

High Pressure Point-of-Use Purification of Corrosive Gases. Effect on Gas Distribution Components

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ABSTRACT

Purification of high-pressure corrosive gases has traditionally utilised a pressure regulator prior to introduction into the purifier. However, reported regulator failures have been shown to be a major disadvantage of using this conventional configuration. Such failures are a great concern owing to the damage they may cause to downstream components such as pressure transducers, mass flow controllers, and on-line monitors. A high-pressure source gas purifier installed before the regulator offers a solution to this common problem. Recently, a new purifier for corrosive gases has been developed that can be used up to full cylinder pressure for source gas purification. This new purifier can also be used at lower pressure for point-of-use purification at the wafer tool. The new purifier reduces moisture concentrations in high- or low-pressure corrosive gases to ppb levels. It has remarkably higher moisture capacity than the first-generation products.

The effect of the high-pressure purifier was investigated under severely corrosive gas conditions. HBr was doped with moisture at concentrations from 1 to 5500 and sent through the experimental gas manifold at pressures of 320 psig (HBr cylinder pressure) and 30 psig. Several line valves and gas sticks were placed upstream and downstream of the purification media. After 18 days of exposure to the corrosive environment, the test components were measured for particles, leak rates, and visual corrosion. The results of this investigation are presented to illustrate the effect of the high-pressure purifier on gas distribution components.

INTRODUCTION

Source gas and point-of-use (POU) purification of process gases is necessary to meet the stringent demands of the semiconductor industry. Gas purity levels are tested in the cylinder at the production site and sometimes at the fab prior to connecting the cylinder to the gas distribution system. However, this only ensures that the gas coming out of the cylinder meets the designated purity specifications of the cylinder. Unfortunately, many contaminants can be added to the process gas prior to it reaching the tool or point of use. It has been reported that the gas distribution system is responsible for 68% of contamination in a CMOS process [1]. Impurity molecules can be introduced into the gas distribution system during cylinder changing, inadequate purge procedures, or exposure to contaminated equipment. It should be noted that although the gas from the cylinder is changed and analysed frequently, the gas distribution components are continuously exposed to the process gas and gradually become contamination sources for the wafer tool. This effect is enhanced when using corrosive process gases.

Source gas and POU purifiers will improve and maintain the purity of a gas or gas mixture by removing impurity molecules. Improvement in the gas quality trans-

lates into an economic enhancement in the manufacturing process, more reliable and consistent equipment performance, and a reduction in equipment maintenance or replacement.

The use of corrosive process gases causes an increase in contamination issues associated with gas distribution systems. Furthermore, the effect of moisture, within the corrosive gas or on the surface of the gas distribution system, has a large impact on contamination contribution. Previous research has shown that adsorbed water is extremely difficult to remove from a metallic surface [2]. Matheson researchers have shown that once moisture is adsorbed onto a metallic surface, it is held quite strongly relative to other gas phase molecules owing to hydrogen bonding [3]. In a simple experiment, it was shown that moisture cannot be effectively removed by purging with a dry inert gas over the course of several hours. Mild heating or addition of any hygroscopic material (such as anhydrous HCl) will cause a significant increase in the moisture concentration [3] owing, in part, to desorption from surfaces. In addition to desorbed moisture, it has been shown that HCl will react with iron oxides to produce iron chlorides and water [4]. Although this reaction subsides as the iron oxides are converted to

iron chlorides, it provides an in situ mechanism for producing moisture within a completely dry gas distribution system.

It is well known that corrosion (accelerated by moisture) is a major problem in the semiconductor industry and causes replacement of gas distribution systems on a regular basis. Metal halides and metallic particulate contamination (caused by corrosion) have a direct impact on the wafer yield. Corrosion is due to moisture reacting with the anhydrous acid gas to produce by-products that are much more corrosive than the anhydrous starting material. It has been reported that 0.1 ppm moisture in HCl does not attack 316L stainless steel, while 200 ppm moisture in HCl can significantly corrode the surface of this material [4]. Corrosion data for HBr revealed that at levels of 0.5 ppm moisture, an electropolished 316L stainless steel showed little reaction over the course of 10 days [5]. At 100 ppm moisture in HBr, the results showed that the same material became pitted and covered in a dense metal bromide scale.

Corrosion in regulators has been demonstrated to be the single largest cause of component failure within a gas distribution system [6]. This was attributed to condensation of moisture and HBr during adiabatic expansion when the gas passes through the poppet of the regulator. Such pressure failures are a great concern owing to the damage they may cause to downstream pressure-sensitive equipment.

