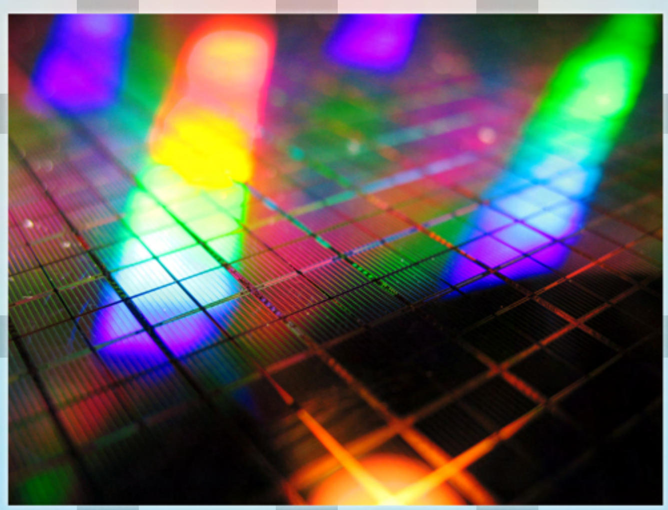
Silicon Coatings for Semicon and Electronics Manufacturing



An eBook from SilcoTek Corporation

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This e-book is dedicated to those who champion design, quality, and efficiency in semiconductor and electronics manufacturing.

1.0: Introduction, About SilcoTek

SilcoTek® is the world's leading provider of high-performance coatings applied by chemical vapor deposition, or CVD. We became a coatings company because we have a passion for helping people and businesses conquer their most difficult material challenges.

In this e-book we take a deep dive into how SilcoTek coatings can improve corrosion resistance, prevent contamination, and improve yield of semiconductor and electronics manufacturing products and systems. We'll discuss how critical manufacturing components benefit from improved corrosion resistance, and purity by using our inert coatings. We'll present data and resources to further support how our coatings help customers save money and enhance the performance of their products and processes. Finally, we'll examine coating applications how to purchase our coatings.

We don't sell coated products because we're solely focused on offering coating services that make your product better. If one of our popular coatings don't fit your application, SilcoTek Scientists and Engineers are often able to craft a custom coating solution that fits your material performance needs. To learn more about our coating process [Watch our video] and see how SilcoTek can improve your products.

Our Mission and ZIP Code

SilcoTek's mission is to provide game-changing coatings for our customers.

What does this mean for you? It means you are doing business with a company where "employees enjoy coming to work as much as going home" and where employees are passionate, enjoy working with each other, and work in an atmosphere based on mutual respect and trust. You'll see the SilcoTek® difference in every order you place.

Our ZIP Code guides the conduct we follow each-and-every day. It creates a culture of success for SilcoTek® and our customers.

What does Z.I.P. mean?

Zero disappointments

We strive for a perfect coating process from quote to shipping, every time.

Integrity in all we do:

We are open, honest, and admit mistakes.

Plus 1 customer service:

We aim to go above and beyond every time you interact with us.

You'll find working with SilcoTek staff easy, helpful, and informative. It's our goal to delight our customers with game changing service and coatings!

Now that you've gotten to know us, let's give you a quick overview of our products.

SilcoTek Coatings Markets and Applications

SilcoTek® coatings are uniquely tailored to solve tough material problems in a variety of industries and applications. We offer over 20 custom coatings for challenging flow path problems. Read about some of our coatings here.

patented process eliminates interactions between materials and a variety of chemicals, liquids, gases, and more to enhance surface performance in a broad variety of applications. Since 1987, SilcoTek has provided surface coating technology solutions to process, analytical, gas & oil, semiconductor, and corrosion applications worldwide.

From prototype to production, SilcoTek offers patented and custom surface coating technologies that allow the user to improve the performance of their processes and products.

A few of our most popular coatings are:

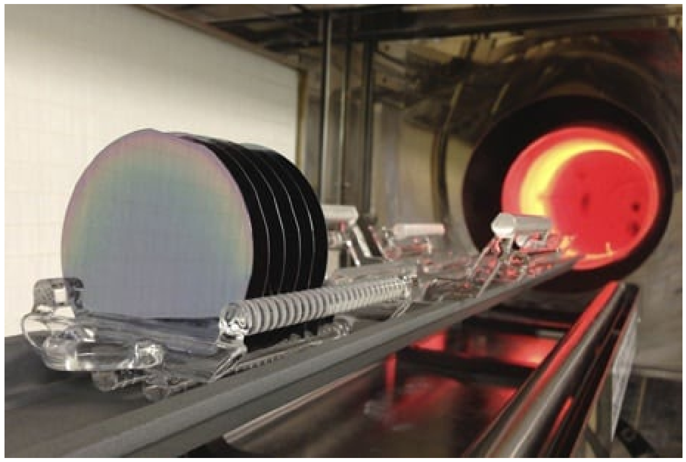
- Silcolloy® : A corrosion resistant silicon barrier coating designed to protect stainless steel surfaces and prevent metal ion contamination.
- Dursan® : A functionalized silica-like coating , NSF certified and FDA compliant process designed to improve the inertness, durability, fouling and corrosion resistance of products ranging from precision instrumentation to severe industrial applications
- Dursox® : A thin but durable silicon oxide (SiO) high purity barrier coating process that prevents semiconductor tool corrosion and erosion

