon the article.

This basically simple process is one which has been known and practiced for many years; with some coating materials, which have a low melting point or a high vapor pressure below the melting point and are not strongly reactive with the surface of heating means or other materials in the vacuum, coatings can be satisfactorily obtained. This is particularly the case where small single articles or small areas of surfaces are to be coated, and where the circumstances of the coating operation, for example in laboratory or small scale conditions, permit a relatively inefficient or expensive short run process to be tolerated.

The problems of producing coating process which is efficient as to material and, to a lesser extent, power consumption for the heating means are substantial, and are greater with certain coating materials, and certain surface to be coated. For example there is a wide need for coating aluminum on thin sheet thermoplastic material, such as polyethylene terephthalate and polyvinyl sheet. Aluminum requires much heat for vaporisation so that in these conditions there is danger of softening or damaging the plastic sheet while it is adjacent the heater.

It is desirable to be able to run a vacuum coating apparatus for as long as possible on each load, and in this respect the nature and operation of the heating elements are important or dominating. The elements normally consist of a number of rods or bars of suitably refractory material, through which the electric heating current has to be passed, disposed across the width of the web to be coated. The material to be deposited is brought into contact with the surface of the heater. The life of such a heater is relatively short, often only a few hours, since the elements are consumed or eroded by the action of the coating material. The elements must be individually fed with coating material; the life of a set of elements is no longer than the life of the shortest, and failure of one element leads to non-uniformity of deposit across the width of the web. In consequence, it is necessary to remove and replace the elements at frequent intervals, re-making the necessary electrical connections to the replaced elements, and this is costly and time consuming.

For use as heating elements carbon rods are attractive because of the refractory nature of the material and comparative cheapness, and elements consisting basically of carbon have been much used in practice. Tungsten as wire or rods can be used but also has short working life and is expensive.

In the past it has been suggested that carbon heaters in the form of carbon fibre woven material could be used, on which material to be evaporated, such as copper, is locally placed. Further reference will be made to this prior proposal.

The present invention has for its object to provide a vacuum deposition process which uses elements which are relatively cheap to manufacture and efficient in use both as to power consumption and efficiency of deposition.

The invention also has for its object to provide a vacuum deposition process which uses elements which permit extended runs, limited only by the material to be coated.

THE INVENTION

The invention is based upon the use of a heater element composed of carbon fashioned into an elongated, flexible form, as a thread or tape or the like in which the carbon thread or filaments or the like extend along the length of the heater without any substantial degree of transverse displacement. The heater carries upon it a quantity of the coating material the material is distributed over at least the heated length of the heater and conveniently over the continuous length of the heater and wholly covers the surface of the heater. By this means a uniform coating material on the surface of the article is secured.

Further objects and advantages of the invention will appear from the following description of embodiments thereof, given by way of example, in conjunction with the accompanying drawings.

THE DRAWINGS

FIG. 1 is a diagrammatic side elevational view of a vacuum deposition apparatus;

FIG. 2 is a diagrammatic sectional view of the same apparatus taken on the line 2—2 of FIG. 1;

FIG. 3 is a sectional view of one form of heater element.

FIG. 4 is an enlarged detail of the heater element of FIG. 3.

FIG. 5 is a view of another form of heater element, and

FIG. 6 is a diagram showing an arrangement for coating both surfaces of a web.

FIG. 1 shows, partly diagrammatically, an apparatus suitable for coating with metal one surface of a thin flexible plastics film **10**, of, for example, polyethylene terephthalate, The film passes from a supply reel **11**, on spindel **12**, over rollers **13, 14** and **15** to take-up reel **16** on spindle **17.** As shown, the film follows a U-shaped path between rollers **13** and **15**, round roller **14.** Motors (not shown) are provided for rotating spindles **12** and **17.** The motor drives are reversible so that the film can be wound and rewound from one reel to the other as may be desired.

The apparatus is mounted in a vacuum chamber. This can take various forms but is shown as comprising a cylindrical body **20**, closed at one end by end plate **21** and at its other end by an end plate **22.** For convenience, the end plate **22** can be mounted on a permanent structure, the cylindrical body **20** and end plate **21** being mounted together on a movable carriage **23.** The body **20** and end plate **22** are held by detachable clamps **24.** Carried on the fixed end plate **22** are the connections, such as **24** to the vacuum pumping equipment, and connections **25** to the electrical supply and control gear for controlling the various driving motors in the apparatus.

