



## 100% chlorine gas corrosion test results at room temperature and 150°C

### Technical Insight

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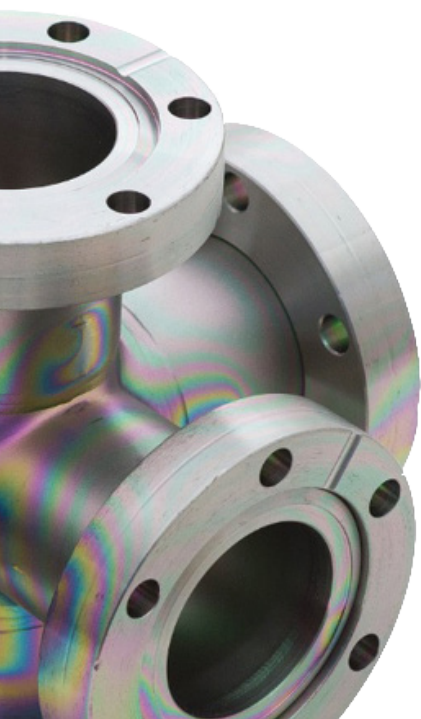
SilcoTek® Corporation

#### Synopsis

This TI summarizes the results of 100% chlorine ( $\text{Cl}_2$ ) gas corrosion test results of four SilcoTek coatings. The test was carried out by an independent lab, and the samples were exposed for 168 hours at either room temperature or 150°C under a constant flow of chlorine gas. Individual sample weight was taken before and after the exposure, and SEM-EDS analysis was performed on representative samples after the  $\text{Cl}_2$  exposure. Uncoated 316L stainless steel (SS) samples were found to exhibit the most corrosion after both temperatures. Uncoated Hastelloy C22 outperformed 316L SS and local corrosion sites were observed under SEM. The degree of protection varied by the coating type and was compared via SEM-EDS analysis.

#### Background

Many of SilcoTek's customers' applications involve gas phase corrosion. SilcoTek has collected a lot of liquid immersion corrosion data but has limited data in gas phase corrosion. This TI marks part of SilcoTek's efforts to build a database for gas phase corrosion. Chlorine was chosen for this study as it is used in a wide variety of industries, including chemical manufacturing, semiconductor, food processing and pharmaceuticals, all of which can benefit from SilcoTek's CVD coatings. An independent testing lab was contracted to carry out the corrosion tests of four SilcoTek coatings, and the results are summarized in this TI.



## Experimental

Four SilcoTek coatings applied to 316L SS coupons were evaluated. The four coatings were Dursan, SL1000 (Silcolloy 1000), SL2000 (Silcolloy 2000) and Siltride 1000. Uncoated and passivated 316L SS coupons and uncoated Hastelloy C22 coupons were included as baseline samples. Two test temperatures were evaluated, room temperature and 150°C. Three coupons per type were tested for each temperature, resulting in 18 coupons total per group.

The test specimens were loosely mounted on PTFE rods separated from each other by PTFE spacers. The rods were attached to legs which kept the specimens suspended within the appropriate test vessel, see Figure 1. The test vessels consisted of glass resin kettles with removable lids. The lids contained ports for gas inlet and outlet and a thermowell for monitoring the temperature. The gas delivery apparatus consisted of a flow meter, associated tubing (stainless steel and PTFE), valves, and fittings. The test vessels were connected in series, with gas from the flowmeter connected to the inlet of the room temperature vessel. The outlet of the room temperature vessel was connected to the inlet of the 150°C vessel. The outlet of the 150°C vessel was routed to a vapor tap and then to a caustic scrubber. The 150°C vessel was placed in a heating mantle that was connected to a temperature controller.



*Figure 1: Test vessels filled with chlorine gas*

Once the test specimens were assembled on the holders and inserted into the test vessels, the vessels were sealed and purged with high purity nitrogen (0.3 ppm O<sub>2</sub> and 1.1 ppm water) for 90 minutes. The chlorine gas (CP Grade, 50 ppm max. moisture) flow was initiated at 200 cc/minute until both vessels were filled with the green colored gas, approximately 30 minutes. The flow rate decreased to 100 cc/minute and the 150°C vessel was heated to the test temperature, approximately 100 minutes. After 7 days (168 hours), the test vessels were purged with nitrogen while the heated vessel cooled to room temperature and the green gas had been purged from the system, approximately 3 hours. The test vessels were opened and the test specimens removed.

