Additive Manufactured Porous Metals
Pore Size Analysis with Mercury Intrusion Porosimetry

Introduction

Porous materials made by Additive Manufacturing (AM), also known as 3D printing, include e.g. building materials, ceramics, wood, and polymers while metal screens and metal foams are produced from metal powders. Porous metals can be used for filtering particles (diesel soot), as catalyst supports or heat exchangers. On the other hand, porous metals can be interesting as components with lower skeletal density and due to their potential weight saving.

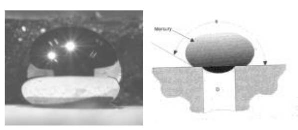
This porosity by purpose in additive manufacturing (AM) is different from porosity to avoid referring to the presence of tiny gaps or voids within the material structure of a printed part. These voids can occur between layers, within layers, or around material particles. Porosity affects the mechanical properties, surface quality, and overall integrity of 3D-printed components, making it a critical concern in the AM process.

Understanding and controlling the porosity of 3D-printed porous parts in additive manufacturing is vital for ensuring quality and performance, especially in industries where component reliability is critical, such as aerospace, automotive, and medical devices. A widely used technique for analyzing open and accessible pores is mercury intrusion porosimetry (MIP), in which the non-wetting mercury is forced into pores at ambient temperature with pressure up to 414 MPa. Any undesirable reaction of the porous metal with mercury can be prevented by passivation, e.g. by oxidation.

Basics of Mercury Intrusion Porosimetry

Mercury Intrusion Porosimetry is described in the ISO standard ISO 15901-1. The non-wetting mercury is forced into accessible pores at ambient temperature with pressures up to 414 MPa entering pores down to ca. 3.6 nm pore diameter (Figure 1). The pore volume corresponds to the intruded amount of mercury. According to the Washburn equation, the pore size (radius, diameter) is inversely proportional to the applied pressure.

$$r = -\frac{2\gamma\cos\theta}{p}$$



With pore radius *r* [m], surface tension of mercury *g*= 0.48 N/m, intrusion pressure *p* [Pa].
With a contact angle *q* =141.3° a simple estimation for the pore radius is obtained

$$r \approx 0.7500/p.$$

BELPORE - the Device for Safe Mercury Intrusion Porosimetry

The BELPORE mercury porosimeters can measure pore diameters from 3.6 nanometers up to 1 millimeter. They consist of 3 models: **BELPORE LP** as mercury filling device and for low pressures, **BELPORE MP** for medium-high pressures, and **BELPORE HP** for high pressures, details see Figure 1.
The BELPORE mercury porosimeters work according to the P.A.S.C.A.L. principle which is not only a pressure unit. In the BELPORE porosimeter, it means the equilibrium-controlled and optimized control of the pressure build-up by "Pressurization by Automatic Speed-up and Continuous Adjustment Logic". This automatic control is driven by the real pore system and allows shorter measuring times with guaranteed equilibrium conditions and detection of all pores within the specification, up to 20,000 measuring points per analysis. Pressure tables are thus superfluous. Since only two types of dilatometers (sample cells for powder or solids) are sufficient for most measuring tasks, and no gases or liquid nitrogen are required, running costs can be kept significantly lower. Other innovations include simplified operation of the BELPORE LP low-pressure porosimeter and employing the ultra-macropore dilatometer an extended measuring range up to pore sizes of 1 millimeter, applicable for thin membranes. Vertical degassing and filling with mercury on the BELPORE LP is not only fast but allows also the degassing pressure to be adjusted. Therefore, it is possible to measure moist samples without changing the material moisture. The 3-D evaluation software PoreXpert is optionally available. With this software, e.g. tortuousness, diffusion, percolation, and much more can be derived from porosimetry data.
A request from electrode manufacturers was incorporated, namely the compression correction and the bulk modulus value which is calculated from the compression.
BELPORE porosimeters are available in pressure ranges from 0.01 kPa to 450 kPa (and mercury filling), 0.1MPa to 228 MPa, or up to 414MPa (Figure 1). High-precision measurements are finished in less than 1 hour, for quality control it can go down to 9 minutes.

