SilcoTek® Coating Solutions

Coatings that Expand Material Limits™

SilcoTek's patented coatings solve the most demanding material challenges found in energy, science, and technology industries.

Features

- Chemically inert
- Corrosion resistant
- Flexible, won't flake
- Thin (<1000 nm)
- Hydrophobic
- Easy to clean
- High temperature

Advantages

- 3D, non-line-of-sight process
- Significant cost savings over exotic alloys or materials
- Enables trace analysis required for regulatory compliance
- Easy to integrate from prototype to production

Benefits

- Improve system efficiency and uptime
- Decrease maintenance
- Increase productivity
- Save money

SilcoTek®

Innovative surface coatings that make the impossible possible

Bring robust corrosion resistance and chemical inertness to the molecular level of stainless steel, glass, ceramic, and other substrates.

FOR CHEMICAL ANALYSIS

SilcoTek's silicon-based coatings are specially tailored for inertness (non-reactivity) to highly active chemical compounds. Required for analyzing trace levels of sulfurs, mercury, emissions, pesticides, etc.

SilcoNert® is the industry-preferred coating for highly sensitive sampling and analysis applications. Dursan® is a tough and versatile coating suited for harsh analytical environments. Modern chemical detectors and analyzers manufactured worldwide rely on these inert coatings to give accurate results.

FOR CORROSION PROTECTION

Protect critical investments in a wide array of corrosive environments. SilcoTek's dense, pinhole-free coatings provide a uniform, molecularly-bonded barrier between substrate and flow path.

Dursan includes oxygen and carbon in the base silicon layer for a ceramic-like, durable, and highly corrosion resistant coating. Suited for pH 0-14 and harsh corrosives like HCl, H_2SO_4, bleach, and more. Silcolloy® adds significant potential lifetime to parts in oxidative environments. Bring exotic alloy performance to stainless steel for a fraction of the cost.

FOR HIGH PURITY

Prevent leaching of metal ions from stainless steel equipment into critical process streams while increasing system uptime. Imperitive in sensitive manufacturing environments e.g. semiconductor.

Silcolloy offers oxidation, chemical, and corrosion protection even at temperatures as high as 1000° C. Dursox™ is a silica-like coating with exceptionally low surface energy and high durability. SilcoGuard® greatly reduces outgassing in high vacuum applications. All three coatings provide better equipment lifetimes and higher product yields to companies with strict purity requirements.

CHOOSING THE RIGHT COATING

Customers should work with SilcoTek's technical experts to help them select the best coating for their application. Some applications require a very specific treatment whereas any SilcoTek coating could work for others. SilcoTek's complete line of coating solutions offers a multitude of surface properties in addition to what's highlighted above:

- Low surface energy
- Anti-coking/anti-fouling
- Hydrophobicity
- Abrasion resistance
- Easy cleaning/anti-stick
- Low outgassing

The recommendation process often involves samples, testing at both customer and SilcoTek sites, technical consultation, visits, and more. The SilcoTek service experience couples technical expertise with coating capability and performance to give customers a solution they (and their customers) can rely on.

Coating Properties

SilcoTek's innovative chemical vapor deposition (CVD) process introduces proprietary process gases into a special oven containing your parts. The gas penetrates torturous passageways and provides a thin, uniform coating even on complex part geometries.

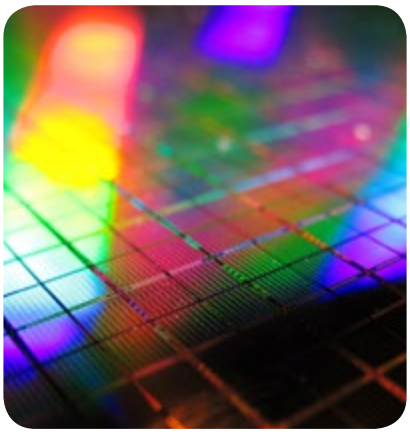
Each standard SilcoTek® coating is tailored to specific applications but can be used successfully in a wide variety of environments. Contact SilcoTek for coating recommendations.

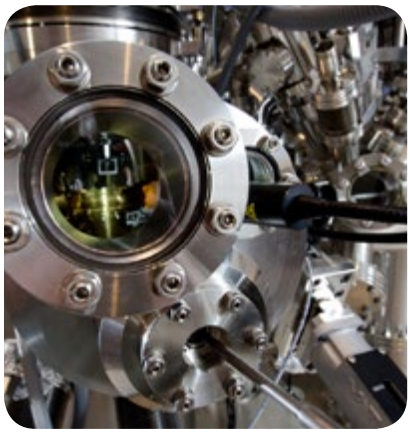
COATING	MATERIAL COMPOSITION	MAXIMUM TEMPERATURE	CONTACT ANGLE*	WHAT IT DOES
SilcoNert® Superior inertness	Silicon (functionalized)	450° C	99°	Makes surfaces non-reactive. A durable, high temperature alternative to fluoropolymers like PTFE or PFA.
Dursan® Corrosion and abrasion resistant, inert, low surface energy	Silicon, oxygen, carbon (functionalized)	450° C	119°	Provides low surface energy and excellent protection in very corrosive environments. Hydrophobic, 2x as wear resistant as stainless steel and easy to clean.
Silcolloy® Oxidation resistant, high temperature	Silicon	1000° C	54°	Protects parts from oxidation while preventing metal ions from leaching out of surfaces. Ideal for high temperature applications.
SilcoKlean® Anti-coking	Silicon (functionalized)	1000° C	90°	Prevents hot fuels and gases from coking or fouling on metal surfaces. Ideal for fuel transfer and exhaust gas applications.
SilcoGuard® UHV low outgassing, high purity	Silicon	1000° C	54°	Isolates materials trapped on or in metal surfaces and prevents them from entering ultra-high vacuum or other high purity environments.
Dursox™ Silica-like, ceramic	Silicon, oxygen** (functionalized) **<2% embedded carbon	450° C	<60°	Gives durabilty, moisture resistance, erosion and corrosion protection to processing equipment. Ideal especially for semiconductor manufacturing equipment.

*Evaluated on 120 grit, 58 rms (µin.) 300-series stainless steel

A Note on Thickness

SilcoTek's chemical vapor deposition (CVD) process has been optimized to produce surface coatings that meet the performance characteristics and material properties listed above, unrelated to thickness. All coatings are typically less than 2000 nm (2µm) thick.





Industries & Applications

Petrochemical

- Process analyzers
- CEMS
- Ethylene and propylene
- Refinery, flare, and stack gas
- ULSD/ULSG
- LNG and CNG
- Environmental sampling

Oil and Gas Exploration

- Well sampling
- Downhole tools
- Offshore instrumentation
- Odorant testing
- Wireline
- Power generation and distribution

Semiconductor Manufacturing

- Etch and deposition
- Epitaxy
- MOCVD and PECVD
- CMP
- OLED
- Ozone
- Moisture analysis

Aerospace and Automotive

- Fuel and injector nozzles
- Feed lines
- Fuel injectors
- Exhaust testing
- Exhaust gas recirculation equipment

Analytical

- Chromatography
- Needles and probes
- Vials
- Sample loops
- Ultra high vacuum
- Flow control
- Food and beverage analysis

Coatings that Expand Material Limits

Whether in the laboratory, plant, or field, SilcoTek's patented coating technologies provide advanced material solutions that save you time, increase your productivity and improve performance, all while lowering operating costs and protecting your critical investments.

SilcoNert® **Dursan®**

Silcolloy® **Dursox™**

SilcoKlean® **SilcoGuard®**

For more information, visit www.SilcoTek.com

Coating Use

All statements, technical information and recommendations contained in this document are based upon tests or experience that SilcoTek believes are reliable. However, many factors beyond SilcoTek's control can affect the use and performance of a SilcoTek coating in a particular application, including the conditions under which the product is used and the time and environmental conditions in which the product is expected to perform. Since these factors are uniquely within the user's knowledge and control, it is essential that the user evaluate the SilcoTek coating to determine whether it is fit for a particular purpose and suitable for the user's method of application.

Limited Liability

Except where prohibited by law, SilcoTek will not be liable for any loss or damage arising from the SilcoTek coating whether direct, indirect, special, incidental or consequential, regardless of the legal theory asserted, including warranty, contract, negligence, or strict liability.

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Information subject to change without notice.

2016-ST-001

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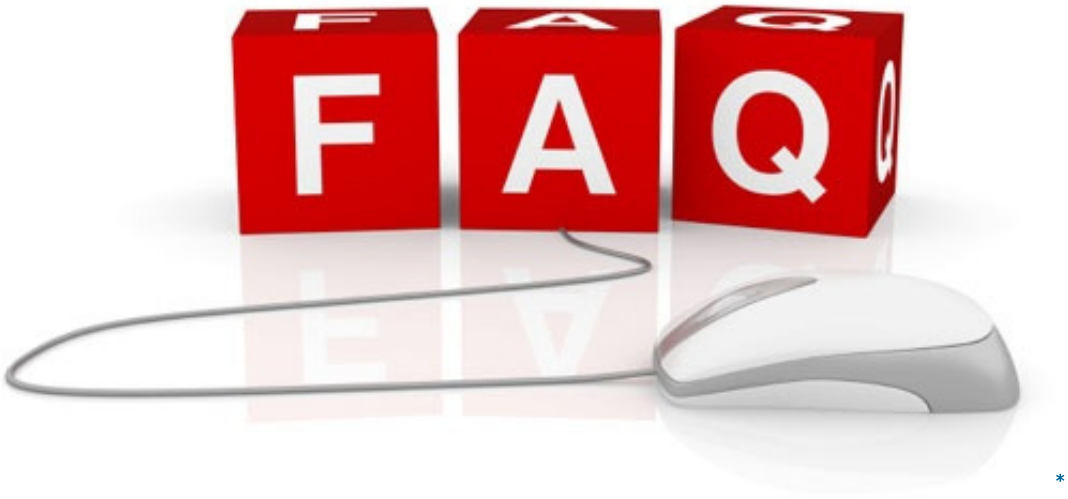
SilcoTek®
Driving Innovation



Frequently Asked Questions

- Can SilcoTek® coat fritted liners?
- Do I need to pre-clean my parts prior to sending them to SilcoTek® for coating?
- Will brazing impact the quality of the coating?
- Where can I order parts already coated by SilcoTek?
- Can SilcoTek® coat assemblies like valves, regulators, and flow controllers?
- How tightly can the tubing be bent?
- Will you re-deactivate my SilcoNert® 2000 treated glass liners or SilcoNert® 1000 metal liners after I clean them?

- **Why do Silco'd treated pieces have different colors?**
- **What is the maximum temperature limit for SilcoNert 2000 deactivated glass?**
- **How thick is the SilcoNert 2000 layer?**
- **Why is SilcoNert® 2000 on glass gold in color?**
- **What are the unique benefits of SilcoNert 2000 deactivation?**
- **What is SilcoNert 2000?**
- **How can I receive pricing for custom surface treatment?**
- **What are the operating temperatures for SilcoTek treated surfaces?**
- **Can sealing surfaces be coated?**
- **Can ferrules be treated?**
- **What materials can be surface treated?**
- **How do I select the most appropriate treatment for my application?**
- **Are SilcoTek's coatings RoHS and REACH compliant? What about their MSDS sheets?**
- **Do SilcoTek coatings change the roughness of the surface?**

How do I select the most appropriate treatment for my application?
The following definitions explain the various treatments currently available and their recommended applications:

- **SilcoNert® 1000** (Silcosteel®)
  A general-purpose passivation layer for steel and stainless steel.
- **SilcoNert® 2000** (Siltek®/Sulfinert®)
  The ultimate passivation of treated surfaces, from glass to high nickel alloys of steel. A required treatment for metal components when analyzing for parts-per-billion levels of organo-sulfur compounds & mercury.
- **Silcolloy® 1000** (Silcosteel®-CR)
  A corrosion resistant layer that increases the lifetime of system components in acidic environments containing hydrochloric acid, nitric acid, or seawater.
- **SilcoKlean®1000** (Silcosteel®-AC)
  Dramatically reduces carbon buildup on stainless steel components.
- **SilcoGuard® 1000** (Silcosteel®-UHV)
  Greatly reduces outgassing from components of ultra-high vacuum systems.
- **Dursan®** Our newest coating! A high durability, wear resistant, corrosion resistant, and inert

coating. Great for field sampling, process, Oil & Gas appications.

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What materials can be surface treated?
Virtually all alloys of stainless steel, ceramic, and most glass materials can be treated. Some other metallic materials, such as nickel, brass, copper, and aluminum, are incompatible with the high (400°C) temperatures used in the process, and should not be treated.

Note: carbon steel, F22 or similar carbon steel, is not recommended for coating in corrosion resistant applications. Treated carbon steel may rust in corrosive environments. Avoid corrosive environments when using treated carbon steel in inert sampling applications.

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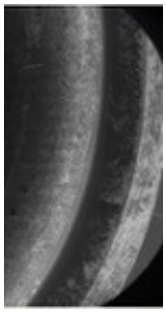
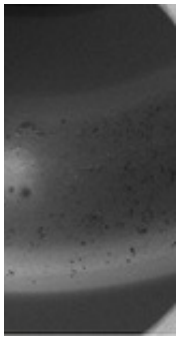
Can sealing surfaces like CF, face seal, threaded and compression fittings be coated?

