Barix Thin Film Encapsulation of OLED's on Flexible and Rigid Glass substrates; high temperature performance and manufacturing aspects.

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Water Barrier coatings, Flexible substrates, thin film encapsulation, high temperature, manufacturing

Abstract

We will discuss encapsulation of OLEDs on both flexible and rigid glass substrates.
Accelerated testing at 6CC/90RH and 85C/85RH is compared and acceleration factors for OLED and Calcium test samples are discussed.We have tested the stability and performance of our barrier coating to much higher temperatures: up to 140 C. Water Vapor Transmission rates at temperatures from 60 to 140 C are presented. Rates and methods for low cost manufacturing on a large scale are analysed

1. Introduction

Thin film encapsulation of OLED Displays does bring a lot of advantages over the existing glass lid/metal can plus dessicant encapsulation technology; it would make the devices roughly half as thick, it would reduce the cost, it enables top emission displays and would also reduce the total periphery space of the display.

But although thin film encapsulation would be an attractive feature, it has not been so easy to achieve that goal in a technically and economically feasible way. The requirements to the layers of being; transparent, totally pinhole and crack free over very large (>1 m^2) surface areas, low stress and high robustness while being deposited at low temperatures well below 80 C, have proven to be very difficult to meet.

For making a flexible display, one not only needs a barrier substrate (even a flexible metal foil can be seen as such), but one also needs to protect the display from the other side. This can be done by thin film encapsulation or by sandwiching the display between two barrier films.

But although thin film barrier coatings on plastic and thin film encapsulation are highly desirable, it has not been so easy to achieve that goal in a technically and economically feasible way. The requirements to the layers of being; transparent, totally pinhole and crack free over very large (>1 m^2) surface areas, low stress and high robustness while being deposited at low temperatures well below 80 C, have proven to be very difficult to meet.

Early attempts to solve this problem with single layer oxides or nitrides, while obtaining some success on small areas, basically failed because of the presence of particles, crack and defects in the layer and residual stress.

Vitex has proposed a multilayer of organic and inorganic layers, Barix ™ [3,6,7,8], to address and solve these problems. The multilayer consists of thicker (0.25 to 4 micron) polymer layers alternated by thin (200-500 nm thick) layers of oxide or nitride. The polymer layers are being deposited in vacuum as a thin liquid film of an acrylate monomer which is polymerized with UV light. These layers fulfill the following functions: because of their initial liquid state they planarise the substrate and because of the fat surface of these films, provide the almost ideal surface to grow a defect free oxide. The polymer layer furthermore covers particles, decouples defects in the oxide layers so that they are not aligned and function as a stress release layer.

The thin films of oxide serve as the barrier layers to oxygen and water. As demonstrated theoretically by G Graff et al[4], the main effect of the multilayer is in increasing the lag time between exposing the top layer to water vapour and the water molecules arriving at the interface between the OLED and the Barix™ encapsulation layer.

Figure 1

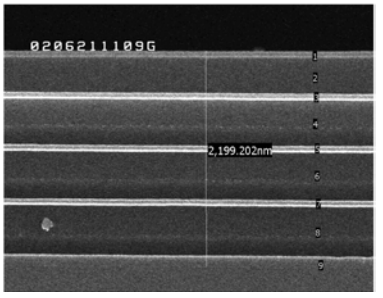

Fig 1. SEM Cross section of a typical Barix multilayer barrier coating. Oxide layers typically are between 30-100 nm and polymer layers 0.25 to 4 micrometers.

The layers are all deposited in vacuum as is shown schematically in Figure 2 [6,7,8.] The organic layers are applied as follows: a mixture of photosensitive acrylate monomers is vaporized, condensed on the substrate and quickly polymerized with UV radiation. The inorganic metal oxide layer, mostly Aluminum oxide, is deposited via a reactive sputtering process. Typically the organic layers vary between 0.25 and 4 micron in thickness and the metal oxide layers between 30 to 100 nm. What is really unique about this process is that the organic phase is deposited as a liquid: the film is very smooth (< 2 Angstrom variation) locally and also has extremely good planarizing properties over particles and high topographical structures like 'cathode separators' 'ink jet wells' and Active Matrix pixel structures. So while the local flatness creates an ideal surface for growing an almost defect free inorganic layer, the liquid takes care of covering topography

