High Vacuum Applications of Silicon-Based Coatings on Stainless Steel

David A. Smith
Restek Corporation
110 Benner Circle
Bellefonte, PA 16823
www.restekcorp.com

Bruce R.F. Kendall
Elvac Laboratories
100 Rolling Ridge Drive
Bellefonte, PA 16823

Performance Coatings

Objective

Evaluate comparative outgassing properties of vacuum components with various surface treatments

Performance Coatings

Outline

- Experimental design
  - Theoretical basis
  - Systems
  - Test components
  - Surface treatments
  - Experimental evolutions
- Outgassing data and discussion
- Conclusions

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Theoretical Basis

- 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
- Basis for comparison measurements

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Our experimental design allows us to isolate and directly compare 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.

Systems Used

- 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
- Ion pump for base pressures to 10^{-10} Torr
  - pumping rate between gauge and pump: 11.7 l/sec (pump alone: 400 l/sec)
  - system under constant vacuum
- Baffle systems used to ensure identical conductance pathways
  - no line-of-site between samples

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Test Components and Surface Treatments

- 1st Generation "N" samples – 304 SS tube, dual flange
  - Raw, as received
  - Heat cleaned
  - Blue Silcosteel®

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This is the first of a series of samples tested in our experimental evolution. The blue sample (far left) was a standard coating commercially available through Restek. However, subsequent improvements in coating technology led to the evaluation of new surfaces that will be available in the future. Note the heating shroud on the sample to the far right. It too evolved in order to prevent heat transfer to uncoated sections of the vacuum system.

Treatment / Coating Types

- Heat clean
  - Ultrasonic cleaning in aqueous caustic surfactant
  - Heat to 400ºC in inert atmosphere
  - Vacuum at 400ºC
- Silcosteel® coatings
  - Silicon-based
  - CVD process
  - Entire surface coverage
  - Manipulation of process parameters key to coating evolution

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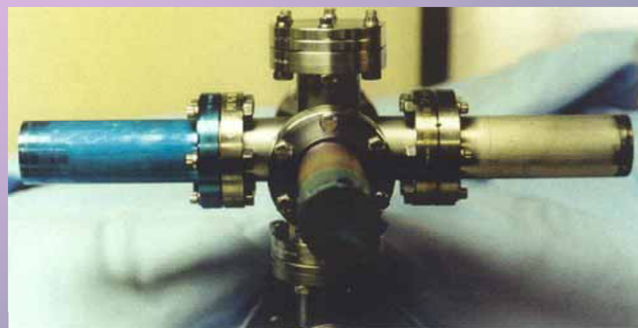
The only difference between heat cleaned and Silcosteel coatings was the coating itself. Both parts were cleaned the exact same way, only the coated parts were exposed to the deposition gases whereas the heat cleaned parts were instead exposed to inert gas. This allowed for an appropriate experimental control to highlight the performance of the coating itself.

Test Components and Surface Treatments (cont.)



- 2nd Generation "P" samples – 4.5" x 1.5" OD 304 SS closed end thimble
  - Heat cleaned
  - Blue Silcosteel®
  - Silcosteel® Beta and Silcosteel®-UHV

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Second generation samples eliminated the terminal Conflat of the first generation pieces. Testing raw, as received samples was also eliminated. The outgassing for raw items was so extreme compared to heat cleaned and coated samples that it was determined that more appropriate comparisons could be highlighted without untreated parts. The first time these parts were tested, a beta version of Silcosteel was used. The second time around, a Silcosteel-UHV coating was used.

Test Components and Surface Treatments (cont.)