Yes! SilcoTek coatings are proven to withstand compression and shear stress common to CF, face seal, and threaded sealing systems.



FESEM (Field Emission Scanning Electron Microscopy) image of the coated pre-sealed surface shows the coating conforms to the sealing area. The raised surfaces of the face seal gland area can be seen. SilcoTek coatings conform to surface contours and small orifices.



After making the metal-to-metal face seal per manufacturers instruction, a second FESEM image of the post sealing surface shows no scarring or scratching, only trace particulate from the silver gasket.

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Can ferrules be treated?
SilcoTek does not recommend coating ferrules as we have found this may lead to leaking problems after coating.

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What are the operating temperatures for SilcoTek treated surfaces?
Maximum temperatures usually will be dictated by the construction material (glass, stainless steel, etc.), not by the surface treatment. In general, temperatures should not exceed those listed in Table I. Temperatures greater than 450°C for SilcoNert®2000 (formerly Sulfinert® or Siltek®) and Dursan® treated surfaces or greater than 600°C for SilcoNert®1000 (formerly Silcosteel®), Silcolloy® (formerly Silcosteel®-CR), or SilcoGuard® (formerly Silcosteel®-UHV) treated surfaces can be used under certain conditions. Heat treatment of parts before applying a Silcolloy, SilcoNert1000, SilcoGuard, coating extends the maximum temperature limit. Please contact technical support at 814-353-1778, for additional information.

SilcoTek® coatings are stable to the following temperatures in an inert atmosphere:

SilcoNert® 2000	450°C
SilcoNert® 1000	600°C
Silcolloy®	600°C
SilcoGuard®	600°C
SilcoKlean®	600°C
Dursan®	450°C

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How can I receive pricing for custom surface treatment?
Click here to request a quotation for custom surface deactivation of sample pathway components, inlet liners, etc. Please forward a scaled drawing or photograph detailing the product to be treated. Upon receipt of this completed worksheet, SilcoTek's Technical Staff will contact you with a quotation, typically within 1-2 business days. When you are ready to send items to SilcoTek for treatment, contact technical support at 814-353-1778 to request service number. When submitting parts for treatment, remember that parts must be completely dissembled, and components that cannot withstand the high (400°C) temperatures associated with the process (rubber o-rings, brass, etc.) must be removed. Note that SilcoTek cannot assume liability for warping of stainless regulators due to process heat.

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How will SilcoTek surface treatments work in my acidic environment?
SilcoNert 1000 and SilcoNert 2000 treated materials have improved corrosion resistance, but

Silcolloy (formerly Silcosteel®-CR) and Dursan surface treatments are optimized to enhance resistance to many common acids; providing a 10-fold improvement in corrosion resistance, compared to untreated stainless steel, and a 4-fold improvement compared to SilcoNert 1000 coated stainless steel.

Note: carbon steel, F22 or similar carbon steel, is not recommended for coating in corrosion resistant applications. Treated carbon steel may rust in corrosive environments. Avoid corrosive environments when using treated carbon steel in inert sampling applications.

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What is SilcoNert 2000?
SilcoNert2000 (U.S. patent 6,444,326) is a passivation process that applies an inert, integral layer to components used for chromatographic analysis. Unlike traditional deactivations, it is not susceptible to cleavage or formation of active silanols and, therefore, greatly reduces bleed, and breakdown or adsorption of active components, compared to conventional surface coatings.

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What are the unique benefits of SilcoNert 2000 deactivation?
SilcoNert2000 creates a unique surface with an inertness range that surpasses all other known surface deactivations used in gas chromatography. In sensitive analyses, SilcoNert2000 deactivated system components provide outstanding results.

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Does anyone else offer an equivalent to SilcoNert 2000 deactivation?
No. SilcoNert 2000 deactivation was created exclusively by SilcoTek and is protected by a US patent (Pat. No. 6,444,326).

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Why is SilcoNert 2000 on glass gold in color?
The color of a surface coating is an expression of the light diffraction qualities and thickness of the layer. The thickness of a SilcoNert 2000 layer determines the degree of darkness, secondary reflectivity, and/or final color of the item. The color can range from light golden-brown (thin coatings) to reflective silver (heavier coatings). We deposit a layer that provides a gold color on liners to aid analysts in inspecting for cleanliness. Chromatographic performance does not depend on the thickness of the layer.

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How thick is the SilcoNert 2000 layer?
Depending on the item, the SilcoNert 2000 layer can be as much as 2,500 Angstroms (250 nanometers) thick. At this thickness, the layer exhibits a reflective silver color on treated glass surfaces or a rainbow on metal surfaces. Chromatographic performance does not depend on the thickness of the layer.

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What is the maximum temperature limit for SilcoNert 2000 deactivated glass?
Maximum temperatures usually will be dictated by the composition of the glass, not by the surface treatment. The SilcoNert 2000 layer is stable at temperatures up to 450°C, but some glasses can soften at lower temperatures. Injection port temperatures normally are well below the SilcoNert 2000 maximum temperature limit, so SilcoNert 2000 treatment is an excellent deactivation for all injection port surfaces.

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Does SilcoTek offer custom SilcoNert treatment?
We are always interested in your specific surface passivation needs. Surface deactivation of glass or metal with a SilcoNert 2000 layer is available on a custom basis, and liner deactivation has become popular with some customers. Please keep in mind that treated surfaces must be able to withstand temperatures up to 400°C, as well as vacuum and pressure environments. It is important to remove any o-rings, seals, or other materials that will not withstand the treatment environment prior to sending items to SilcoTek for coating.
Our SilcoTek Group can provide a quote. Please submit a quote or contact us at 814-353-1778.

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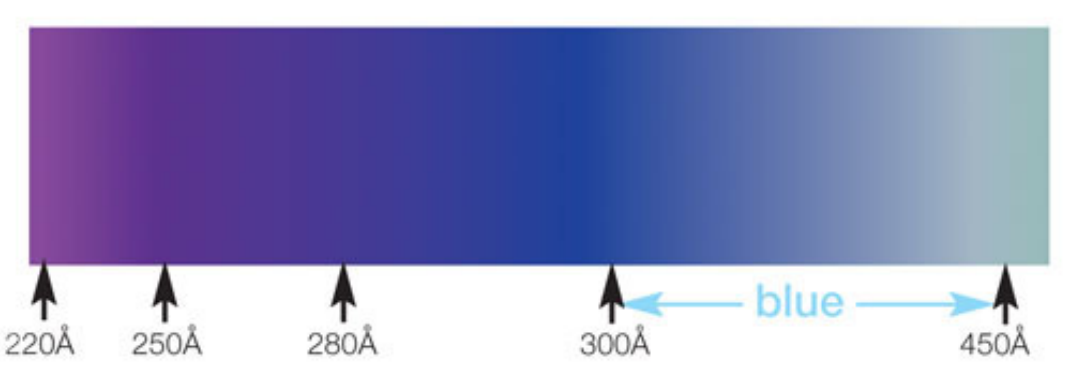
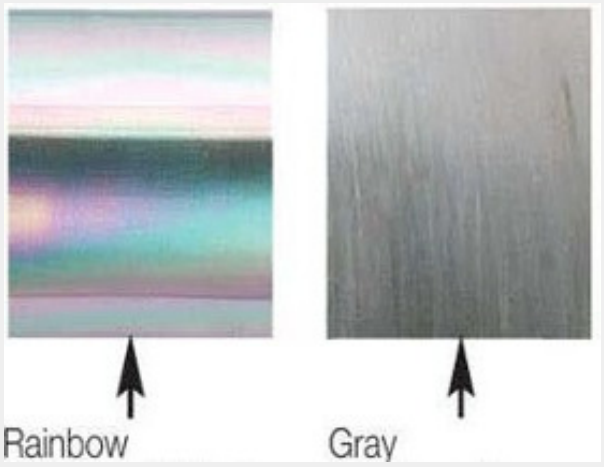
Why do Silco'd™ treated pieces have different colors?
The different colors observed on Silco'd treated parts indicate different layer thicknesses. A blue color corresponds to a 300 to 450 Angstrom layer while a rainbow color indicates a coating of at least 1200 Angstroms (120 nanometers).
Colors associated with layer thickness are:



Depositions used in our Silcolloy and SilcoGuard processes are up to 1µm and have a silver/metallic gray appearance. The photos below show colors created by SilcoNert 1000 (left) and Silcolloy 1000 (right).



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Will you re-deactivate my SilcoNert 2000 treated glass liners or SilcoNert 1000 metal liners after I clean them?
Request re-deactivation of SilcoNert 2000 treated glass liners by contacting SilcoTek's Customer Service department at 814-353-1778. Requesting SilcoNert 2000 re-deactivation of customer supplied liners. A minimum of ten liners is required. Metal inlet liners are designed as inexpensive, disposable products and it is not cost effective to re-treat them. We recommend discarding them when they are no longer serviceable.

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How tightly can the tubing be bent?

OD
<= 1/16"
1/8"
1/4"
3/8"

Min. Bend Radius
1" (2.5 cm)
2" (5.1 cm)
4" (10.2 cm)
6" 15.2 cm)

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Can SilcoTek coat assemblies like valves, regulators, and flow controllers?
Absolutely! SilcoTek can coat the internal and external metal pathways for most flow regulating and metering devices to enhance their inertness and provide corrosion resistance. In order to coat the surfaces, all valves, regulators, and flow meters should be dissembled and all seals or parts which cannot be exposed to temperatures of approximately 400°C must be removed. Disassembly should only be performed by qualified personnel with the appropriate seal kits, tools, and training OR damage or failure to the device will occur. For these reasons, SilcoTek cannot take on the responsibility of disassembly or reassembly of any valve, regulator, or flow meter. If you do not have the experience

and parts necessary to dissemble these devices, we recommend that you get in touch with Swagelok, Parker, or your valve manufacturer and order the coating treatment directly from them. SilcoTek has business relationships with most manufacturers and routinely coats the internal and external surfaces of valves, regulators, and flow meters for them. The manufacturer will send the parts prior to assembling to SilcoTek for coating and will assemble the parts at their factory under the guidance of their quality systems.

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Where can I order products that are already coated by SilcoTek?

SilcoTek does not sell anything besides our coating services, but our Channel Partners work with us to stock their products with our coatings to help you supply chain. **Click here to see product listings.**

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Will brazing impact the quality of the coating?
Yes, brazed joints will outgas during the coating process and will negatively impact the coating quality. SilcoTek does not recommend coating over brazed joints. Coated brazed joints appear as a matte brown finish and can have increased activity/reactivity at the brazed joint. SilcoTek recommends vacuum brazing or welding of joints for best coating quality. Vacuum brazing is a high temperature, flux-free brazing process that results in a contamination free joint. SilcoTek recommends vacuum brazing services from Solar Atmospheres Inc.

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Do I need to pre-clean my parts prior to sending them to SilcoTek for coating?
Your parts are precision cleaned by SilcoTek prior to processing. Additional pre-cleaning by the customer is often unnecessary and at times can be detrimental to our coating process.

In order to provide the highest quality coating and fastest processing; notify SilcoTek of any prior chemical exposure (regardless of whether the part is new or used). Chemicals like cutting oils, pickling acids, solvents, water based cleaning compounds or other hazardous or non hazardous chemicals may impact the coating process. Many times the customer's cleaning process or chemical exposure can be eliminated, saving the customer time and money. Before sending parts to SilcoTek, contact your SilcoTek representative at **silcod@SilcoTek.com** or call us at (814) 353-1778

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Can SilcoTek coat fritted liners?
SPECIAL NOTE: Coating of glass fritted liners: SilcoTek would prefer that all glass liners be sent without deactivation (e.g. silanization) since the deactivation layer will prevent our intermolecular coatings from bonding to the surface. For most glass liners, our cleaning process will remove all prior deactivants. However, SilcoTek cannot put fritted liners through our cleaning process without damaging the frit.

Fritted liners are special because there is no effective way of cleaning glass frits or stripping the coating in case of failure. As we continue to gain experience with glass frits and tailor our processes, we will get better. Our major hurdle is being able to "test" fritted liners prior to coating to ensure they

are clean. We continue to work on this aspect of our operation.

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Are SilcoTek's coatings RoHS and REACH compliant? What about their SDS sheets?

Visit http://www.silcotek.com/compliance-statements to learn more about how our coatings are non-hazardous and in compliance with the law. The treatments do not require SDS sheets.

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Do SilcoTek coatings change the roughness of the surface?

SilcoTek coatings conform to the microstructure of the part surface. There is minimal filling of voids or change to the overall surface roughness. Overall roughness measurement may be reduced slightly (1-2 RA reduction), but not a significant change in roughness. Some SilcoTek coatings have a higher lubricity than stainless steel, so even though surface roughness has not changed, the surface may have a lower friction coefficient.

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*FAQ image courtesy of St. Odilia School, Shoreview, MN

- Resources
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  - Cleaning & Handling

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Protect Your Steel - Dursan® Protective Coating from SilcoTek®

SilcoTek Corporation

Discover the durability, corrosion resistance, moisture resistance and inertness of Dursan. Dursan is a chemical vapor deposition ...