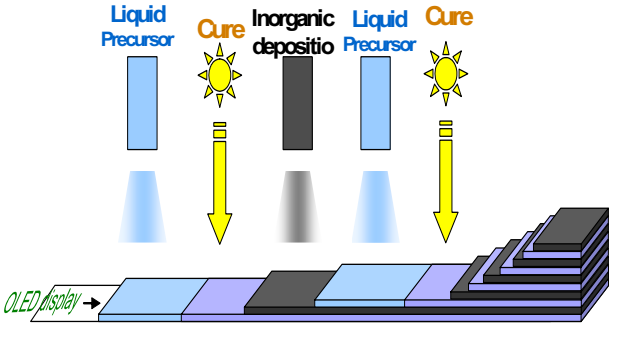

Fig.2 Schematic presentation of the process steps of the Barix encapsulation

It should also be mentioned that while even non-conformal methods to deposit oxides like CVD, have difficulty covering cathode separators without creating voids, they also struggle to coat often more then 4 micron high structures in an acceptable process time.
The multilayer provides redundancy and since the remaining defects in the inorganic layers are few and far in between and not connected, a very long diffusion path to the substrate results as well.[4]

The organic layers also provide a function of stress release layer in thermal shock testing.

An extensive model for the diffusion through this type of barriers has been developed by G Graff et al.[4]

The main findings of this study are that i) high quality inorganic films coupled with a multilayer architecture are necessary to achieve OLED barrier requirements (large spacing between defects) ii)Lag time (transient diffusion), not steady state flux, dominates gas permeation in these multilayer thin films systems. iii) Consideration of steady state , alone, is not sufficient to describe and predict the performance of multilayer barrier films one must consider the transient regime.

The Vitex Barix™ process has been shown to meet telecommunication application specifications for a wide variety of OLED displays: passive and active matrix displays, bottom, top and transparent displays and it works equally wel for small molecule, polymer and phosphorescent OLEDs.[7,8,9]

Fig 3. Flexible AM Matrix Display by LG.Philips and UDC on metal foil substrate, encapsulated with Vitex Barix encapsulation.

2. Results

Now that it has been shown that Barix encapsulation of OLED qualifies for telecommunication application specifications, it makes sense to look at two aspects:

- Higher temperature performance and stability of the barrier in relation to automotive type of specification.
- Manufacturing aspects like, influence of defects, reduction of the nr of layers, throughput and cost.

Using the Calcium test[10] at 60C/90 RH we have established that for barrier films made on a batch machine on a glass substrate typical WVTR of 10^{-6} gr/m^2/day can be obtained with champion values around 10^{-7} gr/m^2/day.

Accelerated tests at 60C/90RH and 85C/85RH

The most demanding tests for thin film barrier encapsulation is the high temperature/ high humidity test. For telecom applications this is typically 500 hrs at 60C/90RH, for automotive applications this test is performed at 85C/85RH.
In order to get a better understanding of the mechanism determining the outcome of the stability test we have tested 1*1 cm^2 OLED pixels and 1*1 inch^2 Calcium buttons on glass substrates at these different temperatures.

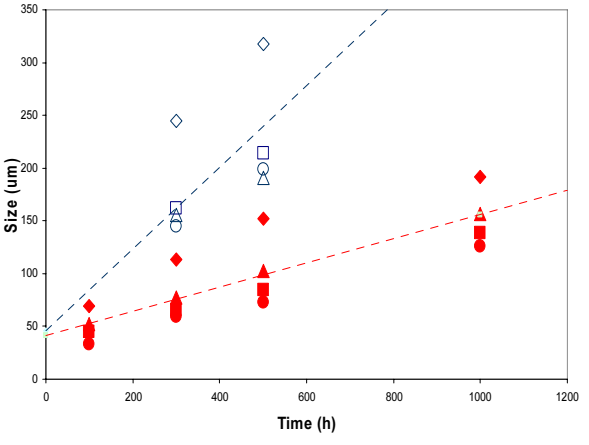

Fig. 4 Blackspot growth in OLED samples encapsulated with Barix encapsulation at 60C/90RH and 85C/85RH.

