Device, system and method for the measurement of a vapor transmission rate through a film of permeable material

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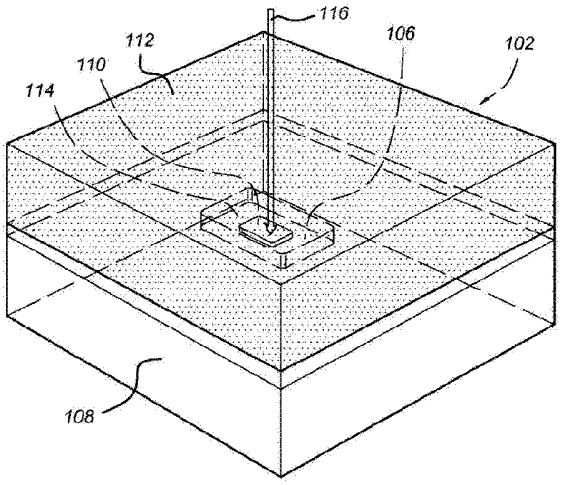
(54) **Device, system and method for the measurement of a vapor transmission rate through a film of permeable material.**

(57) The invention relates to a vapor transmission rate measurement device (102), comprising a pressure sensor (110) and a chamber (106) with a chamber volume V, the chamber (106) having an opening (114) with a surface area A, the opening (114) being arranged to be spanned by a film (112) of permeable sample material. The vapor transmission rate measurement device (102) is arranged for determining an indication of the vapor transmission rate for the film (112) permeated by a vapor (116), based on at least one measurement by the pressure sensor (110) of a pressure P within the chamber (106). A ratio R between the chamber volume V and the surface area A is smaller than 10^{-5} m.
The pressure sensor (110) may for example have a microelectromechanical or nanoelectromechanical resonator (202), or a microelectronic or nanoelectronic Pirani gauge (302).



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Device, system and method for the measurement of a vapor transmission rate through a film of permeable material.

TECHNICAL FIELD

The present invention relates to a vapor transmission rate measurement device, to a vapor transmission rate measurement system and to an electronic device comprising a vapor transmission rate measurement device. Furthermore, it relates to a method of determining a vapor transmission rate.

BACKGROUND

WVTR is the abbreviation for water vapor transmission rate, which is defined as the rate at which water vapor permeates through a layer of material at specified conditions of temperature and relative humidity. In an analogous way, the oxygen transmission rate or OTR is defined. More general, the vapor transmission rate or VTR is used to indicate the rate at which a particular gaseous substance permeates the material.

WVTR and OTR are of great significance for package quality. Electronic equipment for example is prone to deterioration due to corrosion. Therefore, electronic devices are commonly protected by enveloping structures and/or films. With sufficient knowledge of the relevant vapor transmission rates of the protective materials, the probable lifetime of electronic devices can be predicted in a more reliable way. This information facilitates quality control for such devices. The VTR is a particularly important property for flexible electronics, as corrosion induced deterioration is a dominant lifetime reducing mechanism.

From patent document GB 2370649 a method and an apparatus for measuring the rate of transmission of water vapor are known. The apparatus comprises a vacuum chamber enclosing a gas chamber. The gas chamber has an opening for transmitting gas through a material sample. A mass spectrometer is provided for measuring the partial pressures of water molecules in the vacuum chamber. The method involves the introduction of a water droplet inside the gas chamber. The vaporized water subsequently permeates and traverses the material sample and enters the vacuum

chamber, thereby increasing the pressure in the initially vacuumed chamber. The partial pressures measured by the mass spectrometer are used to derive a water vapor transmission rate for the material sample.

Unfortunately, the method and apparatus have a practical lower detection limit for the water vapor transmission rate in the order of milligrams per square meter per day, at least if the VTR determination is based on pressure variation measurements occurring within a day or less. Flexible electronics and organic light emitting diodes (OLED) require better shielding from corrosive agents in order to obtain an acceptable lifetime. A water vapor transmission rate of 10^{-6} g m^{-2} day^{-1} or lower is desired in the OLED industry to achieve an acceptable device lifetime of over 10.000 hours.

SUMMARY

It is an object to provide a method and device for measuring the vapor transmission rate of a sample material, yielding an improved vapor transmission rate sensitivity given a desired measurement time interval, or a shortening of the measurement time interval given an expected fixed value for the VTR.

Therefore, according to an aspect there is provided a device according to claim 1.

The small ratio R between the chamber volume V and the surface area A infers that the characteristic dimensions of the surface area A are substantially larger than the characteristic dimension of the chamber perpendicular to the surface area. By providing a relatively large area for the vapor to enter the chamber having a relatively small transverse dimension, a relatively large amount of vapor particles will be able to penetrate the film into a relatively small chamber volume, thereby contributing to a rapid buildup of the chamber pressure. The relatively shallow chamber partially or completely harbors a highly sensitive pressure sensor that may have microscopic or nanoscopic proportions and is able to detect desired pressure differences within measurement time intervals of a day or less, related to the desired vapor transmission rate of 10^{-6} g m^{-2} day^{-1} or less. Advantageously, this dimensional restriction for the chamber enables sensitive vapor transmission rate measurements on thin films with the desired vapor transmission rates, within an acceptable measurement time.

According to an embodiment, the pressure sensor of the vapor transmission rate measurement device comprises a microelectromechanical (MEM) or nanoelectromechanical (NEM) resonator, wherein the at least one measurement of a pressure comprises a measurement of a resonator quality of the MEM or NEM resonator. Advantageously, the chamber is well suited as a resonator cavity harboring a resonator element by means of which the pressure can be sensed.

According to another embodiment, the pressure sensor of the vapor transmission rate measurement device comprises a microelectronic (ME) or nanoelectronic (NE) Pirani gauge, wherein the at least one measurement of a pressure comprises a measurement of an electrical resistance of the ME or NE Pirani gauge. Advantageously, the chamber is equally well suited as gas chamber harboring a Pirani bridge by means of which the pressure can be sensed.

BRIEF DESCRIPTION OF THE DRAWINGS

Embodiments will now be described, by way of example only, with reference to the accompanying schematic drawings in which corresponding reference symbols indicate corresponding parts, and in which:

FIG. 1 schematically shows a perspective view of a vapor transmission rate measurement device according to an embodiment.

FIG.'s 2A - 2C present perspective, side and top cross sectional views of the vapor transmission rate measurement device according to an embodiment.

FIG.'s 3A - 3C present perspective, side and top cross sectional views of the vapor transmission rate measurement device according to another embodiment.

FIG. 4 shows an embodiment of a vapor transmission rate measurement system.

FIG. 5 presents a perspective view of an embodiment of a flexible electronic device comprising a vapor transmission rate measurement device.

FIG. 6 presents a Q-factor versus pressure P relation for an embodiment of a vapor transmission rate measurement device comprising a MEM resonator.

The figures are only meant for illustrative purposes, and do not serve as restriction of the scope or the protection as laid down by the claims.

DETAILED DESCRIPTION

Definition of VTR

The vapor transmission rate or VTR is defined as the rate at which a vapor permeates through a layer of material at specified conditions for the temperature and relative humidity. The vapor transmission rate may be expressed with the following formula describing the change in total mass m of a vapor within a particular fixed volume V on one side of the permeable material, due to a net particle flux through a constant surface area A of the permeable material, within a continuous time span from t_1 to t_2.

$$VTR(t_1, t_2) = \frac{m_2 - m_1}{A(t_2 - t_1)} \qquad (1)$$

If the variation in mass m corresponds to only one type of gas molecules, then the ideal gas law can be utilized to obtain a relation expressed as a change in the total pressure P within the volume V:

$$VTR(t_1, t_2) = \frac{M(P_2 - P_1)V}{Ak_BT(t_2 - t_1)} \qquad (2)$$

Here, T is the temperature in Kelvin, which is also assumed to be (approximately) constant in time, M is the molar mass expressed in grams per mole, P is expressed in Pascal, and k_B represents the Boltzmann constant in Joules per Kelvin. The VTR is preferably expressed in units of grams per square meter per day (g m^{-2} day^{-1}), requiring the time t to be expressed in units of days. For vapors consisting of distinctive molecular components, the VTR values are determined for each vapor component individually, the pressures in equation 2 being interpreted as the partial pressure contributions from each component to a total pressure present within the volume V.

Formally, the VTR as a function of time and assuming a constant surface is expressed as a derivative. The underlying assumption in the discrete form of equation (1) is that the VTR remains constant during the time interval between the measurements at t_1 and t_2.

Advantageously, the proposed method of measuring the vapor transmission rate results in an improved VTR measurement sensitivity, or equivalently in a shortening of

the measurement time interval required. Apart from the evident benefits of higher measurement time efficiency, a shortened measurement time decreases the measurement errors due to fluctuation of the ambient conditions (e.g. the temperature) and renders the assumption of a static VTR more reliable.

Setup

FIG. 1 schematically shows a perspective view of a vapor transmission rate measurement device 102 according to an embodiment. The vapor transmission rate measurement device 102 shown basically consists of a measurement cell formed by a housing 108. The vapor transmission rate measurement device 102 comprises a chamber 106 with a chamber volume V surrounded by the housing 108, as well as a pressure sensor 110. The chamber has an opening 114 with a surface area A, which can be spanned by a film 112 comprising permeable sample material. The opening 114 constitutes an aperture for a vapor 116 to traverse the film 112, if present, from the surroundings into the chamber 106 (indicated by the solid arrow). The vapor transmission rate measurement device 102 is arranged for determining an indication of the vapor transmission rate for the film 112 permeated by the vapor 116.

A specific property of the chamber 106 is that a ratio R between the chamber volume V and the surface area A is smaller than 10^{-5} m. In particular, the ratio R is preferred to be smaller than $2.5 \cdot 10^{-6}$ m. For a measurement chamber 106 with a basic geometrical shape, for which the volume V can be described as a product of a surface area A for the opening 114 and a depth d (e.g. a cylinder with an opened circular surface, or a box as shown in FIG. 1), the small ratio R implies that the depth d is smaller than 10 micrometer, and preferably smaller than 2.5 micrometer. Alternatively, the chamber 106 may have a more sophisticated shape that cannot be described by $V = A \cdot d$, which may be exploited to improve the ratio R. The VTR measurement device 102 is designed to measure VTR values of 10^{-6} g m^{-2} day^{-1} or less within a measurement period of a day, although it is equally suited to perform measurement on film materials having a VTR above 10^{-6} g m^{-2} day^{-1}. From equation (1) and (2), it is evident that a higher VTR value will shorten the time required between subsequent pressure measurements.

