

PORE STRUCTURE CHARACTERIZATION OF CERAMIC HOT GAS FILTERS

Akshaya Jena and Krishna Gupta
Porous Materials, Inc., 83 Brown Road, Ithaca, NY 14850

ABSTRACT

Ceramic hot gas filters normally have a highly porous support and a coating. The pore structure of the coating determines the flow rates of the gas through the coating and the particle sizes to which the filter acts as a barrier. Pore volume and other relevant flow properties of coating could not be determined by porosimetry. Flow porometry was used. The pores were filled with a wetting liquid and air was allowed to empty the pores and flow through the pores. Pressure and flow measurements yielded all the pore characteristics required for flow.

INTRODUCTION

Ceramic filters are used as hot gas filters because of their inertness, stability and cost effectiveness. The filters are normally composites having a highly porous support and a coating. The substrate provides structural support without increasing flow resistance. The pore structure of the coating determines the flow rates of the gas through the coating and the particle sizes for which the filter acts as a barrier. Therefore, the largest pore size, the constricted pore size, the pore size distribution and the gas permeability of the filter are important characteristics that govern product performance. .

Porosimetry is often used to characterize the pore structure of filters. However, porosimetry was not very effective for accurate determination of pore volume and pore volume distribution of thin coatings. Moreover, porosimetry can not measure permeability and the constricted size of the pore, which determines the barrier properties of a filter. The technique uses the toxic liquid mercury at very high pressures. The novel technique, capillary flow porometry was used to characterize the filter and all the required flow properties of the filter were measured.

TECHNIQUES

Porosimetry

Mercury is non-wetting to most materials because the solid/liquid surface free energy is much higher than the solid/gas surface free energy. Mercury cannot flow into pores spontaneously, but mercury under pressure can be forced into pores. Intrusion volume of mercury is the pore volume. Pore size is obtained from pressure. Equating work done due to displacement of mercury at a location in the pore to the increase in surface free energy:

$$p = - \gamma \cos \theta (dS/dV) \quad (1)$$

where p is pressure, γ is the surface tension of mercury, θ is the contact angle of mercury, dV is incremental intrusion volume in the pore and dS is the corresponding incremental solid/liquid surface area. Pore diameter D is defined as the diameter of a cylindrical opening such that (dS/dV) of the cylindrical opening is equal to the (dS/dV) of that part of pore into which intrusion has occurred. Hence:

$$D = - 4 \gamma \cos \theta / p \quad (2)$$

Sample was placed in the sample chamber and evacuated. Pressure and intrusion volume were measured with better than 0.25 % accuracy. The surface tension and contact angle of mercury were 480 dynes/cm and 140° respectively.

Capillary Flow Porometry

In this novel technique a wetting liquid is allowed to fill all the pores in the sample spontaneously. A non-reacting gas under pressure forces the liquid out and flows through the pore. Accurate measurement of flow rate and pressure are used to compute all the pore characteristics for flow. The procedure [1] for deriving the relation between pressure and pore diameter is similar to that used for Equation 2.

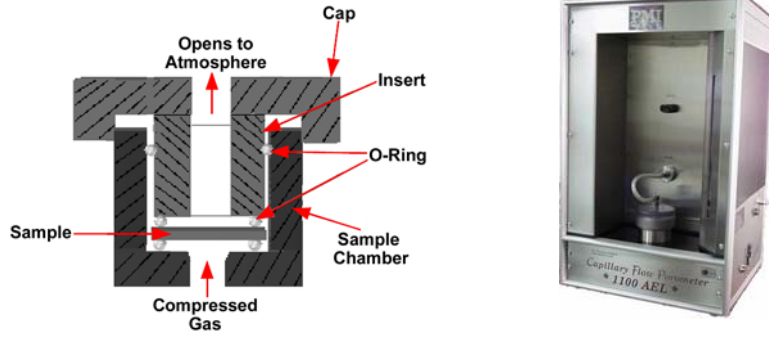
$$D = 4 \gamma \cos \theta / p \quad (3)$$

In case of low surface tension wetting liquids $\cos\theta$ is close to one [2]. Consequently, Equation 3 reduces to:

$$D = 4 \gamma / p \quad (4)$$

The sample to be tested was soaked in the wetting liquid porewick and placed between two o-rings in the sample chamber. Air pressure under the sample was slowly increased. Special design of the sample chamber (Figure 1a) allowed air to

pass through the sample and escape to the atmosphere. The fully automated instrument (Figure 1b) containing state-of-the-art components and many novel design features accurately measured flow and pressure and yielded highly reproducible results [2]. Equation 4 was used with surface tension of 16 dynes/cm.