## Results

The test specimens were examined visually for evidence of corrosion and photographed without any rinsing. Photographs of the specimens are presented in Figures 2 and 3. For the room temperature exposure (Figure 2), the uncoated 316L SS coupons had green deposits present. The green deposits were loosely adhered and after were removed with a subsequent water rinse, there were visible areas of metal loss underneath the deposits. The uncoated Hastelloy C22 coupons showed slight green discoloration in some areas, both front and back. Among the coated coupons, both SL1000 and SL2000 showed a few localized smudges but otherwise unchanged. Dursan and Siltride showed no visual changes before and after the exposure.

For the 150°C exposure (Figure 3), the uncoated 316L SS coupons had small areas of red rust present. The other coupons displayed no visual changes before and after the exposure.



Figure 2: Photographs of all coupons (front and back side) immediately after removal from the room temperature test vessel without any rinsing

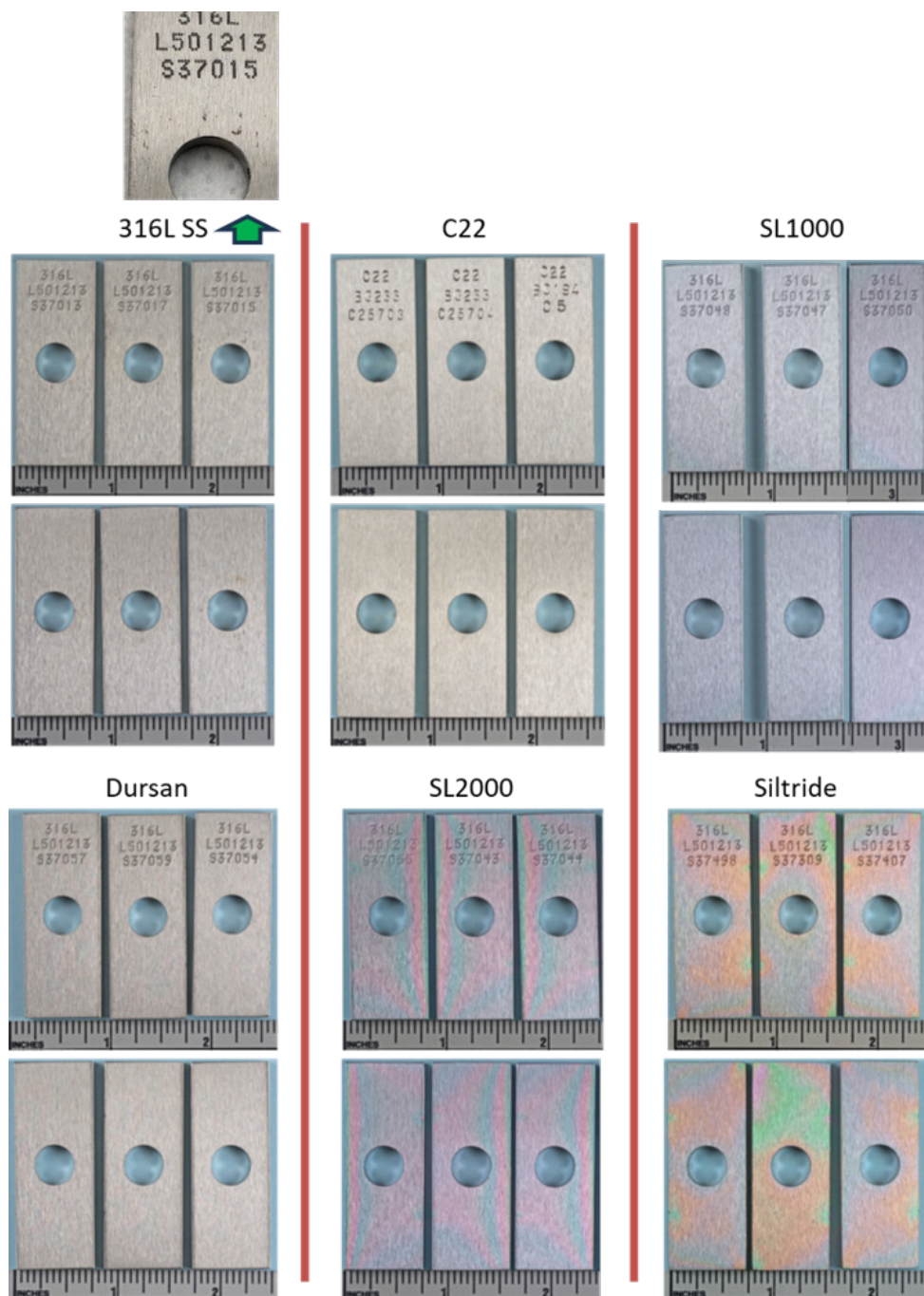


Figure 3: Photographs of all coupons (front and back side) immediately after removal from the 150°C test vessel without any rinsing

