A LOW PRESSURE DROP SINTERED METAL FILTER FOR ULTRA-HIGH PURITY GAS SYSTEMS

Dr. Kenneth L. Rubow
Donna S. Prause,
Mark Eisenmann
Mott Corporation

A Low Pressure Drop Sintered Metal Filter for Ultra-High Purity Gas Systems

AUTHORS BIOGRAPHY

Dr. Kenneth L. Rubow is Vice President - Engineering, Research and Development at Mott Corporation. Prior to joining Mott in 1995, he was Manager of the Particle Technology Laboratory and Associate Director of the Center for Filtration Research at the University of Minnesota. He has conducted extensive research for more than 20 years in the areas of gas and liquid filtration, particle technology, aerosol science and air quality. He has more than 110 publications and edited two books, all dealing with particle technology and filtration. He is a member of 8 professional societies. He is currently on the Board of Directors of the Am. Assoc. for Aerosol Research and the Am. Filtration and Separations Society where he is second Vice Chair. He received his B.S., M.S. and Ph.D. in Mechanical Engineering from the University of Minnesota.

Donna S. Prause joined the Research and Development Department of Mott in 1994 and has been responsible for the development of porous metal media for Ultra High Purity Gaseous Filtration. She is a Research and Development Project Engineer with over 15 years of product design and development experience. She has a B.S.M.E. from the University of Hartford.

Mr. Mark R. Eisenmann joined Mott in 1990 and currently works as a Metallurgist and Senior Research Engineer to develop porous metal filter media for High Purity Filter Applications. He has over 14 years of engineering experience in the Powder Metal Industry and has authored several technical papers. He received a B.S. and M.S. in Material Science from Rensselaer Polytechnic Institute.

ABSTRACT

A low pressure drop, ultra pure, high efficiency all-metal filter, PENTA™[A], has been developed with improved performance over other point-of-use gas filters currently used in semiconductor fabrication, especially Teflon®[B] filters. The filter media is a sintered nickel powder, welded into an electropolished 316L stainless steel housing, and is designed to retain greater than 99.9999999% (log reduction value of >9) of all particles at the most penetrating particle size and at the rated flow.   The new nickel media was developed to have high mechanical strength, high flow / low pressure drop characteristics and to have minimal outgassing of volatile organics and oxygen.

Performance characteristics are presented for five commercially available electronics grade filters designed for ultra-high purity specialty gas distribution systems. Characteristics evaluated include flow performance versus pressure drop across the filter, particle retention, total hydrocarbon contribution and oxygen contribution.

KEY WORDS

High purity gas, electronic grade gas filters, particle filtration, outgassing, contamination, point-of-use.

INTRODUCTION

[A] PENTA™ is a trademark of Mott Corporation.
[B] Teflon® is a registered trademark of DuPont Nemours, E.I. & Co., Inc.
[C] Hastelloy® is a registered trademark of Haynes International, Inc.

The stringent demands of the semiconductor industry require the need for ultra-high purity process gases. These gases, i.e., bulk and electronic specialty gases (ESG), are widely utilized in critical manufacturing processes where contamination can lead to corruption of semiconductor devices.

Contamination can be either in the form of gaseous or particulate matter. Point-of-use filters are extensively used to capture particulate matter, while ideally not contributing particulate or gaseous contamination to the process gas stream. Gaseous contamination typically include moisture, hydrocarbons, and oxygen. Some semiconductor manufacturers are setting specifications for ultra-high purity gases at less than 1 ppb for each critical impurity such as oxygen and hydrocarbons.[1] Furthermore, the filter must be chemically compatible with the electronic specialty gases while minimally affecting the gas pressure.

Filters, with lowest possible pressure drop, are essential for certain electronic specialty gases. This type of filter allows low pressure gas systems adequate pressure to reliably produce the appropriate amount of gas required at the point of reaction. Low pressure drop also means better gas flow through the filtration element, yielding better purging characteristics. Also metal filters lend themselves well to gases such as WF_6 and TEOS when heated lines are used because they are capable of operating at high maximum temperatures and offer material compatibility. Another major benefit of a low pressure drop filter is that it minimizes the required filter volume, thereby limiting potential contamination problems.