SilcoTek Corporation

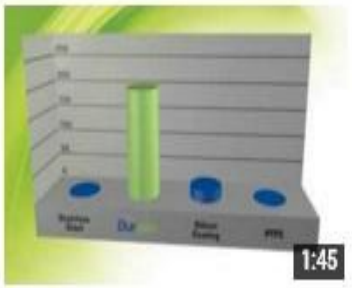
SilcoTek Corporation

- innovators of protection against corrosion, air, water, chemicals, and life.

Bend it, Smash it, Ball it up - SilcoTek® Coatings Won't Flake like Paint

SilcoTek Corporation

No surface treatment matches the strength and durability of protective CVD coatings from SilcoTek®.



Protect your Equipment from Corrosion - Dursan® from SilcoTek®

SilcoTek Corporation

Want to protect your valuable equipment from corrosive attack? Watch how a stainless steel coupon treated with Dursan® from ...

Hydrophobic Coating Demonstration - Dursan® from SilcoTek®

SilcoTek Corporation

See the superior water resistance of SilcoTek®'s most hydrophobic CVD coating, Dursan®. Dursan® is an ultra-thin, corrosion ...

Most Inert Protection for Analytical GCs - SilcoNert® 2000 from SilcoTek®

SilcoTek Corporation

SilcoNert® 2000 is SilcoTek®'s most famous coating technology for a reason.

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SilcoTek Time Lapse

SilcoTek outgrew its present facility and was in need of a new office and manufacturing space. Founder Paul Silvis opted for a ...

Will it Corrode? - Protective Coating Solutions from SilcoTek®

SilcoTek Corporation

This test immerses two stainless steel ladles in hydrochloric acid (HCl). Will the Silco'd ladle from SilcoTek® corrode? Learn more ...

Webinar: How to Choose the Right SilcoTek Coating for Your Application

SilcoTek Corporation

SilcoTek offers a variety of high performance CVD coatings for many different applications, so how do you know which one is best ...

Webinar: SilcoTek Coatings for Semiconductor Applications, June 2015

SilcoTek Corporation

An interactive presentation summarizing the benefits and applications of SilcoTek's coating solutions for the semiconductor ...

Blowtorch Demo - Extreme Heat Resistance of SilcoTek® Coatings

SilcoTek Corporation

Watch a SilcoTek® coated exhaust header get blasted with a blowtorch - and not blink an eye. Learn more at www.SilcoTek.com.

The Flexibility of SilcoTek® Coating Solutions

SilcoTek Corporation

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The Toughest Protective Coating - Dursan® from SilcoTek®

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Abrasion Resistance Demonstration - Dursan® from SilcoTek®

SilcoTek Corporation

Abrasion can be an expensive problem that leads to poor performance in various industries like manufacturing, process, ...

SilcoTek Solutions

by SilcoTek Corporation

Hydrophobic Coating Demonstration - Dursan® from SilcoTek®	1:14
Freeze Test - Temp Durable Coating Dursan® from SilcoTek®	1:16

Freeze Test - Temp Durable Coating Dursan® from SilcoTek®

SilcoTek Corporation

This video demonstrates the coating Dursan®'s strength against extremely low temperatures sometimes found in process ...

SilcoNert® Hates Water - Moisture Resistance of SilcoTek® Coatings

SilcoTek Corporation

See how SilcoNert® from SilcoTek® totally resists water that it is submerged in. This is key for process industries. Learn more at ...

Semiconductor UHV Solutions - SilcoGaurd® from SilcoTek®

SilcoTek Corporation

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Australian Distributors
Importers & Manufacurers
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SilcoTek - US Concrete Building Erection Time Lapse

CoolPixxMedia1

SilcoTek outgrew its present facility and was in need of a new office and manufacturing space. The company opted for a precast ...

Dursan® and Silcolloy® Coatings - Salt Spray Corrosion Resistance

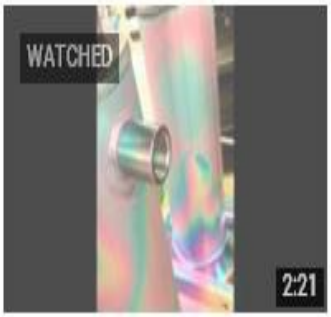
SilcoTek Corporation

The Best Reason to Choose Dursan® over PTFE - Heat Resistance

SilcoTek Corporation

2 years ago • 167 views

Watch PTFE melt away under extreme heat from a blowtorch while Dursan® from SilcoTek® continues to protect the stainless ...



My Ezvid Video

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Coating Electropolished Surfaces

Abstract

SilcoTek's coating processes will not remove or hide defects created during the electropolishing process. Many times, SilcoTek's thin (sub-micron or –half-micron) coatings will highlight small surface differences which exist on a metal surface. One difference which will often be highlighted is an effect referred to as electropolish 'frosting' (sometimes 'pitting'). SilcoTek's coating process will even bring out small features which were not visible to the naked eye.

Electropolish 'Frost'

Electropolish 'frosting' is an effect which is created during electropolishing where some regions are treated at a slightly different rate than others. This 'frost' (or conversely pitting) can be created if residual organics are left on the part surface (blocks electrical current), if the grains of the metal surface are very large, or even if the part is left in the electropolishing bath for too long[iii]. Electropolish 'frost' may be acceptable depending on the final application, but some industries, such as semiconductor and medical devices, have strict 'no frost' requirements.

SilcoTek's Experience

Electropolish 'frost' can exist even when it's not visible to the unaided, naked eye. Microscopic changes in surface height are often responsible for 'frosting' and pieces must be viewed under magnification or with the help of enhanced light sources like white LED. With no light source, often, no 'frost' is visible on the electropolished surface, but when a white LED is shone on the surface, Figure 1 clearly shows some visible differences. The effect can be even greater under magnification.

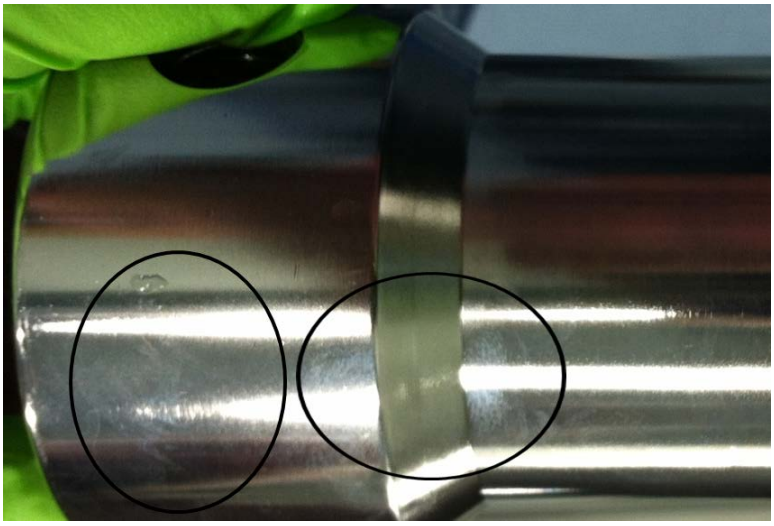


Figure 1 - Electropolish frosting with the aid of white, LED light

Electropolish 'frosting' is not always detected prior to SilcoTek's coating process. Because SilcoTek's pre-coating surface preparation does not affect the 'frost', the surface differences are only found after the CVD coating is applied. After coating, these parts display a distinctly different appearance shown below in Figure 2.

Figure 2 - Electropolish 'frosting' post coating without the aid of a white LED

It is important to note that the electropolish 'frosting' is not on or in SilcoTek's coating, but rather is an effect created by the metal surface finish. The 'frost' should not affect the coating performance within any application where electropolish 'frosting' is acceptable.

Recommendation

Part cosmetics are important for a wide range of end-user applications. SilcoTek recommends that electropolishing be specified to be free of 'frosting' for any parts coming into our coating processes where part cosmetics may be a concern.

[i] http://www.delstar.com/electropolishing.html
[ii] http://www.kepcoinc.com/downloads/Electro_Polishing/LC_cleaning-for-electropolishing.pdf

With the aged nickel tubing, ethyl thioacetate (19.6 min.) disappears completely. Ethyl sulfide (17.5 min.) shows a reduced response while methyl ethyl disulfide (21.2 min.) and diethyl disulfide (23.1 min.) appear. These new components may be the result of catalysis occurring in the presence of an active metal surface.

Conclusions

The Tekmar 3000 Sample Concentrator with new nickel tubing performs well in all of the evaluations performed in this experiment. As nickel ages, however, the possibility for adsorptive losses on the metal surface increases. This aging may result from the hydrochloric acid that many samples are preserved with. The Silcosteel coating shows greater corrosion resistance than electroform nickel. When the 3000 is compared with the Tekmar 3100 Sample Concentrator which possesses a complete Silcosteel sample pathway and improved temperature uniformity, the 3100 outperforms the 3000. Overall, the average response factors for a large number of the analytes in the 502.2 and 524.2 Rev. 4 compounds lists increase when they are evaluated with the Tekmar 3100 Sample Concentrator.

An additional study is planned to follow-up on this work to examine whether improvements can be made by varying the mount temperatures and other purge and trap parameters in order to optimize analyte responses.

The Do's and Don'ts in the Analysis of Sulfur for Polyolefin Producers

Presented as Paper 081 at the Gulf Coast Conference, Galveston Island, TX October 22, 2003

Benjamin Biela[1], Roger Moore[1], Robert Benesch[2], Bruce Talbert[2] & Tracey Jacksier[2]

[1]Equistar, Channelview Complex, 8280 Sheldon Rd., Channelview, TX
[2]Air Liquide, Chicago Research Center, 5230 S. East Ave., Countryside, IL

Agenda

- Introduction
- Ethylene and Propylene
  - ✓ Why is S so important?
  - ✓ What are the stakes?
- Sulfur Analysis
  - ✓ What are the issues?
  - ✓ Analytical Challenges
    - Standard preparation and shelf life
    - How to deliver S to the detector
    - Instrumentation
    - Sample handling
- Summary
- Conclusion

Introduction

- Benjamin Biela, Analytical Specialist
- Equistar Chemicals Analytical Services Laboratory
- Location: Channelview, Texas

Equistar Channelview Analytical Services Lab

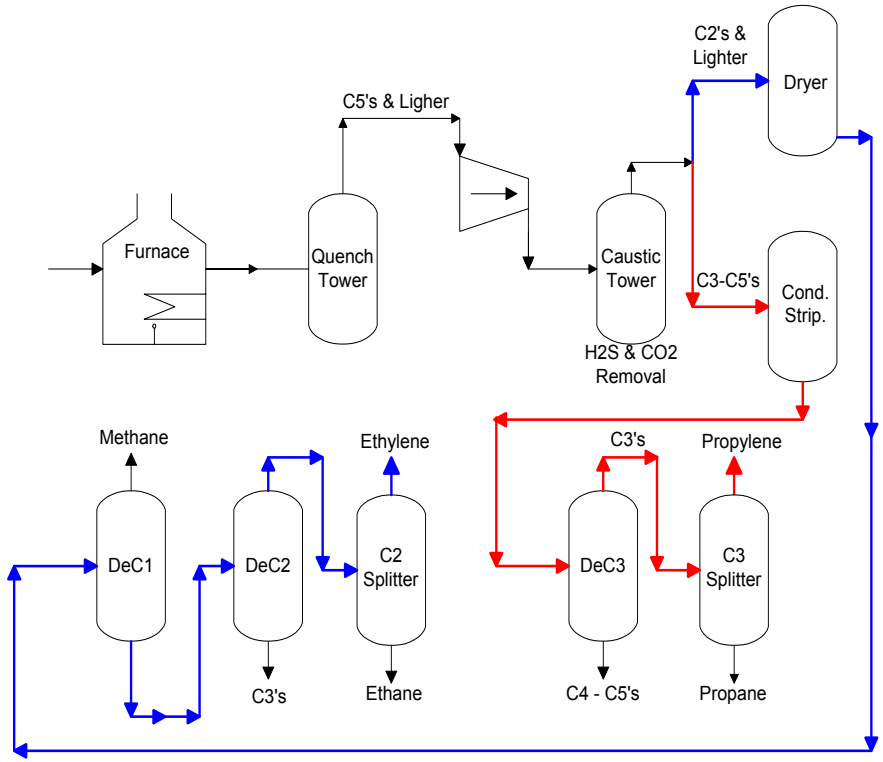
- Support 6 olefin plants producing the following products
  - ✓11.6 Billion lb/yr Ethylene
  - ✓5.0 Billion lb/yr Propylene
- Other products: Butadiene, Benzene, Toluene, Isoprene, Piperylene, Dicyclopentadiene, Alkylate, and Methanol

Sulfur Compounds - Polyolefin Catalyst Poisons

- ppb levels of sulfur compounds present in polyolefin feeds poison catalyst systems and drastically reduce polymer yield
- Reduced yields translate into lost profits **($$$)**
  - ✓Millions of dollars / year
- Typical sulfur impurities
  - ✓Ethylene
    - Hydrogen Sulfide (H_2S)
  - ✓Propylene (Polymer Grade)
    - Carbonyl Sulfide (COS)
    - H_2S (Only during startup or unit upset)

Sulfur in Ethylene

- During steady state operation, sulfur in ethylene product is not normally an issue
- A cracked gas compressor surge or trip sometimes results in a caustic wash system upset allowing carbon dioxide and hydrogen sulfide to enter the ethylene splitter and ethylene product



Sulfur in Propylene

- In the cracking furnaces, most (70 >90%) of the inlet COS is converted to H_2S
- Caustic wash towers do not remove a significant amount of COS
- The majority of COS exiting the cracking furnaces will enter the C3 splitter and exit with the propylene product
- A major unit upset or start-up condition could result in a trace amount of H_2S entering the C3 splitter and exiting with the propylene product.

