United States Patent [19]

Boom

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[54] **VACUUM LOCK FOR PLASMA TREATMENT OF SUBSTRATES**

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[21] Appl. No.: **292,455**

Related U.S. Application Data

[62] Division of Ser. No. 171,282, Aug. 12, 1971, Pat. No. 3,723,289.

[52] **U.S. Cl.** ..... **34/242,** 34/15, 68/5 E, 68/DIG. 1, 277/237 R, 432/242

[51] **Int. Cl.** **F26b 25/00**

[58] **Field of Search** ..... 34/15, 22, 70, 151, 152, 34/155, 242; 432/115, 242; 68/5 E, DIG. 1; 277/70, 237 R

[56] **References Cited**

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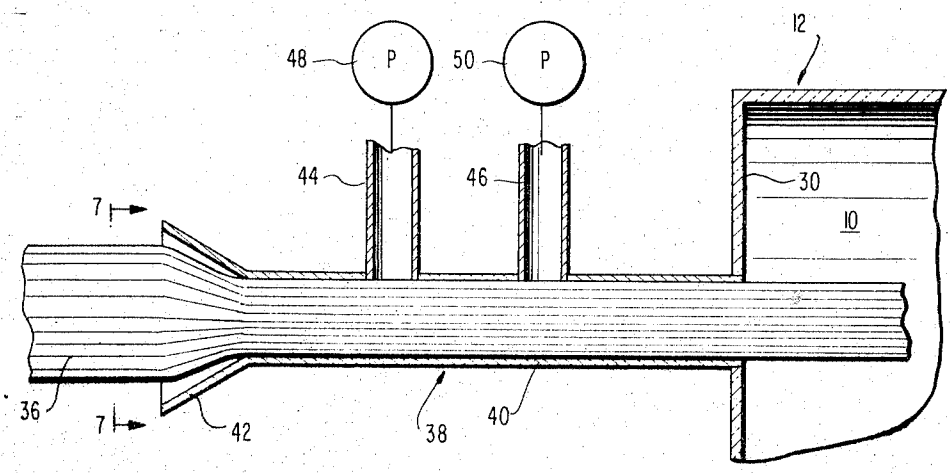
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Assistant Examiner—James C. Yeung

[57] **ABSTRACT**

A method and apparatus for efficiently generating a gaseous plasma particularly for the treatment of substrates. A radio frequency electrical signal is applied to two electrodes disposed exteriorly of an electrically insulative, gas impervious envelope. A central passage extends into the envelope and one electrode is disposed in the central passage. The electrodes are separated at least in part by the envelope and the radio frequency signal applied to the electrodes excites the gas within the envelope to thereby generate a gaseous plasma therein. The gas conditions within the envelope differ from the gas conditions exteriorly thereof and the amplitude of the radio frequency signal is insufficient to generate a plasma outside the chamber defined by the envelope. Since the plasma does not contact the electrodes, efficiency is maximized and the plasma is not contaminated by the electrodes. In addition, the surface areas of the electrodes differ substantially thereby creating a plasma within the envelope which varies in concentration in a predetermined manner, with the concentration being greatest near the center of the envelope. A substrate may therefore be contacted by varying plasma concentration as it passes through the envelope and the outer wall of the envelope is not contaminated by the plasma. A vacuum lock for preventing gas leakage into the envelope is also disclosed.

