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Material Outgassing Kinetics: The Development of a Testing Capability

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Contamination due to outgassing of materials can cause the degradation of critical hardware of a spacecraft. Using outgassing rates, kinetic expressions can be developed and used in models to predict the evolution of molecules and migration of contaminants for specific materials. These models could be used in the selection process of materials to help mitigate the amount of contamination of mission critical hardware for the expected life of the spacecraft. By using the ASTM E1559 test standard this can be achieved. This standard uses the quartz crystal microbalance (QCM) collection approach. A temperature-controlled effusion cell, containing the sample material, impinges outgassing flux onto three QCMs. One QCM is held at LN2 level temperature to measure the total mass loss (TML) as a function of time. The other two QCMs are controlled to selected temperature values to measure the volatile condensable material (VCM) as a function of time.

Using mostly existing vacuum hardware, a design for the ASTM E1559 and stand were created using solid edge. All pieces were spec'd to create a stand for the test chamber itself and a data rack for all the supporting hardware. This includes the design and wiring of an electrical control box and assembling all of the hardware.

Experiment date: 9/1/2019 – Present

Report submitted:

Introduction

Materials must be well understood and well characterized to be allowed in most space applications. Testing requirements for materials to go to the International Space Station or to fly on unmanned spacecraft are rigorous. SDL needed to develop the testing capability to understand these materials better. The American Society for Testing and Materials (ASTM) E1559 standard is used to determine the outgassing kinetics of materials and the corresponding rates of contaminant deposition on nearby surfaces at various temperatures. [1] The objective is to upgrade existing hardware to develop a state-of-the-art ASTM E1559 compliant test chamber. This will allow SDL to evaluate and space-qualify new aerospace materials to be used in critical on-orbit applications.

The ASTM E1559 standard specifies there must be four main components for the test chamber (Fig. 1).

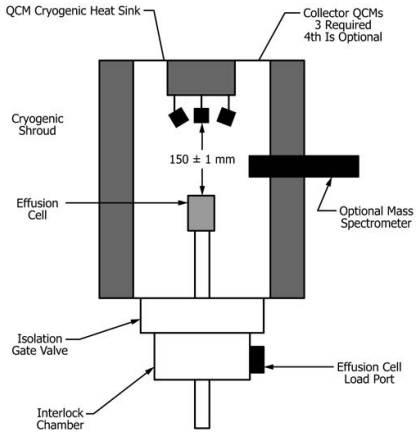


Fig. 1 ASTM E1559 Standard basic internal configuration. [3]

- Vacuum chamber – A outgassing test chamber with a cryogenic shroud connected to a sample introduction "load lock" chamber. Each chamber is independently pumped using a turbo pump backed by an oil-free scroll pump.
- Internal configuration – Three quartz crystal microbalances (QCMs), an effusion cell, a cryogenic heatsink, and a Residual Gas Analyzer (RGA).
    - QCM – Measures material deposition rates by monitoring the frequency change of a quartz crystal oscillator as mass is accumulated. Deposition rates can be measured at various temperatures (Fig. 2).
    - RGA – Quadruple mass spectrometer used to identify chemical composition of outgassing vapors. It is used to identify QCM contaminants by warming the crystal to re-evaporate accumulated deposits (Fig. 3).
- Data acquisition – Used to measure frequencies of QCMs, temperature of QCMs, temperature of effusion cell, and times of data collection at specified intervals. Data is stored for later retrieval for further analysis

- Temperature control system – Able to maintain temperatures of effusion cell and QCMs independently. QCMs have their own internal heaters and are cold-biased by being mounted on the cryogenic heatsink. [2]

