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(54) Title: CAPACITIVE-IMPEDANCE TYPE HUMIDITY/MOISTURE SENSING APPARATUS AND METHOD

(57) Abstract: The moisture content of a gas is sensed by subjecting a moisture-sensing capacitor to the gas and then measuring the impedance of the capacitor. The moisture-sensing capacitor comprises a pair of closely spaced, porous, and parallel metal electrodes having a thin coating of a water-absorbing dielectric, such as zeolite therebetween. In a two-sensor system, the impedance of a dry reference capacitor is differentially compared to the impedance of the moisture-sensing capacitor in order to eliminate any effects that variations in gas temperature and/or gas pressure may have on the measurement of the moisture content of the gas.

CAPACITIVE-IMPEDANCE TYPE HUMIDITY/MOISTURE SENSING APPARATUS AND METHOD

BACKGROUND OF THE INVENTION

Field of the Invention

This invention relates to the measurement of the humidity content, moisture content, or water content that is within a fluid stream (for example, a gas stream) that is used in various manufacturing processes of which a semiconductor manufacturing process is a non-limiting example. More specifically, this invention relates to electrical sensors whose impedance value (i.e., an impedance value having only a resistive term, an impedance value having only a capacitive reactance term, or an impedance value having both a resistive term and a capacitive reactance term) changes as a function of the moisture that is within a fluid mass.

Sensors in accordance with this invention are structurally configured as capacitors that include two electrodes having a moisture-adsorbing dielectric therebetween. Thus, the impedance value of the sensor can be measured and may include the measurement of a significant resistive component value. For example, when measuring the moisture content of ammonia gas, the resistive component of the impedance of sensors in accordance with the invention shows a greater variation as a function of gas moisture content than does the capacitive reactance component of the impedance value. That is, while a utility of the present invention may relate primarily to measurement of the sensor capacitive reactance component, utility is also provided when the sensor resistive component is measured, and utility is also provided when a measured sensor output comprising a complex number impedance. All of these measurement modes are defined as a measurement of the sensor impedance as a function of the moisture content of the environment that is being sampled by the sensor.

Description of the Related Art

Moisture sensors are known in the art. For example, U.S. Patent 4,876,890 provides a pressed zeolite powder compact (zeolite sodium LZ-210) to which gold electrodes, and preferably porous electrodes, are attached by screen printing or sputtering. Wire leads attach the electrodes to an impedance analyzer so that an AC current passes between the electrodes and through at least a portion of the zeolite powder compact. When the zeolite powder compact is placed in a gaseous environment whose humidity is to be determined, and after about 20 minutes, the impedance analyzer passes a continuous AC current thereby measuring the impedance of the zeolite powder compact at the humidity condition that is present in the gaseous environment. This impedance measurement is related to the concentration or partial pressure of water in the gaseous environment. This patent is incorporated herein by reference.

U.S. Patent 3,315,518 provides a pair of stainless steel gauze disks that are stiffened by a pair of nickel-chromium screen disks, wherein the space between the nickel-chromium screen disks is packed with polytetrafluoroethylene granules that are coated with a layer (2500 angstroms) of liquid polyethylene glycol. Gas to be tested for humidity passes through the disk/granule assembly, and a capacitance meter is connected across the stainless steel gauze disks.

U.S. Patent 2,976,728 provides an in-line conductance-type moisture sensor having porous electrode disks between which a hygroscopic material (phosphorous pentoxide) is placed.

U.S. Patent 3,186,225 provides a moisture sensor having a compressed zeolite disk having a metal foil electrode on one side thereof, and a metal gauze electrode on the other side.

The art also provides moisture sensors that include heaters of which U.S. Patents 5,296,819, 5,485,747 and 5,814,726 are examples.

The apparatus/method of the present invention makes use of the water-adsorbing properties of a material that is known as zeolite; i.e., an aluminosilicate of the general chemical composition $M_{2/z}O:Al_2O_3:nSiO_2:xH_2O$, where M is a metal ion, normally Na^+, and z is the valence of the metal ion. It is know that artificial zeolites are made in a variety of forms, and are used as gas adsorbers, drying agents, and catalysts, as well as water softeners.

Zeolite is a molecular sieve (i.e., a group of adsorptive desiccants) which are crystalline aluminiosilicate materials that are chemically similar to clays and feldspars, and belong to the class of materials known as zeolites. A characteristic of these materials is their ability to undergo dehydration with little or no change in crystal structure. The dehydrated crystals are interlaced with regularly-spaced channels of molecular dimensions, which channels comprise almost 50-percent of the total volume of the crystals. The empty cavities that are within activated molecular sieve crystals have a strong tendency to recapture water molecules that have been driven off. Only molecules that are small enough to pass through the pores of the crystal can enter these cavities and be adsorbed on the interior surface thereof, this action being a sieving or screening action. Thus, it is known that the crystals can be used to dry gases and liquids.

While prior art such as above exemplified is of general utility of the purposes stated therein, a need remains in the art for an improved, inexpensive, and highly sensitive in-line or pass-through capacitance-type humidity/moisture sensing apparatus and method.

SUMMARY OF THE INVENTION

This invention provides a capacitance-type humidity/moisture sensor having a variety of uses. A non-limiting example of such a use is to sample the gas output of a gas purifier in order to provide an indication of when the gas purifier is approaching the end of its useful life. Another non-limiting example is to embed a sensor(s) in accordance with the invention within the gas purifier.

A sensor in accordance with this invention includes two generally flat and porous metal plates (for example, two porous stainless steel plates in the shape of generally identical flat disks) each disk being of generally the same uniform diameter and thickness, and each disk having a central axis that extends generally parallel to the plane of the disk.

One surface of each disk is thin coated (by the use of a conventional spray-painting process or a conventional dip-coating process) with zeolite or with a material having water-adsorbing properties.

The two zeolite-coated disk surfaces are then mounted in a coaxial manner (for example, so as to close a gas flow path), and with their two zeolite coatings in intimate physical engagement. The two metal disks are relatively closely spaced due to the thin nature of the zeolite coating that is on at least one of the two disks; thus, advantageously providing a two plate capacitor having a low signal-to-noise ratio, and a relatively high impedance value that is highly sensitive to changes in the humidity level or moisture content of the zeolite coating(s).

In another non-limiting and slower response configuration, the above- described two coaxially-mounted disks only partially obstruct a gas flow path, and in yet another configuration, the disks are mounted so that the plane of the disks are parallel to, or at an angle to, the gas flow path without necessarily closing or obstructing the gas flow path. In these arrangements, the gas to be sensed reaches the sensor through a combination of flow through and diffusion.

An electronic impedance measuring device (for example, an inductance/capacitance/resistance (LCR) meter) is electrically connected to the uncoated surfaces of the two metal disks. When water molecules that are present in the gas flow path adsorb onto or into the zeolite, the impedance value (i.e., a complex number whose real component is resistance and whose imaginary component is capacitive reactance) of the two-disk capacitor increases. This is due to the fact that water has a much higher dielectric constant than do the zeolite coatings.

When a sensor in accordance with this invention is used to monitor the water content of a gas stream at a location that is down stream of a gas purifier, a target or reference impedance value provides a signal of the fact that the gas purifier is spent and should be replaced. In an embodiment of the invention, the reference impedance value is provided by a dry reference sensor that is subjected to the same temperature and/or pressure conditions as the active sensor, and this embodiment of the invention may include placing both the active sensor and the dry reference sensor within the gas purifier.

An important feature of the present invention is that the above-described zeolite layer(s), coat(s) or coating(s) is a relatively thin layer(s) that is “coated” onto at least one porous metal electrode.

As used herein, the term “coat” or “coating” means a relatively thin layer that is produced by spraying, spreading, dipping, or painting a zeolite-containing liquid (i.e., liquid that contains zeolite that has been ground into a finely divided powder) onto a support surface, the liquid containing at least zeolite and water, and perhaps an inert and nonvolatile binder material. After a period of drying, the above-described zeolite-coated disks result. AS 30 collodial silica, the brand Ludox Aldrich 30 wt%, is an example of binder having utility in producing such a “coat” or “coating”.

As such, the term “coat(s) of zeolite” or “coating(s) of zeolite” is critically different than a thin film of zeolite, i.e., a thin layer of zeolite that is one atom or one molecule thick. In addition; the term “coat(s) of zeolite” or “coating(s) of zeolite” is critically different than a bulk mass of zeolite or a bulk compact of zeolite wherein the zeolite particles are usually in the range of 0.5 to 2 mm in diameter, or perhaps smaller. A thin film is dictionary-defined as a coating that is deposited in a layer one atom or one molecule thick.

