



Moisture detection methods and devices

Field of the invention



G00002

The present invention relates to devices and methods for moisture detection, and particular devices and methods which are suitable for detecting moisture
5 permeation through a coated polymer layer.

Background of Invention

Thin film transistors (TFTs) have been widely used in the recent years to reduce physical dimensions of electronic devices and to minimize the fabrication and assembly costs of the components. TFTs are thin current-
10 carrying layered structures that can be made using traditional semiconductor technology. It is believed that such thin integrated structures may make possible breakthroughs in electronic technology for the construction of electronic components on plastics material, to attain lightweight and flexible features for the next generation of handheld electronics. In particular, thin film
15 transistors are expected to make a significant impact on the display industry when plastic substrates are used in place of glass to create thousands or millions of lighting pixels.

For example, consider the case of presently known OLEDs (organic light
20 emitting diodes) which comprise, in order, on a glass substrate, a transparent anode layer, a hole injection layer, an organic light emitting layer, an electron transport layer, and a metal cathode layer. Efforts are presently being made to produce an OLED which is an analogue of this known OLED in which the glass substrate layer, which is generally inflexible, is replaced by a flexible
25 light-transmissive polymer layer.

Development of thin film display technology faces the challenge of degradation due to the damage to the electric circuitry layers (for example to

an organic light-emitting layer) caused by moisture permeating through the plastic substrate. Flexible plastic, commonly known as polymer substrate, unlike glass, contains micro pores because of its soft material structure.

Gases such as water vapour and oxygen in the ambient atmosphere surrounding the TFT are likely to be transmitted through the plastic substrate via these openings and harm the electrical circuitry layers. In most sensitive electronics components, moisture permeation affects the material properties leading to undesired failure or lessens the life of the transistors when oxidation or other forms of deterioration occurs on those thin structured layers.

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Much research has been carried out to develop a good protective coating for polymer substrates to eliminate undesirable effects of permeation through the substrates. In most cases, a plurality of inorganic film layers such as Aluminum Oxide, Silicon Oxide, etc., are coated on one surface of the polymer substrate to prevent penetration of water molecules. Nevertheless, inorganic coatings often contain cracks or other forms of deposition defects due to imperfections in their formation process. Therefore, water permeation is likely to occur through the barrier coating.

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In order to evaluate the effectiveness of various forms of barrier coating, and thus make possible a good choice of barrier coating, an evaluation tool is needed to detect the amount of permeation through a polymer material when provided with such coatings. Such a quantitative check of the moisture permeation is essential for fast screening of the performance of coatings as a moisture barrier.

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Descriptions of such systems can be found in scientific and patent literature. For example, in principle, it might be possible to estimate the level of moisture permeation by inspection of the surface of the coating, but corrosion of sensitive thin film layers often occurs without physical change, and

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deterioration may spread quickly when the barrier resistance is worn out due to extreme ambient conditions. Thus, visual inspection is not preferred as an evaluation technique.

- 5 Alternative, and more successful evaluation techniques are based on measuring the permeation of moisture from a first volume of gas to a second volume of gas, when the first and second volumes of gas are separated by the layer of which the permeability is to be measured. Such methods are based on the well-known phenomenon that moisture levels in the second
- 10 volume of gas can be easily determined by measurements of either the pressure change in the second volume of gas, or mass differences in the second volume of gas measured using a microbalance. However, when these methods are used to evaluation moisture permeation through layers with low permeation, the methods suffer from unacceptable error levels due to the
- 15 imperfections in the testing equipment and in the testing conditions, probably due to transfer of test samples. Other sensitive testing methods using electromagnetic radiation (e.g. microwave, or infra-red radiation) can accurately determine the level of water molecules in the second volume of gas. However, such techniques have disadvantages from the point of view of
- 20 operation safety, and also involve high equipment costs.

In summary, moisture permeation evaluation techniques which are less vulnerable to experimental error generally incur costly hardware or complex evaluation procedures and control of process conditions in order to achieve

25 precision detection. Furthermore, all known detection techniques require that test pieces of the moisture barrier material are tested in a special test location within specialist equipment; for example, it is not feasible to monitor the moisture permeation of barrier material while it is being processed into TFT devices. Thus, there is then a demand for a technique that is both simple and

inexpensive, and preferably suitable for on line monitoring of permeation accurately without complex analytical tools.

Summary of The Invention

5 The invention aims to provide new and useful methods and devices for moisture detection.

In particular the methods and devices to determine the amount of moisture which permeates through a laminar moisture barrier. The moisture barrier is composed of a laminar polymer substrate and usually at least one coating
10 layer.

