



Reducing
Contamination
Points in Clean
Chemical Delivery
from Manufacture
through Point of Use

White paper

INTRODUCTION

As mega-trends, such as artificial intelligence and robotics, smart homes and smart cars, and the Internet of things evolve to meet growing needs for speed, scale, and reliability, they force integrated circuit (IC) manufacturers to increase processor power efficiency and memory size. At the same time, device manufacturers striving to meet new worldwide consumer and business data demands at lower costs, face significant challenges in terms of process control, yield, and economics.

As logic devices migrate to smaller line widths, 3D NAND architectures increase layers, and DRAM memory density increases, sensitivity to contamination and defects have a greater impact on device performance. To achieve optimum wafer yield and reliability, the microelectronics industry needs to address the increased materials consumption requirements and the material purity challenges for these high-performance technologies from chemical manufacture to their point of use.

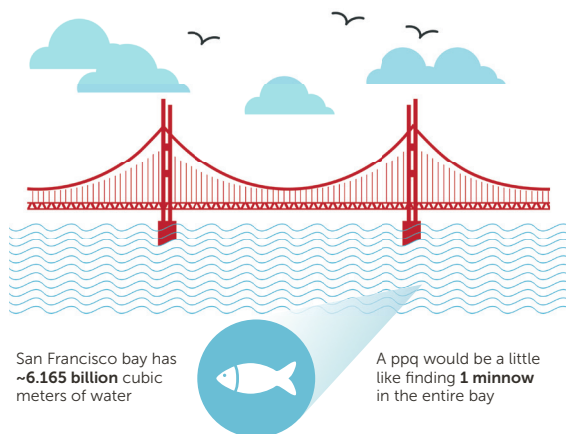


Figure 1. Next generation semiconductor manufacturing purity levels are approaching parts per quadrillion (ppq).

Controlling contamination begins with the chemicals that come into direct contact with every wafer. Increasing chemical purity is the first step in enabling process cleanliness and improving device yield. This is why device manufacturers continue to pressure chemical suppliers to deliver a higher level of purity, soon approaching parts per quadrillion (ppq), and avoid introducing contaminants during chemical packaging, transport, and distribution.

Equally important are material purity and the materials of construction in fluid delivery systems. Without contamination-controlled chemical packaging, filtration, pumps, and fluid handling components, chemicals are vulnerable to recontamination by particles, metals, and impurities. Conducting contamination mapping to understand the contamination source is critical to defect control, yet it can be especially challenging when manufacturers source products with varying cleanliness levels and combine batches from many different vendors.

Chemical manufacturers, OEMs, and device manufacturers need to work together with component suppliers to manage contamination and maintain higher purity levels in chemicals, storage tanks, and fluid delivery systems. This paper looks at purity challenges in two key areas, particularly particle shedding and leachable metallic contamination, and the benefits of investing in cleaner fluid systems to more comprehensively meet the purity specifications that are driven by leading-edge technologies.

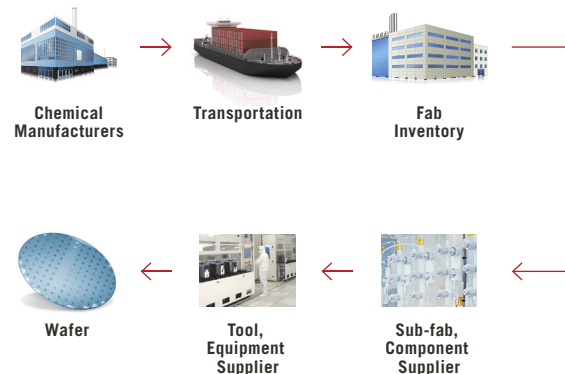


Figure 2. To maintain a clean fluid path from chemical manufacture to point of use, the entire supply chain must work together to manage contamination and ensure clean chemical delivery.

MITIGATE PARTICLE CONTAMINATION WITH CONTAMINATION MAPPING

As the consumption of high-purity chemical increases and the purity requirements become more stringent, chemical manufacturers are having to invest considerably in preventing particle contamination, as well as in maintaining chemical purity during transportation to the fab.

