

Hydrophobic Pore Structure Characteristics of Fuel Cell Components Using Vacuapore

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Abstract: *Important pore structure characteristics of hydrophobic fuel cell components such as Pore volume, pore diameter, pore volume distribution, effects of compressive stress on pore structure characteristics and in-plane pore structure are not measurable by available nonwetting liquid intrusion techniques. A novel Vacuapore technique has none of the disadvantages of the available techniques and can measure all the characteristics.*

Keywords: Pore Volume; Pore Diameter; Pore Distribution; Hydrophobic Pores; Effects of Compressive Stress; In-Plane Pore Structure.

Characterization Requirements

Structural characteristics of hydrophobic pores of fuel cell components determine water management, transport of reactants, reaction rates, and flow rates of reaction products. Fuel cell components are also subjected to considerable compressive stress, which influences the pore structure characteristics [1]. The primary relevant pore structure characteristics of hydrophobic materials used in fuel cell applications are pore volume, pore diameter, pore volume distribution, in-plane pore structure, and effects of compressive stress on pore structure. It is important to be able to measure these pore structure characteristics so that optimum and efficient fuel cells may be designed.

The Technique

Principle: For measurement of pore volume, the sample is surrounded by a nonwetting liquid. Nonwetting liquids can not spontaneously enter the pores of the sample because the sample-nonwetting liquid surface free energy is greater than the sample-vapor surface free energy. Application of pressure on the nonwetting liquid results in the intrusion of the nonwetting liquid into the pores of the sample. The measured decrease in volume of the nonwetting liquid is the pore volume. The measured differential

pressure on the nonwetting liquid yields the pore diameter [2].

$$D = -4 \gamma \cos \theta / (p - p_g) \quad (1)$$

where D is pore diameter, γ is the surface tension of the nonwetting liquid, θ is the contact angle of the nonwetting liquid with the sample, p is the pressure on the nonwetting liquid, p_g is the pressure of trapped gas in the pore, and $(p - p_g)$ is the differential pressure on the wetting liquid.

Limitations of Available Characterization

Techniques: Mercury intrusion porosimetry has been used to characterize pore structure of hydrophobic materials. In this technique, the sample is evacuated and mercury is employed as the nonwetting liquid. Equation 1 is used for computation taking p_g to be zero. This technique requires very high intrusion pressures. It is not possible to accurately apply small intrusion pressures for characterization of large pores, which can not be accurately measured. Also, large intrusion pressures compress small pores. Therefore, diameters and volumes of small pores are not accurately measurable and volume of large pores becomes higher. This technique can not measure the in-plane pore structure and the effects of compressive stress on pore structure.

Water Intrusion Porosimetry or Aquapore technique has also been used to measure pore volume of hydrophobic pores. In this technique, water, which is nonwetting to hydrophobic materials, is used as the intrusion liquid. This technique has the advantage of using very low intrusion pressures. Thus, the undesirable effects of high intrusion pressure are avoided. However, the sample is not evacuated. Therefore, the gas which remains trapped in pores prevents measurement of pore volume and diameter of large pores, which require small intrusion pressures. This technique can not measure the in-plane pore structure and the effects of compressive stress on pore structure.