In general terms, the present invention proposes that a capacitor is formed on one surface (normally a coated surface) of the polymer substrate. The other (uncoated) side of the polymer substrate is exposed to an environment
15 containing moisture. The capacitor includes a water absorbent material as the dielectric, so that as moisture permeates through the moisture barrier it enters the dielectric and changes its dielectric properties. A measurement of the capacitance of the capacitor thus gives an indication of the amount of moisture which has passed through the moisture barrier.

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This proposal enables monitoring of micro-scale permeation because it is possible to determine the moisture concentration accurately by means of electrical detection signal applied to the capacitor.

25 The capacitor structure is typically constructed as a laminated thin film structure. It comprises two conductive films sandwiching a water-absorbent dielectric layer. The proposed architecture makes possible uniform detection by a capacititor having a large area and a fixed narrow separation of the electrode layers, to maximize its sensitivity. This permits a board detection

range of moisture level for multiple scales of wetness density. In addition, a slight change in material property triggers a significant deviation in electrical behavior of the capacitor, so that embodiments of the invention may be highly sensitive.

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Here, a desiccant material is preferred as the dielectric layer due to its strong water absorbing ability. Preferably, the dielectric material is selected to have a no change in its physical dimensions upon absorbing moisture.

- 10 To avoid the desiccant from taking up moisture from its surroundings, it is preferably surrounded by a highly effective water-barrier in all directions except the one facing towards the moisture barrier which is to be tested. Specifically, a highly-effective water barrier such as glass plate may be provided on the laminated capacitor structure facing away from the moisture
- 15 barrier. Additionally, a high-barrier sealant is preferably provided at the inner edges of the water barrier plate, providing a wall to reduce moisture penetration from this direction. In this way, the movement of moisture into the dielectric is confined to moisture which passes into the dielectric through the moisture barrier being studied.

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The proposed structure makes it possible to enable experimental tests to be conducted at higher temperature and pressure settings than in conventional techniques, which in turns accelerates the water permeability of the moisture barrier, and thus shortens the period required for moisture permeation

25 evaluation.

A conventional deposition method may be used to provide the layers of conductive film. Furthermore, a conventional deposition method may be used to provide the desiccant layer. Thus, the proposed device can be fabricated at

30 low cost.

Furthermore, the capacitance measurement employed by the present invention can be performed by a simple independent module including an oscillator, an voltage amplifier, an demodulator and a recorder. No sophisticated testing module is necessary.

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Note that the proposed moisture detection device resembles the structure of thin film electronics transistor. This leads to it having high accuracy in predicting the ability of the moisture barrier to perform when subsequently utilised in a thin film electronics device.

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Furthermore, the present invention makes possible in-situ moisture permeation detection without user operation and handling, and can be performed while the moisture barrier to be tested is in any orientation.

15 Specifically, a first expression of the invention is a method of measuring the moisture permeation through a laminar moisture barrier, the method comprising:

forming a capacitor on one side of the moisture barrier, the capacitor having a water absorbent material as the dielectric;

20 exposing the other surface of the moisture barrier to an environment including moisture; and

measuring changes in the capacitative characteristics of the capacitor.

A second expression of the invention is a moisture permeation measurement
25 device comprising:

a laminar moisture barrier; and

a capacitor formed on one side of the moisture barrier, the capacitor having a water absorbent material as the dielectric.

Brief Description of The Figures

5 Preferred features of the invention will now be described, for the sake of illustration only, with reference to the following figures in which:

FIG. 1 shows the designed construction of device according to the invention;

FIG. 2 shows apparatus used to make a measurement using the
10 device of FIG. 1;

FIG. 3 shows in more detail the circuitry of the tester module of FIG 2;

FIG. 4, which is composed of FIG. 4(a) and FIG. 4(b) shows the permeation of water molecules into the device of FIG. 1;

FIG. 5 exhibits the relationship between the measured capacitance and
15 time, over a period in which the device of FIG. 1 is exposed to a high humidity environment; and

FIG. 6 exhibits the relationship between the moisture content and time, over a period in which the device of FIG. 1 is exposed to a high humidity environment.

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Detailed Description of the embodiments

Referring firstly to FIG. 1, a laminated thin test structure 100 according to the invention is shown. A polymer substrate 101 is shown coated with a barrier
25 coating 102. The device is for studying the moisture permeation of a moisture barrier made up of the combination of the polymer substrate 101 and barrier coating film 102. Note however that alternative embodiments of the invention may be produced which are used to study the moisture permeation of a

polymer substrate 101 without a barrier coating, and in those alternative embodiments the barrier coating film 102 is omitted.

