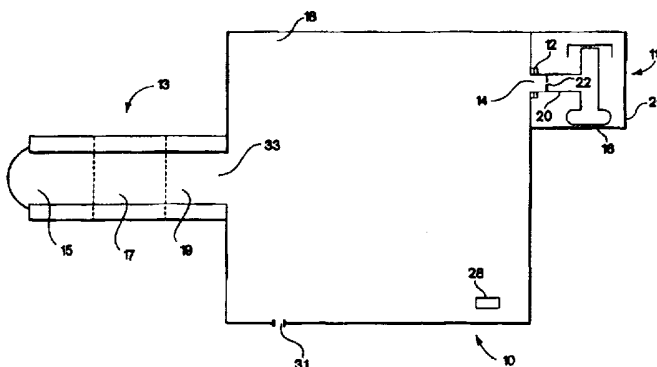




INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

<p>(51) International Patent Classification ⁶ : B05D 7/24, C23C 14/12</p>	<p>A2</p>	<p>(11) International Publication Number: WO 97/45209 (43) International Publication Date: 4 December 1997 (04.12.97)</p>
<p>(21) International Application Number: PCT/US97/09412 (22) International Filing Date: 29 May 1997 (29.05.97) (30) Priority Data: 08/657,820 31 May 1996 (31.05.96) US (71) Applicant: SPECIALTY COATING SYSTEMS, INC. [US/US]; 5707 West Minnesota Street, Indianapolis, IN 46241 (US). (72) Inventor: CRAIN, Kermit; 818 85th Street, Amery, WI 54001 (US). (74) Agent: MIRABITO, A., Jason; Wolf, Greenfield & Sacks, P.C., 600 Atlantic Avenue, Boston, MA 02110 (US).</p>		<p>(81) Designated States: CA, JP, European patent (AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE). Published <i>Without international search report and to be republished upon receipt of that report.</i></p>

(54) Title: CHAMBERS FOR PROMOTING SURFACE ADHESION UNDER VACUUM AND METHODS OF USING SAME



(57) Abstract

In one aspect, the present invention provides a method of depositing a layer of surface coating material. The method is carried out in a vacuum chamber and comprises the steps of depositing a layer of surface adhesion promotion material on a surface of a substrate; and depositing a layer of a surface coating material on a surface of the layer of the surface adhesion promotion material so that the layer of surface coating material has an adhesion of greater than about 3.75 pounds per inch according to the hesiometry test. In another aspect, the present invention provides an article of manufacture. The article comprises a substrate, a layer of surface adhesion promotion material and a layer of a surface coating material. The layer of surface adhesion material is supported by the substrate, and the layer of a surface coating material is supported by the layer of surface adhesion promotion material. The surface coating material has an adhesion of greater than about 3.75 pounds per inch as measured by the hesiometry test. In a further aspect, the present invention provides a vacuum chamber. The chamber comprises a body, a reservoir and plumbing. The body has an orifice with a diameter of less than about 75 mils disposed therein. The reservoir is capable of containing a surface adhesion material. The plumbing is disposed along the perimeter of the orifice of the body to physically connect the plumbing to the body. The plumbing is also physically connected to the reservoir. The plumbing has an orifice disposed therein such that the reservoir is in fluid communication with the body.

FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AL	Albania	ES	Spain	LS	Lesotho	SI	Slovenia
AM	Armenia	FI	Finland	LT	Lithuania	SK	Slovakia
AT	Austria	FR	France	LU	Luxembourg	SN	Senegal
AU	Australia	GA	Gabon	LV	Latvia	SZ	Swaziland
AZ	Azerbaijan	GB	United Kingdom	MC	Monaco	TD	Chad
BA	Bosnia and Herzegovina	GE	Georgia	MD	Republic of Moldova	TG	Togo
BB	Barbados	GH	Ghana	MG	Madagascar	TJ	Tajikistan
BE	Belgium	GN	Guinea	MK	The former Yugoslav Republic of Macedonia	TM	Turkmenistan
BF	Burkina Faso	GR	Greece	ML	Mali	TR	Turkey
BG	Bulgaria	HU	Hungary	MN	Mongolia	TT	Trinidad and Tobago
BJ	Benin	IE	Ireland	MR	Mauritania	UA	Ukraine
BR	Brazil	IL	Israel	MW	Malawi	UG	Uganda
BY	Belarus	IS	Iceland	MX	Mexico	US	United States of America
CA	Canada	IT	Italy	NE	Niger	UZ	Uzbekistan
CF	Central African Republic	JP	Japan	NL	Netherlands	VN	Viet Nam
CG	Congo	KE	Kenya	NO	Norway	YU	Yugoslavia
CH	Switzerland	KG	Kyrgyzstan	NZ	New Zealand	ZW	Zimbabwe
CI	Côte d'Ivoire	KP	Democratic People's Republic of Korea	PL	Poland		
CM	Cameroon	KR	Republic of Korea	PT	Portugal		
CN	China	KZ	Kazakistan	RO	Romania		
CU	Cuba	LC	Saint Lucia	RU	Russian Federation		
CZ	Czech Republic	LI	Liechtenstein	SD	Sudan		
DE	Germany	LK	Sri Lanka	SE	Sweden		
DK	Denmark	LR	Liberia	SG	Singapore		
EE	Estonia						

**CHAMBERS FOR PROMOTING SURFACE ADHESION UNDER VACUUM AND
METHODS OF USING SAME**

BACKGROUND

5

1. **Field of the Invention**

The present invention relates generally to chambers for promoting surface adhesion under vacuum as well as methods of using same, and more particularly to such chambers and methods which may be used with surface adhesion promotion materials as well as surface coating
10 materials.