As one can see because of the transport of the OLED samples we started with samples where the mean size of the blackspots was already 50 micron diameter. We have checked by measuring blackspots before and after encapsulation that the encapsulation process itself does not create new blackspots, nor enlarges them.
As can be seen from the graph the ratio of the slope is 3.4. The acceleration factor between the two conditions is 3.4 ±0.5.
In case of the Calcium test we obtained from the ratio of the thinning of the Calcium over time an acceleration factor of 3.0 ±0.7. Within the margin of error, it is hard to tell if the small difference between the data for Calcium test samples and OLED samples is significant.
The accelerated aging can be caused by two factors, the increase of the water vapour pressure at with increasing temperature and an activation energy in the diffusion process of water through the barrier.
We have created a table of acceleration factors assuming only the influence of vapour pressure and the combined influence of vapour pressure and a small activation energy of 30 kJ/mole.

Condition	Vapor pressure	Vapor pressure +activation energy of 30kJ/mole
60C/90% RH vs. RT 50% RH	15	15 / 0.23 = 65
85 C/85% RH vs. RT, 50% RH	38	38 / 0.1 = 380
60C/90% RH vs. 85 C/85% RH	2.55	2.74
38C/90% vs. RT, 50% RH	4.7	9.58

As can be seen from the table most of the acceleration is caused by the difference in vapour pressure at the different temperatures. A small activation energy between 30 kJ/mole and 60 kJ/mole would be needed to explain the whole effect.
While the contribution of a thermally activated diffusion process is small for this temperature interval of 25 C, it begins to have a large impact for the acceleration factor between 20 C and 60C or 85C. These results predict based on a lifetime of more then

a 1000 hrs at 60 C and 85 C, RT lifetime lifetimes well in excess of 8 years.

Stability and Water Vapour Transmission rates at high temperatures.

We have tested the stability of the barrier layers on Calcium samples on glass at temperatures ranging from 100 C to 140 C.
To test both the stability of the barrier and the WVTR at higher temperatures we also performed Calcium tests of Calcium buttons on glass wafers, encapsulated with Barix (3 and 6 dyads) at temperatures ranging from 100C to 140C in ambient. Obviously the result of this experiment is less accurate as we do not control the humidity of the environment.

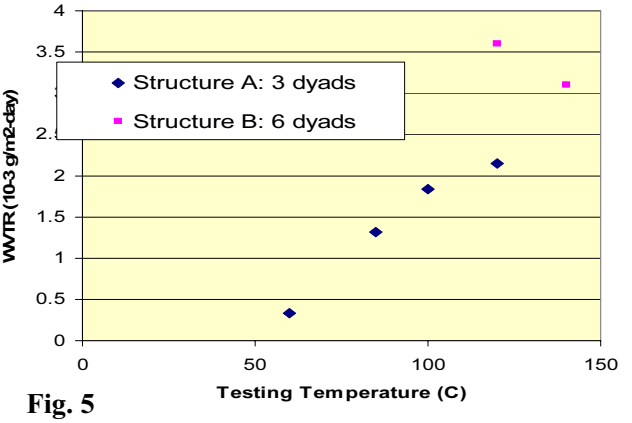

Fig. 5
WVTR values measured for Barix encapsulation in the Calcium test as a function of temperature.

Two important observations can be made:

- The Barix multilayer is very stable even at 140C , this is well above the Tg of the polymer layer which is 120C. Even after 550 hrs at 140C, no pinholes or small cracks could be seen in the Calcium test.
- The WVTR increases roughly a factor of 2 per 20 C

As the humidity is not controlled these WVTR values are only reflecting the effective transmission rates at these temperatures in ambient.

3. Conclusion

We have shown that the Barix thin film encapsulation is stable even at 140C and does not show cracks or degradation. The acceleration of degradation rates is the same for Calcium and for OLEDs within the margin of error of the measurements. The acceleration at higher temperatures and humidity's should be largely attributed to the difference in vapour pressure combined with a small activation energy ranging between 30 kJ/mole and 60 kJ/mole.

4. Acknowledgements

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