

mott *corporation*

84 Spring Lane, Farmington, CT 06032-3159
860-747-6333 Fax 860-747-6739
www.mottcorp.com

SINTERED METAL MICROFILTRATION MEDIA

Dr. Kenneth L. Rubow
Dr. Sunil Jha
Mott Corporation

Presented at the
Seventeenth Membrane Technology/Separations Planning Conference

Newton, MA

December 6-7, 1999

INTRODUCTION

Media for microfiltration, i.e., for filtration of 0.1 to 2 μm size particles in liquids are available commercially in polymers, ceramics and metals. Polymeric membranes can not be used in high chloride environments, extreme pH environments, at temperatures in excess of approximately 200°F and high pressures. Solvents attack polymeric membranes (Cheryan, 1998). The applicability of polymeric membranes is also limited in slurries containing high solids concentration, where the failure may be due to abrasion, or high pressure. The ceramic membranes resolve many of these difficulties. Ceramic membranes are strong but brittle, and must be sealed using polymeric seals. The temperatures and corrosive environments that the seals can withstand limit the use of ceramic membranes. Ceramic membranes have low fracture toughness, therefore, back-pulsing or thermal cycling may introduce cracks, and the cracks may propagate rapidly leading to final brittle failure. Sintered metal media address the concerns described above with ceramic and polymeric membranes. Metal media have higher fracture toughness, high thermal shock resistance and are completely weldable. The sintered metal media are available in different alloys to handle wide-ranging corrosion environments.

Disposal of polymeric and ceramic media often poses an environmental problem, and disposal of contaminated media is difficult and expensive. The sintered metal filters are cleanable, either in-place or by external chemical cleaning. Depending on the nature of the contaminant, the sintered metal filters are completely recyclable.

This paper describes the physical and mechanical properties of 0.1 μm grade sintered metal media in Hiflow™ Nickel, 316L Stainless Steel and Hastelloy C-22. New manufacturing technology developments have enabled the manufacture of highly permeable sintered metal media, with fine porosity and high pore volume fraction.