Heating element assemblies are mounted in the chamber. Normally, two or more of such elements may be used, but as these are similar in construction, one

only will be described. One such assembly is shown in FIGS. 1 and 2. This comprises a main support plate 26 which has two spindles 27 and 28 to receive respectively reels 29 and 30. A filamentary heater 31, initially wound on supply reel 29, passes between a pair of conductive rollers 32 and 33 and then extends in a substantially straight, free and unobstructed path across a cut out portion 26*a* of the plate 26, to pass between the nip of a further pair of conductive rollers 34, 35 and thence to the take-up reel 30. The filament 31 can be advanced from reel 29 to reel 30 electrically under remote control, the rollers 34 and 35 being driven by an electric motor 36 and reduction gear 37. The reel 30 is driven by a take-up motor 38 which is lightly energised, or drives through a friction means such as pad 39, to drive the take-up reel. Reel 29 is under light frictional restraint, by resting on a suitable friction pad 40.

Means are provided for passing an electric current through that part of the filament 31 which spans the rollers, across gap 26*a*. Connection is made from the rollers 32, 33 and 34, 35 to electrical contacts on the plate 26, as at 41 and 42. Rollers 36 and 37 can be water cooled if experience shows this to be desirable. Connections to motors 36 and 38, are made by means of a plug and socket member 43.

The unit shown in FIG. 2 is capable of being readily inserted into and withdrawn from the chamber. The edges of the board are engaged in guides 44 and 45 carried on a support 46 from end plate 22; a suitable latch, can be provided for retaining the plate securely in position in the guides.

When the plate is thus positioned, the filament 31 will be positioned substantially mid-way between the two straight spans of the U-shaped portion of the film web 10. The cut-out 26*a* is of a depth such as to permit ready threading of the web when the apparatus is being prepared for coating of a web of material. When the heater assembly is inserted, connections are made automatically to the heater terminal contacts 41 and 42 for supplying a suitable current to heat the portion of the filament 31 which extends across the cut-out 26*a*. If further heater element assemblies are used, they can be positioned on the same or opposite sides of the chamber, with the filaments in a central position between the spans of the web.

The heater filament 31 is of a nature such that when it is raised to operating temperature, the desired material for deposition is vaporised from the surface of the filament.

In general, this requirement can be met by a wide variety of elements of different shapes, constructions and materials, the most suitable element depending upon circumstances, but the element must be conductive, either superficially or throughout, and must be capable of withstanding the conditions, including temperature, existing in the vacuum chamber during vaporisation. For the purpose of the present invention it is necessary that the element should be sufficiently flexible, to allow the element to be coiled.

The nature of the material of the heater is an important feature of the present invention and before proceeding to describe the construction of the heater it is appropriate to consider the operation of a heater when being used to vaporise a metal in the conditions of vacuum deposition. It is manifest that the heater will be raised to a temperature at which vaporisation takes place, but the conditions that obtain when this temperature is reached vary considerably with different deposition materials and different heater surface composition.

An important consideration is whether the molten material wets the surface of the heater. If the material does not wet the heater surface then when the material has become molten it will run into globules. When this happens it is detrimental to successful commercial coating for several reasons.

An important reason is that when globules are formed, vaporisation takes place from a series of localised areas, that is, the globules. There is little control of the location of such spots and they may change location rapidly and unpredictably; the vaporisation is violent or eruptive. This localised evaporisation is conducive to lack of uniformity of the vaporisation and lack of control of the thickness of the deposited layer of material.

Further, because of the localisation of the globules, the rate of heat loss from the surface of the heater is irregular. In vacuum, the energy dissipated from a unit surface areas of the heater is dissipated substantially entirely by two modes only: by radiation as heat, and by transfer to the molten material to supply the latent heat of vaporisation of the material; there may be a degree of terminal conduction. With a substantially uniform heater heated by current passed through it there will be a substantially uniform dissipation of energy per unit length of the heater, so that more heat will have to be dissipated as radiation from the heater surface between the globules than from the globule-covered surface areas. Not only does this radiated energy present a direct wastage of the power fed to the heater, but the radiated energy will uselessly and sometimes detrimentally heat the nearby surfaces, including the deposition surface. With plastic films, such heating is very undesirable.