A thin layer of electrode film 103 is adhered onto the barrier film 102 by
5 conventional sputtering deposition in a vacuum chamber. It is preferred that the electrode film 103 is made of high electrical conductive material and has a thickness of not more than 500nm. In addition, the material of the electrode layer is chosen such that it has stable chemical properties so that it will not have a disadvantageous chemical reaction with the coated barrier film layer
10 102, and also so that it will not degrade when it is exposed to moisture. A thin layer 104 of non-conductive, porous desiccant material is deposited by a traditional deposition technique on the first electrode layer 103 in a high vacuum enclosure. The desiccant layer 104 functions as a dielectric insulation. It is preferred that the dielectric material is selected such that it
15 absorbs water molecules quickly when exposed to them. Typically, the desiccant layer has a thickness of not more than 5 micrometers. Furthermore, the desiccant layer 104 should preferably have a smooth surface after deposition, and should not have an adverse interaction with the electrode layer 103. Furthermore, the desiccant material should have good
20 chemical properties so that it will not deteriorate or change its physical dimensions when it absorbs moisture. Before the desiccant layer 104 is deposited, it is preferable that the bulk desiccant material be warmed at a high temperature to release the moisture it contains.

25 A second electrode film 105 similar to the first electrode 103 is coated onto the desiccant layer 104, to sandwich the desiccant layer 104. By appropriate masking processes, each of the electrode films 103, 105 is arranged to include a square area, the two square areas facing one another and spaced apart by the desiccant layer 104 which is shaped as an identical square area.
30 Thus, the square portions of the electrode layers 103, 105 and the desiccant

layer 104 together have a capacitor structure. Furthermore, each of the layers 103, 105 additionally a respective lead portion 103a, 105a by which an external voltage can be applied to the respective square portions of the electrode layers 103, 105. The lead portions 103a, 105 are for connection to
5 an external tester module 203 (shown in Fig. 2).

So far as possible, all layers should be deposited free of impurities, which may introduce errors to the detection. These layers are preferably formed in vacuum. Shortly after the test piece is removed from the vacuum, a thin glass
10 slide 106 is placed above the upper electrode layer 105 to cover this flat planar sandwich structure. The glass slide 106 constitutes a moisture impermeable layer (i.e. having a moisture permeation level much lower than that of the moisture barrier, e.g. at least 10 times lower). Preferably this is performed in an enclosed chamber purged with nitrogen gas to shield off
15 moisture as well as surrounding contaminants. An ultraviolet-curable epoxy 107 is then applied at the edge of the glass slide 106, and cured using ultraviolet light. The epoxy 107 provides an extra sealing to restrict moisture permeation. Only the lead portions 103a, 105a of the electrode layers electrode layers 103 and 105 protrude from the volume enclosed by the glass
20 slide 106 and the epoxy 107.

The test piece 100 is loaded into a humidity chamber 201 as shown in FIG. 2. This chamber is maintained at a known relative humidity and temperature setting to replicate moisture aggression conditions. Fine electrical cables 202
25 are used to link up the electrode layers 103, 105 to a remote impedance measurement tester module 203, which can detect the change in the dielectric material characteristics of the desiccant layer 104 when water molecules travel across the barrier coating 102. This in-situ moisture sensor system can be linked to a computer 205 to automate periodic recording of the changes in
30 the dielectric material characteristics.

The basic schematic of the remote impedance tester module 203 connected to the test composite 100 is described as in FIG. 3. An AC electrical voltage is generated by a power supply unit 301. The frequency of the AC voltage is variable by an impedance measurement circuitry to provide an oscillating current signal at a desired frequency. The current induced by the applied voltage is boosted by an amplifier 302 before being applied one of the electrode layers 103 or 105, and this input signal serves as an excitation to examine the material property of desiccant layer 104. The voltage output from the other electrode layer is amplified, and an impedance spectrum is produced from the voltage developed between the electrode layers and is interpreted to indicate the amount of moisture in the test composite 100.

