ZERO DEFECTS

Entegris Newsletter

June 2014

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Entegris, a Leading Supplier of Yield-Enhancing Technology for Advanced Manufacturing

Entegris announced on April, 30 that it has completed its acquisition of ATMI, Inc. (ATMI), creating a leading supplier of products and materials for semiconductor and other advanced manufacturing.

Bertrand Loy, President and Chief Executive Officer of Entegris said, "We are excited to bring together two strong, successful companies with premium brands and incredibly talented teams. As one company, we are creating a very compelling platform for our shareholders, customers and employees. As our customers continue to develop and ramp new, ever more complex and advanced manufacturing processes, we are positioned to leverage our innovative energy and unique strengths to bring a broader portfolio of yield-enhancing solutions to market faster than before."

The combined company, which employs 3,500 people worldwide, has pro forma 2013 revenues of more than $1 billion and adjusted EBITDA of approximately $248 million, adjusted for targeted annualized cost synergies of $30 million, transaction-related costs and other one-time items.

>> Visit www.atmi.com

Entegris @ SEMICON® China

Entegris participated in Semicon China, Shanghai from March, 18-20.

Over 550 people visited the Entegris booth to be informed on our advanced and innovative solutions -see picture of the Entegris Sales Manager Liu Bin introducing products used in photolithography process to a visitor.

Following this successful show we have decided to book a bigger booth for Semicon China 2015. Let's meet next year @ the Shanghai New International Expo Center!

Semicon China
March 17-19, 2015
Entegris Booth #3008
Hall W3

Meet Entegris @ SEMICON® Taiwan

Entegris will present our latest yield-enhancing materials and solutions at SEMICON Taiwan. Please join our SEMICON Taiwan event on Facebook to stay connected with the latest activities announcements before and during the show ! Looking forward to see you this year.

>> www.semicontaiwan.org

>> Exhibition floorplan

Visit Entegris
at Booth #176
Upper Level 4F Area N
September 3-5, 2014

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Publisher: Entegris Asia

Process Stability

CMP Filter Characterization with Leading Slurry Particles

By YiWei Lu, Bob Shie, Steven Hsiao, HJ Yang, Sherly Lee , Asia Application Development and Laboratory - Entegris Taiwan

Chemical mechanical planarization (CMP) slurries contain a small amount of large particles that will contribute to micro-scratches on a wafer's surface. Capturing large particles from slurry with high solids concentration without changing the working particle distribution is one of the main challenges for a slurry filter. In general, filter performance evaluation utilizes Polystyrene Latex (PSL) beads to determine particle-size retention. The PSL retention test provides good resolution under low particle content conditions. However, it is not relevant when simulating high solid content solution, such as CMP slurry. Therefore, developing a new method to fill this technology gap is critical.

This study focuses on characterizing CMP filter performance by using ceria (CeO_2) and silica (SiO_2) particles and comparing PSL bead retention. Based on our study we have developed a new method to evaluate slurry filters. Using this new method further identifies the filtration retention efficiency and emphasizes the discrepancy between commercial slurry and PSL beads. It also helps to advance new sub-100 nm media development for CMP filtration.

Retention Test Materials

Retention with PSL beads

PSL (polystyrene latex) beads are a general method used to define particle removal efficiency of the filters. Retention with PSL beads for CMP filters has been used generally to confirm filtration efficiency.

Retention with slurries

Retention with slurries can be index of particle removal efficiency of CMP filter for slurry. There are various slurries used in applications, so this test can be applied for specific slurry.

Retention with abrasives

Retention with abrasives can be an alternative for retention with slurries once the performance between abrasive and slurry is confirmed. Abrasive type and concentration can be adjusted based for this test type.

Slurry	Abrasive	Chemical Formula	Application
Silica base	Colloidal silica	SiO_2	STI, ILD, metal
	Fumed silica		
Ceria base		CeO_2	STI, ILD

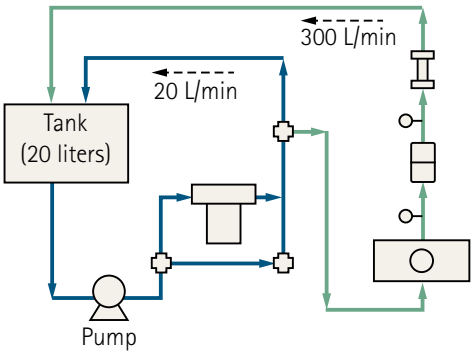
Abrasive Type	Concentration (%)	Shape	pH	Dilution to 1% pH
Colloidal silica (CS)	20	Spherical	7.3	6.8
Ceria (CE)	30	Irregular	6.6	6.4

Experimental

The two abrasives were diluted with DI water to 1% concentration and fully mixed for 40 minutes to prepare for the filtration test. After the mixture is complete, the pH value is measured.