Afterwards, the coupons were sonicated in DI water and dried to remove any residual corrosion products. The coupons were then individually weighed and compared to their pre-test values to calculate corrosion rates. Except for the uncoated room temperature 316L SS samples, mass loss was minimal for all other coupons (< 1 mg), see Figure 4.

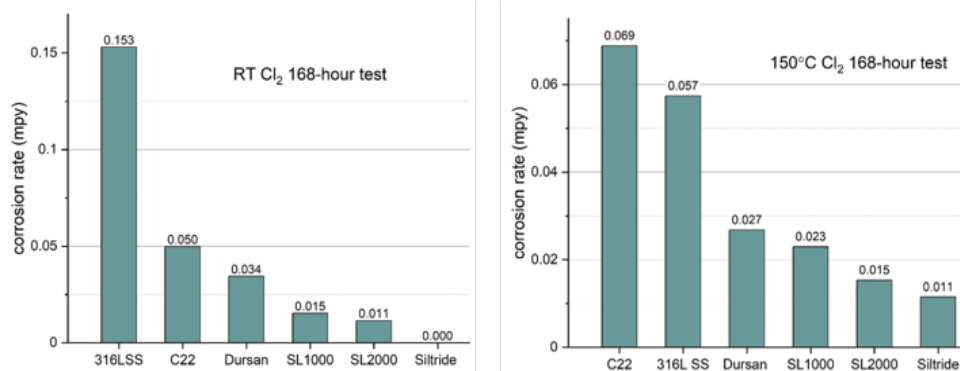


Figure 4: Corrosion rates calculated from weight loss after Cl<sub>2</sub> exposure at two temperatures; except for the 316L SS samples after RT exposure, all samples exhibited a weight loss < 1 mg

SEM-EDS analysis was performed on one coupon from each coating type, and one from each substrate type for the uncoated coupons. During analysis, each sample was first surveyed under a low magnification (500X) to search for signs of corrosion / abnormalities. When located, EDS analysis was performed at 2000X magnification on the site. If no corrosion was identified, a second coupon from the same group was analyzed for confirmation. Figures 5 – 7 show the SEM-EDS results of the samples that saw the one-week room temperature chlorine exposure.