Initialization of the gas distribution system requires a prepurge of the system to minimize or eliminate moisture. Moisture adheres to surfaces forming a monolayer which diffuses into the gas stream slowly over time. This moisture problem is compounded by polymeric materials which tend to shed and retain moisture. All-metal filters allow the system to be quickly purged by exposing components to intense heat which minimizes potential outgassing and residual moisture levels.[2]

A low pressure drop, ultra pure, high efficiency all-metal filter, PENTA, has been developed for improved performance over the Teflon filters currently used in semiconductor fabrication. The demand for operating at elevated temperatures has compounded the need for development of an all-metal filter with superior flow performance characteristics, which are typical of existing Teflon media. The filter was developed not only for characteristic low pressure drop at high flow rates, but also for minimizing any outgassing characteristics resulting in increased hydrocarbon or oxygen contamination. Development efforts also involved providing a media with extremely reliable structural integrity.

This new gas filter has been developed with sintered nickel media welded into an electropolished 316L stainless steel housing. The filter was developed to encompass all of the above discussed characteristics of an ideal point-of-use filter. The media is designed to retain greater than 99.9999999% (log reduction value >9) of all particles at the most penetrating particle size and at the rated flow. The new nickel media was developed to have high flow / low pressure drop characteristics, high mechanical strength and to have minimal outgassing of volatile organics and oxygen. The resulting filter has lower pressure drop characteristics than filters commercially available currently on the market. These include filters with media made of Teflon, ceramic, 316L stainless steel, nickel or Hastelloy®[C] C-22 materials. A family of filters has been developed with this new nickel media with maximum rated flow rates of 2, 10, 30 and 75 SLPM.

Results from an evaluation of five commercially available point-of-use filters are presented in this paper. The objectives of the evaluation were to determine performance characteristics of each major type of media commercially available in electronics grade filters for ultra-high purity specialty gases. These media were sintered nickel, sintered stainless steel fiber/powder, sintered ceramic and Teflon.

Four types of tests were performed on the filters. These tests were flow performance versus pressure drop across the filter, particle retention, total hydrocarbon contribution and oxygen contribution. Furthermore, the structural integrity of three filters was analyzed by comparing the maximum differential pressure and temperature characteristics.

FILTRATION FUNDAMENTALS

Penetration and collection efficiency are terminology used to describe the degree of particle capture from a gas stream passing through filter media. The mechanisms for particle capture, as illustrated in Figure 1, are diffusion, interception, inertial impaction, and electrostatic deposition.

Particles can be removed from the gas stream, as the gas flows around the filter structural elements, via the particle collection mechanisms of diffusion, interception, inertial impaction, and electrostatic deposition. Particle deposition via diffusion results when particles collide with the filter structure due to their random Brownian motion. This motion and hence the degree of particle capture becomes more pronounced as the particle diameter becomes smaller. A particle is deposited via the interception mechanism if a particle of finite size is brought within one particle radius of the filter structure. Collection via this mechanism increases with increasing particle size.   Particles are deposited on the filter structure
by the mechanism of inertial impaction due to the finite mass and momentum of the particles as the gas changes direction as it flows around the filter structure. Collection via this mechanism increases with increasing particle size. Particles deposit via electrostatic deposition if electrical charges on either the particle or the filter, or both, create attractive electrostatic forces.

The concept of maximum penetration and the corresponding most penetrating particle size is illustrated in Figure 2. The most prominent mechanisms of capture, in the vicinity of the most penetrating particle size, are diffusion and interception. The combination of these two capturing mechanisms leads to the "parabolic" shaped particle penetration curve in the vicinity of the most penetrating particle size.

The effect of gas flow rate through a filter on particle penetration is also illustrated in Figure 2. The maximum penetration decreases as the gas flow rate is decreased with a slight increase in the most penetrating particle size.

The most stringent particle retention test is performed at the most penetrating particle size.[3] At the most penetrating particle size, the collection efficiency is the poorest.

TEST FILTERS

Evaluations were conducted on five different types of point-of-use filters. These filters contained five distinct types of filter media. The designation and the materials of construction for each filter are listed in Table 1. Filter A, PENTA, contains a newly developed media which is a much finer matrix of sintered nickel than filter C and is, therefore, distinctively different. Filters B - E are commercially available filters from three other manufacturers. All filter housings are fabricated from electropolished 316L stainless steel.