2. **Background of the Invention**

In many applications, it is desirable to provide a layer of a surface coating material onto a surface to reduce the chemical reactivity of the surface. For example, subcutaneous probes may
15 be formed from materials that are capable of reacting with various chemical species within the human body, but it is desirable to prevent these chemical reactions from taking place. Therefore, it is advantageous to coat such subcutaneous probes with a surface coating material that renders the probes relatively chemically inert.

In recent years, vacuum deposition has become a popular method of applying surface
20 coating materials because this technique results in layers of surface coating materials that are inert and have acceptable adhesion. However, under some circumstances, a surface coating material may not readily bond to a surface of interest, resulting in comparatively poor adhesion of the surface coating material to the surface. As a result, several methods have been developed to increase the adhesion of the surface coating material by applying a surface adhesion promotion
25 material onto the surface prior to depositing the surface coating material.

U.S. Patent No. 3,600,216 discloses a method of applying surface adhesion promotion materials to surfaces using silane primers. According to this technique, the surface of a substrate is treated with a solution produced by dissolving the surface adhesion promotion material in a solvent. The surface of the substrate is then exposed to the solution by dipping. The treated
30 surface is subsequently dried to evaporate the solvent and provide the layer of surface adhesion promotion material. Next, the substrate is placed in a vacuum chamber and the vacuum chamber is pumped down. After the vacuum chamber reaches its base pressure, the surface is exposed to the surface coating material.

While this technique may have some success in producing layers of surface adhesion promotion materials on surfaces, this method has several drawbacks. The use of solvents can involve health and safety factors as well as storage and disposal concerns, resulting in increased cost. Furthermore, since the application of the surface adhesion promotion material is done
5 outside the vacuum chamber, this technique is labor intensive, resulting in decreased convenience and increased costs.

Canadian Patent No. 1,119,056 discloses a method of applying a surface adhesion promotion material, such as gamma-methacryloxypropyltrimethoxysilane (hereinafter "A174"), under vacuum conditions. According to this technique, the A174 is stored in a reservoir that is
10 connected the vacuum chamber through plumbing having a valve. The surface of interest is placed in the vacuum chamber, and the vacuum chamber is pumped down with the valve closed. The valve is then opened to allow to the A174 to enter the vacuum chamber while maintaining the A174 at or below room temperature. The valve is subsequently closed, and the surface coating material is deposited.

15 The inventor of the present application has found that the method disclosed in Canadian Patent No. 1,119,056 may be limited to relatively small sized vacuum chambers which are usually only appropriate for use in a laboratory setting. In particular, when this method is used with vacuum chambers designed for commercial use (i.e., vacuum chambers having a body with a volume of at least about 144 cubic inches), maintaining the A174 at or below room temperature
20 precludes the A174 from evaporating in sufficient quantity to become adequately distributed throughout the vacuum chamber to form an effective coating on the surface. Instead, the A174 has been observed to condense in the plumbing which connects the reservoir to the vacuum chamber, and the A174 either forms an unevenly distributed coating on the surface or does not bond to the surface at all. As a result, the subsequently deposited surface coating material
25 demonstrates inferior adhesion, rendering this method inappropriate for commercial use. In addition, because of the limitation on the size of the vacuum chamber with which this method can be effectively used, the substrates appropriate for use with this method are limited to having an area of at most about 100 square inches.

Therefore, it is desirable to provide commercially useful vacuum chambers that can be
30 used to form layers of surface adhesion promotion materials on surfaces such that subsequently deposited surface coating materials exhibit improved adhesion. It is further desirable to provide methods of using these vacuum chambers to provide layers of coating materials having improved

adhesion.

SUMMARY OF THE INVENTION

Accordingly, it is an object of the present invention to provide a vacuum chamber capable
5 of being used in the formation of layers of surface coating materials that exhibit improved
adhesion.

It is another object of the present invention to provide a method of preparing such layers
of surface coating materials.

It is a further object of the present invention to provide a substrate which supports a layer
10 of surface adhesion promotion material which, in turn, supports a layer of surface coating
material such that the surface coating material exhibits improved adhesion.

In one illustrative embodiment, the present invention provides a method of depositing a
layer of surface coating material. The method is carried out in a vacuum chamber and comprises
the steps of depositing a layer of surface adhesion promotion material on a surface of a substrate;
15 and depositing a layer of a surface coating material on a surface of the layer of the surface
adhesion promotion material so that the layer of surface coating material has an adhesion of
greater than about 3.75 pounds per inch according to the hesiometry test.

In another illustrative embodiment, the present invention provides an article of
manufacture. The article comprises a substrate, a layer of surface adhesion promotion material
20 and a layer of a surface coating material. The layer of surface adhesion material is supported by
the substrate, and the layer of a surface coating material is supported by the layer of surface
adhesion promotion material. The surface coating material has an adhesion of greater than about
3.75 pounds per inch as measured by the hesiometry test.