Experimental procedure:

- Dilution abrasive to 1% concentration
- Install filter into CMP test stand
- Make pressure activation operation
- Used 1% concentration abrasive flush filter and all system 5 min
- Collect downstream sample for LPC measurement
- Collect upstream sample for LPC measurement
- Continue record pressure increase by time



CMP test stand configurations

Results

LPC Result

From this experiment, the results show colloidal silica (CS) and ceria (CE) slurries show a different LPC curve model.

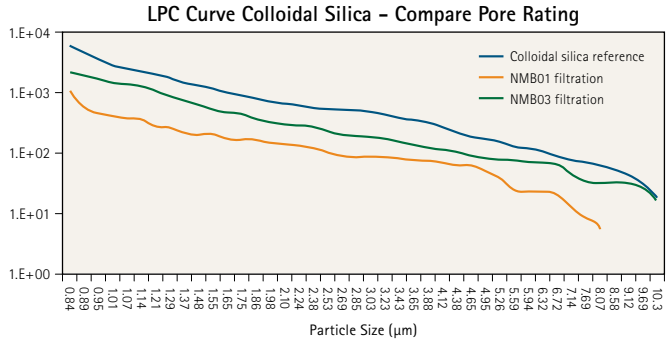
Colloidal silica particles decrease significantly after filtration. The LPC curve from small to large shifts to a low level through this test shows capture of the large particles from the slurry. By comparing different retention ratings, it could help distinguish which is more suitable for the particular product application. This test method is also providing new CMP filter media development evaluation references.

Ceria abrasive particles decrease significantly after filtration with particles larger than 2 µm being completely removed.

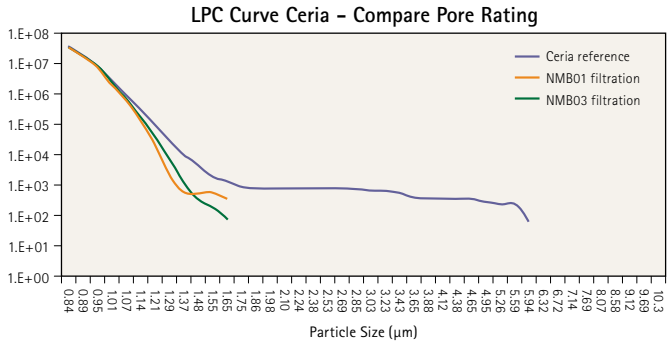
Comparing differential filter pore size ratings, the LPC curve is similar but still can distinguish the improved performance of NMB01 over NMB03.

continued overleaf

Process Stability



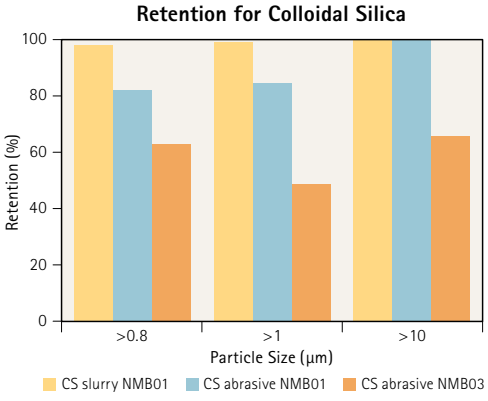
LPC curve colloidal silica—compare pore rating



LPC curve ceria—compare pore rating

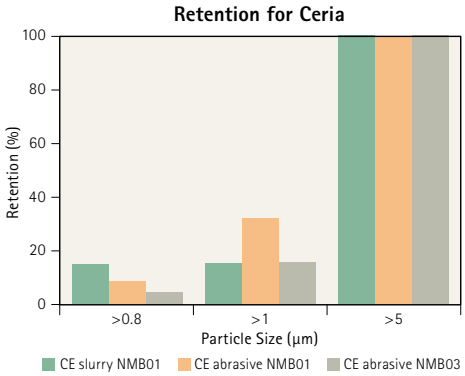
Retention Result

Colloidal silica-based slurry shows a different retention bar chart than ceria-based slurry. Colloidal silica slurry shows very good retention at >0.8 µm, colloidal silica abrasive has similar trend with commercial slurry. We can use colloidal abrasive particle for experiments to study Colloidal Silica-base slurry filtration behavior.



