

[54] **ATMOSPHERIC SAMPLING PROBE FOR A MASS SPECTROMETER**
[75] Inventor: **Robert W. Thomas, Rome, N.Y.**
[73] Assignee: **The United States of America as represented by the Secretary of the Air Force, Washington, D.C.**
[22] Filed: **Apr. 11, 1973**
[21] Appl. No.: **350,259**

Primary Examiner—James W. Lawrence
Assistant Examiner—D. C. Nelms
Attorney, Agent, or Firm—Harry A. Herbert, Jr.;
Henry S. Miller, Jr.

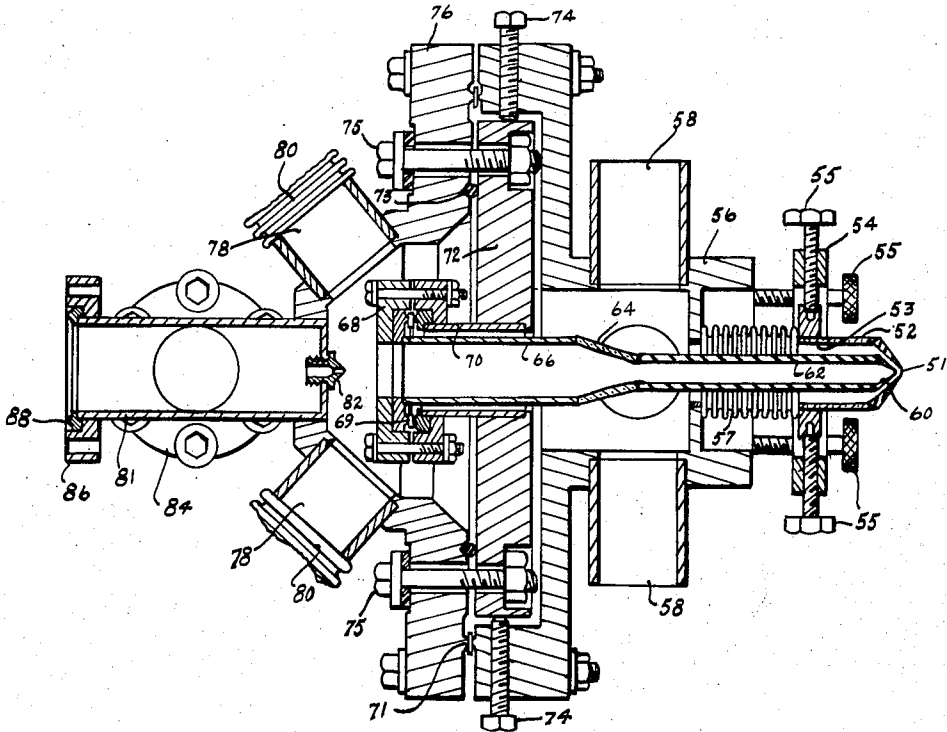
[52] U.S. Cl. **250/288, 250/289**
[51] Int. Cl. **B01d 59/44**
[58] Field of Search **250/283, 288, 289, 428, 250/430, 489**

[56] **References Cited**
UNITED STATES PATENTS

2,721,270	10/1955	Bennett.....	250/289
2,769,912	11/1956	Lupfer	250/289
2,880,323	3/1959	Reinecke	250/289
3,227,872	1/1966	Nemeth	250/289

[57] **ABSTRACT**
An atmospheric sampling probe capable of generating a molecular beam for mass spectrometer analyzation including a quartz gas inlet orifice adjustable in any plane with a first vacuum stage; a quartz skimmer orifice adjustable in one plane having a second vacuum stage and a collimating orifice and ionizing chamber adapted to be connected to a quadrupole mass spectrometer with a third vacuum stage.