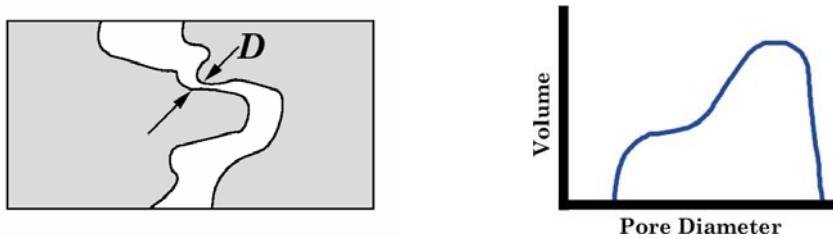


(a) Sample chamber (b) Instrument
Figure 1. The capillary flow porometer.

RESULTS AND DISCUSSION

Porosimetry

Pore size: Size of a pore varies along its length. Therefore, pressure required to intrude mercury into a pore varies according to Equation 2 and a single pore is detected as many pores having a range of diameters. The sketches in Figure 2 illustrate the expected results.



(a) Pore (b) Range of pore diameter

Figure 2. Detection of a pore as many pores with a range of pore diameters.

Pore volume: The volume of intruded liquid is the pore volume. Variation of total pore volume of the filter with pore diameter is shown in Figure 3. The pore volume is due to pores in the support as well as in the coating. It is not possible to separate the characteristics of pores of the coating from those of the support.

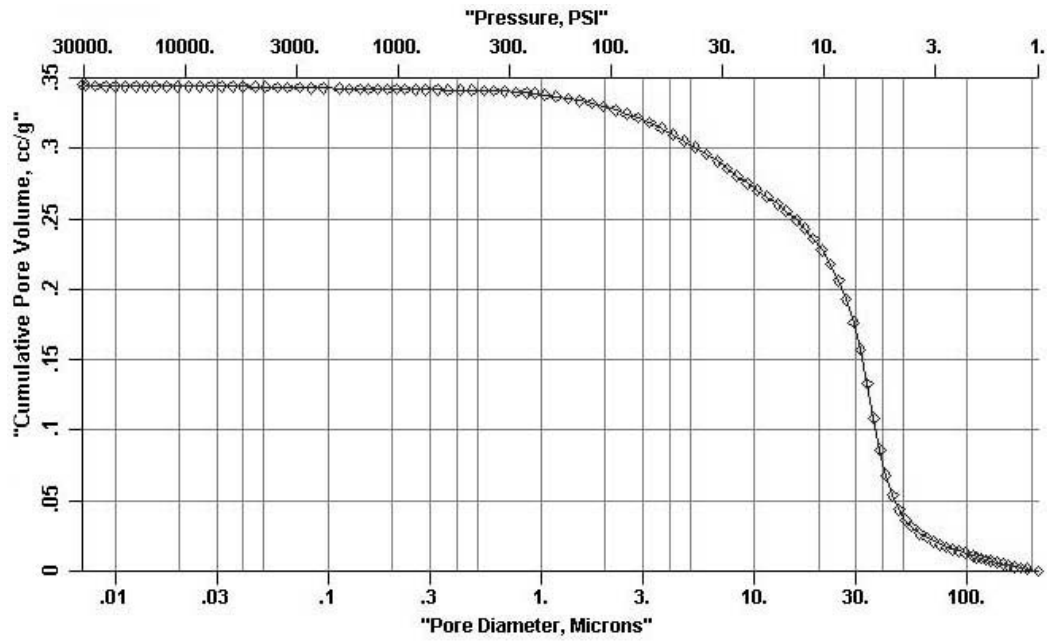


Figure 3. Change of intrusion volume with pore diameter.

Pore volume distribution: The measured pore volume distribution is presented in Figure 4. This figure also does not distinguish the characteristics of pores in the support from those in the coating.