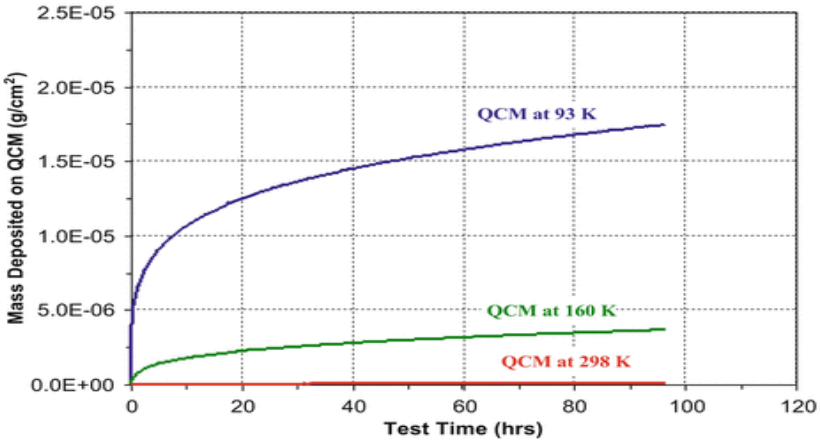


Fig. 2 QCM Deposition Rate vs Time & Crystal Temp. As the test progresses there is less material outgassing from the sample, causing the QCM to collect at a slower rate. Colder temperatures cause more material to collect on QCM surface. [5]

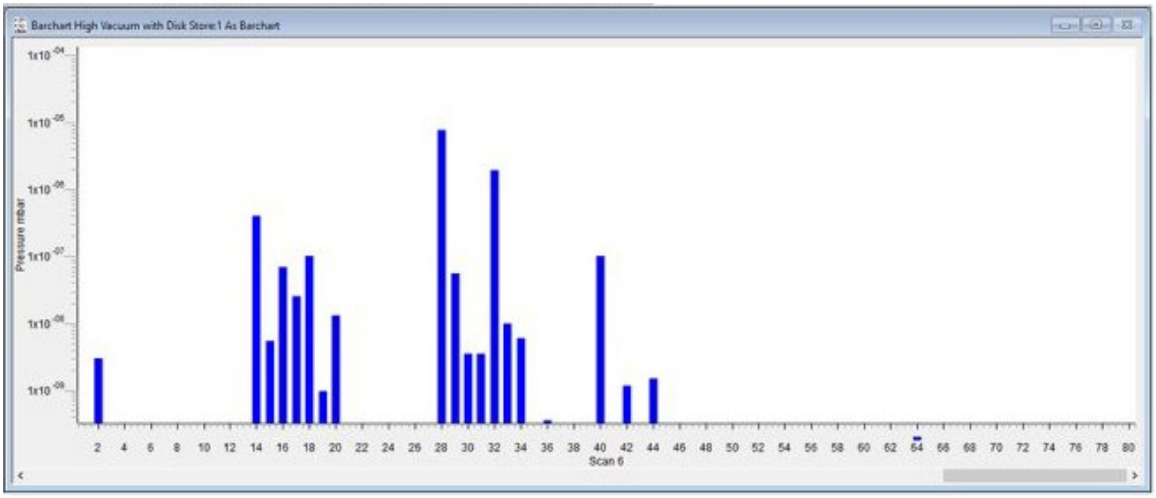


Fig. 3 Mass Spectrum of Residual Gas in Analysis Chamber. Measuring the partial pressure of specific molecules. [6]

Theory

"The ex-situ Total Mass Loss, TML_{ex}, in percent is calculated from the pre- and post-test laboratory balance weighing by the following equation:

$$TML_{ex} = 100 * \left[\frac{m_{s+h}(i) - m_{s+h}(f)}{m_{s+h}(i) - m_h}\right] \quad (1)$$

where *m* indicates a measured mass, the subscripts *s* and *h* indicate the sample and holder, and *i* and *f* indicate initial and final weighing, respectively.

The pertinent data acquired during the isothermal outgassing test are the frequencies of the QCMs and the time since the start of the evacuation of the effusion cell in the interlock chamber. Isothermal outgassing data are calculated from QCM frequency data using the QCM mass sensitivity and the view factor from the QCM to the effusion cell orifice.
The QCM mass sensitivity is used to convert the QCMs output frequency into units of mass deposited on the QCM. The mass deposited on a QCM is calculated from the following equation:

$$m_d(T_q, T_s, t) = K_s[f(T_q, T_s, t) - f(T_q, T_s, 0)] \quad (2)$$

where $m_d(T_q, T_s, t)$ is the mass deposit density (g/cm^2), $f(T_q, T_s, t)$ is the frequency (Hz) of the QCM at temperature T_q with the sample at temperature T_s at time *t*, and K_s is the mass sensitivity factor (g/cm^2/Hz). Since the relationship between frequency shifts and mass deposited on the QCM crystal is linear well beyond the operating regime of the ASTM E 1559 measurements, the mass sensitivity factor, K_s, is constant for a given QCM type. The ASTM E 1559 standard provides K_s values for typical QCMs.

