The Development of Micro-porous Metal – Ceramic Membrane Filters – 9250

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ABSTRACT

Ellipsis and Porvair Filtration Group (PFG) are commercializing sintered 316L stainless steel powdered metal filters with a metal – ceramic membrane layer capable of absolute filtration to 0.5 µm. By the broadest definition, microfiltration includes devices able to separate particulates between 0.1 and 10 microns. Most porous metal filters are incapable of removing particles smaller than 4 µm. The Sinterflo® filters under development will improve filtration by almost an order of magnitude. Scanning electron photomicrographs (SEMs) and porometry, bubble point, permeability and efficiency data for both gaseous and liquid environments are presented.

INTRODUCTION

There are three basic types of metal filters – sintered powder, sintered fiber and sintered woven wire mesh. While polymeric filters are capable of removing particulates from either a gaseous or liquid medium as small as .001 µm, most porous metal filters are incapable of removing particles smaller than 4 µm. With many applications in the nuclear, chemical process and hot gas (e.g. coal gasification & pressurized fluidized bed combustion) industries striving to remove particles smaller than 1 µm in harsh environments including extreme temperatures, the need for improved metal filters is a necessity.

In addition to micron rating or pore size, other critical factors that need be addressed when specifying filters for a given application include pore size distribution, permeability and the ability to clean filters *in-situ*. While most conventional metal filters rely on tortuosity or entrapment to remove particles (depth filters), membrane filters are surface filters that easily allow for back-washing or back-flushing. Using proprietary manufacturing processes, several layers of closely controlled mixed 316L stainless steel and tungsten carbide particles are deposited either on the OD or ID of a conventional sintered tubular filter or on the surface of a conventional sintered sheet filter such that the conventional filter is now the support structure while the sintered membrane layer becomes the active filter that is now easily back-flushable.

RESULTS AND DISCUSSION

The original research & development relating to this unique filtration technology was accomplished via a grant from the Department of Energy and the Colorado Advanced Materials Institute (CAMI) with sample definition and fabrication carried out by the primary author in conjunction with Dr. Steve Landin now with CoorsTek, Golden, Colorado and analytical activities under the direction of Professor emeritus William Krantz, Department of Chemical Engineering and Professor Alan Greenberg, Department of Mechanical Engineering, University of Colorado, Boulder. The objective at the time was to produce a sub-micron robust metal filter capable of removing radioactive actinides from laundry waste water at the DOE Rocky Flats facility in Colorado. This facility is now decommissioned.

Structure

When using only spherical stainless steel spheres for the membrane, filters having an absolute rating of 1µm or less could not be achieved. By adding tungsten carbide particles, a ceramic material that could fit into the interstices of the 316L SS spheres and be simultaneously co-sintered, absolute sub-micron filters could be achieved. The technology is protected by a US Patent[1].

Figure 1 elucidates how the membrane is positioned over and sintered onto a conventional powder metal filter. The sample was fractured rather than cut and polished to provide better definition.

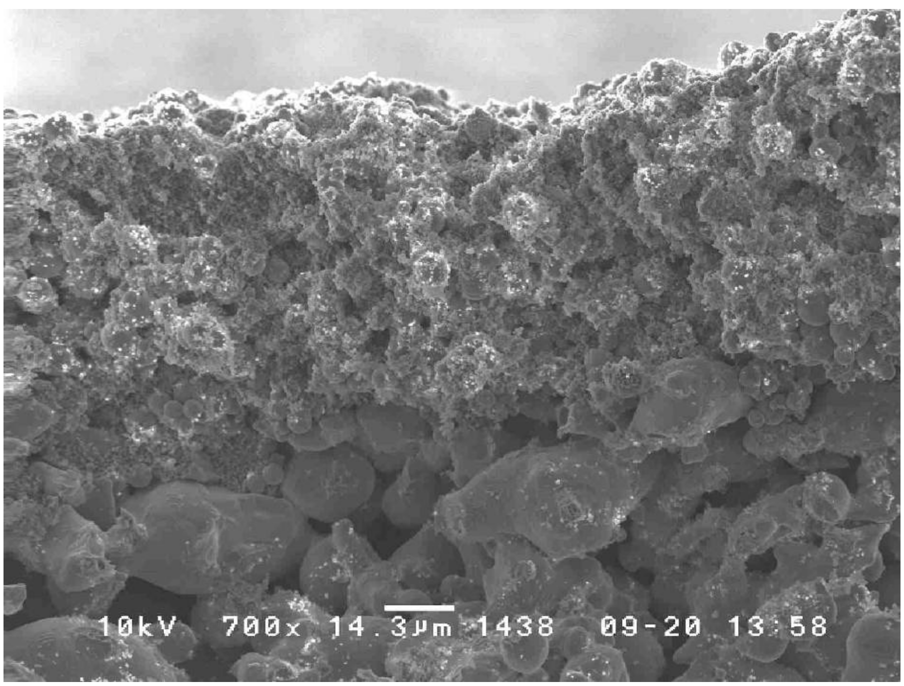

Fig. 1. SEM image of fractured membrane showing the sintered powder interface.

