

Weathering and Corrosion testing of Silcolloy 100 and Dursan

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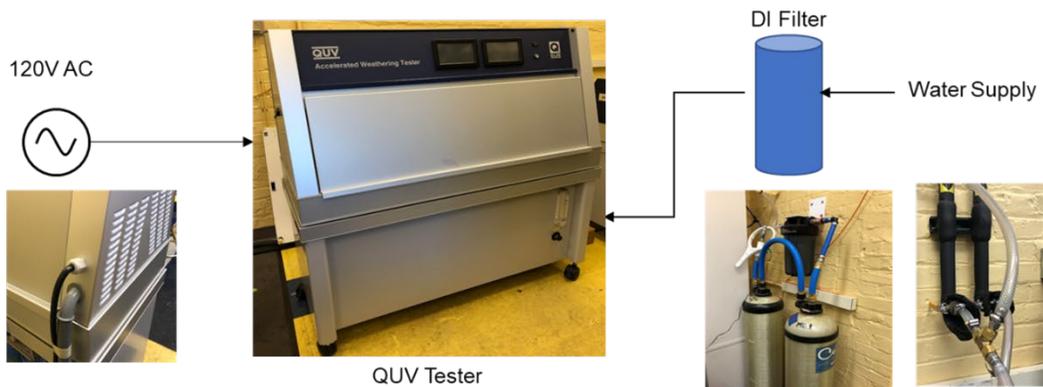
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Weathering Overview

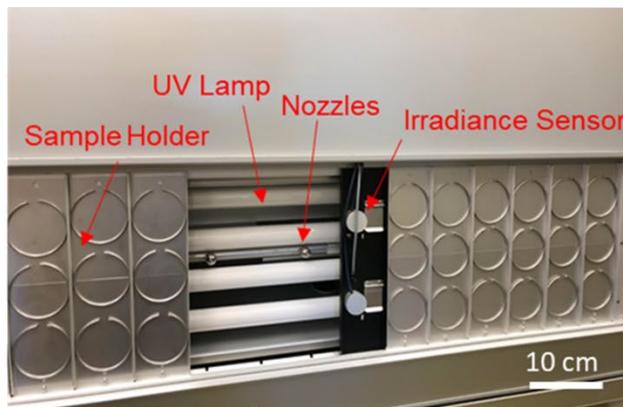
Weathering Tester

The weathering testing was conducted using the QUV Accelerated Weathering Tester (Fig. 1a). Tested coupon-shaped samples were mounted on the sample holders (Fig. 1b, c) using retaining rings and were exposed to harsh weathering conditions of high temperature, UV light, condensation, and water spray. Fluorescent ultraviolet (UV) lamps simulate the natural sunlight and artificial light sources, particularly in the UV region. Rain and dew are simulated by a condensation system and/or a water spray system. Specimen holders form the side walls of the test chamber hold water vapor inside and room air on the back side of the specimens cools them to a few degrees below the vapor temperature causing liquid water to condense on the specimens. The QUV tester can produce effects that might occur over months or years of outdoor exposure in days or weeks.

a



b



c



Figure 1. (a) Picture of the QUV tester setup inside the lab and its connections to utilities. (b) Picture of important components inside the tester. (c) Picture of the sample holder with labeled exposure area

Testing Conditions

We carried out the weathering testing following cycle type 7 exposure conditions of the ASTM G154 standard, the *Standard Practice for Operating Fluorescent Ultraviolet (UV) Lamp Apparatus for Exposure of Materials*. The cyclic test first consists of 8 hours of UV exposure at a temperature of $60 (\pm 3) ^\circ\text{C}$ and at an irradiance of $1.55 \text{ W}/(\text{m}^2\cdot\text{nm})$, which is more than twice the irradiance measured in Phoenix, Arizona at solar noon at the summer solstice with a clear sky. The test is followed by 0.25 hours of water spray at a volumetric flow rate of 7 L/min and then 3.75 hours of condensation at an exposure temperature of $50 (\pm 3) ^\circ\text{C}$. Each test was continued for 750 hours to demonstrate the long-term durability of the tested specimens, and samples were repositioned every 150 hours in accordance with the standard. During the testing, the lab temperature was maintained at $25 (\pm 5) ^\circ\text{C}$.