An alternate to the two above-described metal disks in accordance with the invention is two porous and electrical insulator plates or disks; for example, sintered glass or a flexible material such as woven fiberglass or zirconia cloth. In this embodiment of the invention, each insulator substrate is first coated on one side with a porous thin film or thin electrode of an electrically conductive metal that is inert to the fluid flow being tested, for example the deposition of gold, nickel, copper or carbon by vacuum deposition, and this thin metal film is then coated with a thin coating of zeolite as above described. The above-described LCR meter is then connected to the two thin films of metal.

In this flexible woven sintered glass/fiberglass/zirconia cloth embodiment of the invention, the assembly can be rolled into a physically compact cylinder shape having two protruding wires that are connected to the LCR meter.

As a feature of the invention, an in-situ heating means may be provided to reactivate the zeolite layer by removing or driving off adsorbed water molecules therefrom.

As another feature of the invention, only one thin-coated layer of zeolite as above described may be located between two porous metal plates, porous metal disks, or thin porous metal films.

Advantages provided by the invention include low manufacturing cost, and ease of manufacture. Sensors in accordance with this invention are easily manufactured using conventional paint-spraying techniques wherein the dielectric liquid being spray painted is a dielectric suspension. The simplicity of the manufacturing process of this invention, and the inexpensiveness of the materials, result in an inexpensive, and yet highly-sensitive sensor having the physical appearance of a capacitor. Since the dielectric layer is spray painted onto the metal plates, metal disks, or thin metal film, virtually any dielectric material that can be suspended in a volatile liquid can be employed in the practice of the invention.

Sensors in accordance with this invention find utility in the measurement of the trace water content of various gases of which air, ammonia, argon, arsine, carbon dioxide, chlorine, disilane, tetrafluoromethane (halocarbon 14), hexafluoroethane (halocarbon 16), helium, hydrogen, hydrogen bromide, hydrogen chloride, hydrogen fluoride, hydrogen selenide, nitrogen, nitrogen trifluoride, nitrous oxide, oxygen, phosphine, silane, sulfur hexafluoride, as well as mixtures of theses gases, are non-limiting examples.

Since the metal plates, metal disks, or thin metal films that hold the water-adsorbing dielectric layers are porous, the gas being sampled or analyzed for its moisture content readily flows through the metal plates/disks/films and into intimate contact with the water-adsorbing dielectric layer(s).

These and other features and advantages of the invention will be apparent to those of skill in the art upon making reference to the following detailed description of the invention, which description makes reference to the drawing.

Brief Description of the Drawings

FIG. 1 is a cutaway side view of a first embodiment of the invention wherein a porous or gas permeable water-sensing capacitor structure is placed in line with a gas flow stream whose water concentration is to be monitored.

FIG. 2 is a side view of the two electrode plates or electrode disks of FIG. 1, wherein the two disks are physically separated along a common central axis.

FIG. 3 shows the circular and planar shape of the two electrode disks of FIGS. 1 and 2.

FIG. 4 shows the two disks of FIG. 2 in close physical engagement on the common axis as is also shown in FIG. 1.

FIG. 5 shows an embodiment of the invention wherein only one of the two porous disks carries a thin and porous dielectric coating (zeolite coating).

FIG. 6 shows an embodiment of the invention wherein the two porous disks are each individually formed from a porous non-conductive disk on which a thin and porous metal film is coated as by vacuum deposition, and upon which a thin and porous dielectric coating is then spray painted or dip coated.

FIG. 7 shows an embodiment of the invention wherein one side of two flexible sheet-like non-conductors are coated with a thin porous metal film upon which a thin porous dielectric coating is then coated, a sandwich is then formed of the two flexible sheets, and the sandwich is then rolled into a tube-shaped water sensing capacitor structure in accordance with the invention.

FIG. 8 shows an embodiment of the invention that is similar to FIG. 1 wherein a disk-shaped sensor in accordance with the invention is placed within a non-conductive housing.

FIG. 9 provides a two sensor system in accordance with the invention wherein an active sensor of the invention is subjected to a first portion of a gas flow whose moisture content is to be monitored, and wherein a dry reference sensor that is structurally similar to the active sensor is subjected to a second portion of the gas flow.

FIG. 10 shows the details of an electronic circuit arrangement that provides a two sensor system of the type that shown in, but not limited to, FIG. 9.

Description of the Preferred Embodiment

The present invention provides a two electrode humidity or water sensing capacitor structure whose dielectric is mordenite; i.e., $Na_8Al_8Si_{40}O_{96}(H_2O)_{24}$. However, other materials such as sulfated zirconia, metal oxides, and mixed metal oxides can be used in accordance with the invention.

In principle, any inorganic material that is water adsorbing can be used as the dielectric material of the invention, it being required that the dielectric material adsorb and desorb water in response to variations in the moisture content of a gas in which the dielectric material is immersed or subjected.

An ideal dielectric material for use in the invention is highly water adsorbing so that it operates to produce a measurable impedance change (i.e., a resistance change, a capacitive reactance change, or a combination of a resistance change and a capacitive reactance change) for a small water adsorbed change, thus providing a high signal-to-noise ratio output signal from the sensor. In addition, for gases that are not inert (for example, corrosive gases), the dielectric material must not decompose or volatilize when exposed to the gas. Non-limiting examples of corrosive gases with which the present invention finds utility include chlorine, hydrogen bromide, hydrogen chloride, and hydrogen fluoride.

When using insoluble dielectric materials, such as zeolites and alumina, the material is converted to a powder made into an aqueous or other volatile liquid suspension, and then sprayed or dip coated onto a metallic substrate. A binder may be required in order to improve adhesion of the dielectric material to the metallic substrate.

When using a soluble dielectric material the material is dissolved in water and then sprayed, or dip coated onto a metallic substrate, perhaps also using a binder.

Zeolite dielectric material is chosen for its ability to rapidly adsorb water, and for its resistance to corrosion by corrosive gases, such as hydrogen chloride.

When a sensor in accordance with the invention is used to monitor the water content of a corrosive gas, such as those listed above, sensor components should not be volatilized, or corroded by the corrosive gas, since the material within such components may be transported downstream of the sensor by the gas flow. In microelectronics processes where extremely high gas purity is required, these gas flow impurities can cause chip defects.

In an embodiment of the invention, pellets of mordenite were ground into a fine powder, and the powder was then sieved with a No. 230 sieve, to thus provide particles having a diameter no greater than about 89 microns, but allowing for particles that are smaller than this sieve size.

While a particle size of about 89 microns was used in embodiments of the invention, other particles sizes are useful. In selecting a particle size, the selected size should be large enough to prevent physical contact between the two electrodes and yet small enough to allow the two electrodes to be closely spaced, thus providing an adequate signal from the capacitor structure. The more polished and mutually planar that the dielectric coatings are, the smaller can be the dielectric particle size, and the thinner can be the coatings.

A selected weight of the above-described mordenite powder was mixed with an equal weight of colloidal silica (silicon dioxide or SiO_2), and ten times this weight of deionized water. For example, fused silica is a form of colloidal silica that is used as a thickener, thixotropic and reinforcing agent in inks, resins, rubber, paints, and cosmetics. This three-part mixture was then stirred to achieve a slurry or an aqueous suspension of mordenite particles.

The capacitor substrate in accordance with the invention need only be a rigid or a flexible electrically conductive surface upon which the dielectric layer can be coated.

In an embodiment of the invention, the substrate comprised two porous 316L stainless steel disks that were about 0.062-inch thick, having a diameter in the range of from about 1/4th inch to 2-inch, with about 1-inch being preferred, and having a pore size in the range of from about 2 to about 100 microns.

These stainless steel disks provide a rigid substrate that does not flex, thus minimizing fluctuations in the electronic capacitance measurement that might occur with such mechanical movement of the capacitor electrodes. The porosity of these stainless steel disks allow the gas stream being sampled to freely pass through the substrate and into intimate physical contact with the thin mordenite coating, thus providing a rapid impedance response to water concentration changes in the gas stream being monitored.

Sensors in accordance with the invention also provide a fast response to water content when a major portion of the gas flow being monitored is allowed to bypass the sensor, in which case, the water permeates the sensor by diffusion.