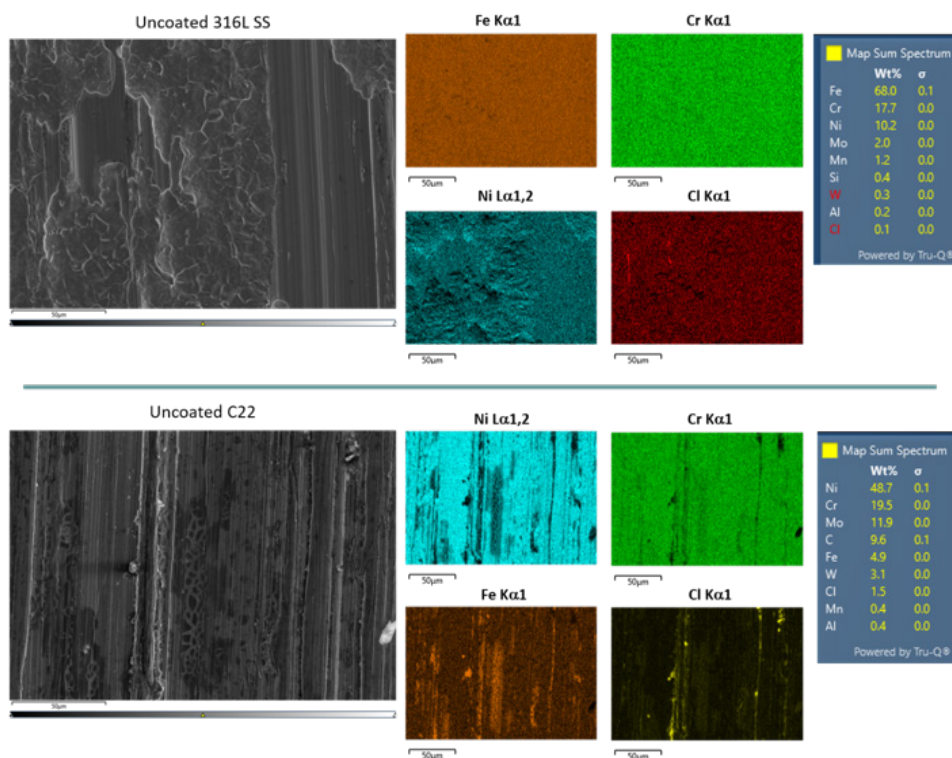


Figure 5: SEM-EDS analysis results of the uncoated 316L SS (top) and Hastelloy C22 (bottom) samples after one week of room temperature Cl<sub>2</sub> exposure, showing corrosion sites and chlorine uptake for both substrates

The uncoated 316L SS coupons exhibited the most visible corrosion spots as seen in Figure 2, and these spots showed up as roughened areas under SEM (Figure 5 top). A slight uptake in Cl (0.1%) was also detected by EDS. The Cl content was likely low because most of the chloride corrosion products were washed away during the previous sonication step. The uncoated Hastelloy C22 coupons showed significantly less corrosion spots than 316L SS. A few corrosion spots were identified and showed up as micro-cracks under the SEM (Figure 5 bottom). A moderate Cl uptake (1.5%) was detected by EDS. The chlorine uptake was attributed to chemisorbed chlorine species to the metal surface, an indication of corrosion initiation but the corrosion reaction did not proceed as far as on the uncoated 316L SS samples.

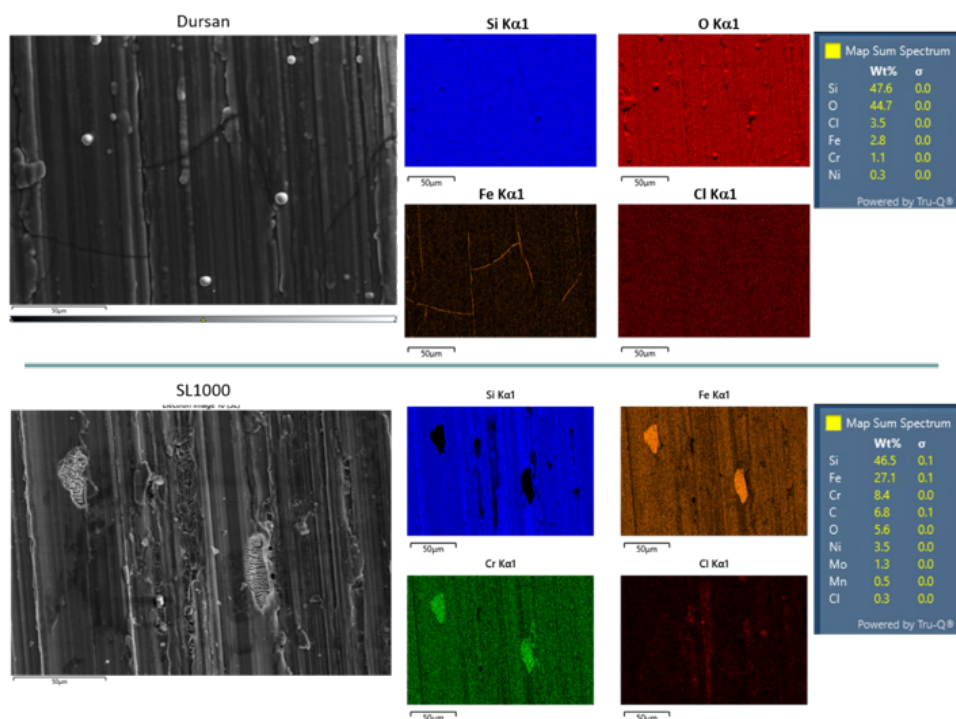


Figure 6: SEM-EDS analysis results of Dursan (top) and SL1000 (bottom) -coated 316L SS samples after one week of room temperature Cl<sub>2</sub> exposure, showing some coating cracks and a relatively high chlorine uptake in the Dursan coating, and a few localized corrosion sites along with a slight chlorine uptake in the SL1000 coating