A large majority of the time and expense of qualifying a new chemical line comes from flushing tubing, fluid components, and filters to eliminate surface contamination. The semiconductor industry spends \$2 billion annually on cleaning chemicals. We estimate that as much as 10% of that is used for non-production activities such as line flushing to clean fluid flow paths and ensure a contamination-free process. Particles are often resistant to initial flushing and as chemical sits in long tubing runs or in re-circulation loops, particles shed off the material, contaminate the fluid stream, and become defects on the wafer. To mitigate the need for this expensive and time consuming flushing, and to reduce long-term particle shedding, chemical manufacturers are very interested in components using ultraclean and stable polymers. Component suppliers are working with them to develop solutions that have lower surface particulates, and to determine what impact that has on their system.

Clean chemical delivery is more important than ever because as circuit line widths continue to shrink and process nodes approach sub-10 nm levels, surface particles become especially detrimental to logic devices. Similarly, as the number of transistors increase in a 3D NAND memory stack, one defect could block more than one cell affecting the performance of the entire device. Consequently, all potential contamination areas must be identified and proper steps taken to avoid defects.

Once a system is qualified and chemical is meeting the required purity specification, the focus turns to maintaining that purity as the material is transported to the fab. Chemicals can be susceptible to particle contamination that may be introduced during chemical transport or transfer from bulk chemical containers to smaller drums, transportation packs, and fluid delivery systems. To maintain chemical purity, all the fluid paths require a contamination-controlled, stable environment.

The choice of chemical storage container plays a large role in maintaining chemical purity. If an initially clean photoresist, for example, fails the particle specification when it arrives at the fab, then the transport container is a likely source of contamination. Sourcing containers based solely on material of construction and volume leaves out the critical aspect of the long-term contamination prevention performance. Manufacturers will benefit from working with materials experts who are constantly testing and developing materials of construction and product manufacturing methods that result in lower particle-generating containers. Investing in contamination-controlled chemical transport and delivery systems can help ensure chemical integrity, increase product yield, and reduce financial loss.

Once delivered to the fab, the equipment that dispenses the chemical out of the container, filters it, and distributes it through the fab must maintain the cleanliness of that chemical and not add contaminants to it. The latest SEMI® World Fab Forecast report shows that in 2018, spending on fab equipment investments will reach an all-time high of \$63 billion. This investment surge is due primarily to Samsung and SK Hynix growth primarily in Korea, with some growth in China, Europe, and the United States¹.

Fab Equipping Spending Over Time Region
(All Front End Facilities, Including New, Used, and In-house)

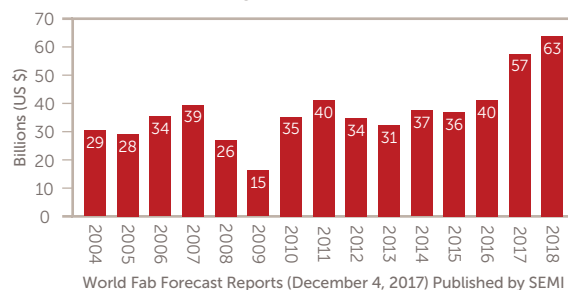


Figure 3.

With so much invested in equipment, it is imperative that this equipment be capable of delivering very high-purity chemical to the fab tools now and into the future. Device makers and OEMs are heavily burdened with evaluating different components to understand relative cleanliness levels and specify components that will maintain the incoming chemical delivery specification. OEMs and fab contractors receive those component specifications and are challenged to choose the right solutions for their equipment that ensure the incoming chemical remains clean throughout their entire system.

