



# Porous Problems: & Ceramics Structural Materials

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**P**orosity is one of those properties with "kill or cure" effectiveness. In the right hands, in the right product, it confers exquisite performance. In the wrong hands or inappropriate application it can stop the process/product, dead in its tracks! Porosity should be well understood, easily identified, quantifiable and controllable. Why is it then, that it can be so easily overlooked or misunderstood?

## A Porous Overview

Porous properties impact/confer on a ceramic, both positive and negative. Porosity is obviously desirable in light-weight products, thermal insulation, catalyst supports, wicking and filtration uses. Negative aspects include friability, loss of strength, undesirable fluid absorption etc.

The forming of ceramics from powders necessarily generates porosity by fixing, in 3-dimensions, positions and relationships of interparticle voids. Low pressure forming methods generate higher porosity and higher pressures produce less. Thermal treatment, be it simultaneous with forming (HIP) or subsequent to it (normal firing) is used to eliminate some, most or all porosity depending on end use. The size of the pores depends on the particle size distribution and shape of the starting powders and various additives (binders, liquids). Of course, the latter creates porosity in the initial stages of firing due to their loss or burning out. As sintering is initiated small pores may coalesce into larger ones with little or no loss of overall pore volume. Loss of pore volume (porosity) must be accompanied by shrinkage of the piece.

## Your Pore Problems Begin

Trouble begins by not putting in place a program of pore volume and size monitoring at all stages of production. From consideration of mass, true density and geometric (envelope) volume one might believe one has all the information needed... just calculate porosity. Maybe even determine it explicitly by water absorption. But not knowing the pore size distribution can lead to serious misunderstandings. You are probably familiar with looking at particle size distributions and making certain deductions. That's OK for loose powders, but even then one cannot deduce how homogeneous the blend is from a size analysis. But when the particles attain a fixed spatial order by pressing, casting, extrusion etc., the resulting pore size distribution plot will reveal the

homogeneity of the blend as it exited the forming process. If it's worth proceeding, you will now be able to understand the path along which the sintering process might take. Small pores will be the most thermally "reactive", larger pores less so. Therefore don't be surprised if the final piece still has porosity - if it started out with predominantly large pores. Remember, the final porosity is not just a function of initial porosity but also of pore size.

## Measuring Pore Size Quickly and Reliably

The best available measurement method is Mercury Intrusion Porosimetry: rapid (orders of magnitude faster than gas sorption) very wide dynamic range (orders of magnitude larger than gas sorption - unrivaled for pores over 0.25 microns) and yielding network structure (more evident than gas sorption because the fluid penetration is sequential or serial in nature). Mercury intrudes from the outside, in, and while there exists a pressure-pore size relationship, it is subject to the arrangement and connectivity of the different pores sizes in the network of internal voids. Therefore, the result not only yields pore size information but also structural or network information.

The forced intrusion of non-wetting mercury into a pore network by applying an external force has been used commercially for the best part of sixty years. So why is it not more widely used? Perhaps it was the lack of automation, the lack of interpretation skills or maybe noise and expense? But that is not now the case. Modern mercury intrusion porosimeters offer high throughput, very wide pore size range, a host of automated features and near-silent operation. Many analyses can be completed in fifteen minutes or less - on two samples at one time.

So, when you need pore size information for R&D, process control and quality assurance in all fields of ceramics, consider using the mercury porosimetry method. Mercury porosimetry may reveal porosity where there should be none, identify off-spec green ceramics and allow them to be removed from the remaining processes, and to optimize firing conditions. After all, kilns are not inexpensive to operate. ■

For more information about pore size and related measurement instruments, contact Quantachrome Instruments by phone: (561) 731.4999, fax: (561) 732.9888, email: [qc.support@quantachrome.com](mailto:qc.support@quantachrome.com) or visit [www.quantachrome.com](http://www.quantachrome.com).

## Mercury Intrusion Porosimetry Facts

Pore size is calculated using the Washburn equation:

$$\text{Pore diameter} = - (4\gamma \cos \theta) / P$$

Where  $\gamma$  = surface tension,

P = pressure and  $\theta$  = contact angle.

Pore size range is from approximately 1mm down to 3.5nm

Pore volume resolution better than 0.1 microlitre.

Pressures required as high as 60,000 psi, achieved using specially designed hydraulic pressure generators.

Featured in a number of standard test methods including ASTM D4284, ASTM D4404, ASTM D2873, UOP578-02, BS7591-1.

## Rid Yourself of Unwanted or Unexplained Porosity In Your Final Product.

The **PoreMaster GT<sup>®</sup>** uses the extremely effective rapid mercury intrusion technique. With automatic pore size and pore volume distribution analyzers, you will never have porosity inconsistency in your products again.

For answers to your porosity related questions and to speak to a Quantachrome consultant, call us at 561.731.4999 or email us at [qc.support@quantachrome.com](mailto:qc.support@quantachrome.com).



## PoreMaster GT<sup>®</sup>

### Advanced Features:

- Automatic air purge
- Vapor trap
- Extremely quiet

### Three Operating Modes:

- Pressure Scanning
- Autospeed
- Step-wise

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