Matheson's Nanochem® series of purifiers offers an effective solution for microelectronic gas purification because of its ability to deal with various gases and many types of impurities. While organometallic Nanochem® purifiers are very efficient for inert gases, inorganic materials are used for purification of corrosive acid gases. Matheson's DPX purifier is known for its high performance under severely corrosive gas conditions, but is limited to relatively low pressures (<100 psig). The HPX purifier can handle the full cylinder pressures of HBr and HCl, but suffers from a lower moisture capacity than the DPX.

Matheson's second-generation corrosive-gas purifiers are based on activated inorganic materials. These new materials are capable of handling cylinder pressures while offering capacities dramatically higher than those of the HPX purifier and even substantially higher than those of the DPX. They have been shown to have a capacity of 17 litres of moisture per litre of purifier (L/L) in 100% N₂, 20 L/L in >99% HCl and 23 L/L in >99% HBr. Such extended capacities ensure long uninterrupted source gas or POU operation. Furthermore, the new materials operate at efficiencies similar to or better than those of previous purifiers and emit practically no metal impurities; all metal emissions are below ICP-MS detection limits. A source gas purifier for high-pressure corrosive gases has a distinct advantage. It is able to protect all components (MFCs, regulators, on-line monitors, etc.) and piping in the gas distribution system immediately after the cylinder valve.

This article focuses on measuring the effect of purification on typical gas distribution components and piping. A literature review in this area reveals much work accomplished on the effect of moisture on gas distribution piping and components, but the end results are typically photographs and SEM and XPS analyses. Although these techniques are quite useful for obtaining a qualitative sense of the degree of corrosion, they cannot provide quantitative analytical data. By contrast, the research contained herein provides direct quantitative information on the

value of using a source gas or POU purifier. This was accomplished by conducting particle counts and leak rate measurements of components before and after exposure to HBr, upstream and downstream of the purifier.

EXPERIMENTAL

Materials

ULSI 5.0 purity (99.999%) grade hydrogen bromide (HBr) was obtained from Matheson Gas Products in Newark, California. Two different source gases were used during the course of the experiments. The first HBr cylinder was used by mixing the outflow from the cylinder with a moisture-doped nitrogen stream. By using the HBr in this manner, it was possible to vary the moisture level from 1 ppm to >5000 ppm. The second HBr cylinder was intentionally doped with moisture such that the resultant concentration in the cylinder was 100 ppm. Over the course of the experiment, 46 lb (216.2 ft³) of HBr was directed through the test samples and purifier.

Dry nitrogen mixed with moisture was used as the matrix gas for moisture dilutions and was used to dope the HBr gas. Dry nitrogen was also used as the purge gas for the manifold before and after the HBr introduction. Nitrogen was obtained from a cryogenic source and was purified using a Nanochem® OMX Bloc Purifier™. Use of the purifier reduced the moisture level in the nitrogen gas to <1 ppb. Unpurified cryogenic nitrogen was used during particle count measurements. The moisture level was determined to be approximately 1 ppm.

Procedures

Manifold purge and drydown

The manifold was purged with purified nitrogen at a flow rate of 5 L/min overnight. While the nitrogen was still flowing, the manifold was wrapped in heat tape and heated for 24 hours at a temperature of 60-70°C. During this portion of the purge sequence the air-actuated valves were cycled in pairs for 30 minute intervals to purge out each leg of the manifold. Heating of the manifold was then terminated and the manifold was allowed to cool. The purified nitrogen was turned off and all the valves were opened. HBr was then introduced into the manifold for 10 minutes. The manifold was then purged with purified nitrogen at a flow rate of 1 L/min for 90 hours. This method was used to ensure that moisture desorbing from the surface and produced from the reaction of HBr with iron oxide was swept away and was not responsible for corrosion in the test samples.

Handling of test components

The gas piping samples (also referred to as gas sticks) and valves were particle-counted prior to exposure to HBr gas to record a baseline value for each component, and again after the exposure. Since the gas sticks and valves were handled in a non-cleanroom environment, before and after exposure to HBr, there were some concerns about particle contamination that could have occurred during transfer to the particle-counting apparatus. To minimise this type of airborne contamination, each gas stick and each valve was purged for 30 minutes in the particle-counting sample apparatus prior to collection of any data.