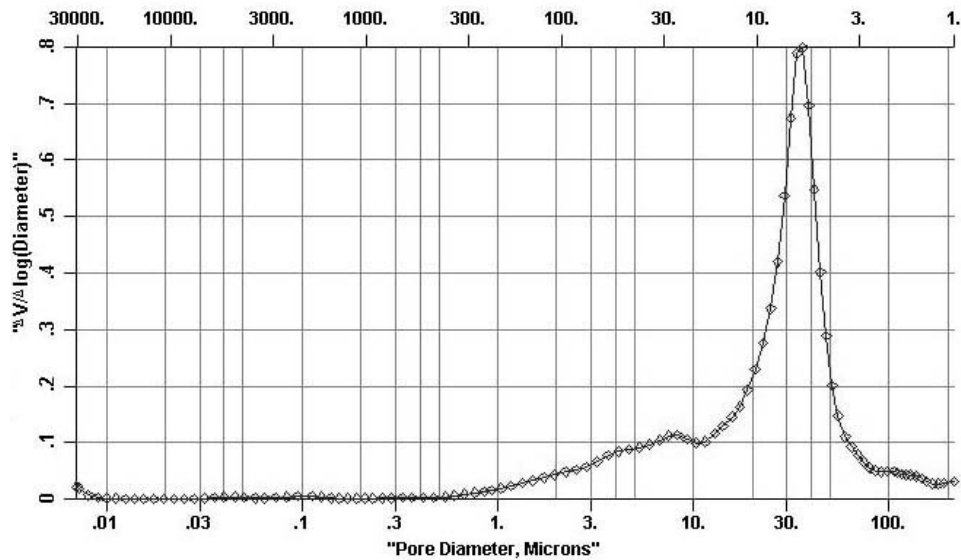


Figure 4. Pore volume distribution.

Flow Porometry

Pore size: Gas pressure required to displace liquid in the pore varies along the length of the pore because of changing pore size. The pressure for displacement of liquid is the maximum at the most constricted part of the pore. Therefore, only when this maximum pressure is reached, liquid is completely removed from the pore, gas increases its flow rate by flowing through the pore and the presence of the pore is detected by the instrument. Thus, each pore is measured as a single pore with a pore diameter equal to the pore's most constricted pore size (Figure 2 a). The pores in the coating are much smaller than the pores in the support. Therefore, the pores in the coating represent the constrictions in the through pores and pore diameters measured by flow porometry are the diameters of constricted parts of pores in the coating.

The largest pore diameter: Figure 5 shows the variation of flow rate with pressure for the sample wetted with porewick. Equation 4 suggests that the pressure at which flow starts, corresponds to the largest pore diameter. The pores measured by flow porometry are in the coating. Therefore, the largest pore in the coating is 83.4 micrometers in diameter.

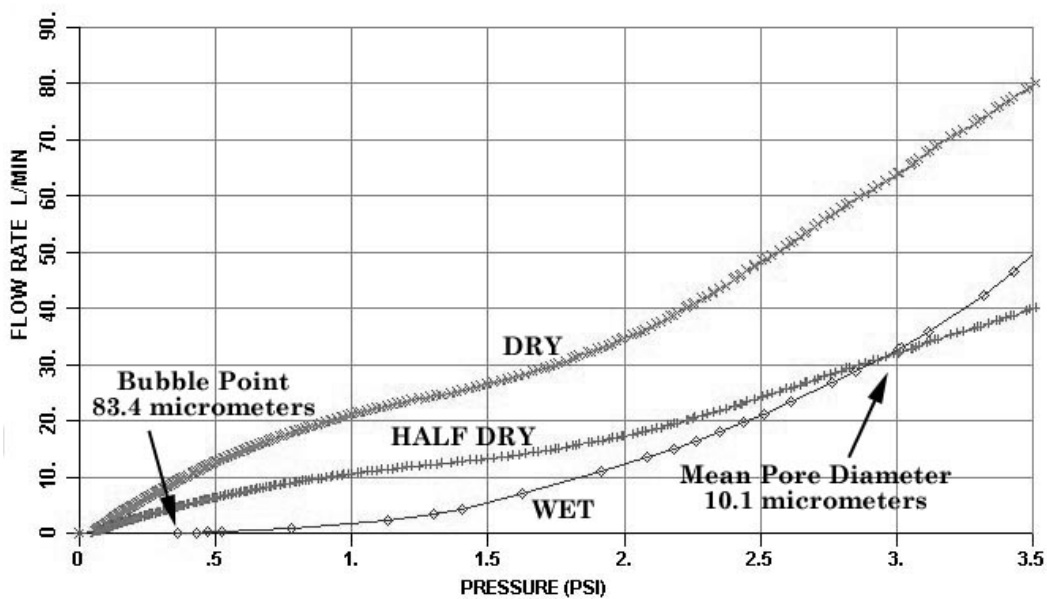


Figure 5. Variation of flow rate with differential pressure

The mean pore diameter: Figure 5 also shows variation of flow rate with pressure for a dry sample and a half-dry curve which is computed from the dry curve to yield half of the flow rate through dry curve at a given differential pressure. The intersection of wet curve and half-dry curve gives the mean flow pressure that corresponds to mean pore diameter. Pores smaller than the mean pore diameter are responsible for half of the flow. The mean pore diameter of the coating is 10.1 micrometers.

Pore size distribution: Equation 5 defines the pore size distribution function, f .

$$f = d[100 \times (F_w / F_d)] / dD \quad (5)$$

where F_w and F_d are flow rates through wet and dry samples respectively at the same differential pressure. The distribution function computed from data in Figure 5 is presented in Figure 6.

