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A novel cup with a pressure-adjusting mechanism for high-temperature water vapor transmission rate measurements

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ABSTRACT

Water vapor transmission rate (WVTR) measurements with the conventional cup method do not yield accurate values at high temperatures because the film specimens deform and are damaged owing to air expansion in the cup. A new cup with a pressure-adjusting mechanism allows measurements at 85 °C and prevents specimen deformation and damage. WVTRs of polypropylene (PP) and polyethylene terephthalate (PET) measured with the new cup method are the same as those measured with the conventional cup method at 40 °C, and gas chromatographic detection method at 60 °C and 85 °C. Arrhenius plots of the water vapor permeability coefficient of PP, polyethylene naphthalate (PEN) and polyimide (PI) with the new cup method show a linear relationship in the range 25–85 °C. In the same range, Arrhenius plots of PET, polybutylene terephthalate (PBT) and polylactic acid (PLA) have bending points corresponding to the glass transition temperatures of the materials.

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1. Introduction

Proper packaging of electronic components prolongs their life and improves their reliability. To achieve high-performance hermetic packaging, high-barrier materials, such as organic–inorganic hybrid films [1–3] and moisture-proof adhesives [4–7], have been widely developed in recent years.

To develop high-barrier materials, the water vapor barrier properties of such materials have to be understood, especially for electronic components that require high reliability in high-temperature environments [8–10]. However, in the existing standard test methods, there is no reference to high-temperature methods for measuring the WVTR. In contrast, there are references to cup methods [11–14], humidity sensing methods [15,16], infrared detection methods [17,18], electrolytic detection methods [19,20] and gas chromatographic (GC) detection methods [21,22]. For this reason, many material developers rely on their own evaluation methods [23–28].

In 2015, to answer the needs of the industry, ISO adopted new methods for measuring WVTRs of plastics at high-temperature and high-humidity conditions [29–31], e.g., 60 °C and 90% relative humidity (RH) and 85 °C and 85% RH.

The cup method (dish method) in ASTM E96 is a basic standard test method for measuring WVTR. It is simple and inexpensive, and can be used to calibrate the standard test pieces for other methods. However, this method is not suitable for making WVTR measurements at high temperatures because of melting of the sealing wax agent (the typical melting point of wax is approximately 50–60 °C) and the high-temperature deformation and damage of the specimens owing to the increasing internal pressure in the cup.

The purpose of this paper is to present a new, reliable high-temperature cup method using a modified cup that accommodates pressure variations. We verify the validity of the method by performing WVTR measurements of a variety of plastic film samples.

2. Experimental

2.1. Materials

The samples used in this study were PP (OPP bag, WorkUp Co., Ltd.), PET (Lumirror S10, Toray Industries, Inc.), PEN (Teonex Q51, Teijin DuPont Films Japan Ltd.), PI (Mordohar PIF, Future

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Technology Co., Ltd.), PBT (Smell blocking bag, Kansai Chemicals Co., Ltd.), and PLA (CF-3S-10P, Lion Office Products Corp.), see abstract for long forms of the abbreviations. Table 1 lists the physical properties of the various film samples. Anhydrous calcium chloride (for U-tube, Wako Pure Chemical Industries, Ltd.) was used as desiccant.

2.2. Test method

WVTR measurements were performed in accordance with the ISO 2528 standard test method. The cup was of screw-type (Imoto Machinery Co., Ltd.), as defined in JIS L 1099 [35]. Anhydrous calcium chloride (desiccant) weighing 20–25 g was placed in the cup. The film specimen was placed on the φ60 mm cup opening at 3 mm from the desiccant and was fixed by a ring and a screw. The cup was kept in a constant climate cabinet (LHL-113, ESPEC corp.) at a predetermined temperature and 90% RH, and it was taken out at predetermined time intervals and weighed. To ensure air tightness, the cup components were coated with oil compound (HIVAC-G, Shin-Etsu Chemical Co., Ltd.) having high thermo-oxidative stability. The total mass of the cup was weighed after 5–30 min of cooling at room temperature in a desiccator that contained silica gel. The time that the cup was taken out of the climate cabinet is subtracted from the total measuring time. The temperature and humidity in the constant climate cabinet were measured with a HYT-221 sensor (Innovative Sensor Technology, accuracy ±0.2 °C, ±1.8% RH). The humidity in the cup was determined by a SHTDL-3 miniature humidity sensor (Syscom Corp.) with a HYT-271 sensor element of Innovative Sensor Technology with a low humidity accuracy of ±1% RH. When the cup–desiccant–PET system was kept at 85 °C, 90% RH until 2 g of water vapor permeated, the relative humidity in the cup was maintained under 1.8%.