Precautions were also taken to ensure that corrosion of lines upstream of the test samples did not deposit corrosion products into the valves or gas sticks. 60 µm VCR®, filter gaskets were installed upstream of each valve and each gas stick prior to HBr

exposure. Figure 1 shows a schematic of the manifold, including the purifier and test components. The gaskets were normal, 1/4 inch, VCR®-style nickel gaskets that had 316L stainless steel filters pressed into the gasket body. The small surface area of the gasket trapped particles larger than 60 µm, while not trapping significant amounts of moisture.

Particle counting of gas sticks

The gas sticks were subjected to three different forms of particle counting: static, dynamic, and impact particle counting. All particle-counting tests were conducted on the basis of ASTM Method F1394-92 [7]. All particle counts were conducted in nitrogen.

Static particle counting was performed on the gas stick by flowing 14 L/min through the test sample and 14 L/min through a bypass leg for a total flow of 28 L/min to the particle counter. Particle counts were recorded once per minute for 20 minutes. The value reported for each individual sample was the average of the particles collected over this 20 minute time period.

Dynamic particle counts were performed similarly with respect to flow rates. However, upstream of the particle filter, on the sample leg, an air-actuated valve was cycled open for ten seconds and closed for two seconds. The valve was cycled five times during a one-minute period for 20 minutes. This action would cause a pressure drop and turbulent airflow within the test sample, causing particles to become dislodged from the test component. The value reported for each individual sample was the average of the particles collected over this 20 minute time period.

Impact particle counting was conducted by flowing 14 L/min through both the sample component and the by-pass leg while striking the test component with a 50 gram steel ball that was attached to a string. The steel ball and string were drawn back to a 45° angle and then released. An apparatus was built to ensure that the impact test could be reproducibly repeated. The steel ball falls in an arc like a pendulum, striking the gas stick and dislodging particles on the internal surface. The impact on the sample was completed once per minute, for ten minutes. The value reported for each individual sample was the average of the particles collected over this 10 minute time period.

Particle counting of line valves

Throughout the duration of the experiment, the valves were cycled nearly 1000 times. This included the cycles necessary to purge the manifold before and after HBr introduction. The test valves were subjected to two different forms of particle counting: static and dynamic particle counting. All particle-counting tests were conducted on the basis of ASTM Method F1394-92 [7]. All particle counts were conducted in nitrogen.

After a 30 minute purge to achieve a baseline value, the test valves were subjected to a static particle count. Static particle counting was performed by flowing 14 L/min through the sample valve and 14 L/min through a bypass leg, for a total flow of 28 L/min to the particle counter. Particle counts were recorded once per minute for 20 minutes.

The test valves were then subjected to dynamic particle counting. During dynamic particle counts, the total flow rate (28 L/min) into the particle counter remained the same throughout the experiment. The test valve was cycled open for ten seconds and closed for two seconds (five times during a one-minute period), for 20 minutes. By actuating the valve this would cause particles to become dislodged from the test component.

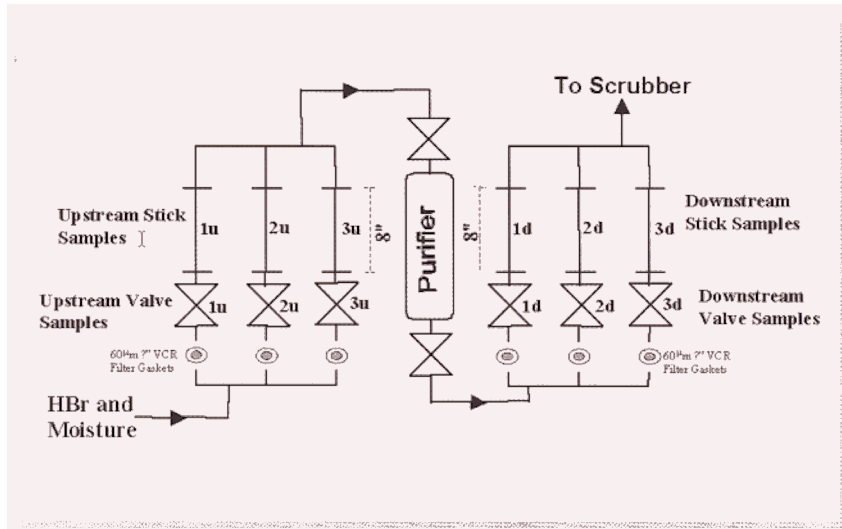


Figure 1 Schematic of the manifold used to conduct experiments

Helium leak rates

The test valves were subjected to two different forms of leak rate testing: “inboard” with the valve open and closed, and “across-the-seat”. For inboard leak testing a cap was used to seal the inlet side of the valve and the leak detector was connected to the downstream side of the valve.

