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Environmental Variation in Contamination Outgas Testing of a Composite Material

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14. ABSTRACT

Molecular contamination degrades sensitive spacecraft surfaces and can adversely affect the useful life of a spacecraft. In order to accurately predict mission performance, a thorough understanding of the emission and condensation of potential spacecraft contaminants is necessary. Potential sources of contamination include composite materials that are often used for large structural components. The large mass of these composite structures can represent the largest outgassing source on a spacecraft. This report documents a series of tests that were performed to investigate the outgassing characteristics of a proprietary composite material. These tests measured the mass outgassed by the material when exposed to a vacuum environment at elevated temperatures and the deposition of the outgassed species on surfaces held at specific temperatures. The results indicate that testing a material under a variety of environmental conditions can provide a valuable array of outgassing information.

15. SUBJECT TERMS

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1. Introduction

This report documents a series of tests conducted at The Aerospace Corporation's Space Materials Laboratory between August and November of 2008. The Contamination Effects Research and Test (CERT) Facility was employed to study the outgassing characteristics of a proprietary composite material. Because of their high strength and low weight, composites are frequently used for large space structures, such as spacecraft, payload fairings, and payload attachment adapters. When used in these applications, composites are the largest mass of non-metallic material on a space vehicle, and thus will be the largest source of outgassing contamination. During this test series, the outgassing process was analyzed using multiple quartz-crystal microbalances (QCMs) to provide information on mass deposition rates of contaminants outgassed from the composite material under various environmental conditions. The QCMs were also employed to perform thermogravimetric analysis on the condensed species. This report presents the results of this test series.

2. Experimental

The CERT facility is a state-of-the-art high-vacuum contamination effects testing facility, designed to allow numerous diagnostic instruments simultaneous in-situ analysis capabilities.[1] The CERT chamber is 61 cm (24 in.) in diameter and 76.2 cm (30 in.) tall. It is pumped to a base pressure of approximately 3.0×10^{-8} torr. A thermal-controlled rotating sample carrier centered in the chamber allows mounting of up to four targets. This radial design allows the targets to be rotated between the deposition source and the various in-situ diagnostic instruments. A cryogenic copper shroud divides the chamber into a deposition section and an analysis section.

The deposition source is a Knudsen-type effusion cell mounted horizontally in the chamber. An internal glass crucible accommodates 40 cm^3 of solid material or 10 cm^3 of liquid material. The effusion cell orifice is 3 mm in diameter. A ceramic lip heater prevents condensation at this orifice, and a cryogenic shutter shields the targets from the source. As shown in Figure 1, the effusion cell orifice is approximately 15.9 cm (6.25 in.) from the targets.

Several cryogenic quartz-crystal microbalance (Mark 18, QCM Research) targets can be mounted in the chamber to monitor molecular flux. These QCMs are actively heated and passively cooled for thermal control between cryogenic temperatures and 373K. During this test series, two or three QCMs were employed to monitor the molecular flux rates depending on the test configuration. As shown in Figure 1, QCM 1 was mounted on the rotating sample carrier, directly on-axis to the effu-

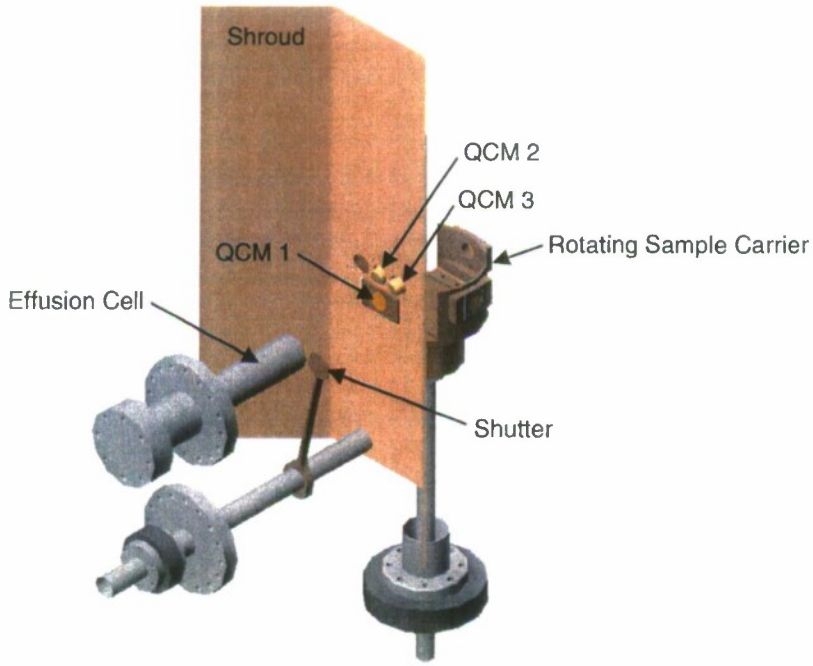

Figure 1. CERT chamber interior deposition section.

sion cell orifice normal. QCM 2 was mounted in the center position on the cryogenic copper shroud at an angle of 15° to the effusion cell orifice normal. QCM 3, when employed, was mounted off-center on the shroud at an angle of 19.2° to the effusion cell orifice normal.

QCMs monitor molecular flux indirectly by measuring the change in frequency of an oscillating crystal when material is deposited on its surface. From the change in resonant frequency, the mass of the deposit can be calculated. If the density of the deposited material is known, the mass-equivalent film thickness can also be determined. The QCM frequency varies as a function of crystal temperature. For thermo-gravimetric analysis (TGA), the QCMs must be calibrated to correct the data for frequency changes associated with varying temperatures during the thermal ramp. For the purposes of these experiments, a temperature of 233K (–40°C) was selected for QCM 1, 113K (–160°C) for QCM 2, and 273K (0°C) for QCM 3 during material outgassing deposition. During TGA, the QCMs were ramped from these temperatures to 373K.

The chamber geometry was designed to comply with the American Society for Testing and Materials (ASTM) *E1559 Standard Test Method for Contamination Outgassing Characteristics of Spacecraft Materials*, Test Method B.[2] However, this test series did not follow the standard ASTM E1559 outgassing procedures because it was intended to provide a broader overview of the outgassing characteristics of the composite material under a variety of environmental conditions. As a result, although the in-situ and ex-situ total mass loss (TML) and in-situ volatile condensable material (VCM) calculations described in Appendix A are similar to those presented in the ASTM E1559 standard, the results herein are distinct from standard ASTM E1559 test results. TML and VCM are a function of the outgassing test time, the test conditions, and the test geometry; as such, this test series provides valuable data on the variation in outgassing measurements for non-standard test procedures and non-standard chamber geometries.