Table 1 lists the material classification and type of media, the media configuration, the type of sealing of the media to the housing, and the rated flow for each of the five filters. The media consist of sintered nickel powder (filters A and C), supported Teflon membrane (filter B), sintered 316L stainless steel fiber/powder (filter D), and sintered ceramic powder (filter E). All five filters were chosen for this study based on their maximum rated flow of 30 SLPM, with the exclusion of filter E (ceramic filter), which has a 50 SLPM rating.
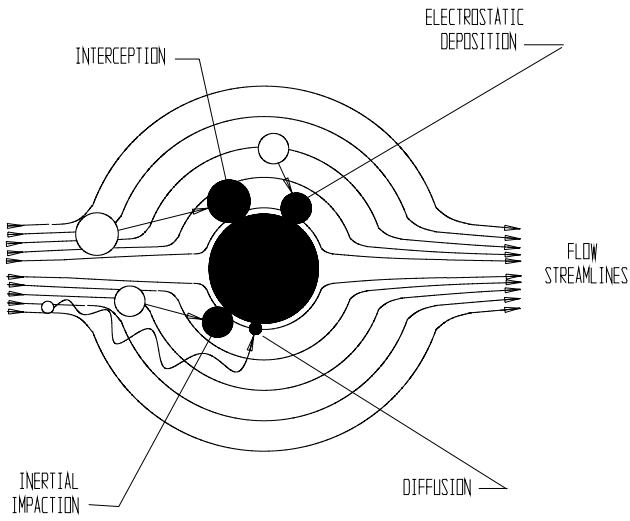
Figure 1. Basic Mechanisms of Particle Capture as Gas Flows Around Filter Elements.
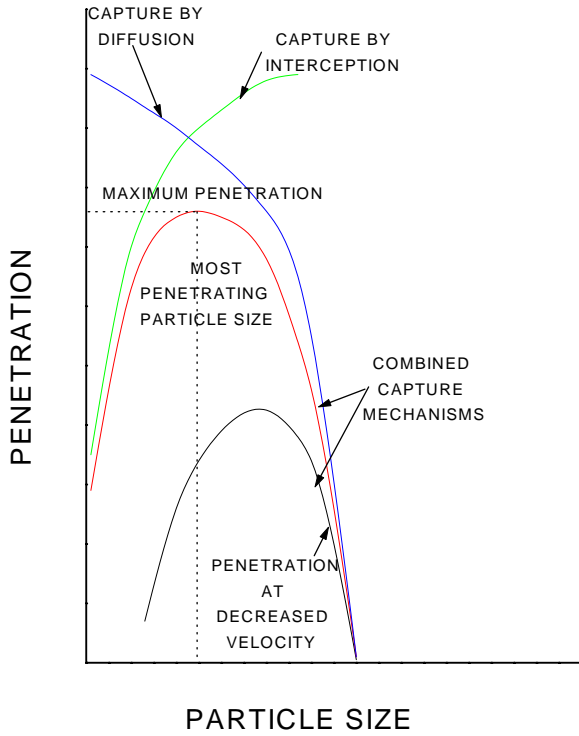

Figure 2. Effect of particle capture mechanisms and flow rate (face velocity) on penetration.

Table 1
Filter Specifications

Designation	Filter Media Classification	Filter Media Material	Media Configuration	Method of Const.	Max. Rated Flow (SLPM)
A	Metal	Nickel	Cup	Welded	30
B	PTFE Membrane	Teflon	PTFE Membrane Supported by a PFA Structure	Welded/ Potted	30
C	Metal	Nickel	Tube	Welded	30
D	Metal	316LSS	Disk	Welded	30
E	Ceramic	Alumina	Tube	Welded/ Teflon Seals	50

EXPERIMENTAL PROCEDURES

Four distinct types of tests were performed on the filters. These tests were flow performance versus pressure drop across the filter, particle retention, total hydrocarbon contribution and oxygen contribution.

Nitrogen flow performance curves were developed for each of the tested filters. The filters were tested for flow performance in order to determine the differential pressure drop across the filter at a given flow rate and system pressure. The tests were performed at inlet pressures of 30, 60 and 90 psig. The filter inlet pressure was regulated by a valve directly downstream of the filter. The nitrogen flow was controlled by a valve upstream of the test filter and monitored by a flowmeter.