Furthermore, the ratio R does not imply that the surface area A is restricted to microscopic or nanoscopic proportions. The surface area A can in principle be of any

size, as long as the characteristic size of the transverse chamber dimension remains sufficiently small.

Film

As shown in FIG. 1, a thickness of the film 112 is considerably larger than the characteristic depth of the chamber 106 of the VTR measurement device 102. The film 112 may comprise polymer materials. Among permeable materials that are suitable films 112 for the claimed measurement method and device are polyethylene naphthalate (PEN) and polyethylene terephthalate (PET). A thickness of a polymer based film 112 may be in the order of 50 μm or larger, typically 100 μm. Furthermore, the film 112 may comprise a vapor diffusion barrier, substantially reducing the permeability of the film to certain vapor particles. Such a vapor diffusion barrier may for example comprise metal oxide, metal nitride, metal oxynitride, metalloid oxide, or metalloid nitride. Examples of barrier materials are MgO, SiO_2 and Si_xN_y. The thickness of a vapor diffusion barrier is commonly in a range of 1 – 100 nanometers.

In order to prevent deformation of the film 112, the surface area A of the chamber opening 114 being spanned by the film 112 may have dimensions comparable to the thickness of the film 112 (i.e. in the order of 100 μm), or smaller. Together with the elastic properties (moduli) of the film, the properties of the film 112 will then be such that the deformation of the film will be negligible, therefore preserving a constant chamber volume V.

Alternatively, the dimensions of the surface area A may be significantly larger than the film thickness, in case deformation may be expected. A calibration or correction for expected changes in the chamber volume V may generally be possible for such cases. Here, it is required that the deformation of the film 112 does not hamper a pressure measurement, for example by obstructing the electronic and/or mechanical components of the pressure sensor 110 inside the chamber 106. The expected deflection of the film 112 spanned on a circular opening 114 may for example be predicted by methods described in document [1].

The lateral extent of the chamber 106 is only relevant in view of the deformation of the film 112, for example caused by a pressure difference between the chamber 106 and the surroundings of the VTR measurement device 102. If such deformation is

undesirable, a chamber 106 having a relatively large volume surface area A and a relatively small depth may incorporate supporting structures for supporting the film 112, in order to prevent the deflection.

General method of VTR measurement

According to an aspect there is provided a method of determining the VTR of a film 112 comprising permeable sample material for a vapor 116 permeating this film 112. This method utilizes a vapor transmission rate measurement device 102, having a chamber 106 with a chamber volume V, a pressure sensor 110, the chamber 106 having an opening 114 with a surface area A that is spanned by the film 112, wherein a ratio R between the chamber volume V and the surface area A is smaller than 10^{-5} m, or preferably smaller than $2.5 \cdot 10^{-6}$ m. The vapor transmission rate measurement device 102 is arranged for determining an indication of the vapor transmission rate for the film 112 permeated by the vapor 116. In general, the method comprises obtaining a first pressure P_1 within the chamber volume V at a first time t_1 and a measurement of a second pressure P_2 within the volume V at a subsequent time t_2. An indication of the VTR is then derived from the first pressure P_1 and the second pressure P_2. Obtaining the first pressure P_1 may be the result of an initial pressure measurement by the pressure sensor 110. Alternatively, the first pressure value P_1 may already be known in advance, for example as an initial condition resulting from the manufacturing of the VTR device 102. In the latter case, only the remaining unknown second pressure P_2 is to be measured by the pressure sensor 110.

According to an embodiment, the indication of the VTR is based on equation (2). Furthermore, the temperature T, the chamber volume V, the surface area A and the molar mass M are assumed to be known. The temperature T, which is assumed to be approximately constant between t_1 and t_2, is preferred to have a value in a range in which the material stability of the film 112 is secured. For a film comprising polymers, the temperature is typically preferred to be kept below 100° C.

Many types of microscopic or nanoscopic scaled pressure sensors may be used, some of which will be discussed below.

MEM/NEM resonator based sensor

According to an embodiment, the vapor transmission rate measurement device 102 has a pressure sensor 110 that comprises a MEM or NEM resonator 202. The term microelectromechanical system (MEMS) is used to refer to electromechanical systems that have typical dimensions in the range of 1 to 1000 micrometer. The term nanoelectromechanical system (NEMS) refers to systems that have typical dimensions in the range of 1 to 1000 nanometers. These typical dimensions relate to the sizes of the functional elements of the MEMS/NEMS, i.e. the mechanically deformable elements and/or the electric components that induce or sense such deformations. It generally does not refer to the electrical or mechanical power sources and/or further sensing, processing and control components coupled to the MEM or NEM system, although such components may be integrated with it.

In the embodiment shown in FIG.'s 2A - 2C, the vapor transmission rate measurement device 102 has a pressure sensor 110 comprising a MEM or NEM resonator 202. The MEM/NEM resonator 202 may have a deformable body as resonator element 204 that is able to vibrate in a resonator cavity 206. This resonator cavity 206 may be partially coinciding or be integrally formed by the chamber 106 of the VTR measurement device 102. The resonator element 204 is suspended within the resonator cavity 206 from a resonator frame 208, the resonator element 204 being mechanically connected at one or several locations to this resonator frame 210. The chamber 106 may have an opening 114 with a surface area A, which is arranged to be spanned by the film 112 of permeable sample material.

The MEM/NEM resonator 202 may be affixed to a substrate 208. This substrate 208 may have electrically and/or thermally insulating properties and may be structurally rigid or be mechanically flexible. The substrate 208 may for instance be organized as a silicon-on-insulator (SOI) structure. An additional electrically insulating layer 218 may be present between the resonator frame 210 and the substrate 208. This insulating layer 218 may for example essentially consist of silicon dioxide (SiO_2) or silicon nitride (SiN). In FIG.'s 2A and 2B, it is shown that the film 112 is applied on the opening 114 that is located on the top side of the MEM/NEM resonator 202 facing away from the substrate 208.

In the example of FIG. 2A – 2C it is further shown that the resonator element 204 is a doubly clamped beam that is allowed to substantially deform in a direction parallel to the substrate 208, although many other resonator configurations are conceivable. In general, the resonator element 204 may be an arbitrarily shaped patch of elastically deformable material, being attached at one or more points to the resonator frame 208. Also, the resonator element 204 may be designed for alternative modes of mechanical resonance than the in-plane transversal deflection depicted by the arrows in FIG. 2, examples being given by longitudinal or torsional resonators. Furthermore, a supporting frame 224 may be present in between the resonator frame 210 and the film 112. The presence of the supporting frame 224 will increase the amount of space between the film 112 and the resonator element 204, reducing friction and the chance of collision during mechanical deformation of the resonator element 204. Furthermore, FIG.'s 2A and 2B illustrate that the presence of the supporting frame 224 as well as the non-elementary shape of the resonator cavity 206 result in an increased surface area A compared to the chamber volume V, yielding in a beneficial decrease in the ratio R.

For a MEM resonator 202, the length L of the resonator element 204 corresponding to a long axis may typically be in the order of 1 - 100 μm. The width W of the resonator element 204 perpendicular to the long axis may be 0.2 – 10 μm. A typical thickness Z of the resonator element 204 in a direction perpendicular to the substrate 208 is 1 – 2 μm, with a preferred gap S of 0.5 μm or larger between the resonator element 204 and the substrate 308. For a stationary resonator element 204, gaps G between the resonator element and the first electrode 212 or the third electrode 216 are present, which may have a size of about 0.3 μm.

The MEMS/NEMS resonator 202 comprises a resonance sensor for measuring the mechanical resonance frequency of the resonator element 204. The resonance sensor may for example incorporate an optical system with a light source aimed at the resonator element 204 and a photovoltaic detector for measuring the amount and location of light reflected by the resonator element 204. In general, a resonance frequency may be detected by various methods like laser interferometry, vibrometry or deflection, or piezoresistive, piezoelectric, capacitive of magnetomotive means.

The resonating motion of the resonator element 204 may be induced by a resonance inducing element or resonance actuator that is designed to drive the resonator element 204 into controlled periodic motion by supplying some or several forms of energy to the resonator element 204. For example, resonance may be generated by an electric or magnetic field source oscillating at the desired resonance frequency, by mechanical means of excitation, by thermal excitation or by thermal-mechanical noise.

In the example shown in FIG.'s 2A - 2C, the resonance actuator is designed to set the resonator element 204 into motion by generating an alternating electric field. Shown are a first electrode 212 and a second electrode 214, which together constitute the electrical field based resonance actuator. The second electrode 214 may comprise or be identical to the resonator element 204. The electric field generated by the resonance actuator induces a mechanical deflection of the resonator element 204, as indicated by the arrows in FIG. 2B. A third electrode 216 is provided to act in conjunction with the second electrode 214 as an electrical capacitance based resonance sensor, which is able to measure a change in an electrical capacity C (or equivalently a potential difference) between the second electrode 214 and the third electrode 216, due to the mechanical deformation of the resonator element 204.

If a harmonic stimulus is applied to the resonator element 204, the resulting oscillating capacity C can be measured using the capacitive based resonance sensor. The amplitude of the oscillating capacity can be studied as a function of the temporal frequency f generated by the resonance actuator, yielding a frequency response of the MEM/NEM resonator 202.

Therefore in this embodiment with a resonator based pressure sensor 110, the pressure P may be derived from a measurement of the resonator quality or Q-factor of the MEM/NEM resonator 202. This Q-factor characterizes the bandwidth of the resonators frequency response relative to its center frequency of oscillation or maximum resonance. A high Q indicates a low rate of energy dissipation relative to the energy represented by the oscillatory motion of the resonator element 204. The excitation method influences the method of determining the quality factor Q of the MEM or NEM resonator. The Q-factor is usually determined by measuring the frequency response of the resonator across a substantial frequency range and extracting

the full width half maximum and centre frequency values. The frequency response may be probed by supplying single frequency actuation signals in a frequency sweep and observing the oscillatory resonator response for each frequency. Alternatively, a broadband actuation signal may be supplied at once, followed by a frequency decomposition of the transient response of the resonator element 204.

As the Q-factor describes the energy dissipation of the resonator, it is dependent of the pressure P inside the resonator cavity 206. In particular, molecules inside the chamber 106 and resonator cavity 206 may extract mechanical energy from the coherent oscillatory motion of the resonator element 204, representing a damping mechanism for the resonator. A larger amount of molecules increases the damping, which reduces the Q-factor. The physics of the damping of the resonator usually exhibits different regimes when going from the high vacuum to atmospheric pressure, as will be understood by the skilled person. In the current application, the molecular regime is of particular importance, as it represents the pressure range in which the Q factor of the resonator is approximately inversely proportional to the pressure P in the resonator cavity 206.