3. General Test Procedure

The test procedure for this test series was based loosely on the ASTM E1559 standard, with significant, test-specific modifications.

1. Pre-Test Chamber Preparation
   - 1.1 Vent chamber
   - 1.2 Remove effusion cell from chamber
   - 1.3 Load test material into effusion cell crucible
     - 1.3.1 Weigh test material
   - 1.4 Install effusion cell in chamber
   - 1.5 Evacuate chamber
     - 1.5.1 Start data acquisition
     - 1.5.2 Cool effusion cell to below 293 K
     - 1.5.3 Allow chamber pressure to reach 5×10^{-7} torr

2. Testing
   - 2.1 Cool shroud and shutter to cryogenic temperatures
   - 2.2 Deposition
     - 2.2.1 Cool QCMs to specified temperatures
     - 2.2.2 Allow several hours for temperatures to stabilize and background data to be collected
     - 2.2.3 Open the effusion cell shutter
     - 2.2.4 Heat the effusion cell to the specified temperature
     - 2.2.5 Deposit outgassed contaminants on the QCMs for a specified time period
     - 2.2.6 Close the effusion cell shutter
     - 2.2.7 Cool the effusion cell to below 293 K
   - 2.3 Thermogravimetric Analysis (TGA)
     - 2.3.1 Heat QCMs from their specified temperatures to 373 K at 1°/min
   - 2.4 Cool QCMs to ambient

3. Post-Test Chamber Work
   - 3.1 Warm all components in chamber
   - 3.2 Vent chamber once all components are above 288 K
   - 3.3 Remove effusion cell from chamber
   - 3.4 Remove test material from effusion cell crucible
     - 3.4.1 Weigh test material to determine mass loss during testing
   - 3.5 Install effusion cell in chamber for next test

4. Results

This test series was intended to investigate the outgassing kinetics of contaminants outgassed by a composite material. A panel of the material was provided from which four 3.5 in. by 1 in. rectangular samples were cut for testing at specific conditions. A single sample is shown in Figure 2.

A total of four samples were tested in this test series under various conditions. The kinetic outgassing results for each sample are presented below. Effusion cell temperature calibration results are presented in Appendix B. Thermogravimetric analysis (TGA) results are not included in this report due to poor calibration of the QCM frequency changes associated with varying temperatures during thermal ramps.

4.1 Sample 1

Sample 1 was loaded into the effusion cell for testing on 18 August 2008. The mass of Sample 1 before testing was measured as 3.916 g. During testing, this sample was heated to 323K and allowed to outgas for 61.9 h. The temperature profile and flux measured by the QCMs during testing of Sample 1 are shown in Figure 3 and Figure 4. The QCM output is reported in Hertz (Hz). Assuming a deposited-mass density of 1 g/cm^3, a change of 5 Hz on a QCM represents a change of approximately 1 Å in mass-equivalent film thickness. The TML and VCM analyses for Sample 1 are summarized in Table 1 and Table 2.

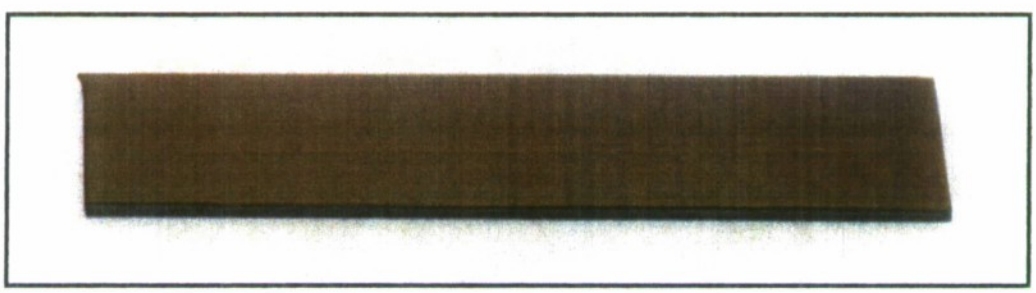

Figure 2. Sample of the composite material prepared for testing.

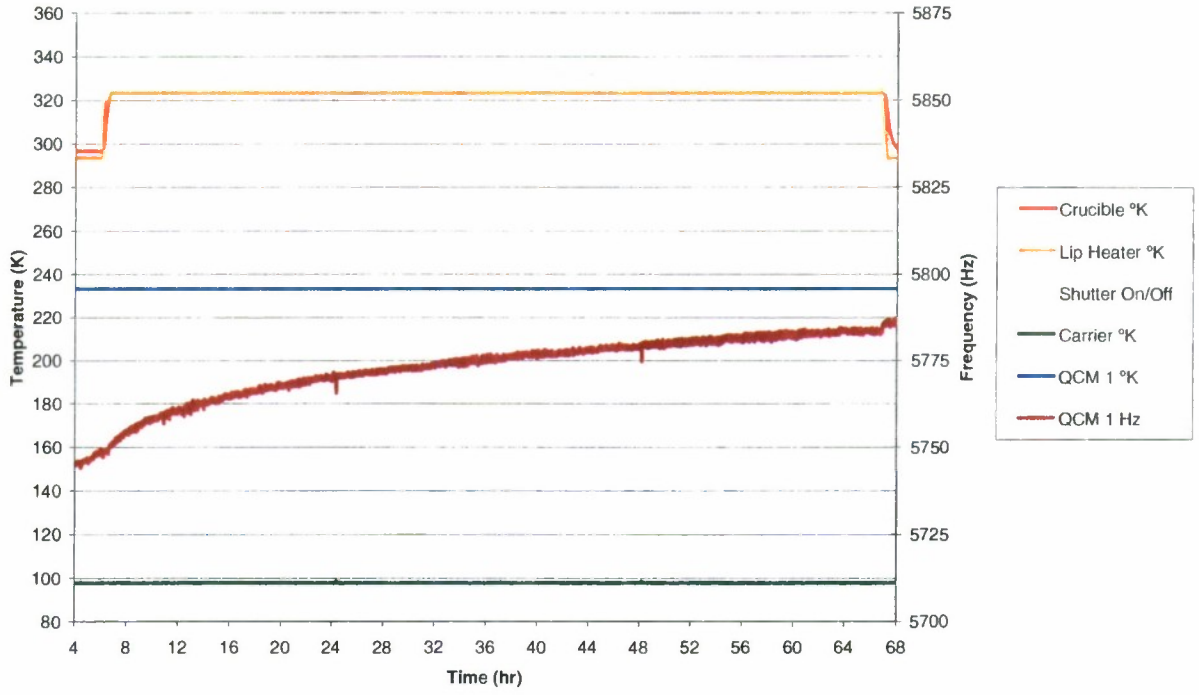

Figure 3. Deposition of contaminants outgassed by Sample 1 on QCM 1 (233K).