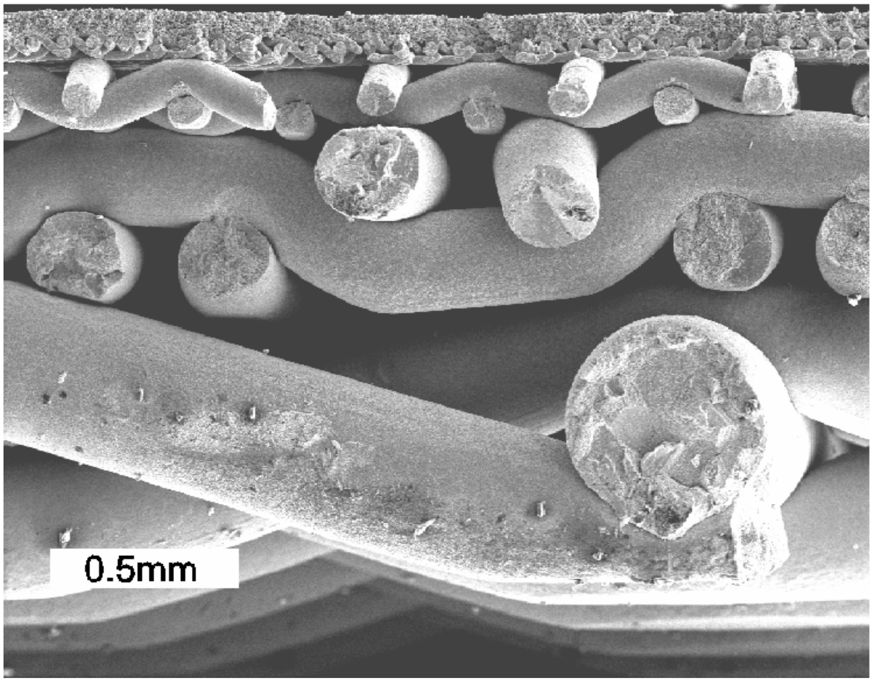

Fig. 2 SEM image of a fractured membrane showing the sintered wire mesh interface.

Figure 2 is provided to demonstrate a better perspective of the relative thicknesses of membrane and substrate.

Absolute vs. Depth Filtration

For a filter to be a true absolute filter, it is imperative that the pore-size distribution be held to a minimum. The experimental filter shown below in Figure 3 shows essentially all pores smaller than 0.5 µm and most clustered between 0.3µm and 0.4µm.