During the inboard leak rate procedure, the valve was placed in an environment composed of helium. While the valve was closed, a vacuum was established inside of the valve and a leak rate recorded. After recording the leak rate with the valve closed, the valve was opened and the leak rate was recorded again with the valve open.

Across-the-seat leak checks were conducted by connecting a 5 psig helium source to the inlet side of the valve. The valve was kept closed, while a vacuum was established on the outlet side of the valve. The valve remained in this state for a period of 15 minutes with the leak rate information being recorded every minute. The data reported herein were collected at the tenth minute.

Moisture doping

Experiments using full cylinder pressures were conducted by using the aforementioned intentionally doped cylinder (100 ppm moisture) and a standard ULSI grade HBr cylinder (≤ 1 ppm moisture). Experiments using low-pressure HBr were conducted by using in-house, custom-built, dynamic gas dilution manifolds and moisture permeation devices. Use of this equipment allowed a variation in moisture level from parts per billion to parts per thousand.

Moisture-in-HBr exposure conditions

The test components (gas sticks and line valves) and purifier were exposed to various experimental conditions. The purpose of the variation was to simulate different conditions that an actual gas distribution system could encounter during normal operations. This included HBr pressure exposures of 30 psig to 320 psig (full cylinder pressure), moisture levels from 1 ppm to 5500 ppm, and flow variations from 160 ml/min to 500 ml/min. In all cases, as illustrated by the data, the components downstream of the purifier were essentially unaffected by the exposure to the HBr. The components upstream of the purifier encountered severe degradation in performance and were visibly observed to exhibit severe corrosion. Table 1 presents the experimental parameters used during this study.

TABLE 1.
EXPERIMENTAL PARAMETERS USED DURING THE STUDY

H ₂ O concentration (ppm)	HBr EXPOSURE time(days)	HBr PRESSURE (psig)	BHBr USED (lb)	HBR USED (ft ³)
1	7	320	18.5	87
100	3	320	14	66
225	7	30	12	57
5500	1	30	1.5	7
Total	18 days		46lb	217 ft³

Figure 2 (below)
Average static particle counts of gas sticks before and after exposure to HBr

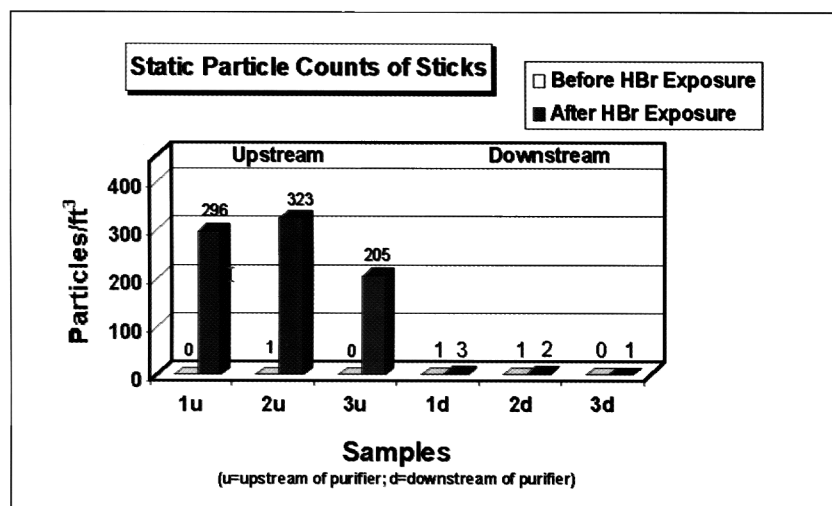
DATA AND RESULTS

Particle concentrations

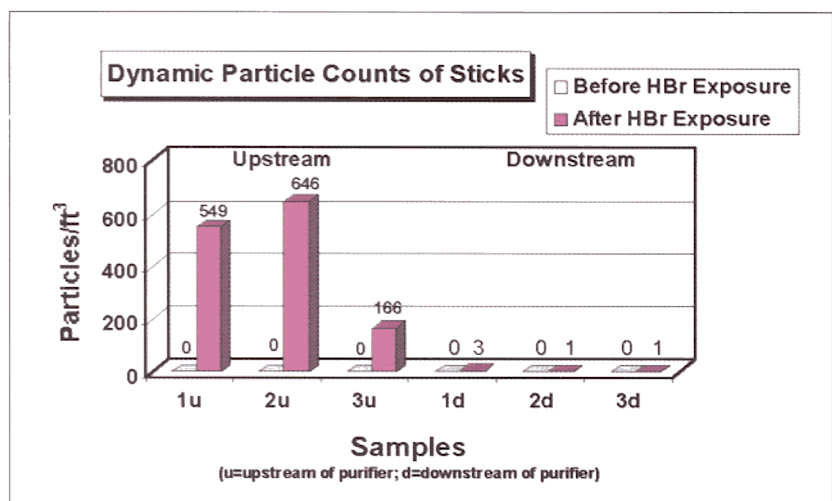
Particle counting of gas piping samples
The gas piping samples (gas sticks) were placed upstream of the purifier and revealed a dramatic difference in static particle measurements from the downstream gas sticks. The average particle count