The Dursan coupon exhibited micro-cracks under the SEM (Figure 6 top), exposing underneath SS substrate, and the cracks were present on > 50% of the surface area. A relatively high (3.5%) Cl uptake was detected by EDS, indicating that chlorine was able to penetrate through the coating to create enough stress at the substrate / coating interface to cause coating cracks. A second Dursan sample was analyzed and had similar observations. The SL1000 coupon appeared mostly intact under the SEM, with only a few corrosion spots identified (Figure 6 bottom). The corrosion sites showed local coating loss and the exposed substrate exhibited micro-cracks. A slight (0.3%) Cl uptake was detected by EDS.

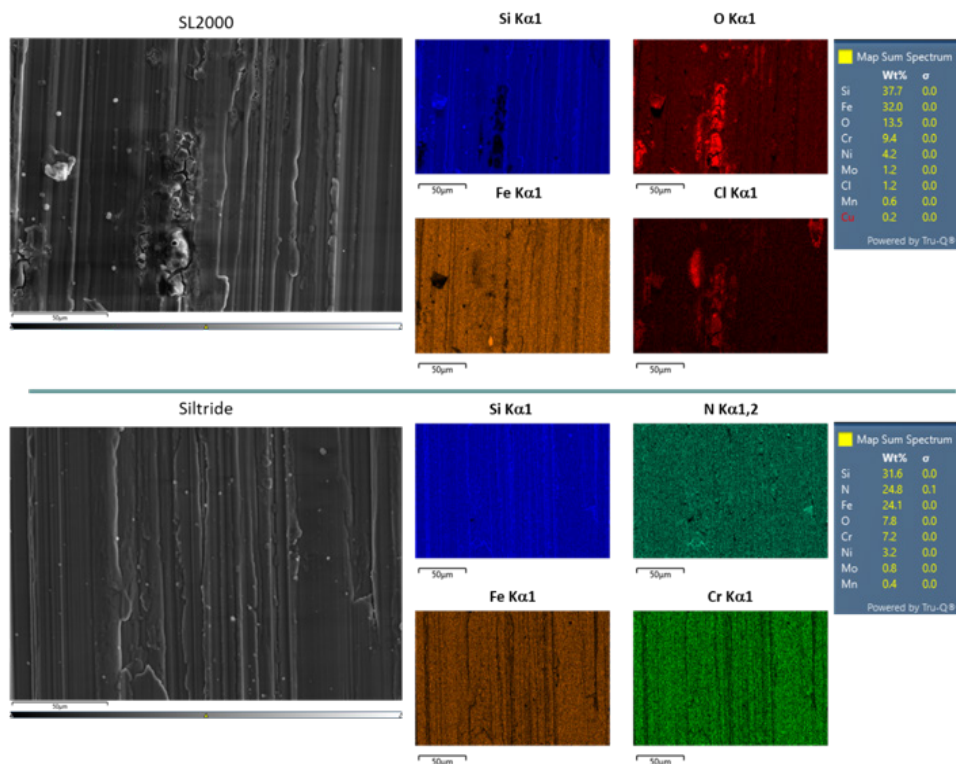


Figure 7: SEM-EDS analysis results of SL2000 (top) and Siltride (bottom) -coated 316L SS samples after one week of room temperature Cl<sub>2</sub> exposure, showing a few localized corrosion sites along with a moderate chlorine uptake in the SL2000 coating, and an intact coating with no chlorine uptake in the Siltride coating

The SL2000 coupon appeared mostly intact under the SEM, with only a few corrosion spots identified (Figure 7 top). The corrosion sites showed local coating loss and micro-cracks. A moderate (1.2%) Cl uptake was detected by EDS, higher than what was detected on SL1000, indicating that SL1000 was superior compared to SL2000 in preventing chlorine penetration. The Siltride coupon appeared intact with no corrosion spots identified under the SEM (Figure 7 bottom), and zero Cl uptake detected by EDS. The remaining two Siltride coupons were analyzed to confirm the results, and no corrosion spot was observed on those two samples either.