The relation between the pressure P and the resonator quality Q may initially be determined by a calibration measurement in a controlled environment in which the pressure P is known from a distinct manometer. From such calibration measurements, the MEMS/NEMS resonator has been found to be sensitive to pressure differences of 100 Pa or even less.

The graph of FIG. 6 illustrates a Q-factor versus pressure P sensitivity that can be achieved by an embodiment of a vapor transmission rate measurement device 102 with a clamped-clamped beam type MEM resonator as described with reference to FIG.'s 2A – 2C, having a typical chamber volume V of $2 \cdot 10^{-14}$ m^3, and a surface area A of about 10^{-8} m^2.

In a further embodiment, the VTR measurement device 102 with a MEM/NEM resonator 202 may have a resonator element 204 comprising a material that is chemically reactive with the vapor 116. Among candidates that are suitable for sensing oxygen and/or water permeation through the film 112 are alkali metals and alkaline earth metals. Water-reactive polymeric materials may also be used. For example, the resonator element 204 may be coated with calcium or sodium. A chemical reaction i.e.

corrosion of a calcium layer on the resonator element 204 with water molecules inside the resonator cavity 206, will alter the frequency response of the MEM/NEM resonator 202. This may involve a change in only the centre resonance frequency, a change in the frequency bandwidth, or a combination thereof. In general, a change in the resonator quality Q can be expected, which may be exploited to derive a VTR.

Pirani gauge based sensor

In another embodiment shown in FIG.'s 3A – 3C, the vapor transmission rate measurement device has a pressure sensor 110 comprising a micro electrical (ME) Pirani gauge 302. The Pirani gauge 302 operates on the principle that the pressure P is derived from a measurement of the electrical resistance R of the Pirani gauge 302, as will be understood by the skilled person. The Pirani gauge 302 shown in FIG.'s 3A – 3C comprises an elongated conducting bridge 304 and a gas chamber 306, which is surrounded by a frame 307 of laminar structures. The laminar structures of the frame 307 flanking the conducting bridge 304 may be designed to be efficient thermal conductors forming first and second heat sinks 310, 312. The frame 307 comprising the first and second heat sinks 310, 312 are provided on a substrate 308. A thermally insulating layer 316, may be provided in between the substrate 308 and the frame 307. Alternative or in addition to the presence of the first and/or second heat sinks 310, 312, the substrate 308 may have considerable thermal conductivity, representing a third heat sink.

The gas chamber 306 of the Pirani gauge 302 has an opening 114, which is arranged to be spanned by the film 112 of permeable sample material. In FIG.'s 3A and 3B, the film 112 is applied on the opening 114 that is located on the side of the Pirani gauge 302 facing away from the substrate 308. A supporting frame 318 may be provided in between the frame 307 and the film 112. This supporting frame 318 may increase the spatial separation and therefore thermal and electrical resistivity between the film 112 and the conducting bridge 304.

The gas chamber 306 of the ME or NE Pirani gauge 302 may be partially coinciding or be completely formed by the chamber 106 of the vapor transmission rate measurement device 102, with the conducting bridge 304 completely located inside the chamber 106.

A length L of the conducting bridge 304 along a longest axis may typically be in the order of 100 - 500 μm. The width W of the conducting bridge 304 perpendicular to the longest axis and parallel to the substrate 308 may be 1 – 5 μm. A typical thickness Z of the conducting bridge 304 in a direction perpendicular to the substrate 308 is 1 – 2 μm. A distance or gap S between the conducting bridge 304 and the substrate 308 may also be typically be 1 – 2 μm. In document [2] it is described that pressures of down to 10 Pa may be detected with a ME Pirani gauge 302 having a conducting bridge 304 with typical dimensions of L = 336 μm, W = 3 μm, Z = 1.4 μm and a gap S of 1 μm between the conducting bridge 304 and the substrate 308.

The Pirani gauge 302 operates on the principle that a current is driven through the conducting bridge 304, while a change in pressure P inside the gas chamber 306 will result in a change in heat transfer from the conducting bridge 304 to any present heat sinks 310 – 314. This in turn will result in a change of the local temperature and therefore the electrical resistance R of the conducting bridge 304. The electrical resistance R and its evolution in time are measurable by known methods.

In addition, the ME or NE Pirani gauge 302 may be designed to comprise materials that are chemically reactive to the vapor 116 that has traversed the film 112. In particular, the conducting bridge 304 may comprise chemically reactive material, whereby a chemical reaction between the vapor and conducting bridge 304 alters the electrical resistance R of the ME or NE Pirani gauge 302. For example, the conducting bridge 304 may be coated with an alkali metal or alkaline earth metal. The corrosion of a calcium layer applied to the Pirani bridge 304 due to water (oxygen) molecules that have permeated the film 112 and entered the gas chamber 306 during use of the VTR measurement device 102 will alter the electrical resistance R.

Further remarks

According to a further embodiment, the vapor transmission rate measurement device 102 comprises a pressure sensor 110 and a chamber 106 with a chamber volume V, the chamber 106 having an opening 114 with a surface area A, the opening 114 being arranged to be spanned by a film 112 of permeable sample material, wherein the vapor transmission rate measurement device 102 is arranged for determining an indication of the vapor transmission rate for the film 112 permeated by a vapor 116,

based on at least one measurement by the pressure sensor 110 of a pressure P within the chamber 106, and wherein the pressure sensor 110 comprises a MEM or NEM resonator 202. Here, the at least one measurement of a pressure P comprises a measurement of a resonator quality Q of the MEM or NEM resonator 202.

Alternatively, the vapor transmission rate measurement device 102 comprises a pressure sensor 110 and a chamber 106 with a chamber volume V, the chamber 106 having an opening 114 with a surface area A, the opening 114 being arranged to be spanned by a film 112 of permeable sample material, wherein the vapor transmission rate measurement device 102 is arranged for determining an indication of the vapor transmission rate for the film 112 permeated by a vapor 116, based on at least one measurement by the pressure sensor 110 of a pressure P within the chamber 106, and wherein the pressure sensor 110 comprises a ME or NE Pirani gauge 302. Here, the at least one measurement of a pressure P comprises a measurement of an electrical resistance R of the ME or NE Pirani gauge 302.

Vapor transmission rate measurement system

According to another aspect, there is provided a vapor transmission rate measurement system 402, having a control system 404 with an electronic processor 406, wherein the vapor transmission rate measurement system 402 further comprises at least one VTR measurement device 102 as described above. The control system 404 is arranged to communicate with the at least one VTR measurement device 102, and is also arranged to derive an indication of the VTR based on measurements by the at least one vapor transmission rate measurement device 102. Furthermore, the control system 404 may be able to record and/or further process measurement values.

Shown in FIG. 4 is an embodiment of a VTR measurement system 402 having a first VTR measurement device 408, a second VTR measurement device 410 and the control system 404. A portion of the control system 404 may for instance be formed by a computer arrangement (not shown). The computer arrangement may comprise a processor unit for performing arithmetical operations. The processor unit is connected to a memory unit that may store instructions and data, the memory unit may for instance be one or more of a tape unit, a hard disk, a Read Only Memory (ROM), an

Electrically Erasable Programmable Read Only Memory (EEPROM) and a Random Access Memory (RAM). The processor unit is also connected to one or more input devices, such as a keyboard and a mouse, one or more output devices, such as a display and a printer and one or more reading units to read data carriers, such as a DVD. The computer arrangement may also comprise an input output (I/O) device that is arranged to communicate with other computer systems via a communication network. However, it should be understood that there may be provided more and/or other memory units, input devices and read devices known to persons skilled in the art. Moreover, one or more of them may be physically located remote from the processor unit, if required. The memory unit may comprise instruction lines that are readable and executable by the processor unit to provide it with the functionality according to the embodiments of the method of measuring the VTR. The processor unit may comprise several processing units functioning in parallel or controlled by one main processor unit that may be located remote from one another, as is known to persons skilled in the art. All or any of the connections may be physical connections or be made wireless, the general purpose being the ability of the computer arrangement units to communicate with one another in some way.

According to embodiments of the method of determining a vapor transmission rate, the pressure sensor 110 comprises a MEM or NEM resonator 202 or a ME or NE Pirani gauge 302. The control system 404, and in particular the computer arrangement, may be provided with reference data representing pressure P versus the resonator quality Q information of a MEM or NEM resonator 202 and/or representing pressure P versus the electrical resistance R information of a ME or NE Pirani gauge 302. This Q versus P and/or R versus P relations may have been obtained by calibration phases preceding an execution method of determining the VTR.

In the embodiment shown in FIG. 4, the first VTR measurement device 408 comprises a MEM/NEM resonator 202, and the second VTR measurement device 410 comprises a Pirani gauge 302, both shown in perspective view. In general, any number and/or type of VTR measurement devices 102 may be present in a VTR measurement system 402.

For a VTR measurement system 402 comprising a MEM/NEM resonator 202, the resonator quality Q required for deriving a pressure P, may for instance be determined by electrical capacity or voltage measurement methods. For this purpose, the control system 404 may comprise a voltage source 412 which is arranged to generate an input potential difference V_{in} between the first and second electrodes 212, 214 if connected. Voltage source 412, with first and second electrodes 212, 214 together act as a resonance actuator. The voltage source 412 may be controllable, for example by the electronic processor 406 or another part of the control system 404, and may have a function generator that is able to output excitation signals that are harmonic, comprise many frequency components, have a pulsed character or even consist of broad band noise. The control system 404 may further comprise a first voltmeter 416 which is arranged to measure a first electrical potential difference $V_{out,1}$ between the second and third electrodes 214, 216, if connected. By measuring the potential difference $V_{out,1}$ as a function of the temporal frequency f of the potential difference V_{in} generated by the resonance actuator, the frequency response of the MEM/NEM resonator 202 may be determined.

For a VTR measurement system 402 comprising a Pirani gauge 302, the electrical resistance R of the Pirani gauge 302 may for instance be determined by a four point probe measurement. In FIG. 4 it is shown that the control system 404 may further comprise a current source 414, which is arranged to apply a current I_{in} across the conducting bridge 304 of the Pirani gauge 302. The current source 414 may be adjustable, for example being controllable by the electronic processor 406 or another part of the control system 404. The small size of the Pirani gauge 302 results in low power consumption, typically in the range of 5 – 100 μW.

In addition, the control system 404 may comprise a second voltmeter 418, which is arranged to measure a second electrical potential difference $V_{out,2}$ across the conducting bridge 304. The resistance R of the conducting bridge 304 is then determined by $V_{out,2} / I_{in}$, and may be monitored in time.

