High Vacuum Applications of Silicon-Based Coatings on Stainless Steel

Gary A. Barone, David A. Smith, Marty Higgins:
Restek Corporation, Bellefonte, PA

Bruce R.F. Kendall:
Elvac Associates, Bellefonte, PA

Introduction

Ultra-High Vacuum (UHV) environments of 10^{-7} Torr or lower and high vacuum environment of 10^{-5} Torr or lower are critical for many instruments and semi-conductor manufacturing processes. Under these vacuum conditions, steel and aluminum components outgas large quantities of water, CO_2, CO, and other contaminant molecules. Large pumping systems and extensive bake-out treatments are required to remove these materials in order to attain and maintain low vacuum environments. This research investigates the use of an amorphous silicon coating to reduce outgassing of components in high vacuum and UHV environments.

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.

- 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 directly illustrate the difference incurred by the applied coatings

System

- 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

System

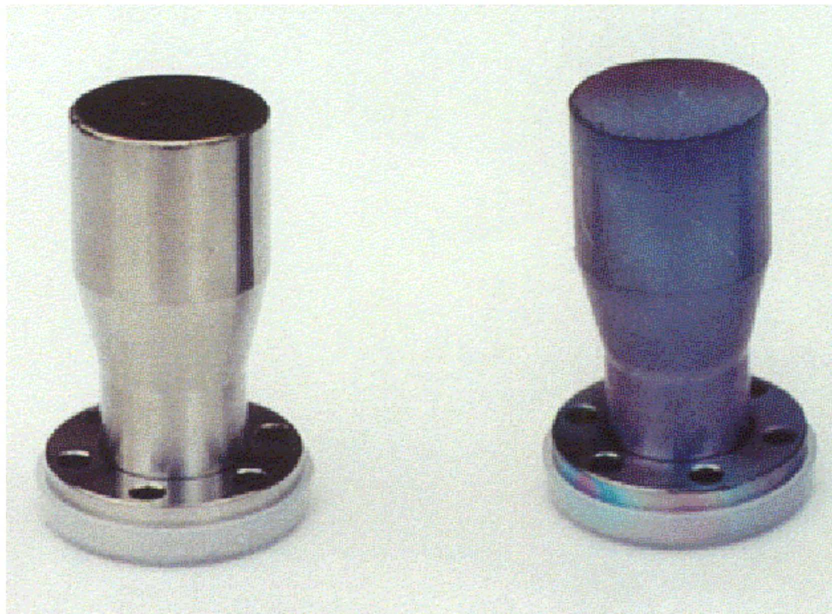
- Cleaning of all components as follows:
  - Ultrasonic cleaning of all components in aqueous caustic surfactant.
  - Heat to 400ºC in inert atmosphere
  - Vacuum at 400ºC
- Silcosteel® coatings (amorphous silicon) applied to components, after previous cleaning process, to reduce outgassing. Coating process:
  - CVD process
  - Entire surface coverage
  - Manipulation of coating process parameters reduce outgassing
- Figure 1 is a photograph of a non-coated and coated component used in one of the studies.

Experimental

Both the “HEAT CLEANED” only and Silcosteel®-UHV treated components were attached to an ion pump system. The systems was then pumped down to an initial base pressure. Several variants of coating were studied to understand the impact of coating composition and depth.

The “HEAT CLEANED” only and Silcosteel®-UHV treated components were individually heated, and the resultant pressure increase was a measured effect of materials outgassing from the substrate into the vacuum environment