For the 150°C tested samples, similar SEM-EDS analysis was performed on one coupon from each coating type, and one from each substrate type for the uncoated coupons. During analysis, each sample was first surveyed under a low magnification (500X) to search for signs of corrosion / abnormalities. When located, EDS analysis was performed at 2000X magnification on the site. If no corrosion was identified, a second coupon from the same group was analyzed for confirmation. Figures 8 – 10 show the SEM-EDS results of the samples that saw the one-week 150°C chlorine gas exposure.

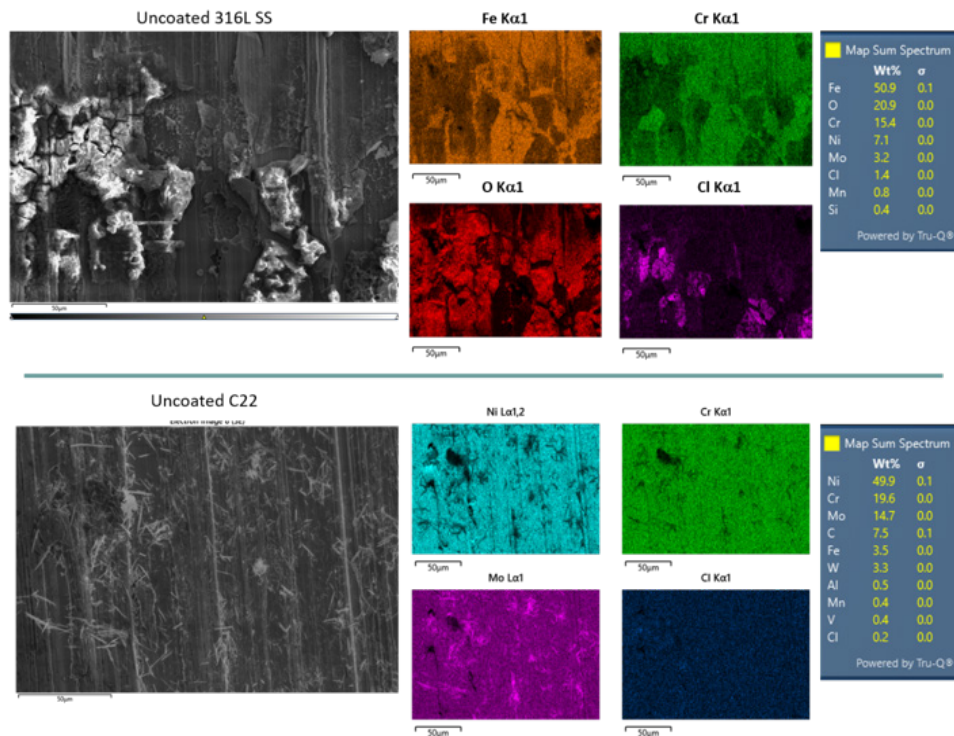


Figure 8: SEM-EDS analysis results of the uncoated 316L SS (top) and Hastelloy C22 (bottom) samples after one week of 150°C Cl<sub>2</sub> exposure, showing corrosion sites and chlorine uptake for both substrates

Among the 150°C tested samples, the uncoated 316L SS coupons once again exhibited the most visible corrosion spots as seen in Figure 3, although they appeared less prominent than on the room temperature tested bare 316L SS samples (Figure 2). The corrosion spots showed up as micro-cracks under SEM (Figure 8 top). A moderate uptake in Cl (1.4%) was detected by EDS. The uncoated Hastelloy C22 coupons appeared mostly intact under the SEM, with only a few corrosion spots identified (Figure 8 bottom). The corroded area had a “hairy” appearance, and the hairy regions were depleted in Ni and Cr but enriched in Mo, indicating a selective etch of Ni and Cr by the chlorine species. A slight Cl uptake (0.2%) was detected by EDS.