The electrical connections between the control system 404 and the first or second VTR measurement devices 408, 410 are only schematically represented in FIG. 4 as passing through the film 112. Actual connections may be realized by common means, like electrical contacting regions (e.g. electrode pads) that are located remote

from the actual VTR measurement devices 408, 410, or that are protruding from side or bottom faces of the VTR measurement devices 408, 410.

For a determination of the VTR according to equation 2, a value for the temperature T is required. For obtaining this value, a temperature sensor may be provided in or near any or all of the VTR measurement devices 102, and which may be read out by the control system 404. In an envisioned application of the proposed VTR measurement devices or system, a determination of the VTR for a permeable film 112 is performed at or near room temperature. For a VTR measurement system comprising a MEM/NEM resonator 202, a temperature change within the activated MEN/NEM resonator 202 is expected to be negligible. For a VTR measurement system with at least one Pirani gauge 302, the temperature increase within the activated Pirani gauge due to local heating of the conducting bridge 304 is expected to be small compared to the reference temperature of about 293 K.

According to an embodiment, a vapor transmission rate measurement system 402, has at least two vapor transmission rate measurement devices 102 with non-overlapping chambers 106. The at least two VTR measurement devices 102 may be arranged to individually measure indications of a vapor transmission rate. This enables the measurement of possible variations of the VTR across the surface of the film 112, for example due to the presence of material defects within the film 112, the spatial distribution of which may require a statistical description.

Furthermore, at least one of the at least two vapor transmission rate measurement devices 102 in the vapor transmission rate measurement system 402 may be arranged to measure a first vapor transmission rate corresponding to a first vapor, and at least one of the remaining vapor transmission rate measurement devices may be arranged to measure a second vapor transmission rate corresponding to a second vapor that is different from the first vapor. This circumvents the need for separating the total pressure buildup in a single chamber 106 due to the permeation of a vapor 116 consisting of distinct molecular components into partial pressure contributions from the individual components. For this purpose, the film 112 may locally comprise additional vapor diffusion barriers, each spanning the opening 114 of a chamber 106 of a

particular VTR measurement device 102. Such an additional diffusion barrier is arranged to only allow the vapor component of which the VTR is to be measured to permeate the film 112 and enter the respective chamber 106.

Electronic device with VTR system

A VTR measurement system 402 as described above may be incorporated within an electronic device 502. The electronic device 502 may comprise one or more vapor transmission rate measurement devices 102, these VTR measurement devices 102 being arranged to derive an indication of the expected lifetime of the electronic device 502 during use by monitoring the VTR. In addition, subsequent VTR measurements may be integrated to estimate the age of the electronic device 502. Such an electronic device 502 is schematically shown in FIG. 5. The electronic device 502 may for example be a solar collector panel or a flexible electronic device, like a display. In the latter case, the characteristic dimensions of any of the at least one VTR measurement device 102 being part of the VTR measurement system 402 may preferably be well below the expected flexural curvatures of the electronic device 502. For instance, a flexible display device with expected characteristic curvatures of centimeters may incorporate VTR measurement devices 102 with characteristic sizes in the order of millimeters or smaller.

The descriptions above are intended to be illustrative, not limiting. It will be apparent to the person skilled in the art that alternative and equivalent embodiments of the invention can be conceived and reduced to practice, without departing from the scope of the claims set out below.

REFERENCES

[1] S. Timoshenko and S. Woinosky-Krieger, "Theory of Plates and Shells", McGraw Hill Classic Textbook Reissue (1987)

[2] Q. Li, J.F.L. Goosen, J.T.M. van Beek, F. van Keulen, "A novel SOI Pirani sensor with triple heat sinks", proc. Chem. 1(2009), pp 160–163 (Proc Eurosensors XXIII)

LIST OF FIGURE ELEMENT

102	vapor transmission rate measurement device
104	cell
106	chamber
108	housing
110	pressure sensor
112	film
114	opening
116	vapor
V	chamber volume
A	surface area
R	ratio

202	MEM/NEM resonator
204	resonator element
206	resonator cavity
208	substrate
210	resonator frame
212	first electrode
214	second electrode
216	third electrode
218	resonance sensor
220	resonance actuator
222	electrically insulating layer
224	supporting frame

302	Pirani gauge
304	conducting bridge
306	gas chamber
308	substrate
310	first heat sink
312	second heat sink

314 third heat sink
316 insulating layer
318 supporting frame

402 vapor transmission rate measurement system
404 control system
406 electronic processor
408 first vapor transmission rate measurement device
410 second vapor transmission rate measurement device
412 first voltage source
414 current source
416 first voltmeter
418 second voltmeter
420 temperature sensor
422 heater element
424 temperature controller

502 electronic device

CONCLUSIES

1. Dampdoorlaatsnelheidmeetinrichting (102), omvattende een druksensor (110) en een kamer (106) met een kamervolume V, waarbij de kamer (106) een opening (114) heeft met een oppervlakte A, waarbij de opening (114) is ingericht om te worden overspannen door een vlies (112) van doordringbaar monstermateriaal, waarbij de dampdoorlaatsnelheidmeetinrichting (102) is ingericht voor het bepalen van een indicatie van de dampdoorlaatsnelheid voor het vlies (112) welke wordt doordrongen door een damp (116), gebaseerd op ten minste een meting door de druksensor (110) van een druk P binnen de kamer (106), **met het kenmerk dat**
een verhouding R tussen het kamervolume V en de oppervlakte A kleiner is dan 10^{-5} m.

2. Dampdoorlaatsnelheidmeetinrichting (102) volgens conclusie 1, waarbij de verhouding R kleiner is dan $2.5 \cdot 10^{-6}$ m.

3. Dampdoorlaatsnelheidmeetinrichting (102) volgens conclusie 1 of 2, waarbij de druksensor (110) een micro-elektromechanische MEM of nano-elektromechanische NEM resonator (202) omvat, en waarbij de ten minste een meting van een druk P een meting van een resonatorkwaliteit Q van de MEM of NEM resonator (202) omvat.

4. Dampdoorlaatsnelheidmeetinrichting (102) volgens conclusie 3, waarbij de MEM of NEM resonator (202) een resonatorelement (204) heeft dat gelegen is binnen de kamer (106).

5. Dampdoorlaatsnelheidmeetinrichting (102) volgens conclusie 3 of 4, waarbij het resonatorelement (204) een materiaal omvat dat chemisch reactief is met de damp (116), waarbij een chemische reactie tussen de damp en het resonatorelement (204) een frequentieresponsie van de MEM of NEM resonator (202) wijzigt.

6. Dampdoorlaatsnelheidmeetinrichting (102) volgens conclusie 1 of 2, waarbij de druksensor (110) een micro-elektronische ME of nano-elektronische NE Pirani meter (302) omvat, en waarin de ten minste een meting van een druk P een meting van een elektrische weerstand R van de ME of NE Pirani meter (302) omvat.

7. Dampdoorlaatsnelheidmeetinrichting (102) volgens conclusie 6, waarbij de ME of NE Pirani meter (302) een geleidende brug (304) heeft die gelegen is binnen de kamer (106).

8. Dampdoorlaatsnelheidmeetinrichting (102) volgens conclusie 6 of 7, waarbij de geleidende brug (304) materiaal omvat dat chemisch reactief is met de damp (116), waarbij een chemische reactie tussen de damp en de geleidende brug (304) de elektrische weerstand R van de ME of NE Pirani meter (302) wijzigt.

9. Dampdoorlaatsnelheidmeetsysteem (402), omvattende een besturingssysteem (404) met een elektronische processor (406),
met het kenmerk dat
het dampdoorlaatsnelheidmeetsysteem (402) verder ten minste een dampdoorlaatsnelheidmeetinrichting (102) volgens een van de conclusies 1 – 8 omvat, waarbij het besturingssysteem (404) is ingericht om te communiceren met de ten minste een dampdoorlaatsnelheidmeetinrichting (102), en om een indicatie van de dampdoorlaatsnelheid af te leiden op grond van metingen door de ten minste een dampdoorlaatsnelheidmeetinrichting (102).

10. Dampdoorlaatsnelheidmeetsysteem (402) volgens conclusie 9, voor zover afhankelijk van een van de conclusies 3 – 8, waarbij het besturingssysteem (404) is voorzien van referentiedata die een relatie tussen de druk P en de resonatorkwaliteit Q voor de MEM of NEM resonator (202) of tussen de druk P en de elektrische weerstand R voor de ME of NE Pirani meter (302) voorstellen.

11. Dampdoorlaatsnelheidmeetsysteem (402) volgens conclusie 9 of 10, omvattende ten minste twee dampdoorlaatsnelheidmeetinrichtingen (102) volgens een van de

conclusies 1 – 8, waarbij de ten minste twee dampdoorlaatsnelheidmeetinrichtingen (102) niet samenvallende kamers (106) hebben en zijn ingericht voor het individueel meten van indicaties van dampdoorlaatsnelheden.

12. Dampdoorlaatsnelheidmeetsysteem (102) volgens conclusie 11, waarbij een eerste dampdoorlaatsnelheidmeetinrichting (102) is ingericht voor het meten van een eerste dampdoorlaatsnelheid corresponderende met een eerste damp, en een tweede dampdoorlaatsnelheidmeetinrichting (102) is ingericht voor het meten van een tweede dampdoorlaatsnelheid corresponderende met een tweede damp die verschilt van de eerste damp.

13. Elektronische inrichting (502), omvattende ten minste een dampdoorlaatsnelheidmeetinrichting (102) volgens een van de conclusies 1 – 8, waarbij de ten minste een dampdoorlaatsnelheidmeetinrichting (102) is ingericht voor het afleiden van een indicatie van de verwachte levensduur van de elektronische inrichting (502) tijdens gebruik.

14. Werkwijze voor het bepalen van een dampdoorlaatsnelheid voor een vlies (112) met doordringbaar monstermateriaal, welke wordt doordrongen door een damp (116), de werkwijze omvattende:
- het verkrijgen van een eerste druk P_1 binnen een kamervolume V op een eerste tijdstip t_1;
- het meten van een tweede druk P_2 binnen het kamervolume V op een later tijdstip t_2;
- het afleiden van een indicatie van de dampdoorlaatsnelheid uit de eerste druk P_1 en de tweede druk P_2;
gekenmerkt door
- het voorzien van een dampdoorlaatsnelheidmeetinrichting (102) met een druksensor (110) en een kamer (106) met het kamervolume V, waarbij de kamer (106) een opening (114) heeft met een oppervlak A dat is overspannen door het vlies (112), waarbij een verhouding R tussen het kamervolume V en het oppervlak A kleiner is dan 10^{-5} m.