Inspection

The samples after the weathering testing were inspected through visualization, wettability characterization and X-ray photoelectron spectroscopy (XPS) analysis, and the results were compared with those of untested reference samples. The apparent advancing contact angle of DI water on the samples was measured using micro-goniometry (MCA-3, Kyowa Interface Science Co., LTD).

Results

After 750 hours of weathering, neither sample showed significant visual change (figure 2). The advancing contact angle of SL1000 sample changed from $47 (\pm 1)$ degrees to $53 (\pm 1)$ degrees. And the advancing contact angle of Dursan sample changed from $53 (\pm 2)$ degrees to $49 (\pm 1)$ degrees. This small increase/decrease in wettability could be a result from absorption of volatile organic compounds (VOC) rather than indication of coating degradation. Further analysis of elements present in the coatings through XPS also suggests that coatings did not degrade after the 750-hour weathering testing, as shown in figure 3.

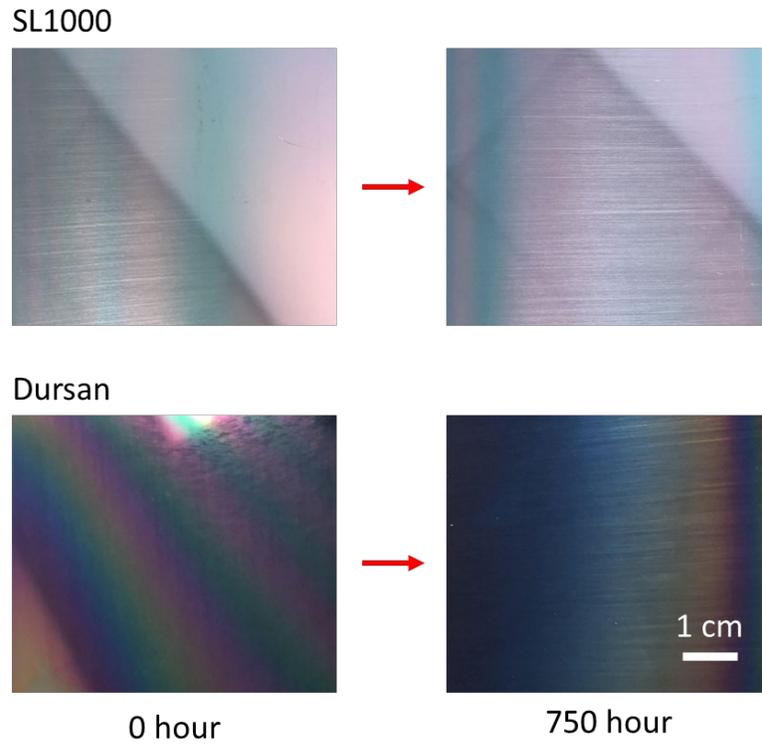
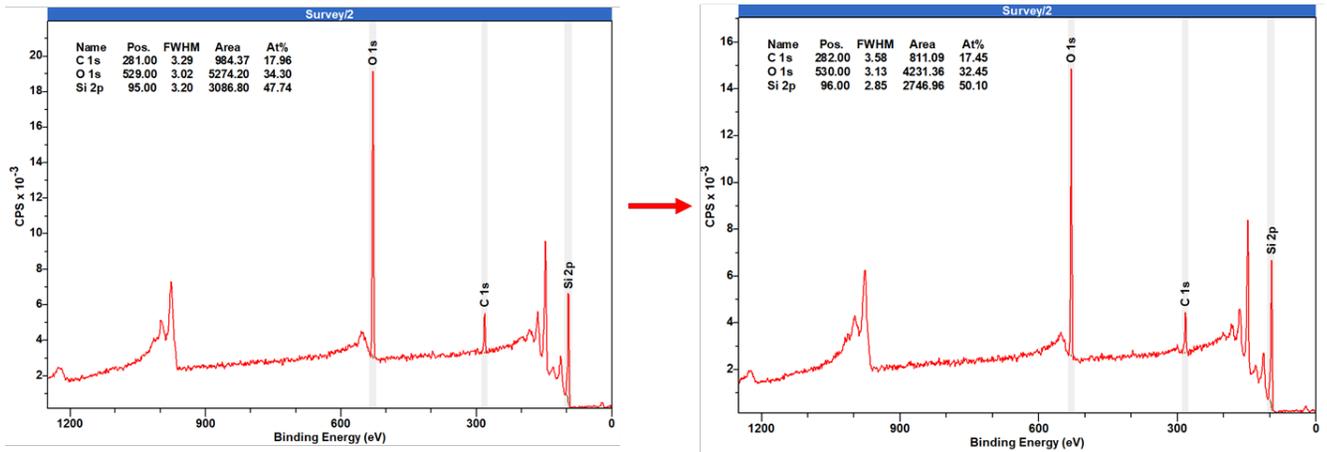
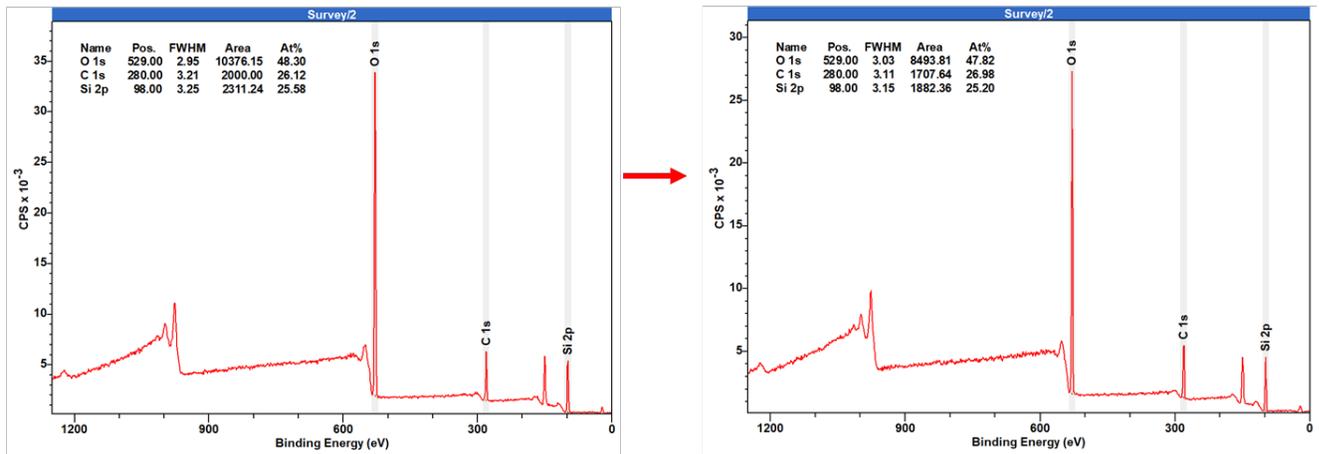


