Low Outgassing of Silicon-Based Coatings on Stainless Steel Surfaces for Vacuum Applications

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Restek Performance Coatings

This presentation was developed through the collaborative efforts of Restek Performance Coatings and Bruce Kendall of Elvac Labs. Restek applied the coatings to vacuum components and Dr. Kendall developed, performed and interpreted the experiments to evaluate the coating performance.

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Objective

Evaluate comparative outgassing properties of vacuum components with and without amorphous silicon coatings

Performance Coatings

Theoretical Basis – Heat Induced Outgassing

- Outgassing rate (F) in monolayers per sec:

$$F = [\exp(-E/RT)] / t'$$

t' = period of oscillation of molecule perp. to surface, ca. 10^{-13} sec
E = energy of desorption (Kcal/g mol)
R = gas constant

source: Roth, A. Vacuum Technology, Elsevier Science Publishers, Amsterdam, 2nd ed., p. 177.

- Slight elevation of sample temperature accelerates outgassing rate exponentially

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The design of the first set of experiments allowed the isolation and direct comparison of outgassing rates with increasing temperature. By applying heat, the outgassing rates are exponentially increased for the purpose of timely data collection. These comparisons with experimental controls will directly illustrate the differences incurred by the applied coatings.

Experimental Design – Heated Samples

- Turbo pump for base pressures to 10^{-8} Torr
  - pumping rate between gauge and pump: 12.5 l/sec (pump alone: 360 l/sec)
  - system vent with dry N_2 between thermal cycles
- Comparative evaluation parts
  - a-silicon coated via CVD (Silcosteel®-UHV); 3D deposition
  - equally treated controls without deposition

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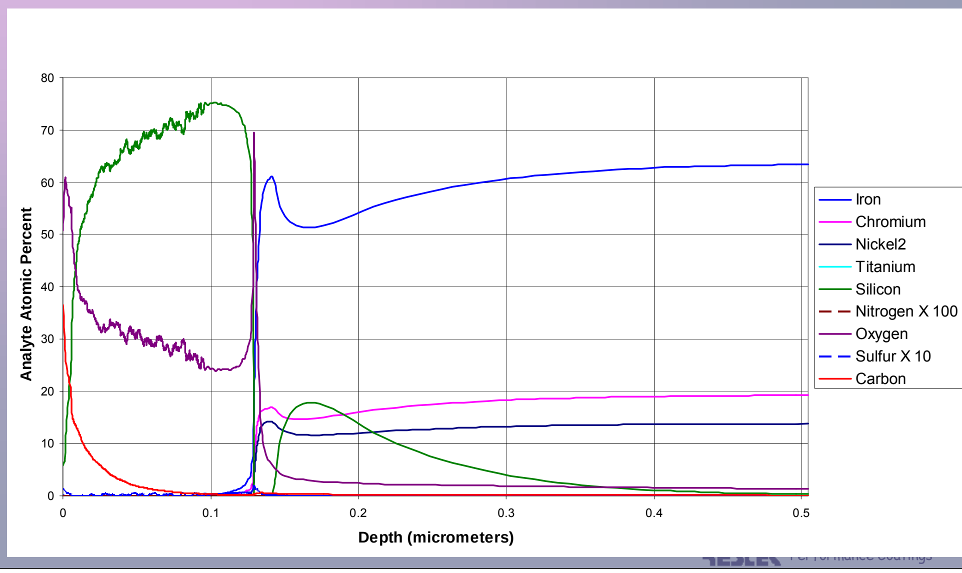
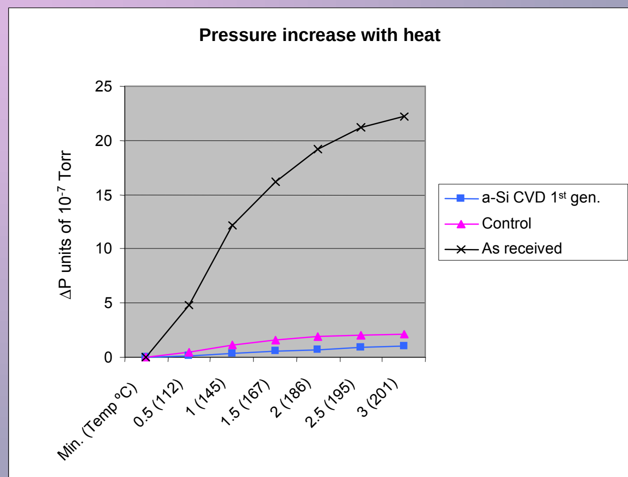
The blue sample (left) was a standard coating commercially available through Restek. However, subsequent improvements in coating technology led to the evaluation of new surfaces (center thimble pointing out of the slide). Note the heating shroud on the sample to the right. It was used in order to heat only the component being measured and prevented heat transfer to neighboring sections of the vacuum system. The only difference between controls and Silcosteel coatings was the coating itself. Both parts were cleaned the exact same way, but the coated parts were exposed to the deposition gases whereas the control parts were instead exposed to inert gas. This allowed for an appropriate experimental design to highlight the performance of the coating itself. System conductance was equivalent to each thimble via a baffle system.

