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FLEXIBLE CONDUIT MEANS FOR CONNECTING AN ELECTRON MICROSCOPE TO A VACUUM PUMP

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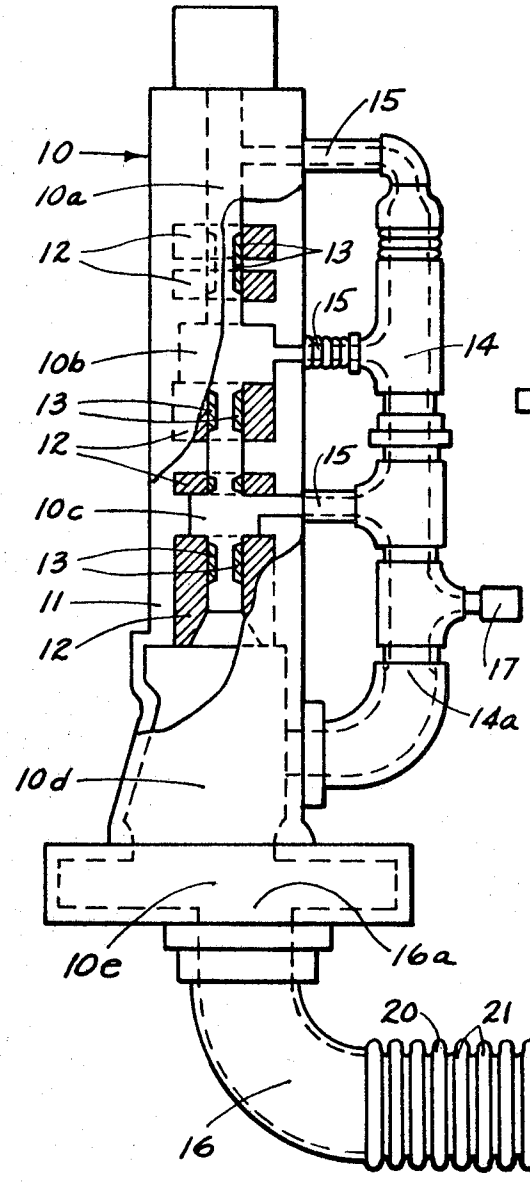


Fig. 2.

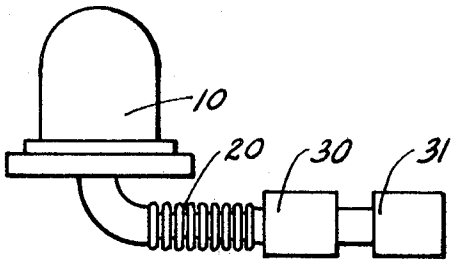


Fig. 1.

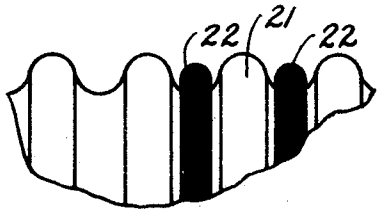


Fig. 3.

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FLEXIBLE CONDUIT MEANS FOR CONNECTING AN ELECTRON MICROSCOPE TO A VACUUM PUMP

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U.S. Cl. 250—49.5 6 Claims

ABSTRACT OF THE DISCLOSURE

A mechanically stable high vacuum system is provided for operationg at 10^{-6} torr, comprising a vacuum chamber connected to a turbine molecular pump by way of a shock attenuating coupling. The coupling is a flexible metal tube or conduit formed with axially aligned circumferential pleats. The spaces between the outside of the pleats are filled with resilient flexible material (e.g., rubber) such that when the vacuum is drawn on the system the pleats are prevented from making metal-to-metal contact. Vibration imparted by pumping is thus attenuated and kept from reaching the vacuum chamber so that the chamber remains mechanically stable. The reduced pressure atmosphere (vacuum) within the chamber, unlike vacuum systems using an oil pump, is oil-free, since the turbine pump contains no working fluid to contaminate the system. In a preferred form of the invention the chamber is an electron microscope column preferably equipped with high capacity pumpout manifold means.

SUMMARY AND DETAILED DESCRIPTION

This invention relates to novel vacuum systems providing extremely high vacuum with relatively short pumpout times. More particularly, the invention relates to a vacuum system providing pressures of 10^{-6} to 10^{-7} torr (1 torr by definition being equivalent to 1 millimeter of mercury) or lower under conditions making for mechanically stable operation.