Advantageously, the 316L type stainless steel material is highly resistant to corrosive gases. Although the pore size of the stainless steel substrate is not particularly important to the measurement of impedance change, pore size is important to achieving a relatively low gas pressure drop across the sensor. Stainless steel of the 316 type is made up of 16-18 Cr, 10-14 Ni, 0.03 C, 2.0 Mn, 1.0 Si, 0.045 P, 0.030 S, and 2.0-3.0 Mo.

In coating one flat surface of each of the two stainless steel disks, it is important that the exposed surface of the coating be as flat as possible; i.e., that this exposed flat surface not include a hill/valley profile having upward- protruding surfaces. To aid in achieving this surface smoothness, it may be desirable to polish the mating surfaces of each of the two metal disks. While this polishing step is usually not required, the flatter the two parallel dielectric surfaces are, the thinner the two dielectric coatings can be, thereby increasing the base or starting impedance of the sensor.

The flatness of the exposed mordenite/zeolite dielectric surface of each metal disk ensures that these two exposed dielectric surfaces can be mounted in intimate and full surface physical contact, thus ensuring a maximum base, or starting capacitance for the sensor. In addition, sharp protruding physical features on one, or both, of the flat dielectric disk surfaces may potentially contribute to failure of the sensor due to an electrical short occurring between the two stainless steel disks.

An important feature of the invention is the process by which the stainless steel disks are coated with the mordenite dielectric layer. In practice, the three-part slurry containing No. 230 sieved mordenite/zeolite powder is spray painted onto one flat surface of each of the two stainless steel disks, and the resultant spray coated layer is then dried using a hot air gun. After drying the first layer in this manner, a second layer is spray painted onto the first layer, and this second spray-coated layer is then dried in the same hot air gun manner. While five such spray-coated layers are preferred, a more general teaching of the present invention is that the resulting multi-layer dielectric layer should be thin enough to minimize the physical separation of the two stainless steel disks, and thus provide a high base capacitance value, and yet the multi-layer dielectric layer must be thick enough to prevent an electrical short from occurring between the two stainless steel disks. In accordance with the invention, the final dielectric coating thickness is in the range of from about 0.1 micro to about 200 microns.

By way of a non-limiting example, a Badger brand air brush was used to accomplish the above-described spray painting, with the air brush operating at about 30 psi.

While preferred embodiments of the invention, as above described, utilize spray painting steps, dip coating is a viable alternative to spray painting.

By way of example, when a 0.75-inch diameter disk is coated as above described, the weight of the disk coating is about 6 mg, and the thickness of the disk coating is about 12.4 microns. When the largest zeolite particle within the slurry is about 89 microns and when all voids are removed from the coating to thereby produce a monolithic coating, the nominal thickness of the coating is about 12 microns. Importantly, for the two-disk structure of the present invention, the total two-coating thickness in the range of from about 24 microns (i.e. 2x12) to about 178 microns (i.e. 2x89) is much larger than a dictionary-defined monolayer film thickness, and much smaller than a power compact (assuming the powder compact is a monolithic structure).

Once two such capacitor plate disks are produced in the above-described manner, the two disks are placed in a holder, conduit or housing with the flat dielectric faces or surfaces in intimate and full surface physical contact. This housing is constructed and arranged to physically support the two metal disks while at the same time, electrically isolating the two disks from each other.

When the housing is formed of an electrical insulator, no insulator members are required to hold the disk-shaped substrate members. When the housing is formed of an electrically-conductive material, such as stainless steel, one or more insulator members, for example one or more quartz rings, are provided to hold and electrically isolate the two disk-shaped substrate members from each other.

Electrical contact is then provided to the back, or uncoated flat side of each disk, and these two electrical conductors are connected to an impedance measuring device of conventional and known construction. A non-limiting example of a suitable impedance measuring device is the HP4263B inductor/capacitor/resistor (LCR) meter.

As stated, it may be desirable to include a heating means into this housing. For example, an electrical heater which, when electrically energized, operates to heat the two disks and their two mordenite dielectric coatings to a temperature of from about 100 to about 350-degrees centigrade for the purpose of driving off water that may be strongly bonded to the dielectric coatings.

In its broader concept, the sensor of the present invention provides a pair of rigid or flexible metal members that constitute two physically-opposed plates of a water molecule sensitive capacitor. The sensitivity of this capacitor to water molecules is provided by a thin sprayed-on coating of zeolite powder that is applied to at least one of the two physically engaging surfaces of the two metal members.

The sprayed-on dielectric coating acts as the dielectric of the capacitor. Zeolite has a high affinity for water. The dielectric constant of the sprayed-on coating of zeolite, as initially coated onto the two metal members, is very different from the dielectric constant of the sprayed-on coating of zeolite after it has collected or adsorbed water. This change in the dielectric constant of the dielectric coating is easily detected using a conventional LCR meter.

Dielectric coating(s) of the invention usually do not require thermal activation (i.e., heating to drive off adsorbed water). This is true because the dielectric coating(s) reversibly equilibrates within a reasonably short period of time with any water that is within the gas being monitored.

After making the dielectric coating(s) as above described, the coating(s) may contain a relatively large amount of adsorbed water. However, when the sensor is thereafter placed within a gas to be monitored, the amount of coating-adsorbed water rapidly adjusts to an equilibrium with the water concentration that is within the gas being monitored. That is, when the gas is drier than the sensor dielectric coating(s), water is desorbed from the dielectric coating(s), and when the sensor dielectric coating(s) is drier than the gas, water is adsorbed by the dielectric coating(s). In both cases, the result is a change in sensor impedance.

When using certain dielectric materials to produce a coating(s) in accordance with the invention, and mordenite is an example, the above- described equilibrium condition may occur relatively slowly due to the fact that the various adsorption sites that are within the dielectric material have different bonding strengths. Therefore, in order to accelerate water removal from the dielectric coating(s), it is desirable to heat the dielectric coating(s) as the coatings are concomitantly subjected to a dry gas environment. Once the sensor is activated in this manner, it is desirable to maintain the sensor in a dry atmosphere prior to the sensor being used in a desired sensing environment. As an alternative, sensors having a relatively wet dielectric coating(s) may be placed in the sensing environment, and then heated in-situ. In addition, a sensor that includes this type of dielectric should be heated after being used in a first sensing application, and before it is used in a second sensing application.

An additional property of dielectric coatings that have some water- adsorbing sites with a high bonding strength is that water that is too strongly bonded does not appreciably contribute to an increase in the dielectric constant of the sensor dielectric coating(s). If dielectric coatings of this type are dried too much, the strong bond sites loose their water, and when the sensor is thereafter used in a sensing environment, the first sites to fill with water are these strong bond sites that do no appreciably contribute to a sensor impedance change. As a result, a delay may occur when measuring very low water concentrations, such as 1-2 PPM and less.

One utility of the above-described capacitor sensor in accordance with the invention is as an in line humidity sensor that is be placed within a gas purifier or within a gas line that exists from the gas purifier.

An additional use of capacitor sensors in accordance with the invention is in the manufacture of semiconductor and microelectronic materials wherein it is common to apply various gases to the materials being manufactured. It is highly desirable to minimize any contaminants within the gases that are being applied to the semiconductor materials. One of the most common contaminants is water. Accordingly, it is typical to provide a gas purifier device at some point in the gas line(s) that supply a gas to the manufacturing process.

A typical gas purifier includes a hollow and elongated vessel that is filled with a material that collects and retains water from gas that passes through the vessel. In this way, gas exiting the gas purifier has had water/moisture removed therefrom. It can be appreciated that the water collecting agent that is within the gas purifier eventually becomes spent, and as a result, the gas purifier no longer serves its intended purpose of purifying the gas by removing water therefrom.

It is desirable that an indication be provided when the gas purifier is nearly spent so that an alternate means of purifying the gas can be switched into the gas line; for example, by replacing the spent or partially spent gas purifier with another gas purifier.

Since the water-removing agent that is within the gas purifier becomes spent in a gradual time wave that moves across the gas purifier in the direction of gas flow from gas inlet to gas outlet, it is possible to place a humidity sensor at or near the gas outlet in order to detect when gas passing through the gas outlet, or at least a portion of the gas passing through the gas outlet, begins to have water or humidity therein.