Glow Discharge Surface Analysis of Coating – Bulk Depth Profile



Glow discharge optical emission spectroscopy was used to characterize the silicon deposition layer. Key elements to observe are silicon (coating), oxygen (surface oxides and bulk contamination) and iron, chromium and nickle (bulk stainless steel). Note that after the silicon surface layer up to 0.13 microns, there is a diffusion of silicon in the stainless steel bulk from 0.14 to 0.4 microns at the expense of iron, chromium and nickel. This shows a significant penetration in to the bulk, which highlights the bonding mechanism to be less like a surface adherence and more of a physical diffusion in to the bulk. This helps explain why the coating is flexible with substrate thermal expansion and mechanical bending.

Outgassing Data – Heated Samples



- Turbopump, 1 x 10^{-7} Torr base pressure
- 10hr under vacuum

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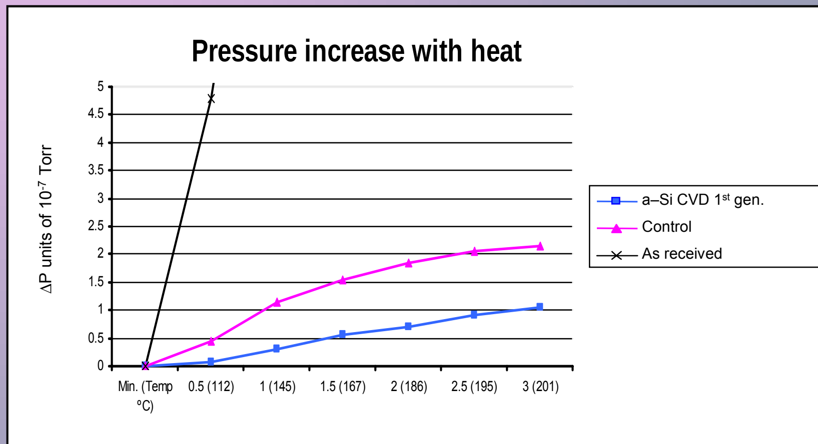
Data output graphs show pressure differences on the y-axis. Therefore, higher increases in pressure indicate higher outgassing rates. The x-axis shows units of time during which pressure readings were performed. Parenthetical values are surface temperatures (in Celsius) at the indicated time.

Note the highly improved performance of both the control and coated parts compared to raw.

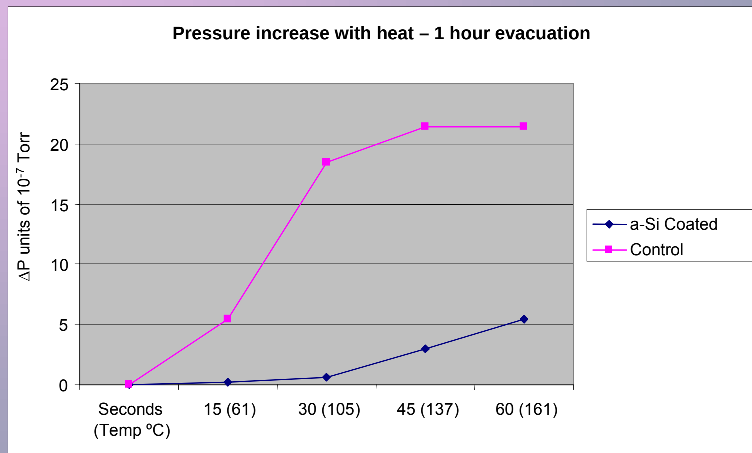
Outgassing Data – Heated Samples



- 7.5 fold improvement at 112ºC
- Subsequent experiments down to 1.2×10^{-10} Torr base pressure (ion pump)
  - 6.4 fold improvement at 61ºC (7.0×10^{-12} Torr ΔP)

To highlight the differences between heat cleaned and coated, the y-axis is expanded. The stock Silcosteel coating maintains a significant outgassing advantage over the control throughout the temperature range. Pressure increases are in units of 10^{-7} Torr. Later experiments applied an ion pump to experiment in to the 10^{-10} Torr range. Even at that level of vacuum, there was a significant outgassing advantage for coated components.