- SilcoNert® : An inert barrier coating designed to improve analytical sensitivity and reliability
- SilcoGuard® : A high purity silicon barrier coating process designed to minimize vacuum system outgassing and contamination in ultra-high vacuum chambers and semiconductor process flow paths.
- SilcoKlean® : A high temperature precision anti-coking coating, designed specifically to reduce carbon coking and improve fouling resistance on stainless steel and specialty alloy surfaces.
- Notak® : Improves surface repelling properties to resist unwanted build-up of water, oils, sticky hydrocarbons, and other foulants.

We also offer specialized coating processes for specific materials or applications. Contact us to learn more.

2.0 Semiconductor Manufacturing Applications and Benefits

How silicon barrier coatings can be used in semiconductor manufacturing.



2.1 Background

With 3D NAND flash technology entering the mainstream, increased utilization of etching (staircase, hard mask, gate trench, channel hole, staircase contact, etc.) has created many quality and operational challenges.[1] Several of these challenges are being addressed through innovative advances with etch equipment. Others have required increased dependence on aggressive etch chemistries to create the necessary chip geometries.[2–5] Unfortunately, that aggressive etch gas can interact with the gas delivery piping (often stainless steel) and lead to corrosion and corrosion byproducts (metal ions or particulate) reaching the wafer surface. The corrosive action ultimately results in lost productivity, frequent maintenance and component replacement, and potential product yield problems. Consequently, improving the corrosion resistance of the gas delivery system has become a higher priority for manufacturers.

Utilizing amorphous silicon barrier coatings deposited by industrial thermal CVD can offer some unique advantages. The inert silicon layer acts as a barrier between interactions of the process gases and the metal tubing. This barrier prevents metal ions (iron, chromium, etc.) from reaching the wafer surface. Additionally, silicon is a commonly used material in semiconductor manufacturing which is, in many steps, being removed already. This makes silicon a non-invasive material option for many processes related to semiconductor production.

The silicon thermal CVD application process does not suffer from line-of-sight limitations like spray-on coatings or physical vapor deposition (PVD) processes, allowing for the coating of complicated three-dimensional geometries without issues like incomplete or insufficient coating coverage.

Components that undergo flexing or bending, e.g., tubing and bellows, can also be coated. The amorphous nature of the silicon coating will allow a significant amount of flexing without the risk of coating fracture or delamination.

Amorphous silicon coatings have a long track record of use in analytical and industrial applications. Historically, coatings like Silcolloy® and Dursan® have been used extensively in liquid and gas chromatography applications to limit negative surface interactions where stainless steel surfaces can cause problems in complex analysis. The oil and gas industry were an early adopter of inert coating technology for the ease of use and accuracy it provides when measuring sulfur content in crude oil in harsh environments.[6]

Inertness, moisture control, and corrosion resistance properties can all be improved by coating the entirety of a sample or process flow path.[7,8] These features can also provide benefit within semiconductor etch applications and other manufacturing processes such as atomic layer deposition, epitaxy, and ozone generation.

2.2 Benefits in Semiconductor Manufacturing

SilcoTek's coating technology is used within the industry to protect against stainless steel corrosion and contamination both during processing and cleaning cycles, for example, in epitaxial depositions where hot (400-500° C), gaseous chlorine (Cl2) is used to clean the chamber following deposition. Chlorine can 'stick' to the stainless steel and convert to hydrochloric acid (HCl) when exposed to moisture during maintenance.

HCl reacts with the stainless steel creating corrosion byproducts including metal particles and ions that contaminate the chamber and epi wafers. Corrosion and its effects currently result in maintenance cycles which can take a tool offline for several hours while the chamber is cleaned and, in severe cases, re-machined. The value SilcoTek coatings like Silcolloy® and Dursan® provide include:

- Reduce number of maintenance cycles per year
- Improve time per maintenance cycle
- Reduce cost per maintenance cycle
- Reduce time from maintenance mode to service mode

In addition, Silcolloy and Dursan coatings improve manufacturing processes by enhancing test quality and reliability and protecting product feedstock from contamination. Examples include:

- Ensure accurate readings in cleanroom VOC testing by preventing adsorption of test compounds by stainless steel flow paths.
- Improve vacuum pump down efficiency and prevent metal interaction with the wafer.
- Protect process gas delivery lines from corrosion and contamination. This will improve product yield and process control.
- Reduce barrier contamination and burn-in while increasing lifespan in Etch processes. Silcolloy and Dursan do not require burn-in or seasoning to reach optimal performance.
- Enhance purity and reduce carryover and corrosion in ALD processing. Silcolloy and Dursan prevent interaction of process materials with stainless steel.
- Eliminate particulate generation and improve stainless steel corrosion resistance in EPI applications. Silcolloy and Dursan improve corrosion resistance of stainless steel by orders of magnitude.
- Improve yield and efficiency in OLED operations by preventing corrosion and contamination.
- Stabilize the flow path in Ozone processes and improve system reliability.
- Increase the uptime of exhaust scrubber and burn-box systems by improving the corrosion resistance of duct and other system components.