11 Claims, 8 Drawing Figures



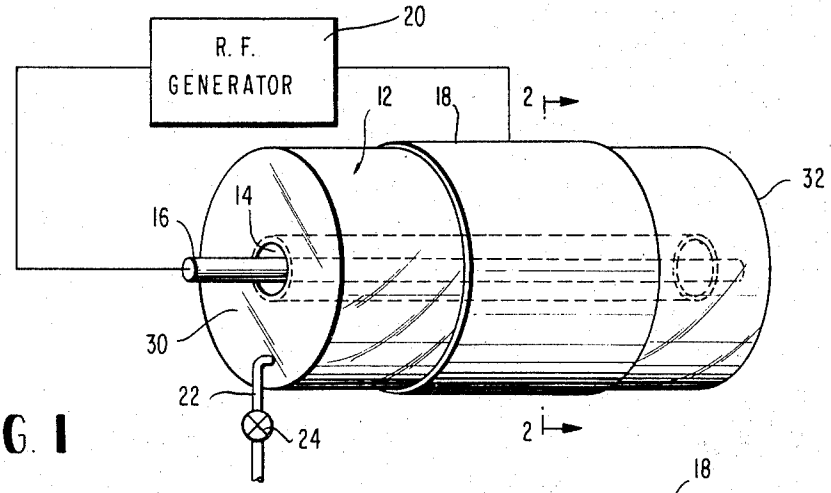


FIG. 1

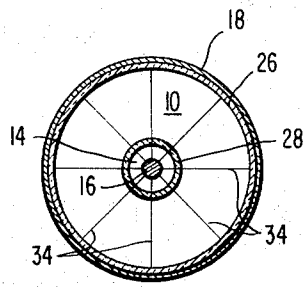


FIG. 2

FIG. 3

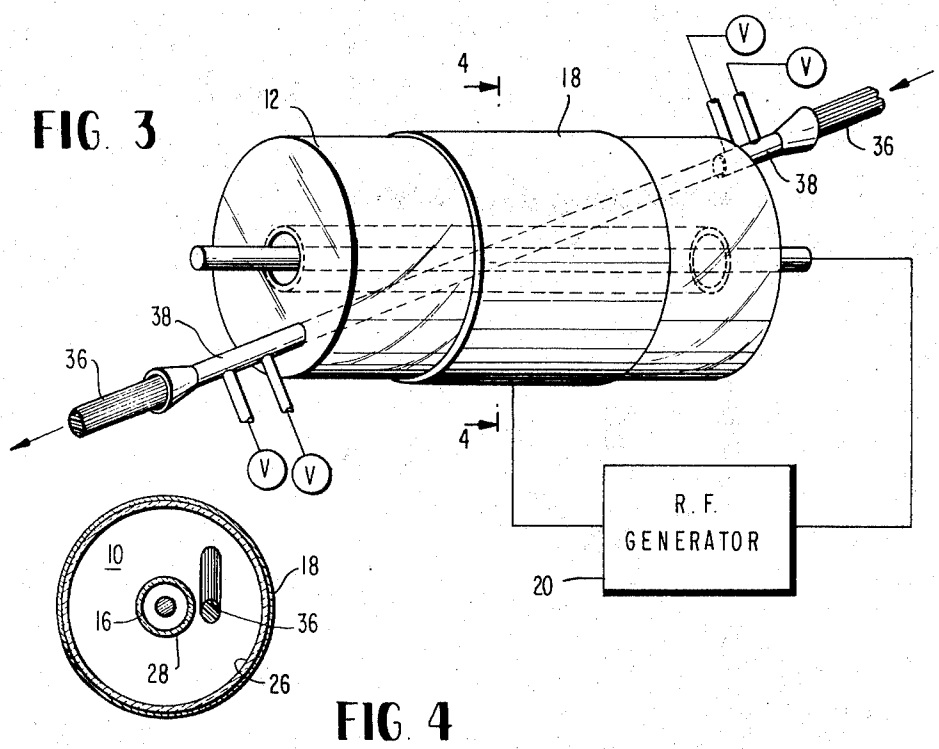


FIG. 4

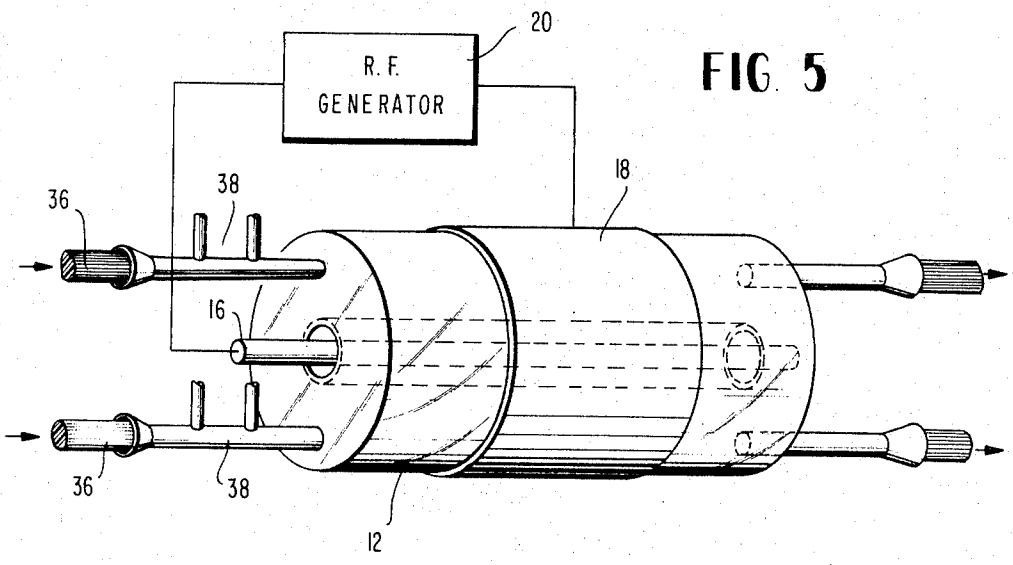


FIG. 5

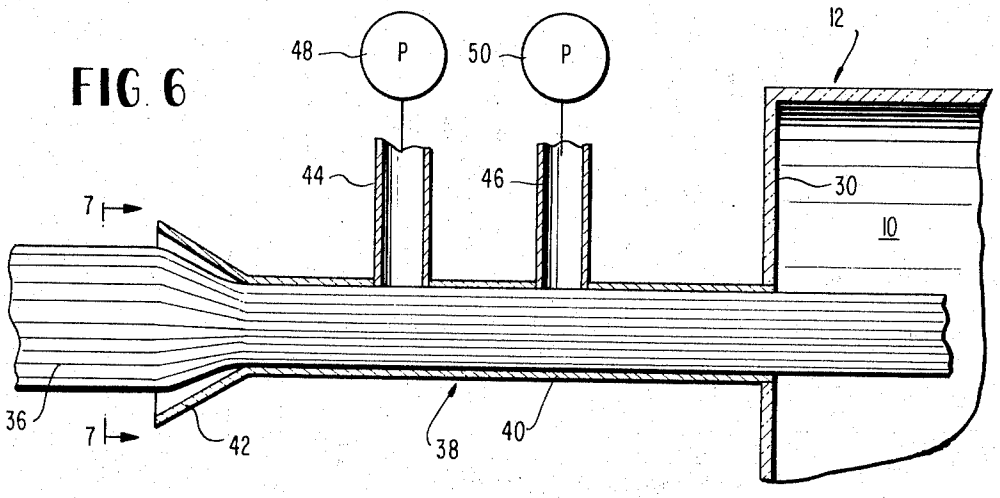


FIG. 6

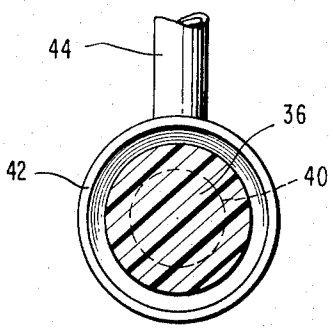


FIG. 7(A)

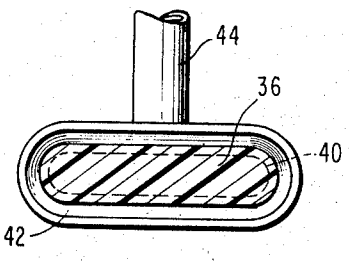


FIG. 7(B)

VACUUM LOCK FOR PLASMA TREATMENT OF SUBSTRATES

This is a division of application Ser. No. 171,282 filed Aug. 12, 1971, now issued as U.S. Pat. No. 3,723,289 and assigned to the assignee hereof.

BACKGROUND OF THE INVENTION

The present invention relates to a vacuum lock for apparatus for treating substrates.

It is often desirable to maintain the pressure of the gas within a reaction chamber at a pressure lower than that of the gas outside the chamber. The pressure differential between the interior and exterior of the chamber may create problems in introducing the substrate into the chamber. This may be a particular problem where expensive and/or harmful gases are utilized for treatment of the substrates since the escape of these gases or the leakage of undesired gases into the chamber may increase the cost of the process, result in hazardous working conditions, and/or adversely affect the treatment of the substrate.

It is therefore an object of the present invention to provide a novel method and apparatus for passing a substrate from a zone at one pressure into a zone at another pressure without substantial leakage therebetween.

These and other objects of the present invention will become apparent to one skilled in the art to which the invention pertains from a perusal of the following description when read in conjunction with the appended drawings.

THE DRAWINGS

FIG. **1** is a schematic representation of a reaction chamber embodying the present invention;

FIG. **2** is a view in cross section of the reaction chamber of FIG. **1**, taken along the line **2—2**;

FIG. **3** is a schematic representation of the reaction chamber of FIG. **1** with a substrate being treated therein;

FIG. **4** is a view in cross section of the reaction chamber of FIG. **3**, taken along the line **4—4**;

FIG. **5** is a schematic representation of a reaction chamber similar to the chamber shown in FIG. **3** with a plurality of substrates being treated therein;

FIG. **6** is a view in partial cross section of the reaction chamber of FIG. **5** illustrating the vacuum lock of the present invention; and,

FIGS. **7A** and **7B** are end views of the vacuum lock of FIG. **6**, taken along the line **7—7** thereof, and illustrate two of the alternative shapes which the vacuum lock may have.