The second principal piece of information required for analyzing the isothermal outgassing test data is the view factor from the QCM to the effusion cell orifice. This view factor relates the mass deposit on the QCM to the mass lost from the sample and can be calculated from the following equation:

$$F_q = \frac{\pi r^2 W_{L/R}}{B(\phi_1)\cos(\phi_1)\cos(\phi_2)} \tag{3}$$

where
F_q = the view factor for QCM_q to the effusion cell orifice (cm^2)
r = the distance from the cell orifice to the QCM_q crystal (cm)
ϕ_1 = the angle between the QCM_q-to-orifice line of sight and the orifice normal
ϕ_2 = the angle between the QCM_q-to-orifice line of sight and the QCM_q normal
L = the length of the effusion cell orifice (mm)
R = the radius of the effusion cell orifice (mm)
$B(\phi_1)$ = the directivity of the angular flow from the effusion cell orifice
$W_{L/R}$ = the transmission probability of the orifice

The $B(\phi_1)$ and $W_{L/R}$ functions account for the deviation from a cosine distribution for molecular flow leaving an orifice of finite length. The angular flow directivity, $B(\phi_1)$, can be calculated from the following equations:

$$B(\phi_1) = 1 - \frac{2}{\pi}(1-\gamma)\left[sin^{-1}(\rho) = +\rho\sqrt{1-\rho^2}\right] + \frac{4}{3\pi}(1-2\gamma)\frac{1-(1-\rho^2)^{3/2}}{\rho} for\ \rho < 1 \tag{4}$$

and
$$B(\phi_1) = \gamma + \frac{4}{3\pi}\frac{1-2\gamma}{\rho} for\ \rho > 1 \tag{5}$$

where
$$\rho = \frac{Ltan(\phi_1)}{2R} \tag{6}$$

and
$$\gamma = \frac{\sqrt{L^2+4R^2}-L}{2R + \frac{4R^2}{\sqrt{L^2+4R^2}}} \tag{7}$$

The other flux distribution related function in Equation 3 that needs to be established is the transmission probability, $W_{L/R}$. This factor represents the fraction of molecules entering the upstream face of the effusion cell orifice which exit the downstream face towards the QCMs.

$W_{L/R}$ is a complex function of the geometry and dimensions of the orifice. Calculated values are briefly summarized in the ASTM E 1559 standard.

The in situ Total Mass Loss (TML) expressed in percent of sample mass is calculated from the mass density deposited on the 90 K QCM and normalized with respect to the initial mass of the sample using the following equation:

$$TML(T_q, T_s, t) = 100 * \left[\frac{F_1 m_d(T_q, T_s, t)}{m_{s+h}(i) - m_h}\right] \quad (8)$$

Equation 8 assumes that all of the outgassing flux impinging on this QCM is condensed. Some highly volatile outgassed species such as atmospheric gases will not deposit on the QCM but the error introduced by this assumption is acceptably small." [6]

In some cases, it is desirable to have the TML normalized to the surface area, A_s, of the sample. This would give units of total mass lost from the sample per unit area of sample (μg/cm^2). This is shown with the following equation:

$$TML(T_1, T_s, t) = 10^6 * \left[\frac{F_1 m_d(T_1, T_s, t)}{A_s}\right] \quad (8)$$

"The Volatile Condensable Material (VCM) expressed in percent of sample mass is calculated from the mass density deposited on the higher temperature QCMs and normalized with respect to the initial mass of the sample using the following equation:

$$VCM(T_q, T_s, t) = 100 * \left[\frac{F_q m_d(T_q, T_s, t)}{m_{s+h}(i) - m_h}\right] \quad (9)$$

where q corresponds to the higher temperature QCMs." [7] As with TML, we can express VCM in μg/cm^2 with the following equation:

$$VCM(T_q, T_s, t) = 10^6 * \left[\frac{F_q m_d(T_q, T_s, t)}{A_s}\right] \quad (10)$$

"The time-dependent outgassing rates for species condensable on a QCM is computed by first differentiating the mass density deposited on a QCM as a function of time to give the deposition rate on the QCM. This can be obtained from a point-by-point differentiation of the QCM data as given in Eq 12.