In a further illustrative embodiment, the present invention provides a vacuum chamber.
25 The chamber comprises a body, a reservoir and plumbing. The body has an orifice with a
diameter of less than about 75 mils disposed therein. The reservoir is capable of containing a
surface adhesion material. The plumbing is disposed along the perimeter of the orifice of the
body to physically connect the plumbing to the body. The plumbing is also physically connected
to the reservoir. The plumbing has an orifice disposed therein such that the reservoir is in fluid
30 communication with the body.

BRIEF DESCRIPTION OF THE DRAWINGS

Fig. 1 is a schematic representation of one embodiment of a vacuum chamber according to the present invention;

Fig. 2 is cross-sectional view of one embodiment of a substrate having disposed thereon a layer of surface adhesion promotion material which has disposed thereon a layer of surface coating material; and

Fig. 3 is a schematic representation of one embodiment of an apparatus used for performing hesiometry tests according to the present invention.

DETAILED DESCRIPTION

In one aspect, the present invention relates to vacuum chambers that can be used to form layers of surface adhesion promotion materials on surfaces such that subsequently deposited surface coating materials exhibit improved adhesion. Fig. 1 depicts one embodiment of a vacuum chamber 10 which includes adhesion promotion deposition compartment 11. According to the present invention, compartment 11 is designed to allow a relatively even distribution of a surface adhesion promotion material to be disposed onto the surface of a substrate 28, located within the body 18 of chamber 10, such that a subsequently deposited layer of a surface coating material exhibits improved adhesion.

Compartment 11 includes a weldment stub 12 which is welded to vacuum chamber 10 around the perimeter of a hole 14 which is drilled into body 18 of chamber 10. A reservoir 16, containing A174, is in fluid communication with body 18 orifice 22 and plumbing 20 which is connected to stub 12. Heating apparatus 26, which is held in place by mounting brackets (not shown), surrounds reservoir 16. Pump exhaust 31 of vacuum chamber 10 is disposed such that exhaust 31 is located in a diagonal direction across chamber 10 as far as possible from hole 14. In addition, it is to be noted that reservoir 16 is in a high position relative to exhaust 31.

With this arrangement, a layer of A174 is coated onto the surface of substrate 28 as follows. The pressure of vacuum chamber 10 is reduced to its minimum pressure using standard methods as known to those skilled in the art. The temperature of reservoir 16 is subsequently increased from room temperature to about 190°C by use of heating apparatus 26. At this temperature, the A174 is evaporated from reservoir 16 in a controlled fashion. The evaporated A174 then travels through plumbing 20 to orifice 22 and passes through orifice 22 to enter body 18 of chamber 10. Since the A174 was evaporated at an elevated temperature, it contains a large

amount of kinetic energy relative to A174 at or below room temperature, so the A174 easily travels within body 18 and is exposed to a substantial portion of the volume of body 18.

Furthermore, because substrate 28 is located the maximum diagonal distance from hole 14, the density of the evaporated A174 is approximately homogeneous in the region around the surface of substrate 28. Thus, the A174 which deposits onto the surface of substrate 28 forms a relatively evenly distributed layer. It is believed that the formation of a relatively evenly distributed layer of surface adhesion promotion material on the surface of substrate 28 allows a subsequently deposited layer of surface coating material to exhibit improved adhesion. It is to be noted that, during the process of deposition of A174 on the surface of substrate 28, the vaporized A174 may also coat portions of the inner walls of various parts of vacuum chamber 10.

It has been unexpectedly found that vacuum chamber 10 can be pumped down to its minimum pressure without valving off reservoir 16 which contains A174 at room temperature. In particular, contrary to the conventional wisdom, the vapor pressure of A174 has been found to be low enough that evaporation of A174 does not prevent vacuum chamber 10 from reaching its minimum pressure when the A174 is held at room temperature. It is believed that orifice 22 is capable of reducing the conductance of A174 from reservoir 16 to body 18 which assists chamber 10 in obtaining its minimum pressure without complications due to the evaporation of A174.

One common problem with deposition steps in vacuum chambers is associated with the steps of pumping out and subsequently valving off the reservoir and associated plumbing prior to pumping down the body of the vacuum chamber. In many instances, an operator of a vacuum chamber forgets to first pump out the reservoir and associated plumbing prior to valving them off. As a result, when the valve to the reservoir and associated plumbing is opened after pumping down the main chamber, the vacuum chamber pressure increases dramatically. This can lead to venting of the vacuum chamber, destruction of vacuum chamber equipment (e.g., pressure sensitive electronics stored within the vacuum chamber) and/or contamination of the substrate surface. This results in a loss of a substantial amount of labor and resources, increasing the costs associated with this process. Therefore, by eliminating the need of a valve between reservoir 16 and body 18, vacuum chambers in accordance with the present invention can provide a more efficient and less expensive process.

While one particular arrangement of adhesion promotion deposition compartment 11 has been disclosed herein, the present invention is intended to include other arrangements. The

particular arrangement of the components of compartment 11 is limited only in that compartment 11 should be capable of producing a sufficiently evenly distributed layer of surface adhesive material on the surface of substrate 28 such that a subsequently deposited layer of surface coating material exhibits improved adhesion.

5 For example, in embodiments in which body 18 of chamber 10 is relatively long and horizontal, reservoir 16 should be located near orifice 33 of pyrolysis chamber 19 (described below).

Reservoir 16 may be any vacuum compatible container that is capable of housing any surface adhesion promotion material. Reservoir 16 should also be chemically inert to the surface
10 adhesion promotion material contained therein at or below the temperature used to deposit the surface adhesion promotion material. One example of such a reservoir is a 100 mL 304 stainless steel tube.