15. Werkwijze voor het bepalen van een dampdoorlaatsnelheid volgens conclusie 14, waarbij het afleiden van een indicatie van de dampdoorlaatsnelheid VTR is gebaseerd op de vergelijking

$$VTR(t_1, t_2) = \frac{M(P_2 - P_1)V}{Ak_B T(t_2 - t_1)}$$

waarbij k_B de Boltzmann constante, M de moleculaire massa en T de temperatuur in Kelvin voorstelt.

Fig 1

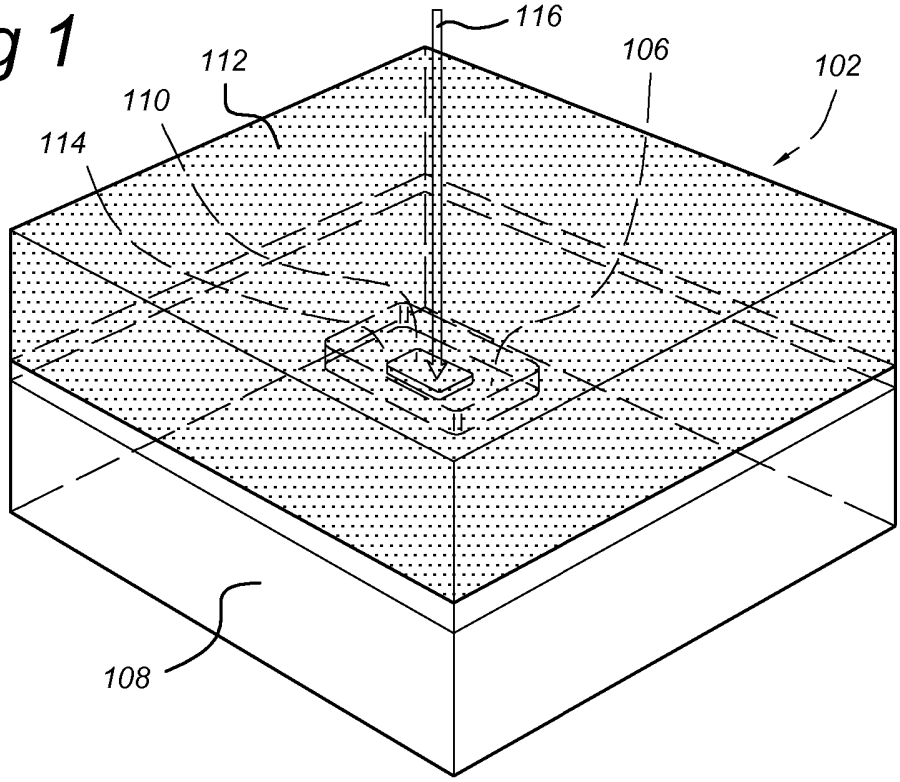

116
112
106
102
110
114
108

Fig 2a

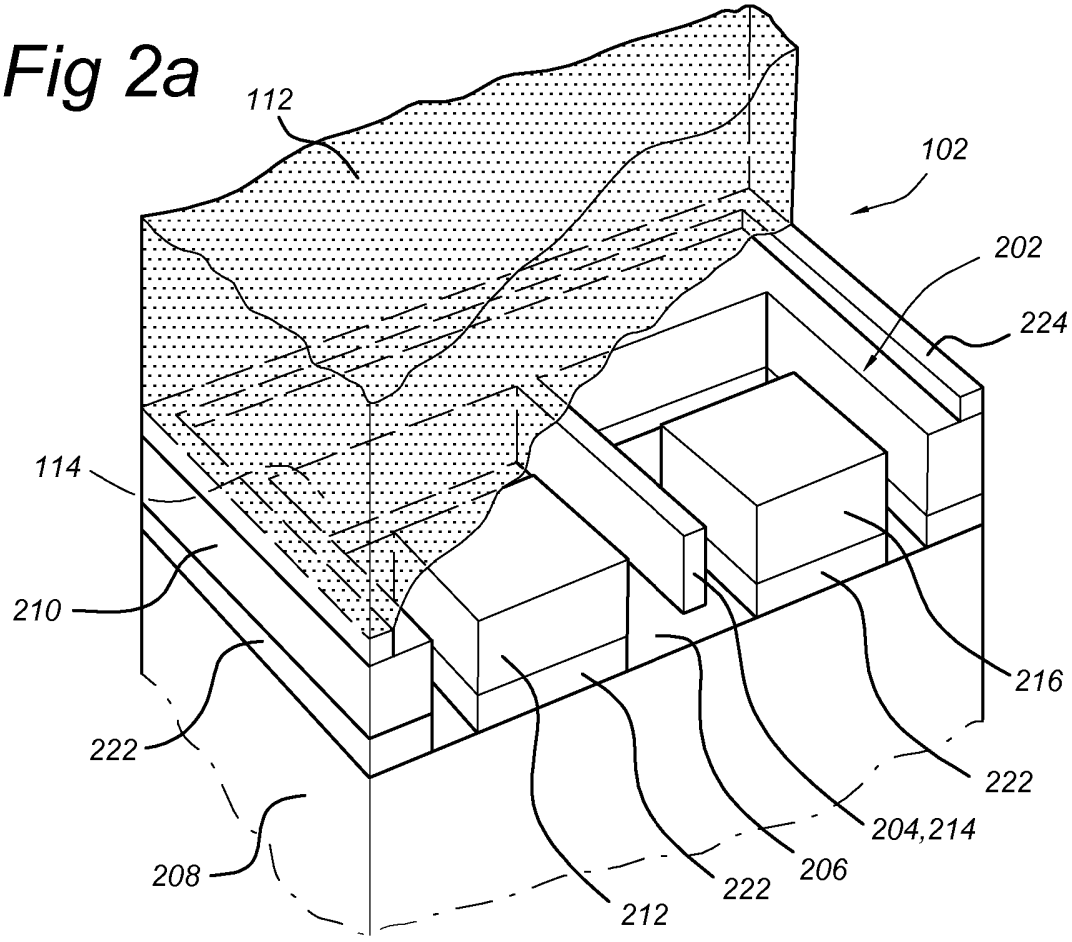

112
102
202
224
114
210
222
216
222
208
204,214
206
222
212

Fig 2b

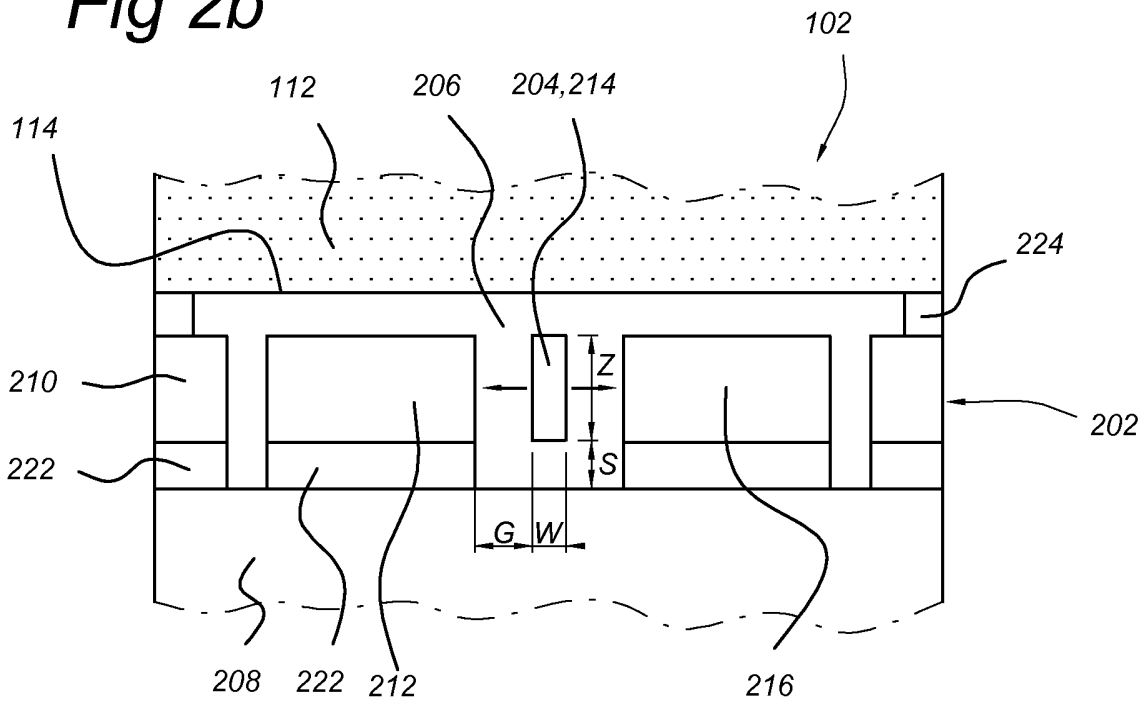

102
112
206
204,214
114
224
210
Z
202
222
S
G
W
208
222
212
216

Fig 2c

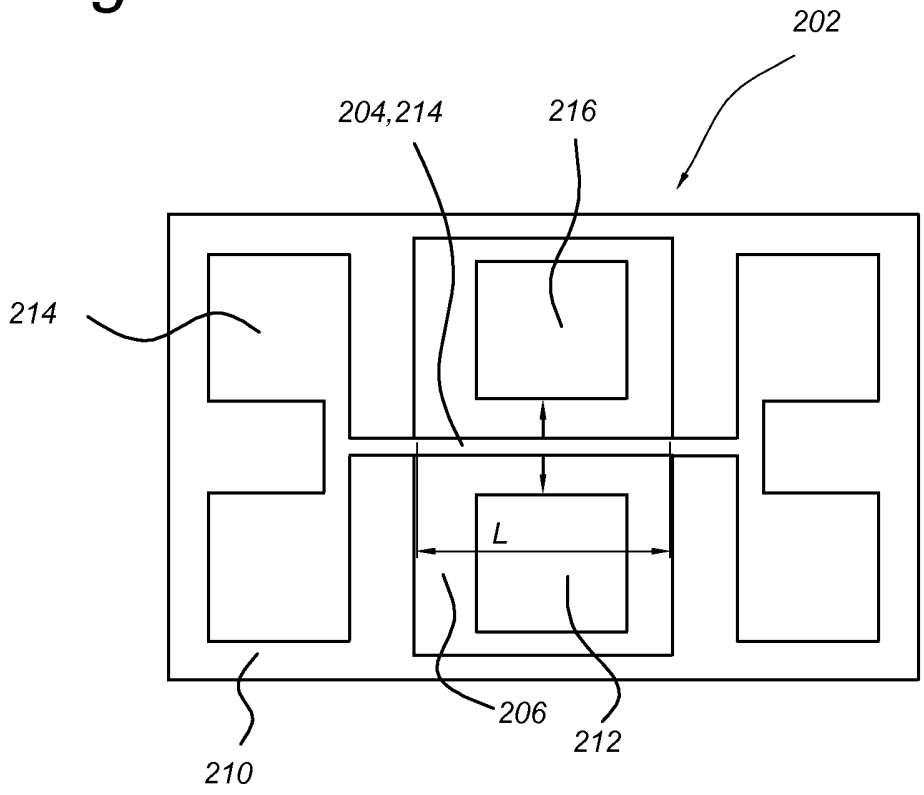

202
204,214
216
214
L
206
212
210

Fig 3a

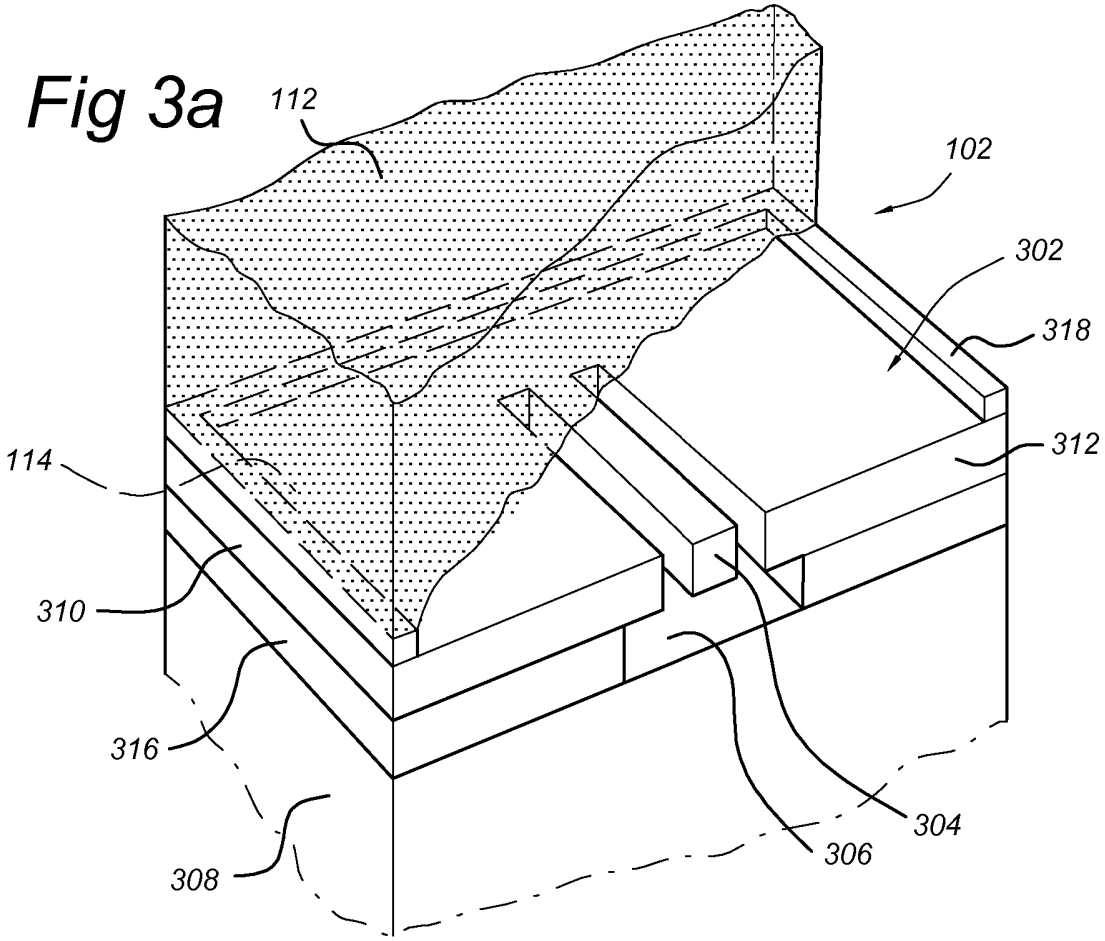

112
102
302
318
312
114
310
316
304
306
308

Fig 3b

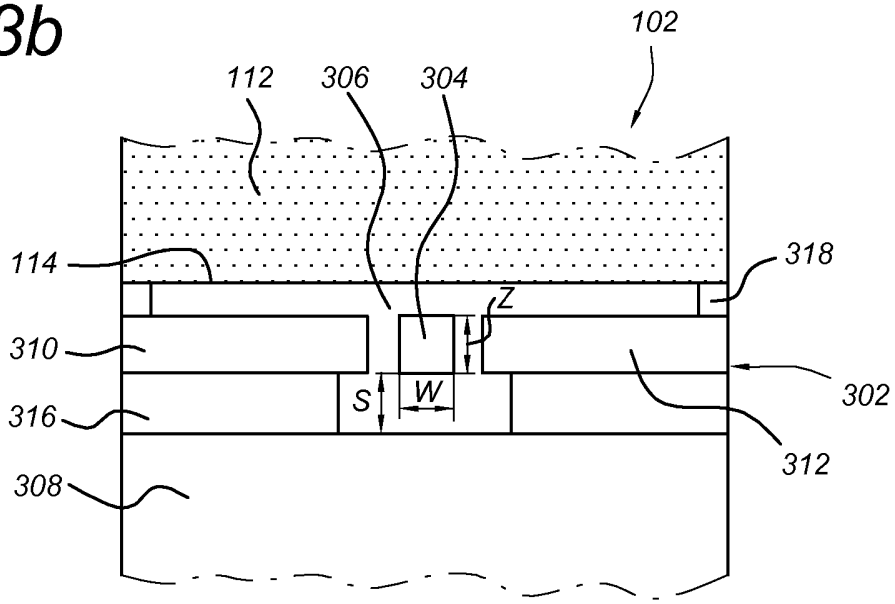

102
112
306
304
114
318
Z
310
302
S
W
316
312
308

Fig 3c

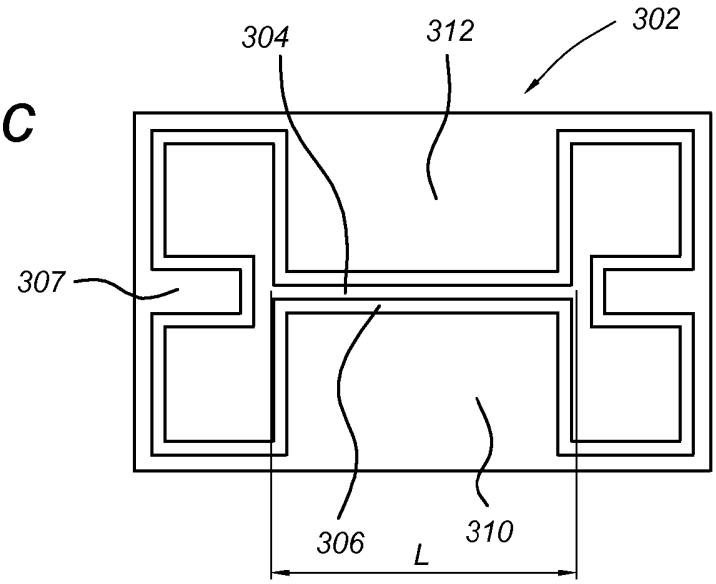

304
312
302
307
306
L
310

Fig 4

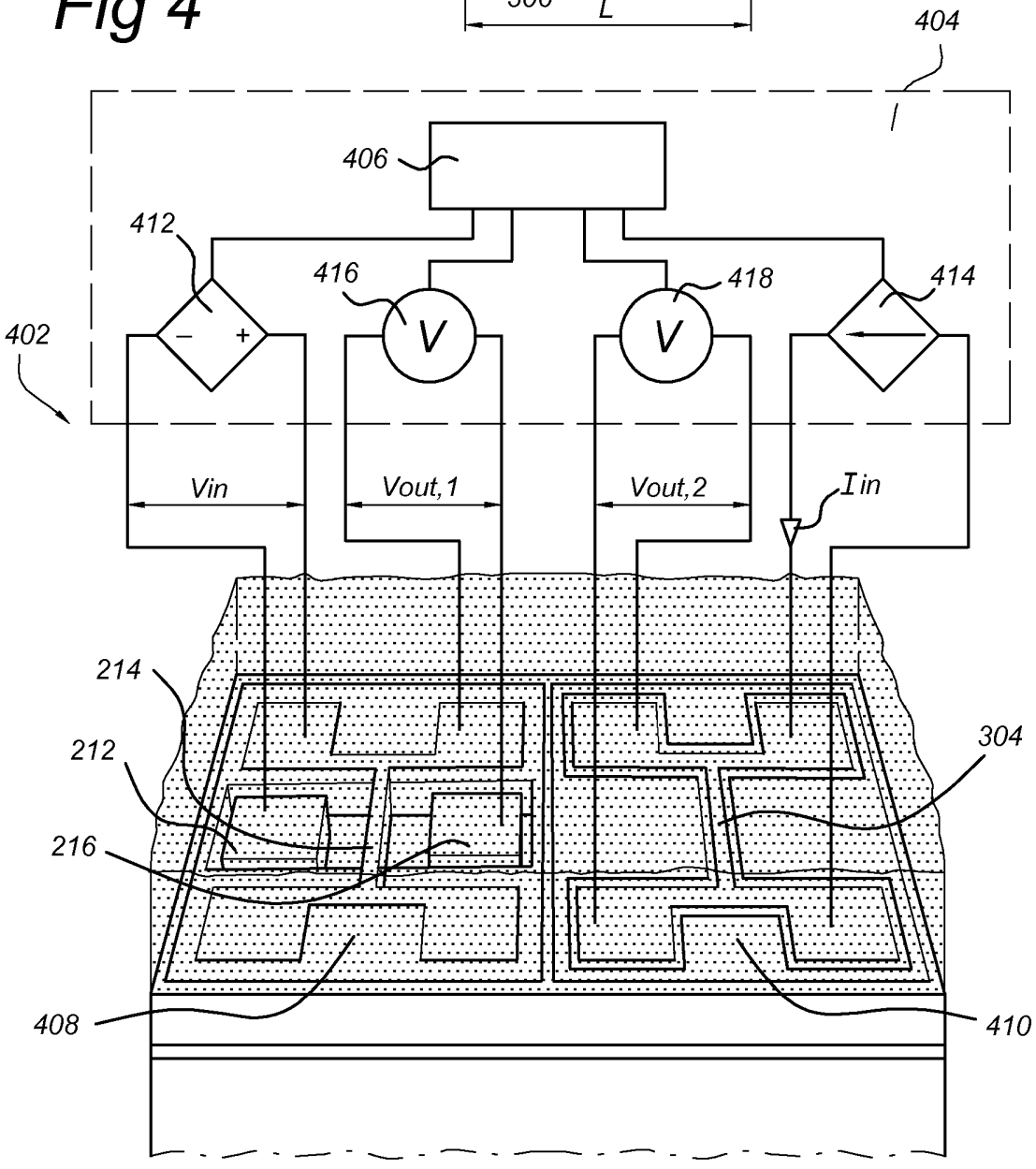

404
406
412
416
418
414
402
V
V
Vin
Vout,1
Vout,2
Iin
214
212
216
304
408
410

Fig 5

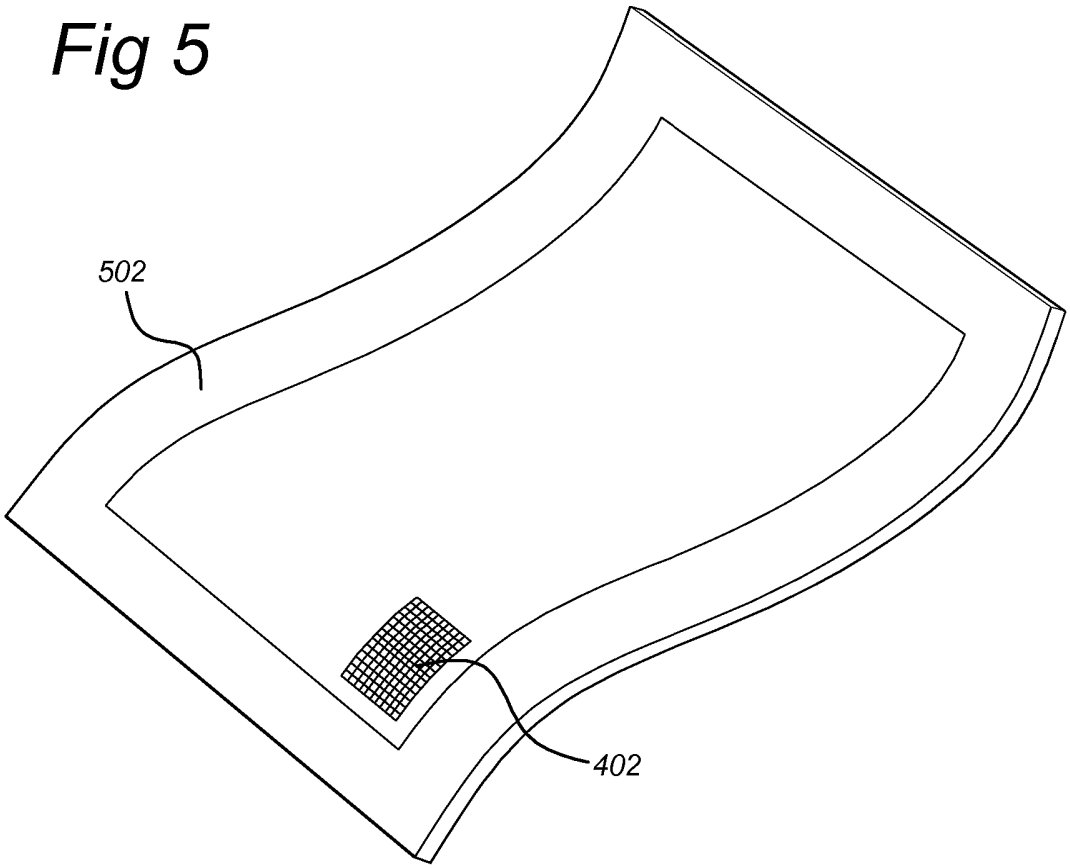
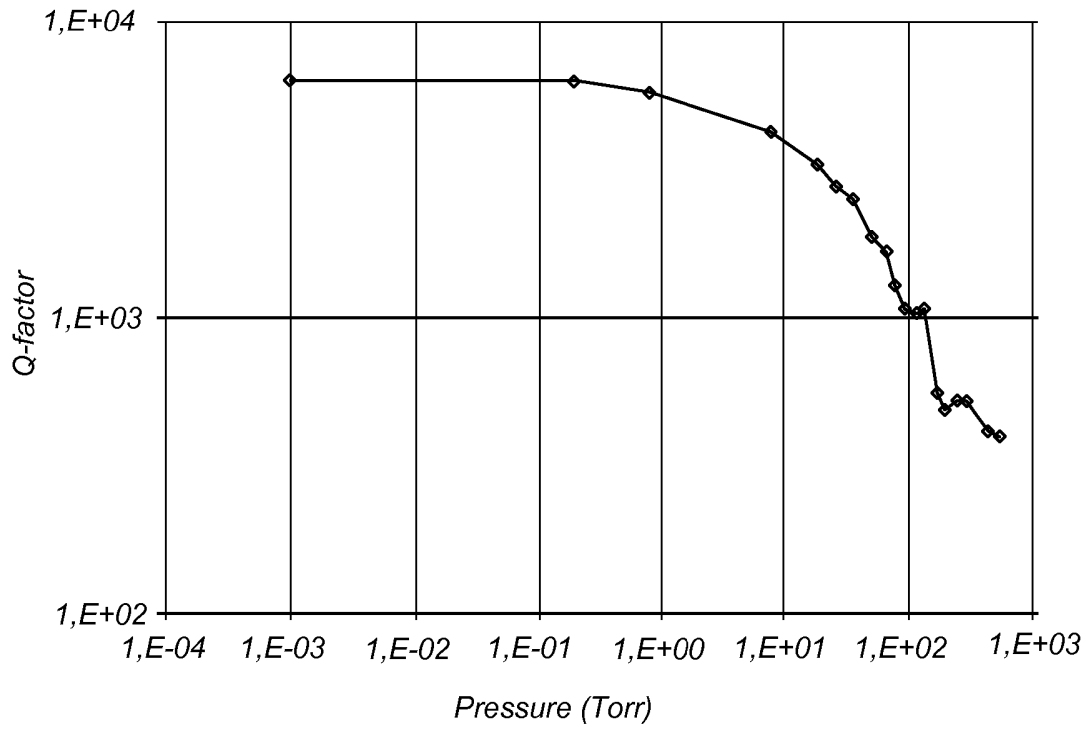

502
402

Fig 6


1,E+04
1,E+03
1,E+02
Q-factor
1,E-04
1,E-03
1,E-02
1,E-01
1,E+00
1,E+01
1,E+02
1,E+03
Pressure (Torr)

