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Short communication

Helium gas permeability measurements of polymeric membranes using the difference method

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ABSTRACT

This paper presents a new permeation measurement facility based on the difference method, which is shown to be capable of measuring gas fluxes through different polymeric membranes. In order to correct for the background pressure increase in the measurement chamber, a reference chamber has been adapted and connected to it. The permeation flux was examined by measuring the pressure difference between the reference and measurement chambers. Pure helium permeation was performed through polydimethylsiloxane (PDMS) and polyethylene terephthalate (PET) membranes using this method. The uncertainty of the permeability was evaluated. The results show that the introduction of reference chamber can mitigate the influence of the residual pressure in the measurement chamber. This new method allows simplified and visualized measurement of the permeation fluxes.

The investigation of gas permeation through different polymeric films is of great interest. Several industrial applications, such as organic light emitting diode (OLED) package and stratospheric airship, are based on the permeation properties of membranes. Transport of gas through the polymeric membranes is described by Fick's first law. In order to obtain the transport properties given by the diffusion coefficient (D), permeability (k) and the solubility (S), permeation flux needs to be measured precisely.

The classical time-lag permeation measurement is the most commonly method which can be found in several articles [1–4], and has also been adopted by the American Society for Testing Materials [5]. According to this method, the experimental facility consists of two chambers separated by the specimen. Gas is introduced into the chamber and then penetrates in normal direction to the polymeric membrane surface by the pressure difference. The increasing pressure of the measurement chamber and its relationship with time were recorded. As the test duration should be long enough, the residual pressure in the measurement chamber will increase with the ongoing process due to desorption and outgassing, which obviously results in the increase of fictitious pressure. To eliminate this measurement deviation, several researchers have described apparatuses to measure the partial pressure increase based on the use of quadrupole mass spectrometer (QMS) [6–8]. A new permeation apparatus was developed by Tremblay et al. [9] to measure the gas permeation characteristics of polymeric membranes, in which a residual gas analyzer was used as a selective pressure transducer to measure pressure changes of the ultra high vacuum (UHV) chamber. However, the system with two UHV chambers may need extensive work before performing permeation experiments, and the laboratory equipment is expensive due to the introduction of the residual gas analyzer calibration and UHV hardware.

This paper aims to propose a new gas permeability test facility based on the difference method, which has been used to directly measure the net outgassing rate [10] and the flow rate of standard leak [11,12]. To exclude the effect of background outgassing, a reference vacuum chamber was connected to the measurement chamber. The permeation flux could be achieved according to the pressure difference between the reference and measurement chambers. The dates were dealt with Fourier series method to get helium permeation parameters. PDMS and PET membranes were used to perform permeation experiments to demonstrate the validity of this approach.

The solution-diffusion mechanism describes transport of gas through the membrane. The driving force behind the transport process is the gradient concentration between the two sides of the polymeric membranes, and this process is described by Fick's laws of diffusion [13]. The permeating flux, J (mol/m^2/s), may be

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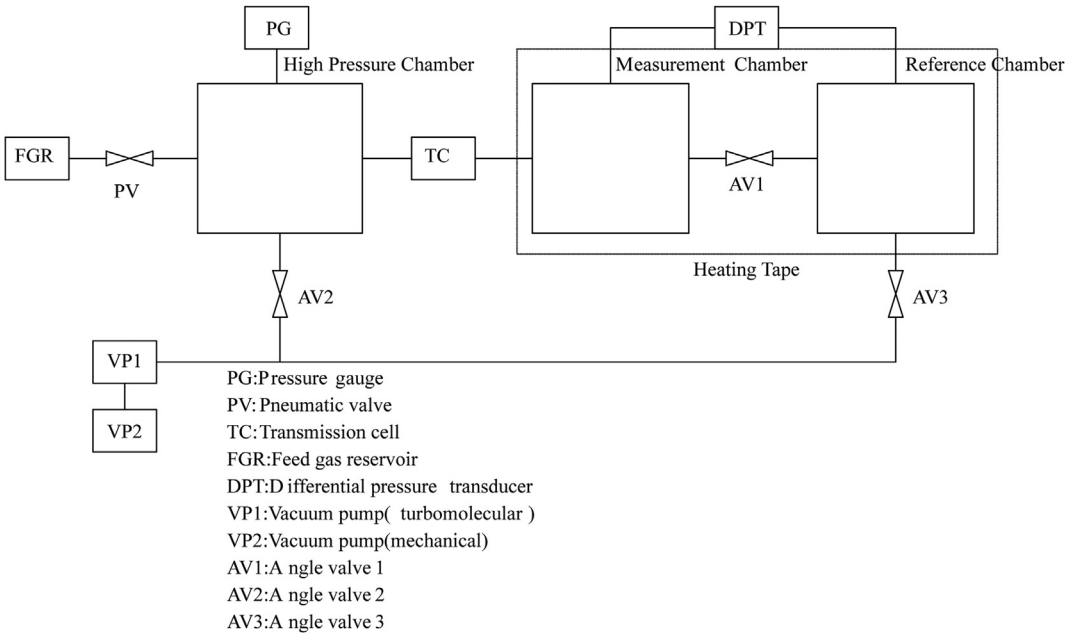