5 Claims, 2 Drawing Figures



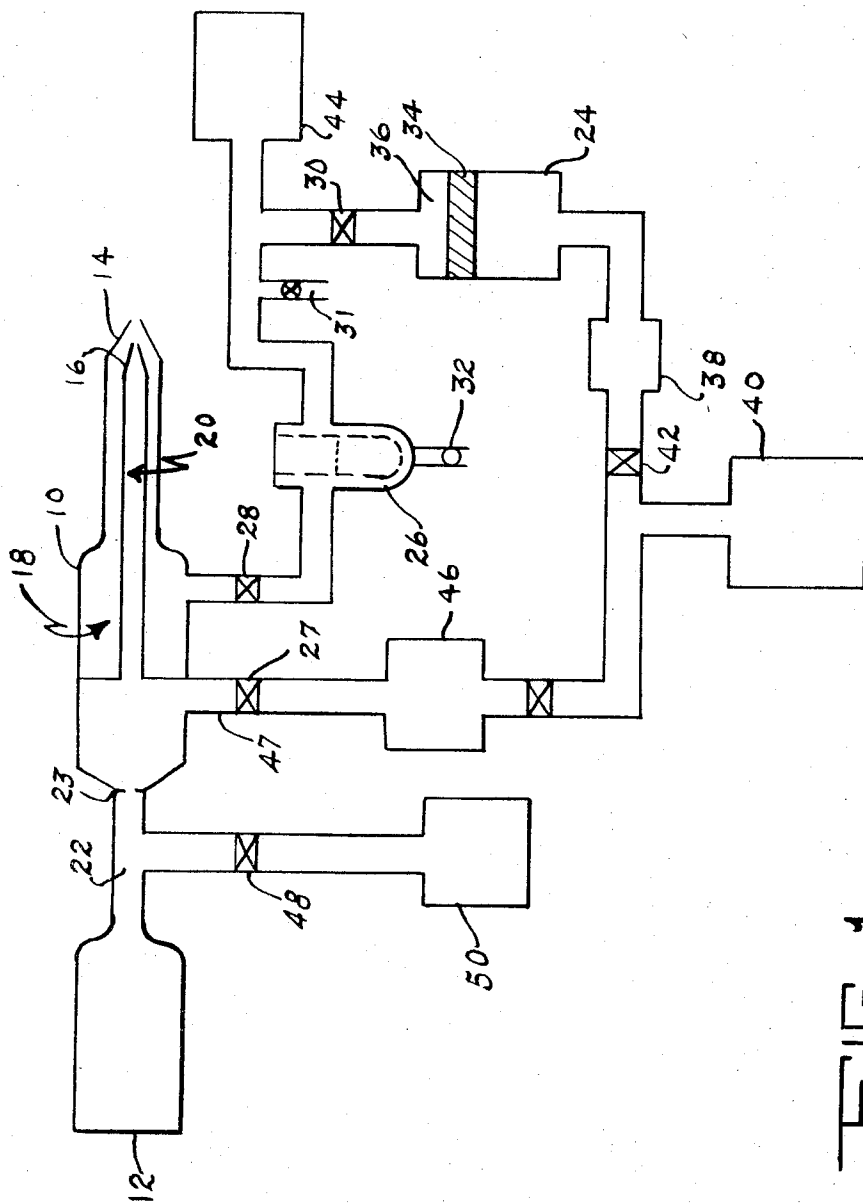
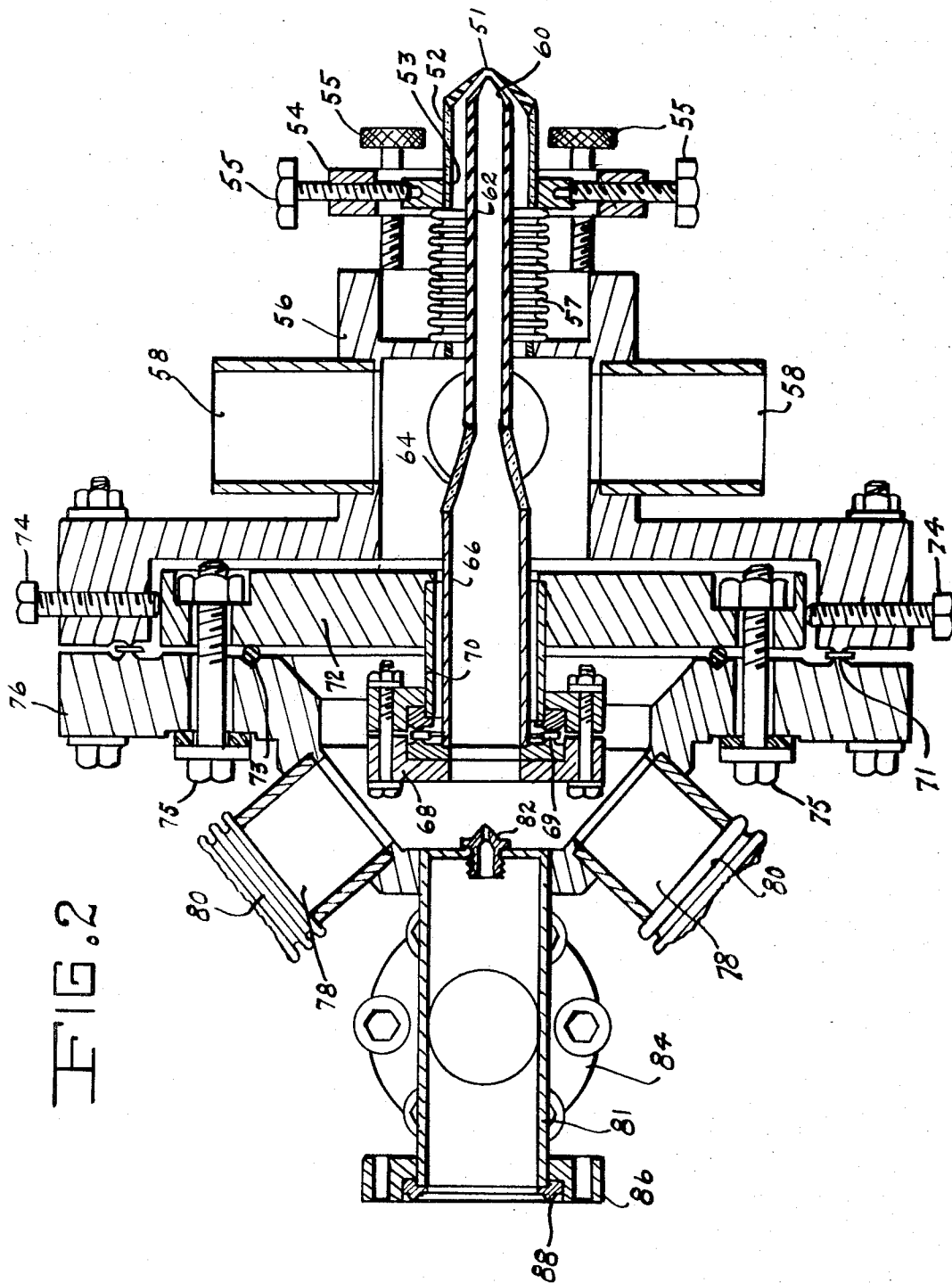