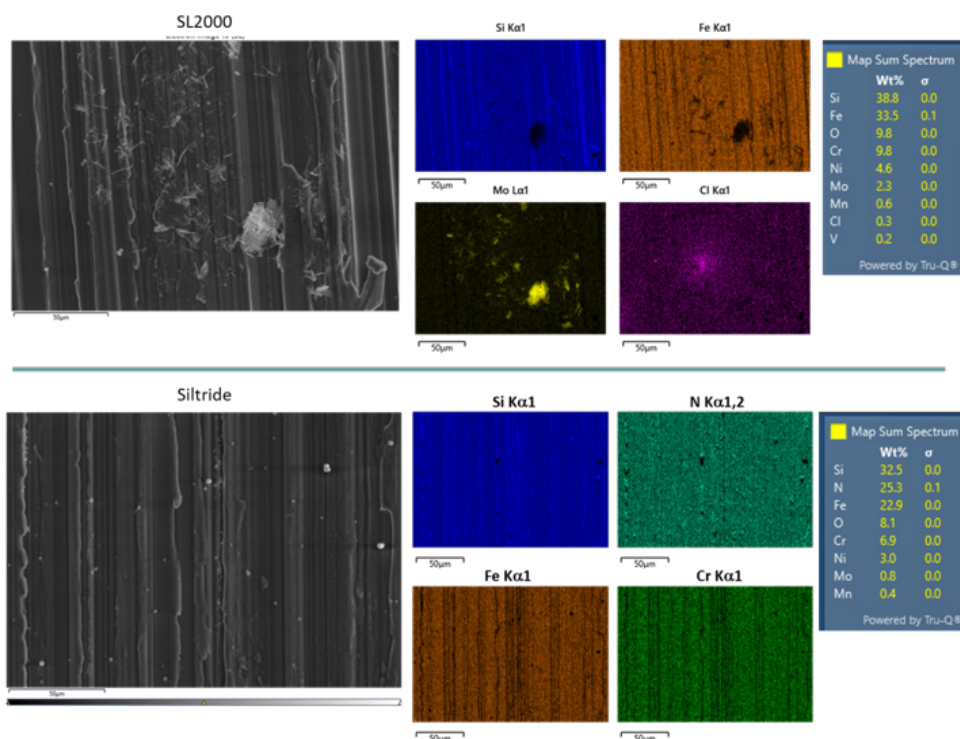


Figure 10: SEM-EDS analysis results of SL2000 (top) and Siltride (bottom) -coated 316L SS samples after one week of 150°C Cl<sub>2</sub> exposure, showing a few localized corrosion sites along with a slight chlorine uptake in the SL2000 coating, and an intact coating with no chlorine uptake in the Siltride coating

The SL2000 coupon appeared mostly intact under the SEM, with only a few corrosion spots identified (Figure 10 top). The corrosion sites had a “hairy” appearance corresponding with local coating loss and the exposed SS substrate surface was depleted in Fe but enriched in Mo. This is similar to the observations on the C22 surface (Figure 8 bottom), showing that the less corrosion-resistant components in the metal were selectively etched away by chlorine whereas the more corrosion-resistant Mo stayed behind. A slight (0.3%) Cl uptake was detected by EDS. The Siltride coupon appeared intact with no corrosion spots identified under the SEM (Figure 10 bottom), and no Cl uptake detected by EDS. The remaining two Siltride coupons were analyzed and similar observations were made.