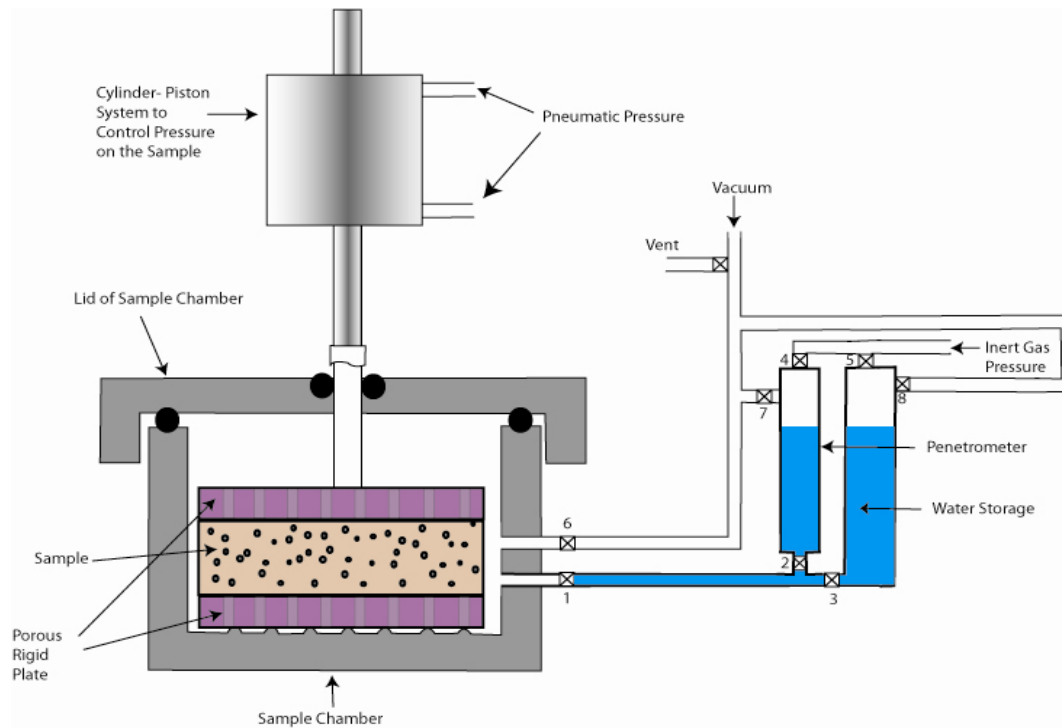


Figure 1. Important features of the Vacuapore

The Novel Vacuapore Technique

In the novel vacuapore technique, water is used as the nonwetting liquid for characterization of hydrophobic materials, so that the intrusion pressure is low and the drawbacks associated with the use of high intrusion pressures are avoided. The sample is evacuated so that the limitations due to air trapped in the pores are eliminated. A novel instrument design is incorporated so that it becomes possible to test effects of compressive stress on pore structure and to measure the in-plane pore structure.

The Instrument: The layout of the instrument is shown in Figure 1. The sample is held between two rigid plates with large holes. The bottom plate is placed on corrugated bottom of the sample chamber. The top plate is connected to a rod that passes through a pressure tight seal in the lid and is linked to a piston-cylinder device pneumatically operated for accurately applying controlled compressive stress on the sample. This arrangement permits sample to be kept under desired compressive stress of zero or higher and intrusion of water in to pores of the sample from all sides. The sample chamber is in communication with a penetrometer through valves 1 and 2. On application of pressure on water in the penetrometer, water intrudes in to pores of the

sample. The decrease in the volume of water in the penetrometer is the pore volume. For measuring the change in the volume of water, the change in the position of a magnet floating on water in the penetrometer is measured. The penetrometer is filled with water from a water reservoir connected to the penetrometer through valve-3. The penetrometer and water reservoir are connected through valve-4 and valve-5 respectively to a gas line capable of supplying an inert gas at an adjustable controlled pressure for pressurization of water. The sample chamber containing the sample is in communication with a vacuum line through valve-6. The vacuum line is also in communication with the penetrometer through valve-7, and to the storage vessel through valve-8 for evacuation above the water level. A vent valve is provided in the vacuum line. This unique design permits evacuation of sample chamber and the space above water in the penetrometer to be performed independently and separately so as to permit elimination of problems due to trapped air in the system. In order to create small sub-atmospheric pressures, a special device called vacuum regulator is used for controlled release of vacuum and creation of the desired sub-atmospheric pressures on water.

Procedure: The sample is loaded in the clean sample chamber. The sample chamber cap is sealed. Air pressure is used to operate the piston-cylinder device to apply desired compressive stress on the sample. The sample chamber is evacuated. The penetrometer and water storage are also evacuated. Water is

allowed to enter the sample chamber. The pressure on water is increased. The pressure on water and the decrease in volume of water in the penetrometer due to intrusion in pores are measured.

Application

Low Test Pressure for Intrusion: Figure 2 shows the change of cumulative pore volume with increase in differential pressure. The results from a mercury intrusion porosimeter are also shown in the figure for comparison with the results of the water intrusion test. The pressures for water intrusion are much less.

Through Pore and Blind Pore Volume: The pore volume shown in Figure 2 is the volume of through and blind pores because water intrudes both pores. Because of evacuation, the air trapped above water in the sample chamber is negligible and any increase in intrusion volume because of compression of the trapped air at higher pressures is also negligible. The initial intrusion pressures are close to the vapor pressure 0.3 psi of water at the test temperature of 18°C. The intrusion pressure is increased rapidly the vapor may not get time to condense. Therefore, even at intrusion pressure as low as 1 psi, at the most only a small part of the pore will be undetected because of vapor contained in the pore.