Heating apparatus 26 may be any device that is capable of heating reservoir 16 and the surface adhesion material contained therein in a controlled fashion. Apparatus 26 may also be
15 capable of being mounted to chamber 10 by brackets 24a and 24b, or the like, while supporting reservoir 16. In certain embodiments, apparatus 26 may surround reservoir 16. One example of such a heating apparatus is a small oven.

Plumbing 20 may be formed from any vacuum compatible material that is capable of fluidly connecting reservoir 16 to hole 14. Plumbing 20 should also be chemically inert to A174.
20 In one embodiment, plumbing 20 is formed from a Viton® O-ring, a 1/8" male coupling and a VCO female connector.

Hole 14 and weldment stub 12 may have any size diameter so long as they are capable of being welded together to form a vacuum tight seal that allows reservoir 16 to be fluidly
connected to body 18. Since stub 12 typically is available in predetermined sizes (e.g., 1/8"), it
25 may be advantageous for hole 14 to have a diameter which corresponds to one these predetermined sizes. In one embodiment, hole 14 and weldment stub 12 each have a diameter of about 1/8". In certain embodiments, weldment stub 12 may be replaced by an equivalent connecting device, such as a 1/8" Union or a female fractional tube adjuster.

It is believed that orifice 22 may reduce the conductance of vaporized surface adhesion
30 promotion material from reservoir 16 to body 18 such that, subsequent to being heated by apparatus 26, the flow of vaporized surface adhesion promotion material into body 18 produces a relatively homogeneous density distribution of vaporized surface adhesion promotion material

throughout portions of body 18. This allows the vaporized surface adhesion promotion material to form a relatively evenly distributed layer on the surface of substrate 28, allowing a subsequently deposited layer of surface coating material to exhibit improved adhesion.

Therefore, orifice 22 may be of any size so long as orifice 22 is capable of restricting the flow of evaporated surface adhesion promotion material from reservoir 16 to body 18 so that the surface adhesion material forms a relatively evenly distributed layer on the surface of substrate 28 and so that a subsequently deposited layer of surface coating material exhibits improved adhesion. It has been found that, in some embodiments, orifice 22 is ineffective if it has a diameter greater than about 100 mils (1mil = 0.001"). Hence, for such embodiments, orifice 22 should have a diameter of less than about 75 mils. In one embodiment, orifice 22 has a diameter of about 16 mils.

Vacuum chamber 10 may be any vacuum chamber which is capable of being modified to include compartment 11. In addition, chamber 10 should include surface coating deposition compartment 13 (Fig. 1). Preferably, body 18 of chamber 10 has a volume of at least about 144 cubic inches, more preferably at least about 288 cubic inches and most preferably at least about 432 cubic inches. Such vacuum chambers include, but are not limited to, the following commercially available vacuum chambers, each of which is available from Specialty Coating Systems, Inc, Clear Lake, WI: model 1050 (part no. 2169800); model 1030 (part no. 216802); model 2090 (part no. 2169803); model 2010 (part no. 2169804); and model 2060 (part no. BM02616).

As shown in Fig. 1, surface coating deposition compartment 13, which is appropriate for use with the parylene family of surface coating materials, should include vaporization chamber 15, pyrolysis chamber 17 and post-pyrolysis chamber 19. Vaporization chamber 15 evaporates the parylene dimer, pyrolysis chamber 17 cleaves the parylene dimer to form a gas including parylene monomer, and post-pyrolysis chamber includes at least one cooled baffle to remove any parylene dimer from the gas. Such deposition compartments are disclosed in commonly assigned U.S. Patent Serial No. 08/549,087 which is herein incorporated by reference. While certain embodiments of compartment 13 have been disclosed herein, it is to be understood that compartment 13 is not limited by these embodiments. Compartment 13 may include any combination of devices that are capable of acting in unison to deposit a layer of surface coating material onto a layer of surface adhesion promotion material disposed on the surface of substrate 28. Such compartments are known to those skilled in the art and are intended to be within the

scope of the present invention.

Methods of depositing a layer of surface coating material using compartment 13 include the steps of vaporizing the parylene dimer, pyrolyzing the parylene to form a gas including monomers of the parylene, passing the gas through at least one cooled baffle to remove any parylene dimer in the gas, and depositing the monomer of parylene on the layer of the surface
5 adhesion promotion material which is disposed on the surface of substrate 28. Such methods are described in, for example, U.S. Patent Application Serial No. 08/549,087. Other methods of depositing a layer of surface coating material are known to those skilled in the art and are intended to be within the scope of the present invention.

10 Fig. 2 depicts a cross-sectional view of one embodiment of substrate 28 having a surface 30 on which a layer 32 of surface adhesion promotion material is deposited. A layer 34 of surface coating material is disposed on layer 32. Surface 30 may have any area so long as layer 32 can be deposited thereon such that subsequently deposited layer 34 exhibits improved adhesion. While Fig. 1 depicts chamber 10 having one substrate 28 disposed therein, it is to be
15 understood that chamber 10 may have a plurality of substrates disposed therein. In certain embodiments in which a plurality of substrates are disposed within chamber 10, the total surface area of the substrates is preferably at least about 100 square inches.