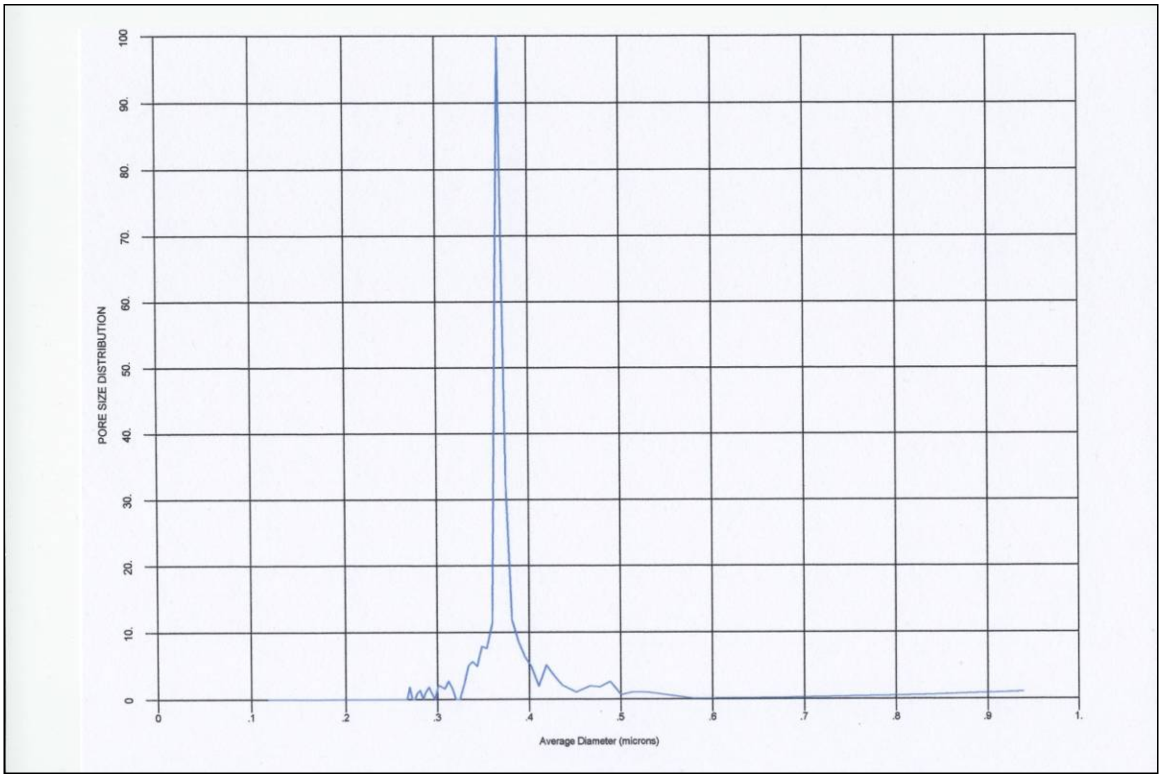

Fig. 3. Pore size and pore size distribution as measured by porometry.

Back-Flushing or Blow-Back

To demonstrate the back-flushing or blow-back capabilities of these surface membrane filters, a cyclical experiment was setup whereby ultra-fine dust was impinged onto the surface of the filter, then back-pulsed to determine the extent of particulate containment. As noted below in Figure 4, virtually all of the ultra-fine dust (UFD) was expelled from the surface of the membrane filter. Compositionally, greater than 97% of the UFD volume had particles 10 µm or less; 100% were 20 µm or less.

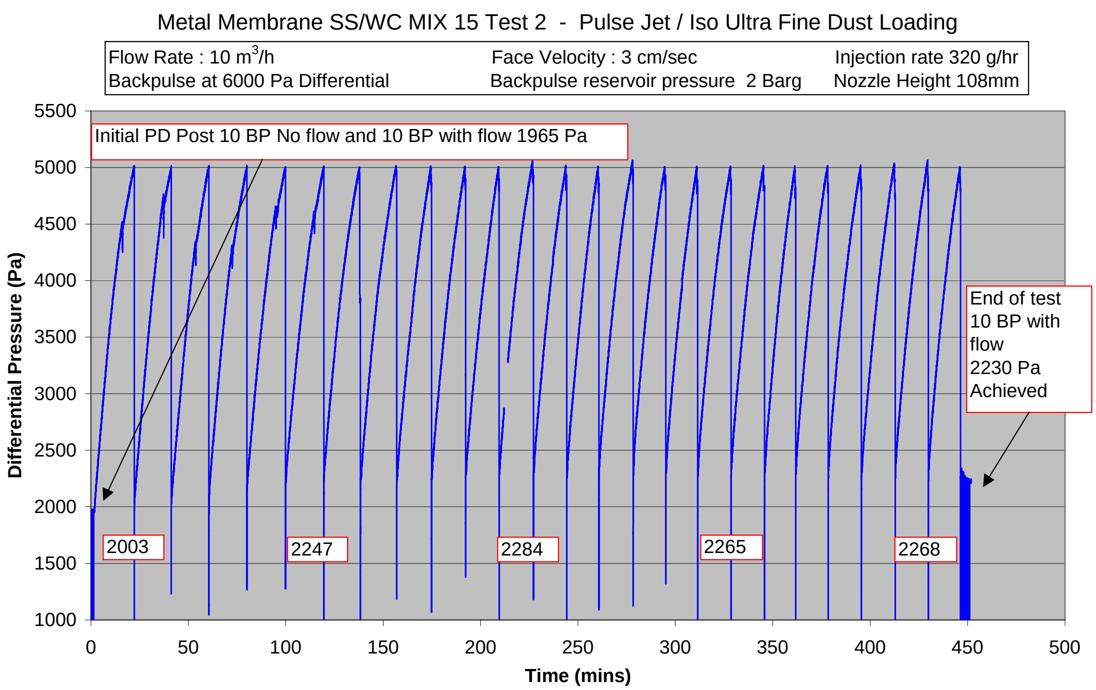

Fig. 4. Blow-back chart for filter cleaning showing back-pulsing for removal of ultra-fine dust.