Fig. 1. Schematic diagram of the permeation measurement facility.

described by Ref. [14]:

$$J = D(C_0 - C_1)/h \tag{1}$$

where D (m^2/s) is the diffusion coefficient, C_0(mol/m^3) and C_1 are the permeant concentration on the two sides of the membrane, h (m) is the thickness of membrane.

In this experiment, the gas partial vapor pressure can replace the concentration when the gas diffuses through a membrane, and then the permeating flux is usually described by Ref. [14]:

$$J = k(p_0 - p_1)/h \tag{2}$$

Where k (mol/Pa/m/s) is the permeability and p_0–p_1(Pa) is the pressure difference between the two sides of the membrane.

In order to obtain the diffusion coefficient D, permeability k and the solubility S ($mol/Pa/m^3$), the Fourier series method will be adopted. Pressure difference between the two sides of the membrane was measured by differential pressure transducer and meets the following physical model [4]:

$$dp/dt = JART/V \tag{3}$$

where dp/dt (Pa/s) is the pressure increase rate, A (m^2) is the effective area of the membrane, V is the volume of the measurement chamber ($1.15 \times 10^{-4} m^3$), R is the gas constant (8.315 J/K/mol) and T is temperature (293 ± 1 K). The relationship between the helium leak rate, Q (Pa m^3/s), and pressure increase rate is:

$$Q = Vdp/dt \tag{4}$$

As a consequence, combining Eq. (2), Eq. (3) with Eq. (4), k can be expressed by:

$$k = \frac{dp}{dt}\frac{Vh}{ARTP} \tag{5}$$

Where P is the pressure difference between the two sides of the polymeric membranes ($P = p_0 - p_1 = 1.01 \times 10^5 \pm 300$Pa).

After permeating, the gas pressure in the measurement chamber (p(Pa)) is obtained by using the Fourier series method [4]:

$$p = P\frac{ADS}{Vh\lambda}\left(t - \frac{h^2}{6D} - \frac{2h^2}{\pi^2 D}\sum_{n=1}^{\infty}(-1)^n \frac{\exp\left(-\frac{n^2\pi^2 D}{h^2}t\right)}{n^2}\right) \tag{6}$$

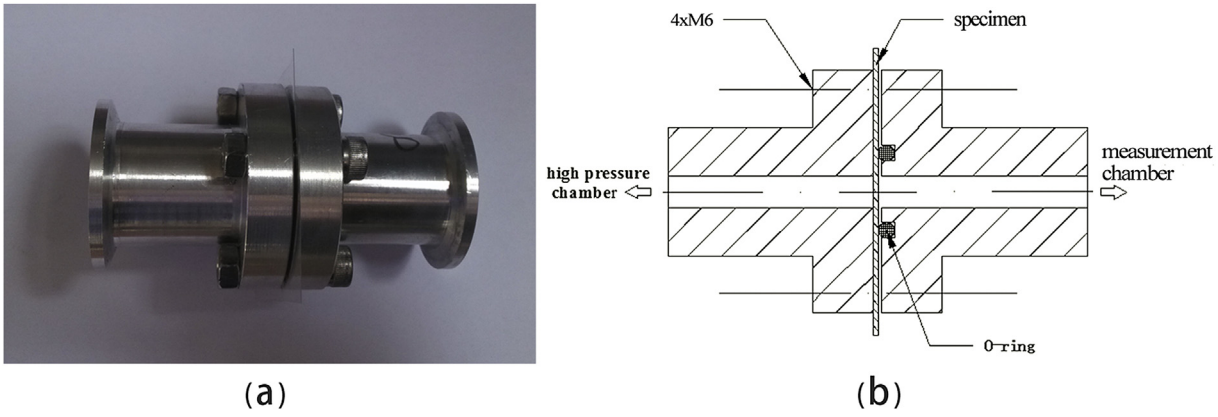

Fig. 2. The membrane mounting design: (a) photo taken of the transmission cell (b) schematic layout in cross section with the position of the membrane.