The eruptive vaporisation of the material also conduces to inefficient deposition, and less of the vapor is deposited on the surface of the article. This leads to wastage of the coating material, which may or may not be economically tolerable, but it also results in deposits on parts of the apparatus, calling for more frequent cleaning, and resultant down time.

With the invention the heater element is composed of carbon fashioned into a filament, which may be a thread or tape, which is flexible. The material is applied to the element before the element is heated to vaporising temperature. By making the element of fine carbon fibres it will be given an adequate flexibility.

The degree of flexibility required is that the heater must be capable of being coiled and handled within the vacuum chamber without adversely affecting its mechanical and physical properties. As will appear, the use of a multifilament carbon fibre heater is advantageous in this as well as other respects. Suitable carbon fibres can be obtained by the carbonising appropriate organic compounds, in known manner; a large number of the fine diameter fibres are combined mechanically as by twisting, before carbonising the organic material of the fibres. Good results have been obtained by carbonising a material sold, for domestic knitting, as double ply material, under the trade mark Courtelle. This consists of artificial fibres, about 6 microns in diameter; after carbonising in known manner, the carbonised thread is about one-sixteenth inch in diameter. The fibres are staple fibres, with only a small degree of twist, so that the fibres all lie substantially along, or

only at a small angle to the length of the thread. A flat tape material made up of threads of this kind side by side, is particularly suitable. It is important that the length-wise fibres of the filament should be disposed substantially along the length of the filament without transverse displacement such as occurs, for example, with a conventional textile weave, where the warp threads are bent by the weft threads. The purpose of this is that the heater should be dimensionally stable, in the direction along its length, when heated.

The heater carries on or near its surface region the material to be vaporised; the material can be embodied by a mechanical or chemical means on the surface of the element by alloying or by forming a compound which is pyrolysable, but in any case the material is applied wholly over the surface of the heater. A heater element of this kind is shown in diagrammatic form, in FIGS. **3** and **4.** In FIG. **3** the four filaments **50** are comprised of individual fibres **51;** the adjacent edges of the four filaments can be made to adhere sufficiently to form the tape by light combing, whilst preserving substantially longitudinal disposition of the fibres.

The coating on the individual fibres is shown at **52** in FIG. **4.** It will be appreciated that the fibre and coating dimensions shown in FIGS. **3** and **4** are not to scale.

In some cases, material to be vaporised can be applied to the heater filament by a conventional coating method, including plating by electrolytic, electroless or mechanical plating. The material can also be applied to the filament as a powder coating, the filament being first treated with a suitable adhesive, or as a paint in suitable solvent or vehicle. The process used for applying the coating must preserve the flexibility of the material not only in the cold state, but also when heated, and the heater must remain dimensionally stable in the heated condition. It is for this reason that the lengthwise fibres should be axially disposed as described, for then the heater will retain its lengthwise dimension and will not sag or contract. This also precludes the use of a conventionally woven material where the warp threads yield to the weft threads. Such a material is not sufficiently stable when heated. Any deformation may cause the coating to become detached from the heater.

The coating material must not wet the surface of the heated element. However, by restricting the quantity of material on the heater the possiblity of the molten material running into droplets or blobs as described is obviated. By using a large number of fibres to form the filament heater, the quantity of coating material, within this limit, that can be used is increased. As the material on the heater reaches its melting point it will tend to form globules, but the globules are formed in initially a very small size, and there is insufficient material to permit aggregation of the small globules to form the large and localised detrimental globules as described above. The heat radiation is maintained substantially uniform and highly uniform vaporisation over the heated length of the heater takes place.

The very large numbers of very small diameter fibres make it extremely difficult to estimate the coating thickness, and the high temperature conditions in which the coating forms the globules and the fact that globulation is followed rapidly by evaporation, makes equally difficult an estimation of globule size. It is clear however by empirical methods to determine if the coating is of an excessive thickness then the globules, of excessive size, give rise to visible nonuniformity of heating or deposition or both.