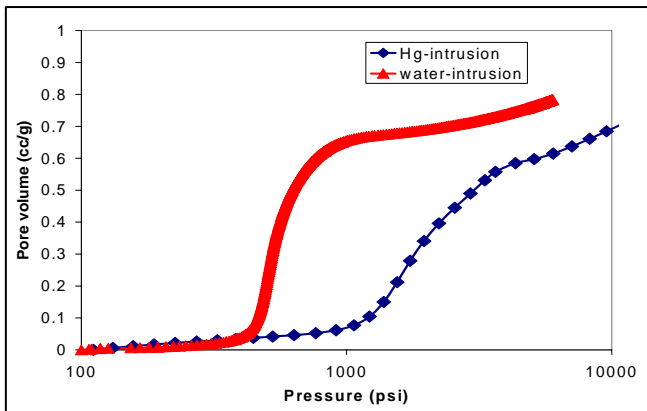


Figure 2. Change of pore volume with intrusion pressure for water intrusion and mercury intrusion

Through Pore and Blind Pore Diameter: The pore diameter is computed using Equation 1 (Figure 3). The pressure in the pore, p_g , used in this equation will be equal to or less than the small pressure of gas left in the pore after evacuation because part of the air left in the pores will be absorbed by the intruding evacuated unsaturated water. Because of short test duration, water is unlikely to have sufficient time to evaporate and

maintain its equilibrium vapor pressure in pores. If water is able to main its equilibrium vapor pressure of 0.3 psi during the normally short test, the pressure of air in the pore, p_g , will be 0.3 psi. This small pressure is mostly negligible and pore diameters are accurately measured. When intrusion pressure is increased, diameters of large pores may not be measurable if the vapor does not condense.

Through Pore and Blind Pore Volume Distribution:

The through pore and blind pore volume distribution is given in terms of the distribution function, $f_v(\log D)$ in terms of cumulative pore volume, V .

$$f_v(\log D) = - (dV / d \log D) \quad (2)$$

The area under the distribution function in any pore diameter range is the volume of pores in that range (**Figure 4**).

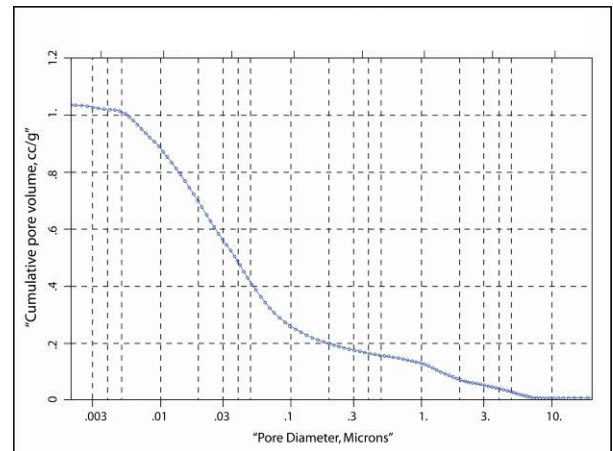


Figure 3. Change of cumulative pore volume with pore diameter

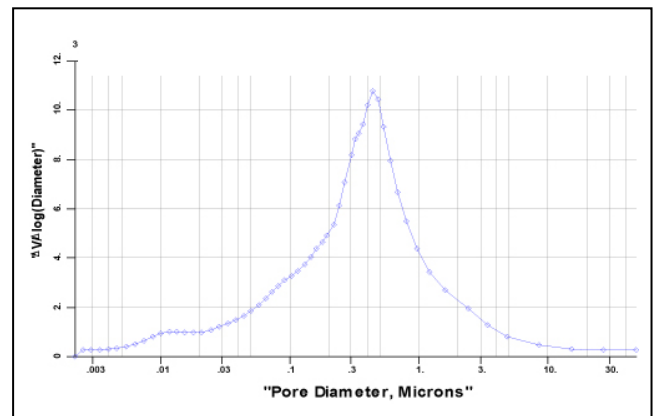


Figure 4. Pore volume distribution

In-Plane Pore Structure: The sample is sandwiched between two rigid nonporous plates having no holes. Therefore, water radially intrudes the pores in the sample in the x-y plane. The pore structure of radial pores are evaluated.

Effects of Compressive Stress on Pore Structure: Sample is put under desired compressive stress and pore structure is characterized. Thus, the influence of compressive stress on pore structure is evaluated.

Summary & Conclusions

A novel technique, Vacuapore, has been described. It can measure pore volume, pore volume

distribution, in-plane pore structure, and pore structure of the in-plane. Results have been presented to illustrate the application

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