 According to the present invention, substrate 28 may be any object or body having surface 30 onto which a surface adhesion promotion material may be deposited by at least one of
20 the methods of the present invention. Substrate 28 may be formed from organic materials or inorganic materials including, for example, polymeric materials, semiconductor materials, metallic materials, oxides of metallic materials and the like. Such organic materials and inorganic materials include, but are not limited to, aluminum, iron, steel, molybdenum, aluminum oxide, titanium oxide, lead oxide, copper oxide, iron oxide, beryllium oxide,
25 manganese oxide, tungsten oxide, tantalum oxide, vanadium oxide, silicones, natural rubbers, plastic composites, cellulosic materials, epoxy-containing compounds, thermosetting compounds, thermoplastic compounds, silicon oxide (e.g., sand, fly ash, hydrated silica, silica, quartz, aerogel, xerogel, fumed silica) and the like. Substrate 28 may also be formed from a vacuum compatible liquid. By "vacuum compatible" it is herein meant to refer to a material that
30 has a vapor pressure at room temperature such that the minimum pressure to which a vacuum chamber can be pumped is independent of the presence of the vacuum compatible material. One example of such a vacuum compatible liquid is A174. Other such vacuum compatible materials

will be apparent to those skilled in the art and are intended to be within the scope of the present invention.

In certain embodiments, substrate 28 may be a printed circuit board, a silicon wafer, paper, a key pad, a catheter, a pacemaker cover, a subcutaneous probe, a bird feather, a silicone
5 O-ring or the like.

Layer 32 of surface adhesion promotion material may be formed from any vacuum compatible material that is capable of simultaneously bonding to surface 30 and layer 34 such that layer 34 has an adhesion of greater than about 3.75 pounds per inch (lbs/in) as measured according to the hesiometry test, more preferably at least about 5 lbs/in and most preferably at
10 least about 8 lbs/in.

As used herein, the term "hesiometry test" refers to a test which was developed by W. K. Asbeck, *Paint and Varnish Prod.* p. 23, 1970. Fig. 3 represents the setup used in the hesiometry test. Substrate 30 is placed on a motorized stage 40 which moves in a direction indicated by the arrow. As stage 40 moves, a knife-like device 42 cuts channels in layers 32/34. Device 42 is
15 pushed through the interface between substrate 28 and layers 32/34, and the horizontal force (F_h) is measured by a transducer 44. Substrate 28 is returned to its starting point and run again so that the contribution due to the frictional force between device 42 and surface 30 can be measured. The difference between the horizontal force and the frictional force is equal to the adhesion of layer 34.

20 Although layer 32 of surface adhesion promotion material may be formed from any material having the properties discussed above, layer 32 is preferably formed from an organosilane, more preferably from an alkoxysilane, and most preferably from A174 (i.e., gamma-methacryloxypropyltrimethoxysilane).

In some embodiments, layer 32 is formed from a multi-layer of molecules of surface
25 adhesion promotion material. In these embodiments, some molecules of surface adhesion promotion material bond with surface 30 while other molecules of surface adhesion promotion material do not bond with surface 30. A "bond" as used herein refers to any combination of chemical bonding, physical entanglement or physical bonding, such as hydrogen bonding or van der Waals bonding.

30 For embodiments in which layer 32 is formed from a multi-layer of molecules of surface adhesion promotion material, layer 32 should have a thickness of at least about 20 Å as measured

using a quartz crystal thickness monitor or an equivalent device. It is to be noted that if layer 32 is less than about 20 Å thick, layer 34 may not have sufficient adhesion.

In some embodiments, molecules of surface adhesion promotion materials have a polar end and a nonpolar end. For example, A174 has a polar end (trimethoxy) and a nonpolar end (methacryloxy). While it is not fully understood, it is believed that, when such surface adhesion promotion materials are used with surfaces that are more polar than the nonpolar end of the molecules of the surface adhesion material, the polar ends of the molecules of the surface adhesion material bond to the surface and the nonpolar ends of the molecules of the surface adhesion material are directed away from the surface. With this configuration, the molecules of the surface coating material are exposed to the nonpolar ends of the molecules of the surface adhesion material rather than the comparatively polar surface. If the surface molecules of the surface coating material are nonpolar relative to the surface, the surface molecules of the surface coating material may bond more readily to the nonpolar end of the molecules of the surface adhesion material than the relatively polar surface.

Layer 34 of surface coating material may be formed from any material that is capable of bonding to layer 32 and which is capable of forming a layer of material that is chemically inert relative to surface 30. Such materials are known to those skilled in the art and include, for example, poly-p-xylylene materials. An illustrative and nonlimiting list of such poly-p-xylylenes includes parylene D, parylene N, parylene C and octafluoro-[2,2] paracyclophane such as disclosed in commonly assigned U.S. Patent Application Serial No. 08/544,831 which is hereby incorporated by reference.

In certain embodiments, layer 34 may have different physical properties than surface 30. For example, layer 34 may have a different roughness, electrical conductivity and/or thermal conductivity than surface 30.

The following examples are intended for illustrative purposes and is not to be construed as limiting.

Example 1

A substrate without a layer of surface coating material was deposited on a substrate as follows. A borosilicate substrate was cleaned by hand washing with a 2% solution of Microsoap® in water. The substrate was rinsed with de-ionized water and placed in an autoclave. The substrate was heated in the autoclave at 250°F for about one hour in the presence of de-

ionized water. The substrate was removed from the autoclave, and any remaining liquid was blown off with ultrapure nitrogen gas. The substrate was then vacuum baked at a temperature of about 50°C at a pressure of less than 100 mTorr.