$$\dot{m}_d(T_q, T_s, t) = \left[\frac{m_d(T_q, T_s, t+1) - m_d(T_q, T_s, t)}{(t+1) - (t)}\right] \tag{11}$$

where:

(t + 1) and *(t)* = the times that data were collected at consecutive intervals (s) and

$\dot{m}_d(T_d, T_s, t)$ = the deposition rate on QCMs 1, 2, and 3 ($g \cdot cm^{-2} \cdot s^{-1}$)

The deposition rate on a QCM $\dot{m}_d(T_q, T_s, t)$, is then used with the QCM-to-cell orifice view factor, F_q, and the sample surface area, A_s, in the following equation to calculate the total outgassing rate from the sample, $OGR(T_q, T_s, t)$, in units of g·cm−2·s−1 ." [8]

$$OGR(T_q, T_s, t) = \frac{F_q \dot{m}_d(T_q, T_s, t)}{A_s} \tag{12}$$

Procedure

Solid Edge was used to model all existing hardware so that it could be integrated into an efficient test stand. The hardware was adapted to a vertical configuration to improve the

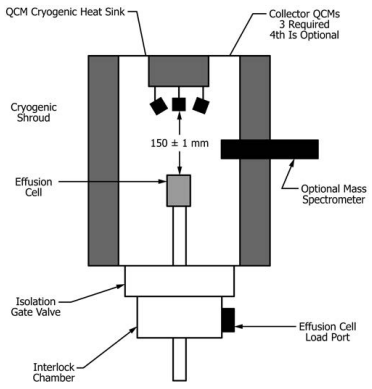


Fig. 4 From ASTM Standard showing basic internal configuration. [3]

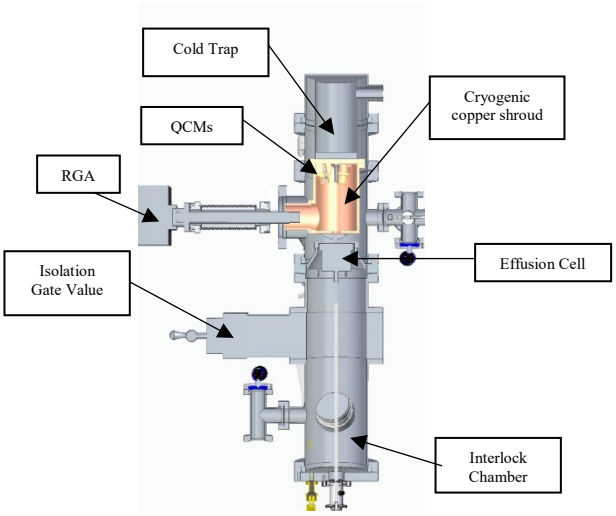


Fig. 5 Cutout of SDL Design.

cryogenic performance. Fig. 4 and Fig. 5 show side-by-side comparison of the ASTM standard and a cutout of SDL's design.

An instrumentation chassis was designed to integrate the vacuum gauge controllers, the vacuum pump control, the heater control, QCM controller, turbo pump controllers, power distribution system, Agilent data acquisition unit, and computer. Fig. 6 shows the current assembly of the instrumentation chassis.