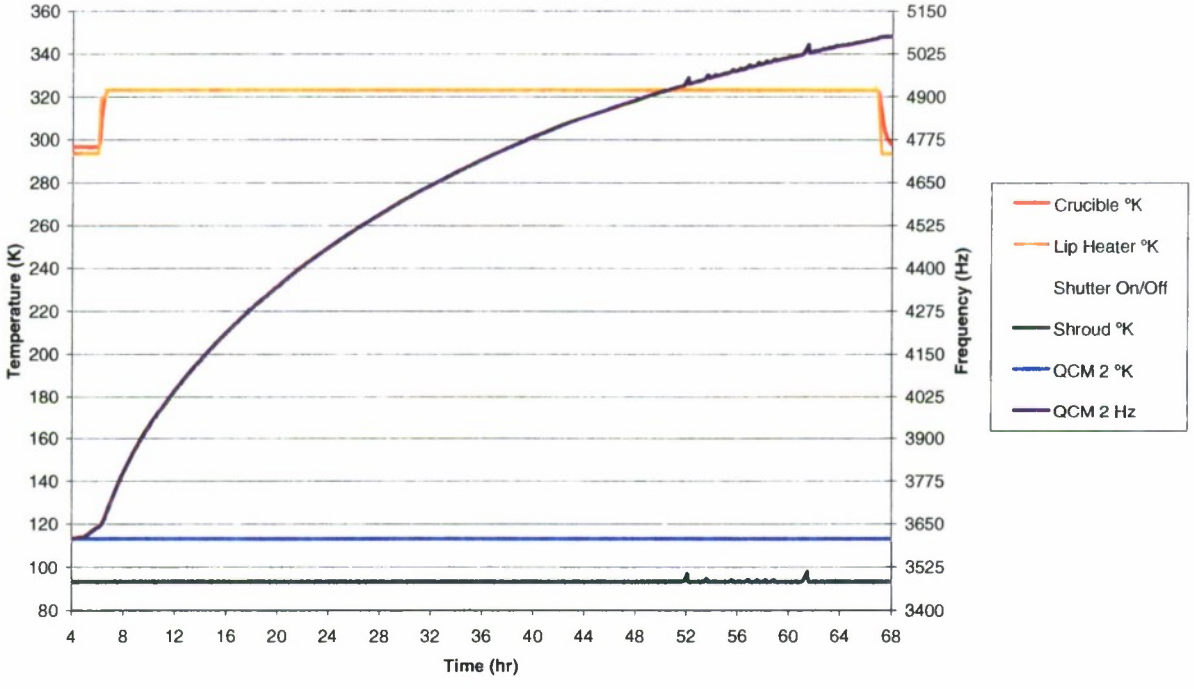

Figure 4. Deposition of contaminants outgassed by Sample 1 on QCM 2 (113K).

Table 1. In-situ TML and VCM Analysis of Sample 1.

Parameter	QCM 1 VCM Analysis	QCM 2 TML Analysis
r	15.9 cm	15.9 cm
ϕ_1	0°	15°
ϕ_2	0°	0°
L	0.05 mm	0.05 mm
R	1.5 mm	1.5 mm
L/R	0.0333	0.0333
$W_{L/R}$	0.9984	0.9984
p	0	0.0045
γ	0.4918	0.4918
$B(\phi_1)$	1	0.9972
F_q	790.5 cm^2	820.7 cm^2
K	1.965×10^{-9} $g/cm^2/Hz$	1.965×10^{-9} $g/cm^2/Hz$
f_{end}	5784 Hz	5072 Hz
f_0	5746 Hz	3610 Hz
m_d	7.466×10^{-8} g/cm^2	2.872×10^{-6} g/cm^2
m_s	3.916 g	3.916 g
	In-situ VCM = 0.0015%	**In-situ TML = 0.060%**

Table 2. Ex-situ TML Analysis of Sample 1.

Parameter	Ex-situ TML Analysis
$m_s(i)$	3.916 g
$m_s(f)$	3.914 g
	Ex-situ TML = 0.051%

4.2 Sample 2

Sample 2 was loaded into the effusion cell for testing on 25 August 2008. The mass of Sample 2 before testing was measured as 3.973 g. During testing, this sample was heated to 373K and allowed to outgas for 25.6 h. The temperature profile and flux measured by the QCMs during testing of Sample 2 are shown in Figure 5 and Figure 6. The TML and VCM analyses for Sample 2 are summarized in Table 3 and Table 4.