Outgassing Data – Approaching "Real World" Evacuation



- Turbopump, 4.6 x 10^{-7} Torr base pressure
- 1hr under vacuum (ΔP1)

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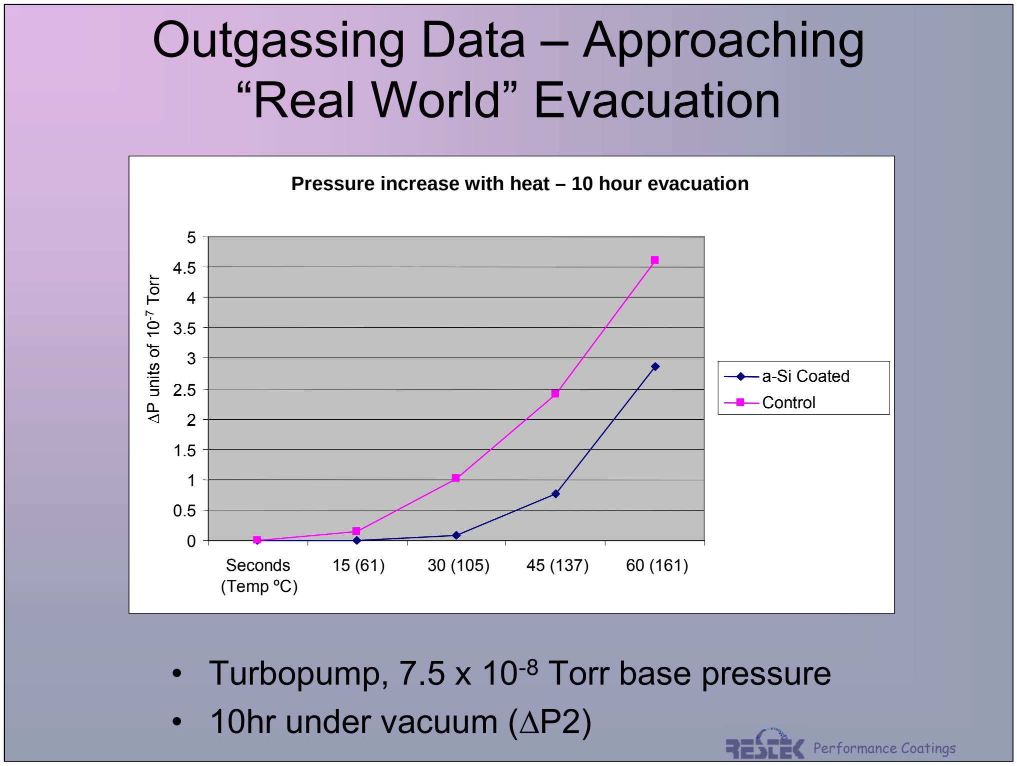
At the request of a vacuum component manufacturer, comparative outgassing measurements were performed after 1hr and 10hrs of pumpdown. This figure illustrates a significant decrease of outgassing rate when comparing the control to Silcosteel-UHV after 1 hour of pumpdown. Note the operating base pressure of 4.6 x 10^{-7} Torr.



At hour 10 of pumpdown on a turbopump system, the Silcosteel-UHV coating still shows a significant improvement over the control. Base pressure is now in to the 10^{-8} Torr range.

Outgassing Calculations

- For the system (P_A), sample area = 125cm^2, conductance = 12.5 l/sec;
  therefore, $\Delta Q = \Delta P(12.5/125) = \Delta P/10$
- At 1 hour, 61ºC:
  ΔQ_1 (control) = 5.4 x 10^{-8} Torr l sec^{-1} cm^{-2};
  ΔQ_1 (a-silicon) = 0.2 x 10^{-8} Torr l sec^{-1} cm^{-2}
  <u>27x improvement</u>
- At 10 hours, 61ºC:
  ΔQ_{10} (control) = 0.14 x 10^{-8} Torr l sec^{-1} cm^{-2};
  ΔQ_{10} (a-silicon) = 0.01 x 10^{-8} Torr l sec^{-1} cm^{-2}
  <u>14x improvement</u>