Impact of S on Polymer Yield

Polypropylene Yield Data: Ziegler Natta Catalyst
Poison Level for Indicated Effect[1,2]

	Yield Loss		
Poison	5%	10%	20%
Carbonyl Sulfide	30	60	220
Hydrogen Sulfide	2800	3400	4600

1. *Equistar yield loss data*
2. *Poison level (ppb, wt) for indicated effect*

Specifications

- Poison levels for Metallocene catalyst: Propylene
  - ✓Acceptable maximum sulfur levels
    - Carbon Disulfide (CS_2): 50 ppb
    - Carbonyl Sulfide (COS): 10 ppb
    - Dimethyl Sulfide (C_2H_6S): 1 ppm
- Manufacturing specifications for polymer grade monomer
  - ✓Ethylene
    - Hydrogen Sulfide (H_2S): 50 wt. ppb
  - ✓Propylene
    - Carbonyl Sulfide (COS): 20 wt. ppb

Analytical Challenges

- If no measurable sulfur response is noted at the analyzer, what does that mean?
  - ✓The hydrocarbon stream is free from S impurities
  - ✓"Purified" the hydrocarbon gas stream
    - Reacted out the S
- What are the issues to consider here?

Analytical Challenges

- Preparation of accurate ppb level sulfur standards
- Reasonable shelf life of ppb level sulfur compounds
- Delivery of sulfur compounds to the analytical instrument
  - ✓ Regulator
  - ✓ Tubing
- Analysis
  - ✓ Inlet system
  - ✓ Column

What Exactly is the Challenge?

- Accurate sulfur standards are now available
- Equipment to measure ppb S levels currently exists
- What about the rest of the story?
  - ✓Regulators
  - ✓Transfer tubing
  - ✓GC inlet system
  - ✓Column

→***How do these impact S analysis?***

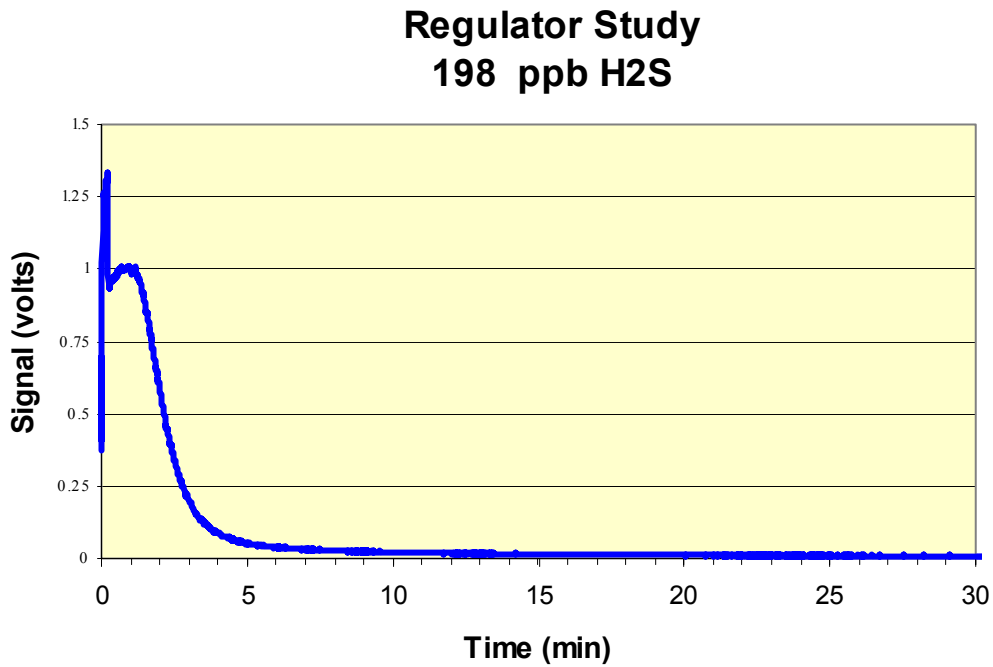
Summary: Storage

- Because of the long contact times associated with storage (minutes to years), any small reactivity of the sulfur compounds with the storage materials becomes critical
  - ✓ A shelf life study was performed with nominal 100 ppb H_2S in N_2 balance mixtures[1]
    - Excellent Materials
      - *Silcosteel™, Sulfinert ™*
        - » Bonded inert layer to surface of stainless steel
      - *ALPHATECH™ - Air Liquide America*
        - » Cylinder treatment process that yields a shelf life >1 year
    - Poor Materials
      - *Aluminum, Carbon Steel, Stainless Steel*

[1] *Benesch, R., Jacksier, T."The Preparation of Low Concentration Hydrogen Sulfide Standards" GCC 2002, paper 050*

Regulators

- Materials not as critical as for storage because of decreased contact times
  - ✓ A regulator comparison study was performed[1] with ppb H_2S in N_2
    - Stainless steel regulators work well once "passivated"
      - *Smaller volume SS regulators are preferred*
    - Materials with brass should be avoided at all costs
    - Sampling flow rates should exceed 100 mL/min

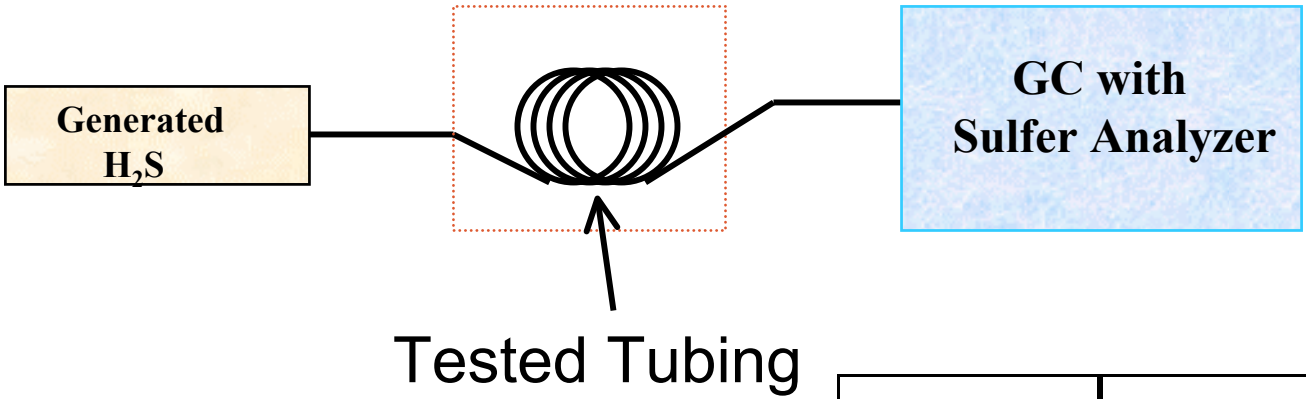


→Signal totally lost with utilization of incorrect regulator

[1] *Benesch, R., Haouchine, M., Tabert. B., Jacksier, T. "Low Concentration Hydrogen Sulfide Standards: Is it Possible to Obtain and Regulate at Concentrations below 100 ppb?" Abstract 40 Wednesday Oct 22nd Bluebonnet 9:20 am*

Tubing

- Materials are not as critical as for storage because of decreased contact time



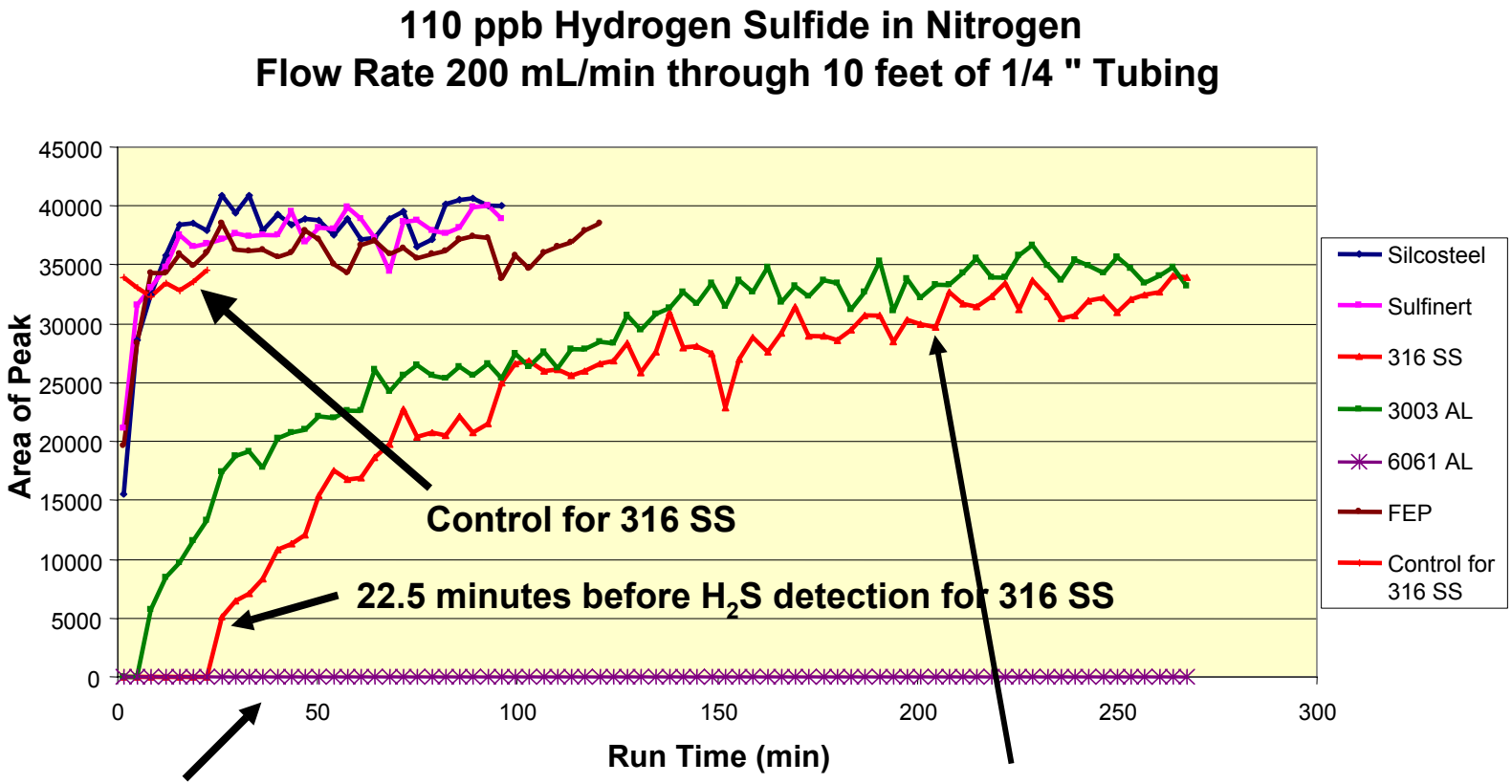
- Tubing
  - ✓Materials – Present in analysis systems
  - ✓Sizes 1/4”, 1/8”, 1/16”

1/4 inch	1/8 inch	1/16 inch
Silcosteel™	Silcosteel™	Silcosteel™
Sulfinert ™	Sulfinert ™	Sulfinert ™
316 SS	316 SS	316 SS
3003 Alum.	3003 Alum.	x
6061 Alum.	x	x
FEP	FEP	x
x	PEEK	PEEK

Procedure

- Control
  - ✓Prior to testing – control experiments were run without the tubing to determine "control" signals
- Testing
  - ✓Tubing piece attached and analysis started
    - Pretreatment of tubing
      - *Used "as is" from manufacturer*
      - *Left exposed to air until tested*
      - *No purging after attachment*
    - Analysis finished when signal approached control value