Because of the porous nature of the substrate members of the above-described capacitor in accordance with the present invention, the above- described capacitor can be placed in a position relative to the outlet of the gas purifier so that outlet gas, or at least a portion thereof, must pass therethrough. A meter, such as a LCR meter, is then electrically connected to the above-described capacitor, thereby providing an indication when the impedance thereof (i.e., the resistance, the capacitance, or the resistance and capacitance) has changed due to water being absorbed by the capacitor dielectric. It is possible to place the sensor of the invention within the gas purifier, or in an output gas line that is located outside of the gas purifier. It is also possible to place the sensor in a manner to sample only a portion of the purifier output gas flow.

An embodiment of an in-line sensor capacitor 10 in accordance with the invention is shown in FIG. 1. Sensor 10 includes a hollow, tubular, and non-porous stainless steel sleeve 12 having an inwardly-protruding shoulder 13 formed near end 11 thereof.

An annular quartz ring 20 is received within sleeve 12 and positioned against shoulder 13. Ring 20 provides an annular-shaped cavity for receiving the two capacitor electrode plates 14 and 16, and ring 20 prevents metallic and electrical contact of metal plates 14 and 16 with metal sleeve 12. A first porous stainless steel, circular, and flat disk-shaped electrode plate 14 is received within the ring 20. A second electrode plate 16 (generally similar in construction and arrangement to first electrode plate 14) is also received within ring 20.

As shown in FIG. 2, the opposed parallel and flat surfaces 15 and 17 of plates 14 and 16 are each coated with a sprayed-on thin layer 18 of powdered zeolite in the manner above described. As described above, surfaces 15 and 17 may be polished to insure that these two surfaces are flat and coplanar.

An annular insulator ring 22 is next received within sleeve 12, and an externally-threaded and hollow annular nut 24 having an external thread pattern is then screwed into the end 19 of sleeve 12 that is opposite to shoulder 13. Sleeve 12 is provided with an internal thread pattern that mates with the external thread pattern that is carried by nut 24. Nut 24 is manually- screwed sufficiently far into sleeve 12 to maintain plates 14 and 16 in close physical proximity. The force by which plates 14 and 16 are held together is not critical. However, operation of nut 24 should not produce a high force that operates to fracture dielectric coating(s) 18.

Two electrical conductors 26 and 28 are individually electrically connected with one conductor to each of the two plates 14 and 16. Electrical conductors 26 and 28 are connected to a conventional LCR meter 30 that monitors the value of the impedance of the capacitor that is created by the combination of plates 14 and 16 and dielectric zeolite material 18.

Any of a variety of gases can be allowed to pass through sensor device 10 in either direction, as is indicated by double-headed flow arrow 32. In this manner, the gas passes through porous plates 14 and 16 and thin zeolite coating(s) 18. Thus, zeolite coating(s) 18 operates to collect water that may be in the gas passing therethrough.

In order to activate zeolite coating 18, thus making it more sensitive to the water content of the gas, it may be desirable to heat zeolite coating 18 prior to use, or after extended use. This heating operation provides that zeolite coating 18 will give up any water therein that has accumulated over time. Zeolite coating 18 may be heated at the time of sensor manufacture, or an electrical heater 31 may be provided as a portion of sensor 10 to periodically activate/reactivate zeolite coating 18 as desired.

While zeolite is preferred, capacitance-type impedance moisture sensors in accordance with the invention may utilize almost any type of water- adsorbing dielectric material 18. It is only necessary to form the dielectric material into a fine powder, perhaps add an amount of a binder, add an amount of water, form a slurry, and then coat the slurry onto electrode/capacitor plates 14, 16.

It may be desirable to provide an output comparison network (see 34 of FIG. 8) to which the electrical output 35 of meter 30 is connected. This comparison network would operate to compare signal 35 with a reference impedance signal. When the impedance of capacitor 14, 16, 18 bears a predefined relationship to the reference signal (for example, when the impedance of capacitor 14, 16, 18 becomes greater than the reference signal), an output from comparison network becomes active. For example, when gas stream 32 comprises the output of a gas purifier (not shown), when the impedance value of capacitor 14, 16, 18 becomes greater than the reference impedance signal, the output of the comparison network becomes active to indicate that the gas purifier should be replaced.

FIG. 2 is a side view of the two stainless steel disks 14, 16 of FIG. 1 in a spaced-apart relationship. FIGS. 2 and 3 show that each of the two circular disks 14, 16 are generated about a centrally-located axis 39 that extends generally perpendicular to the parallel and planar faces or surfaces 41, 42 of disks 14, 16, and that axis 39 also extends perpendicular to the exposed surface 40 of each of the thin dielectric coatings 18, exposed surfaces 40 being mutually parallel and parallel to surfaces 41 and 42.

FIG. 4 shows the two stainless steel disks 14, 16 in their FIG. 1 operating condition wherein operation of nut 24 has brought the two parallel dielectric surfaces 40 into an intimate and full surface abutting relationship.

FIG. 5 is a view similar to FIG. 2 wherein only disk 14 carries a thin dielectric coating 18 in accordance with the invention on its inner surface 15; i.e., disk 16 does not carry such a dielectric coating. When the disks 14, 16 of FIG. 5 are assembled in the manner of FIGS. 1 and 4, the surface 40 of zeolite coating 18 physically engages the parallel surface 17 of disk 16 in a full surface engaging manner.

FIG. 6 is a side view of an embodiment of the invention having two thin and flat disk-shaped capacitor plates as above described relative to FIGS. 1-5. For example, the embodiment of FIG. 6 operates as a substitute for capacitor 14, 16, 18 of FIG. 1.

The embodiment of FIG. 6 includes two porous and electrical insulator disks 44 and 45 that are each formed of a rigid non-conductor, such as sintered glass or a like material. The flat and mutually parallel inner surface 46 of each sinter glass disk 44, 45 is coated with a porous thin film 47 or porous thin electrode 47 that comprises an electrically-conductive metal that is inert to the fluid flow being tested; for example, vacuum-deposited porous electrodes 47 of gold, nickel, copper, or carbon.

The mutually parallel surfaces 50 of one, or both, of the porous electrodes 47 is then coated with a thin coating of porous dielectric 18 in the manner above described. Porous and thin electrodes 47 are then electrically connected to FIG. 1 meter 30 as above described. An example thickness of glass disks 44 and 45 is from about 0.5 mm to about 5 mm.

FIG. 7 is an end view of an embodiment of the invention wherein the dielectric support substrate of a water-sensing capacitor in accordance with the invention comprises a flexible non-conductor material, such as woven fiberglass or zirconia cloth. In this embodiment of the invention, each length of the woven material is first coated on one side with a thin film or thin electrode of an electrically-conductive metal that is inert to the fluid flow being tested; for example, electrodes 47 as above described.

This thin metal film 47 is then coated with a thin coating of dielectric as above described. The resulting flexible capacitor structure is then rolled into a cylinder form 60, and gas being sampled is passed axially of cylinder 60. Above-described LCR meter 30 is then connected to the two thin films of metal 47.

FIG. 8 provides yet a further embodiment of the invention. In this embodiment, a capacitor sensor 73 in accordance with the invention is located within a hollow, generally tubular shaped and closed housing 70 that is made of a non-conductive material. Housing 70 defines an internal gas inlet compartment 71 and an internal gas outlet 72 compartment.

Compartments 71 and 72 are separated by a humidity-sensing capacitor 73 in accordance with the invention, capacitor 73 being of the type above described relative to FIGS. 2, 3 and 4. As gas passes through housing 70 from a gas inlet 75 to a gas outlet 76, water molecules within the gas are adsorbed by thin dielectric coatings 18, and an impedance signal 35 is generated by meter 30 as above described. If desired, this signal 35 can be compared at 34 with a reference signal 36, to thereby produce and output signal 38 when signal 35 bears a predefined relationship to reference signal 36.

The FIG. 1 embodiment of the invention employs a single sensor 10. However, sensor 10 may be susceptible to changes in measuring conditions, such as temperature and pressure, that do not relate to the concentration of water that is within the gas being monitored. FIG. 8 provides a reference impedance value 37, and in order to prevent errors from occurring in the FIG. 8 embodiment as a result of changes in non-water measuring conditions of this type, reference impedance network 36 must be constructed and arranged to compensate for changes in the non-water measuring conditions.