for the upstream gas sticks after exposure to moisture-laden HBr was 275 particles/ft³. The average particle count for the downstream gas sticks after exposure to moisture-laden HBr was 2 particles/ft³. Figure 2 exhibits individual particle levels for each sample tested. The results represent over two orders of magnitude of difference in the amount of particle contamination between the purified and non-purified gas sticks.

Figure 3 (bottom)
Average dynamic particle counts of gas sticks before and after exposure to HBr



The dynamic particle count results revealed similar results. The average particle count for the upstream gas sticks after exposure to moisture-laden HBr was 453 particles/ft³. The average particle count for the downstream gas sticks after exposure to moisture-laden HBr was 2 particles/ft³. Figure 3 depicts individual particle levels for each sample tested.



The impact particle count results were the most dramatic. The average particle count for the upstream gas sticks after exposure to moisture-laden HBr was 3493 particles/ft³. The average particle count for the downstream gas sticks after exposure to moisture-laden HBr was 6 particles/ft³. Figure 4 represents individual particle levels for each sample tested. The trend of increasing particle levels when comparing static, dynamic, and impact tests was an expected result. This result was due to the fact that each test was more aggressive in dislodging particles than the previous test. The results from the impact particle count tests represent a difference of over 500 times in the amount of particle contamination between the purified and non-purified gas sticks. What is even more impressive is the fact that the particle levels for the downstream samples are near zero. This indicates that the purifier has a large impact on control of particulate contamination (owing to its effect on corrosion). Furthermore, use of the source gas purifier allows the gas sticks to operate in a nearly particle-free environment.

The gas sticks were cross-sectioned and examined visually. The upstream gas sticks exhibited much more discoloration, and appeared to have a thin scale or film present on the surface. The reddish-brown discoloration was thought to be due to iron bromide formation on the surface. High-magnification photographs showed the surface morphology to be rough with deposits and debris present. The downstream samples appeared to be slightly dulled or turbid compared with new electropolished tubing. There was no visible evidence of corrosion, discoloration, scale, or debris for the downstream samples.

Although the upstream and downstream samples exhibited significant differences in appearance, the degree of corrosion of each sample remained a subjective evaluation. Particle counting and leak rate measurements offer objective quantitative data on the degree of corrosion. The visual results obtained demonstrate the need for quantitative methods of analysis, such as particle counting. As pointed out earlier, SEM and XPS analysis are useful techniques, but they are not able to ascertain quantitative information on the degree of corrosion. The fact that a gas line can undergo an impact test with a 50 gram steel ball, while not emitting particles, is irrefutable evidence that minimal corrosion has occurred.

Particle counting of line valves

The diaphragm line valves that were placed upstream of the purifier revealed a dramatic difference in static particle measurements compared with the downstream valves. The average particle count for the upstream valves after exposure to moisture-laden HBr was 2114 particles/ft³. The average particle count for the downstream valves after exposure to moisture-laden HBr was <1 particle/ft³. Figure 5 shows the individual particle levels for each sample tested. The results represent over three orders of magnitude of difference in the amount of particle contamination between the purified and non-purified valves.

The dynamic particle count results revealed a similar trend. The average particle count for the upstream valves after exposure to moisture-laden HBr was 4790 particles/ft³. The average particle count for the downstream valves after exposure to moisture-laden HBr was 5 particles/ft³. Figure 6 depicts the individual particle levels for each sample tested.

The trends observed for the static and dynamic particle levels show the expected result of higher particle levels for the dynamic tests. The rationale for this behaviour was mentioned previously. The results from the static and dynamic particle count tests represent differences in the amounts of particle contamination between the purified and non-purified valves of >2000 and ≈1000 times, respectively. This result is even more surprising than the results from the gas sticks. The greater total number of particles for the valves than for the gas sticks was an expected result. However, the near-zero particle levels in the downstream valves were quite unexpected. These data indicate, again, that the purifier has a large impact on control of particulate contamination (owing to its effect on corrosion), and clearly demonstrate the value of using POU and source gas purifiers within gas distribution systems.