1/4 Inch Tubing

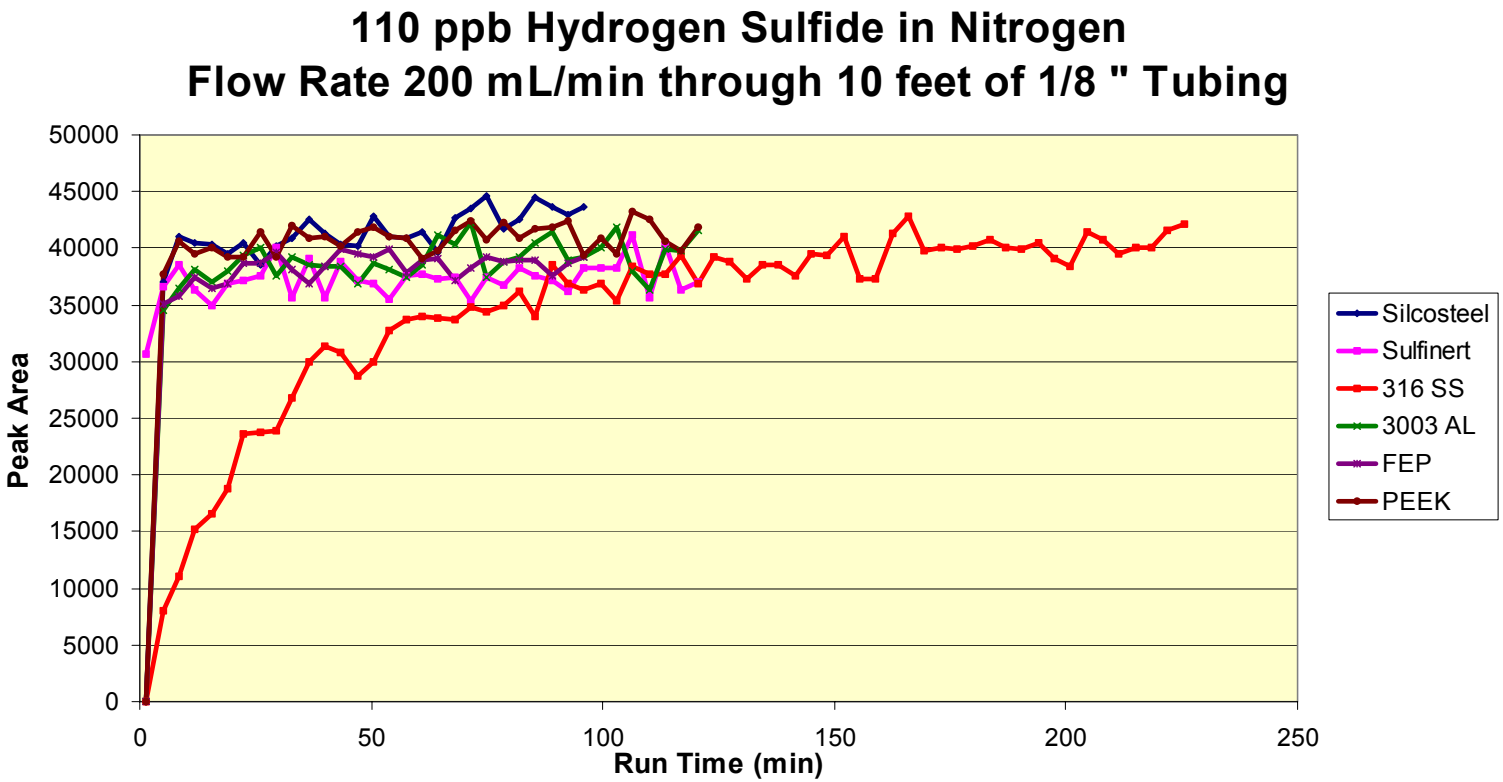


Surface of 6061 AL was very rough

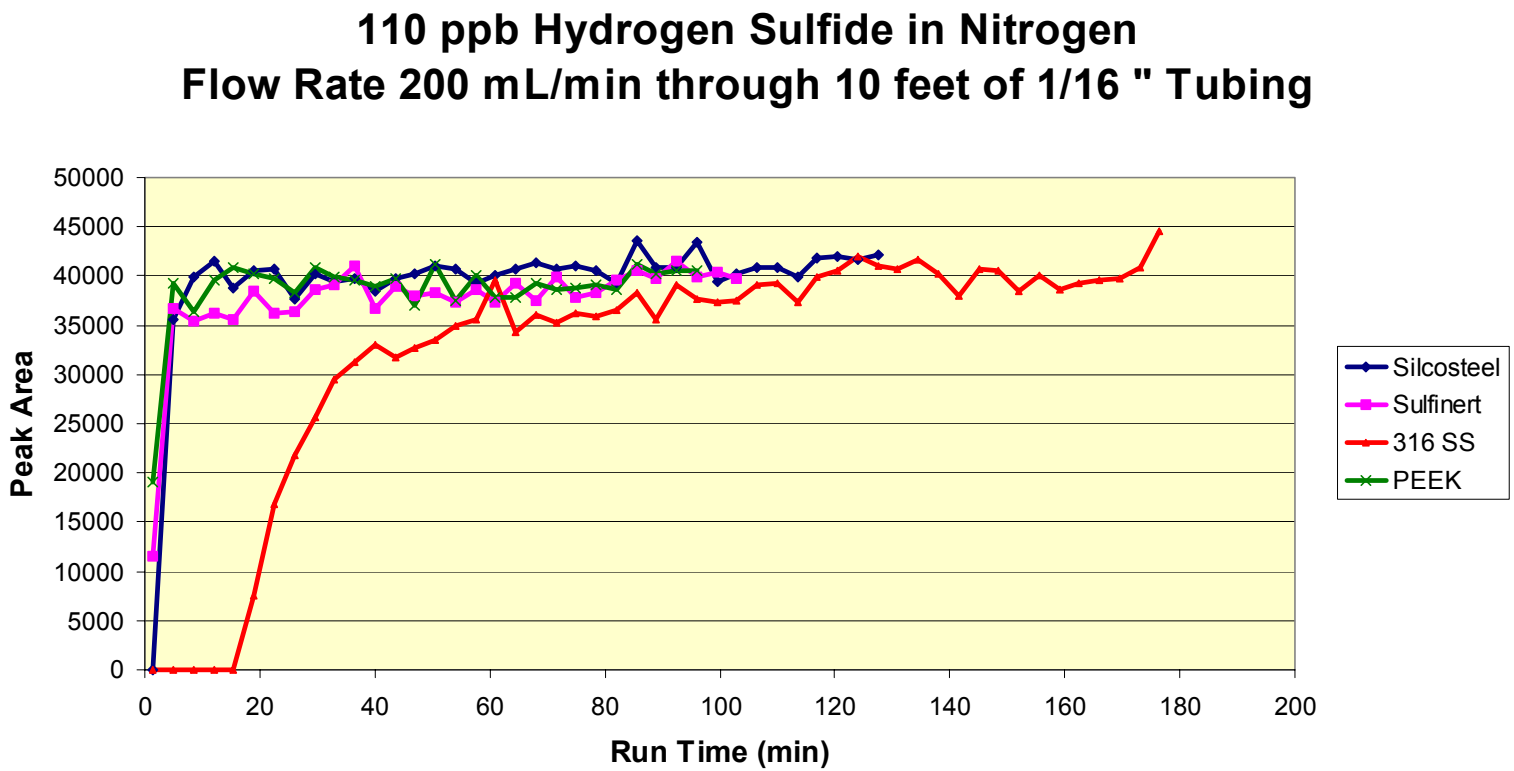
> 200 minutes before control value was reached

→Residence time of the gas in the tubing < 1min

1/8 Inch Tubing



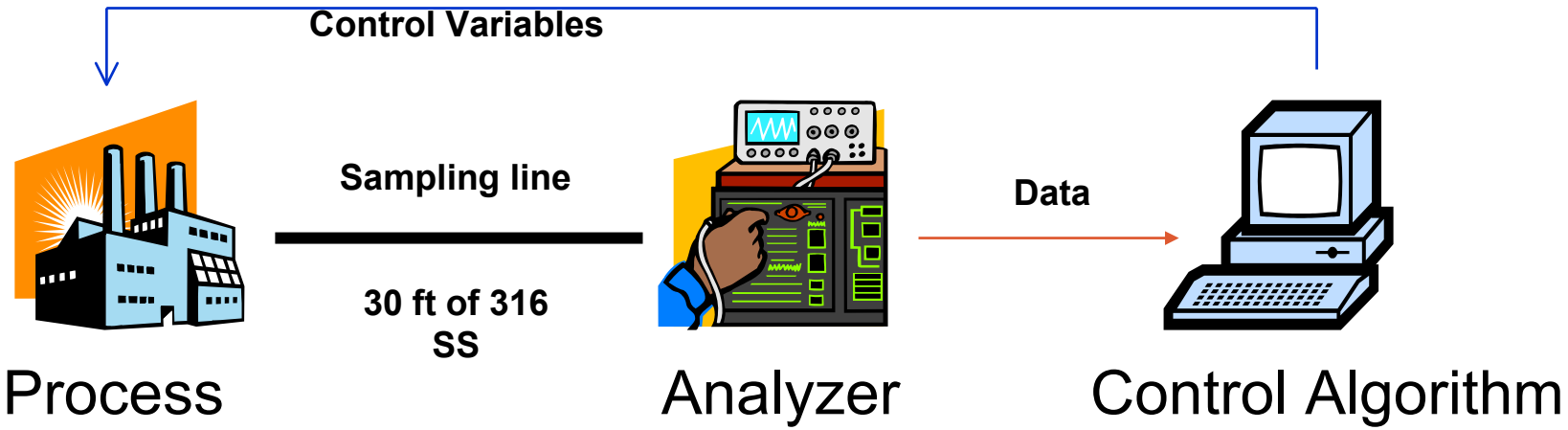
1/16 Inch Tubing



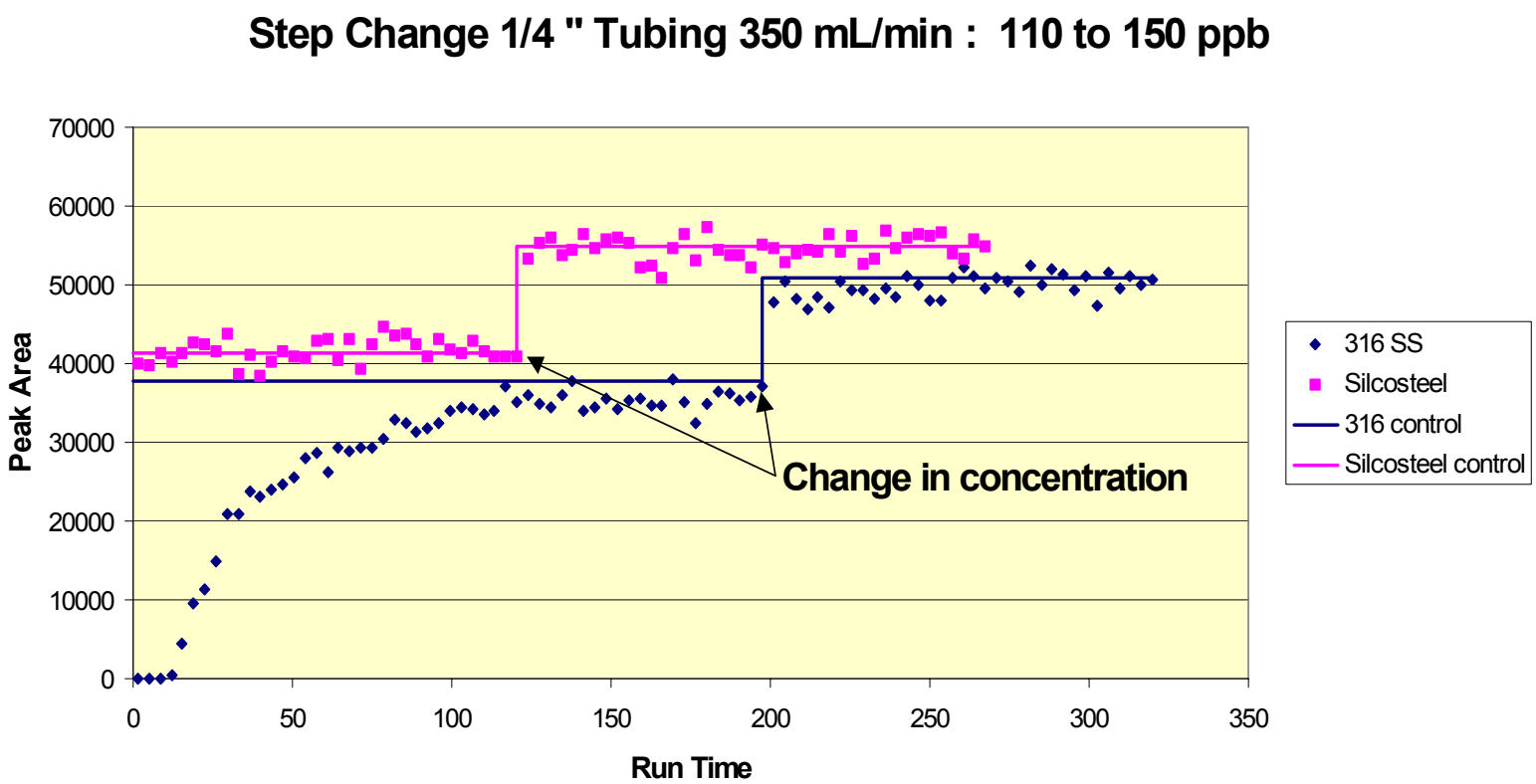
→ 1/16” SS is not EP and increased time for appearance of S signal maybe related to rougher tubing surface

Step Change in Concentration

- How will a change in the inlet concentration be observed in the outlet concentration?
  - ✓Will there be a significant time delay in analysis?
    - For 316 SS, up to 200 minute delay per 10 ft of tubing
      - *Lead to process problems*