In operation, low frequency interference and equipment noise are produced within the lines 202 connected to the capacitor 103, 104, 105, and these noise sources are attenuated by means of a filter 303 to give a purer signal. Subsequently, the output current is converted to a corresponding direct current by a demodulator 304. A comparator 305 then determines changes in the dielectric properties of the desiccant layer 104, to give an indication of the moisture absorbed in the capacitor 103, 104, 105. Active monitoring of the dynamic current output of the capacitor 103, 104, 105 helps to provide continuous detection during the moisture aggression process, whereby the increase in moisture content can be constantly read and recorded by a computer 205. A series of digital detection values can be presented by computer 205 to identify the profile of the characteristics of the dielectric layer when moisture penetrates the barrier coating 102.

The operation of the structure 100 will now be explained with reference to FIG. 4, which is composed of FIG. 4(a) and FIG. 4(b) which shows a portion of FIG. 4(a). The plastic substrate 101 is exposed to an atmosphere 2 having

a high humidity. Water molecules 21 in the atmosphere 2 gradually permeate through the polymer 101 and through the barrier coating 102 over a period of exposure at high humidity environment, and these molecules 21 are entrapped and stored in the desiccant layer 104, resulting in a change in wetness density of this medium. This increase in moisture changes the electrical characteristics of dielectric layer 104 under the influence of an alternating electric field when an oscillating signal is applied at the electrode layers 103, 105. The amount of absorption of electric flux between the electrode layers 103 and 105 is known as permittivity of the dielectric material, and is modified owing to the reorientation of water molecules 21 caused by their dipole moment. The rising permittivity of the dielectric medium due to moistening of the dielectric layer 104 tends to reduce the electric field between the electrode layers 103, 105.

This phenomenon can be distinguished in the form of measured capacitive values over time, as shown in FIG. 5. The capacitance is according to the equation,

$$\text{Capacitance} = \text{Permittivity} \times \frac{\text{Area}}{\text{Width}} \quad (1)$$

where 'Area' refers to the surface area of the dielectric layer, and 'Width' refers to the separation gap between the electrode layers. The wetness density of the desiccant medium rises gradually as moisture penetrates through the barrier coating 102, and this is reflected by a rise in the measured capacitance, as shown in Fig. 5.

The dielectric properties of multiple standard samples of desiccant having known respective moisture contents can be used to correlate moisture content with measured impedance. Given this correlation, the microprocessor of the

computer 205 can easily calculate the values of the water content of the desiccant layer 204 corresponding to the capacitance values of Fig. 5. This comparison then makes possible a continual monitoring of the moisture level as shown in Fig. 6, and thus a continuous monitoring of the permeation
5 through the barrier coating 102.

Note that the moisture content measurement of FIG. 6 is a one-time measurement. That is, the water cannot be expelled from the desiccant layer 104, and hence the measurement structure 100 can only be used once.



Claims

1. A method of measuring the moisture permeation through a laminar moisture barrier, the method comprising:
 - 5 forming a capacitor on one side of the moisture barrier, the capacitor having a water absorbent material as the dielectric;
 - exposing the other surface of the moisture barrier to an environment including moisture; and
 - measuring changes in the capacitative characteristics of the capacitor.
- 10 2. A method according to claim 1 in which the capacitor comprises a first conductive layer formed on the moisture barrier, a dielectric layer of water-absorbent material formed over the first conductive layer, and a second conductive layer formed over the dielectric layer.
3. A method according to claim 1 or claim 2 in which the water-absorbent
 - 15 material is a dessicant.
4. A method according to claim 1, claim 2 or claim 3 in which the water-absorbent material is porous.
5. A method according to any preceding claim in which the capacitor is encapsulated within a structure which inhibits ingress of water from all
 - 20 directions apart from through the moisture barrier.
6. A method according to any preceding claim in which the capacitive properties of the capacitor are observed at a plurality of times, and used to produce data indicating the moisture transmitted by the moisture barrier over time.
- 25 7. A moisture permeation measurement device comprising:

a laminar moisture barrier; and

a capacitor formed on one side of the moisture barrier, the capacitor having a water absorbent material as the dielectric.

8. A device according to claim 7 in which the capacitor comprises a first
5 conductive layer formed on the moisture barrier, a dielectric layer of water-absorbent material formed over the first conductive layer, and a second conductive layer formed over the dielectric layer.
9. A device according to claim 7 or claim 8 in which the water-absorbent material is a desiccant.
- 10 10. A device according to claim 7, claim 8 or claim 9 in which the water-absorbent material is porous.
11. A device according to any of claims 7 to 9 further comprising a moisture impermeable layer provided over the surface of the capacitor facing away from the moisture barrier.
- 15 12. A device according to claim 11 comprising a resin layer sealing the moisture impermeable layer to the moisture barrier and encircling the capacitor.