2.3 Component Applications

Treating gas delivery and precursor system components with amorphous silicon coatings results in a robust manufacturing platform that ultimately improves yield and reduces operational maintenance cost. The CVD process is scalable from small fittings and very high surface area parts (metal frits, filters, etc.) to very large, fabricated parts (vacuum chambers, etc.). The focus of this e-book is on stainless steel parts, but the industrial CVD process is compatible with coating any material able to withstand the thermal requirements (stable to about 500 °C). This will include aluminum and its alloys, titanium, super alloys (Hastelloy®, Inconel®, etc.), glass, and ceramics. Examples of components that should be coated to assure performance include:

- Gas delivery tubing and manifolds

- Weldments
- Exhaust bellows
- Gas abatement ducts
- Gas regulators and mass flow controllers
- Valves and fittings
- Tanks and canisters including:
    - Liquid precursor canister
    - Liquid source canister
    - Shuttle canister
    - Ultra-high purity stainless steel canisters

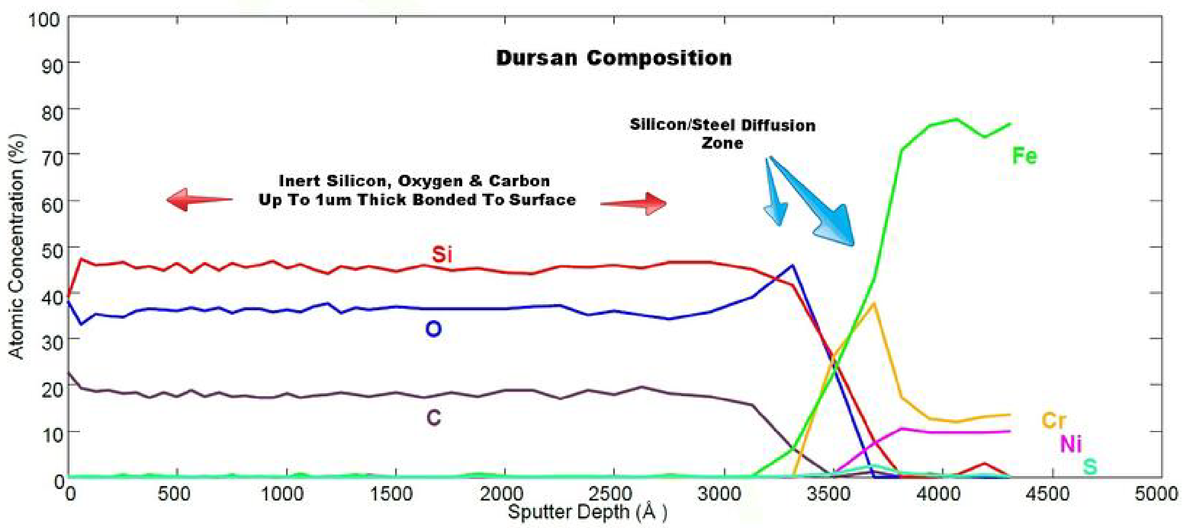
SilcoTek scientists and engineers are here to provide custom solutions to your material challenges. We offer custom manufacturing and coating options to meet your specifications. In chapter 3 we'll review and discuss some real-world tests to demonstrate the effectiveness of coatings like Dursan® and Silcolloy® in semiconductor and electronics manufacturing applications.

3.0 SilcoTek Coating Performance

The SilcoTek CVD coating process builds an inert barrier of silicon onto critical flow path surfaces. In this chapter we'll take a deep dive into coating performance testing to better define how coatings like Dursan and Silcolloy perform under extreme environments. After reading through the data, you'll gain an understanding of the performance of the coatings and how to better select the best coating for your application.

3.1 Coating Composition, Physical Properties and Bond

The thermal CVD deposition process creates a surface coating that is bound to the substrate, creating a small diffusion layer between the coating and the material surface. Auger depth profiling of a Dursan treated surface shows a slight diffusion zone after the 3500 angstrom coating thickness.

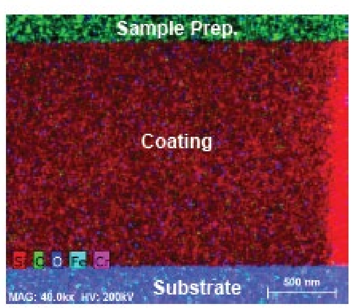


The coating composition in the depth profile indicates a silicon oxide with carbon in the coating matrix.

TEM/EDS analysis (below) shows the coating composition is uniform throughout the coating.