SINTERED METAL MICROFILTRATION MEDIA PROPERTIES

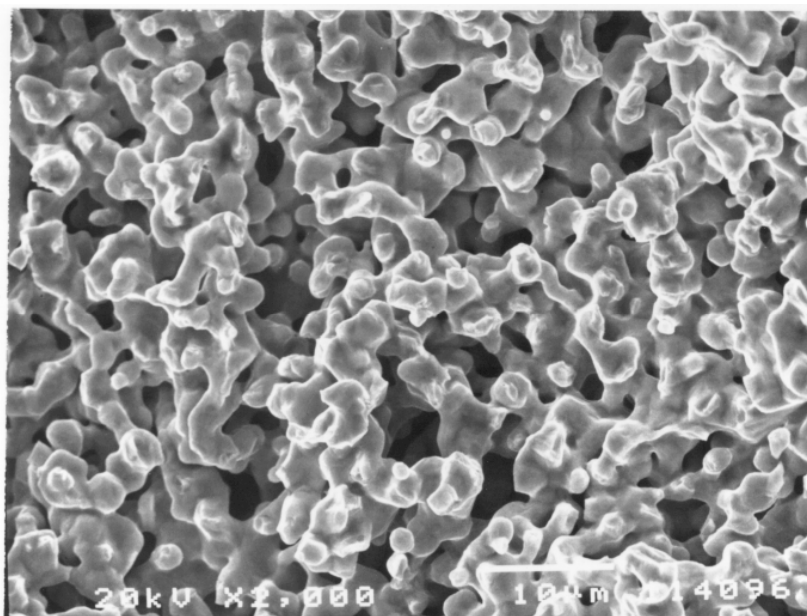
Strength and Microstructure

A novel manufacturing process, proprietary to Mott Corporation, is applied to fabricate Hiflow™ Nickel media that has high pore volume fraction and fine porosity for 0.1 μm particle filtration (Jha and Rubow, 1999). The pore volume fraction in Hiflow Nickel media ranges from 40 to 65%. This level has not been obtained previously in sintered porous metal media for industrial liquid filtration. Figure 1 shows a scanning electron micrograph of the Hiflow Nickel media. The flow pores are tortuous in nature and flow path lengths are long. Therefore, even though the pore dimensions on the porous surface appear to be micrometer size due to high surface roughness, the finer subsurface pores, high tortuosity and long flow paths provide effective obstacle to the passage of sub-micron particles through the media. The micrograph in Figure 1 shows that the metal powder particles used to fabricate the media are well sintered providing intrinsically high mechanical strength. Table 1 shows the tensile strength of the media, typical dimensions and porosity fraction. The strong media can be back-pulsed to dislodge the particle cake formed during fluid filtration and increase the permeate flow. The high media strength provides a high collapse strength (> 1000 psi for 0.5" outer diameter, 24" long tube) and burst pressure. The high strength aids in the construction of robust filter modules,

which can be operated at high pressures and temperatures approaching 300°C, depending on the application.

Table 1: Physical Properties of Sintered Metal Microfiltration Media					
Mott Grade	Material	Tube Inner Diameter (Inches)	Wall Thickness (Inches)	Porosity %	Tensile Strength (psi)
0.1 μm	Hiflow™ Ni	0.35	0.090	40	12,000
0.1 μm	316L SS	0.35	0.055	25	27,000
0.1 μm	Hastelloy C-22	0.36	0.085	20	35,000

Figure 1: Scanning Electron Micrograph of 0.1 μm Hiflow™ Nickel Media



Clean Flow Permeability

Figures 2 and 3 show the liquid and gas permeabilities of 0.1 μm grade sintered metal media. The clean liquid permeabilities of Hiflow Nickel and 316L SS media are considerably higher than that of the 0.2 μm grade alumina membrane. The permeability of 0.1 μm grade Hiflow Nickel is considerably higher than that of similarly graded 316L SS or Hastelloy C-22 due to the high porosity in the Hiflow Nickel media.

For consistent manufacturing of the sintered metal media, the bubble point pressure and air permeabilities of the media are controlled within specified ranges.

Corrosion Resistance

Figure 4 shows the galvanic series of various common metals and alloys depicting their relative corrosion resistance. The corrosion guidelines for Hiflow Nickel media are based upon

Figure 2: Isopropanol Permeability of Sintered Metal Microfiltration Media

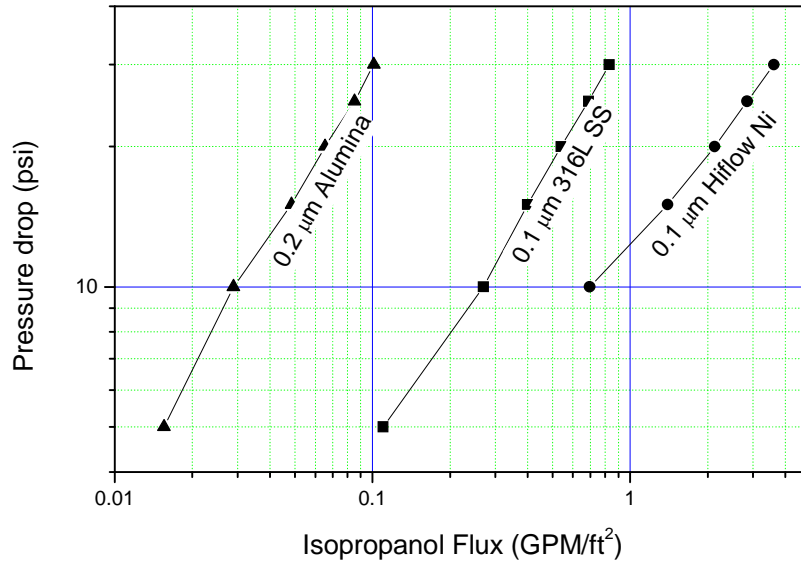
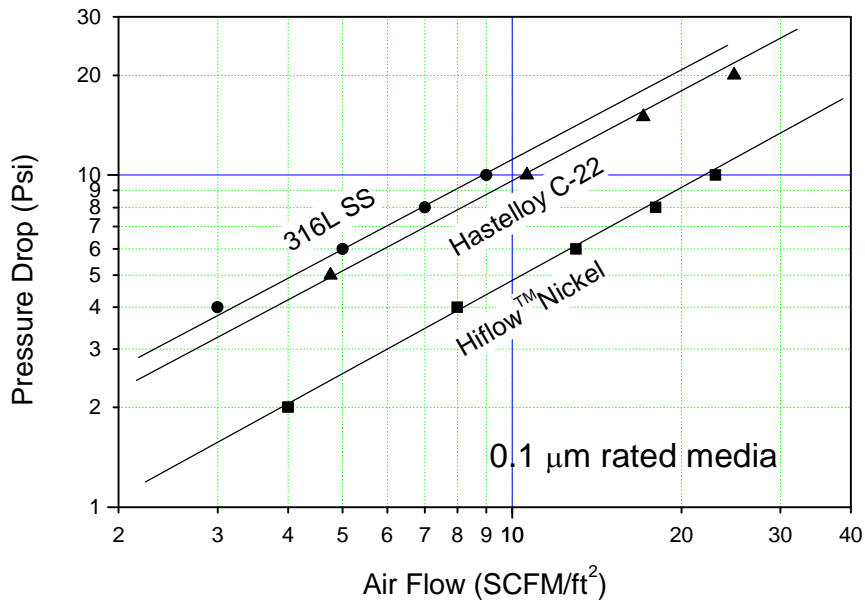