A schematic diagram of the particle retention test system is presented in Figure 3. Tests with each filter consisted of flowing clean particle-free nitrogen gas through the filter at the filter's rated flow for at least a period of 5 minutes after zero particles were detected downstream from the test filter. Polydispersed NaCl with a mean particle size of 0.08 µm was then delivered to the test filter at the rated flow rate. Particle concentration downstream of the filter was measured with a TSI Model 3762 condensation particle counter (CPC) (TSI, Inc., St. Paul, MN). The upstream particle concentration was monitored by a TSI Model 3022A CPC. Both CPCs detect particles larger than 0.01 µm in diameter and concentrations of up to 9.99×10^6. The upstream particle concentration ranged from 10^5 - 10^7 particles/cm^3.
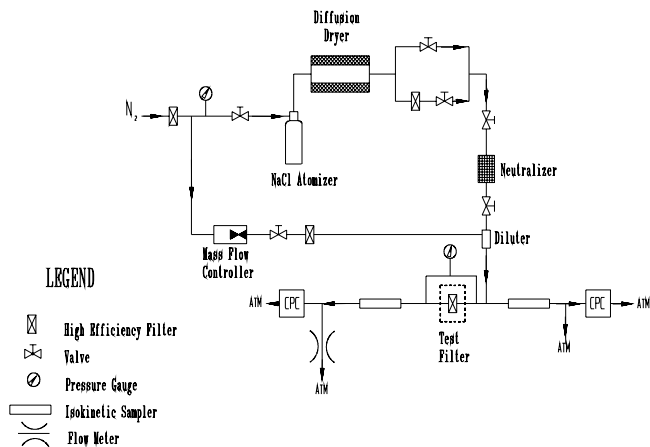

Figure 3. Schematic Diagram of Particle Retention System.

Additional testing was performed on specially prepared samples of the media in filter A to determine the location of the most penetrating particle size. This work was conducted using the system described in the preceding paragraph, but augmented to use monodisperse test particles.[4,5] These particles were obtained with an electrostatic classification method.   The most penetrating particle size for filter A is 0.08 µm.

A concern for semiconductor manufacturing processes is the outgassing of volatile organics and oxygen from filters. This type of microcontamination can lead to the corruption of semiconductor devices and premature failure of process

equipment. Therefore, the filters were tested for total hydrocarbon and oxygen contribution. The total hydrocarbon (THC) and oxygen contribution tests were performed in accordance with the SEMASPEC Standards 90120396B, and 90120398B. The test gas was pure particle-free dry nitrogen. System pressure throughout the test was maintained at 30 psig. The flow rate throughout testing was 1.3 SLPM. At initiation of the test, a spool piece was exchanged with the test filter in a glove box. Prior to the exchange, the glove box was purged with greater than five volumes of pure dry particle-free nitrogen test gas. Ambient temperature nitrogen continued to flow through the system during the exchange and after the filter was installed. After installation was completed, the "out-of-box" THC and oxygen content of the filter were measured. Hydrocarbon concentrations above 100 ppb were recorded. Oxygen concentrations above 20 ppb were recorded. The test was repeated at elevated temperatures of 158° F. Total hydrocarbon and oxygen concentrations were analyzed with a Rosemount Analytical Inc. Model 400A hydrocarbon analyzer and a Teledyne Model 396 ultra trace oxygen analyzer. The minimum limit of detection for each instrument is 10 ppb.

EVALUATION OF RESULTS

The objectives of this study were to determine differential pressure drop, particle penetration, outgassing (hydrocarbons and oxygen) and structural characteristics of high purity filters.

Flow performance was determined for each of the filters at inlet pressures of 30, 60 and 90 psig and nitrogen gas flow rates of 10, 20 and 30 SLPM.   Flow performance information was obtained for filter B, Teflon media, from the manufacturer's published literature. The remaining four filters were tested for flow performance. The differential pressures across the filters are illustrated in Figures 4, 5 and 6. Typical pressure drop results at an inlet pressure of 30 psig and at 30 SLPM for filter A is 3 psid, filter E is 5.5 psid, filter D is 6 psid, filter B is 6.5 psid, and filter C is 15 psid. Filter A (newly developed nickel media) has less than half the pressure drop of filter B (Teflon media). Similar findings exist for inlet pressures of 60 and 90 psig.