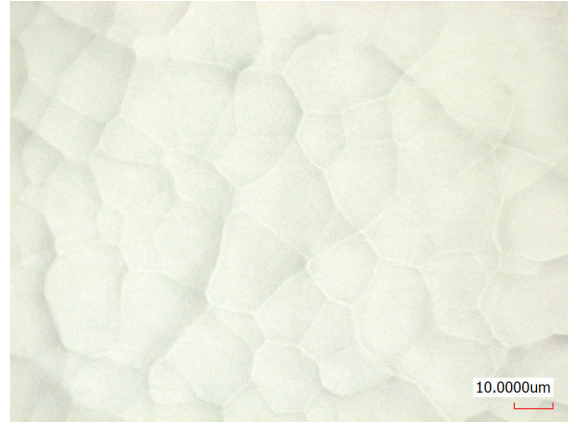
With every fab startup and new build, an enormous amount of fluid handling systems are installed that require pre-flushing, testing, and purging before any chemical is run through them. Pre-flushing is used to leak test the system and rid the system of surface contaminants. Once chemical is going through the system, fab engineers continually monitor chemical purity and wafer defects, and flush the chemical and exercise their process until they get the desired yields. All of this consumes time, wastes chemical, and is very expensive. The goal is to reduce the time and expense of qualifying a process line by sourcing cleaner components and employing metrology early for better defect detection and prevention.

Like chemical manufacturers, device makers will benefit from sourcing a contamination-controlled fluid system that has been evaluated to ensure it is not contaminating the process, but rather improving it if possible. As particle sensitivities increase for more complex memory and logic devices, the need to remove these particles becomes critical. One proven method of controlling contaminants in process streams is through sophisticated liquid filtration. Filter membrane technology has advanced to enable sub-10 nm particle removal, which is vital to improving overall operating efficiency and enabling leading-edge technologies. Through novel polymer design, diverse membrane manufacturing techniques, and advanced cleaning technologies, filter solutions enable device makers to tailor their contamination control based on the chemicals used, and the conditions required. Using process monitoring, statistical analysis, and customer collaboration, these fluid components provide a stable and repeatable solution to control contaminants.

Not all components are the same in terms of particle cleanliness and contamination. Not all HDPE drum suppliers are created equal and this is particularly evident in understanding the importance of resin selection and continued testing, and how to employ process controls to maintain drum purity consistency. This can be achieved by conducting daily particle testing. Additionally, using an assortment of tubing suppliers can significantly increase the risk of contamination variability, as shown in Figure 4. It is important to select suppliers that have a proven understanding of fluoropolymer processing and regularly engage in contamination mapping evaluations to understand potential sources of contamination and how to control them.

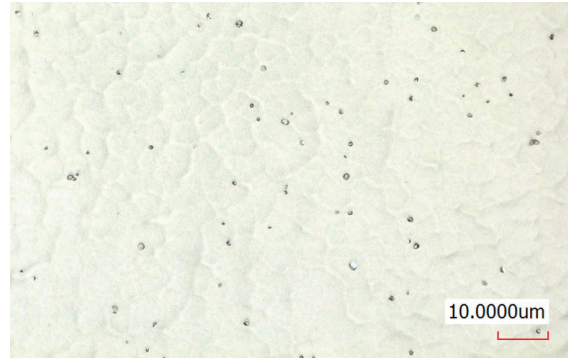
Surface Analysis 3/4" Supplier A

No observable surface contamination in specimen, magnification 100x



Surface Analysis 3/4" Supplier B

Surface contamination present throughout the specimen, magnification 100x



Surface Analysis 3/4" Supplier C

Surface contamination present throughout the specimen, magnification 100x

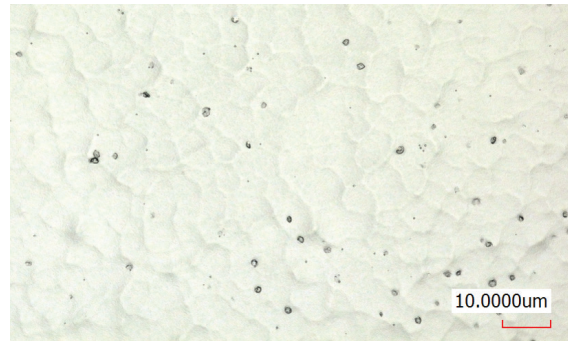


Figure 4. Not all components are the same in terms of particle cleanliness and contamination. Surface analysis shows purity levels can vary significantly from supplier to supplier.