- 3rd Generation “Q” samples – miniature metal envelope Bayard-Alpert ion gauge housing (Televac)
  - Heat Cleaned
  - Silcosteel®-beta

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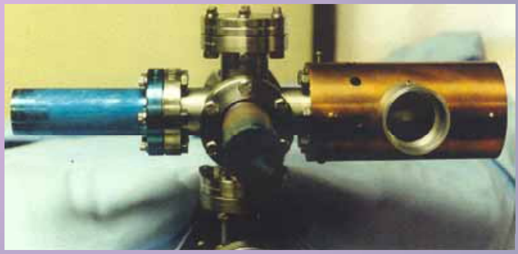
There was also a comparison of Heat Cleaned vs. Silcosteel-UHV coated gauge housings on an Ion pump system. Thanks to Televac for supplying the housings.

Experimental Evolution



- "P" samples with heating shroud
  - Hot air source; monitored with thermocouples
- Version 1 – heated gasket/flange (untreated)
- Version 2 – isolated gasket/flange w/ insulation
- Version 3 – Version 2 + cool air circulated on untreated gasket/flange

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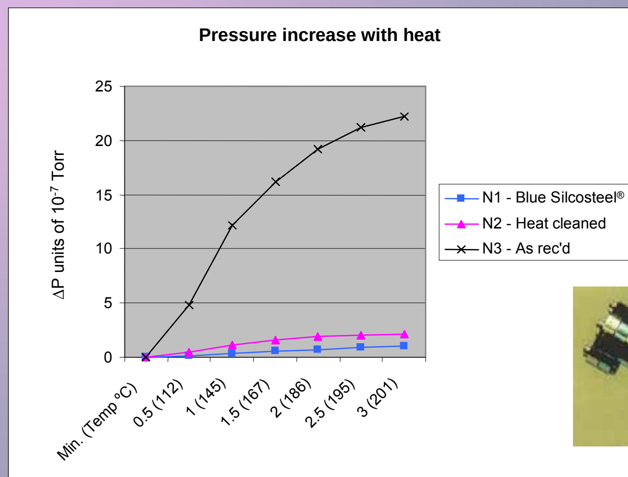
Heating shrouds evolved to prevent heating of non-treated areas and give more accurate outgassing data from only the coated surfaces.