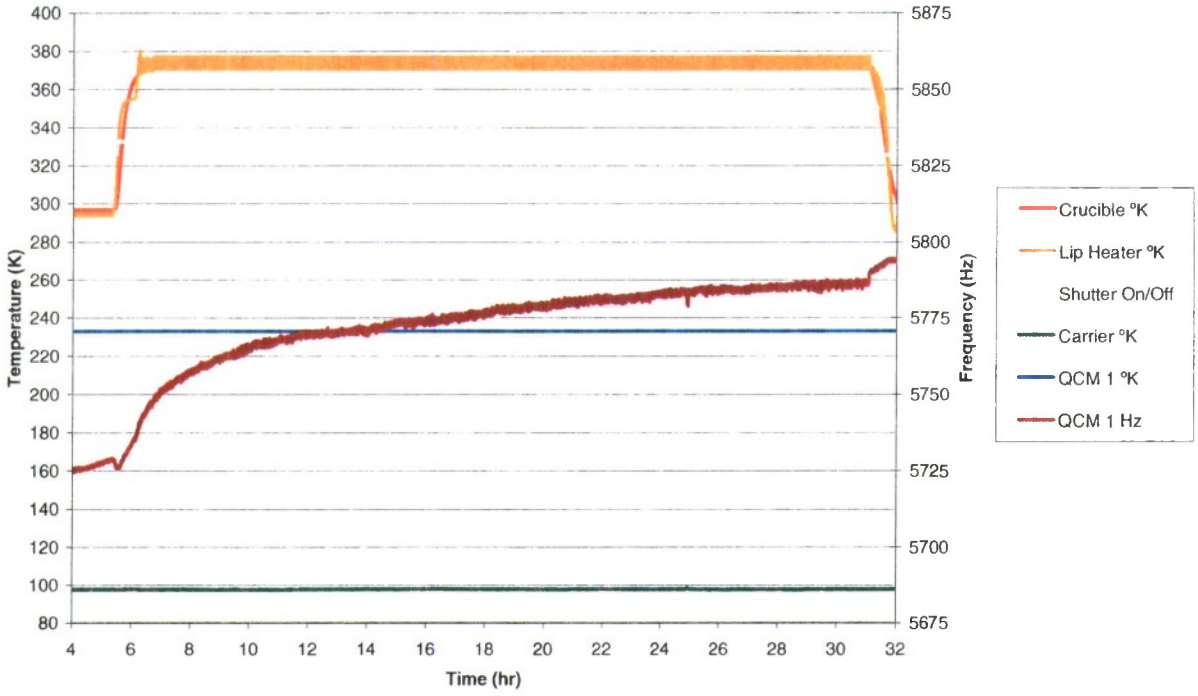

Figure 5. Deposition of contaminants outgassed by Sample 2 on QCM 1 (233K).

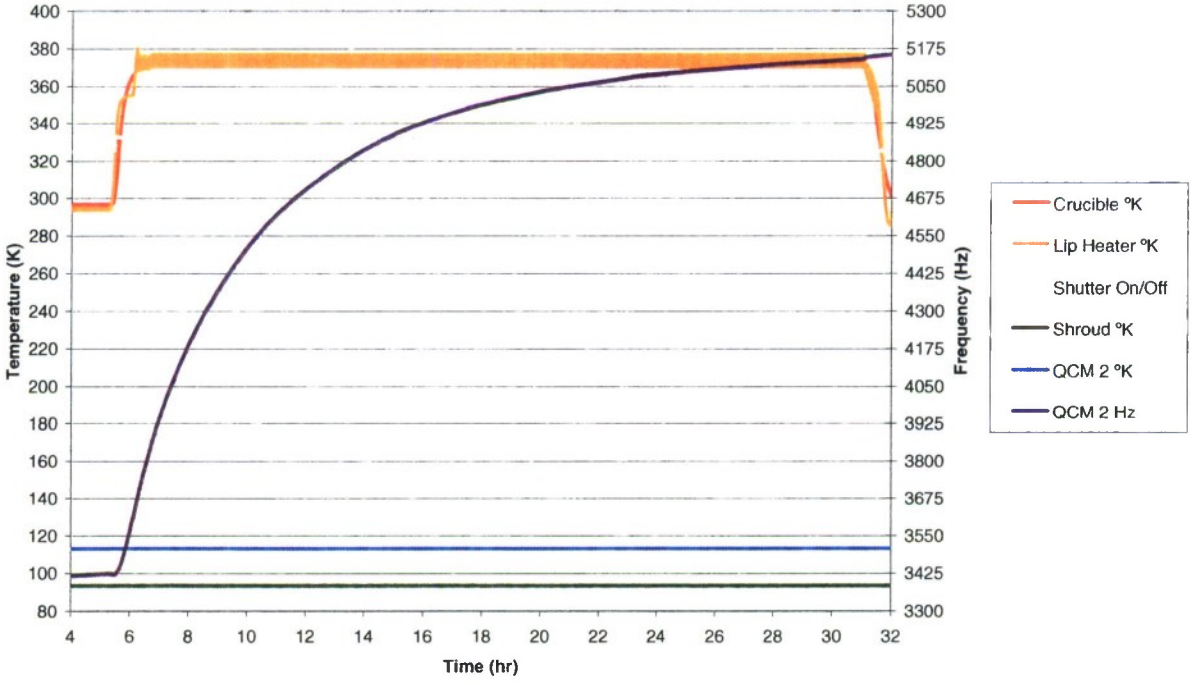

Figure 6. Deposition of contaminants outgassed by Sample 2 on QCM 2 (113K).

Table 3. In-situ TML and VCM Analysis of Sample 2.

Parameter	QCM 1 VCM Analysis	QCM 2 TML Analysis
r	15.9 cm	15.9 cm
ϕ_1	0°	15°
ϕ_2	0°	0°
L	0.05 mm	0.05 mm
R	1.5 mm	1.5 mm
L/R	0.0333	0.0333
$W_{L/R}$	0.9984	0.9984
p	0	0.0045
γ	0.4918	0.4918
$B(\phi_1)$	1	0.9972
F_q	790.5 cm^2	820.7 cm^2
K	1.965×10^{-9} $g/cm^2/Hz$	1.965×10^{-9} $g/cm^2/Hz$
f_{end}	5789 Hz	5145 Hz
f_0	5728 Hz	3419 Hz
m_d	1.198×10^{-7} g/cm^2	3.391×10^{-6} g/cm^2
m_s	3.973 g	3.973 g
	In-situ VCM = 0.0024%	**In-situ TML = 0.070%**

Table 4. Ex-situ TML Analysis of Sample 2.

Parameter	Ex-situ TML Analysis
$m_s(i)$	3.973 g
$m_s(f)$	3.971 g
	Ex-situ TML = 0.050%

4.3 Sample 3

Sample 3 was loaded into the effusion cell for testing on 27 August 2008. The mass of Sample 3 before testing was measured as 3.846 g. During testing, this sample was heated to 348K and allowed to outgas for 40.5 h. The temperature profile and flux measured by the QCMs during testing of Sample 3 are shown in Figure 7 and Figure 8. The TML and VCM analyses for Sample 3 are summarized in Table 5 and Table 6.