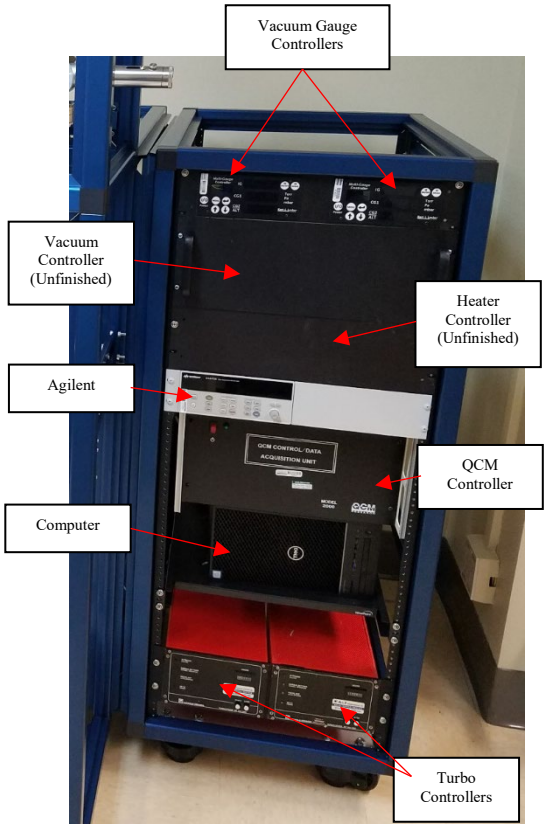
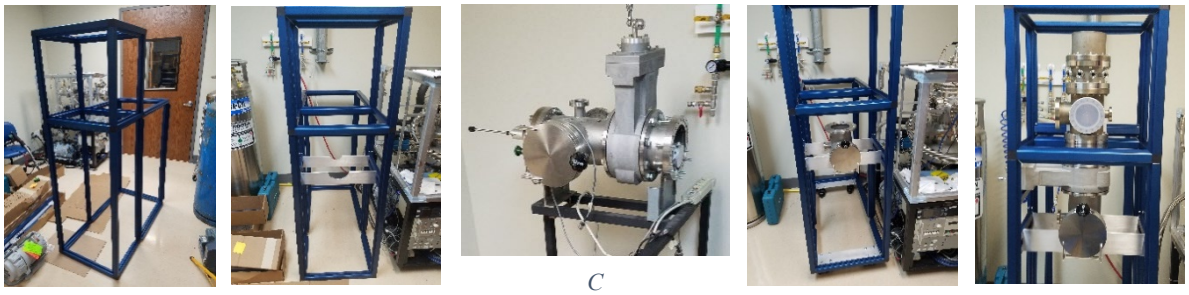


Fig. 6

A vacuum system controller was designed to turn on and off the pumps, open and close the pneumatic gate valves, and manage the liquid nitrogen level controller. Easy to see indicator lights show the status of gate valves, scroll and turbo pumps, LN2 controller, water flow, and pneumatic air. The pneumatic gate valves are interlocked to protect the turbo pumps. For a gate valve to remain open there must be sufficient water flow for cooling, turbo must be operating normally, chamber pressure sufficiently low, and enough air pressure to operate the gate valve. If any of these criteria fail, the gate valves will close automatically and protect the pumps.

After finishing the design and ordering all needed parts, assembly was started. Putting the frame together was straight forward, it was designed to go together easily (A in Fig. 7). Once the frame was assembled, the cross braces and saddles were mounted for the chamber (B in Fig. 7). This involved drilling, lining up, and tapping all the holes. The upper cross brace and saddle were able to be attached once the vacuum chamber was in the frame to help center it. Next was getting the vacuum chamber placed in the frame. All vacuum chamber components were removed from existing frame (C in Fig. 7), disassembled, and precision cleaned. The interlock

chamber was placed first in the bottom saddles (D in Fig. 7) and built up to the test vacuum chamber, including the copper shroud (E in Fig. 7).



A *B* *C* *D* *E*

Fig. 7

Unfortunately, the copper shroud was eclipsing the effusion cell (Fig 8). The cold trap had sagged from originally being mounted in a horizontal position and the QCM mounting plate was slightly angled. This would impede the QCMs' line of sight, which would invalidate test results. We were able to bend the cold trap to align with the centerline of the chamber, and we machined the QCM mounting plate to remove the tilt, which corrected the eclipsing issue (Fig 9).

Fig. 8 Misalignment of cryogenic shroud initially eclipsed apertures

Fig. 9 No eclipsing of apertures after significant rework of target chamber

Additionally, the data rack needed assembly. Four data rails were mounted inside of the supporting hardware frame. This was done by drilling and tapping holes in the frame. Once the data rails were mounted, it was a simple matter of mounting the vacuum gauge controllers, turbo controllers, and a shelf for the computer and QCM controller. The front panels for the control box and heater control were mounted for aesthetics.

Results

Unfortunately, time prohibited the ability to get the chamber fully assembled to run a material test. Progress is still being made however. QCMs are ready to be mounted, vacuum control box is set to be wired, the effusion cell is ready to be installed, and the RGA is ready to be attached. Fig. 10 shows the current state of the chamber next to the completed 3D model.