Particle retention testing was performed using the following procedure. Each filter was challenged at its maximum rated flow with polydispersed NaCl particles. The mean size of the particles was 0.08 μm which is in the vicinity of the most penetrating particle size. All filters were purged with compressed filtered dry nitrogen gas at ambient temperatures. The particle background counts were maintained at zero prior to initialization of the particle challenge portion of the test. The particle retention results are listed as log reduction value (LRV). LRV is the log of the ratio of particle concentration upstream of the filter to particle concentration downstream of the filter. For example, a filter with a LRV of 9 is equivalent to a removal of 99.9999999% of all particles.

Results of the particle penetration test are listed in Table 2. All filters were tested at a rated flow of 30 SLPM with the exception of filter E, which was tested at the rated flow rate of 50 SLPM. Filter A had a LRV of >9. The LRV for filter B was not available; however, an LRV of >9 was obtained from previous testing of filters with Teflon media. Filters C and D followed with an LRV of 8 while filter E (ceramic) had a LRV of 5.7.   Similar results for ceramic media has been reported.[6]
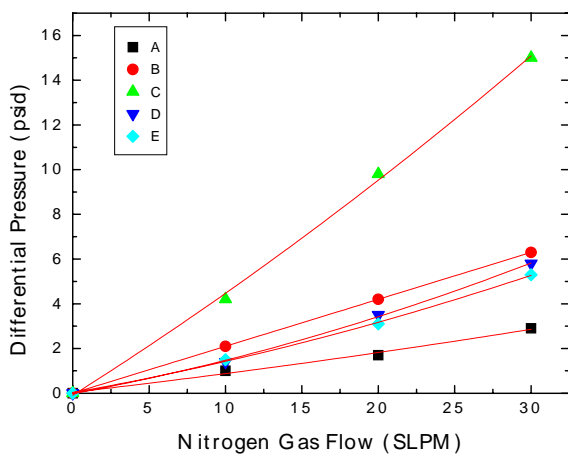

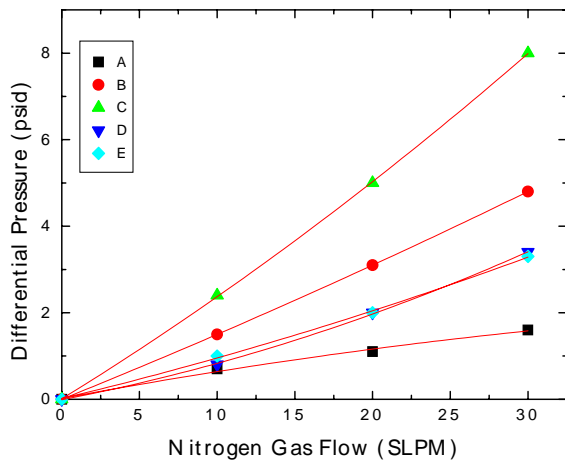Figure 4.   Flow Performance for Inlet Pressure of 30 psig.

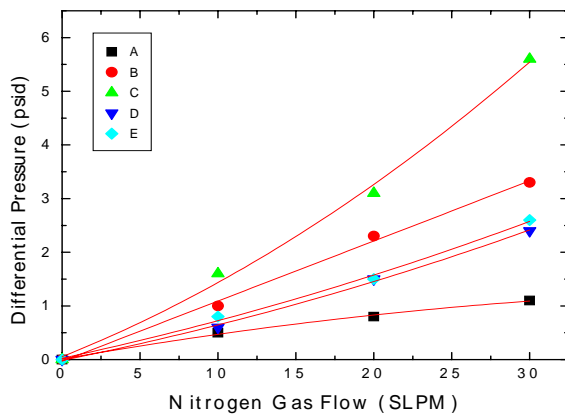Figure 5.   Flow Performance for Inlet Pressure of 60 psig.

Figure 6.   Flow Performance for Inlet Pressure of 90 psig.

Table 2
Particle Retention Results

Designation	Filter Media Material	Particle Retention LRV	Rated Flow (SLPM)
A	Nickel	>9.0	30
B	Teflon	N/A*	30
C	Nickel	8.0	30
D	316LSS	8.0	30
E	Alumina	5.7	50

*LRV >9 obtained in previous studies.

Results of the hydrocarbon and oxygen contribution are listed in Table 3. Each filter was unpackaged in a purged glove box and tested for total hydrocarbon and oxygen contribution. Initial contribution of oxygen and hydrocarbon concentration from each unpackaged filter is listed in the table. The interval of time required, for each filter to obtain 10 ppb oxygen concentration, at a flow rate of 1.3 SLPM, is also listed in Table 3.