Compare colloidal silica challenge particle

Ceria slurry shows very good retention large particle scale, Ceria abrasive also has similar trend with commercial slurry. We can use this method to distinguish which one is more suitable for the particular product application.



Compare ceria challenge particle

Conclusion

PSL, slurry and abrasive are suitable for filter evaluation, but which one is closer to real condition? Comparing these three methods, we can see the retention results are different, but have a similar trend.

PSL retention is a more suitable representation of micron-scale pore rating filter performance. Slurry/abrasive retention is a suitable representation of micron to nano scale pore rating filter performance.

Selection of a pure abrasive particle is effective for filter evaluations. Abrasive not only distinguishes different pore size rating performance, it also provides the end user and slurry matching reference. For future study we can add some activate to adjust slurry condition to help CMP filter upgrade.

Process Stability

Advanced FOUP Purge Using Diffusers for FOUP Door-Off Application

By Huaping Wang , Manager, Technology Characterization Laboratory - Entegris

As the IC fabrication technology node advances, the need for minimizing the exposure of in-process wafers to airborne molecular contamination including moisture and oxygen has become so stringent that some processes require FOUP to be purged while the FOUP door is off on an Equipment Front End Module (EFEM) loadport.

In this paper, published at ASMC™ 2014, we presented **Entegris' latest experimental study on understanding the unique challenges of FOUP door-off purge and the excellent test results of newly designed advanced FOUP with purge flow distribution manifolds (diffusers).**

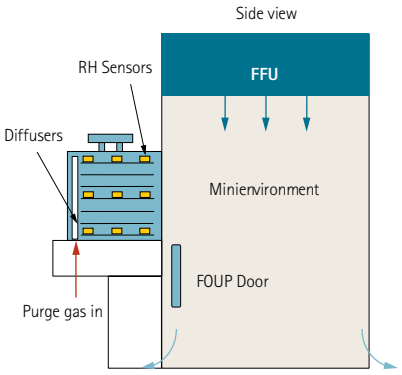
Background

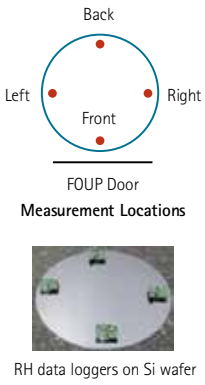
Classic purge scheme used in door-closed purge of introducing purge gas at high velocity through holes (inlets) on bottom of FOUP and mixing with air inside to dilute and carry away the contaminants no longer works for door-open purge. The ambient air tends to enter the FOUP through the front opening due to vortices created by the downward airflow inside the EFEM. The classic purge scheme would enhance the vortices and draw more ambient air into the FOUP during purge primarily through the bottom half of the FOUP (see the flow visualization photo to the right).

Purge with distribution manifolds (diffusers) which creates a plug flow can effectively push out the contaminants uniformly and quickly.

Test System and Method

- RH sensor: wireless RH data loggers (Dickson® model TK550 with casing removed)
- Purge gas: Nitrogen
- Minienvironment: air flow velocity 0.4 m/s, pressure 2.5 Pa