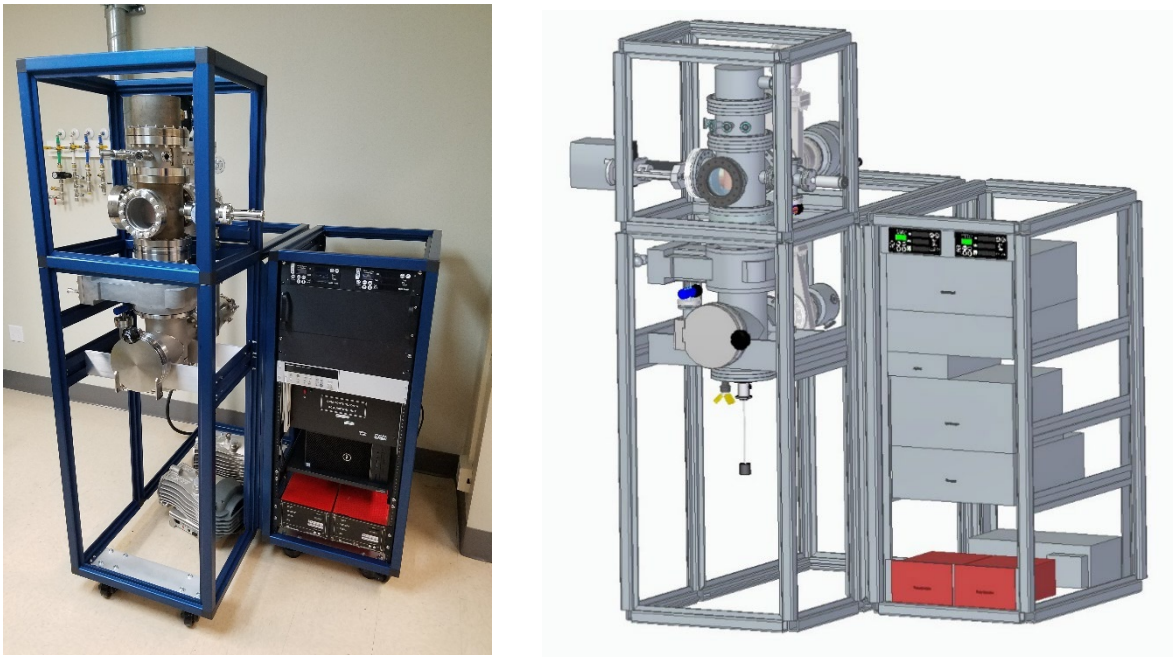


Fig. 10

The initial testing, once assemble is complete, will compare SDL measurements of common aerospace materials to previously published reference data, i.e. arathane 5753 or Kapton permacel p224 tape. Once satisfied with the performance of the chamber, testing of materials will begin. This will include new or novel materials from programs as innovation moves forward. This will help certify these new or novel materials for space-flight use.

References

[1] ASTM International. Standard Test Method for Contamination Outgassing Characteristics of Spacecraft Materials. 2016; section 1.1

[2] ASTM International. Standard Test Method for Contamination Outgassing Characteristics of Spacecraft Materials. 2016; section 5.1

[3] ASTM International. Standard Test Method for Contamination Outgassing Characteristics of Spacecraft Materials. 2016; section 1.1 FIG.2

[4] Vitali Issoupov, Sergey Horodetsky, Jacob Kleiman. 17 May 2017. ASTM E1559 Comparative Testing of Low-Outgassing Silicone-Based MAP Thermal Control Coatings.

[5] MKS. https://www.mksinst.com/f/vision-2000-p-xd-process-monitor

[6] J. W. Garret, A. P. M. Glassford, and J. M. Steakley Lockheed Missiles & Space Company. AStm E 1559 For Measuring Material Outgassing/Deposition Kenitics Has Applications to Aerospace, Electronics, and Semiconductor Industries.

[7] J. W. Garret, A. P. M. Glassford, and J. M. Steakley Lockheed Missiles & Space Company. AStm E 1559 For Measuring Material Outgassing/Deposition Kenitics Has Applications to Aerospace, Electronics, and Semiconductor Industries.

[8] ASTM International. Standard Test Method for Contamination Outgassing Characteristics of Spacecraft Materials. 2016; section 10