Figure 1



Results and Discussion

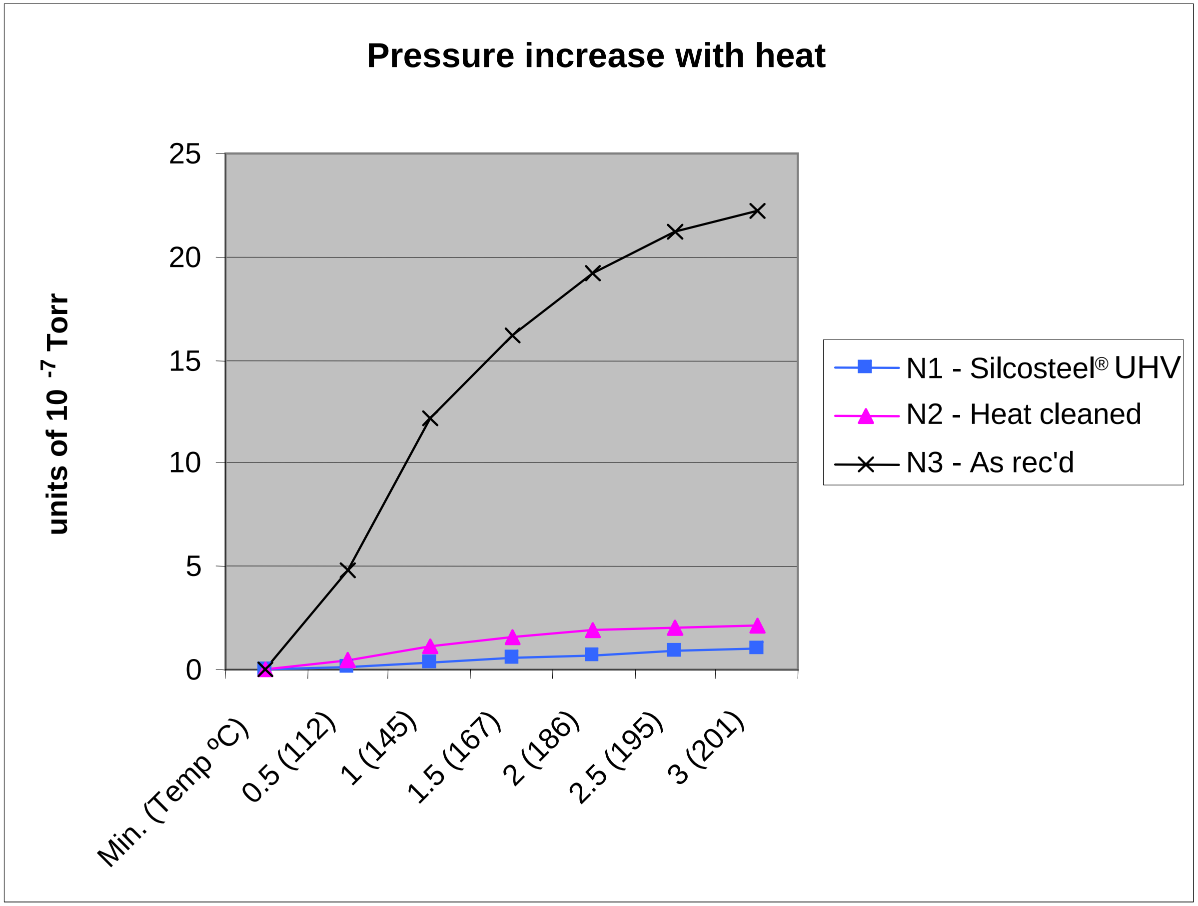
INITIAL PUMPDOWN RESULTS (10^{-7} Torr)

Figure 2 charts the pressure increase vs. time/temperature as a result of heating the vacuum components. Base pressure was 10^{-7} Torr and the system was under vacuum for 10 hours.

The vacuum components that were used as received from the manufacturer demonstrated a large amount of outgassing causing a large pressure increase. It was impractical to study these components at any lower levels of base pressure due to immense outgassing.

The heat treated and coated components both exhibited little pressure increase in the 10^{-7} environment with heating

Figure 2



Pressure increase with heat
units of 10 -7 Torr
25
20
15
10
5
0
Min. (Temp oC)
0.5 (112)
1 (145)
1.5 (167)
2 (186)
2.5 (195)
3 (201)
N1 - Silcosteel® UHV
N2 - Heat cleaned
N3 - As rec'd

Results and Discussion

EXTENDED VACUUM STUDY (10^{-9} Torr)

Figures 3,4 and 5 demonstrate pressure increase of vacuum components exposed to heating after extended time under pumpdown