Uniform, Inert Amorphous Silicon Surface

TEM / EDS

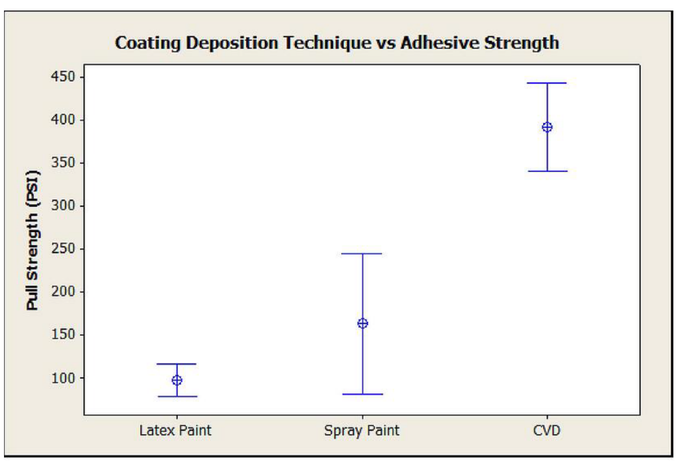


Additional purity testing by laser ablation ICP-MS shows the layer does not contain significant metal contamination.

Chemically Pure Coating Layer
Laser Ablation ICP-MS

Element	Coating Contents
Aluminum	<20 ppm
Chromium	0 ppm
Iron	<100 ppm
Nickel	<10 ppm
SilcoTek® Silicon CVD Coating	99.98% purity

CVD coatings have also shown improved adhesive strength over other deposition techniques[11]. Pull strength adhesion testing results (below) show silicon coatings applied by chemical vapor deposition perform significantly better than traditional coating application techniques.



process failures. The use of an amorphous coating with strong adhesion and the ability to flex with the part offers a significant benefit to overall workability in manufacturing and use.

SilcoTek coatings are ultra-thin; consequently, they improve surface properties without negatively affecting the mechanical properties or tolerances of the substrate materials or work pieces. This feature allows tool manufacturers to utilize the coating to derive benefits such as extending preventative maintenance (PM) intervals or increasing the mean time between failures without the need to redesign or reformat critical components. Table 1 compares some of the physical characteristics of common coating materials in etch applications.

Table 1: Physical Properties of common surface treatments used in semiconductor manufacturing

Property	**a-Si**[14]	**Yttria** [15,16]	**PTFE** [14]
Maximum Temperature	650° C*	1800° C	260° C
Low pH limit	0	2	0
High pH limit	7	13	14
Nominal Thickness	0.5 µm	0.5 µm	25 µm
Adhesion/Flexibility [12]	Very good	Poor	Poor

*Onset of crystallization. Go to our Silcolloy Data Sheet for more information.

The comparison data shows that surface treatments like PTFE or yttria-based coatings can be prone to physical damage that may lead to costly failures from particulate contamination. Substitution with more expensive superalloy materials, other steel alloys, or pure metals may be an option, but those materials can still succumb to aggressive etch chemistry corrosion and present additional fabrication challenges. To further demonstrate how our silicon coatings perform, we tested and compared the coatings to learn how Silcolloy, Dursan, and other SilcoTek coatings perform under extreme conditions.

3.2 Metal Contamination Testing

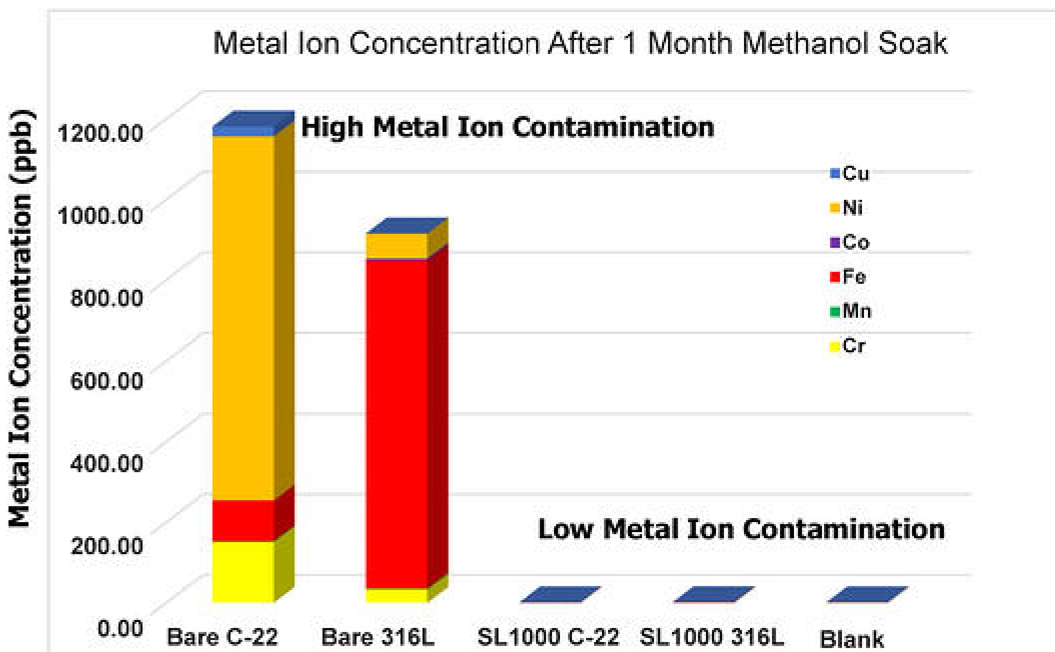
Silcolloy and Dursan act as a barrier between the process stream and the mechanical equipment to minimize or eliminate corrosion, metal ion contamination, and outgassing. The coatings reduce or prevent the ability of process fluids (or vacuum conditions) to react with free iron, moisture, or other elements on the substrate. Let's review testing data to show how coatings can improve yield and product quality by preventing metal ion contamination.