The concept of contamination mapping is to examine the entire fluid system, break it down into individual components, and map out where the particle contaminants come from in the materials used and/or in the processing of them. If the end chemical has a particle spike, mapping the components can help detect which component is contributing the particles. Once identified, the component supplier conducts a contamination mapping evaluation of their process, including the equipment, the materials, and the processes used to manufacture the component. If it is determined that the material is contributing the contamination, then the process goes back one step further in the supply chain to the material supplier who then maps their manufacturing process to determine if the contaminants are in their raw feed materials or in their processing of the polymer. The goal is to take active steps throughout the supply chain to remove contaminant sources.

A key step toward ensuring overall system cleanliness is for end users to partner with component and equipment suppliers that have conducted contamination mapping up the supply chain to understand and lessen contamination sources. Leveraging the contamination analysis capabilities of component suppliers is also key to ensuring consistently clean component delivery to OEMs and fabs.

In addition to evaluating materials of construction that are less prone to particle shedding, there should be continued focus on component configurations and minimizing dead legs and entrapment areas, as well as reducing particles generated by moving parts from valves, pumps, and filters. Computational fluid dynamics analysis is another tool that component manufacturers can use to optimize product designs and improve component flushing performance.

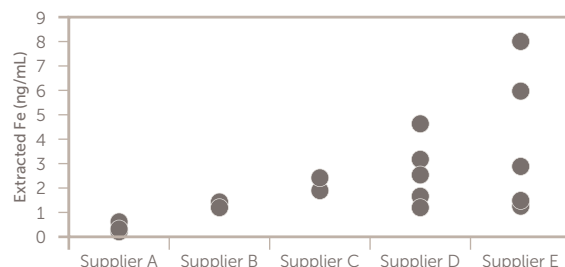
OPTIMIZE CLEAN FLUID PATHS BY REDUCING LEACHABLE METALLIC CONTAMINATION

Semiconductor processes, such as photolithography and wet etch and clean, have become more metal sensitive at advanced process nodes. In all fluoro-polymer delivery systems, metals leaching from material in components such as PFA tubing, valves, and purifiers impact device yield.

No matter how much time or money is invested in flushing systems to rid them of particle contamination, a much longer-term problem exists that flushing will not resolve. As chemical sits in storage containers and long tubing runs, metallic ions are being pulled off or leach out of the material and contaminate the fluid stream. Consequently, in addition to identifying causes for particle contamination, chemical manufacturers and device makers spend significant time interrogating their entire chemical delivery system to determine where metals are coming from.

It can take months for metal contaminants to leach out of materials. Containment vessel manufacturers are looking at ways to reduce metallic contamination by testing innovative polymers that have lower metal content. Component manufacturers are also working to reduce contamination in tubing and other components by researching ultra-clean PFA materials. Chemical manufacturers and fabs are both very interested in clean tubing and are engaged in studies with component suppliers to evaluate tubing materials to determine what impact they have on their systems. It is important to note that not all suppliers perform metals extraction testing on their product, nor have they optimized their processes to reduce contamination. Regular component analysis is critical to detecting process and material excursions that lead to metallic contamination. See Figure 5.

Tube Metal Extraction Using 5% Nitric Acid



Tube Metal Extraction Using 5% Nitric Acid

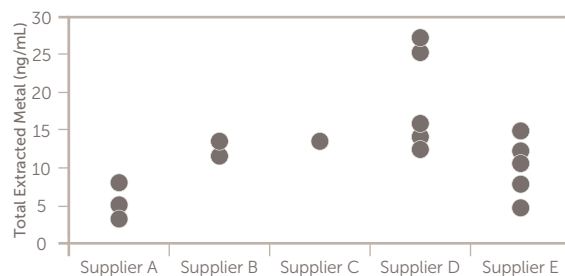


Figure 5. Material grades and process protocols differ, so performing metals extraction testing is critical to detecting process and metal excursions that lead to metallic contamination.

Different PFA grades and how those materials are processed result in dissimilar purity levels. A contaminant that starts in the raw feed can end up in the fluoropolymer, which can then be extruded into the tubing or valve wall, or into the filter membrane where it then moves through the fluid delivery system ending up on a wafer and causing a defect. Contamination mapping can be instrumental in finding the contaminant source.