FIG. 9 provides a two-sensor system in accordance with the invention wherein an active sensor 90, as above described, is subjected to a first portion 91 of the flow 92 of a gas whose moisture content is to be monitored, and wherein a reference sensor 93 as above described is subjected to a second portion 94 of gas flow 92.

Sensors 90 and 93 are preferably identical in construction and arrangement, each sensor having two electrode plates 14, 16 and a thin coating 18 of dielectric between plates 14, 16, to thus form an active sensing capacitor 90 and a reference capacitor sensor 93.

Active sensor 90 is contained within a housing so as to at least partially obstruct gas flow 91. While FIG. 9 shows that all of gas flow 91 passes through active sensor 90, this construction and arrangement is not required. All that is required is that the thin dielectric coating 18 of active sensor 90 be subjected to any water content that may be in gas flow 92, and thus in gas flow 91, preferably with a minimum pressure drop occurring across active sensor 90.

Reference sensor 93 is shielded from the water content of gas flow 92, 94 by surrounding reference sensor 93 with a drying agent, or desiccant material 96. In this way, reference sensor 93 is kept in a dry environment within a housing 97. Again, it is not required that reference sensor 93 and its surrounding drying agent 96 intercept all of gas flow 94, as is shown in FIG. 9, and it is preferred that reference sensor 93 and its surrounding drying agent 96 provide little or no pressure drop in gas flow 94. All that is required is that reference sensor 93 be exposed to the same fluctuations in temperature and pressure as is active sensor 90, thus enabling reference sensor 93 to provide an dry gas impedance signal that reflects any changes in temperature, and/or pressure that may occur in the sensing environment.

A differential impedance measuring network 99 has a first input 100 that is connected to electrodes 14, 16 of active sensor 90 and a second input that is connected to electrodes 14, 16 of reference sensor 93. Network 99 operates to measure the differential between the two impedances of the two sensors 90, 93. That is,. the resistance, capacitive reactance, and/or resistance plus capacitive reactance of reference sensor 93 is subtracted from the resistance, capacitive reactance, and/or resistance plus capacitive reactance of active sensor 90. Thus, temperature and/or pressure fluctuations that may occur in the sensing environment do not affect the output 102 of network 99, output 99 then being an indication of only the water content of gas flow 92.

While FIG. 9 shows a construction and arrangement having the two flow paths 91 and 94, such is not required by the invention. That is, a single flow path, such as flow path 92, can be provided wherein active sensor 90 is placed in flow path 92 so as not to be isolated from the water/moisture content of the gas flow, and wherein reference sensor 93 is placed closely adjacent to active sensor 90, so that both of the sensors 90, 93 experience the same gas temperature and gas pressure, but wherein reference sensor 93 is housed so as to be isolated from the water/moisture content of the gas flow.

FIG. 10 shows the details of an electronic circuit arrangement 110 that is usable to provide a two-sensor system such as is shown in, but is not limited to, FIG. 9, wherein circuit arrangement 110 operates to provide a DC output 111 that is not affected by temperature and/or pressure changes that may occur at a moisture content sensing environment.

Circuit arrangement 110 compares a sensor impedance value 112 that is provided by an active sensor, such as 90 of FIG. 9, to a reference-impedance value 113 that is provided by a reference sensor, such as 93 of FIG. 9. It is to be noted that active sensor 90 is subjected to the moisture, temperature and pressure of the gas environment being monitored, but reference sensor 93 is subjected only to the temperature and pressure of the gas environment being monitored.

Again it is to be noted that impedance values 112 and 113 are preferably provided by sensor construction and arrangements that provide only a minimal pressure drop at the moisture content sensing environment, that sensor-impedance value 112 reflect moisture at the moisture content sensing environment (for example, gas flow 92 of FIG. 9), and that reference impedance value 113 reflects only temperature and/or pressure changes that may occur at this same moisture content sensing environment. That is, reference impedance value 113 is derived from a moisture-free environment that shares temperature and pressure changes with the moisture that generates sensor impedance value 112.

A reference oscillator 114 supplies an AC sine wave or square wave current to a series combination of sensor impedance generator 90 and reference impedance generator 93. Sensor impedance generator 90 operates to generate an impedance value 112 that is responsive to the moisture, the temperature and the pressure of the gas environment being monitored, whereas reference impedance generator 93 operates to generate an impedance value 113 that is responsive only to the temperature and pressure of the gas environment being monitored.

Differential amplifier 115 receives sensor impedance value 112 at a first input 116 and reference impedance value 113 at a second input 117. Amplifier 115 now operates to subtract reference impedance value 113 from sensor impedance value 112. This subtraction operation removes from AC amplifier output 118 all common impedance components of each of the sensors 90, 93 that are sensitive to temperature and/or pressure changes at the moisture-sensing environment. Thus, AC output 118 is proportional only to the moisture at the moisture-sensing environment.

Mixer 119 operates to modulate AC output 118 to a DC output 120. For example, mixer 119 is implemented by an analog multiplier, or by switching a gain block between +1 and -1 on phase-shifted zero crossings of reference oscillator 114.

Variable phase shifter 121 receives the output 122 of reference oscillator 114 and selectively operates to shift the phase of output 122 in the range of from 0 to 90-degrees as a function of the position of manual control knob 125, to thereby select various resistance /reactance components that are within sensor impedance value 112.

A phase shift of 0-degrees operates to select the resistance component of sensor impedance value 112, a phase shift of 90-degrees operates to select the capacitive reactance component of sensor impedance value 112, and phase shifts that lie between 0-degrees and 90-degrees operate to select varying combinations of the resistive component and the capacitive reactance component of sensor impedance value 112.

Depending on the particular gas that is being monitored for moisture content at the moisture-sensing environment, sensing resistance, sensing capacitive reactance, or sensing a combination of both resistance and capacitive reactance may provide an output 111 that is more sensitive to changes in the moisture content of the particular gas.

Low pass filter 123 operates to remove any spurious double frequency components that may exist in DC output 120, such that output 111 comprises a pure DC value that is proportional to the moisture that is experienced by active sensor 90.

The invention has been described in detail while making reference to embodiments thereof. However, since it is known that other skilled in the art will, upon learning of the invention, readily visualize yet other embodiments that are within the spirit and scope of the invention, the above detailed description is not to be taken as a limitation on the invention.

What is claimed is:

CLAIMS

We Claim:

1. A sensor for use in measuring the quantity of water that is present in a gaseous environment comprising:

a first porous metal electrode having a first smooth surface;

a thin dielectric coating formed of a dielectric material that adsorbs water on said first surface;

said thin dielectric coating having a second surface that physically engages said first surface and having a third surface that is physically spaced from and parallel to said second surface;

said thin dielectric coating having a dielectric constant that varies as a function of the quantity of water adsorbed by said dielectric material; and

a second porous metal electrode having a fourth smooth surface physically engaging said third surface;

said first metal electrode, said thin dielectric coating, and said second metal electrode forming a capacitor that is subjected to said gaseous environment such that the impedance of said capacitor varies as a function of the quantity of water adsorbed by said thin dielectric coating.

2. The sensor of claim 1 wherein said first, second, third and fourth surfaces are generally flat surfaces.

3. The sensor of claim 1 wherein said first and second electrodes are formed of a porous metal.

4. The sensor of claim 3 wherein said porous metal is stainless steel.

5. The sensor of claim 4 wherein:

said stainless steel has a pour size in the range of from about 2 to about 100 microns in diameter; and

said thin dielectric coating includes a powdered dielectric material having particles that are no greater than about 89 microns in diameter.

6. The sensor of claim 5 wherein said powdered dielectric material is an inorganic water adsorbing dielectric material.

7. The sensor of claim 5 wherein said powdered dielectric material is selected from the group zeolite, sulfated zirconia, metal oxide and mixed metal oxide.

8. The sensor of claim 5 wherein said powdered dielectric material is mordenite.

9. The sensor of claim 8 wherein said mordenite coating is formed by painting a uniform thickness layer of a slurry containing powdered mordenite and a non-volatile binder on said first surface.

10. The sensor of claim 8 wherein said mordenite coating is formed by painting a uniform thickness layer of a slurry containing powdered mordenite and a non-volatile binder on both said first surface and said fourth surface.

11. The sensor of claim 10 including:

a heater for heating said mordenite coating so as to cause said coating to desorb water therefrom.

