

SilcoTek in-house corrosion tests

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Sulfuric acid (H₂SO₄)

• All H₂SO₄ concentrations are volume percentages. A volume to weight percentage conversion for sulfuric acid is listed in the table below.

Vol%	Wt%
9.1	15
15	24.5
25	38
50	64.8
85	91.2

2/3/2016



Summary of H₂SO₄ tests - 1

- Result is highly temperature and concentration-dependent.
- Five concentrations were tested (9, 15, 25, 50 and 85 vol%) for both room temperature 24-hour exposure, and 80°C 7-hour exposure. The 25% solution was also tested for 50°C 7-hour exposure.
- Room temperature 24-hour: 50% H₂SO₄ is the most corrosive concentration to 316L SS. Dursan outperforms SL1000 under all concentrations and should be the coating to recommend.
- Temperature increases reactivity exponentially. At 80°C, the corrosivity increases with concentration, peaks at 25%, then decreases as concentration goes further up.



Summary of H₂SO₄ tests - 2

- <u>Coating is not a solution for permanent exposure to hot sulfuric acid!</u>
- As concentration goes ≥ 50%, the benefit of the coating becomes more significant in hot acid (80°C). The amount of delay in corrosion offered by coating may be valuable to some customers. In general, Dursan outperforms Silcolloy at elevated temperatures too.
- Tests for extended exposures at room temperature still ongoing.

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9% H₂SO₄ room temperature

- No solution color change for either the uncoated or coated coupons after a 24-hr room temperature immersion.
- Uncoated coupon is discolored after exposure; coated coupons show no change.
- Corrosion rates are minimal for all coupons (graph on the right).



9% H₂SO₄ at 80°C



- Uncoated coupon showed severe corrosion after a 7-hour immersion at 80°C.
- Coated coupons were corroded as well at the end of the 7-hour immersion, but at a reduced rate of corrosion.
- <u>Coatings are not a solution for permanent exposure to 9% hot sulfuric acid!</u>



15% H₂SO₄ room temperature

- No solution color change for either the uncoated or coated coupons after a 24-hr room temperature immersion.
- No changes on the coupons after exposure (coated or uncoated).
- Corrosion rates are minimal for all coupons (graph on the right).



15% H₂SO₄ at 80°C



- Coated coupon solution started to change color after about 4-hour immersion.
- Uncoated coupon solution started to change color after about 40-minute immersion.
- All coupons showed severe corrosion after a 7-hour immersion at 80°C.
- <u>Coatings are not a solution for permanent exposure to 15% hot sulfuric acid!</u>



25% H₂SO₄ room temperature

• Observed solution color changes for both uncoated and SL1000-coated coupons (slight for SL1000); but no color change for Dursan-coated coupons after a 24-hour immersion.

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• Dursan provides better protection than SL1000 under this condition.



25% H₂SO₄ at 50°C



- Uncoated coupon started reacting instantly, whereas coated coupons had a delayed reaction, but coating eventually broke down over time.
- All coupons showed severe corrosion after a 7-hour immersion at 50°C.
- <u>Coatings are not a solution for permanent exposure to 25% hot sulfuric acid!</u>



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25% H₂SO₄ at 80°C - 1

- Uncoated coupon started reacting instantly, whereas coated coupons had a delayed reaction, but coating eventually broke down over time.
- All coupons showed severe corrosion after a 7-hour immersion at 80°C.
- <u>Coatings are not a solution for permanent exposure to 25% hot sulfuric acid!</u>



- 20 minutes: uncoated coupon reacted vigorously, giving off hydrogen gases; the SL1000 and Dursan-coated coupons showed very little reaction. The picture also shows Dursan's hydrophobicity repelling water wetting.
- 2 hours: solution very dark in color. Coatings can be seen to start breaking down.

2/3/2016



25% H₂SO₄ at 80°C - 2

- Coating delayed reaction, but eventually broke down over time.
- Once coating breaks down, corrosion proceeds aggressively, and the benefit of coating from the initial hours gets washed off over time.
- All coupons showed severe corrosion after a 7-hour immersion at 80°C.
- <u>Coatings are not a solution for permanent exposure to 25% hot sulfuric acid!</u>



50% H₂SO₄ room temperature

• Solution of uncoated coupon turned green after a 24-hour immersion; solutions of coated coupons showed no color change.