Figure 3, data after 8 days of pumpdown

Figure 4, data after 41 days of pumpdown

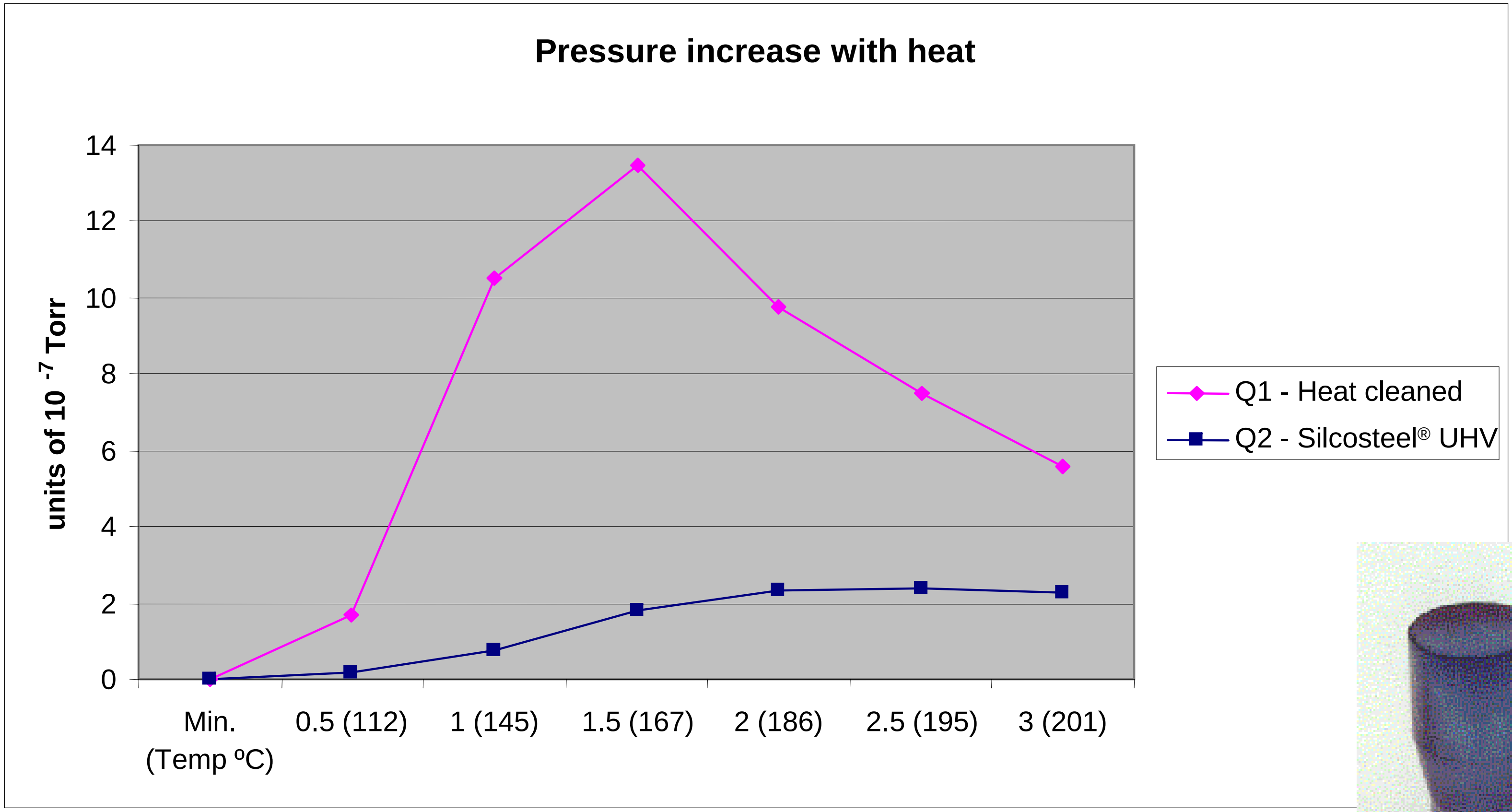
Figure 5, data after 156 days of pumpdown

Ion pump used with base pressure of 1 x 10^{-9} Torr

Amorphous silicon components continued to experience a lesser degree of outgassing material even after long periods of time under vacuum.

NOTE: Magnitude of Y-axis in each figure diminishing as better vacuum is achieved in the test system and overall outgassing of components decreases

Figure 3



Pressure increase with heat
units of 10 -7 Torr
14
12
10
8
6
4
2
0
Min. (Temp ºC)
0.5 (112)
1 (145)
1.5 (167)
2 (186)
2.5 (195)
3 (201)
Q1 - Heat cleaned
Q2 - Silcosteel® UHV