The valves were disassembled and examined visually. The results of this examination revealed that the upstream samples suffered severe corrosion. The upstream valves appeared highly discoloured on the seat and diaphragm, and exhibited significant amounts of corrosion products within the valve cavity. The downstream samples appeared slightly dulled, but showed no signs of corrosion products in the valve cavity.

Helium leak rate data

Inboard and across-the-seat helium leak rates were measured for each of the valves as previously described. The average inboard helium leak rate for the upstream valves after exposure to HBr (in the closed position) was found to be 1.8×10^{-10} cc/sec. The average inboard helium leak rate for the downstream valves after HBr exposure (in the closed position) was found to be 1.9×10^{-10} cc/sec. The inboard leak rates for the sample valves did not

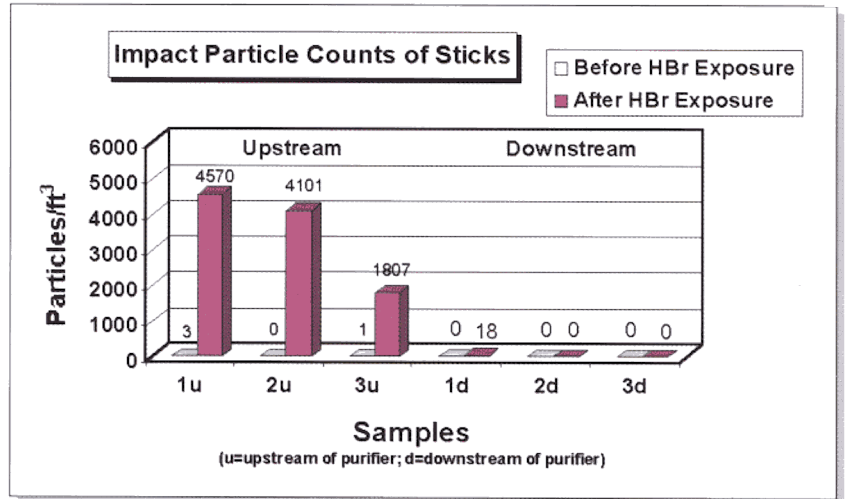


Figure 4 Average impact particle counts of gas sticks before and after exposure to HBr

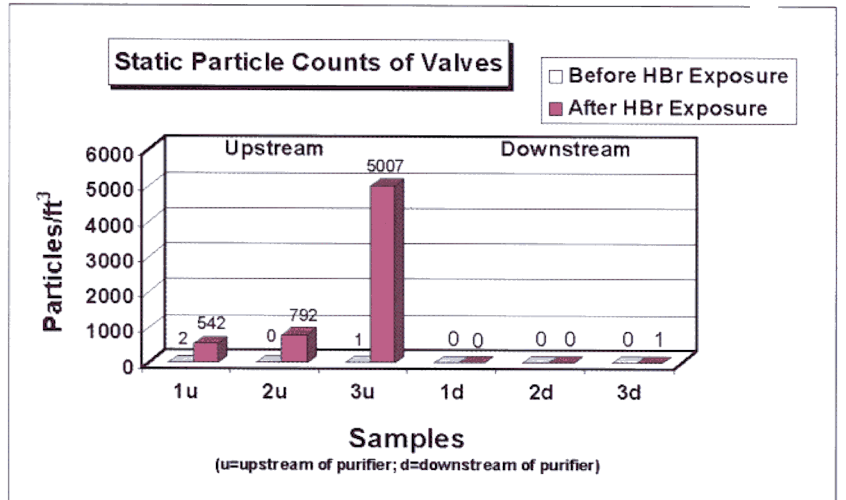


Figure 5 Average static particle counts of valves before and after exposure to HBr