FIG. 1



ATMOSPHERIC SAMPLING PROBE FOR A MASS SPECTROMETER

BACKGROUND OF THE INVENTION

This invention relates generally to gas sampling probes and in particular to a probe adapted to sample gases at atmospheric pressure by means of a molecular beam generator combined with a mass spectrometer.

The evolution of solid state electronics has created formidable problems relating to the manufacture of solid state devices. Cost necessitates that there be a minimum of waste, reliability and accuracy require more stringent controls in the assembly process, supply and demand call for more speed in the manufacturing process. Generally, in the production of an article these requirements become ultimately mutually exclusive, in that an increase in one requirement necessarily produces a decrease in an other.

In the field of solid state electronics manufacture, it has been found that the analytical tools available lack sufficient sensitivity to maintain direct and absolute control over a semiconductor processing environment, such as the epitaxial growth process. It is readily apparent that the only satisfactory technique to substitute for the presently used cut and try procedure, is a real-time monitoring of the epitaxial reaction, which would drastically reduce the time required to achieve optimum epitaxial growth.

The same real time monitoring system could serve equally as well as a means for studying the mechanisms of chemical vapor decomposition; for sampling localized laser evaporations; for measuring exhaled human breath as a disease diagnostic tool and for quantitatively measuring condensible air pollutants.

SUMMARY OF THE INVENTION

In order to solve the problems of inefficiencies in the prior art, and to provide a new and improved method for sampling gases at atmospheric pressure, this invention offers a sampling probe which, when coupled with a mass spectrometer allows a chemically reactive or condensible species of gas to be transported from atmospheric pressure to 10^{-8} torr without collision with other molecules or walls of the probe. The unique design of this system allows adjustment of a first orifice in any plane and a second or skimmer orifice in one plane while the system is operating under vacuum. Another unique feature is the utilization of materials such as stainless steel and quartz which will allow a 400°C bakeout of the unit while operating.