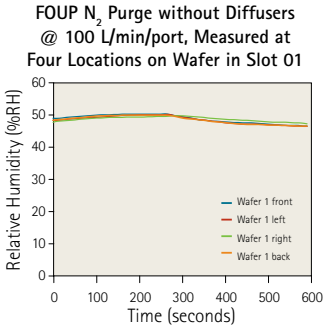


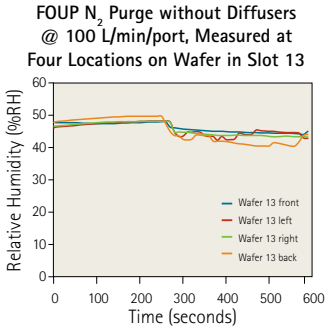


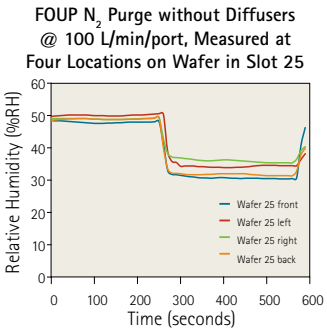
RH data loggers on Si wafer

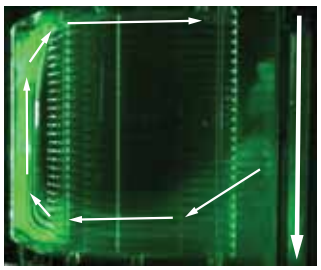
Results/Findings

Classic purge can't effectively control the RH inside a FOUP during door-open purge.



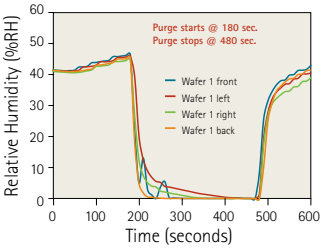


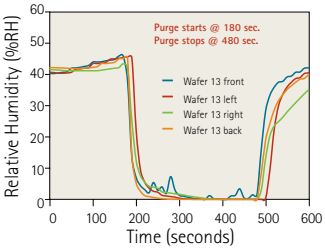


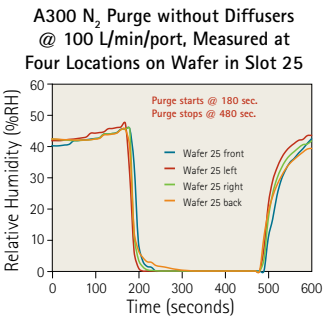


Vortices inside an open FOUP due to downward airflow of the minienvironment

FOUP with diffusers can control RH to below 10% level uniformly during door-open purge.







A300 FOUP with diffusers

Conclusion

Entegris' advanced diffuser purge solution implemented in A300 FOUP platform is shown to be very effective in creating and maintaining a below 10% RH environment in all wafer slots during door-off purge. The same diffuser solution is also being implemented in Entegris' 300 mm Spectra™ FOUP and 450 mm FOUP.

Yield Improvement

A Product Offering to Monitor Chemical Mixtures of Raw Feedstock to Proprietary Blends for Chemical, Process and Equipment Engineers

By Chris Farmer BSEE/MBA, Global Product Specialist - Entegris

Metrology is nice to have until your team needs it. Inline NX-148 process chemical monitoring gives assurance that chemical concentrations are at target. Bulk Chemical and Process Engineers have many metrics to keep track of and real time monitoring can reduce grab sample frequency saving time and money. But more importantly can give an added layer of control over incoming raw/bulk chemicals from suppliers. If incoming ratios are off how can process engineers accurately mix by volumetric flow?

In this note we illustrate **how the NX-148 refractive index technology can verify incoming bulk chemicals and accurately monitor point of use additives. Our purpose was to find root cause of wafer defect.**

Incoming Bulk Chemical Tested

Seven Barrels of incoming Chemicals from sub-fab delivery unit.

Experimental

To determine cause of chemical concentration change a NX-148 was installed on a chemical delivery tool which took samples from seven different bulk containers.

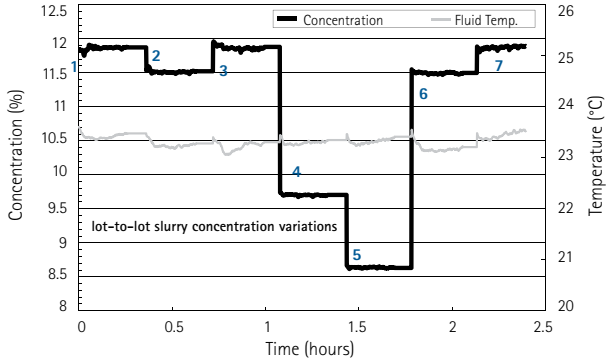
Step 1: Install sensor, including calibration, temperature compensation, and rezero.

Step 2: After initial setup monitoring began by sampling barrels as they were consumed and mixed into the system.