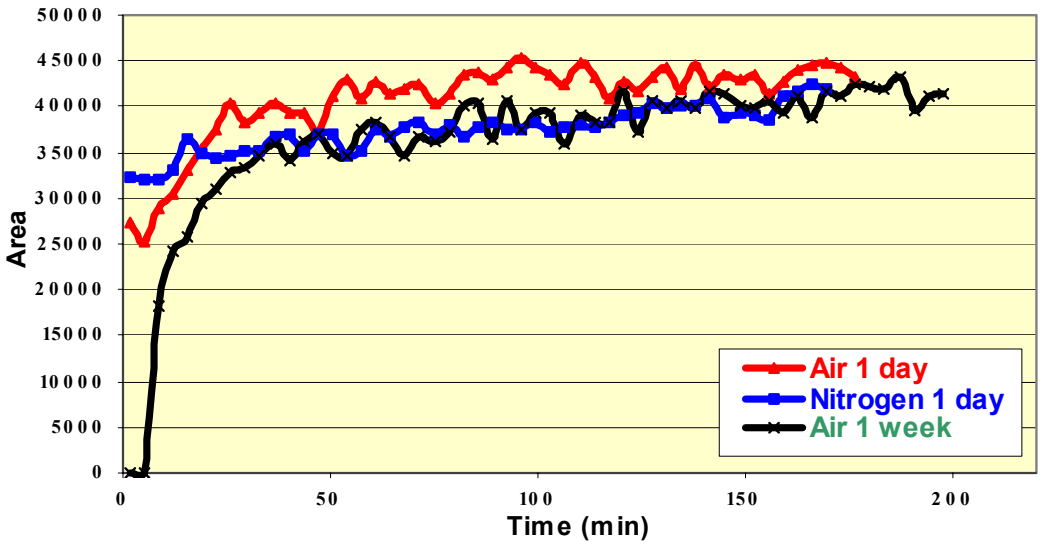
Step Change In Concentration



- ✓Silcosteel showed rapid increase
- ✓316 SS showed rapid increase once passivated

Passivation of SS

- Is the passivation of stainless steel permanent?
  - ✓Three pieces of 316 SS passivated and left to sit under different conditions
    - 1 - Exposed to N_2 for one day
    - 2 - Exposed to air for one day
    - 3 - Exposed to air for one week



✓After exposure – retested with 100 ppb H_2S in N_2 at 350 mL/min

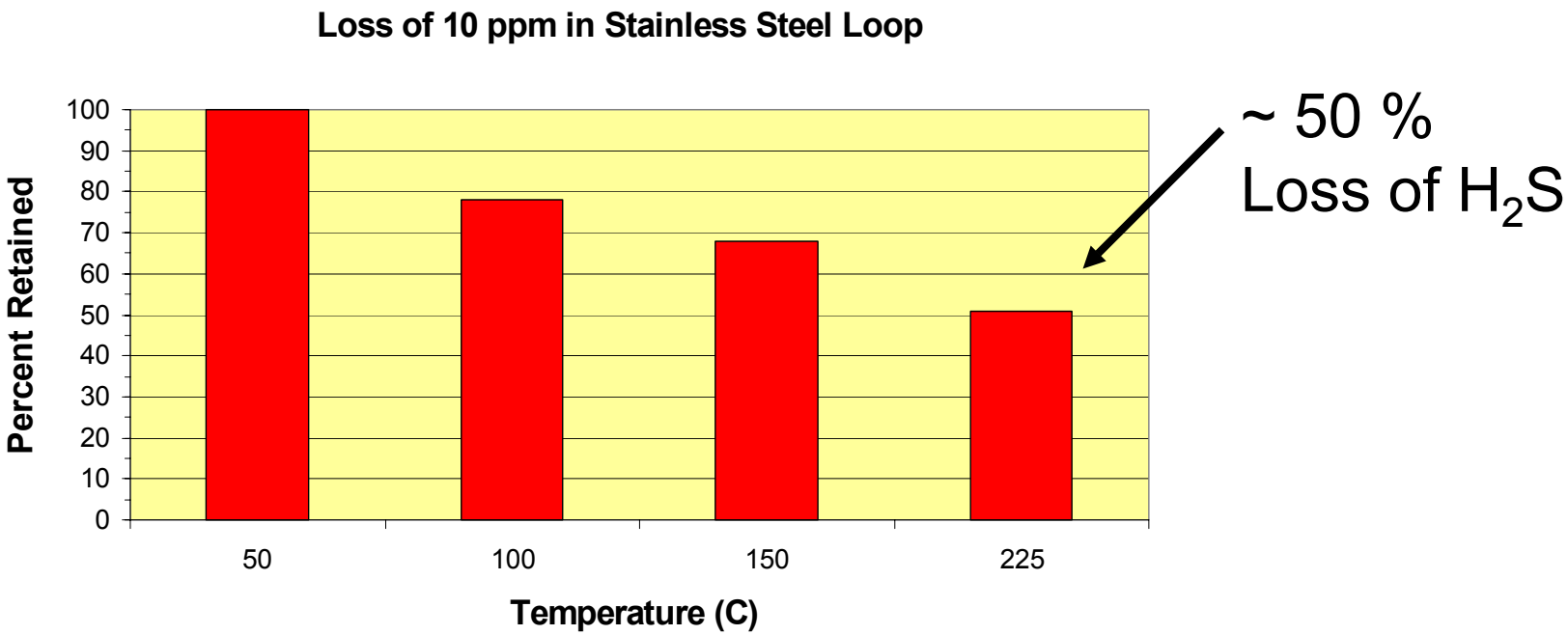
✓Long exposures (> 1week) will require repassivation

GC: Valve

- Loop materials
  - ✓ Stainless Steel - requires passivation
  - ✓ Silcosteel™, Sulfinert ™ - good material
- Temperature
  - ✓ Loops - often heated to prevent adsorption of compounds – how does this effect the reactive sulfurs – 2 concentration levels – 100 ppb and 10,000 ppb
    - Low concentration 100 ppb H_2S in N_2
      - *Silcosteel™ – no noticeable effects from 50 - 225 °C*
      - *Stainless steel – complete loss of signal when heated to temperatures above 100 °C, even after passivating*

GC: Sample Loop

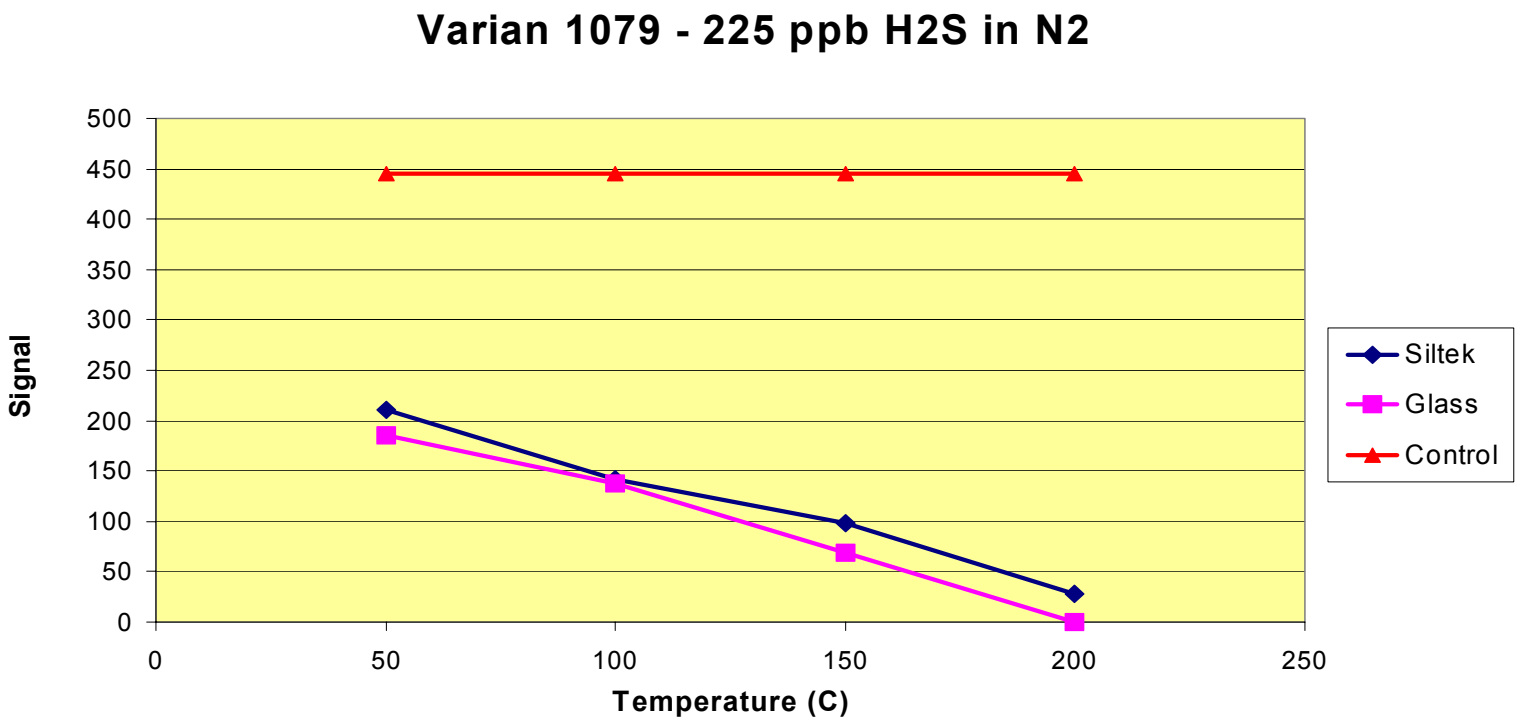
- High concentrations 10 ppm
  - *Silcosteel™ - no noticeable effect 50 °C to 225 °C*
  - *Stainless Steel – loss of signal*



GC: Inlet

- On column injection is preferred because of minimal sample contact
- Inlets Tested – materials and temperature effects
  - ✓Concentration tested – 225 ppb H_2S in N_2
    - Varian 1079
      - *Glass - insert*
      - *Siltek™ insert (Restek)*
    - Agilent Volatiles Interface

GC: Inlet

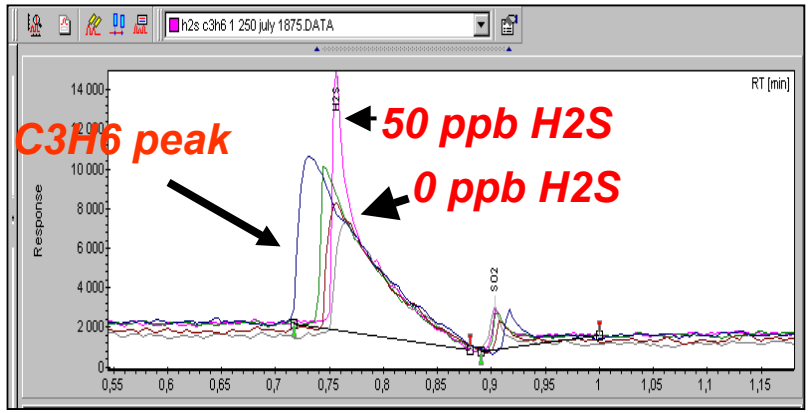


- ✓"Control" determined by using a Silcosteel™ Tee as the inlet

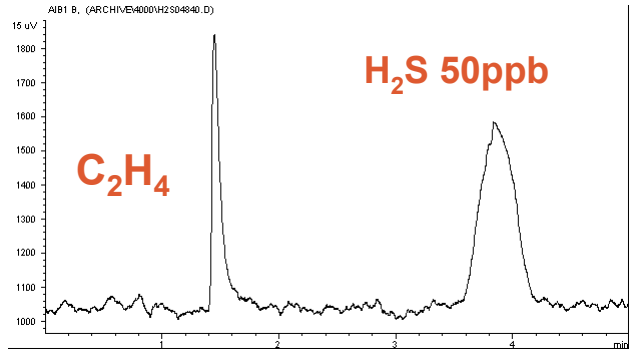
- Volatiles Interface showed no temperature effects (Agilent)

GC: Columns

- Even though the SCD is "sulfur specific", high concentrations of olefins will produce peaks
- Hydrocarbon peaks must be separated from the S compounds[1]
  - ✓ Ethylene (& propylene) and H_2S coelute at temperatures above 0°C
  - ✓ Good separation at elevated temperatures with correct column selection



100 % dimethyl polysiloxane
split ratio 1:1
-50°C

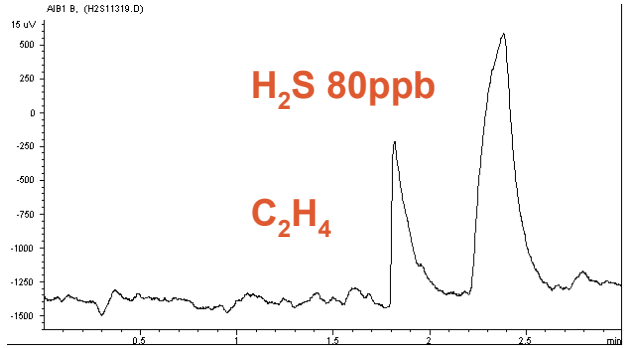


[1] *Grimberg, A., Benesch, R.,Haouchine, M., Richard, S., Coffre, E., Jacksier, T. "Analysis of Sulfur Compounds in Hydrocarbon Matrices", Pittcon 2002, paper 740-2*

GC: Column

- Ethylene analysis
  - ✓ 100 % Dimethyl polysiloxane – often recommended for sulfur analysis
    - Coelution of ethylene and H_2S at non cryogenic conditions
  - ✓ Varian Silicaplot
    - Good separation
  - ✓ J&W Gas Pro
    - Good separation at temperatures >100°C

Gaspro column
split ratio 4:1
100°C



Summary

- Low concentrations of sulfur will poison Ziegler-Natta and Metallocene catalysts
- Store sulfur compounds in containers with Sulfinert, Silcosteel, or ALPHATECH™ surface treatment technology
- Low wettable surface SS regulators work well
  - ✓"*Traditional*" SS regulators can be used if passivated and if the flow rate exceeds 100 mL/min
- Tubing should be Sulfinert, Silcosteel, or PEEK
- Column selection is critical for accurate analysis in hydrocarbon matrices
- Sample loop should be coated
- Utilization of "on column" or volatile interface for sample inlet

Conclusion

- Sulfur analysis is not trivial …the devil is in the details
  - ✓Correct component selection is the hallmark for a successful analysis

- Feel sympathy for those who do trace level sulfur analysis

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IMPROVING THE RELIABILITY OF ANALYTICAL AND SAMPLING SYSTEMS IN CHALLENGING AND CORROSIVE ENVIRONMENTS

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KEYWORDS

Carboxy-Silane, silicon, corrosion control, sulfur, mercury, adsorption, inertness, chlorides, sulfuric acid, off-shore corrosion, salt water corrosion

ABSTRACT

Process analyzers and process sampling systems often times are exposed to challenging environments both internally and externally. Many sample streams are corrosive or contain active compounds that reduce equipment lifetime or require extended preventative maintenance. Some systems are exposed to environments such as sea water, which cause rapid deterioration of equipment, requiring extra costs to keep them operating. For systems that are required to give accurate, reliable and repeatable data in such conditions, the cost of upkeep and maintenance is much larger than systems in more benign environments. This paper reports on data using wear resistant, chemically inactive surface treatment to greatly reduce maintenance cycles and improve analytical reliability.