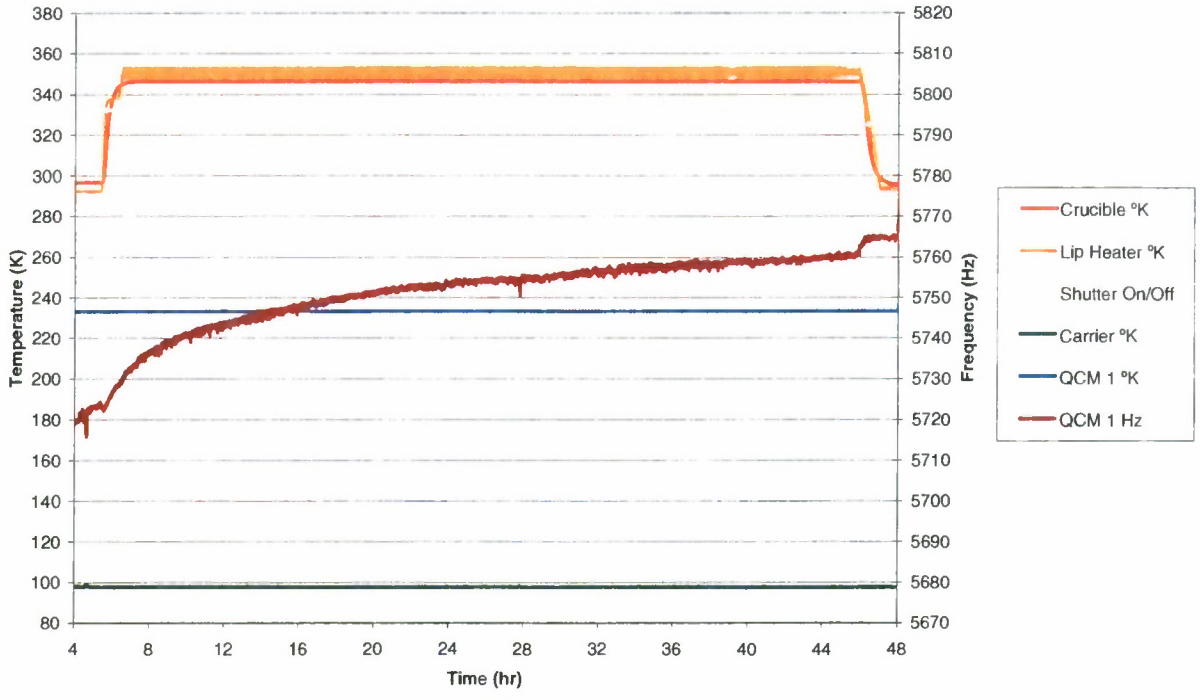

Figure 7. Deposition of contaminants outgassed by Sample 3 on QCM 1 (233K).

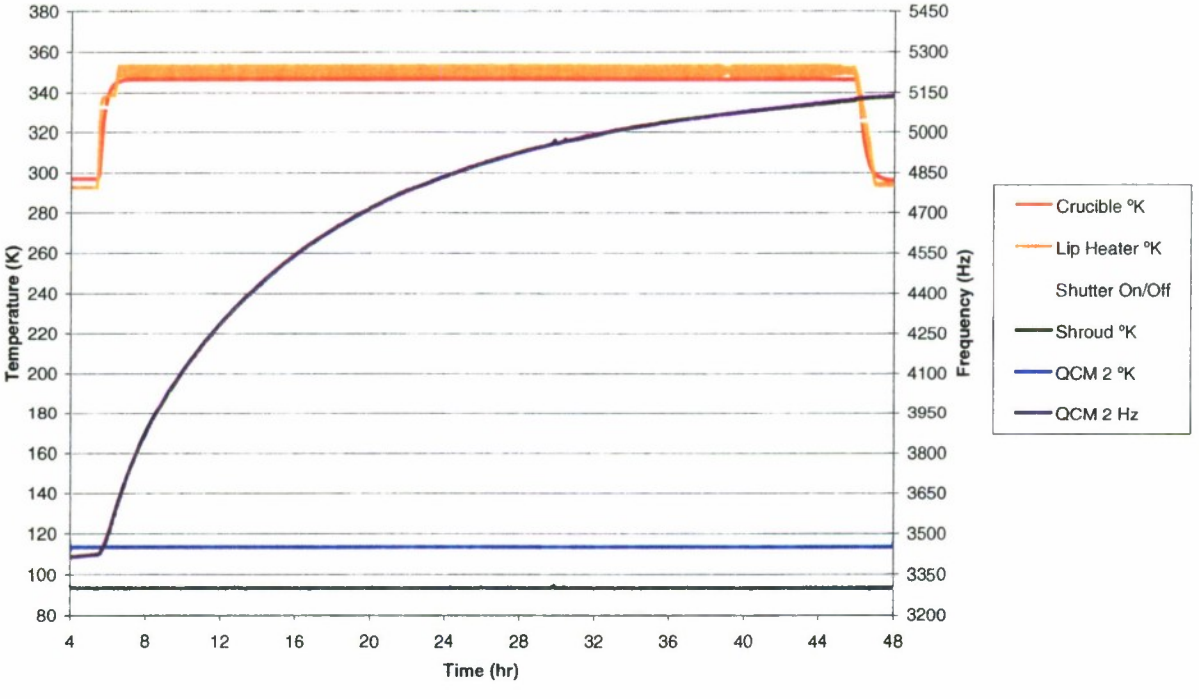

Figure 8. Deposition of contaminants outgassed by Sample 3 on QCM 2 (113K).

Table 5. In-situ TML and VCM Analysis of Sample 3.

Parameter	QCM 1 VCM Analysis	QCM 2 TML Analysis
r	15.9 cm	15.9 cm
ϕ_1	0°	15°
ϕ_2	0°	0°
L	0.05 mm	0.05 mm
R	1.5 mm	1.5 mm
L/R	0.0333	0.0333
$W_{L/R}$	0.9984	0.9984
p	0	0.0045
γ	0.4918	0.4918
$B(\phi_1)$	1	0.9972
F_q	790.5 cm^2	820.7 cm^2
K	1.965×10^{-9} $g/cm^2/Hz$	1.965×10^{-9} $g/cm^2/Hz$
f_{end}	5763 Hz	5122 Hz
f_0	5722 Hz	3418 Hz
m_d	8.055×10^{-8} g/cm^2	3.348×10^{-6} g/cm^2
m_s	3.846 g	3.846 g
	In-situ VCM = 0.0017%	**In-situ TML = 0.071%**

Table 6. Ex-situ TML Analysis of Sample 3.

Parameter	Ex-situ TML Analysis
$m_s(i)$	3.846 g
$m_s(f)$	3.844 g
	Ex-situ TML = 0.052%

4.4 Sample 4

Sample 4 was loaded into the effusion cell for testing on 27 October 2008. The mass of Sample 4 before testing was measured as 3.904 g. During testing, this sample was heated to 348K and allowed to outgas for 73 h. In contrast to the previous tests, this test employed a third QCM. The temperature profile and flux measured by the QCMs during testing of Sample 4 are shown in Figure 9, Figure 10, and Figure 11. The deviations in the data beginning at approximately 20 h are due to a power interruption that resulted in an extended loss of thermal control. This fluctuation in temperature has a small but uncalibrated effect on the QCM frequency recordings, and introduces an unknown error into the results. The TML and VCM analyses for Sample 4 are summarized in Table 7 and Table 8.