The cleaned substrate was then coated with parylene C by first vaporizing the solid dimer of parylene C. The methylene-methylene bonds of the parylene were then cleaved in the pyrolysis chamber at a temperature of about 680°C to yield monomers of the parylene C. The monomer was subsequently deposited onto the layer of A174. Upon deposition, the monomers of A174 polymerized. The coated substrate was removed from the vacuum chamber stressed by placement in an autoclave for a time period of about one hour at a temperature of about 250°F in the presence of de-ionized water.

The adhesion of the coating of parylene C material was then measured using the hesiometer test described above. This experiment was repeated four times to achieve a total of five data points. The average adhesion was measured to be about 0.25 lbs/in according to the hesiometry test.

15

Example 2

A substrate having a layer of surface adhesion promotion material deposited in substantial accordance with the method disclosed in U.S. Patent No. 3,600,216 and a further layer of a surface coating material was prepared as follows. A borosilicate substrate as described in Example 1 was cleaned according to the method disclosed in Example 1. A layer of A174 was applied to the surface of the borosilicate substrate by dipping the in a solution of 0.5% A174, 49.75% de-ionized water and 49.75% isopropanol. This dipping procedure was carried out for about 15 minutes. The substrate was then dried for about 15 minutes and subsequently rinsed with neat isopropanol. The substrate was next air dried for about one half hour.

The layer of A174 provided by the above method was coated with parylene C and stressed as described in Example 1. The adhesion of the coating of parylene C material was then measured using the hesiometer test described above. This experiment was repeated four times to achieve a total of five data points. The average adhesion was measured by the hesiometry test to be about 2.25 lbs/in.

30

Example 3

A substrate with a layer of surface adhesion promotion material was prepared as follows. A borosilicate substrate as described in Example 1 was cleaned according to the method disclosed in Example 1. A borosilicate substrate as described in Example 1 was cleaned
5 according to the method of Example 1. The substrate was then placed in a vacuum chamber, and a layer of A174 was applied to the surface of the substrate under vacuum conditions. The coated substrate was then removed from this vacuum chamber and transferred to a second vacuum chamber in accordance with methods described herein (i.e., the coated substrate was exposed to atmospheric conditions prior to the deposition of the surface coating material).

10 The layer of A174 was then coated with parylene C and stressed as described in Example 1. It is to be noted that the coated substrate was exposed to a substantial amount of dynamic pumping in the second vacuum chamber prior to the deposition of the surface coating material. The adhesion of the coating of parylene C material was then measured using the hesiometer test described above. This experiment was repeated four times to achieve a total of five data points.
15 The average adhesion was measured by the hesiometry test to be about 3.75 lbs/in.

Example 4

A borosilicate substrate as described in Example 1 was cleaned according to the method of Example 1. The substrate was then placed in a model 1050 parylene generation vacuum
20 chamber (part no. 2169800, Specialty Coatings Systems, Inc., Clear Lake, WI) which was modified as shown in Fig. 1. The pressure of the chamber was reduced to about 50 milliTorr. About 2.5 mL of A174 was vaporized at a temperature of about 190°C, and the thickness of the coating of A174 was monitored using a quartz crystal thickness monitor (Model No. TM 100, available from Maxtek). The layer of A174 was then coated with parylene C and stressed as
25 described in Example 1. The layer of A174 was about 227 Å and the layer of parylene C was from about 4000 Å to about 5000 Å. Adhesion of the coating of parylene C material was then measured using the hesiometer test described above. This experiment was repeated four times to achieve a total of five data points. The average adhesion was measured to be about 8 lbs/in according to the hesiometry test.

30 As indicated by Example 1, when a layer of A174 is not applied to the borosilicate surface prior to the deposition of the parylene material, the parylene material has the poorest

adhesion. Furthermore, when a layer of A174 is applied from solution (Example 2), the subsequently deposited layer of parylene material exhibits poor adhesion. In addition, when the A174 coated borosilicate substrate is exposed to atmospheric conditions prior to the deposition of the parylene material (Example 3), the layer of parylene material exhibits inferior adhesion.

5 However, when the process of the present invention is used to apply the layers of A174 and parylene material to the borosilicate substrate (Example 4), the layer of parylene material exhibits better adhesion than when the methods of Examples 1-3 are used.

Having thus described certain embodiments of the present invention, various improvements, modifications and alterations will be obvious to those skilled in the art. Such
10 improvements, modifications and alterations are intended to be within the spirit and scope of the present invention. Accordingly, the foregoing description is by way of example only and is not intended as limiting. The invention is limited only as defined in the following the claims and equivalents thereto.

What is claimed is:

CLAIMS

1. A method of depositing a layer of surface coating material, the method comprising the steps of:
 - 5 depositing, within a vacuum chamber, a layer of surface adhesion promotion material on a surface of a substrate; and
 - depositing, within the vacuum chamber, a layer of a surface coating material on a surface of the layer of the surface adhesion promotion material so that the layer of surface coating material has an adhesion of greater than about 3.75 pounds per inch according to a hesiometry
 - 10 test.

2. The method according to claim 1, wherein the step depositing a layer of surface coating material includes depositing the layer of surface adhesion promotion material so that the layer of surface coating material has an adhesion of at least about 5 pounds per inch as measured using
- 15 the hesiometry test.

