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MEASUREMENT OF ULTRA-LOW MOISTURE PERMEATION THROUGH EPOXY SEALING IN OLED ENCAPSULATION

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Abstract: As the next generation flexible display elements are very vulnerable to moisture, securing proper encapsulation is a decisive factor in enabling a long working life. Therefore, together with the recent development of plastic barrier film with very low permeability, interest in the permeability of sealant used for periment sealing has been increasing. In this study, the equipment to measure moisture permeability in periment sealing was established, and the permeability of different sealants was measured, whose resolution was approximately ~10^{-7} g·day^{-1}.

Keywords: Moisture Permeation, Water Vapor, Tritium, Sealant, OLED Display .

1. INTRODUCTION

The flexible display is a new-concept, next-generation display technology that can be bent, crooked or rolled without causing any damage to the image, and it is accomplished by using a thin, light, and flexible substrate such as plastic film, instead of glass substrate. However, one important factor in using the polymeric film substrate for display purposes is the encapsulation technology to prevent the entry of moisture or oxygen. The OLED element is very sensitive to moisture or oxygen. The organic luminiscent material easily loses its luminiscence once it is exposed to moisture, and the highly reactive cathode with low work function will be easily corroded by moisture and oxygen. In order to acquire 10,000 hours of working life, the water vapor transmission rate (WVTR) required of the barrier layer structure to sufficiently protect an OLED is reported to be less than 10^{-6} [g·m^{-2}·day^{-1}]. For this reason, it is absolutely necessary to measure the moisture transmission rate to evaluate a plastic barrier substrate. However, the minimum moisture rates that can be measured with current commercial equipment are 10^{-3} [g·m^{-2}·day^{-1}] [1].

Recently, the use of tritium to measure extremely-low water vapor permeation has been reported [2]. And to meet the demand of industry, KRISS has established a low water vapor permeability measurement system using tritium, a radioactive isotope [3]. This is available up to the vertical vapor permeability of plastic barrier film, 5 × 10^{-7} [g·m^{-2}·day^{-1}]. However for the vapor permeability into the flexible display element, not only the vapor permeability of the plastic barrier film, but also the sealing part of film perimeter cannot be neglected, and recently the measurement of the vapor permeability of the sealing part has been required by industry. This study aims to develop the moisture permeability measurement technology for the sealant used at the sealing part of film perimeter, to support the industry.

2. MEASUREMENT EQUIPMENT

As water has a permanent dipole moment and hydrogen bond, it is generally more difficult to accurately measure moisture than other physical properties. In particular, the measurement of traces at the level of ~ppb is very difficult. Therefore, to increase the accuracy of measurements, the developed technique uses water including tritium (HTO), which has more energy than the water. The established measurement system using tritiated water is shown in Fig 1. Vapor permeability at an OLED display can occur not only through the plastic substrate, but also through the periment sealing of the substrate. Usually, the permeability through the plastic substrate is called Z-direction permeability, while that through the periment is XY-direction permeability. Fig. 2 shows the schematic diagram of XY-directional vapor permeability measurement. The periments of the two differently-sized substrates of XY-directional specimen were bonded with sealants, whose lower part is perforated. The water including tritium (HTO) is put into the lower part of the divided space of the prepared XY-direction measurement specimen, the carrier gas is flowed into the upper part, the HTO having permeated through the specimen is carried to the tritium detector, and the quantity of permeated moisture is measured.

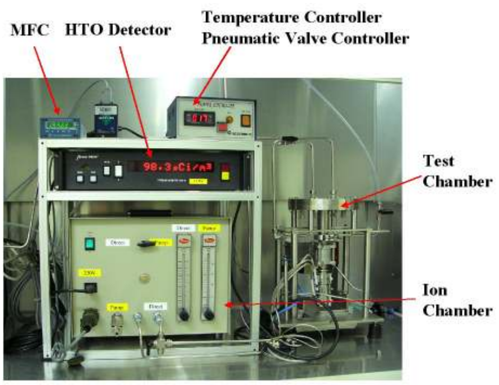

Fig. 1 Water vapor permeation measurement system

If the specific radioactivity of the HTO specimen used for measurement is A [Ci·cm^{-3}], the amount of water [g] contained in C [Ci·m^{-3}] radioactivity of this water can be calculated as:

$$m_w = \frac{1}{A} \cdot C \cdot \sqrt{\frac{M_T}{M_H}} e^{\frac{\ln 0.5}{12.5} Y} = \frac{1.054}{A} \cdot C \cdot e^{\frac{\ln 0.5}{12.5} Y} \qquad (1)$$

The numerator of the first term in the above expression shows the difference in vapor permeability by the difference in molecular weight of HTO and H_2O, the last term is due to weakened radioactivity followed by the tritium radioactive decay, and the half life of tritium is 12.5 years, in the expression Y=year.