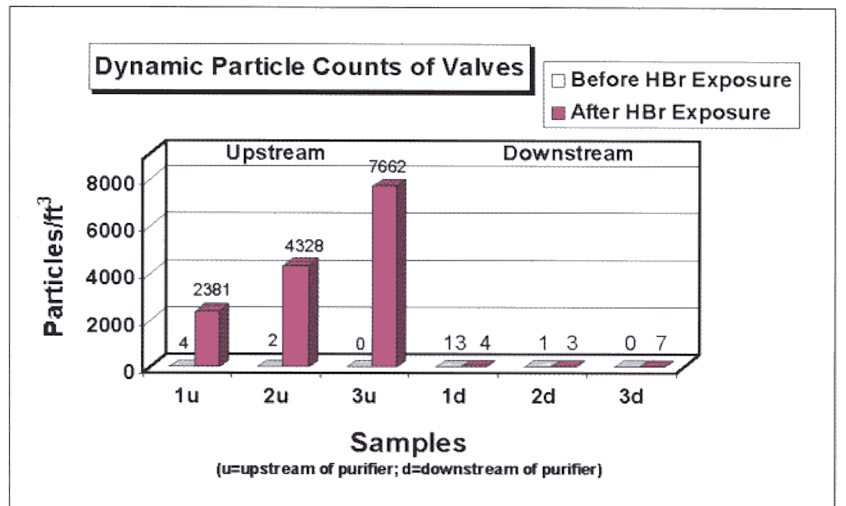


Figure 6 Average dynamic particle counts of valves before and after exposure to HBr

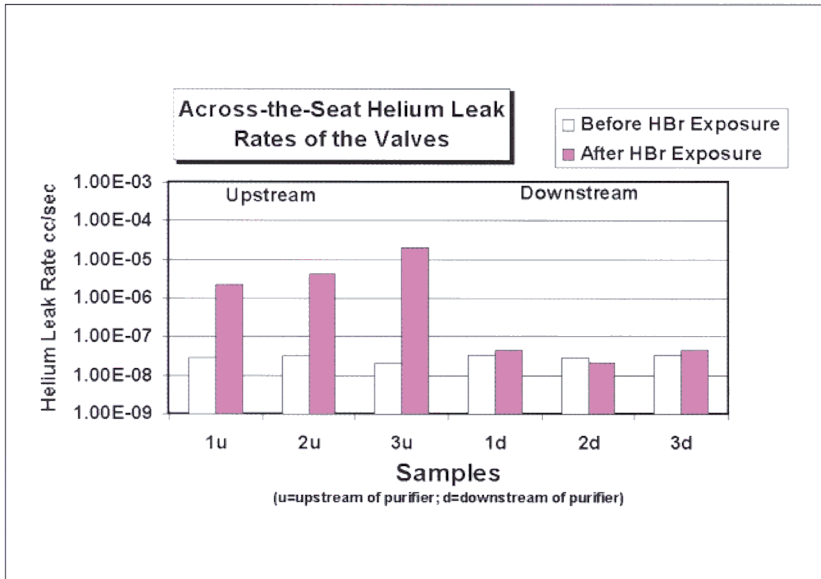


Figure 7 (above)
Across-the-seat helium leak valves before and after exposure to HBr

Figure 8 (right)
Polymer seat of a line valve, after exposure to HBr, placed upstream of the purifier

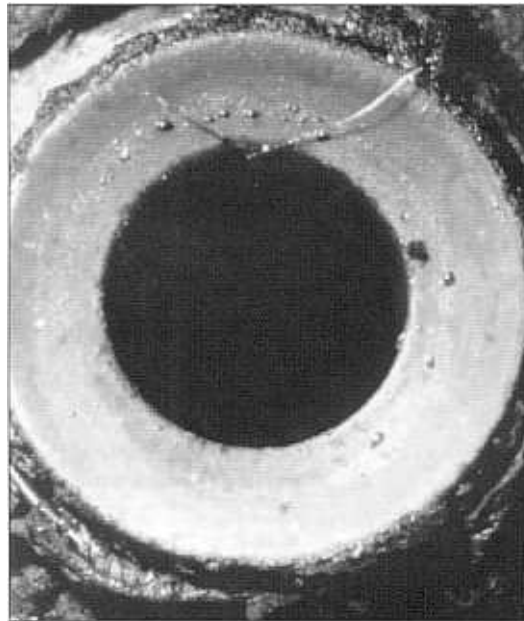
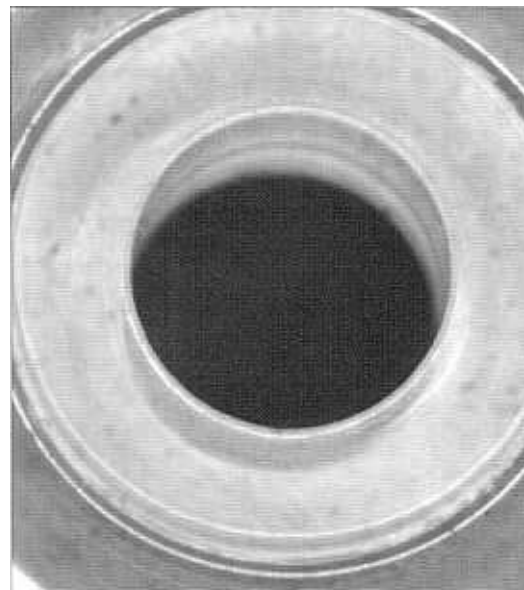


Figure 9
Polymer seat of a line valve, after exposure to HBr, placed downstream of the purifier



change significantly with the valves in the open position. The inboard leak rate data (before and after exposure to wet HBr) reveal that the HBr did not have an effect on this parameter.