SAMENWERKINGSVERDRAG (PCT)

RAPPORT BETREFFENDE NIEUWHEIDSONDERZOEK VAN INTERNATIONAAL TYPE

IDENTIFICATIE VAN DE NATIONALE AANVRAGE	KENMERK VAN DE AANVRAGER OF VAN DE GEMACHTIGDE
	P6025905NL
Nederlands aanvraag nr. **2004419**	Indieningsdatum **17-03-2010**
	Ingeroepen voorrangsdatum
Aanvrager (Naam) **St.Materials innovation institute (M2i**	
Datum van het verzoek voor een onderzoek van internationaal type **12-06-2010**	Door de Instantie voor Internationaal Onderzoek aan het verzoek voor een onderzoek van internationaal type toegekend nr. **SN 54332**

I. CLASSIFICATIE VAN HET ONDERWERP (bij toepassing van verschillende classificaties, alle classificatiesymbolen opgeven)

Volgens de internationale classificatie (IPC)

G01N15/08

II. ONDERZOCHTE GEBIEDEN VAN DE TECHNIEK

Onderzochte minimumdocumentatie

Classificatiesysteem	Classificatiesymbolen	
IPC 8	**G01N**	**G01L**

Onderzochte andere documentatie dan de minimum documentatie, voor zover dergelijke documenten in de onderzochte gebieden zijn opgenomen

III. ☐ **GEEN ONDERZOEK MOGELIJK VOOR BEPAALDE CONCLUSIES** (opmerkingen op aanvullingsblad)

IV. ☐ ***GEBREK AAN EENHEID VAN UITVINDING*** *(opmerkingen op aanvullingsblad)*

ONDERZOEKSRAPPORT BETREFFENDE HET RESULTAAT VAN HET ONDERZOEK NAAR DE STAND VAN DE TECHNIEK VAN HET INTERNATIONALE TYPE