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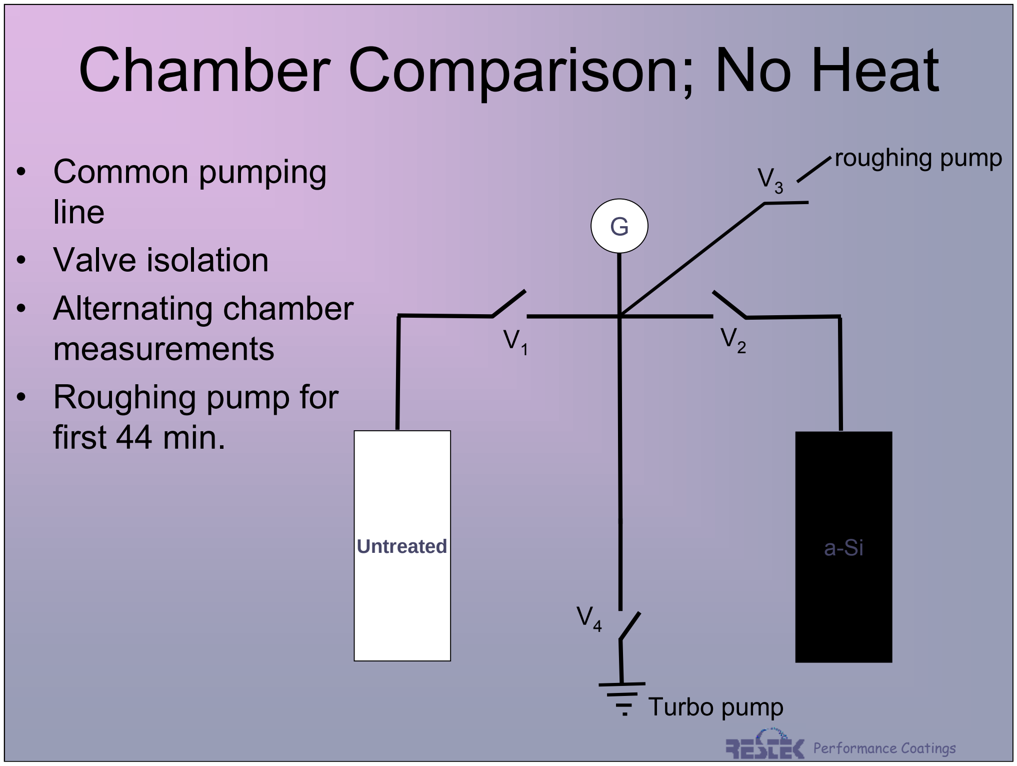
The previous two figures visually compared the variation in outgassing rates for Heat Cleaned and Silcosteel-UHV parts relative to increasing temperature. At the first data point, 61ºC, the figures show a seemingly small difference in outgassing. However, if we compare these results numerically, the differences are impressive. After 1 hour, the Silcosteel-UHV has a 27-fold improvement in outgassing rate (Torr l set^{-1} cm^{-2}) and even after 10 hours under vacuum, the Silcosteel-UHV maintained a 14-fold improvement.



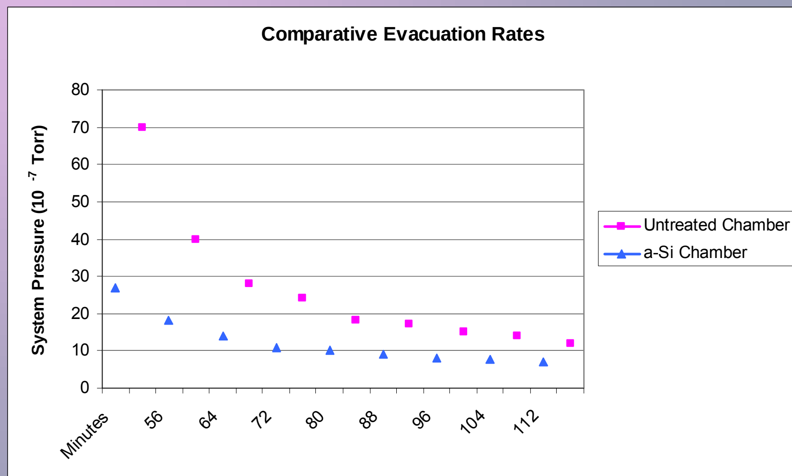
Most recent experimentation has focused on larger chamber outgassing comparisons without the benefit of preheating nor heating during evacuation. The design of comparative chamber pumpdown rates is critical in order to negate possible variables such as pumps, leaks, contaminated components, etc. By using a common evacuation source and valve switching during evacuation, each chamber was alternately isolated for 4-minute periods and each system's pressure was noted.