DETAILED DESCRIPTION

Referring to FIGS. **1** and **2** wherein a preferred embodiment of a reaction chamber is illustrated, a reaction chamber **10** is formed by a substantially gas impervious, generally electrically non-conductive or insulative envelope **12** into which a central passage **14** extends. An electrode **16** extends into the central passage **14** and is isolated from the chamber **10** by the radially inward wall of the envelope **12.** An electrode **18** is disposed radially outward of the envelope **12,** and is separated at least in part from the centrally disposed electrode by at least a portion of the envelope **12,** thereby defining an area within the envelope **12,** i.e. at least a portion of the chamber **10,** which is disposed between the electrodes **16** and **18.**

A high frequency electrical potential is applied between the electrodes **16** and **18** from a suitable source such as a variable frequency and amplitude, radio frequency (RF) generator **20** to thereby subject the chamber as defined by the envelope **12** between the electrodes **16** and **18** to a selectable time varying electrical field. A suitable fill tube **22** may be provided communicating with the chamber **10** through the envelope **12** and having a valve or other suitable closure means **24** therein to selectively control the nature and pressure of the gas within the envelope **12.**

With continued reference to FIGS. **1** and **2,** the envelope **12** defining the chamber **10** preferably comprises an outer elongated hollow glass cylindrical member **26,** an inner elongated hollow glass cylindrical member **28,** and apertured end plates **30** and **32** sealed therebetween in a suitable conventional manner. The cylindrical member **28** illustrated is substantially coextensive with the member **26** and is disposed in telescoping relationship thereto coaxially within the member **26** to define a chamber annular in cross section as is shown in FIG. **2.**

The central electrode **16** is preferably an elongated metallic cylindrical member, e.g. a wire, telescoped within the central passage **14,** but may be hollow. The outer electrode **18** is preferably a hollow cylindrical electrically conductive member circumferentially disposed round at least a portion of the insulative member **26** and may, for example, be a metallic foil conformed to the radially outer surface of the envelope. The central electrode **16** preferably extends axially into the central passage **14** sufficiently so that an elongated annular portion of the chamber **10** is located between the electrodes **16** and **18.**

The application of a potential between the electrodes **16** and **18** creates an electric field between these electrodes, as is indicated by the lines **34** in FIG. **2.** The electrode configuration, i.e. the relative positions of the electrodes and the relative dimensions thereof, cause the electric field to be more concentrated or dense in the vicinity of the central electrode **16** near the axis of the annular chamber **10.**

If the intensity of the electric field is sufficient, the gas in the chamber **10** will be excited sufficiently to create a gaseous plasma in the chamber. The plasma generally comprises highly reactive species such as ions, electrons and neutral fragmented particles in highly excited states. Since the exciting of the gas by the electric field creates the plasma, the plasma concentration or density generally conforms to the electric field concentration or density. Thus, the concentration or density of the plasma generated within the gas impervious envelope **12** varies between the outer cylindrical member **26** and the inner cylindrical member **28** in a manner related to the electric field concentration of density.

The plasma is thereby concentrated around the inner cylindrical member **28** rather than being dispersed evenly throughout the chamber **10.** This central concentration permits more efficient utilization of the plasma for treating substrates and permits selective exposure of the substrate to the plasma as will hereinafter be described. In addition, this central concentration of the plasma prevents excessive buildup of material on the inner wall of the outer cylindrical member **26.**

The relationship between the gas conditions within the envelope **12** and the gas conditions exteriorly thereof is desirably such that the plasma may be confined to the chamber **10.** The electric potential applied to the electrodes **16** and **18** may thus be lower and the current density will be correspondingly less. This desirable relationship may be obtained by utilizing selected gases at predetermined pressures within the chamber **10,** while exposing the electrodes outside the envelope **12** to the atmosphere.