The system consists of two quartz microorifices, the first orifice is used to generate a pure molecular beam by free expansion of the sample at atmospheric pressure into a vacuum of 10^{-5} torr. The beam of particles generated by the orifice is skimmed by the second orifice to form a pure beam of the sampled gas. The skimmer effectively removes gas molecules which have reacted with the walls of the first orifice to form unwanted secondary combinations of atoms. The beam, after entering the second orifice, is traveling at Mach. 5. At a vacuum of 10^{-5} torr the molecules in the beam have a mean free path of sufficient length to be detected by the quadrupole mass filter before suffering a collision with another molecule. The design therefore, insures the purity of the sampling beam. The beam en-

ters the third collimating orifice before it is detected by the mass filter and electron multiplier.

The system was designed completely from stainless steel and quartz to allow bakeout at 400°C. This provides an effective control over contamination which might arise from the probe assembly itself. A particularly difficult problem arose where both orifices must be adjustable for beam alignment. The successful fabrication of the microorifices and the design of the bakeable stainless probe assembly are the major innovative achievements in the design of the probe system.

It is therefore an object of the invention to provide a new and improved sampling probe that is more accurate than those of the prior art.

It is a further object of the invention to provide a new and improved sampling probe that is capable of providing a molecular beam input to a mass spectrometer.

It is still another object of the invention to provide a new and improved sampling probe that allows adjustment of the orifices while the system is in operation.

It is still a further object of the invention to provide a new and improved sampling probe that will allow unaffected operation of the unit at 400°C.

It is another object of the invention to provide a new and improved sampling probe that is simple in design and more reliable than those known in the art.

It is another object of the invention to provide a new and improved sampling probe which is economical to produce.

These and other advantages, features and objects of the invention will become more apparent from the following description taken in connection with the illustrated embodiment in the accompanying drawing.

DESCRIPTION OF THE DRAWING

FIG. 1 is a schematic representation of a system utilizing the invention.

FIG. 2 is a crosssectional view of the invention.

DESCRIPTION OF THE PREFERRED EMBODIMENT

Referring now to FIG. 1, a typical application of the invention is shown with an appropriate vacuum system for atmospheric sampling. The probe is shown at 10 in combination with a quadrupole mass spectrometer 12. The probe is shown with a gas inlet orifice 14 and a skimmer orifice 16. A vacuum differential is maintained between the first stage chamber generally indicated as 18 (10^{-3} torr) and the second stage chamber generally shown as 20 (10^{-5} torr). The probe is connected to the mass spectrometer through an adapter 22 which contains a collimating orifice 23 and serves as an ionizing chamber for molecules entering through the probe. The mass spectrometer is a third stage vacuum chamber and is maintained at 10^{-8} torr.

The pumping system for the first stage utilizes a six inch diffusion pump 24 to achieve 10^{-3} torr. Due to the large throughput of the first stage, a high capacity pump is required to protect the system from such extraneous matter, as for example readily condensible material. Flow from the first or inlet orifice is fed into two open liquid nitrogen dewars 26 (only one of which is shown) where condensation built-up can be then observed during system operation, as well as the level of the liquid nitrogen. After operation, the dewars can be isolated by closing the gate valve 30 and the butterfly valves 28. A stopcock 32 is provided for purging the

dewars with dry nitrogen via inlet valve 31 into a fume exhaust system. Hence, a large percentage of the condensibles are not allowed to enter the diffusion pump 24. Similarly, a water baffle 34 and liquid nitrogen trap 36 at the diffusion pump decreases the amount of hydrocarbons backstreaming into the first stage of the probe. A bakeable molecular sieve foreline trap 38 is used to isolate mechanical pump oil from the diffusion pump and protect the mechanical pump 40 from condensation contamination. In the event of a power loss, a solenoid valve 42 isolates the diffusion pump from the mechanical pump. A glass bell jar 44 is utilized as ballast to prevent overload of the diffusion pump when the orifice is first open.

The second stage requires a higher degree of cleanliness as well as a moderately high pumping speed. A turbomolecular pump 46 connected through valve 41, performs this function and has negligible memory effect and prevents the back-streaming of the mechanical pump oil.

The second stage is connected to the turbo pump by means of stainless steel tubing butterfly valves 27, and bellows 47 to insure low outgassing rates into a molecular flow region. Under load, the turbomolecular pump has the capacity to maintain a pressure of 10^{-5} torr, thus insuring a mean free path of sufficient length to allow molecules in the beam to travel from the skimmer to the third collimating orifice 23 without collision. This is necessary to maintain beam integrity from skimmer to detector.