12. The sensor of claim 1 wherein said first and second porous metal electrodes respectively comprise:

a first and a second porous flexible sheet of insulating material, each sheet having a thin and porous metal film on one side thereof, to thereby form said first and said fourth surfaces;

said thin porous metal films and said dielectric coating that is located intermediate said thin porous metal films forming said capacitor.

13. The sensor of claim 12 wherein:

said powdered dielectric material is mordenite;

said first and second porous sheets and said porous metal films have a pour size in the range of from about 2 to about 100 microns in diameter; and

said powdered mordenite includes particle that are no greater than about 89 microns in diameter.

14. The sensor of claim 13 wherein said mordenite coating is formed by painting a uniform thickness layer of a slurry containing powdered mordenite, a non-volatile binder, and deionized water on one or both of said first surface and said fourth surface.

15. The sensor of claim 14 including:

a heater for heating said mordenite coating so as to cause said coating to desorb water therefrom.

16. The sensor of claim 14 wherein said first, second, third and fourth surfaces are parallel curved surfaces.

17. A method of measuring the water content within a gaseous environment comprising the steps of:

providing a first porous metal electrode having a first smooth surface;

forming on said first surface a thin dielectric coating of a dielectric material that adsorbs water;

said thin dielectric coating having a second surface that physically engages said first surface and having a third surface that is physically spaced from and parallel to said second surface;

said thin dielectric coating having a dielectric constant that varies as a function of the quantity of water that is adsorbed by said dielectric material;

providing a second porous metal electrode having a fourth smooth surface that physically engages said third surface;

said first metal electrode, said thin dielectric coating, and said second metal electrode forming a water sensitive capacitor;

subjecting said water-sensitive capacitor to said gaseous environment such that the impedance of said water-sensitive capacitor varies as a function of the quantity of water that is adsorbed by said thin dielectric coating; and

measuring the impedance of said water-sensitive capacitor.

18. The method of claim 17 including the step of forming said first and second metal electrodes of a porous metal.

19. The method of claim 18 wherein:

said porous metal has a pour size in the range of from about 2 to about 100 microns in diameter; and

said thin dielectric coating includes a powdered dielectric material having particles that are no greater than about 89 microns in diameter.

20. The method of claim 19 wherein said powdered dielectric material is mordinite.

21. The method of claim 20 including the steps of:

forming a slurry containing said powdered mordinite, a non-volatile binder, and water; and coating said on said first surface using a process selected from the group spray painting and dip coating.

22. The method of claim 21 including the step of:

coating said slurry on said fourth surface to thereby form a thin dielectric coating on said fourth surface.

23. The method of claim 21 including the steps of:

providing a electric heater for heating said mordenite coating so as to cause said coating to desorb water therefrom; and

electrically energizing said electric heater prior to subjecting said capacitor to said gaseous environment.

24. A sensor for use in measuring the quantity of water that is present in a gaseous environment comprising:

a first porous and non-metallic member having a first smooth surface;

a first porous metal film formed on said first smooth surface, said first porous metal film having an exposed second smooth surface;

a thin dielectric coating of a dielectric material that adsorbs water on said first surface formed on said second smooth surface;

said thin dielectric coating having an exposed third smooth surface that is physically spaced from and parallel to said first and second smooth surfaces;

said thin dielectric coating having a dielectric constant that varies as a function of the quantity of water adsorbed by said dielectric material;

a second porous and non-metallic member having a fourth smooth surface physically engaging said third smooth surface;

a second porous metal film formed on said fourth smooth surface, said second porous metal film having an exposed fifth smooth surface that is parallel to and that physically engages said third smooth surface;

said first and second porous metal films and said thin dielectric coating forming a capacitor that is subjected to said gaseous environment such that the impedance of said capacitor varies as a function of the quantity of water adsorbed by said thin dielectric coating.

25. The sensor of claim 24 wherein said first and second non-metallic members are flexible, and wherein said capacitor is cylindrical in shape.

26. The sensor of claim 24 wherein said dielectric coating is a powdered inorganic water-adsorbing dielectric material.

27. The sensor of claim 26 wherein said powdered dielectric material is selected from the group zeolite, sulfated zirconia, metal oxide and mixed metal oxide.

28. The sensor of claim 27 wherein said powdered dielectric material is mordenite.

29. A method of measuring the water content within a corrosive gas, comprising the steps of:

providing a first porous stainless steel member having a first smooth surface;

forming a first thin mordinite dielectric coating on said first smooth surface;

said first thin mordinite coating having an exposed second smooth surface that is physically spaced from and parallel to said first smooth surface;

providing a second porous stainless steel member having a third smooth surface;

forming a second thin mordinite dielectric coating on said third smooth surface;

said second mordinite coating having an exposed fourth smooth surface that is physically spaced from and parallel to said third smooth surface;

said first and second mordinite coatings having dielectric constants that vary as a function of the quantity of water that is adsorbed by said mordinite coatings;

placing said second and fourth smooth surfaces in physical contact to thereby form an assembly;

subjecting said assembly to said corrosive gas such that the electrical impedance of said assembly varies as a function of the quantity of water that is adsorbed by said first and second thin mordinite coatings; and

connecting an electrical impedance-measuring circuit to said first and second stainless steel members.

30. The method of claim 29 including the step of selecting the corrosive gas from a group consisting of chlorine, hydrogen bromide, hydrogen chloride, and hydrogen fluoride.