Step 3: To build confidence in the readings different batches were sampled.

Step 4: As a control measure we requested temperature to be controlled down to a 0.2°C change. To accurately monitor concentration temperature must be controlled or compensated for.

Results

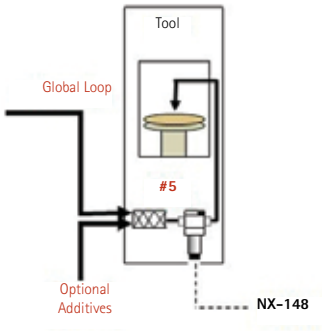


Variation from batch to batch is easily noticeable, nearly a 3.5% range. Five of the samples monitored were roughly the same concentration percentage, but even small differences can lead to product yield defects. Customers often require monitoring capabilities into the hundredths place. We quote our NX-148 product down to a 0.01 wt% resolution. With incoming chemical variations of this magnitude the NX-148 product will have no problem detecting and signaling an engineer.

Possible causes of lot-to-lot change could be evaporation, moisture absorption from ambient humidity, mixing errors from supplier, or mixing errors from components on tools, such as flow controllers.

Point of Use Chemical injection

To help with evaporation and absorption the NX-148 can be used to monitor and spike in additives close to the process chamber or point of use (POU). The challenge here is to create an accurate calibration table. Once calibration is complete monitoring point of use additives can be accomplished.



Conclusion

Results show and customer references conclude, **in-line monitoring gives instant feedback to process chemical concentration. Process engineers can take data captured by the NX-148 product and overlay with product yield results confirming efficiency and reduce defects.**

A Method for the Combined Measurement of Volatile and Condensable Organic AMC in Semiconductor Applications

By Charles M. Miller, Emily C. Zaloga and Jürgen M. Lobert - Entegris, Inc.

As lithography processes advance and become more sensitive, the need for accurate measurements of particularly volatile organic AMC is critical to gauge EUV system exposure to total carbon load and evaluate where AMC mitigation is necessary.

The current industry standard uses a polymer-based sample trap type that is not suitable for measuring volatile compounds. It also produces substantial artifacts when heated or exposed to inorganic acids in fab environments.

This study, presented at SPIE® Advanced Lithography conference, shows results from a new type of carbon based sample trap, which offers superior performance across the entire range of organic compounds. Accurate and artifact-free results lead to a better understanding of total organic AMC load for process control and can help reduce costly carbon deposition on EUV exposure system mirrors.

Development of the analytical method was divided into four parts, (a) finding the most suitable sample trap, (b) developing a similar cold trap for pre-concentration, (c) finding the most suitable analytical column and (d) developing a suitable separation method with optimized resolution for all compounds in a reasonable time. **We propose to establish this analytical method as a new industry-wide standard for the combined measurement of volatile and condensable organic AMC**. Please contact Entegris for licensing details. Key abstracts of the study are highlighted below. If you are interested to get the original poster, please contact europe@entegris.com.

Sample and Preconcentration Trap Selection

Stage one consisted of identifying a sample trap with strong adsorbent beds capable of capturing volatile organic AMC and a weak bed capable of capturing and releasing condensable organics up to hexacosane (C26). To find the most suitable, commercially available sample trap, we compared six trap types using standard LeanSigma® tools and slowly narrowed the selection until we concluded that there is only one good solution that is commercially available. Commercial availability was important, as we set out with a new industry standard in mind.

Trap Identification	AMC	Outgassing (µg/g)
2,6-diphenylene-oxide polymer resin	D3	0.2
	D4	0.3
Carbonaceous sample trap	SO_2	0.1
Carbonaceous cold trap	CO_2	0.2

Table 1: Adsorbent Trap Outgassing Results

Adsorbent Cleanliness and Artifacts

In contrast to polymer-based traps, adsorbents used for the carbonaceous traps did not exhibit any organic outgassing (Table 1). Minor amounts of carbon dioxide were detected in the cold trap and some sulfur dioxide was found in the sample trap, neither affects organic AMC analysis.

Each adsorbent trap was sampled with a mixed challenge of acetic acid, nitrous acid, trifluroacetic acid and limonene, commonly known to cause artifacts on Tenax®.[1] Artifact concentrations (Table 2) were determined by subtracting the results from challenge testing from the outgassing values. No such artifacts were found in the carbonaceous sample or cold traps.