Figure 2. Pictures of visual comparison between tested and fresh SL1000 and Dursan samples

SL1000



Dursan



0 hour

750 hour

Figure 3. XPS results for SL1000 and Dursan samples before and after the weathering testing

Corrosion Testing

Cyclic Corrosion Testing

To investigate the anti-corrosion performance of the PDMS coating, we used the ASTM G85 Annex 3 (A3) corrosion testing, which is also known as salt fog (cyclic) test or Sea Water Acetic Acid Test (SWAAT). To prepare the salt spray solution for the SWAAT test, synthetic seawater was first prepared according to the ASTM D1141-98 standard by dissolving sea salt (SS15-10, Instant Ocean) in de-ionized (DI) water. Then, 10 ml of glacial acetic acid (CAS: #64-19-7, Sigma Aldrich) per liter of solution was added. Finally, an appropriate amount of sodium hydroxide (CAS#: 1310-73-2) was added to adjust the pH between 2.8 and 3.0. Briefly, the test consists of 2-hour cycles consisting of 30 minutes of spray of the acidified seawater solution, followed by incubation in 98% relative humidity at 49°C. The corrosion test was carried out in a Q-fog CRH-600 salt spray chamber shown in Fig. 4(a). Fig 4(b) shows how samples are arranged inside the tester during the test.

Images of the corroded samples over the course of the experiment are shown in Figures 5 and 6 for the 3 × 1 inch and 6 × 3-inch samples.