Outgassing Data – N Samples



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

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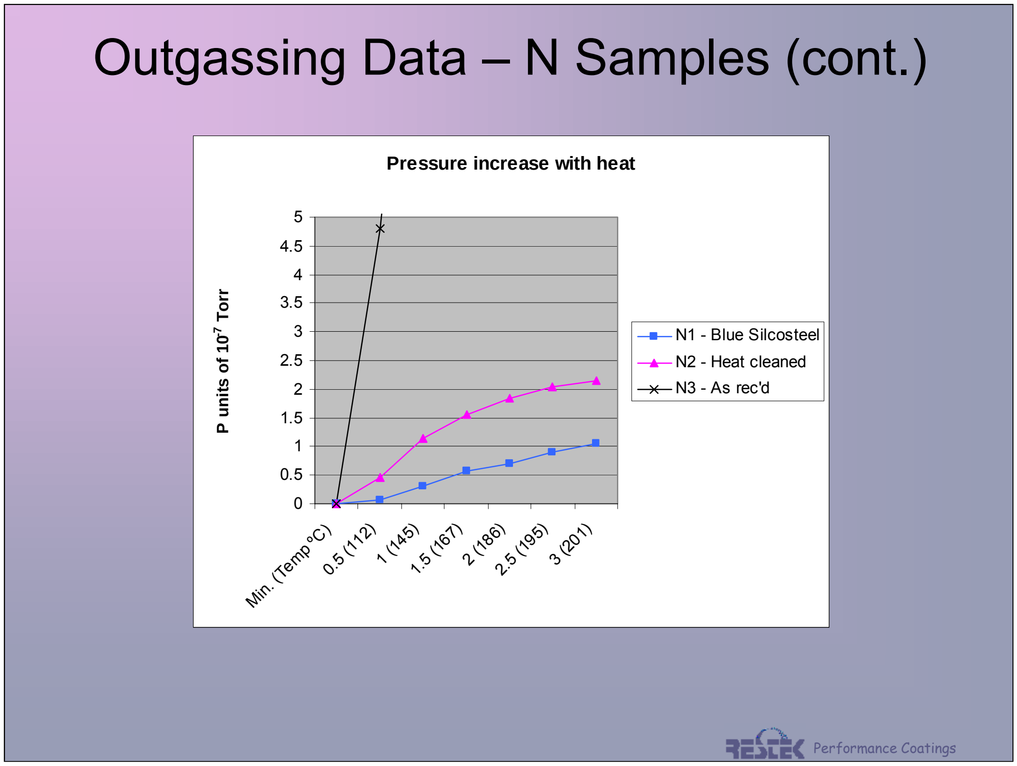
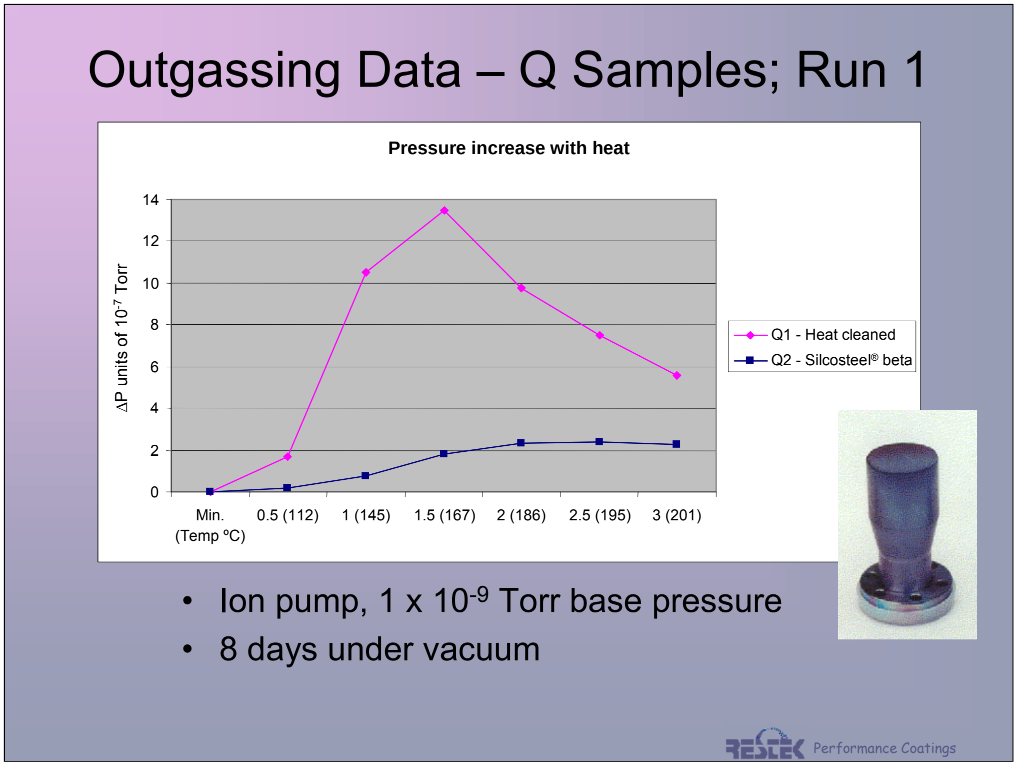
Data output graphs show pressure differences on the y-axis (base pressures are indicated in text at bottom of slide). Therefore, higher increases in pressure indicate higher outgassing rates. The x-axis shows units of time during which pressure readings were performed. Parenthetical values are surface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