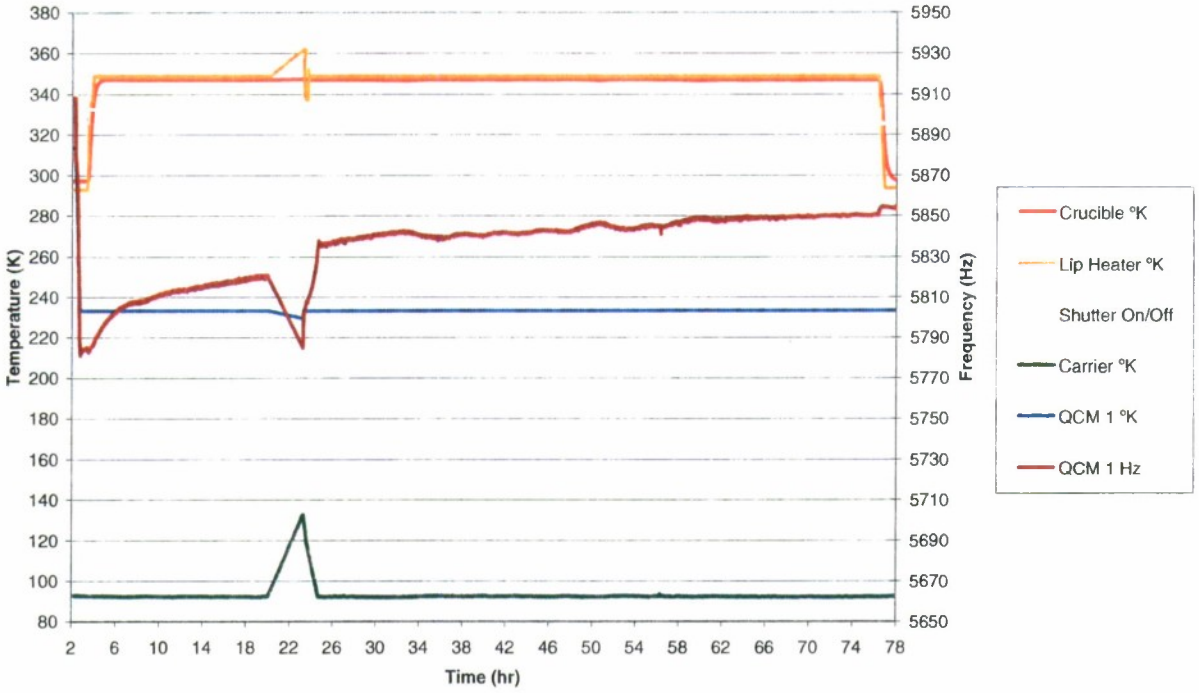

Figure 9. Deposition of contaminants outgassed by Sample 4 on QCM 1 (233K).

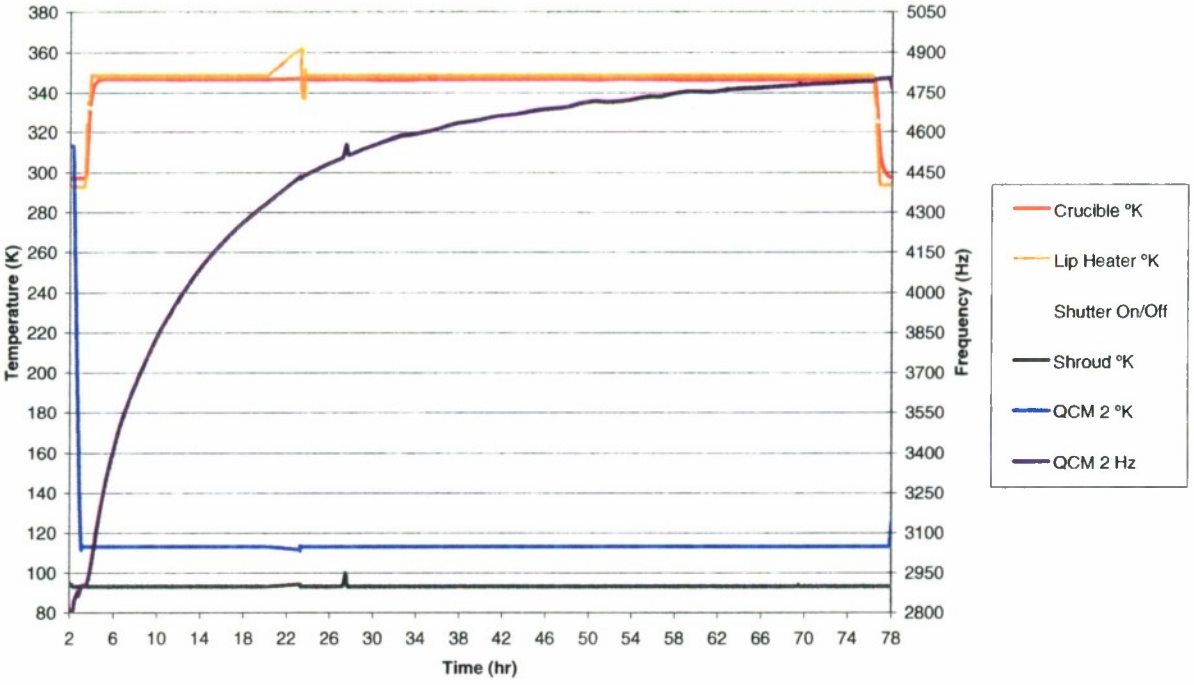

Figure 10. Deposition of contaminants outgassed by Sample 4 on QCM 2 (113K).