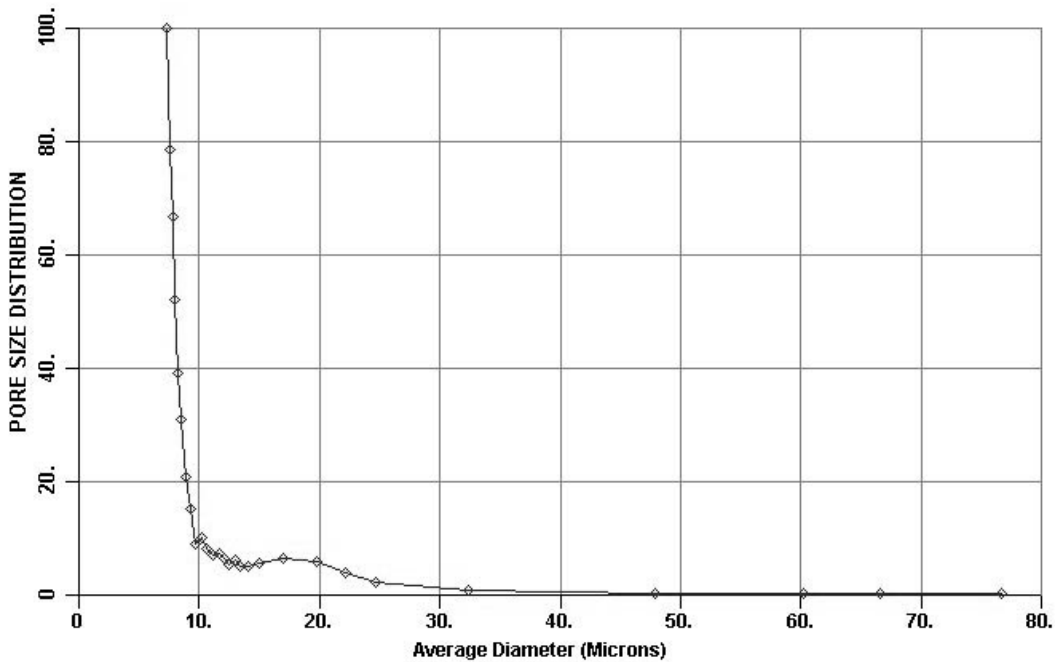


Figure 6. Pore distribution by porometry

The distribution function is such that the area under the curve gives the percentage flow through pores in any pore size range (Equation 6). The distribution is of pores in the coating.

$$[100 \times (F_w/F_d)]_{D1} - [100 \times (F_w/F_d)]_{D2} = - \int_{D1}^{D2} f dD \quad (6)$$

Gas permeability: Permeability, k of air through the sample is given by the following equation.

$$k = (q \mu l p_o) / (p a p) \quad (7)$$

where q is flow rate in volume at standard temperature and standard pressure p_o , μ is viscosity, l is sample thickness, p is average pressure and a is sample area. Substituting the flow rate through dry sample at 100 psi differential pressure and other parameters, permeability is computed to be $0.148 \times 10^{-9} \text{ cm}^2$ (0.015 darcies).

Comparison of Techniques

The results obtained in this study demonstrate the ability of porosimetry and porometry to determine characteristics of coatings of hot gas filters. Table 1 summarizes the capabilities of the two techniques.

Table 1 Comparison of porosimetry and porometry for characterization of coating

Characteristic of coating	Measurable by	
	Porosimetry	Porometry
The largest pore diameter	no	yes
Mean pore diameter	no	yes
Pore size distribution	no	yes
Permeability	no	yes
Pore volume	no	no
Pore volume distribution	no	no

Flow porometry has a number of advantages over porosimetry. The toxic mercury used in porosimetry is not used in flow porometry. Dry air at low pressure is used for flow. Rapid testing and high-speed data acquisition are other important features of porometry.

CONCLUSIONS

Hot gas ceramic filters consisting of a membrane and a support were investigated by porosimetry and flow porometry. Porosimetry could not measure any of the characteristics of the pore structure of the coating. Flow porometry measured coating's all relevant flow properties including the largest pore size, mean pore size, pore size distribution and permeability. Flow porometry had a number of advantages and was the appropriate technique for analysis of the pore structure of coatings on hot gas filters.

REFERENCES

- ¹A.K. Jena and K.M. Gupta, "In-plane Compression Porometry of Battery Separators", *Journal of Power Sources*, **80** 1&2 46-52 (1999).
- ²Vibhor Gupta and A.K. Jena, "Substitution of Alcohol in Porometers for Bubble Point Determination", *Advances in Filtration and Separation Technology*, **13b** 833-44 (1999).