• Both SL1000 and Dursan provide good protection under this condition.



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50% H₂SO₄ at 80°C



- Uncoated coupon showed severe corrosion after a 7-hour immersion.
- Coatings greatly reduced corrosion, and provided significant benefit.
- SL1000 showed a larger variation than Dursan.
- **Dursan provides better protection for 50% hot sulfuric acid under the testing conditions.**



85% H₂SO₄ room temperature

- Silcolek Driving Innovati
- Solution of uncoated coupon turned green after a 24-hour immersion; solutions of coated coupons showed no color change.
- Both SL1000 and Dursan provide good protection under this condition.



85% H₂SO₄ at 80°C



- Uncoated coupon showed severe corrosion after a 7-hour immersion.
- Coatings reduced corrosion by over 10x.
- Dursan provided better protection than Silcolloy under this condition.
- Dursan and Silcolloy both provide benefits and reduce corrosion by 10x.





To be tested

 Extended sulfuric acid exposure at room temperature (2 concentrations – best and worst)



Hydrochloric acid (HCl)

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6M HCl at room temperature

- Solution of uncoated coupon turned green after a 24-hour immersion; solutions of coated coupons showed very slight color changes.
- Both SL1000 and Dursan provide good protection under this condition, with Dursan slightly outperforming Silcolloy.



6M HCl at 50°C

- Silcolek. Driving Innovation
- Uncoated coupon had severe corrosion after a 7-hour immersion at 50°C.
- Coating greatly delayed and reduced corrosion.
- Dursan outperformed Silcolloy in this test.
- Dursan offers great benefits under this condition.



6M HCl at 80°C - 1



- Uncoated coupon started reacting instantly, whereas coated coupons had a delayed reaction, but coating eventually broke down over time.
- All coupons showed severe corrosion after a 7-hour immersion at 80°C.
- <u>Coatings are not a solution for permanent exposure to 6M hot hydrochloric acid!</u>





6M HCl at 80°C - 2

- Coating delayed reaction, but eventually broke down over time.
- Once coating breaks down, corrosion proceeds aggressively, and the benefit of coating from the initial hours gets washed off over time.
- All coupons showed severe corrosion after a 7-hour immersion at 80°C.
- <u>Coatings are not a solution for permanent exposure to 6M hot hydrochloric acid!</u>





Extended HCl exposure at room temperature

2/3/2016



Summary of observations

- Dursan outperformed SL1000 in the extended room temperature HCl corrosion test.
- Despite using a larger volume of acid per coupon in this run, the corrosion rate of uncoated 316L coupon still decreases over time.
- For coated coupons, taking intermittent mass measurements significantly increased the corrosion rate, compared to coupons that were only measured at the end of the exposure. For uncoated coupons, the opposite trend was observed. No explanation yet.



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Effect of intermittent measurements



- The corrosion rates reported here are average rates throughout the whole 8-day exposure period.
- Intermittent measurements to determine corrosion rate vs. time evolution (including sonication rinse – dry steps) have adverse effects on the corrosion rates of the coated coupons. The effect on uncoated coupons is opposite – do not know why.

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Effect of intermittent measurements



 At the end of 8-day exposure upon removal from the acid, the SL1000-coated coupon that did not see intermittent measurements (C) appeared to still have coating on. A closer look revealed severe blisters on the coating and most of the coating fell off upon sonication.



Discussion

- The SL1000 coating tested in this experiment was produced in DGS oven-9. Plan to repeat corrosion test with SL1000 produced in a non-DGS oven (e.g. oven 3).
- Need to discuss with marketing what data to present and how. Once we determine that, future extended exposure tests will all use the same parameters.
- Increasing the volume of acids did not help to maintain high corrosion rates in uncoated coupons (rate still drops over time). The corrosion rate vs. time may not be the best format to present the data. Instead an average corrosion rate comparison over the exposure period (i.e. slide 4) may be a better format.
- Thoughts and other comments?