INTRODUCTION

This paper presents laboratory corrosion and chemical inertness test results for a variety of chemically deposited coatings used by the process analyzer manufacturers. Through improvements in chemical composition, the properties of existing and new coatings will be evaluated in environments common to the petrochemical, refining and off-shore industries.

Process analyzers used in the refining, petrochemical and off-shore environments are exposed to a variety of potentially damaging compounds. Sulfuric acid, hydrochloric acid, caustic streams and salt-water exposure are environments that will be evaluated with stainless steel and stainless steel surfaces treated with silicon and carboxysilane materials.

In addition to corrosion resistance, process analyzers in these applications must also maintain chemical inertness for the sampling of reduced sulfur compounds.

DISCUSSION

In choosing a substrate enhancing coating, selection of material properties is important. Different surface treatments will have different useful ranges of exposure and chemical reactivity. For applications where inertness to reduced sulfur compounds, for example H_2S, is required, modified amorphous silicon treatments are ideal. Applications with particulate and harsh environments requiring part-per-million stability of reduced sulfurs, a carboxysilane material can be used.

Using Auger Spectroscopy, the atomic composition of amorphous silicon (Figure 1) and a carboxysilane coating (Figure 2) are analyzed. The amorphous silicon coatings are usually surface enhanced to deliver low part-per-billion inertness to common active compounds. The silicon substrate, though chemically inactive, has a low level of wear resistance. The carboxysilane surface treatment is a uniform composition of silicon, oxygen and carbon throughout the matrix. Though less chemically inert, it maintains a very corrosion resistant and wear resistant layer. Table I lists the physical properties of both coatings. Both of the coatings diffuse 400 to 500 angstroms into the lattice of the steel which provides for an excellent adherence to the surface.

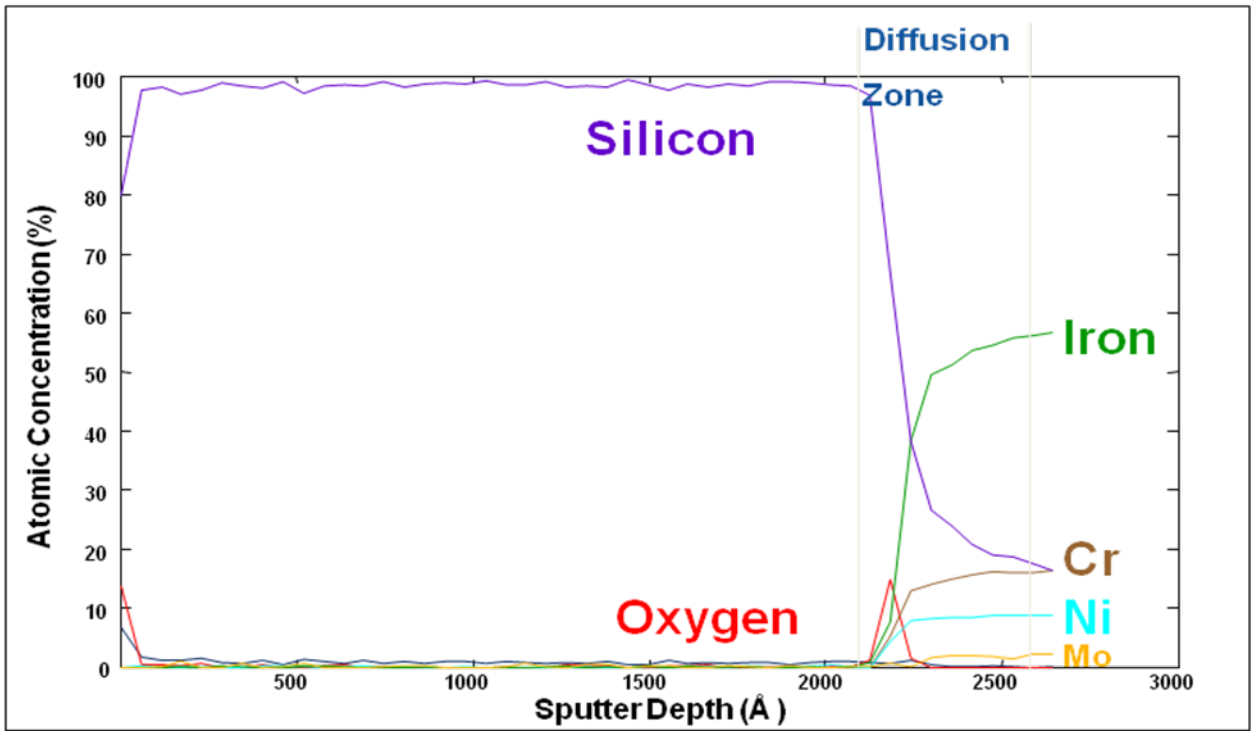

FIGURE 1: AUGER DEPTH PROFILE OF SILICON COATED STAINLESS STEEL

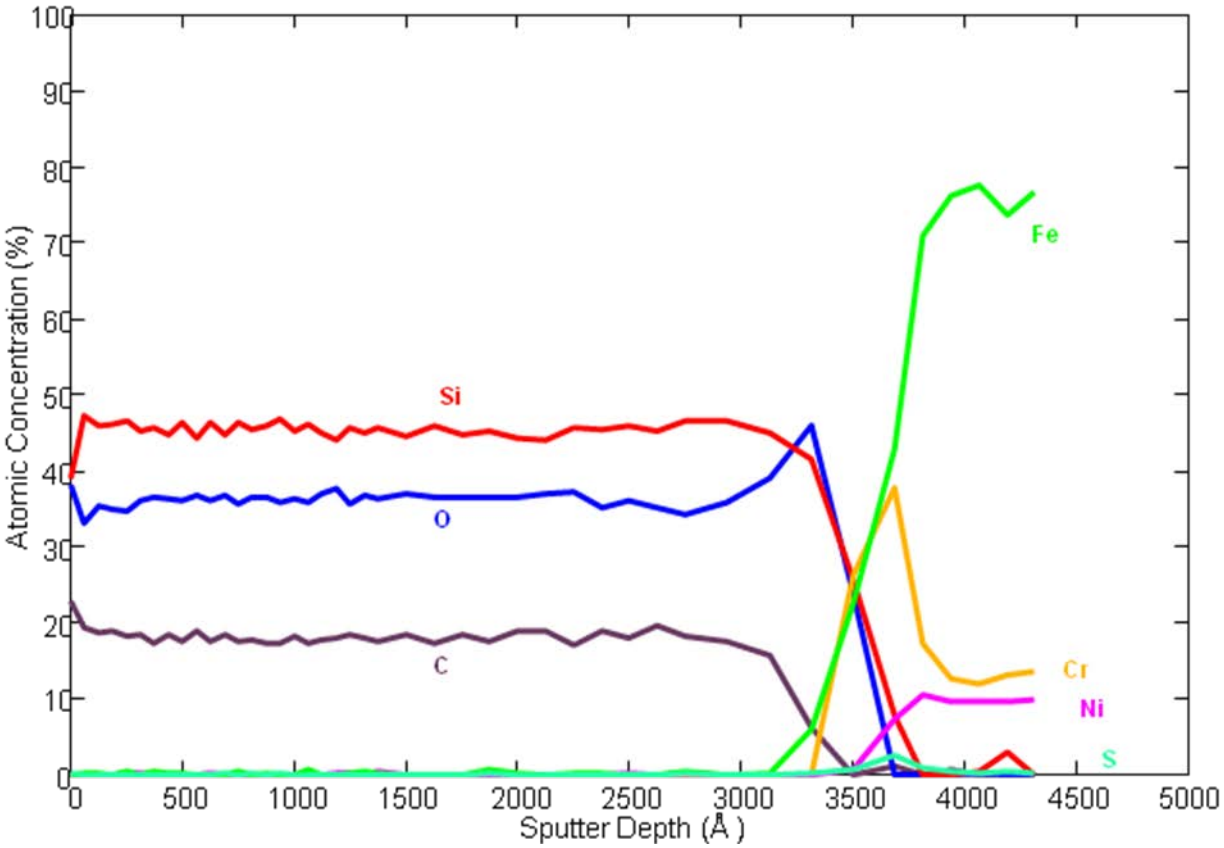

FIGURE 2: AUGER DEPTH PROFILE OF CARBOXYSILANE COATED STAINLESS STEEL

TABLE I: PHYSICAL PROPERTIES OF COATINGS

Coating	Silicon: SilcoNert®	Carboxysilane : Dursan®
Maximum Temperature	1000°C	450°C
Minimum Temperature	-196°C	-40°C
Low pH limit	0	0
High pH limit	7	14
Thickness	500nm	2000nm
Adhesion	Excellent	Excellent

EXPERIMENTAL

.

Chloride environments and chloride containing streams can greatly reduce the lifetime of process systems. Coatings, paints and costly super alloys have been used to increase the lifetime of components in salt water and/or chloride containing environments. Table II provides the results obtained from ASTM G31 testing. This method is an immersion test for 24 hours in a 6M HCl (18%) solution at room temperature and pressure. After immersion, differential weighing allows the amount of material loss to be determined. The sample size for each configuration was 3 samples. The amorphous silicon coated stainless steel shows greater than 20 times the resistance of non-treated stainless steel in these environments and the carboxysilane treatment creates greater than 200 times the resistance. Any loss in the coated samples occurred as a result of pitting corrosion. The pitting is an indication that there are still pin-holes present in the surface which allowed corrosive attack to initiate.

TABLE II: WEIGHT LOSS AFTER 24 HOUR EXPOSURE TO 6M (18%) HCL

24hr; 6M HCl; 22ºC	**304 SS**	**Silicon coated**	**Carboxysilane coated**
MPY (mils-per-year)	389.36	16.31	1.86
Improvement Factor	---	23.9	209.8

Another factor for consideration is the wear resistance of coatings applied to analytical sampling equipment. This factor is critical, especially in applications where there is mechanical rubbing with valve movements or process abrasion via particulate moving through the sampling equipment at high velocity. Valve seat movements or particulate in these applications can quickly erode a soft coating such as silicon creating sites for adsorption to occur. Table III summarizes the data obtained from wear studies conducted on both non-treated and treated surfaces. Data was generated using a pin-on-disk tribometer (Nanovea, Irvin, CA). The experiment uses a flat plate loaded onto the test rig and the indenter applies a precise force to the surface. The plate is then rotated and forces are measured between the pin and the disc. Results from this experimental method can produce wear behavior and friction coefficients of the plate surface[1]. Results from this study demonstrate that the carboxysilane coatings wear less than untreated steel and silicon coated surface. The improved wear resistance as a result of the coating will lead to longer lifetimes of system components in extreme environments.

TABLE III: PHYSICAL PROPERTIES OF COATINGS

Pin on Disc; 2.0N	**316 stainless steel**	**Carboxysilane coated 316 stainless steel**	**Silicon coated 316 stainless steel**
Wear rate ($x10^{-5}mm^3/N\ m$)	13.810	6.129	2
Improvement Factor over SS	---	2 times	1/3 times

For analytical systems used in sampling and transfer of sulfur containing species, system inertness must be addressed when stainless steel components are used. In most refining and petrochemical streams, analysis in the ppm level they are acceptable. Figure 3, demonstrates that even at concentrations of 50ppm, hydrogen sulfide sampling requires passive surfaces[2]. In this analysis sample cylinders tested were either sourced from the manufacturer, non-coated, or treated with a carboxysilane, commercial name Dursan™. Figure 4, demonstrates the need for coating during the sampling, storage and analysis of part-per-billion levels hydrogen sulfide. In critical applications, the ultimate inertness of components is enhanced using silicon based coatings at the cost of physical durability.