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

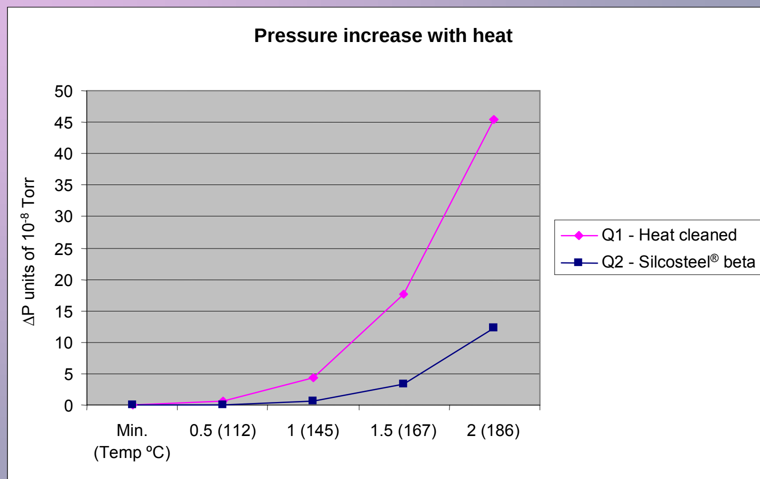


To highlight the differences between heat cleaned and coated, the y-axis is expanded. The stock Silcosteel coating maintains a significant outgassing advantage over the heat cleaning throughout the temperature range. Pressure increases are in units of 10^{-7} Torr.



The 3rd iteration of samples were the first to use an ion pump system. With an initial base pressure in the 10^{-9} Torr range, the Silcosteel beta component exhibits a significantly lower outgassing rate throughout the temperature increase.

Outgassing Data – Q Samples; Run 2



- Ion pump, 1 x 10^{-9} Torr base pressure
- 41 days under vacuum

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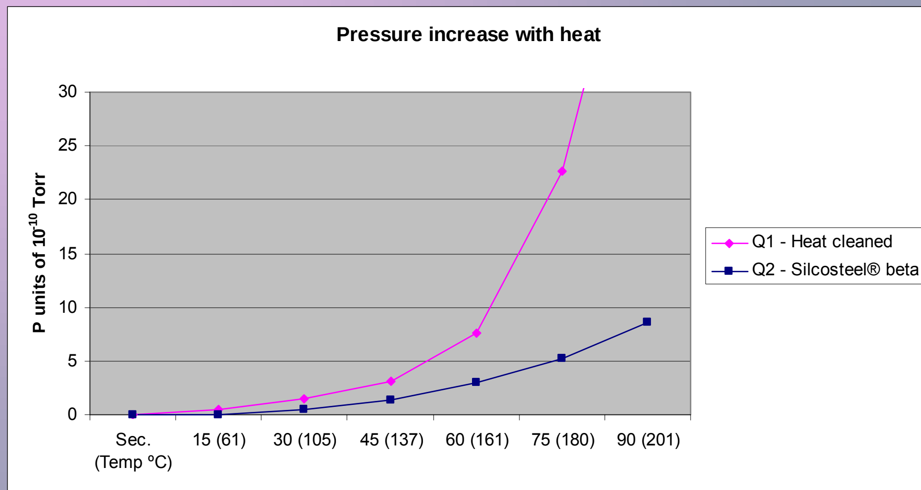
The improvement for Silcosteel beta continues. Note the degree of pressure increase is now in the low 10^{-9} Torr range (y-axis).

Outgassing Data – Q Samples; Run 3



- Ion pump, 1.2 x 10^{-10} Torr base pressure
- 156 days under vacuum (5^{th} baking cycle)