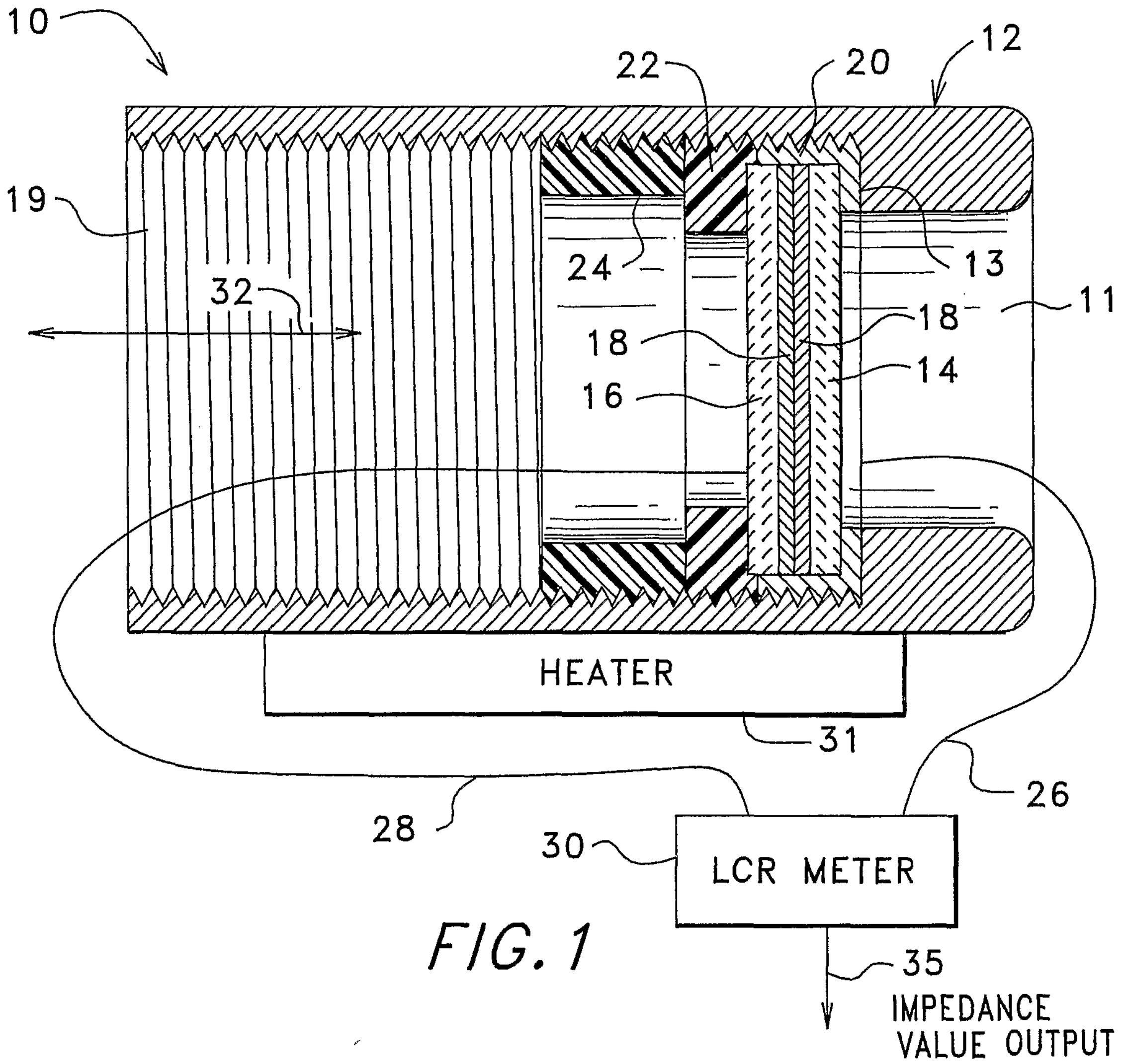

FIG. 1

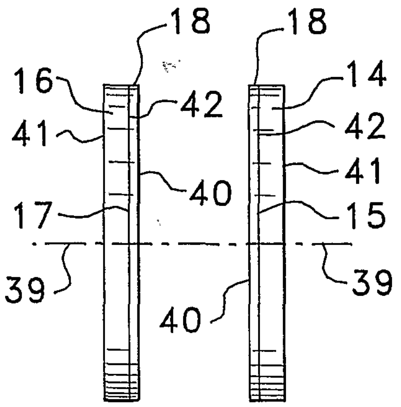

FIG. 2

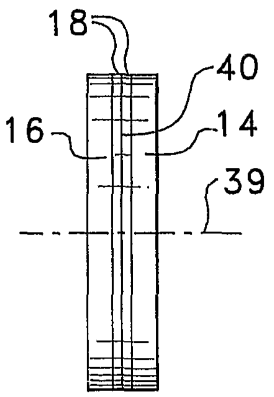

FIG. 4

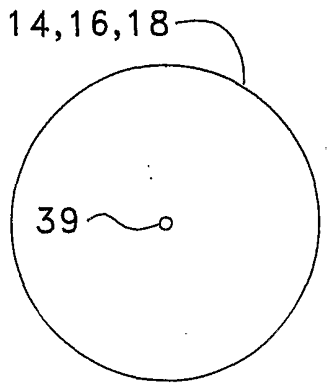

FIG. 3

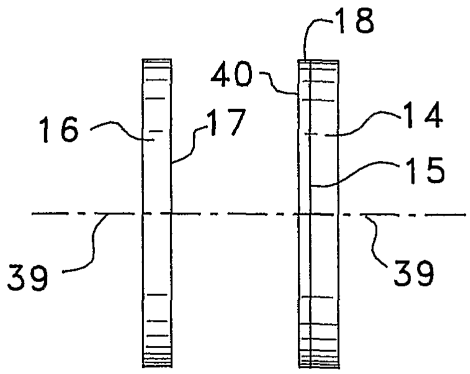

FIG.5

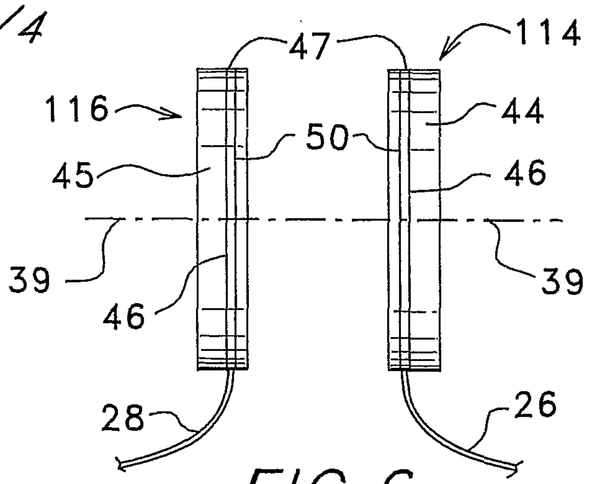

FIG.6

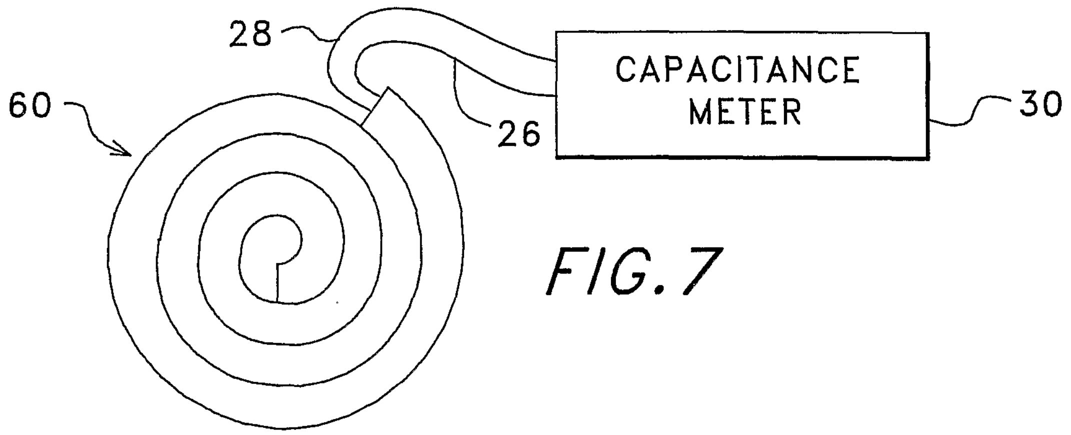

FIG.7

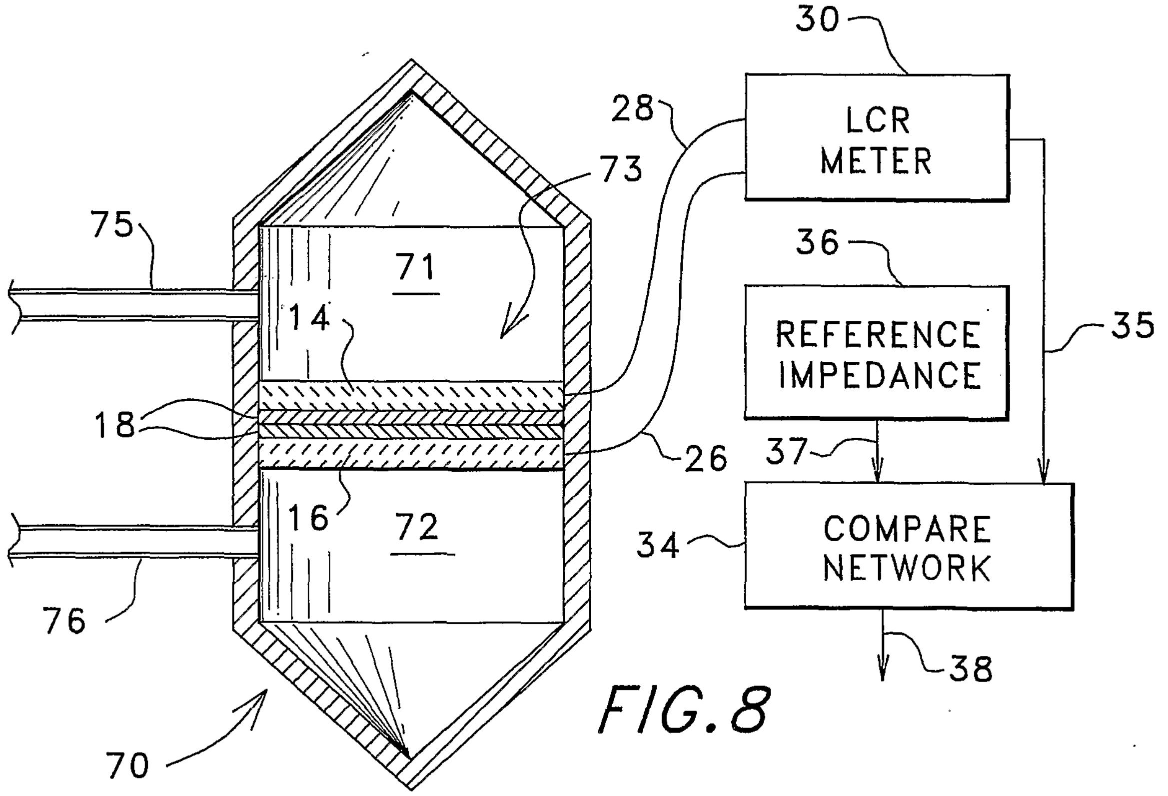

FIG.8

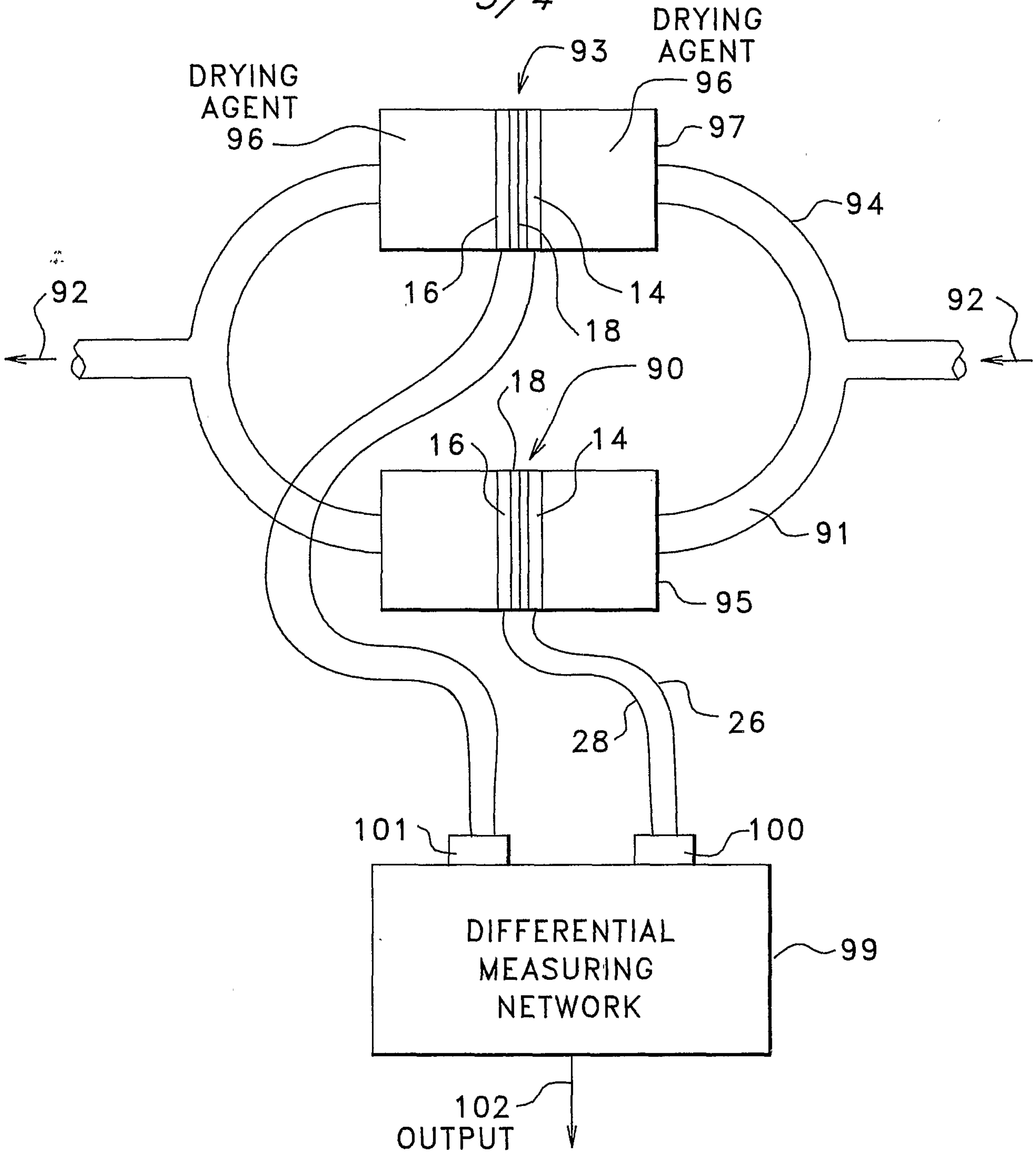