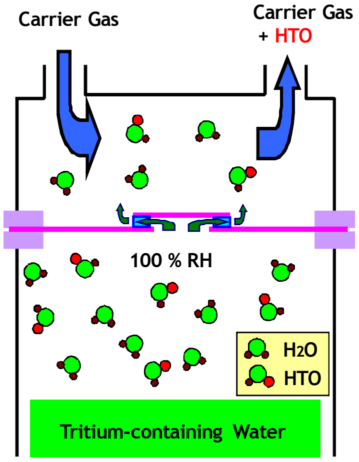

Fig. 2 Schematic diagram of the water vapor permeation measurement through periment sealing of substrate with tritium-enriched water.

3. MEASUREMENT RESULTS

Fig. 3 shows the specimen prepared for the measurement of XY-directional vapor permeability. A square type hole with width 1.5 ㎝ is perforated at the center of a glass substrate with width 10 ㎝ and thickness 0.7 ㎜. On it, there is the same type of glass substrate with width 3.5 ㎝, whose gap is bonded with sealant. The thickness of sealant is approximately 100 ㎛, with width 1~4 ㎜.

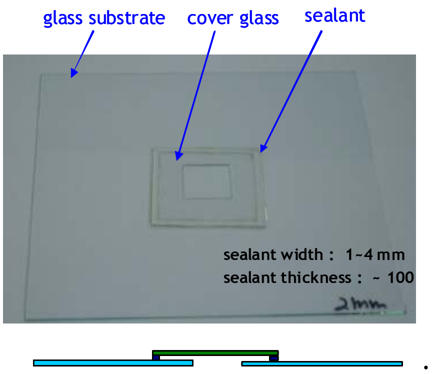

Fig. 3 Specimen for water vapor permeation measurement in XY-direction

Fig. 4 shows the result of the measurement of vapor permeability of XY-directional specimen prepared using silicone sealant (part no. 40557 by Loctite Co., Ltd.). In the graph, (a) shows the increase in tritium signal detected by beta-ray detector, (b) shows the quantity of water permeated and calculated from the increase in tritium signal, and (c) shows the vapor permeability converted into the quantity permeated for a day. In other words, the vapor permeability of silicone sealant measured through the periment of 35 ㎜ × 35 ㎜ quadrangle was 1.3×10^{-4} [g·day^{-1}]

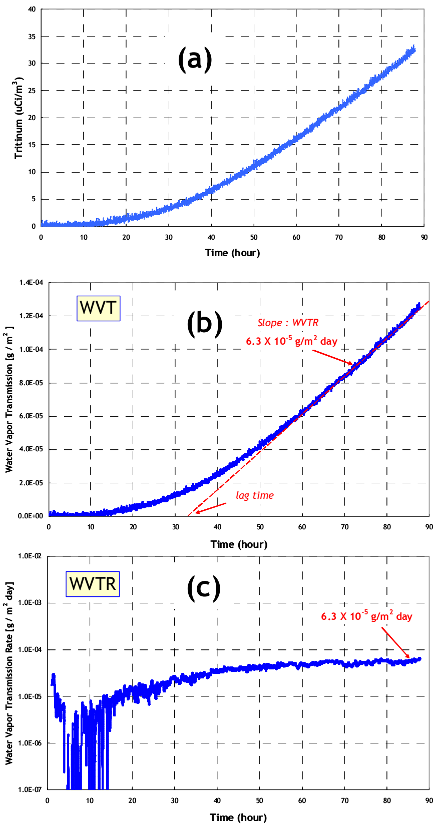

Fig. 4. Water vapor permeation curves for Silicon resin. (a) detected tritium signals, (b) calculated transmitted water vapor, (c) calculated water vapor transmission rate with time

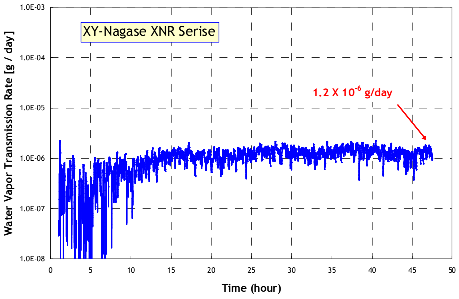

Fig. 5 Water vapor transmission rate for UV epoxy(Nagase XNR serise)

Fig. 5 shows the measurement result of XY-directional specimen prepared by UV epoxy (Nagase XNR series) numerously used by the recent LCD, OLED display. The measurement of the UV epoxy for 3.5 cm x 3.5 cm size showed very low vapor permeabilities of 1.2×10^{-6} [$g \cdot day^{-1}$].

4. CONCLUSION

In order to meet the demands of the display industry KRISS has developed ultra-low vapor permeability measurement equipment using tritiated water, and measures the vertical directional (Z-direction) vapor permeability of plastic barrier film. However, recently the industry has required not only Z-directional vapor permeability, but also XY-directional vapor permeability, at the periment of the bonding surface. Accordingly, this study established the XY-directional vapor permeability measurement equipment, and the test on different specimens was finished, whose resolution was $\sim 10^{-7}$ [$g \cdot day^{-1}$] or so. This will make a great contribution to technical development by supporting not only the display industry, but also sectors requiring moisture-proof encapsulation, such as the semiconductor and solar cell industries.

5. REFERENCES

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