The average across-the-seat helium leak rate for the upstream valves before HBr exposure was found to be 2.7×10^{-8} cc/sec. The average across-the-seat helium leak rate for the upstream valves after HBr exposure was found to be 8.9×10^{-6} cc/sec. This corresponds to an increase in the leak rate of over 300 times for the valves placed upstream of the purifier. The average across-the-seat helium leak rate for the downstream valves before HBr exposure was found to be 3.3×10^{-8} cc/sec. The average across-the-seat helium leak rate for the downstream valves after HBr exposure was found to be 3.7×10^{-8} cc/sec. This corresponds to an insignificant increase in the leak rate for the valves placed downstream of the purifier. Figure 7 gives individual leak rate values for each sample tested. The large increase in the leak rates for the upstream valves was attributed to corrosion material being deposited between the valve body and polymer seat. Examination of the upstream valves revealed the sealing area to be highly contaminated with what was presumed to be iron bromide. Examination of the downstream valves revealed the sealing area to be free from any debris, with minimal discoloration. Figures 8 and 9 visually illustrate the observable differences between the upstream and downstream valves.

CONCLUSIONS

The research described here provides direct quantitative information on the value of using a POU or source gas purifier. This was accomplished by conducting particle counts and leak rate measurements of components before and after exposure to HBr, upstream and downstream of the purifier. The effect of the high-pressure purifier was investigated under severely corrosive gas conditions.

The test components (gas sticks and line valves) and purifier were exposed to various experimental conditions. The purpose of this variation was to simulate different conditions that an actual gas distribution system could encounter during normal operations. In all cases, as illustrated by the data, the components downstream of the purifier were essentially unaffected by the exposure to the HBr. The components upstream of the purifier encountered severe degradation in performance and were visibly observed to exhibit severe corrosion.

Further evidence to support the value of using POU purification is illustrated in Figure 10. The photograph shows the wetted diaphragms of two line valves that were in HBr service for three years. The top diaphragm was upstream of the purifier and shows signs of severe corrosion, while the bottom diaphragm was downstream of the purifier and appears to have been unaffected by the gas.

The gas piping samples that were placed upstream of the purifier revealed a dramatic difference in static, dynamic, and impact particle measurements compared with the downstream gas sticks. The results from the static, dynamic, and impact particle count tests represent differences in the amounts of particle contamination between the purified and non-purified gas sticks of >100, >200, and >500 times, respectively. What is even more impressive is the fact that the particle levels for the downstream samples are near zero. This indicates that the purifier has a large impact on control of particulate contamination.

Particle levels for the valves revealed similar trends. The data for static and dynamic particle levels show the expected result of higher particle levels for the dynamic tests. The results from the static and dynamic particle count tests represent differences in the amounts of particle contamination between the purified and non-purified valves of >2000 and ≈1000 times, respectively. This result is even more surprising than the results from the gas sticks. The increase of total particles in the valves compared with the gas sticks was an expected result. However, the near-zero particle levels in the downstream valves were quite surprising.

Helium leak rates (across-the-seat) increased over 300 times for the valves placed upstream of the purifier compared with the downstream valves. The large increase in the leak rates for the upstream valves was attributed to corrosion material being deposited between the valve body and polymer seat. Examination of the upstream valves revealed the sealing area to be highly contaminated with what was presumed to be iron bromide. Examination of the downstream valves revealed the sealing area to be free from any debris, with minimal discoloration.

The data conclusively indicate that the purifier has a large impact on control of particulate contamination, and clearly demonstrate the value of using POU and source gas purifiers within gas distribution systems.

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Figure 10
Wetted surfaces of upstream (top) and downstream (bottom) valve diaphragms from a purifier that was in HBr service for three years



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Dr. Robert Torres is Manager of Gas Technology at the Matheson Tri-Gas Advanced Technology Center. His research group is focused in two areas: understanding microcontamination in electronic gases and development of gas purification materials.

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