Exposure to acids or common solvents like methanol or even deionized water can leach a significant amount of metal into the process flow stream. Silcolloy and Dursan nearly eliminate ion contamination in process streams. Comparative immersion testing (described below) in methanol shows significant leaching by exposed Hastelloy and stainless steel.

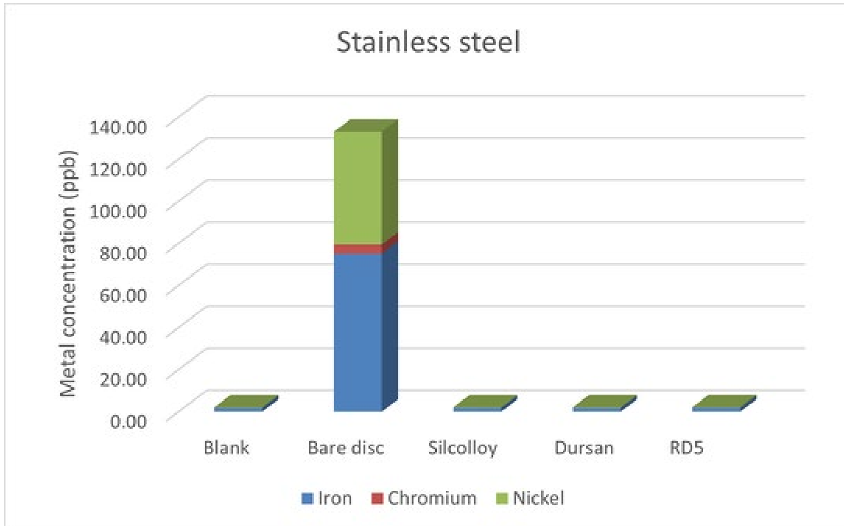
Porous C-22 Hastelloy and 316L stainless steel discs with 10 µm nominal pore sizes were purchased from Mott Corporation. They were coated with Silcolloy silicon barrier coating. After coating, the discs were placed in 50 mL of HPLC grade methanol and sealed. Uncoated discs as well as a container with no disc (a solvent blank test control to establish baseline response) were also filled with methanol and sealed. After one month of soak time, the porous disks were removed from the methanol and the samples were delivered to the Energy and Environmental Sustainability Laboratories at Penn State University. Samples were then prepared by evaporating 10 mL of the methanol in a PTFA vial. The remains were dissolved in 5 mL of dilute nitric acid which was then analyzed using a Thermo Fisher iCap RQ ICP-MS.

Figure 1 shows the total metal ion concentrations from the solution of each container. The blank represents the level of metal ions in the methanol bottle, leached from the sealed container, sample preparation, or absorbed via environmental sources. The bare Hastelloy and stainless steel discs represent the level of metal ions leached into the methanol without any coating protection provided by processing at SilcoTek Corporation. The level of metal ion contamination for the uncoated discs was significant, with the Hastelloy discs exceeding 1200 ppb contamination.

The coated discs showed little to no appreciable level of contamination.



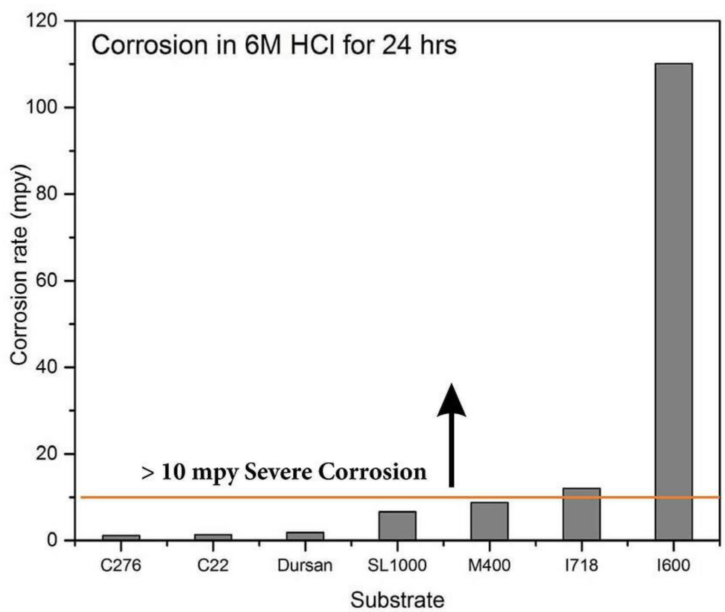
Additionally, exposure to relatively less aggressive solutions like deionized water can leach a significant amount of metals from stainless steel.