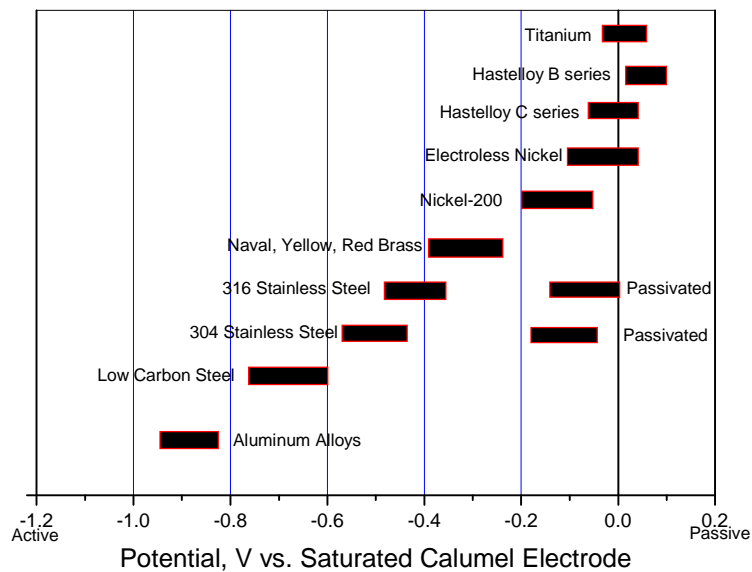
Figure 3: Nitrogen Gas Permeability of Sintered Metal Microfiltration Media



the exposure of wrought Nickel 200 metal (UNS N02200). Figure 4 shows the galvanic series for various common metals and alloys, and it shows that the corrosion potential of Nickel in 5% NaCl solution is comparable to that of passivated stainless steels (Fontana, M.G., 1986). A

porous material will have a higher corrosion rate than a corresponding solid material of the same chemistry due to its high exposed surface area. In the case of Nickel, in general, reducing conditions retard corrosion attack, whereas oxidizing conditions promote corrosion. Nickel porous materials can be used in handling bromine, halogen gases, and chlorinated solvents. Nickel is used widely in the food processing industry. Nickel is resistant to neutral and mild acidic solutions, but is readily attacked by oxidizing acids, such as nitric acid. Alkaline solutions and mild atmospheric conditions do not affect Nickel. Nickel has limited corrosion resistance in seawater and brackish water.