The collimating orifice 23 immediately in front of the quadrupole 12 separates the second and third stage vacuum systems. The third vacuum stage maintains an ultra-clean vacuum in the detector by means of a high capacity ion pump 50 valved at 48. During operation of the beam, this pump maintains an ultra-high vacuum of 10^{-8} torr. This section of the system is maintained at 100°C during probe operation to prevent buildup contamination of the walls of the quadrupole mass spectrometer.

Concerning FIG. 2, there is shown an enlarged cross-sectional view of the sampling probe. The gas inlet orifice is shown at 51 and is formed of quartz material which is bonded to pyrex glass 52 and the stainless steel 53. The stainless steel portion of the orifice structure by friction engages the first adjustment plate 54 in a friction fit. The adjustment screws 55 move both the plate and orifice structure in any plane to insure proper alignment of the various apertures. The first orifice structure is welded to the frame 56 by a metal bellows 57. Pumping ports 58 allow a vacuum to be created in that region of the apparatus.

A skimmer orifice 60 is positioned internally of and adjacent to the gas inlet orifice 51. The skimmer in a manner similar to the gas inlet orifice is formed of fused quartz 62; pyrex glass 64 and a stainless steel base 66. The base is mounted in the flange portion 68 which is in turn mounted on an extender 70 protruding from the second adjustment plate 72. The adjustment screws 74 relative to the frame members 56 provide orthogonal movement of the skimmer orifice. Second orifice adjustment plate 72 is sealed by gold O-Ring 73 to frame member 76 by head sealing screws 75. O-Ring 73 and gasket 69 isolate chamber 1 and chamber 2. Copper

gasket 71 seals chamber 1 from atmosphere.

Pumping parts 78 connect through bellows 80 to a vacuum pumping system to allow control of the pressure in the skimmer chamber.

5 An adapter 80 is connected to the frame 76 and provided with a collimating aperture 82. A vacuum pumping port is provided at 84. The adapter is connected to a quadrupole mass spectrometer at the flange 86 and sealed at 88.

10 In operation, referring to FIG. 1, gas at atmospheric pressure enters the orifice 14 where it is expanded and a quantity of gas is removed from the system via the first stage vacuum system. The remaining gas passes through the skimmer orifice 16 where high velocity molecules travel along the axis of the chamber and enter the collimating orifice 23 after which the molecules are ionized and analyzed in the quadrupole mass spectrometer 12. The orifices are aligned by monitoring the intensity of a nitrogen molecular beam and adjusting the orifices until maximum intensity is reached as indicated by the mass spectrometer.

Having thus described my invention, I submit the following claims thereon:

1. A system for continuously sampling gases at atmospheric pressure comprising: a sample probe means, including a first microorifice mounted on a frame and movable in any plane, a second microorifice positioned in line with said first microorifice, adjacent thereto and movable in one plane and a third orifice positioned in line with said first and second microorifices and spaced therefrom; a first stage vacuum system for creating a vacuum; a first vacuum chamber, connected between said first microorifice and said first stage vacuum system; a second stage vacuum system for creating a vacuum of greater magnitude than the first stage vacuum system; a second vacuum chamber positioned within said first vacuum chamber connecting the second stage vacuum system and said second microorifice; a third stage vacuum system for creating a vacuum of greater magnitude than said second stage vacuum system; a third vacuum chamber, connected between said third stage vacuum system and said third orifice, in juxtaposition to said second vacuum chamber and a mass spectrometer detector means connected to the said third vacuum chamber whereby gases entering the sampling probe means pass therethrough forming an uncontaminated molecular beam for detection in the mass spectrometer.

2. A system for sampling gases according to claim 1 wherein said first and second orifices are formed of quartz.

3. A system for sampling gases according to claim 2 wherein said first stage vacuum system includes a pump, means for removing condensibles from said stage, and ballast means between the pump and condensibles removing means to prevent overload to the pump when the probe means is open to atmospheric pressure.

4. A system for sampling gases according to claim 3 wherein said second stage vacuum system includes a turbomolecular pump.

5. A system for sampling gases according to claim 4 wherein said third stage include an ion pump.

* * * * *