Figure 4: (a) Q-fog cyclic corrosion chamber. (b) Arrangement of samples inside the testing chamber

While it is difficult to calculate an acceleration factor for the SWAAT test, the SL 1000 and Dursan samples performed very well in the test. We observe that for both coatings, corrosion initiates at the edges and propagates inwards. The edge failure occurs due to coating imperfections near the edges or sharp corners of the substrate. These edge imperfections are introduced by the inward pull (towards the center of the face) during the temperature change after deposition due to surface tension. The thinning of the coating also leads to formation of micro/nano pinholes and defects where corrosion initiates and propagates underneath the coating, leading to coating delamination and failure. It was observed that edge effects were more prominent in the smaller samples (due to a larger perimeter to face area ratio). Overall, the anti-corrosion performance is observed to be very similar for the two coatings.

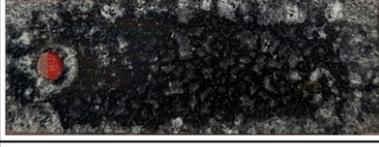
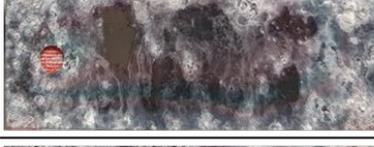
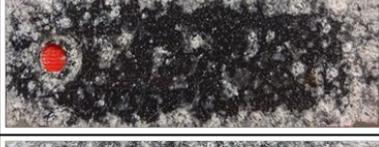
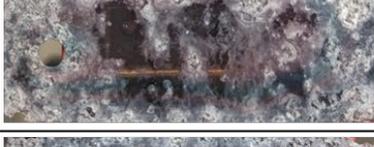
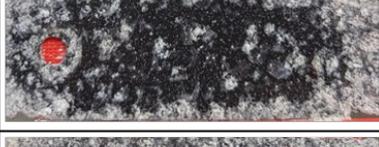
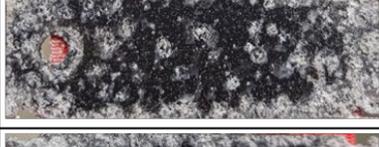
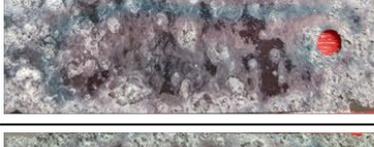
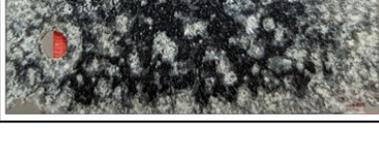
	SL1000 3×1-inch Tab	Dursan 3×1-inch Tab
Day 0		
Day 5		
Day 10		
Day 15		
Day 20		
Day 25		
Day 30		
Day 35		
Day 40		
Day 45		

Figure 5: Images of the corroded 3 × 1-inch coupons over the course of the corrosion test

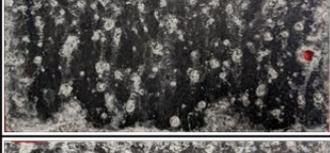
	SL1000 6×3-inch Tab	Dursan 6×3-inch Tab
Day 0		
Day 5		
Day 10		
Day 15		
Day 20		
Day 25		
Day 30		
Day 35		
Day 40		
Day 45		

Figure 6: Images of the corroded 3 × 1-inch coupons over the course of the corrosion test

Flow Fouling

For the flow fouling tests, the coated tubes were immersed in a stainless-steel bath filled with silicon oil (polydimethylsiloxane Oil M 10, Momentive Performance Materials). The silicon oil is heated with an immersion heater (1489 N38 1 kW, McMaster) using a proportional integral differential (PID) controller to maintain the external surface of the test tube at 80 °C. The oil bath heated with an immersion heater was implemented to ensure the total length test tubes are maintained at uniform temperature as the foulant solution passes through it. The flowrate and pressure drop across the test section tube were monitored using a variable area flowmeter (KSK-2050GT1000, Kobold Instruments Inc.) and a differential pressure transducer (PX409-005DDUI, Omega Instruments). The temperature of the foulant solution flowing through the loop is measured at the inlet and the outlet of the test section tube using T-type thermocouples (TMQ316SS-062G-6, Omega Instruments) and the foulant solution flowing out of the tube was cooled down to 25°C using a brazed plate heat exchanger (35115K61, McMaster-Carr) and a 50:50 water:ethylene glycol chiller (6100 series, Polyscience). A schematic of the test facility is shown in Fig 7 (A).