Figure 4: Galvanic Series of Metals and Alloys in 5% NaCl Solution



APPLICATIONS OF SINTERED METAL MICROFILTRATION MEDIA

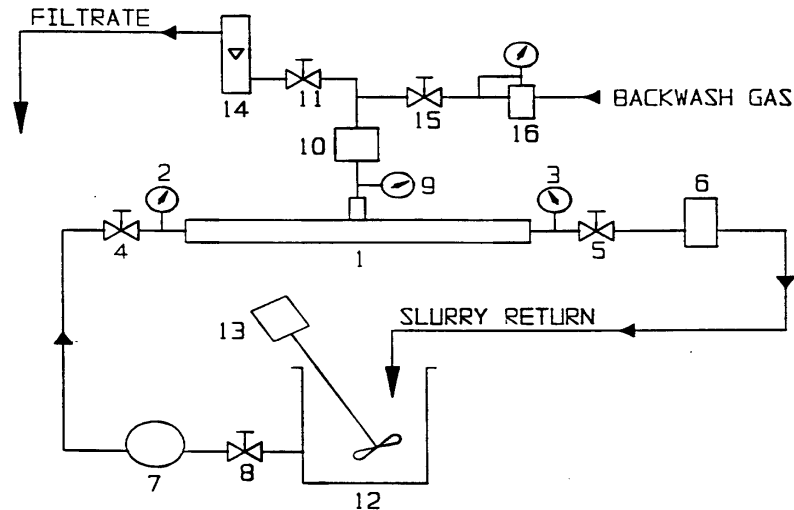
Filtration of Titanium Dioxide slurry:

Titanium di-oxide particles are produced in very high volumes, and have a myriad of applications in industrial and consumer products. They are widely used in paints, and often sold as concentrated slurry. Cross flow filtration of titanium dioxide slurry has been used to concentrate up to 50 to 80% (Trendell et al., 1997). Wastewater recovery of titanium oxide particle containing waste is also of potential interest. Previous attempts for barrier filtration of TiO₂ particles at Mott's R&D laboratory were unsuccessful and the particles were found to readily coat and plug the filter media. Successful cross flow filtration of TiO₂ slurry has now been demonstrated with 0.1 μm Hiflow Nickel and 316L SS media. A surfactant was used to keep the slurry deflocculated. The pH of the slurry was 6.8, and at this pH, the particles have been found to remain well-dispersed (Marchant and Wakeman, 1997).

Figure 5 is a schematic diagram of the cross flow test stand used for filtering TiO₂ slurry. 0.1 μm grade Hiflow Nickel and 316L SS media were used to perform the filtration tests. The pore volume fraction of the two media is considerably different (40% for Hiflow Nickel and 25%

for 316L Stainless Steel). The purpose of the test was to evaluate filtration characteristics, backwash efficiency and the steady state flow characteristics of the media.

Figure 5: Schematic Diagram of Cross Flow Filter



- | | |
|--------------------------|----------------------------|
| 1. MOTT CAT No. 7000 LSX | 9. FILTER PRESSURE GAUGE |
| 2. INLET PRESSURE GAUGE | 10. FILTRATE RESERVOIR |
| 3. OUTLET PRESSURE GAUGE | 11. FILTRATE CONTROL VALVE |
| 4. INLET CONTROL VALVE | 12. FEED TANK |
| 5. OUTLET CONTROL VALVE | 13. AGITATOR |
| 6. FLOWMETER | 14. FILTRATE FLOWMETER |
| 7. PUMP | 15. BACKWASH VALVE |
| 8. PUMP SHUT-OFF VALVE | 16. BACKWASH REGULATOR |

The mean particle size of the 1% TiO₂ slurry in tap water was determined by a Horiba Laser Scattering Particle Size Analyzer and was found to be 2.6 μm with a standard deviation of 1.0 μm. Figure 6 shows a scanning electron micrograph of the TiO₂ particles. These particles tend to settle in the slurry, if left standing for a period of time. Figure 7 shows the TiO₂ cake that formed on the Hiflow Nickel media. It is clearly seen that the particle penetration in the media is no more than 20 μm from the media surface. The cake itself is approximately 50 μm