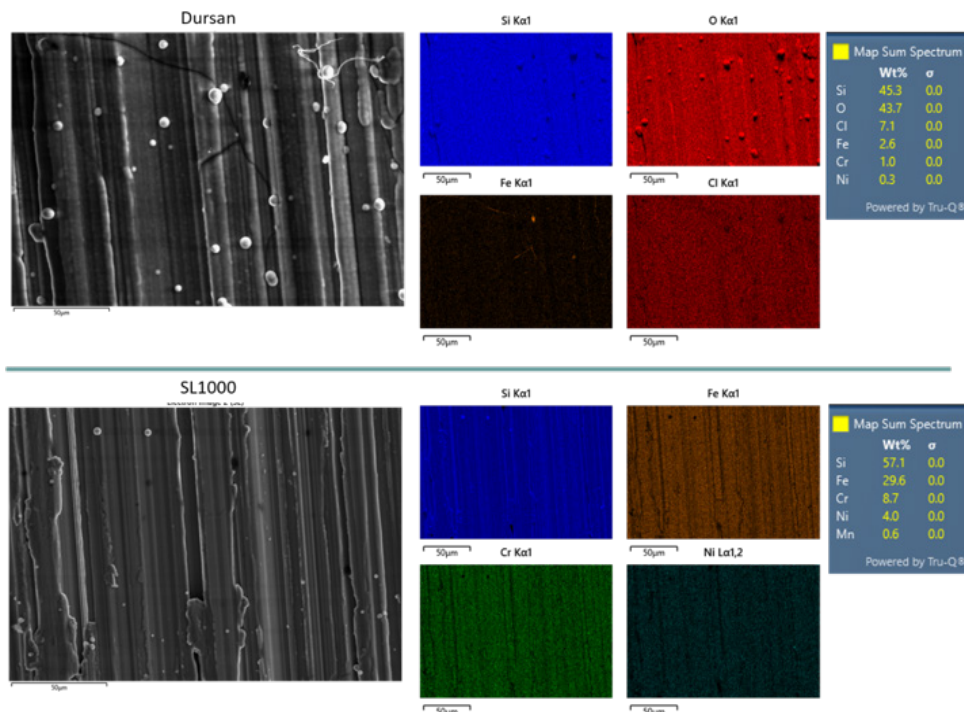


Figure 9: SEM-EDS analysis results of Dursan (top) and SL1000 (bottom) -coated 316L SS samples after one week of 150°C Cl<sub>2</sub> exposure, showing some coating cracks and a high chlorine uptake in the Dursan coating, and an intact surface and no chlorine uptake in the SL1000 coating

Like the room temperature Dursan samples, the 150°C exposed Dursan coupon exhibited micro-cracks under the SEM (Figure 9 top), exposing underneath SS substrate, and the cracks were present on > 50% of the surface area. A high (7.1%) Cl uptake was detected by EDS, roughly doubling the amount detected on the room temperature Dursan coupon. A second Dursan coupon was analyzed and had similar observations. The SL1000 coupon appeared intact under the SEM, and no corrosion spots or chlorine uptake were detected (Figure 9 bottom). A second SL1000 sample was analyzed and similar observations were confirmed.

## Summary

This TI summarizes the results of 100% dry Cl<sub>2</sub> gas corrosion test results of four SilcoTek coatings. The test was carried out by an independent lab, and the samples were exposed for 168 hours at either room temperature or 150°C under a constant Cl<sub>2</sub> flow.

Among all room temperature tested coupons, bare 316L SS showed the most visual corrosion. Bare C22 outperformed bare 316L, but local corrosion sites were observed under SEM. Among the coated coupons, Dursan showed the highest Cl uptake, indicating that chlorine was able to penetrate through the Dursan coating most effectively, creating enough stress at the substrate/coating interface to cause coating cracks on over half of the surface area. SL2000 showed the second highest Cl uptake, but otherwise appeared mostly intact except for a few local corrosion spots under the SEM. SL1000 only showed a slight Cl uptake and appeared mostly intact except for a few local corrosion spots under the SEM. Siltride looked intact with no corrosion spots or Cl uptake detected by SEM-EDS.

Among all 150°C tested coupons, bare 316L SS once again showed the most visual corrosion. Bare C22 outperformed bare 316L, but local corrosion sites were observed under SEM, showing selective etch of Ni and Cr while the more corrosion-resistant Mo was left behind. Among the coated coupons, Dursan showed the highest Cl uptake and corresponding coating cracks on over half of the surface area. SL2000 showed a slight Cl uptake, but otherwise appeared mostly intact except for a few local corrosion spots under the SEM. SL1000 and Siltride both looked intact with no corrosion spots or Cl uptake detected by SEM-EDS.

Under the test conditions outlined in this TI, the performance of the four coatings was ranked as Siltride > SL1000 > SL2000 > Dursan. Room temperature exposure appeared to create more visual corrosion on the testing specimens likely due to a higher probability of moisture presence under the lower temperature testing conditions.



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