3. The method according to claim 1, wherein the step of depositing a layer of surface adhesion promotion material includes reducing the pressure of the vacuum chamber while exposing the vacuum chamber to a reservoir of the surface adhesion promotion material.
- 20

4. The method according to claim 1, wherein the step of depositing a layer of surface adhesion promotion material includes heating a reservoir of the surface adhesion promotion material to a temperature above room temperature.

- 25 5. The method according to claim 1, wherein the step of depositing a layer of surface adhesion promotion material includes depositing a layer of surface adhesion promotion material having a thickness of at least about 20 Å.

6. The method according to claim 1, wherein the step of depositing a layer of surface
- 30 adhesion promotion material includes passing the surface adhesion promotion material through an orifice having a diameter of less than about 75 mils.

7. The method according to claim 1, wherein the step of depositing a layer of surface adhesion promotion material on a surface of a substrate includes depositing a layer of surface adhesion promotion material on a surface of each of a plurality of substrates, a total surface area
5 of the surface of each of the plurality of substrates being at least about 100 square inches.
8. An article of manufacture, comprising:
a substrate;
a layer of surface adhesion promotion material supported by the substrate; and
10 a layer of a surface coating material supported by the layer of surface adhesion promotion material, the surface coating material having an adhesion of greater than about 3.75 pounds per inch as measured by a hesiometry test.
9. The article according to claim 8, wherein the surface coating material has an adhesion of
15 at least about 5 pounds per inch as measured by the hesiometry test.
10. The article according to claim 8, wherein the surface coating material has an adhesion of at least about 8 pounds per inch as measured by the hesiometry test.
- 20 11. The article according to claim 8, wherein the surface adhesion promotion material is formed from gamma-methacryloxypropyltrimethoxysilane.
12. The article according to claim 8, wherein the surface adhesion promotion material has a
25 thickness of at least about 20Å.
13. A vacuum chamber, comprising:
a body having an orifice disposed therein, the orifice of the body having a perimeter;
a reservoir capable of containing a surface adhesion material; and
plumbing disposed along the perimeter of the orifice of the body to physically connect
30 the plumbing to the body, the plumbing being physically connected to the reservoir, the plumbing having an orifice disposed therein such that the reservoir is in fluid communication

with the body.

14. The vacuum chamber according to claim 13, further comprising a heating apparatus physically connected to the body, the heating apparatus disposed adjacent the reservoir so that
5 the heating apparatus can heat the reservoir.
15. The vacuum chamber according to claim 13, wherein the heating apparatus is capable of heating the reservoir to a temperature of at least about 190°C.
- 10 16. The vacuum chamber according to claim 13, wherein the orifice of the plumbing has a diameter of less than about 75 mils.
17. The vacuum chamber according to claim 13, wherein the body has a volume of at least about 144 cubic inches.
15
18. The vacuum chamber according to claim 13, wherein the body has a volume of at least about 288 cubic inches.
19. The vacuum chamber according to claim 13, wherein the body has a volume of at least
20 about 432 cubic inches.
20. The vacuum chamber according to claim 13, further comprising surface coating deposition compartment.