Figure 6: SEM Micrograph of TiO₂ particles

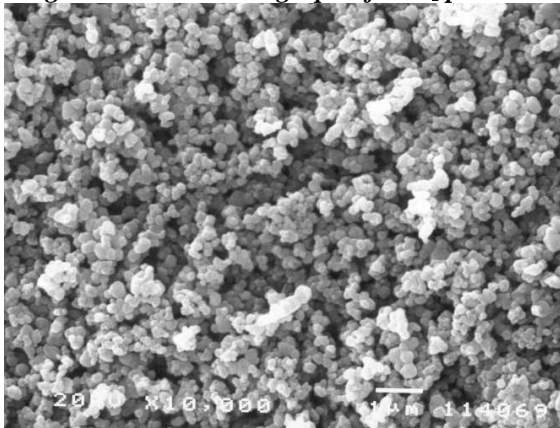
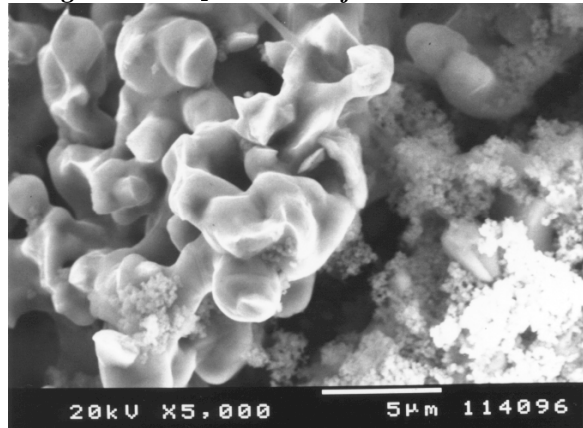


Figure 7: TiO₂ cake on Hiflow Nickel media



thick, and the cake thickness is a strong function of the mainstream flow rate during filtration. Figures 8 and 9 show the permeate flux and associated trans-membrane pressure (TMP) drop observed during the cross flow filtration tests. The cross flow filtration was started at low TMP, and then TMP was increased to 20 – 25 psi. Every peak in the filtrate flux curve represents a blowback. The data show that permeate flow is recovered effectively after blowback. Permeate turbidity with both media was less than 1 NTU. The results on Hiflow Nickel media show that there is a slight dependence of the steady state permeate flux on the TMP. The steady state permeate flux through 316L SS media was somewhat independent of the TMP. At a TMP of 22 psi, the steady state permeate flux through the 0.1 μm Hiflow Nickel media was twice that through the 0.1 μm 316L SS media. This is primarily due to the difference in the porosity of the two media. A more porous media, in general is expected to show higher flux and better response to blowback.

Figure 8: Cross Flow Filtration of TiO_2 slurry

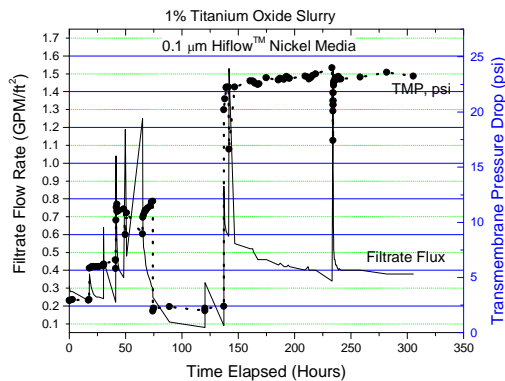
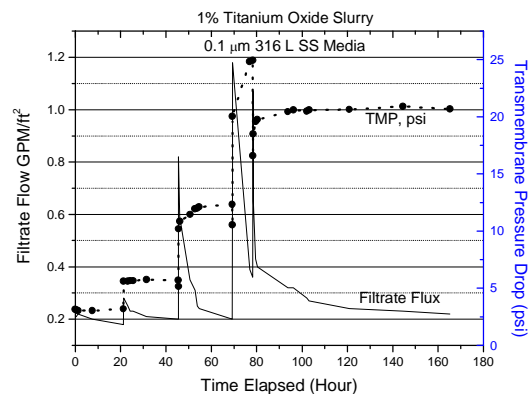