For the experiments, we prepared a 2 L solution of synthetic seawater and added 5g of calcium sulphate (CaSO_4 , CAS# 7778-18-9, Sigma-Aldrich) to it followed by stirring the solution for 15 minutes at 600 rpm. CaSO_4 is chosen as the foulant since its solubility reduces with increasing temperature (~ 2.4 g/L below 80°C). While the synthetic seawater prepared already contains calcium and sulphate ions (present as 1.4 g/L of CaCl_2 and 4.09 g/L of Na_2SO_4 according to the standard), the extra 5g of CaSO_4 added is meant to supersaturate the solution once it enters the tubes heated to 80°C. While only ~ 1.4 g of the additional CaSO_4 added dissolves in the solution, the remaining (undissolved) salt observed at the bottom of the reservoir is meant to replenish the CaSO_4 in solution as fouling occurs over the course of the experiment.

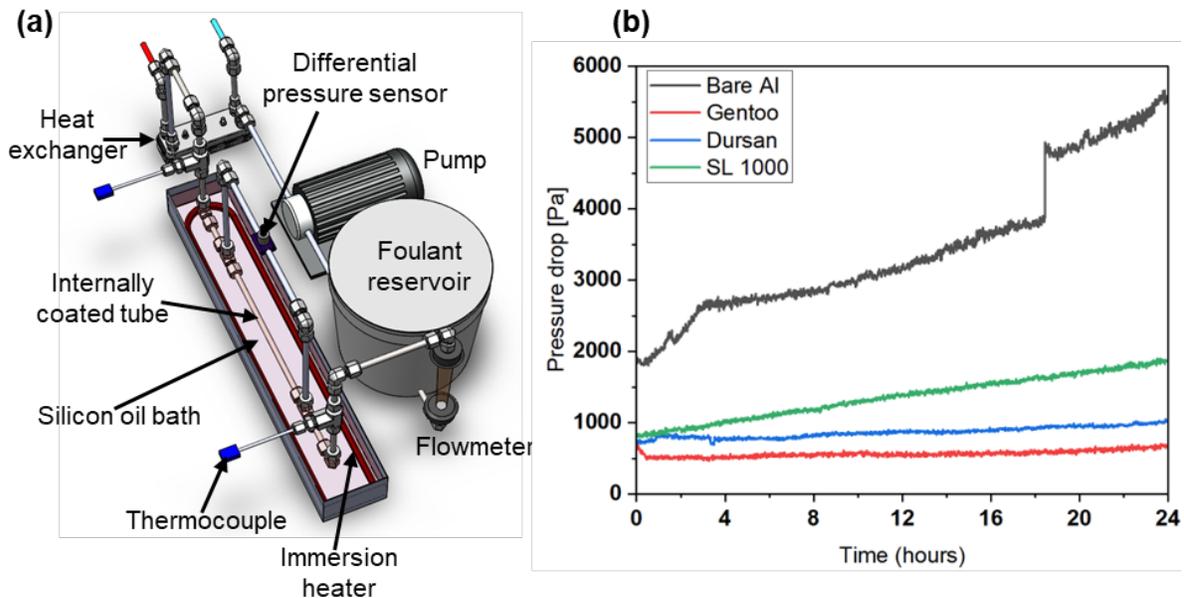


Figure 7: (a) Schematic of the custom-built flow fouling test facility. (b) Plot showing pressure drop across various tubes over time during the flow fouling experiment.

Figure 7(b) shows the pressure drop observed for different tubes over the course of the fouling experiments. The performance of the Silcotek samples is compared with the commercially available coating Gentoo. The initial pressure drop for the bare Al tube is roughly three times that of the coated tubes. This can be attributed to the higher surface roughness of the bare Al surface

in contrast with the smoother coated surfaces. For the bare Al tube, the pressure drop across the tube increased by ~202% after 24 hours of flow fouling test. For SL 1000 and Dursan coatings, the pressure drop across the tubes at the end of the experiment was ~ 60% and ~74% less than the bare Al tube respectively. In comparison, the gentoo coating demonstrated ~ 88% less pressure drop increase than that of bare Al tube.