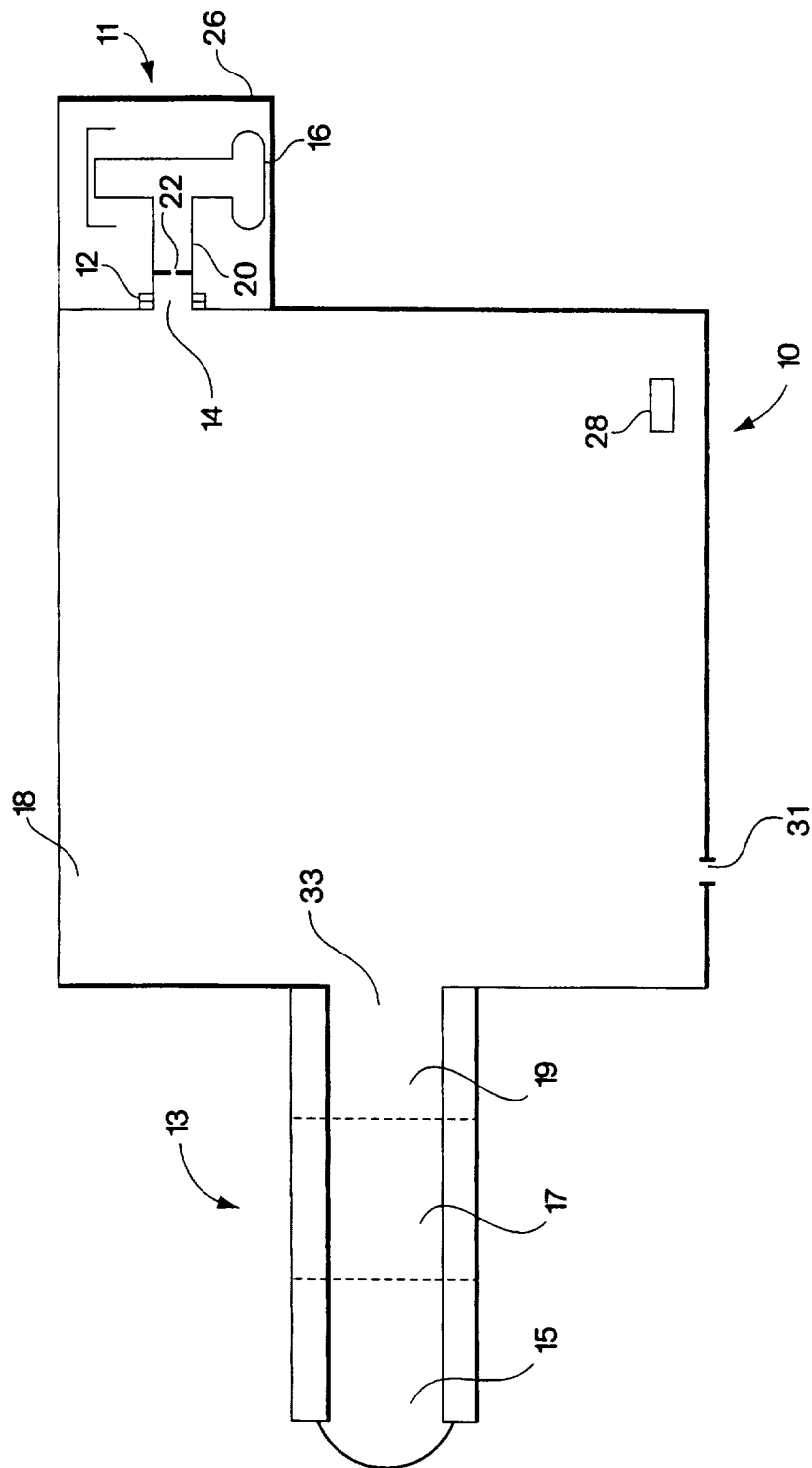


Fig. 1

2/2

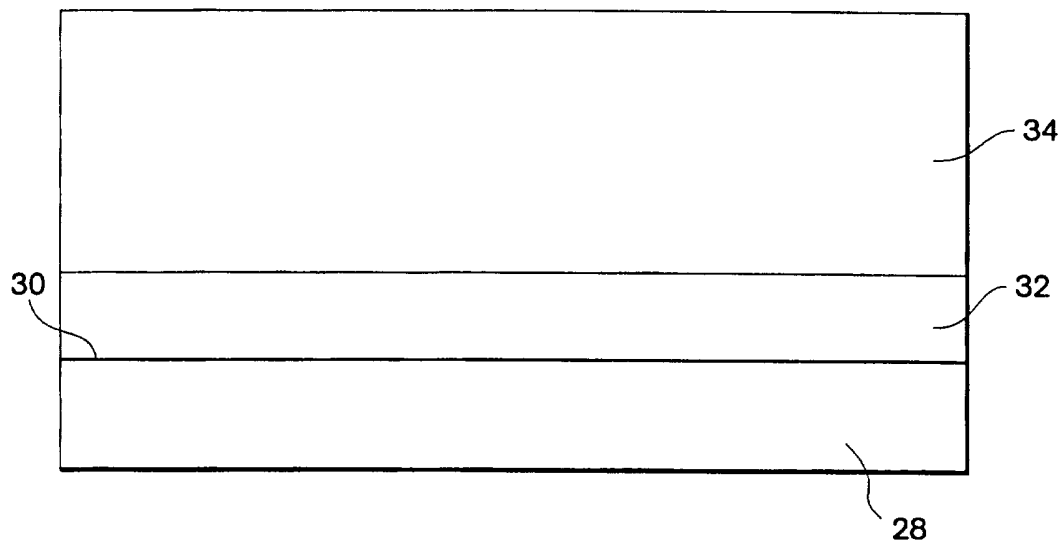


Fig. 2

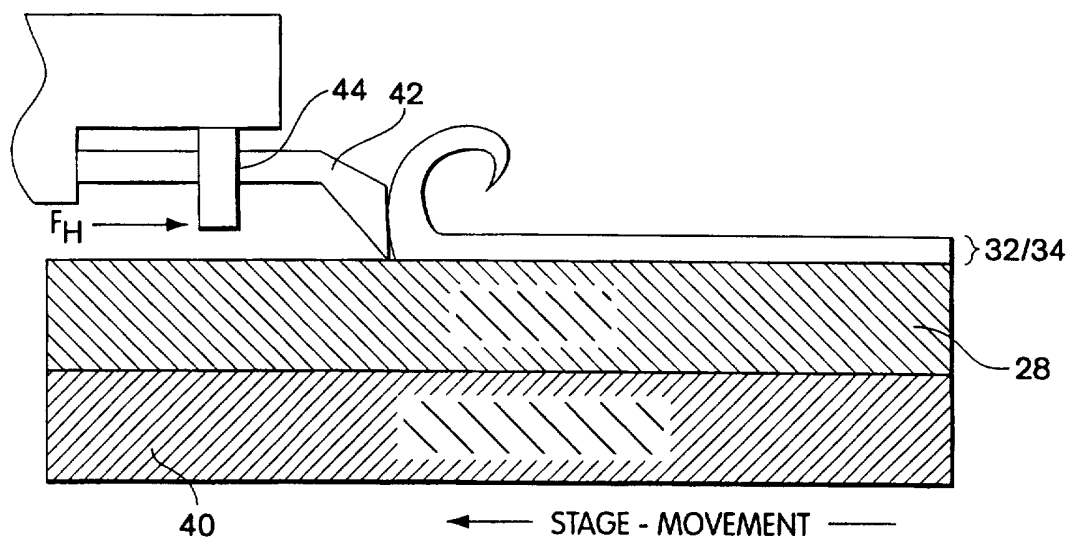


Fig. 3