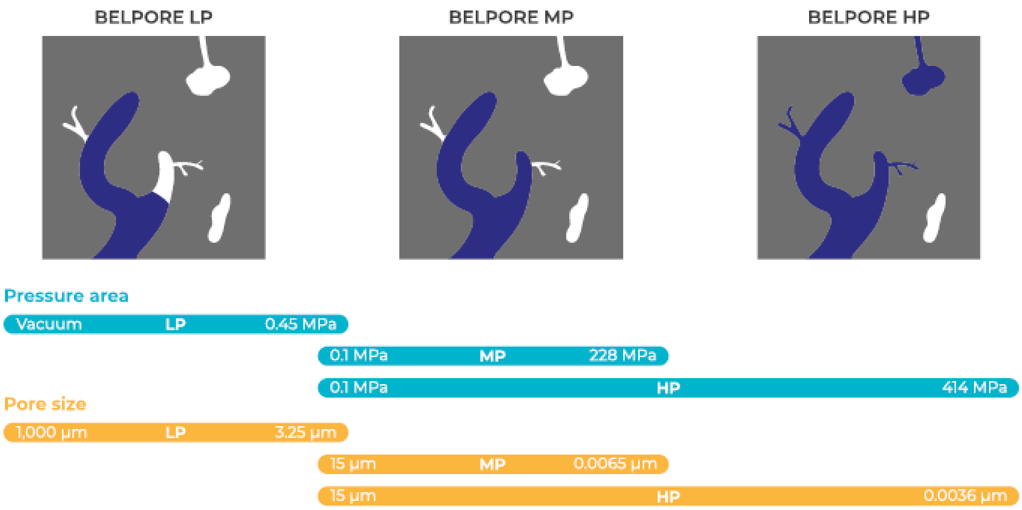


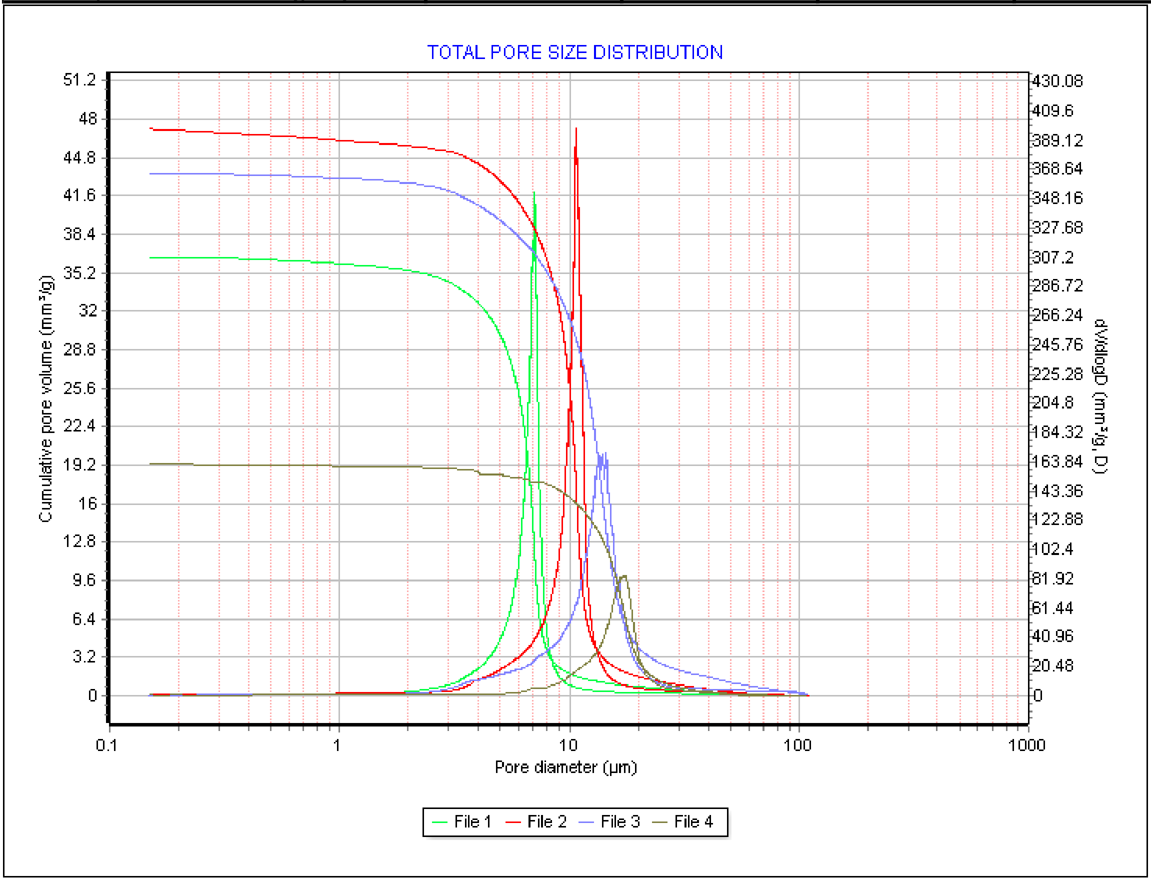
Figure 1. Pressure ranges and accessible pore sizes of the BELPORE porosimeters

Results

An example of the pore size distribution of sintered metal sieves and polymer-metal mixtures as used for 3D printing is shown below in Table 1 and Figure 1. Since compression effects occurred, in particular for the metal-polymer mixes during the measurements up to 414MPa, the maximum pressure for data evaluation was set to 10MPa. Therefore, all relevant pores with diameters larger than 150nm are included.
The effect of the addition of polymer can be seen from the decrease in density (files 3 and 4). Further, the modal pore diameter was adjusted to larger sizes up to ca. 13µm and 17µm for the polymer-metal mixes, while the pure metal sieves exhibit smaller pore diameters between 7µm and 11µm.
Remark to Amalgam Reactions: The mercury used in these measurements with metals could not be recycled in the lab with the Mercury siphon due to the risk of amalgam formation.

Table 1: Pore size distribution results of sintered metal resp. metal-polymer sieves

RESULTS	File 1	File 2	File 3	File 4
Total intruded volume (cm³/g)	0.036	0.047	0.044	0.019
Apparent density (g/cm³)	8.442	9.437	4.267	1.832
Porosity by Hg intrusion (%)	23.530	30.810	15.660	3.420
Modal pore diameter (µm)	7.060	10.698	13.408	17.340



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Conclusion

The use of Mercury Intrusion Porosimetry (MIP) is essential for analyzing the pore structures of additive-manufactured porous metals.

The BELPORE porosimeters, with their advanced features and wide pressure ranges, provide accurate and comprehensive measurements of pore sizes and volumes. This technology ensures optimal sieve performance by enabling precise control over the pore characteristics of the materials used. The results demonstrate significant differences in pore volumes and sizes between the various polymer-metal powder mixes.

Overall, the BELPORE porosimeters offer a reliable and efficient solution for the analysis of porous AM metals.

BELPORE analysis of additive manufactured AM metal sieves stands for

- **Mercury Intrusion Porosimetry according to ISO 15901-1**
- **Fast and accurate measurements of pore size and volume.**
- **Safe and easy handling**
- **Small lab space**
- **Advanced results for improving metal sieve performance**