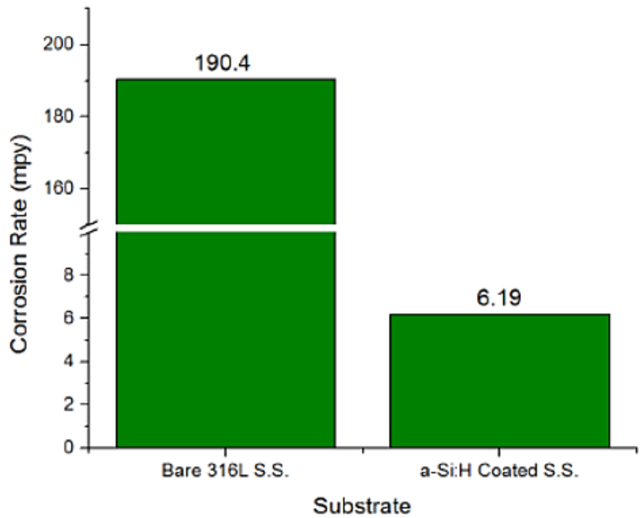
3.3 Corrosion Control

Selection of coatings and surface treatments in semiconductor manufacturing applications is typically derived from a function of performance and process compatibility. High performance alloys such as Hastelloy® or Inconel® may provide superior corrosion protection in some environments, but they are often prohibitively expensive and difficult to machine. SilcoTek coatings reduce replacement costs and offer sustainability and cost-effective product improvement and refurbishment pathways that otherwise don't exist in fab environments.

Stainless steel corrosion can also be minimized by coating the stainless substrate with Dursan, or Silcolloy. The graph below compares various alloys, including Hastelloy, with Dursan and Silcolloy coated stainless steel. After exposure to concentrated hydrochloric acid for 24 hours, the coated stainless steel coupons performed comparably to Hastelloy and exceeded performance of other alloys.

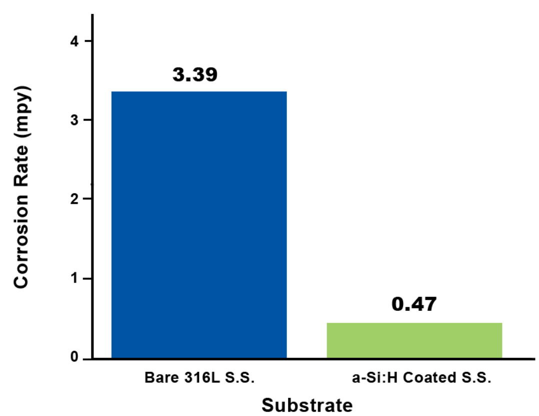


Further, 24 hour ASTM G31 HCl immersion testing shows Silcolloy (a-Si:H) coated 316L coupons creating a 30x improvement in corrosion resistance compared to uncoated 316L stainless steel.



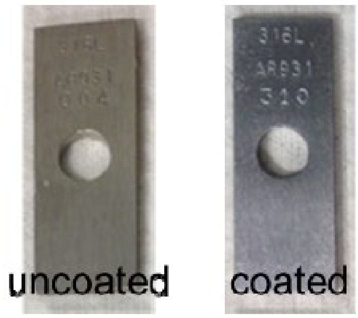
Comparison of a-Si:H coated stainless steel vs. uncoated stainless steel show a 30x improvement in hydrochloric acid corrosion resistance.

Additionally, HBr corrosion resistance is improved by 7x or more when coating 316L stainless steel with Silcolloy (a0=-Si:H). Samples were measured after 72 hours of exposure to 6M hydrobromic acid (HBr gas dissolved in water) at room temperature per ASTM G31 methodology.



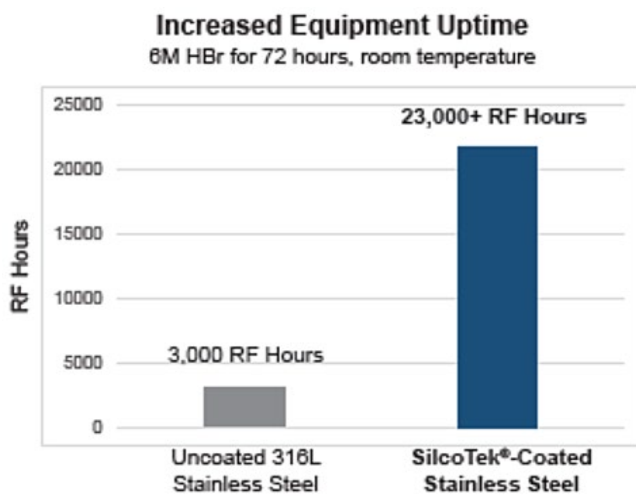
ASTM G31 72 hour immersion testing shows a 7x improvement in HBr corrosion resistance.

A visual inspection of the test coupons(below) reveals slight pitting and discoloration of the uncoated coupon while the Silcolloy coated coupon shows no effect.