Artifact Identification	Artifact (µg/g)
Benzene	2.0
D3	0.6
Benzaldehyde	1.8
D4	2.2
D5	1.0
2,5-diphenyl 2,5-cyclohexadiene 1,4-dione	97

Table 2: Artifacts produced on polymer-based sample trap

Precision and Accuracy

Our method development targeted an accuracy within 10% of actual concentration. When sampled on Tenax, 95% of the acetone was lost and retention and recovery were less than 5% of the actual amount. When sampled using the carbonaceous sample trap, capture efficiency and release were within 97% of challenge concentration.

When using statistical software to analyze accuracy data, the probability of measuring acetone outside ±10% of known value on our carbonaceous trap was only 0.8%, the equivalent of one measurement outside the control limits in 125 at 99.97% confidence. Precision and accuracy measurements for acetone are depicted in Figure 1.

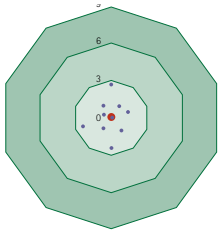


Figure 1: Precision and accuracy measurements for acetone: deviation (in ppbV) from actual 53 ppbV challenge.

Even though trimethylsilanol (TMS), a volatile compound, was fully retained after collecting 32 liters of air, recovery of TMS varied dramatically and cannot be considered accurate,[2] see next section. Capture efficiency and recovery was tested with additional compounds and results were consistent and similar to acetone.

Desorption Efficiency/Recovery

Desorption efficiency of each carbon trap was determined by comparing the response of an absolute concentration (µg) from an on-column injection to two manually spiked sorbent traps (one purged with 40 liters of XCDA®). Desorption efficiency for C26 was greater than 94% for each trap (Table 3).

Trap Type	Desorption Efficiency
Cold trap	95%
Sample trap	94%

Table 3: C26 desorption efficiency - comparison to on-column injection

Desorption efficiency for each trap was compared and averaged 99.7 ±0.32% (Table 4). Desorption efficiency of octacosane (C28)

continued overleaf

was also studied using similar techniques, but recovery of the compound was less than 85%.

Desorb Cycle	Trap 1	Trap 2	Trap 3	Trap 4
1	100.0%	100.0%	99.4%	99.5%
2	0.0%	0.0%	0.6%	0.5%
3	0.0%	0.0%	0.0%	0.0%

Table 4: C26 desorption efficiency measured by multiple desorption cycles

As mentioned, we found TMS recovery to vary substantially with the age of the trap, we consistently find TMS being retained and not released in the gas phase on aging carbonaceous sample traps, which was the reason for developing a stand-alone analytical method for TMS.[2] With each thermal desorption cycle of the trap, recovery of TMS appears to diminish further.

Performance Under Humidified Conditions

Hydrophobic adsorbents were used for both the new sampling and cold traps. Performance of the proposed traps was excellent at zero and 50% relative humidity (Table 5). This is an important result, as the traps are used for AMC detection in both dry supply gases (CDA, N_2, CO_2) and humidified environments (air handlers, cleanrooms, subfabs, AMC filter cabinets).

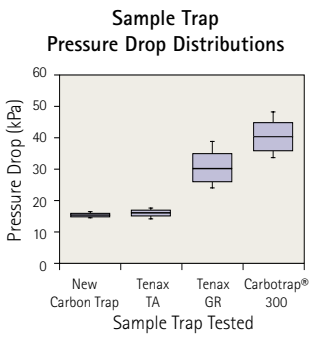
Compound	RH	Challenge (ppbV)	Measured (ppbV)	Capture Efficiency
Acetone	0%	53	53	101%
Acetone	50%	26	26	101%
Benzene	0%	41	43	104%
Benzene	50%	57	56	98%
Hexadecane	0%	7.9	8.1	103%
Hexadecane	50%	17	17	100%

Table 5: Analysis results for volatile and condensable organic AMC on the proposed multi-layered sample trap

Sample Trap Pressure Drop

Sample trap pressure drop is important to ensure that standard, low cost pump equipment such as Pocket (SKC) can be used for AMC sampling.

The trap we propose has a pressure drop very similar to that of the polymer traps, allowing for the use of all types of and including legacy pumps. Distribution of pressure drop measurements is listed in the Figure. Box "whiskers" indicate high and low reading, the box itself represents plus/minus one standard deviation.