93
DRYING AGENT 96
DRYING AGENT 96
97
94
16
14
18
92
92
18
90
16
14
91
95
28
26
101
100
DIFFERENTIAL MEASURING NETWORK
99
102
OUTPUT

FIG. 9

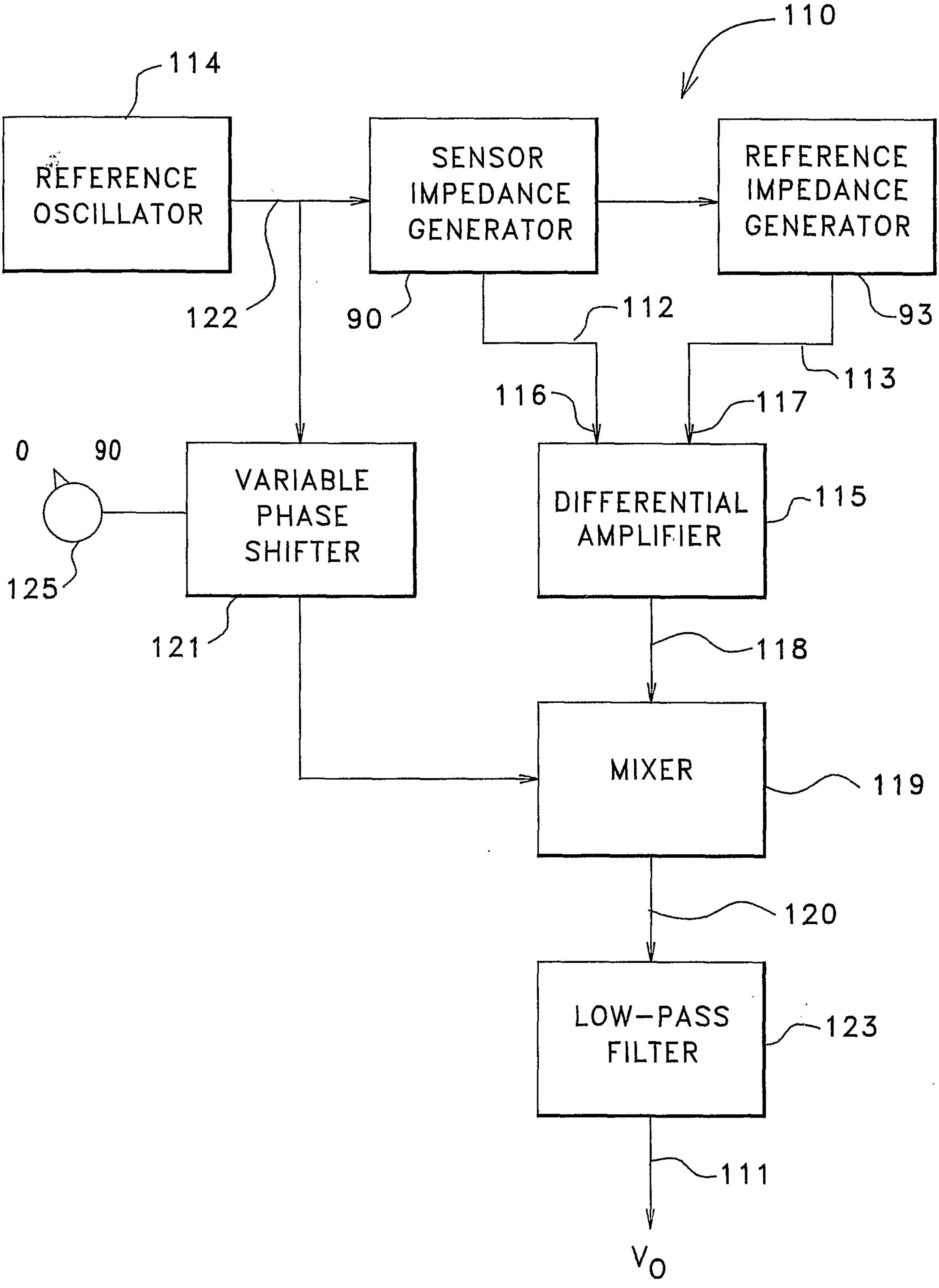
110
114
REFERENCE OSCILLATOR
SENSOR IMPEDANCE GENERATOR
REFERENCE IMPEDANCE GENERATOR
122
90
112
93
113
116
117
0
90
VARIABLE PHASE SHIFTER
DIFFERENTIAL AMPLIFIER
115
125
121
118
MIXER
119
120
LOW-PASS FILTER
123
111
V_O

FIG. 10