Figure 9: Cross Flow Filtration of TiO_2 slurry



Other Commercial Applications:

In addition to 0.1 μm grade sintered metal media, 316L Stainless Steel media in filtration grades ranging from 0.2 to 2 μm are also available commercially. The applications where these cross flow filtration media have been successfully applied are as follows:

- a. Separate Iron Oxide (Fe_2O_3), 95% less than 1 μm size, from water and concentrate. This application permits recycling of water resulting in saving several million gallons of water per year in a critical drought area.
- b. Separate and concentrate photo color pigment from wastewater. The sintered metal elements in this instance lasted more than ten years before replacement.
- c. Separate carbon catalyst fines, 70% less than 1 μm size, in a solid/liquid reactor. The liquid phase is recycled.
- d. As a sampling filter, operated continuously in cross flow mode, for withdrawing clean particle free liquid sample from reactors for analysis.
- e. Removal of fine particles from wine before packaging.
- f. Removal of sludge and filter aid from wastewater in a nuclear facility.
- g. Pt on Carbon catalyst particle recovery, 2.5 μm size, from a wastewater stream with trace amounts of organic solvent.

SUMMARY

Sintered metal microfiltration media have been used for wastewater recovery and other industrial liquid/solid separation processes for a long time. The benefits of using sintered metal media are their long life, low maintenance, and predictable behavior. Advances in manufacturing processes have enabled the development of 0.1 μm grade media with high porosity fraction, enabling high throughput and reduced media fouling. Sintered metal microfiltration media have been developed in various alloys, namely Nickel, Stainless Steel and Hastelloy C for application in increasingly corrosive environments. The sintered metal media are completely weldable; therefore, the temperature and corrosion resistance of the seals does not limit the applications.

Many inorganic membranes have been reported to show erosion damage by hard particles present in the slurry. Sintered metal microfiltration media have less propensity of erosion damage due to the high toughness of the media. The thin ceramic membranes deposited on coarser ceramic substrates may erode due to the friable nature of the ceramic. In similar circumstances, sintered metal membranes may not show damage due to the galling nature of metal surfaces. One of the major problems with ceramic and other inorganic membranes that are coated on ceramic or carbon substrates is their brittle behavior and inability to withstand sustained mechanical shock or thermal shock. The sintered metal media, with sound mechanical design for the filtration system, can withstand mechanical and thermal shock due to its high toughness, and can be frequently back pulsed to recover filtrate flux. The application is then limited by the strength and fatigue limits of weld joints and the porous metal media.

Sintered metal microfiltration media would be an excellent material for pre-filtering the wastewater streams, before the water is purified using ultra and nanofiltration grade polymeric membranes. The sintered metal media can remove the hard particles that would damage the membranes. Membrane modules located downstream of the sintered metal cross flow filters would remove solutes or higher molecular weight materials. This application would be particularly attractive for clarifying wastewater generated from high volume machining operations.

REFERENCES

- Cheryan, M., 1998, "Ultrafiltration And Microfiltration Handbook", Technomic Publishing Co.
- Jha, Sunil, and Rubow, Kenneth, "Advances in Filtration and Separation Technology", Vol. 13a, 1999, Wallace Leung and Thad Ptak, editors, American Filtration and Separations Society, pp. 512 to 520.
- Fontana, M.G., 1987, "Corrosion Engineering", Third Edition, McGraw-Hill.
- Trendell, Michael J., Drury, Kevin, Spruce, Stephen R., Davis, Martin R.B., Robson, Keith, United States Patent No. 5,622,628, Issued Apr. 22, 1997.
- Marchant, J.Q., Wakeman, R.J., "Crossflow Microfiltration of Concentrated Titania Suspension", IchemE, The 1997 Jubilee Research Event, Apr. 1997, Vol. 2, pp. 1061-1064.