316L stainless steel sample coupons after 72 hour exposure to 6 M HBr at room temperature

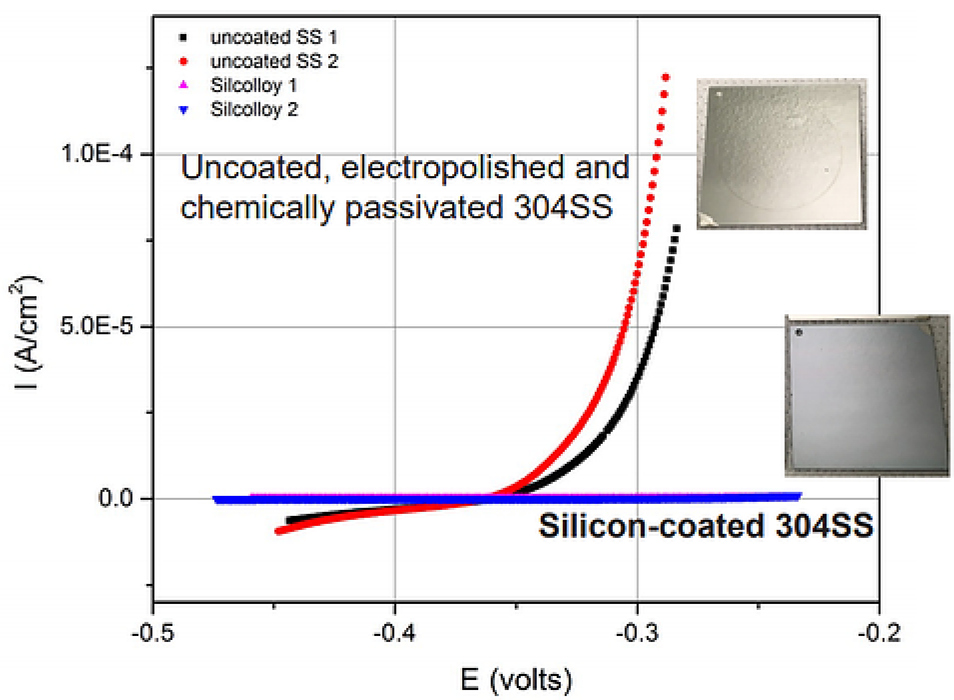
Calculating the benefit of the substanital improvement in corrosion resistance can be translated to better machine performance and efficiency. If we assume that the corrosion rate of 316L stainless steel in HBr solution approximates 3000 RF hours of uptime, the Silcolloy coated part would allow over 23,000 RF hour before failure. This may not be a perfect estimate but it is enough to suggest the Silcolloy system would stop metal ion leaching over a wide range of conditions.



Dursan® and Silcolloy® coatings also improve the corrosion protection from other acids including hydrofluoric, nitric, phosphoric, and sulfuric acid. Protection from a variety of acids can substantially improve the lifetime of exhaust and abatement systems used in semiconductor manufacturing operations.

	5% HF		70% Nitric		85% Phosphoric		25% Sulfuric	
	MPY rate	Factor	MPY rate	Factor	MPY rate	Factor	MPY rate	Factor
316L SS	120.00	-	0.78	-	0.62	-	91.8	-
Dursan	80.38	1.49	0.1	7.5	0.08	8.00	1.8	51
Silcolloy	44.26	2.71	0.36	2.14	0.28	2.18	15.7	5.85

Polarization scan testing (below) shows the silicon coated 304 stainless steel coupon resists pin-holing and the potential for metal ion leaching, assuring processes and analytical test streams remain free of ion contamination. The silicon-coated stainless steel coupon (blue line) shows no change in electrochemical current throughout the voltage range, while the uncoated stainless steel coupon shows a significant increase in pitting potential.



3.4 VOC Analysis in Cleanrooms and Exhaust Analyzer Systems

Protecting cleanroom environments is also critical to product yield. Analysis of VOC contaminants in ambient air and at point source processing is critical to maintaining statistical process control and product quality. Additionally, keeping exhaust analyzer systems in peak operation is critical to regulatory compliance. Testing by German based researchers DWD (Deutscher Wetterdienst) below show that silicon coatings like SilcoNert® (called by our other tradename Sulfinert®) improve detection and prevent adsorption of VOCs in the test flow path, showing no adsorption or desorption. A coated flow path improves test repeatability and sensitivity while improving system corrosion resistance.