Efficiency

To provide quantitative data regarding the efficiency of the membrane filters, a smoke generator was used to generate 0.3 µm dioctylphthalate (DOP) particles. The DOP smoke was next impinged onto the surface of a cylindrical membrane filter at varying flow rates. In all cases the efficiency for DOP particle removal was 99.999%

Permeability

Figure 5 graphs comparative differential pressure (y axis) vs. flow rate (x axis) for a standard filter without a membrane coating (average) vs. a thicker walled filter without a membrane coating to achieve a 3 µm nominal rating (3µm SMP) vs. standard filters with membrane coatings. The graph defined as Medium (**yellow)** has a 3 µm absolute rating. As one would anticipate, as the absolute pore size decreases the permeability decreases as well. One note of interest is that the 3µm membrane filter has superior flow characteristics with a much tighter controlled pore size distribution than the 3 µm SMP sample.

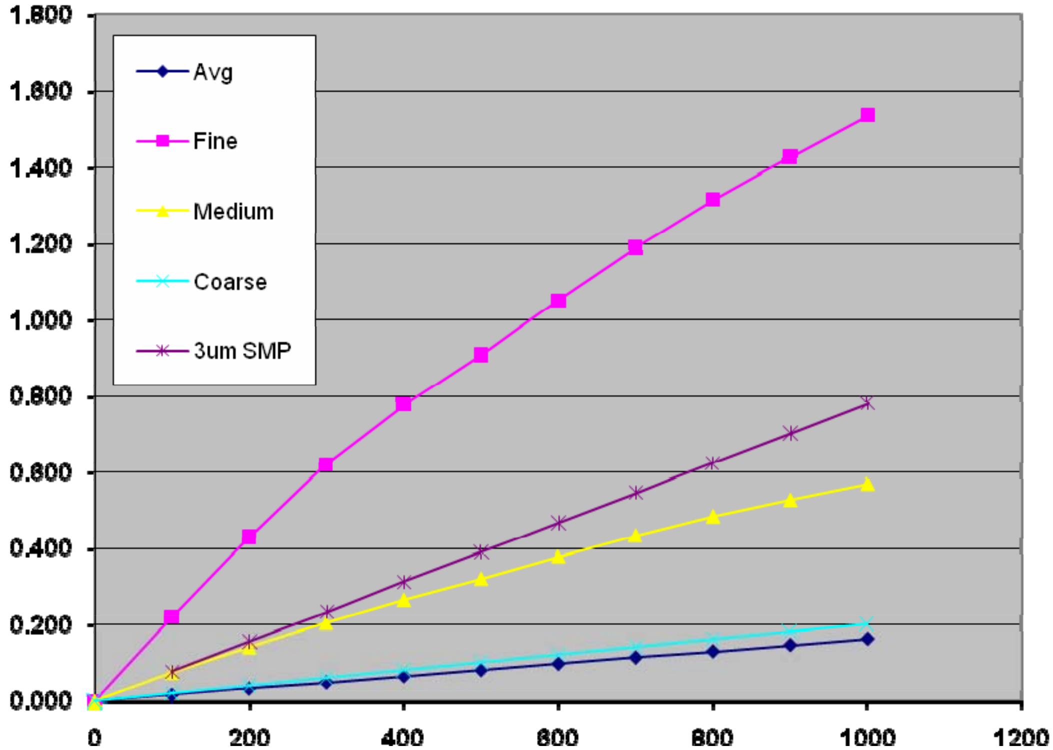

Fig. 5. Differential pressure vs. flow rates for standard vs. membrane filters.

CONCLUSIONS

As regulatory agencies impose ever more stringent requirements for the control of emitted particles from either gaseous or liquid mixtures, especially in hostile environments, the need for sub-micron filters capable of operating at temperatures of 500^0C and beyond or in noxious or hazardous environments will become more and more apparent. As precious metal catalysts become ever more expensive and are used in greater amounts, finer filters will definitely be considered as alternatives for higher yield recoveries. By sintering a metal – ceramic membrane to the surface of a conventional sintered powder metal filter, different pore sizes can be achieved meeting a variety of industrial requirements and challenges.

REFERENCES

[1] US Patent #6,309,546   Herrmann et al