Selection of Preconcentration Trap

The second stage focused on identifying adsorbents to create a cryogenic focusing trap (cold trap) capable of analyzing both volatile and condensable organics.

To replace the commonly used Tenax-style cold/preconcentration trap in GCMS systems, we looked at available solutions and were not able to find a commercially available trap. However, we developed an easy way to manufacture these traps in small lab settings, using empty cold trap tubes as supplied by GCMS vendors for the desorption system and filling them with several layers of different carbons to mimic the sample trap behavior and capabilities. The multi- layered, carbon-based cold trap that we developed eliminates the formation of organic artifacts and also reduces moisture retention.

Selection of Separation Column

Stage three was to identify a low-bleed separation column capable of separating isopropyl alcohol and acetone as well as other low boiling "refractory" compounds (such as volatile, halogenated refrigerants) commonly measured in semiconductor fabs.

The separation column we selected for our new method completely resolved IPA and acetone, while maintaining low column bleed at the upper temperature limit. Resolution for low boiling point, halogenated ("refractory") compounds was substantially improved, allowing for increased accuracy of their measured concentrations and reporting of individual compounds rather than that of a merged signal.

Conclusions

By using the proposed carbon-based sample and cold traps, improvements to AMC measurements can be made in these areas:

- **Adsorbent cleanliness** – carbon-based sample and cold traps do not exhibit any organic based artifacts and produced only minor inorganic AMC.
- **Accuracy and capture efficiency for volatile organic AMC** – carbon-based sample and cold traps fully retain all organic compounds, including IPA, for four or more hours of sampling at 0.15 L/min (36 liters of volume).
- **Desorption of high molecular weight organics** – the desorption efficiency of hexacosane, the upper end of the combined organics range, was about 95%.
- **Cost reduction** – combining the sampling and measurement of both volatile and condensable organic AMC into one analytical method saves cost and makes adoption of this method as an industry standard more likely.

1. Kleno et al, Degradation of adsorbent Tenax TA by nitrogen oxides, ozone, hydrogen peroxide, OH radicals and limonene oxidation products, Environ Sci. Technol., 2002,36, 4121-4126.

2. Lobert et al., Measurement of low molecular weight silicon AMC, Proc. SPIE, 7272-81 (2009).

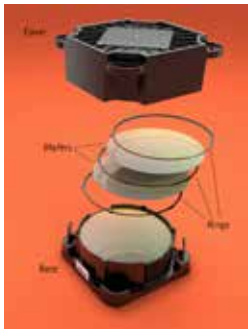
Product Highlight

Contactless Horizontal Wafer Shipper and Moisture Barrier Bags

The industry requirements towards smaller, higher performing and lower cost device confirgurations have lead to the contactless Horizontal Wafer Shipper designed by Entegris for shipping of thin, 3D, lensed or bumped wafers/substrates (see Zero Defects - September 2013):

- Less components to order and inventory
- Improved shipping density
- Improved lead time
- Better quality control
- No chemical contamination and mechanical surface contact on the wafer
- Better shipping density
- Designed for auto-compatibility

▸ Combined with a secondary packaging Entegris ensures your wafers are safe and clean during shipping and storage.



Secondary Packaging: moisture barrier bag, cushion and shipping box

Moisture Barrier Bags

The SmartStack® horizontal wafer shipping system is designed to reduce wafer breakage, die bond corrosion and particle contamination, increasing IC fab yield and profit.

Our moisture barrier bags are designed to protect wafers while used with our secondary packaging.

They are ideal for medium to full vacuum packaging, shipping and handling of ESD-sensitive devices with contour surfaces.

Features and Benefits

Part Numbers	Features	Benefits
HWS-CMB-BAG & HWS300-CMB-BAG	Clear bag ESD shielding plus medium level moisture barrier	Clear barrier to see product, ID labels and bar codes inside the sealed bag Long-term ESD and moisture protection in Controlled Discharge Environment Puncture resistance for more handling steps
HWS-MB-BAG & HWS300-MB-BAG	Metallic bag ESD shielding plus medium level moisture barrier	Long-term ESD and moisture protection in Controlled Discharge Environment Puncture resistance for more handling steps

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