Chamber Comparisons; No Heat

- System conductance: 7.4 l/sec
- 360 l/sec turbomolecular pump
- Cold cathode gauge

The left side chamber is not coated and used as-is from a component supplier with the exception of removal of some gross particulate contamination. The right side chamber is coated with amorphous silicon deposition. Valves and downstream components are not coated with the exception of a Televac cold cathode gauge visible in the right-hand photo. A common turbo pump (and roughing pump) was used. Leakdown studies showed a relatively insignificant leak on the valve/coated chamber flange connection, which could only give a slight negative bias to the amorphous silicon data.

Chamber Comparisons; No Heat



- Alternate-pumpdown system pressures
- 80-84 minute range: 2.4-fold improvement

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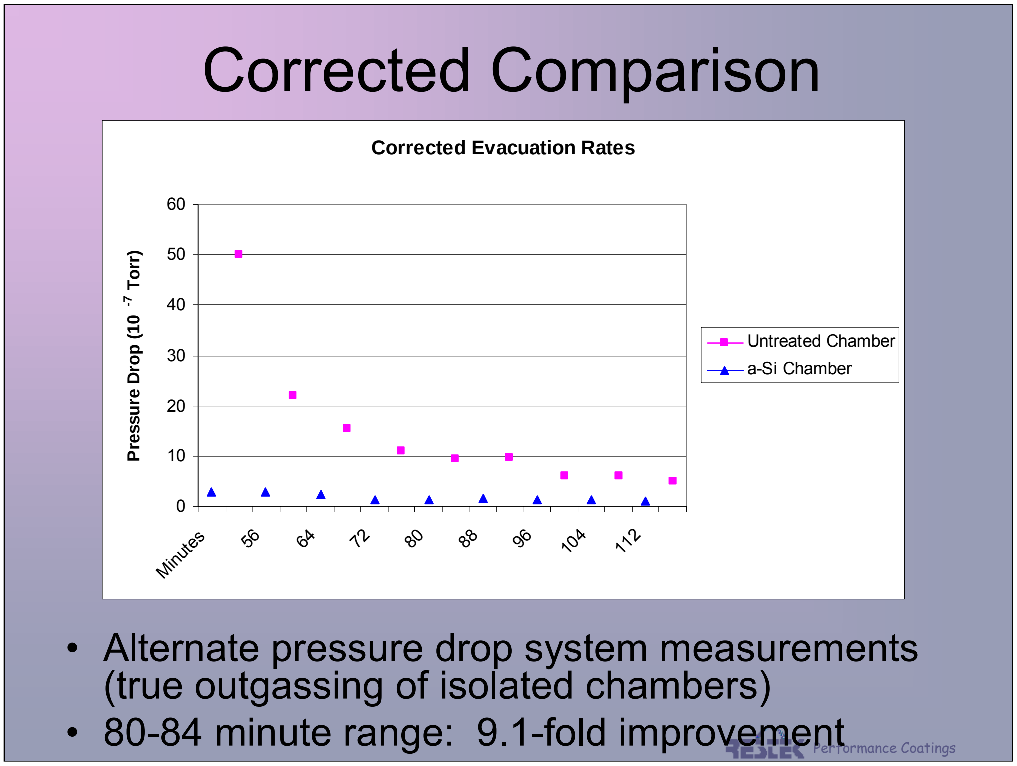
With alternating isolation of the vacuum system every 4 minutes to each chamber (using valves), the pump-down rates of each chamber is shown. This is the raw system pressures without correction. Nevertheless, there is a significant improvement in pumpdown rate and base pressure for the coated chamber (2.4 fold after 82 minutes of evacuation).



The outgassing contribution of uncoated components downstream of the chambers of interest can be used to calculate true pressures of the coated vs. uncoated chambers alone. With this corrected data, an even more dramatic difference is illustrated. At 82 minutes, there is a 9.1-fold improvement for the coated chamber over the uncoated one. Not that even at the initial point of measurement (52 minutes), the coated chamber is already close to the absolute base pressure, whereas the uncoated chamber is not. This data further illustrates that an amorphous silicon-coated chamber can be evacuated much more rapidly and to lower base pressures than standard untreated stainless steel chambers.

Conclusions / Future

- Outgassing rates of vacuum system components can be dramatically reduced with CVD amorphous silicon coating
- Allows for a more rapid evacuation rate to lower base pressures

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Acknowledgements

- Swagelok®
- Televac

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