Nummer van het verzoek om een onderzoek naar de stand van de techniek
NL 2004419

A. CLASSIFICATIE VAN HET ONDERWERP
INV. G01N15/08
ADD.

Volgens de Internationale Classificatie van octrooien (IPC) of zowel volgens de nationale classificatie als volgens de IPC.

B. ONDERZOCHTE GEBIEDEN VAN DE TECHNIEK

Onderzochte miminum documentatie (classificatie gevolgd door classificatiesymbolen)
G01N G01L

Onderzochte andere documentatie dan de mimimum documentatie, voor dergelijke documenten, voor zover dergelijke documenten in de onderzochte gebieden zijn opgenomen

Tijdens het onderzoek geraadpleegde elektronische gegevensbestanden (naam van de gegevensbestanden en, waar uitvoerbaar, gebruikte trefwoorden)
EPO-Internal, WPI Data

C. VAN BELANG GEACHTE DOCUMENTEN

Categorie °	Geciteerde documenten, eventueel met aanduiding van speciaal van belang zijnde passages	Van belang voor conclusie nr.
A	SCHMIDT M ET AL: "Gas diffusion barrier layers for transparent polymer packages for optical MEMS" PROCEDIA CHEMISTRY, ELSEVIER, deel 1, nr. 1, 1 september 2009 (2009-09-01), bladzijden 1523-1526, XP026799847 ISSN: 1876-6196 [gevonden op 2009-09-01] * alinea [0003]; figuur 1a *	1-15
A	WO 2009/096504 A1 (RITSUMEIKAN TRUST [JP]; KIMATA MASAFUMI [JP]) 6 augustus 2009 (2009-08-06) * het gehele document *	1-15

-/--

[X] Verdere documenten worden vermeld in het vervolg van vak C.