By way of example, a monatomic inert gas, such as argon or helium at atmospheric or slightly less than atmospheric pressure may be utilized in the chamber **10.** When the RF signal is applied to the electrodes **16** and **18,** a plasma will be more readily generated within the chamber **10** than exteriorly thereof. With the potential of the RF signal applied to the electrodes set at a value corresponding to the potential required to generate a plasma within the chamber **10,** but below the potential required to generate a plasma in the vicinity of the electrodes **16** and **18** externally of the chamber **10,** the current which flows between the electrodes **16** and **18** will not be appreciably affected by the ion flow within the highly electrically conductive plasma since these electrodes are electrically isolated from the plasma. The plasma within the chamber **10** is not contacted by the electrodes **16** and **18** and therefore not contaminated by the electrodes.

Referring now to FIG. **3,** a substrate **36** to be treated within the generated plasma may be introduced into the chamber **10** through a vacuum lock **38** subsequently described in greater detail in connection with FIGS. **6** and **7.** The substrate **36** may be passed through the chamber **10** in contact with the plasma therein at a rate determined by the particular treatment process to which the substrate is being subjected. For example, the substrate **36** may be an organic polymeric fiber, such as a thermoplastic or thermosetting polyester, polyamide, cellulosic or polyolefin material, the surface of which is to be treated in the plasma to obtain a particular surface modification as is described in greater detail in the previously discussed United States patent application Serial No. **93,350,** by Forschirm et al. The substrate **36** may alternatively be a carbonaceous fibrous material to be treated in the plasma within the chamber **10** as is described in greater detail in the previously discussed U.S. Letters Pat. No. 3,745,104, issued July 10, 1973 to , by Kenneth C. Hou. In a further application of the present invention to the treatment of substrates, a coating may be deposited on a suitable substrate by generating a suitable gaseous plasma and contacting the substrate with this plasma. A more detailed description of the substrate and gases utilized in one such coating technique may be had by reference to the previously discussed United States Letters Pat. No. 3,677,799, issued July 18, 1972 to, by Kenneth C. Hou. The above referenced Forschirm and Hou patent applications are hereby incorporated herein by reference.

The substrate **36** may be introduced into the chamber **10** at an a angle with respect to the central electrode **16** as is illustrated in FIG. **3.** The substrate **36** might thereby follow a path generally indicated at **40** which subjects the substrate **36** to varying concentrations of the plasma as it passes through the chamber **10.** Alternatively, as is shown in FIG. **5,** one or more substrates **36** may be passed through the chamber **10** substantially parallel to the electrodes **16** at a selected radial distance therefrom, thereby permitting the exposure of the substrates **36** to a selected plasma concentration.

Referring now to FIGS. **6** and **7** wherein the vacuum lock **38** of FIGS. **3** and **5** is illustrated in greater detail, a hollow tube **40** sealed to the end plate **30** of the envelope **12** communicates interiorly with the chamber **10** and provides a passage through which the substrate **36** may be introduced into the chamber **10.** The substrate **36** may be, for example, a loosely packed fiber bundle through which air leakage ordinarily occurs during the passage thereof between chambers at different pressures.

The tube **40** generally conforms in cross-section to the shape of the substrate, i.e. the bundle of fibers, but is slightly smaller in cross-section than the bundle causing the fibers to be inwardly compressed against each other and against the internal wall of the tube **40.** For example, if the fiber bundle is generally circular in cross-section as in FIG. 7(A), the tube **40** may also be circular in cross-section with a slightly smaller diameter than that of the bundle. Likewise, if the bundle is elliptical in cross-section as in FIG. 7(B), the tube **40** preferably conforms to that shape and is scaled down to slightly smaller dimensions.

One end **42** of the tube **40** is flared or funnel-shaped providing a transition zone for gently compressing the fiber bundle without damage thereto. If desired, the tube **40** may also narrow slightly along the length thereof to further compress the fiber bundle during the introduction thereof as illustrated in the uppermost inlet cube **38** of FIG. **5** into the chamber **10.** It should be noted that when the substrate is a tightly packed fiber bundle or a single filament substrate, the diameter of the tube **40** may be the same or slightly larger than the substrate to prevent damage thereto.

At least two fluid passages **44** and **46** are spaced along the length of the tube **40** and communicate with the interior thereof. Each of the passages **44** and **46** is connected to associated pressure sources **48** and **50,** respectively.

