

ECN Permporometer

Introduction

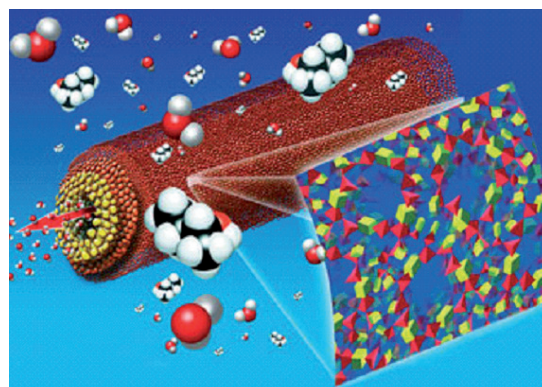
Knowing the pore size and size distribution of selective layers is essential in the development and application of ceramic membranes. The ECN Permporometer offers a quick, cheap and effective method for the characterization of micro- and mesoporous membranes. The Permporometer has been developed by the Energy research Centre of the Netherlands, and has been proven to be essential in the successful development of novel ceramic membranes.

The Permporometer uses the permeation of humidified helium to assess the pore size distribution of very thin layers on gross bulk supports. As it measures the behavior of the membrane as applied in practice, this method provides more information on the characteristics of a micro- or mesoporous membrane than alternative methods (more information on humid helium permeation can be found in the text box). Because the method only tests the selective layer, the supporting layers do not influence the measurement.

The ECN Permporometer enables the determination of membrane pores as small as the size of a water molecule ($d = 0.31 \text{ nm}$), and is extremely useful for performing comparative studies on different membranes and troubleshooting in applications. It is even very well suited for testing of defective membranes, and renders an estimate of the intrinsic pore size distributions. The influence of defects can also be easily recognized.

Humid helium permeation

Humid helium permeation is a non-destructive method based on capillary condensation of the water vapor in the fine pores at a given humidity. As a consequence, the helium permeation through these pores is blocked by water. Since the active pore distribution of membranes are asymmetric in nature (a small number of pinholes left in a membrane possibly affect the membrane performance significantly), this method is preferred over a gas adsorption method.



Jaap Vente (Head of ECN's Molecular Separation Group):

"Without the Permporometer we could never have adjusted the pore size of the revolutionary HybSi membrane as accurately".

The ECN Permporometer uses water vapor and helium instead of toxic chemicals such as hexane. Also, a dynamic measuring principle is used to execute the pore size tests within a short time period.

The program unit Energy Efficiency in Industry of ECN has used this device in the current set-up since 2003. The ECN Permporometer has proven to be a reliable and a valuable asset in the successful development of novel membranes.

The Permporometer is especially an essential tool for university based and institutional research groups, as well as manufacturers of porous membranes in the field of ultrafiltration, nanofiltration and molecular separation.

Measurable Properties

The standard ECN Permporometer offers the following capabilities:

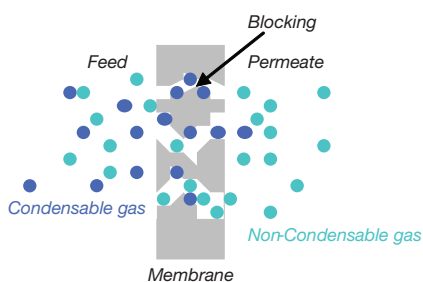
- Determination of intrinsic pore size distribution (0.3 - 50 nm, of which 0.3 - 2 nm can be reliably assessed and 2 - 50 nm can be determined), for defect free as well as non-perfect membranes, using water as the condensable phase
- Determination of defect permeance. This provides essential information to guide the research towards higher selectivity of membranes: intrinsic pore size distribution tuning or defect reduction
- Dry helium permeance.

Upon request, ECN can adapt the Permporometer to include the following features:

- Hexane as condensable phase
- Water and/or hexane permeance
- Various non-condensing gases including all conventional permanent gasses
- Separation Factor of condensable gas and permanent gas
- Ideal perm selectivity of the various non-condensable gasses.

Principle & Formula

The basic principle of Nano-PermPorometry is the relation between relative vapour pressure and the pore diameter where blocking of the pore by the vapour occurs (see figure).



This relation is described by the Kelvin equation:

$$d_K = - \frac{4\sigma \cos\theta}{\bar{v}RT \ln\left(\frac{p}{p_0}\right)}$$

In this equation d_K is the Kelvin diameter, $\bar{v}$ is the molar volume, σ the liquid surface tension, θ the contact angle, p the vapor pressure and p_0 the saturated vapour pressure, R the gas constant and T the temperature. This relation is valid above 2 nm and yields an accurate value for the pore size. Further corrections are possible using the thickness of a so-called t -layer. At smaller pore diameters, the Kelvin relation provides only a qualitative estimate of the pore size distribution.

Features

- The method is non-destructive and occurs at low pressures
- Automated tests running overnight are possible
- Short test times are possible
- Adsorption and desorption runs can be performed to detect hysteresis effects
- Permeance of dry membranes can be measured
- Defect flow can be detected accurately
- Hydrophilic and hydrophobic membranes can be tested.

Application

The Permporometer is suitable for both micro- and mesoporous membranes in plate and tube shapes.

Technical specifications

Pore size: 0.2 - 50 nm

Permeability: 10^{-10} – 10^{-5} mol/m².s.Pa

Max.Sample size:

Tubes: diameter 45 mm cross, length: 100mm

Flat plate: 70x20 mm²

Power supply: AC230V 50Hz

Contact details

For more information please contact:

Dr. Jaap Vente

ECN Energy Efficiency in Industry

e-mail: vente@ecn.nl

telephone: +31 224 564916

web: www.ecn.nl/en/serviceloket/