[X] Leden van dezelfde octrooifamilie zijn vermeld in een bijlage

° Speciale categorieën van aangehaalde documenten

"A" niet tot de categorie X of Y behorende literatuur die de stand van de techniek beschrijft

"D" in de octrooiaanvrage vermeld

"E" eerdere octrooi(aanvrage), gepubliceerd op of na de indieningsdatum, waarin dezelfde uitvinding wordt beschreven

"L" om andere redenen vermelde literatuur

"O" niet-schriftelijke stand van de techniek

"P" tussen de voorrangsdatum en de indieningsdatum gepubliceerde literatuur

"T" na de indieningsdatum of de voorrangsdatum gepubliceerde literatuur die niet bezwarend is voor de octrooiaanvrage, maar wordt vermeld ter verheldering van de theorie of het principe dat ten grondslag ligt aan de uitvinding

"X" de conclusie wordt als niet nieuw of niet inventief beschouwd ten opzichte van deze literatuur

"Y" de conclusie wordt als niet inventief beschouwd ten opzichte van de combinatie van deze literatuur met andere geciteerde literatuur van dezelfde categorie, waarbij de combinatie voor de vakman voor de hand liggend wordt geacht

"&" lid van dezelfde octrooifamilie of overeenkomstige octrooipublicatie

Datum waarop het onderzoek naar de stand van de techniek van internationaal type werd voltooid	Verzenddatum van het rapport van het onderzoek naar de stand van de techniek van internationaal type
9 november 2010	

Naam en adres van de instantie	De bevoegde ambtenaar
European Patent Office, P.B. 5818 Patentlaan 2 NL – 2280 HV Rijswijk Tel. (+31-70) 340-2040, Fax: (+31-70) 340-3016	van Lith, Joris

1

ONDERZOEKSRAPPORT BETREFFENDE HET RESULTAAT VAN HET ONDERZOEK NAAR DE STAND VAN DE TECHNIEK VAN HET INTERNATIONALE TYPE

Nummer van het verzoek om een onderzoek naar de stand van de techniek
NL 2004419

C.(Vervolg). VAN BELANG GEACHTE DOCUMENTEN

Categorie °	Geciteerde documenten, eventueel met aanduiding van speciaal van belang zijnde passages	Van belang voor conclusie nr.
A	EP 0 429 397 A2 (ORBISPHERE LAB [CH]) 29 mei 1991 (1991-05-29) * bladzijde 5, regel 21 - bladzijde 6, regel 44 *	1
A	WO 98/03850 A1 (GETTERS SPA [IT]; MANINI PAOLO [IT]) 29 januari 1998 (1998-01-29) * samenvatting * * bladzijde 5, regels 25-26 *	1
A	US 2004/250625 A1 (KOGAN YAKOV [US] ET AL) 16 december 2004 (2004-12-16) * samenvatting *	3,4

1

ONDERZOEKSRAPPORT BETREFFENDE HET RESULTAAT VAN HET ONDERZOEK NAAR DE STAND VAN DE TECHNIEK VAN HET INTERNATIONALE TYPE

Informatie over leden van dezelfde octrooifamilie

Nummer van het verzoek om een onderzoek naar de stand van de techniek
NL 2004419

In het rapport genoemd octrooigeschrift	Datum van publicatie	Overeenkomend(e) geschrift(en)	Datum van publicatie
WO 2009096504 A1	06-08-2009	EP 2237009 A1	06-10-2010
EP 0429397 A2	29-05-1991	CH 679890 A5	30-04-1992
		DE 69031901 D1	12-02-1998
		DE 69031901 T2	06-08-1998
		JP 3000296 B2	17-01-2000
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		US 5144831 A	08-09-1992
WO 9803850 A1	29-01-1998	IT MI961489 A1	19-01-1998
US 2004250625 A1	16-12-2004	US 2007074574 A1	05-04-2007

OCTROOICENTRUM NEDERLAND

WRITTEN OPINION

File No.	Filing date *(day/month/year)*	Priority date *(day/month/year)*	Application No.
SN54332	17.03.2010		NL2004419

International Patent Classification (IPC)
INV. G01N15/08

Applicant
Stichting Materials innovation institute (M2i)

This opinion contains indications relating to the following items:

- ☒ Box No. I Basis of the opinion
- ☐ Box No. II Priority
- ☐ Box No. III Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
- ☐ Box No. IV Lack of unity of invention
- ☒ Box No. V Reasoned statement with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
- ☐ Box No. VI Certain documents cited
- ☐ Box No. VII Certain defects in the application
- ☐ Box No. VIII Certain observations on the application

	Examiner
	van Lith, Joris

Application number

WRITTEN OPINION

NL2004419

Box No. I Basis of this opinion

1. This opinion has been established on the basis of the latest set of claims filed before the start of the search.

2. With regard to any **nucleotide and/or amino acid sequence** disclosed in the application and necessary to the claimed invention, this opinion has been established on the basis of:

   a. type of material:

   ☐ a sequence listing

   ☐ table(s) related to the sequence listing

   b. format of material:

   ☐ on paper

   ☐ in electronic form

   c. time of filing/furnishing:

   ☐ contained in the application as filed.

   ☐ filed together with the application in electronic form.

   ☐ furnished subsequently for the purposes of search.

3. ☐ In addition, in the case that more than one version or copy of a sequence listing and/or table relating thereto has been filed or furnished, the required statements that the information in the subsequent or additional copies is identical to that in the application as filed or does not go beyond the application as filed, as appropriate, were furnished.

4. Additional comments:

Box No. V Reasoned statement with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty	Yes:	Claims	1-15
	No:	Claims	
Inventive step	Yes:	Claims	1-15
	No:	Claims	
Industrial applicability	Yes:	Claims	1-15
	No:	Claims	

2. Citations and explanations

see separate sheet

Reference is made to the following documents:

D1 SCHMIDT M ET AL: "Gas diffusion barrier layers for transparent polymer packages for optical MEMS" PROCEDIA CHEMISTRY, ELSEVIER, deel 1, nr. 1, 1 september 2009 (2009-09-01) , bladzijden 1523-1526, XP026799847 ISSN: 1876-6196 [gevonden op 2009-09-01]

D2 WO 2009/096504 A1 (RITSUMEIKAN TRUST [JP]; KIMATA MASAFUMI [JP]) 6 augustus 2009 (2009-08-06)

D3 EP 0 429 397 A2 (ORBISPHERE LAB [CH]) 29 mei 1991 (1991-05-29)

D4 WO 98/03850 A1 (GETTERS SPA [IT]; MANINI PAOLO [IT]) 29 januari 1998 (1998-01-29)

D5 US 2004/250625 A1 (KOGAN YAKOV [US] ET AL) 16 december 2004 (2004-12-16)

Document D2 has been assessed using the automated translation as provided by Thomson Scientific.

Re Item V

Reasoned statement with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1 For the purpose of assessing novelty and inventive step the following remarks apply:

1.1 The search examiner has carefully considered the clarity of Claim 1.

In first instance it appears as if claim 1 does not meet the requirement of clarity because it would appear that the passage "a ratio R between the chamber volume V and the surface area A is smaller than 10^{-5} m" attempts to further define the subject-matter of this claim by specifying a "result to be achieved". In view of the acknowledged prior art (description, page 1, line 25 - page 2, line 3) it is obvious to the skilled man that this ratio needs to be minimized in order to optimize the sensitivity of the vapor transmission rate measurement device. Therefore the statement that the ratio should be smaller

than 10^{-5} m would appear to merely amount to a statement of the underlying problem, without providing the technical features necessary for achieving this result.

The ratio however represents the average height of the measurement chamber, i.e. the distance between the opening of the chamber that is to be covered by the sample material and the opposite side of the measurement chamber. The ratio thus does not represent a vague parameter that should have a desired value, but it clearly describes a specific geometry of the measurement chamber. In addition, the small dimension of the average height implies the use of micro-technology, similar to that with which MEMS are created. These days micro-technology is well known and it would be clear to the skilled man how the achieve the desired ratio.

Claim 1 is therefore clear.

2 The subject matter of claim 1 meets the criteria of patentability.

2.1 The document D1 is considered to be the closest prior art to the subject matter of claim 1.

2.1.1 D1 discloses:

A device for evaluating the permeability of a film by measuring the pressure increase in a chamber, defined partially by that film (paragraph 3).

2.1.2 The subject matter of claim 1 differs from this known in that the ratio between the chamber volume and the surface area is smaller than 10^{-5}m.

The subject matter of claim 1 is thus novel.

2.1.3 The technical effect caused by this difference is an increased response time or a higher sensitivity (application, page 2, line 14-17)

2.1.4 The problem to be solved by the present invention may therefore be regarded as how to improve the response time or sensitivity.

2.1.5 It is well known in the field the response time or the sensitivity relate directly to the ratio between the chamber volume and the surface area. The skilled man would therefore attempt to reduce the volume of the chamber in D1. The materials, geometry, the bulging of the film under the pressure difference (see Figure 1(a) and the type of pressure sensor used, i.e. not a micro-sensor, do however not allow such a large reduction of the ratio.

To arrive at the subject matter of claim 1, the skilled man has to completely redesign the permeability measuring device of figure 1(a), i.e. to change the geometry, materials and sensor.

Neither D1 nor the cited documents hint at radically changing the design of similar permeability measuring devices fabricated with the conventional macro-technology, so as to allow a device fabricated using micro-technology.

Starting from D1 as the closest prior art the subject matter of claim 1 is thus inventive.

2.2 Document D2 can also be considered to represent the closets prior art to the subject matter of claim 1.

2.2.1 D2 discloses:

a micro vacuum Pirani gauge mounted in a package for a MEMS device for evaluating the quality of the vacuum in that package (paragraph "description prior art" and "disclosure of the invention").

The presence of an opening is implicit and it corresponds to the space occupied by the package, which usually consists of a film covering the chip on which the MEMS device is fabricated.

The fact that the gauge is a micro-gauge and intended to be used in a package for a MEMS device, implies the use of micro technology.

The device is in first instance not used to measure the vapor transmission rate of the package. It is only used to measure the quality of the current vacuum. It is however obvious that from the current vacuum, rough conclusions, e.g. "good" or "bad", can be drawn on the permeability of the package.

2.2.2 The subject matter of claim 1 differs from this known in that the ratio between the chamber volume and the surface area is smaller than 10^{-5}m.

The subject matter of claim 1 is thus novel.

2.2.3 The technical effect caused by this difference is an increased sensitivity to the permeability of the packaging, i.e. the pressure inside the package increases faster.

2.2.4 In view of the device in D2 the skilled man is however not interested in improving the sensitivity of the device to the permeability of the packaging. To the contrary, he would want to make the sensitivity as low as possible, in order to keep the quality of the vacuum as good as possible, for as long as possible. As such there is no motivation for the skilled man to change the dimensions of the packaging or MEMS to achieve a ratio between the chamber volume and the surface area that is smaller than 10^{-5}m.

Starting from D2 as the closest prior art the subject matter of claim 1 is thus also inventive.

3 Independent claim 14 relates to the same subject matter as claim 1.

The same argumentation applies mutatis mutandis.

This independent claim therefore also meets the requirements of patentability.

4 The dependent claims contain all the features of claims 1 or 14 and therefore also meet the requirements of patentability.