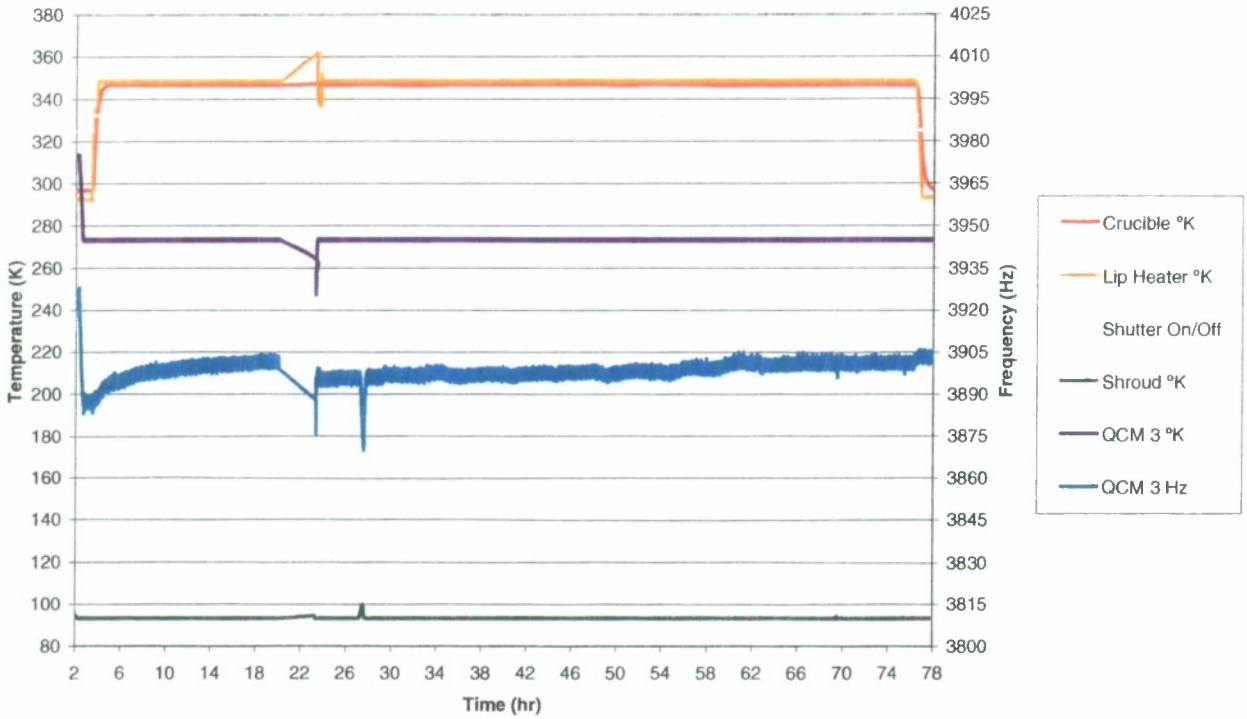

Figure 11. Deposition of contaminants outgassed by Sample 4 on QCM 3 (273K).

Table 7. In-situ TML and VCM Analysis of Sample 4.

Parameter	QCM 1 VCM Analysis	QCM 2 TML Analysis	QCM 3 VCM Analysis
R	15.9 cm	15.9 cm	15.9 cm
ϕ_1	0°	15°	19.2°
ϕ_2	0°	0°	0°
L	0.05 mm	0.05 mm	0.05 mm
R	1.5 mm	1.5 mm	1.5 mm
L/R	0.0333	0.0333	0.0333
$W_{L/R}$	0.9984	0.9984	0.9984
P	0	0.0045	0.0058
γ	0.4918	0.4918	0.4918
$B(\phi_1)$	1	0.9972	0.9963
F_q	790.5 cm^2	820.7 cm^2	840.1 cm^2
K	1.965×10^{-9} $g/cm^2/Hz$	1.965×10^{-9} $g/cm^2/Hz$	1.965×10^{-9} $g/cm^2/Hz$
f_{end}	5853 Hz	4798 Hz	3903 Hz
f_0	5783 Hz	2900 Hz	3887 Hz
m_d	1.375×10^{-7} g/cm^2	3.729×10^{-6} g/cm^2	3.143×10^{-8} g/cm^2
m_s	3.905 g	3.905 g	3.905 g
	In-situ VCM = 0.0028%	**In-situ TML = 0.078%**	**In-situ VCM = 0.0007%**

Table 8. Ex-Situ TML Analysis of Sample 4.

Parameter	Ex-situ TML Analysis
m_h	21.412 g
$m_{s+h}(i)$	25.316 g
$m_s(i)$	3.904 g
$m_{s+h}(f)$	25.312 g
$m_s(f)$	3.900 g
	Ex-situ TML = 0.10%

5. Conclusion

The results from the test series are summarized in Table 9, along with the specific test parameters for each sample. Testing the composite material under a variety of environmental conditions provides a broader collection of outgassing data that is useful in understanding complex contamination problems associated with this material. Standardized testing at a single set of conditions does not provide the breadth of information obtained through testing under multiple conditions. These results show that environmental conditions have a large influence on the outgassing kinetic measurements obtained. In addition, this collection of data indicates that materials in the generic composite family produce small amounts of outgassing contamination.

Table 9. Summary of Test Series Results

	Sample 1	Sample 2	Sample 3	Sample 4
Date	8/18/2008	8/25/2008	8/27/2008	10/27/2008
Effusion Cell Temperature	323 K	373 K	348 K	348 K
Outgassing Time	61.9 hr	25.6 hr	40.5 hr	73 hr
Sample Mass (Initial)	3.916 g	3.973 g	3.846 g	3.904 g
QCM 1 Temperature	233 K	233 K	233 K	233 K
QCM 1 Location	Carrier	Carrier	Carrier	Carrier
QCM 2 Temperature	113 K	113 K	113 K	113 K
QCM 2 Location	Shroud Center	Shroud Center	Shroud Center	Shroud Center
QCM 3 Temperature	-	-	-	273 K
QCM 3 Location	-	-	-	Shroud Side
QCM 1 In-situ VCM	0.0015%	0.0024%	0.0017%	0.0028%
QCM 3 In-situ VCM	-	-	-	0.0007%
QCM 2 In-situ TML	0.060%	0.070%	0.071%	0.078%
Ex-situ TML	0.051%	0.050%	0.052%	0.10%

Appendix A—TML and VCM Analysis

ASTM E1559 provides standard procedures for determining standardized in-situ and ex-situ total mass loss (TML) and in-situ volatile condensable material (VCM) values from the outgassing data collected from a sample material. This allows kinetic outgassing information of different materials to be compared. TML and VCM measurements are time-, temperature-, and configuration-dependent; as such, the data collected in this test series can be used to determine TML and VCM values, but these values are not comparable to standardized ASTM E1559 results. This appendix presents in-situ and ex-situ TML and in-situ VCM calculation procedures used in this test series. The results for each sample are presented in Section 4, where the non-standard results are qualified by the total outgassing time and the test conditions.

A.1 In-situ Measurements

The time-dependent, in-situ total mass loss (%) determined by mass deposition on the cryogenically cooled QCM (QCM 2) is given by

$$TML = 100 \cdot \left(\frac{F_{q,2} m_{d,2}}{m_s} \right), \tag{A1}$$

where m_s is the measured sample mass before the test, and all quantities denoted with the subscript *2* refer to QCM 2. This definition of TML assumes that essentially all the outgassing flux impinging on the cryogenic QCM 2 is condensed.