Figure 4

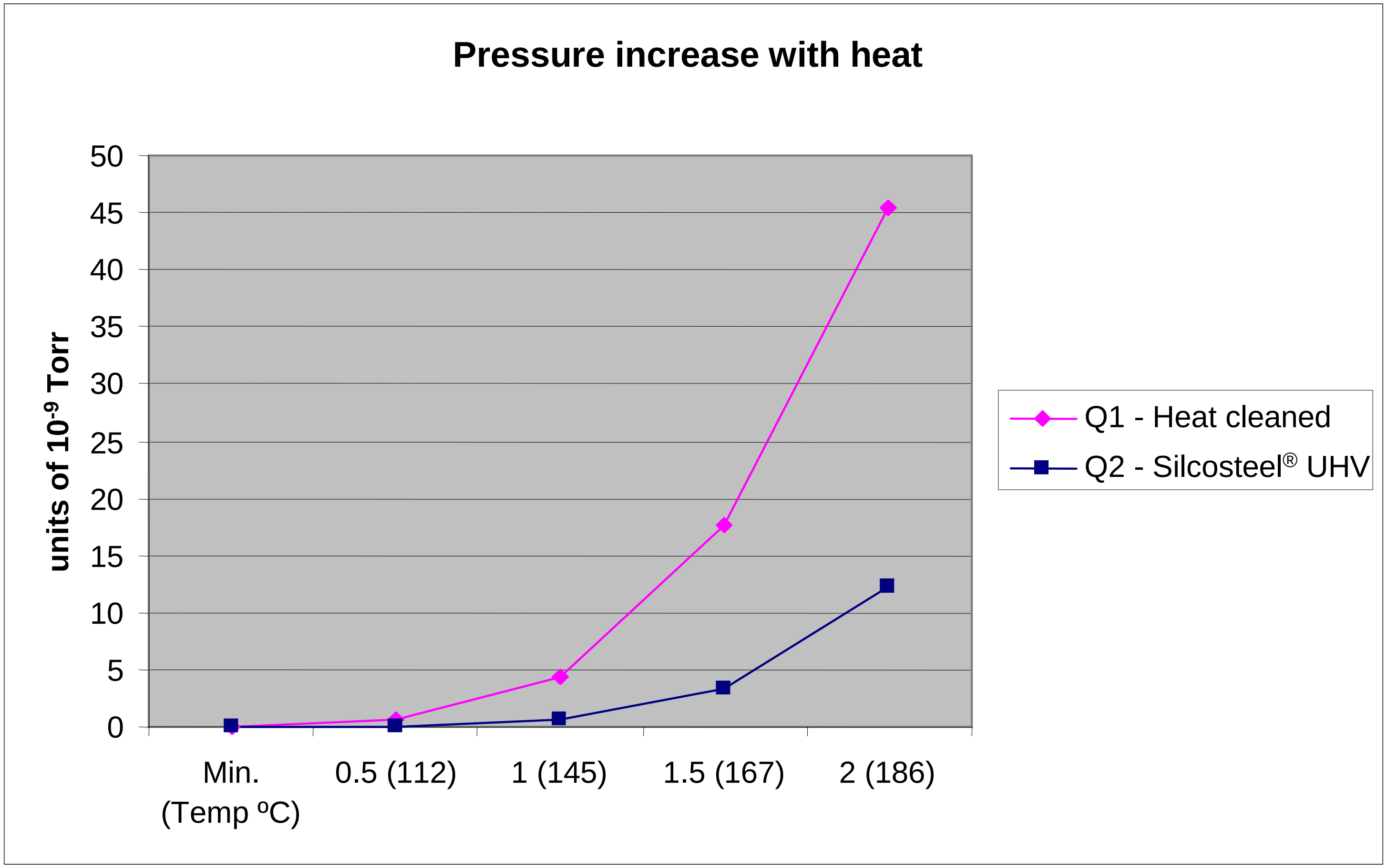
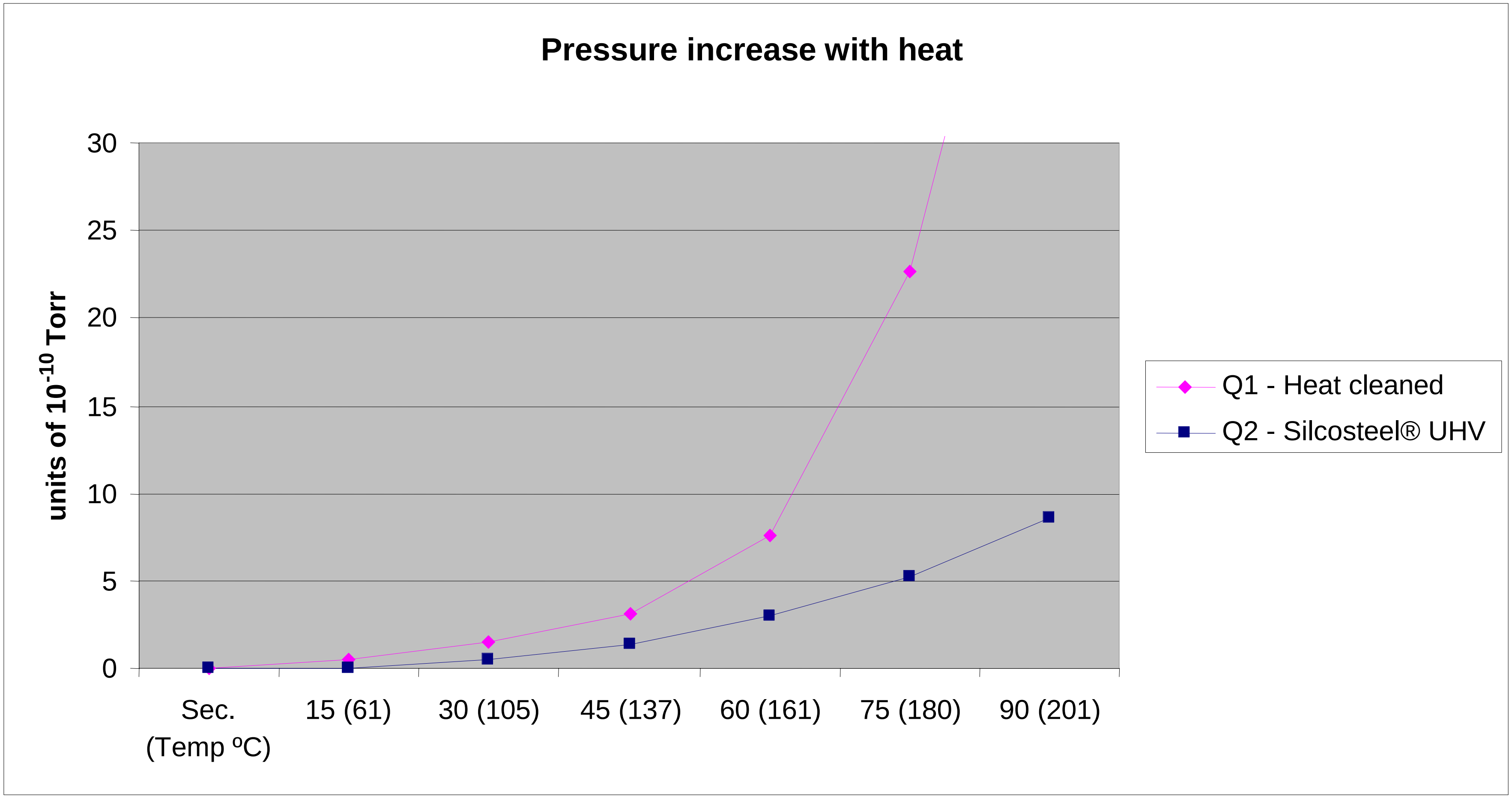

Pressure increase with heat
units of 10^{-9} Torr
50
45
40
35
30
25
20
15
10
5
0
Min.
(Temp ºC)
0.5 (112)
1 (145)
1.5 (167)
2 (186)
Q1 - Heat cleaned
Q2 - Silcosteel® UHV

Figure 5


Pressure increase with heat
units of 10^{-10} Torr
30
25
20
15
10
5
0
Sec.
(Temp ºC)
15 (61)
30 (105)
45 (137)
60 (161)
75 (180)
90 (201)
Q1 - Heat cleaned
Q2 - Silcosteel® UHV

Conclusions

This studied has illustrated the need for pre-treatment of vacuum components prior to installation and use. Vacuum components as received from the manufacture outgas greatly. As such, use in high vacuum and UHV vacuum environments is impractical.

The use of a caustic cleaning system and aggressive heating in an inert/vacuum condition reduces outgassing of new vacuum components by orders of magnitude.

This study also demonstrated the advantage of using an amorphous silicon coating (Silcosteel-UHV) to reduce outgassing of vacuum components.

The coatings act as a barrier, trapping volatile materials such as carbon monoxide, carbon dioxide and water. As such, these volatile materials are kept from entering the vacuum environment

Acknowledgements

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