In general, however, it is estimated that the coating will be only a few microns thick, and the globules only a few microns in diameter.

In a practical case, using a tape made of six threads as described, a suitable weight of coating has been estimated to be 15 to 25 mg per square cm. of heater surface.

Materials which are not electrically conductive can be used by applying the coating to the heater so as to leave the conductive heater exposed at intervals. Thus, in FIG. **5** the heater **31** is shown diagrammatically as coated with material **53,** leaving gaps at **31***a* at which contact can be made to the conductive heater surface by the contact rollers. In this case, the element is advanced step by step.

The process and apparatus described make possible improvements in other respects. For example by using a plurality of heaters with different evaporation materials composite deposits can be produced; such deposits may be homogeneous or may be in the nature of successive layers of different materials.

The coating material should not "wet" the heater, for if this occurs there is brought about a mechanical deformation which may be severe, or very severe. If the heater is wetted, the surface tension effect is great enough to distort the heater and affect vaporising operation. Uniformity of vaporisation or control, may be lost.

The invention can be used to produce magnetic coatings on base material, suitable for use in magnetic recording, by deposition of magnetic material including iron, cobalt, nickel and chromium, deposited as metals, or alloys, or solid solutions of such metals. The alloys or solid solutions can best be deposited directly from a plurality of heaters. When such materials are deposited, the deposit is initially of a very fine structure, such that the degree of resolution, as a magnetic recording medium, is very high, but the film is extremely thin. If the film thickness is increased, the magnetic particles agglomerate and the fineness of structure is diminished.

However, if a layer of non-magnetic material is deposited over the first magnetic layer, before the second deposition is made, the resolution is retained and the volume of magnetic material is increased. A very suitable non-magnetic material is nylon, having good anti-friction properties which are advantageous when contact-type magnetic heads are used. There may be as many repetitions of deposition steps as may be desired, the film being wound and rewound between reels **11** and **16** as necessary, by means of the reversible drive described.

Uniformity of deposit is improved if two, or an even number of heaters are used moving in opposite directions across the width of the web. Two identical heat assemblies inserted in the apparatus from opposite sides, give good uniformity.

Depending upon the size and arrangement of the vacuum chamber it is also possible to use relatively large supply and take up reels for the heater element wire, with sufficient wire to last for many operations of the apparatus. In this case, the removable element assembly will not offer any further substantial economic advantage.

Both sides of a web can be coated, with an arrangement as shown in FIG. **5;** filament heaters **31, 31***a* disposed between parallel spans of the web **10.** Two or more webs can be coated simultaneously for example to avoid contact of the coating surface of the web with

roller **14,** by using suitable guide roller arrangements.

The invention has been found to make a major improvement in vacuum deposition giving a much higher output from a given equipment and a product of improved uniformity. A wider range of materials, under closer control, can be deposited.

I claim:

1. A method of coating a surface of an article by the evaporation of a vaporizable coating material in a vacuum chamber, which method comprises the steps of carbonizing or graphitizing organic material to provide a flexible elongated electrically conductive heater element in the form of a tape which is inextensible under the conditions to which it is subjected in carrying out said method, forming a solid coating on the surface of said element with a quantity of said vaporizable material sufficient to substantially wholly cover the surface of said heater but insufficient to permit the formation of globules of said coating material more than a few microns in size, said vaporizable material having the property of not wetting the heater when at the vaporizing temperature of said material, said coated heater element retaining its flexibility when coated, disposing said article and said element with said material thereon in said chamber, providing electrical connections to regions of said element spaced along its length, heating that part of the element between said connections by passing electrical current through said connections and said part to bring said part of the element to a temperature which causes uniform vaporization of the material from substantially the full length of said part and deposition of said vaporized material on said surface of the article and, while maintaining a vacuum in said chamber, moving said element lengthwise to bring a different part of the element between said connections, and heating said different part.

2. A method as claimed in claim **1** in which said tape is fibrous.

3. A method as claimed in claim **1** in which said vaporizable material is a metal.

4. A method as claimed in claim **3** in which said metal is a magnetic metal.

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