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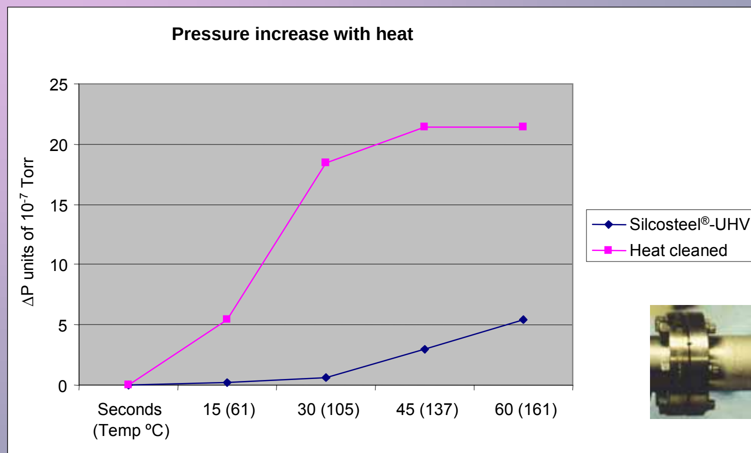
After 156 days under vacuum, the base pressure and pressure increases are down to the low 10^{-10} Torr range. Even at its lowest temperature reading (61ºC), the Silcosteel beta shows a pressure increase of 0.07 x 10^{-10} Torr, whereas the Heat Cleaned increased 0.45 x 10^{-10} Torr. This is a 6.4-fold improvement for the coated system.

Outgassing Data – P_A Samples; Run 1



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

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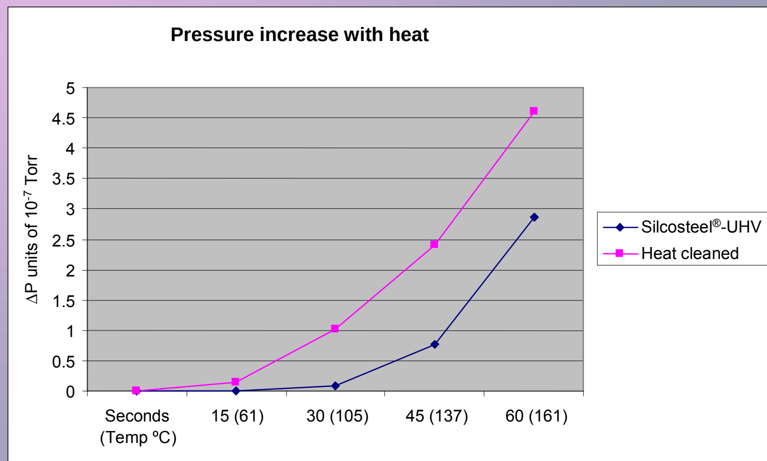
The most recent advance in Restek's coating technology is called Silcosteel-UHV. This surface was applied to the Generation 2 pieces for evaluation under a typical turbopump startup process. The treated parts were tested for outgassing rates after 1hr and 10hrs of pumpdown. This figure illustrates a significant decrease of outgassing rate when comparing the heat cleaned part to Silcosteel-UHV. Note the operating base pressure of 4.6 x 10^{-7} Torr.

Outgassing Data – P_A Samples; Run 2



- Turbopump, 7.5 x 10^{-8} Torr base pressure
- 10hr under vacuum (ΔP2)

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

Outgassing increase sample 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 (heat cleaned) = 5.4 x 10^{-8} Torr l sec^{-1} cm^{-2};
  ΔQ_1 (Silcosteel®-UHV) = 0.2 x 10^{-8} Torr l sec^{-1} cm^{-2}
  27x improvement
- At 10 hours, 61ºC:
  ΔQ_{10} (heat cleaned) = 0.14 x 10^{-8} Torr l sec^{-1} cm^{-2};
  ΔQ_{10} (Silcosteel®-UHV) = 0.01 x 10^{-8} Torr l sec^{-1} cm^{-2}
  14x improvement