The prior art vacuum systems provide a high vacuum by combination of a mechanical fore pump and an oil diffusion pump. The mechanical fore pump or rough pump (such as Welch rotary pump, Model No. 1397B, for example) works against a pressure "head" of 760 torr. It operates on the principle of decreasing the volume of gas admitted to its compression chamber thereby increasing the gas pressure sufficiently to actuate its exhaust valve and pass through into the atmosphere.

The oil diffusion pump, on the other hand, can work only against pressure heads well below atmospheric pressure usually beginning at about 10^{-1} torr. It operates by trapping the gas which diffuses into it. However, a certain amount of back diffusion or back streaming of oil vapors undesirably is produced from the diffusion pump into the area being evacuated. This back diffusion of oil vapor has been a major source of hydrocarbon contamination. Back streaming can be prevented to a degree wih suitable trap materials, i.e., zeolites or large liquid nitrogen-cooled metal surfaces, none of which are practical in the present application.

The main advantages of oil diffusion pumps are that they do not contain mechanically moving parts (thereby being relatively free of vibration) and are inexpensive to operate. Other high vacuum pumps have certain advantages such as high pumpout rate. In general, however, these other pumps are costly, cause excessive vibration or have other disadvantages making them unsuitable, such as operation at liquid nitrogen temperatures unsuitable in general laboratory practice, as well as operation with high

magnetic fields which are intolerable in association with sensitive instruments such as electron microscopes.

The difficulty with the prior art systems also is that the time required for pumping out is unduly only and more importantly for special applications such as electron microscopes, the accompanying oil vapor contamination of operating components in the vacuum system is excessive. Equipment becomes contaminated to the point where the components need to be dismantled and cleaned frequently. Also, contamination causes short filament life due to corrosive effects of hydrocarbon deposits on the tungsten of the filament. The downtime involved in repairs is often considerable since typically an instrument can be out of service for days at a time.

Concerning the construction of high vacuum systems, the electron microscope is an example of extreme measures taken to assure in a practical sense the utmost in low pressure attained rapidly. The enclosed envelope which comprises the microscope column is constructed of a number of carefully tested vacuum-tight sections. These sections are joined together with synthetic rubber gaskets of a special type having low vapor pressure characteristics. The seals for the joints are of two types: for a static seal using rubber gaskets, i.e., for joints where there is no relative motion of the parts no lubrication is used with the gaskets; for a dynamic seal, i.e., for joints where two sections can be moved with respect to one another, a vacuum grease is used as a lubricant. Every effort is made in providing these seals to prevent out-gassing or deposition of grease vapor or other organic contamination. Despite these efforts the prior art systems have required frequent servicing and dismantling, as indicated.

It is therefore an object of the present invention to provide vacuum systems operating under high vacuum of the order of 10^{-6} torr or higher.

It is another object of the invention to provide vacuum systems which are serviceable over long periods and require little maintenance.

It is also an object of the invention to provide vacuum systems in which the vacuum chamber is mechanically stable and free of vibration.

Still another object of the invention is to provide vacuum systems which are comparatively free of organic contamination and the like.

Yet other objects of the invention are to provide means for improving resolution and definition of pictures in electron microscopes in respect to reduction in exposure time and increase in filament life.

Other advantages, objects and features of the invention will be seen in reference to the following specification and the accompanying drawing in which:

FIG. 1 is a schematic representation of a simple vacuum system according to the invention;

FIG. 2 is a diagrammatic representation partly cut away to expose a section taken on the central axis, of an electron microscope column connected to the pump assembly through flexible conduit means; and

FIG. 3 is an enlarged view of a portion of the conduit of the type shown in FIG. 2.

Referring in greater detail to the drawing, the vacuum system of FIG. 1 includes an airtight chamber **10** connected by way of an opening in its base to the flexible conduit **20** which in turn is connected to the fine pump **30** and rough pump **31**. The conduit **20** is a flexible metal tubing or bellows of suitable thickness having a plurality of circumferential pleats **21**.

Between the pleats **21** are provided shock attenuating spacing means **22**. The spacing means can be used in any of various forms such as cords, O-rings, molded ensleeving jackets with digital spacing pieces, films, tapes, especially films and tapes having a pressure-sensitive adhesive

surface for attachment as by winding, and other equivalent forms. O-rings and especially rubber O-rings are preferred; sizes which approximate the circumference of the bellows groove advantageously are used. For installation onto the conduit the O-rings are stretched open slightly, guided one by one lengthwise along the conduit and released into the several open bellows grooves, so that each groove receives a single O-ring whereby the normal tension of the O-ring controllably retains the ring in position for cooperation with the bellows and the other assembled O-rings.