Summary results for tubing at 50°C and 100°C

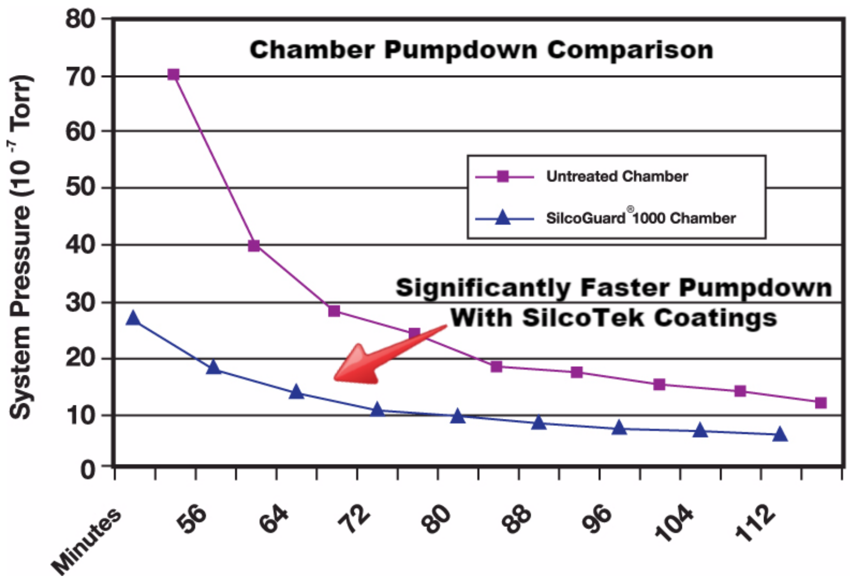
Deutscher Wetterdienst
Wetter und Klima aus einer Hand

Mole fraction of 3 µmol/mol, flow rate 15 ml/min

Tubings (1/8 inch OD)	50°C results	100°C results
Stainless steel 321 (untreated)	strong adsorption/desorption	no methanol detectable, destruction on the hot surface?
Electropolished stainless steel 316	strong adsorption/desorption	slightly better
Sulfinert® passivated stainless steel 304	no adsorption/desorption	no adsorption/desorption
Sulfinert® passivated stainless steel 316	no adsorption/desorption	no adsorption/desorption
PFA tubing	acceptable, but intensive flushing required	high outgassing of VOCs
PEEK tubing	some adsorption/desorption	some adsorption/desorption, slight outgassing of VOCs

3.5 Ultra High Vacuum and Moisture Management

SilcoTek offers low outgassing coatings with moisture repelling properties. These coatings improve moisture detection and reduce moisture contamination in vacuum systems as well as other systems sensitive to water contamination. Testing below shows a significant reduction in system outgassing under UHV conditions.

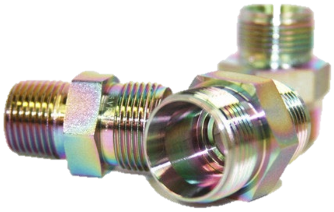


3.6 Conclusion

Coating critical process flow paths of semiconductor manufacturing equipment reduce failures related to corrosion and contamination which can lead to higher uptime and higher yields. As the industry continues scaling down to below 10 nm and smaller nodes, inert, high-purity surfaces and pathways become an essential element of the production tools. Thermal chemical vapor-deposited amorphous, hydrogenated silicon coatings provide a versatile material solution that inhibits corrosion and provides a higher degree of mechanical flexibility than alternative surface treatments while allowing equipment manufacturers to use preferred and affordable materials of construction.

In chapter 4 we'll summarize the benefits of using SilcoTek coatings in semiconductor manufacturing applications. We'll also provide additional reference material sources that provide a more detailed review of specific applications.

4.0 Continued Reading and Conclusion



Silicon coatings like Dursan® and Silcolloy® are proven to improve corrosion resistance, extend component life, and reduce contamination in semiconductor manufacturing systems . The data presented in this e-book is just a fraction of the coating performance information available to manufacturers. Go to our Learning Center to read studies, whitepapers, presentations, and technical insights about our coatings and learn more about how our coatings perform in your application.

- For more information about how our coatings perform in semiconductor manufacturing applications, go to our Semiconductor Applications Page.
- For more information about metal contamination, go to our Metal Contamination Prevention Page.
- To learn more about our corrosion resistant coatings, go to our Corrosion Resistant Coating Properties Page.
- You can learn more about how to send parts to our coating facility in Bellefonte, PA, USA by going to our Ordering Options web page.

We've also teamed up with leading manufacturers to offer SilcoTek coated products directly from the manufacturer, so you don't have to take the time to send parts to our facility for coating service. You can learn more about how to buy coated products from the manufacturer by going to our Buy Coated Products web page. There you'll get a listing of approved partners who offer coated products. Just click on the partner's name to learn more about how to purchase coated products from that supplier. SilcoTek partners with component manufacturers, international resellers, and fab-facing integrators to streamline the supply chain for getting coated products into your semiconductor manufacturing process. You can find a SilcoTek Sales Representative for your location by going to our International Sales Representative Web Page.

Thanks for taking the time to read our e-book and learn about how our coatings improve the lifetime and performance of semiconductor manufacturing components and systems. If you'd like to read more of our insightful e-books you can go to our Coating E-book web page. There you can access informative e-books for applications ranging from analytical testing, process sampling, heat exchanger, filtration, and general coating properties.   As applications grow and new coatings are developed, we'll be adding to our e-book library, technical insights and other information in our coating resource library so check back to our website frequently!

Finally, the SilcoTek Team would like to thank all our dedicated customers for making the advancement of coating material science possible.

End

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