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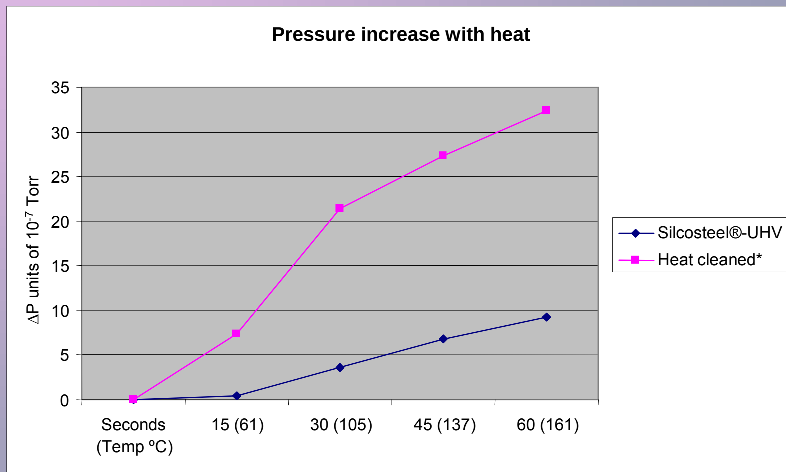
The previous two figures visually compared the variation in outgassing rates for Heat Cleaned and Silcosteel-UHV parts relative to increasing temperature. At the first data point, 61ºC, the figures show a seemingly small difference in outgassing. However, if we compare these results numerically, the 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.

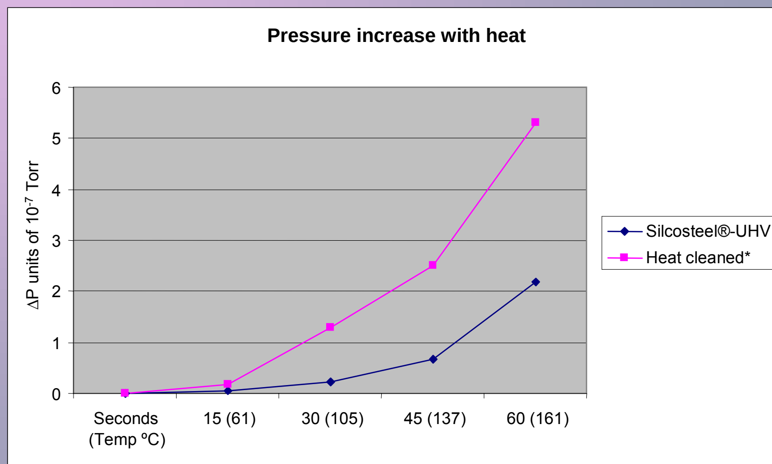
After air/humidity exposure



- 70ºF, 58% relative humidity, 114 days
- Turbopump, 2 x 10^{-7} Torr base pressure
- 1hr under vacuum (ΔP1); 18.5 fold improvement

Performance Coatings

After air/humidity exposure



- Turbopump, 8 x 10^{-8} Torr base pressure
- 10hr under vacuum (ΔP2); 4.5 fold improvement

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Preliminary Electronic Performance Data

- Resistivity perpendicular to film range 12-60 Ω/cm^2
- Pinholes allow conductivity through film to substrate

- Practical applications
  – Comparison of coated vs. uncoated miniature cold cathode gauge housings showed no difference in performance
    - Current density (est.) = 10^{-6} amp/cm^2
      resulting from secondary electron emission from ion impact in gauge
  – Comparison of vacuum tubes with alternative anodes of uncoated vs. coated steel showed no detectable performance difference
    - Internal filament produced 1ma electron current
    - Anode voltage = 200 V
    - Anode area = 23.5 cm^2
    - Anode current density approx. 4 x 10^{-5} amp/cm^2

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Initial electronic performance data indicates that although the Silcosteel layer itself is not conductive along a parallel axis, there are microscopic pinholes that allow the penetration of charge through the thin film and then conductivity along the steel substrate. Measurements on coated pieces have confirmed this phenomenon.

Conclusions / Future

- Studied outgassing properties of stainless steel parts coated with evolving silicon-based coatings
- Comparative outgassing rate improved by > order of magnitude
- Electronic, galling, H_2 permeability properties
- Evaluate completely coated system

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The coatings studied have illustrated a decrease in cleaned Stainless Steel outgassing greater than an order of magnitude. Initial studies in electronic and galling characteristics have been favorable and are ongoing. Future evaluation plans include hydrogen permeability and the performance of a completely coated system.

Acknowledgements

- Televac

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Our gratitude to Televac for supplying gauge housings for this study.