For spacing, any substance can be used which prevents the pleats from contacting each other in an axial direction and is resilient and shock absorbent. For purposes of illustration, some examples of suitable resilient substances are natural rubber, synthetic rubber (e.g., butyl rubber, neoprene, GR–S, GR–I, especially Buna®), plastic elastomer (e.g., polyurethane elastomer) and the like. The substance should be sufficiently resilient and shock absorbent such that compression of the conduit (e.g., from the position shown in FIG. 3 to the position shown in FIGS. 1 and 2) results in controlled, self-limiting spacing of the pleats from one another whereby vibration at the pump side of the conduit means is dampened out. In operation, the dampening effect can readily be appreciated simply by touching the opposite ends of the conduit: the pump end vibrates noticeably to the touch whereas the vacuum chamber end is essentially static.

The invention is useful for various types of vacuum systems and is particularly applicable to precision instruments of the type illustrated by the electron microscope. For purposes of illustration, the specification which follows will therefore describe the invention with particular reference to its application in electron microscopes, although it will be realied that the invention is applicable broadly to vacuum systems and is not limited to the field of electron microscopy.

Referring to the preferred embodiment of the invention illustrated in FIG. 2, the electron microscope chamber **10** includes the bore **10***a* which intersects the specimen chamber **10***b*, the intermediate lens chamber **10***c*, viewing chamber **10***d* and the photo chamber **10***c*. The microscope column **11** includes several lenses or magnets **12** (for condensation and focussing of the electron beam) as well as associated aperturing or pole pieces **13**. The term "bore" as used in this connection refers to the overall opening extending along the center axis of the column **11** which opening for purposes of definition is considered to be substantially uniform and defined by the average of the inner dimensions of the magnets (exclusive of the pole pieces **13**). The column is provided with a manifold **14** which communicates directly with open areas in the vacuum chamber by means of the pumpout ports **15**. Each pumpout port is adapted for evacuating a particular open area in relation to the adjacent aperturing associated with the bore. A vacuum gauge **17** is located on the pumpout manifold. At the base of the column the chamber **10** communicates through elbow **16** to the fine pump **30** by way of conduit means **20**. The fine pump **30**, according to the invention, is a turbine molecular pump of a conventional type. One such suitable pump is the turbo molecular pump (Model 3103–C) supplied by the Welch Scientific Co., Skokie, Ill., which operates at a pumping speed of about 260 liters per second over pressures ranging from 10^{-2} to 10^{-8} torr; ultimate blank-off pressure is rated at approximately 10^{-9} torr. Other equivalent turbine molecular pumps would be satisfactory for purposes of the invention.

Since molecular pumps of the type described operate at speeds in excess of 15,000 revolutions per minute and since they uniformly cause vibration both mechanical and harmonic, their use with precision equipment requiring absolute mechanical stability would have seemed impractical. For high resolution work of the type required in electron microscopy, the specimen for example should not move at the rate of more than a few hundredths of an angstrom unit per second. In particular, movement of the specimen with respect to the photographic plate should be avoided entirely. Nothwithstanding this, random external vibration often is unavoidably encountered causing a distortion of the magnitude of several angstroms. Such undesirable distortion is one of the major practical limitations on resolution. Consequently, it was heretofore considered important in electron microscopy to keep every source of vibration away from the microscope column. Conventional precautions which have been taken for this purpose have been to keep the mechanical rotary pump at a distance remote from the microscope and to switch off the pumps during exposure.

With the present invention, on the other hand, no such precautions need be taken. The pumps are allowed to operate continuously. More surprisingly, there is remarkable improvement in the resolution, or definition, of pictures taken. Back-diffusion or back streaming of oil or oil vapor has been eliminated. The operating vacuum of the system is improved by at least one decade from 2×10^{-5} to 10^{-6} torr or more. Moreover, there is a substantial increase in filament life, for example, for service instruments in constant use from a maximum of 100 hours to as long as 300 to 400 hours.

The vacuum chamber can according to the invention be located directly alongside the turbine molecular pump, as illustrated. The mountings for the chamber and the pump are preferably in solid vibration-free concrete or other suitable foundation. The relative location of the chamber and the pump can be selected to suit individual needs but in general sufficient space should be provided to permit required equipment servicing. The distance from the chamber to the pump **30** should, however, be as short as practical. In this connection it is one of the several advantages of the invention that the space occupied by the conduit **20** is minimal. Short lengths, e.g., 10–12 inches, are adequate.