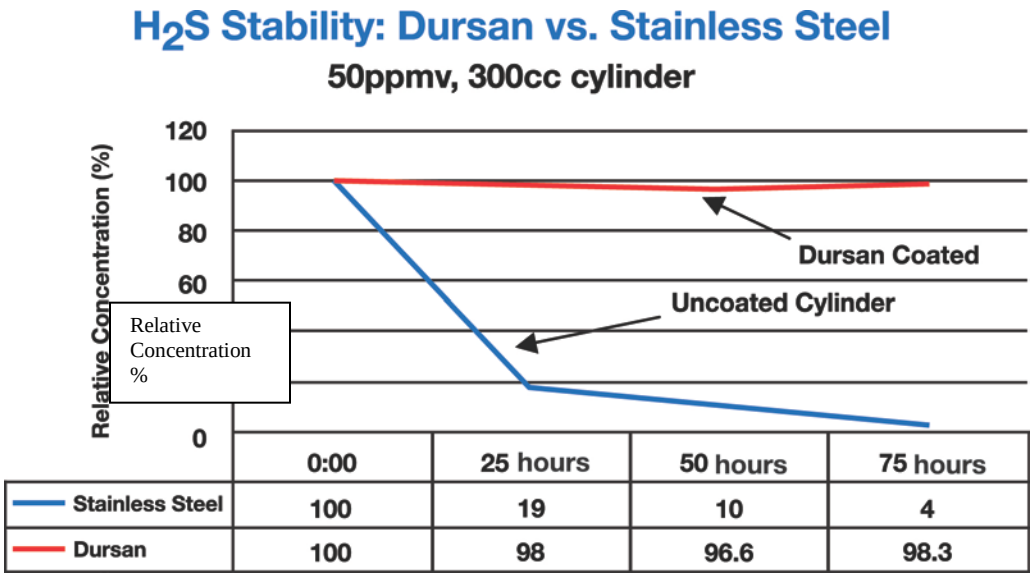

FIGURE 3: SULFUR COMPOUNDS AT 50 PARTS-PER-MILLION IN CARBOXYSILANE TREATED STAINLESS STEEL CONTAINERS VERSUS NON-TREATED CYLINDER

In Figure 3 and 4, the degradation of hydrogen sulfide on bare stainless steel is rapid and irreversible: Both at 50ppm and 17bbp levels, H_2S is lost within 24 hours.

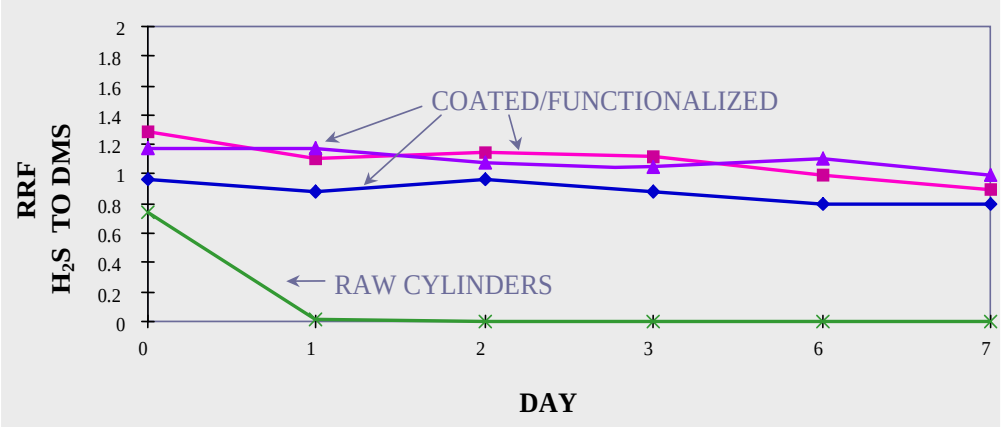

FIGURE 4: SULFUR COMPOUNDS AT 17PPBV IN AMORPHOUS SILICON TREATED STAINLESS STEEL CONTAINERS

Effect of passivation on sulfur storage and transport has consistently been raised. Passivation is a technique that is based on the assumption that if all active areas of a transport vessel or storage vessel are taken up by sulfur compounds, then they are made inert to sulfur compounds. There have been studies to support this at low temperature for gas phase transport through low surface area regulators[3]. It was demonstrated that purging a component with clean gas can reduce the inertness of the passivation with measurable impact occurring within 1 day and complete within 1 week. Additional data in the same study also demonstrate that heated stainless does not passivate and complete adsorption of sulfurs will occur no matter the conditions and previous exposure to sulfur compounds.

In work to test the stability of sulfur compounds during static sampling, as in sample cylinders, the use of gases such as silane (SiH_4) along with multiple day exposure to 5000ppm H_2S was required to create a passive cylinder for storage[4]. Much of the data in these studies was done to demonstrate stability for the use of creating low-level standards.

Commercially available inert coated components have eliminated the need for passivation and are now recognized as a “use out of the box” solution to sulfur sampling and transport. This eliminates the need for working with dangerous materials such as high concentration H_2S or pyrophoric gases such as silane. The value delivered by coating solutions cannot be taken for granted in comparison to passivation techniques which increase the risk of obtaining poor analytical results.

A surface that is hydrophobic is critical in refining and petrochemical applications. Many of the streams are very dry but an upset in process conditions will lead to moisture in the sampling system. This moisture will adversely affect analysis because of the polarity of the water in the system. The faster a system can “dry” of any moisture, the faster the analytical system will begin to generate reliable data. Figure 5, shows images of water droplets applied to 304 stainless coupons as well as coated 304 stainless surfaces. The coatings impart a hydrophobic

characteristic to the stainless steel substrate. The hydrophobic surfaces are easier to purge free of water. This is critical in refining and petrochemical operations when upsets occur, as moisture in analyzer systems lead to poor and unreliable data.

FIGURE 5: COATING OF 304 STAINLESS RESULTS IN ABILITY TO INCREASE HYDROPHOBICITY

CONCLUSION

Coatings are well accepted as a means to improve analytical system accuracy and durability in demanding applications. To select the proper coatings, properties such as acid exposure, particulate exposure and the needs for chemical inertness must be know. When coatings are properly matched to the physical and chemical demands of an application, years of accurate and reliable results can be expected.

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2. Barone, G., Smith, D., Higgins, M., Rowan, S., Gross, W., Harris, P., “Impact of Sampling and Transfer Component Surface Roughness and Composition on the Analysis of Low-Level Sulfur and Mercury Containing Streams”, Restek Corp., O’Brien Corp., Haritec LLC, ISA Symposium, October, 1995.
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Removing Rust Prior to Coating

By Jim Mattzela

Abstract

Silcolloy® and Dursan® coatings are both applied to improve the corrosion resistance of stainless steel and other alloys. The effectiveness of the coating as a barrier to corrosion can be compromised if the substrate surface has pre-existing rust or corrosion spots (stains). Once noted, rust can easily be removed prior to coating by utilizing some simple chemical techniques to create an optimum surface.

Stainless Steel Rust

Stainless steel is typically specified because of its ability to resist corrosion in a variety of environments. Unfortunately, stainless steel is not fully stain- or rust-proof, it is just more *resistant* to corrosion. Exposure to high salinity environments such as sea water can remove the native protective layer (chromium oxide) and allow oxidation/corrosion to occur at the substrate[i]. Another common factor which can cause the stainless steel to rust or corrode is so-called 'free iron' on the surface[ii]. This iron residue can come from a variety of sources such as transfer of tooling particulate that remains after machining and is very susceptible to corrosion if not handled properly.

SilcoTek's Experience

Any pre-existing surface corrosion can degrade the performance of SilcoTek-coated parts. We have found that even minor rusting should be removed prior to coating for optimum value. Pre-coating surface treatments are selected so that the iron oxide (rust) is removed while the majority of the substrate is unaffected. SilcoTek employs two different treatments to achieve this result: exposure to phosphoric acid or exposure to acetic acid.

Phosphoric Acid

Phosphoric acid will dissolve iron oxide without attacking the other components of the steel (chromium and chromium oxide, nickel, and iron). The rust is dissolved by the following reaction:

$$2\,H_3PO_4 + Fe_2O_3 \rightarrow 2\,FePO_4 + 3H_2O$$

Phosphoric acid is readily available from a number of commercial suppliers (SilcoTek uses 85% 'food grade') and it can be used with no dilution because of its non-aggressive nature. Clean up after acid treatment is done by rinsing parts in deionized water thoroughly and then returned to the coating process. Unfortunately, this technique is not 100% effective on all the forms of iron oxide and other surface preparation treatments could be necessary.

Acetic Acid

Another acid treatment which is effective for removing some iron oxides uses acetic acid. Acetic acid is also available commercially (SilcoTek uses glacial acetic acid which is greater than 99.7% pure) and requires no dilution prior to use as a rust remover. The reaction with acetic acid is often slower than phosphoric acid and can be a better choice when the underlying corrosion/staining affects a larger percentage of the substrate. Following the acid exposure, parts must be rinsed thoroughly with deionized water prior to re-entry into the coating process.

Some Results

Below is an example of a part which showed rusting prior to coating and the results from our recommended acid treatments.

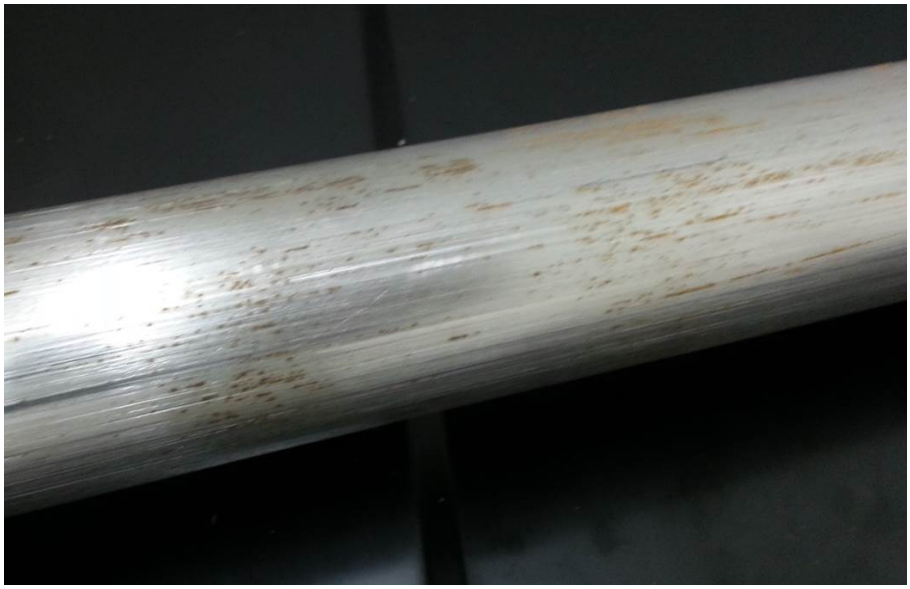

Figure 1 - Stainless steel part showing rust of surface free iron.

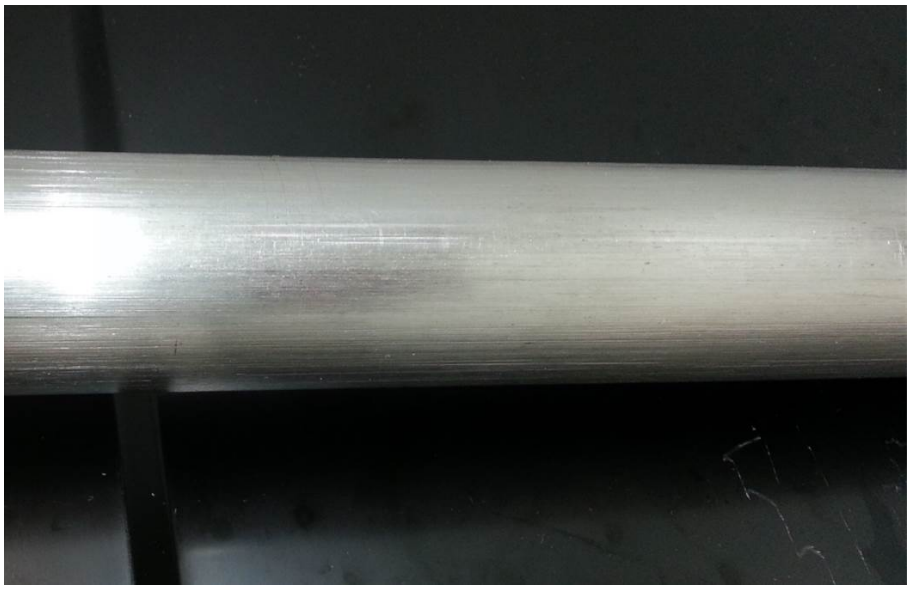

Figure 2 - Stainless steel part following treatment with phosphoric acid removal of surface rust.

Recommendation

The best substrate to coat is one that is clean and does not show rust. If there is a potential for free iron on your part's surface, SilcoTek recommends addressing it prior to shipping to us. The easiest method to remove free iron is through passivation (nitric or citric acid exposure). Any oxidized free iron (rust) can then be removed by one of the methods described above. If it is not possible to apply the removal techniques in-house, SilcoTek's sales group can provide a quote for the acid cleaning process upon request.

[i] http://www.stainless-steel-blog.com/2013/09/06/why-is-stainless-steel-stainless/
[ii] http://www.stainlessfoundry.com/FreeIron.asp