The gas pressure applied to the passage **46** preferably approximates the pressure in the chamber **10,** while the pressure applied through the passage **44** is preferably slightly higher than the pressure in the chamber **10,** thereby creating a pressure differential along the interior of the tube **40.** This pressure differential, together with the mechanical compression of the substrate, prevents gas leakage into the chamber **10** when, for example, the pressure in the chamber **10** is less than the pressure outside the chamber **10.**

With the two passages **44** and **46** illustrated in FIG. **6,** gas leakage into the chamber **10** is minimized since a very slight pressure differential, e.g. **1** mm. Hg., can be maintained between the chamber **10** and the passage **46.** An even smaller pressure differential between these two points may be obtained by increasing the number of lateral fluid passages **44** and **46,** thereby providing even greater gas integrity between the spaces.

SUMMARY OF ADVANTAGES AND SCOPE OF THE INVENTION

It is apparent from the description of the invention that numerous advantages result therefrom. For example, continuous substrates may be treated without ad-

verse effects on the conditions within the reaction chamber since isolation is provided between the interior and exterior of the envelope. For example, the substrate may pass from an area at one pressure, into the envelope which may be at another pressure, and then into an area at yet a different pressure without any substantial gas leakage.

The present invention may thus be embodied in other specific forms without departing from the spirit or essential characteristics thereof. The presently disclosed embodiments are therefore to be considered in all aspects as illustrative and not restrictive, the scope of the invention being indicated by the appended claims rather than by the foregoing description, and all changes which come within the meaning and range of equivalency of the claims are therefore intended to be embraced therein.

What is claimed is:

1. Apparatus for continuously passing a substrate from a first space at a first gas pressure into a second space at a second gas pressure while maintaining the pressure differential between the spaces comprising:

first means defining an elongated passage for providing communication between the spaces, said passage and said substrate generally conforming in cross section throughout the length of said passage;

transition means at the end of said first means in the first space for introducing the substrate into said passage; and,

a plurality of pressure sources in fluid communication with said elongated passage at spaced points along the length thereof.

2. The apparatus of claim **1** wherein the pressure of each of said sources are different.

3. The apparatus of claim **2** wherein the second gas pressure is lower than the first gas pressure and wherein the relative pressures of said sources vary progressively from higher to lower along the length of said passage from the first space to the second space.

4. The apparatus of claim **1** wherein said first means comprises a substantially hollow, elongated tube and said transition means comprises a flared end portion on the end of said tube in the first space.

5. The apparatus of claim **4** wherein the substrate is a fiber bundle and the substrate dimensions slightly exceed the internal dimensions of said tube in cross-section whereby the substrate is mechanically compressed as it passes through said passage.

6. The apparatus of claim **4** wherein the substrate is substantially incompressible without damage thereto and wherein the substrate dimensions are substantially equal to the internal dimensions of said tube in cross-section.

7. Sealing apparatus for passing a substrate from a first space at a first gas pressure into a second space at a second gas pressure while maintaining the pressure differential between the spaces comprising:

a hollow tube of generally circular cross section having a substantially constant internal diameter throughout the length thereof for providing communication between the first and second spaces;

hollow transition means at the upstream end of said hollow tube for introducing the substrate into said hollow tube, said transition means having an internal diameter at the upstream end thereof larger than the internal diameter of said hollow tube and having an internal diameter at the downstream end thereof substantially equal to the internal diameter of said hollow tube; and,

pressure means in fluid communication with the interior of said hollow tube at spaced points along the length thereof, said means being adapted for fluid communication at one of said spaced points with a source of pressure substantially equal to the pressure of the second space.

8. The apparatus of claim **7** wherein said pressure means includes means for establishing a pressure differential between said spaced points along the length of said hollow tube.

9. Sealing apparatus for passing a substrate from a first space at a first gas pressure into a second space at a second gas pressure while maintaining the pressure differential between the spaces comprising:

a hollow tube for providing communication between the first and second spaces, the internal cross section area of said hollow tube being continuously reduced throughout the length thereof from the first space to the second space; and,

pressure means in fluid communication with the interior of said hollow tube at spaced points along the length thereof, said means being adapted for fluid communication at one of said spaced points with a source of pressure substantially equal to the pressure of the second space.

10. The apparatus of claim **9** wherein said pressure means includes means for establishing a pressure differential between said spaced points along the length of said hollow tube.

11. In a method of passing a substrate from a first space at a first gas pressure into a second space at a second gas pressure while maintaining the pressure differential between the spaces wherein the substrate is passed through an elongate hollow tube of substantially constant internal cross section, the improvement comprising the establishment of fluid communication of the interior of the hollow tube with a source of pressure at each of a plurality of spaced points along the length of the hollow tube, the pressure of the source at one of the plurality of spaced points being substantially equal to the pressure of the second space.

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