In assembling the various components, the conduit is mounted in the compact or compressed form shown in FIG. 2. The conduit is a conventional item supplied commercially in various forms. It is constructed of metal, preferably stainless steel, bronze or brass. Stainless steel is preferred. The joints to the elbow **16** and pump **30** may be made by any suitable means such as welding with silver solder or other conventional means for obtaining vacuum-tight seals. One type of conduit suitable for the invention is the metal bellows known under the trademark "Sylphon" supplied by Detroit Flexible Metal Products Company, Detroit, Mich. Another such bellows is supplied by High Voltage Engineering Corp. of Burlington, Mass.

According to a further preferred embodiment of the invention as shown in FIG. 2 (in which the column typifies one commercially available unit supplied by the Radio Corporation of America, New York, Model EMU–3g), the vacuum chamber is provided with an external manifold **14** which connects through the pumpout ports **15** with open areas (that is, specimen chamber **10***b*, intermediate lens chamber **10***c* and viewing chamber **10***d*) in the bore **10***a* of the chamber **11**. According to this embodiment and in order to achieve optimum pumpout rates practical for general use, the cross-sectional area or lateral opening in the pumpout ports **15** is at least seventy-five times as great as that of the aperturing of the bore. Further, the cross-sectional area of the entrance opening **16***a* from elbow **16** to the photo chamber **10***e* is at least four times the area of the exit opening **14***a* of the manifold **14** into the back of the viewing chamber **10***d*; also the cross-sectional area of the manifold is at least four times the cross-sectional area of the pumpout ports **15**. Thus, to take an illustrative case, the openings of the aperturing and pole pieces expressed as inside diameter are from 0.0012 to 0.01 inch; of the

pumpout ports, about ¾ to 1 inch; of the manifold and exit opening, about 2 inches; and of the elbow entrance opening (and in fact the elbow itself and the conduit means) about 4 inches. With this preferred embodiment pumpout to 10^{-6} torr is accomplished in short periods, for example, from about 2 to 6 minutes, whereas with prior art instruments pumpout to a minimum of 10^{-4} to 10^{-5} torr generally requires from 1 to 2 hours or longer. The time required for vacuum pumpout varies depending on the degree of vacuum required but ordinarily pumpout to vacuum as high as 10^{-5} to 10^{-6} torr is accomplished in about 1.5 to 3.0 minutes. In contrast to this, prior art systems for routine use have seldom operated at vacuum as high as 10^{-6} torr since the pumpout time for achieving such vacuum has been excessive and has been prevented by back streaming of oil vapor equilibrating with and limiting the pumping rate of the oil diffusion pump.

The invention also contemplates a preferred construction wherein the manifold communicates directly with the conduit means and turbine molecular pump so that pumpout by way of the viewing chamber and photo chamber is not required. In this case, the cross-sectional area of the conduit means should be at least four times that of the manifold, the inside diameter in this case being, for example, 4 inches and 2 inches, respectively.

While the invention in vacuum systems have been described in considerable detail in the foregoing specification, it will be realized by those skilled in the art that wide variation can be made in such detail without departing from the spirit of the invention.

I claim:

1. In combination, an electron microscope column including a vacuum chamber; turbine molecular pump means; and flexible metal conduit means adapted to communicate between the vacuum chamber and the pump means for drawing a vacuum on the chamber, the conduit means constituting a plurality of axially aligned circumferential pleats and having external resilient shock attenuating spacing means for the pleats whereby vibration associated with pumping is absorbed before reaching the chamber.

2. A vacuum system according to claim 1 wherein the bore of the column includes aperturing and pole pieces intermediate enlarged zones located at spaced sections along the bore, and the column comprises an external manifold having pumpout ports adapted for pumpout of at least some of the zones, the cross-sectional area of the pumpout ports being at least seventy-five times that of the aperturing of the bore thereby providing for optimum pumpout of the column.

3. A vacuum system according to claim 1 wherein the column comprises an external manifold having ports adapted for pumpout of the column, the cross-sectional area of the manifold being at least four times that of the ports, thereby providing for optimum pumpout.

4. A vacuum system according to claim 1 wherein the chamber is included in an electron microscope column having an external manifold communicating with the chamber, the cross-sectional area of the conduit means being at least four times that of the manifold thereby providing for optimum pumpout of the chamber.

5. The combination of claim 1 wherein the conduit means includes rubber spacing means.

6. The combination of claim 1 wherein the means for spacing adjacent pleats comprises O-ring means.

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U.S. Cl. X.R.

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