Resin manufacturing starts with a monomer, which may already have its own contamination, and then is put into a polymerization chamber, which if made of metal, can contribute contamination to the polymer powder. The polymer is then dried and extruded into a pellet, which can also introduce more contaminants into the polymer pellet. When the component manufacturer converts the pellet into tubing, that extrusion process adds another potential contamination source to the final product. Here again, performing contamination mapping of the material supply chain can be critical in identifying the metallic contamination source so immediate corrective action may be taken. See Figure 6.

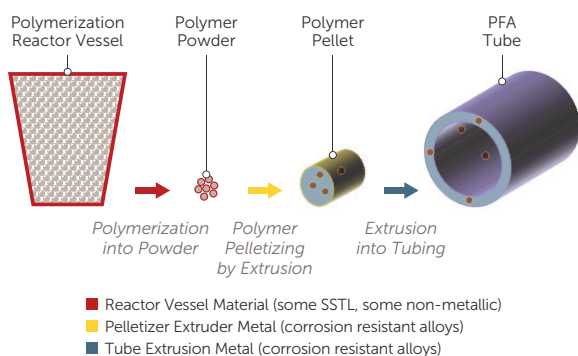


Figure 6. Metal contamination is an impurity, or foreign body in the polymer that can be introduced when polymerizing, pelletizing, or extruding the resin. Identifying where the impurity is being introduced is a critical first step in contamination reduction efforts.

Cleaning process tools and components like photoresist dispense pumps can also reduce the impact of metallic contamination. Photoresist and other lithography solvents do not have a strong affinity to pull out metal contaminants, but they still leach out over time, and chemical interactions between photoresist and metal ions create gel defects and microbridged circuits, which cause electrical shorts on wafers. At advanced logic nodes, these contaminants are detrimental to device yield. The use of solvent purifiers can be especially effective at removing both dissolved and colloidal metal contaminants from a variety of ultrapure, polar, and non-polar solvents used in photoresist applications. Purifiers today remove sub-ppt contaminants and successfully reduce metal-induced defects from raw material manufacturing to wafer dispense.

SUMMARY

Not all material handling components are equal in terms of particles and metals content. Evaluating mechanical components and materials of construction will increase purity levels and help maintain a safe and contamination-controlled chemical delivery environment. It starts with chemical manufacturers around the world working to meet or exceed stringent purity specifications while controlling particle and metallic contamination in containment vessels and distribution systems.

Another key step toward ensuring overall system cleanliness throughout the fluid stream is for end users to partner with component and equipment suppliers who have conducted contamination mapping up the supply chain to understand and lessen contamination sources. Evaluating component cleanliness and sourcing the cleanest chemical delivery, filtration, metrology, and fluid handling systems is an important part of the contamination reduction effort.

The entire semiconductor industry stands to benefit from chemical manufacturers, and equipment and materials suppliers providing contamination-controlled, clean products at each step throughout chemical manufacture to point of use. The result being better device performance, increased yield, and reduced costs.

ENTEGRIS SOLUTIONS FOR ACHIEVING CLEAN CHEMICAL DELIVERY FROM MANUFACTURE THROUGH POINT OF USE

Entegris understands the unique challenges of clean chemical delivery and works with customers to develop and optimize operations by identifying potential sources of contamination and the solutions that remove particle and metal impurities. From safe and efficient chemical storage and delivery systems to a broad range of fluid handling products to expertise in filtration and purification technology, passion for purity leads to integrity of processes and helps increase yield.

ABOUT ENTEGRIS

Entegris is a leader in specialty chemicals and advanced materials solutions for the microelectronics industry and other high-tech industries. Entegris is ISO 9001 certified and has manufacturing, customer service and/or research facilities in the United States, China, France, Germany, Israel, Japan, Malaysia, Singapore, South Korea, and Taiwan. Additional information can be found at www.entegris.com

References

¹ SEMI World Fab Forecast projects new highs in equipment spending, Control Design web site:
<https://www.controldesign.com/industrynews/2018/semi-world-fab-forecast-projects-new-highs-in-equipment-spending/>

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