WVTR (g m^{-2}d^{-1}) and the water vapor permeability coefficient P (mol m^{-1}s^{-1}Pa^{-1}) were calculated using the following equations,

$$\mathrm{WVTR} = \frac{\Delta q}{A\Delta t}$$

$$\mathrm{P} = \frac{\Delta q}{A\Delta t}\,\frac{l}{\Delta p}$$

where Δq/Δt is the mass change of the cup per time (g d^{-1} or mol s^{-1}), A is the transmission area (m^2), l is the sample thickness (m), and Δp is the difference in partial water vapor pressure between the two sides of the film specimens. Three replicates of each film type were tested.

2.3. Pressure-adjusting mechanism

To prevent specimen deformation and damage owing to the internal pressure variations in the cup, we built a pressure-adjusting gasket and attached it to a conventional cup. The gasket shown in Fig. 1 consists of a polypropylene gasket with side air vents and a laminated aluminum foil bag (HRS-1422S, Meiwa Pax Co., Ltd., the thickness of the Al layer is 9 μm) for collecting the air leaking from the vents. The gasket was sandwiched between the conventional cup and ring, and coated with oil compound where it overlapped to prevent air leakage. The new cup with the gasket has sufficient air tightness for making WVTR measurements because the measured value for a nonpermeable 30 μm thick aluminum sheet is less than 0.2 g m^{-2}d^{-1} at 85 °C and 90% RH. The new cup was weighed with an electronic balance, and the laminated bag of the gasket was folded over the cup.

2.4. Pressure measurement in the cup

The air pressure in the conventional cup and the new cup were measured continuously for 24 h at 85 °C and 90% RH by a pressure sensor module (CQ30A-G101, TAISEI Co., Ltd.), which was attached to the cups. Air tightness was maintained.

2.5. WVTR measurements with the GC detection method

WVTR measurements based on the equal pressure method were performed in accordance with ISO 15105-2 using a GC detector sensor (GTR-20FXA, GTR Tec corp.) at 60 °C and 90% RH and 85 °C and 90% RH with a gas flow rate of 30 cc min^{-1}.

2.6. Characterization

The storage modulus E′ and loss modulus E″ of each film specimen were investigated during heating at 2 °C min^{-1} at an amplitude of 30 μm, frequency of 3 Hz, and temperature range of 25–85 °C using a PZ-Rheo NDS-1000 (TAISEI Co., Lid.). The glass transition temperature of PET, PBT and PLA was determined from the bending of the dynamic viscoelasticity curves with increasing temperature. PBT and PLA were measured without any

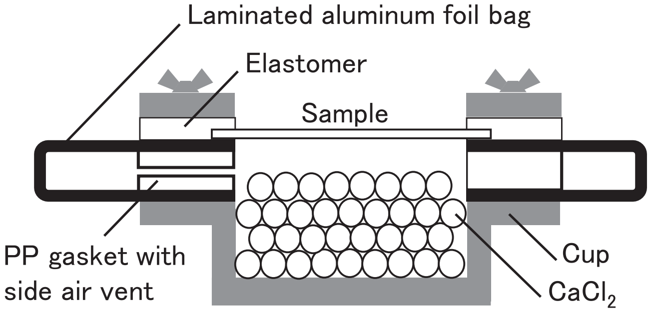

Fig. 1. Schematic of the pressure-adjusting gasket attached on a conventional screw cup.

Table 1
Physical properties of various film samples. Asterisk denotes data from Refs. [32–34]. Double asterisk indicates cm^3(STP)cm^{-1}s^{-1} cmHg^{-1} at 35 °C. The gas transition rate was estimated from the thickness of the samples and the gas permeability.

Sample	Thickness [μm]	Glass transition temperature [°C]	Gas permeability* [cm^3(STP) mm m^{-2}d^{-1}atm^{-1}]		Gas transition rate [mol m^{-2}s^{-1}Pa^{-1}]	
			O_2 (23 °C)	N_2 (23 °C)	O_2 (23 °C)	N_2 (23 °C)
PP	30	−7*	107	20.5	2×10^{-11}	3×10^{-12}
PET	25	65	0.91	0.2	2×10^{-13}	4×10^{-14}
PEN	25	125*	0.525	–	1×10^{-13}	–
PI	100	210–270*	10	2.0	5×10^{-13}	1×10^{-13}
PBT	50	49	15.2	3.04	2×10^{-12}	3×10^{-13}
PLA	125	57	3.36×10^{-11}**	5.40×10^{-12}**	9×10^{-12} (35 °C)	1×10^{-12} (35 °C)

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