All filters tested had an initial detectable level of oxygen and hydrocarbons; however, the hydrocarbon concentration dropped below 10 ppb within 5 minutes of initiation of the testing for all filters. The oxygen content for only two filters, filters A and B, dropped below 10 ppb after initiation of the testing. The other three filters required more than 10x longer to reduce the oxygen concentration to a level of less than 10 ppb.

Maximum differential pressure drop across the filter is a measure of the structural integrity of the media. The published maximum differential pressures across filters A, B and C, at ambient temperature, for the forward and reverse flow direction are listed in Table 4, along with maximum operating temperatures. The nickel media of filter A is more than 8x stronger than the Teflon media,

Table 3
Oxygen and Total Hydrocarbon Contribution

Designation	Media Material	Initial O_2 Reading (ppb)	Time Required To Obtain <10 ppb O_2 Conc. (min)	Initial Hydrocarbon Reading (ppb)
A	Nickel	13,600	<5	200
B	Teflon	40,000	<5	40,000
C	Nickel	33,600	50	910
D	316LSS	22,400	55	780
E	Alumina	26,200	70	12,400

Table 4
Structural Integrity of Media

Designation	Media Material	Maximum Rated Diff. Pressure (psid)		Maximum Operating Temperature (C°)
		Forward	Reverse	
A	Nickel	500	500	400
B	Teflon	60	10	120
C	Nickel	290	290	400

filter B, in the forward flow direction and more than 50x stronger in the reverse flow direction. Furthermore, the maximum operating temperature of filter A is more than 3x higher than for filter B. The integrity issue is important from a service life and system upset flow condition. These could lead to rupture of the filter media and/or particle shedding caused by media flexing over time.

SUMMARY

Performance characteristics have been obtained for five commercially available point-of-use filters. The media used in these filters were sintered nickel, sintered stainless steel fiber/powder, sintered ceramic and Teflon. Characteristics evaluated include flow performance versus pressure drop across the filter, particle retention, total hydrocarbon contribution and oxygen contribution. Lastly the structural integrity was analyzed by comparing the maximum differential pressure and temperature characteristics of the filters.

Overall filter A, PENTA, with nickel media has clear advantages in terms of flow/pressure drop, overall size, particle retention, oxygen content, and total hydrocarbon content. At rated flow of 30 SLPM, the pressure drop across the filter at atmospheric pressure is 6 psid. The pressure drops across filter A, for rated flow of 30 SLPM, at 30, 60 and 90 psig system pressures are 3.0, 1.4, and 1.0 psid, respectively. Other filters tested have pressure drops that are 1.8 to 5 times higher. Filter A was shown to retain greater than 99.9999999% (LRV of >9) of all particles at the most penetrating particle size and at the rated flow. The strength of the filter A media exceeds that of filter B (Teflon) by more than 8 times. Lastly, the media structure of metal filters are advantageous over that of polymer (Teflon) filters because they do not tend to shed and retain moisture.

REFERENCES

1. A. R. Homyak, S. N. Ketkar, and B. J. Shemanski, "Delivering Hydrogen to Meet < 1 ppb Impurity Levels without the Use of Purifiers", Solid State Technology Supplement, pp. S5-S9, Oct 1995.

2. S. D. Cheung, G. L. Mooney, and D. L. Jensen, "Designing, Installing a Gas Distribution System in a Sub-0.5-μm Facility", Micro, Vol. 13, No. 9, pp. 59-67, Oct 1995.

3. D. C. Grant, and K. L. Rubow, "Particle Removal Rating of Process Gas Filtration Media", Solid State Tech. 31 pp. 53-55, May 1988.

4. K. L. Rubow and C. B. Davis, "Particle Penetration Characteristics of Porous Metal Filter Media for High Purity Gas Filtration", Proc. of the 37th Annual Technical Meeting of the Institute of Environmental Sciences, pp. 834-840, 1991.

5. K. L. Rubow, R. A. Hogle, and C. B. Davis, "Characteristics of Metal Nickel Filter Media for High Purity Corrosive Gas Filtration", Proc. of Microcontamination 91 Conference, pp. 373-386, 1991.

6. W. Plante and J. B. Jailet, " Downstream Cleanliness of Inorganic Filters for Ultra High Purity Gases", Proc. of the 37th Annual Technical Meeting of the Institute of Environmental Sciences, pp. 712-718, 1991.