Similarly, the time-dependent, in-situ volatile condensable material (%) determined by mass deposition on QCM 1 or QCM 3 is given by

$$VCM_{QCMx} = 100 \cdot \left(\frac{F_{q,x} m_{d,x}}{m_s} \right), \tag{A2}$$

where x is *1* or *3* such that all quantities denoted with the subscript x refer to QCM 1 or QCM 3.

The quantities used to calculate *TML* and *VCM* are defined, in general, in the subsequent sections, and were calculated for the specific QCM of interest. It should be noted that these in-situ TML and VCM measurements are not the same as the ex-situ TML and CVCM measurements determined through ASTM E595 testing.[3]

The QCM-to-effusion cell orifice view factor (cm^2) is defined as

$$F_q = \frac{\pi r^2 W_{L/R}}{B(\phi_1)\cos(\phi_1)\cos(\phi_2)}, \tag{A3}$$

where:

r = distance from the orifice to the QCM crystal (cm),

ϕ_1 = angle between the QCM-to-cell orifice line of sight and the orifice normal,

ϕ_2 = angle between the line of sight and the QCM normal,

L = length of the effusion cell orifice (mm),

R = radius of the effusion cell orifice (mm),

$W_{L/R}$ = "Clausing transmission probability" for the effusion cell orifice (Table A1), and

$B(\phi_1)$ = "Clausing angular flow distribution" for the effusion cell orifice and the QCM position.

The Clausing transmission probability is defined in Table A1.[2]

Table A1. Values of Clausing Transmission Probability, $W_{L/R}$.

L/R	$W_{L/R}$
0	1
0.1	0.9524
0.2	0.9092
0.3	0.8699
0.4	0.8341
0.5	0.8013
1.0	0.6720
1.5	0.5810
2.0	0.5136
5.0	0.3146
10.0	0.1973

The Clausing angular flow distribution is defined as follows. For $\rho < 1$,

$$B(\phi_1) = 1 - \frac{2}{\pi}(1-\gamma)\left[\sin^{-1}(\rho) + \rho\sqrt{1-\rho^2}\right] + \frac{4}{3\pi}(1-2\gamma)\frac{1-\left(1-\rho^2\right)^{3/2}}{\rho}. \qquad \text{(A4)}$$

For $\rho > 1$,

$$B(\phi_1) = \gamma + \frac{4}{3\pi}\frac{1-2\gamma}{\rho}. \qquad \text{(A5)}$$

In the limit that $\rho \rightarrow 0$,

$$B(\phi_1) = 1. \qquad \text{(A6)}$$

For calculating $B(\phi_1)$, ρ and γ are defined as

$$\rho = \frac{L\tan(\phi_1)}{2R} \tag{A7}$$

and

$$\gamma = \frac{\sqrt{L^2+4R^2}-L}{2R+\dfrac{4R^2}{\sqrt{L^2+4R^2}}}. \tag{A8}$$

The deposited mass density (g/cm^2) on each QCM x is defined as

$$m_{d,x} = K\left(f_{end,x} - f_{0,x}\right), \tag{A9}$$

where f_0 is the frequency (Hz) of the QCM at time zero, f_{end} is the frequency (Hz) at the end of the deposition period, and K (g/cm^2/Hz) is the mass sensitivity factor of the QCM. The QCMs employed in this test series have a mass sensitivity factor of 1.965×10^{-9} g/cm^2/Hz.

A.2 Ex-situ Measurements

Time-dependent ex-situ total mass loss (%) is determined by the change in sample mass as measured prior to and after the sample is tested in the vacuum. The mass measurements were performed with a Sartorius TE313S-DS Analytical Microbalance. Samples 1, 2, and 3 were measured individually such that ex-situ TML is given by

$$TML_{ex} = 100\cdot\left(\frac{m_s(i)-m_s(f)}{m_s(i)}\right), \tag{A10}$$

where m indicates a measured mass, the subscript s refers to the sample, and i and f indicate measurements before and after the vacuum test, respectively. In contrast, Sample 4 was measured with the sample holder as described in ASTM E1559 such that ex-situ TML is given by

$$TML_{ex} = 100\cdot\left(\frac{m_{s+h}(i)-m_{s+h}(f)}{m_{s+h}(i)-m_h}\right), \tag{A11}$$

where the subscript h refers to the holder. For this test series, the holder was the effusion cell crucible.

Appendix B—Effusion Cell Temperature Calibration

Prior to testing the four samples, a calibration of the effusion cell temperature was performed. A sample of the composite material was instrumented with a thermocouple and loaded into the effusion cell. The effusion cell temperature was then stepped to 323K, 348K, and 373K to determine any lag between the temperature reported by the effusion cell controller and the temperature of the sample itself. Figure B1 shows the sample used in the calibration. It should be noted that the wire lead to the thermocouple was threaded through the effusion cell orifice during the test since this is the only opening into the crucible when it is installed in the chamber.

The temperature profile from this calibration is presented in Figure B2. The temperature of the sample is shown to be quite close to the temperature reported from the crucible. The largest differences between the two thermocouples occurred during ramping of the effusion cell, with a maximum lag between the crucible temperature and the sample temperature of approximately 5K. During the soak periods, the differences were much smaller. At 323K, the difference in the two thermocouples was approximately 2K because the lip heater was not being actively heated. At 348K and 373K, with the lip heater working properly, any difference was virtually undetectable. In the results presented herein, the crucible temperature is reported because the temperature of the sample could not be measured directly during testing.

QCM data recorded during the calibration is presented in Figure B3 and Figure B4 for completeness.

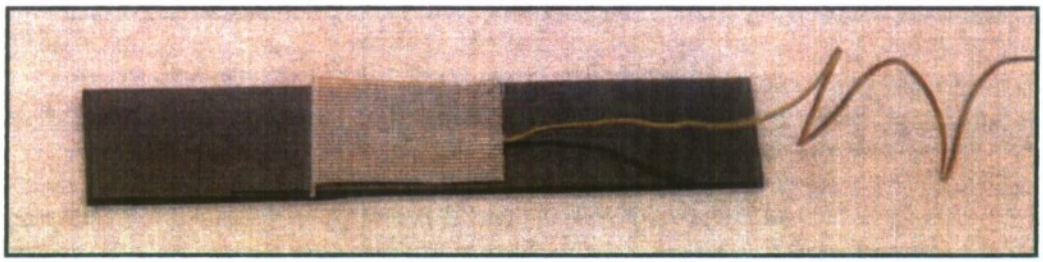

Figure B1. Sample of the composite with a thermocouple affixed to one side for calibration testing.

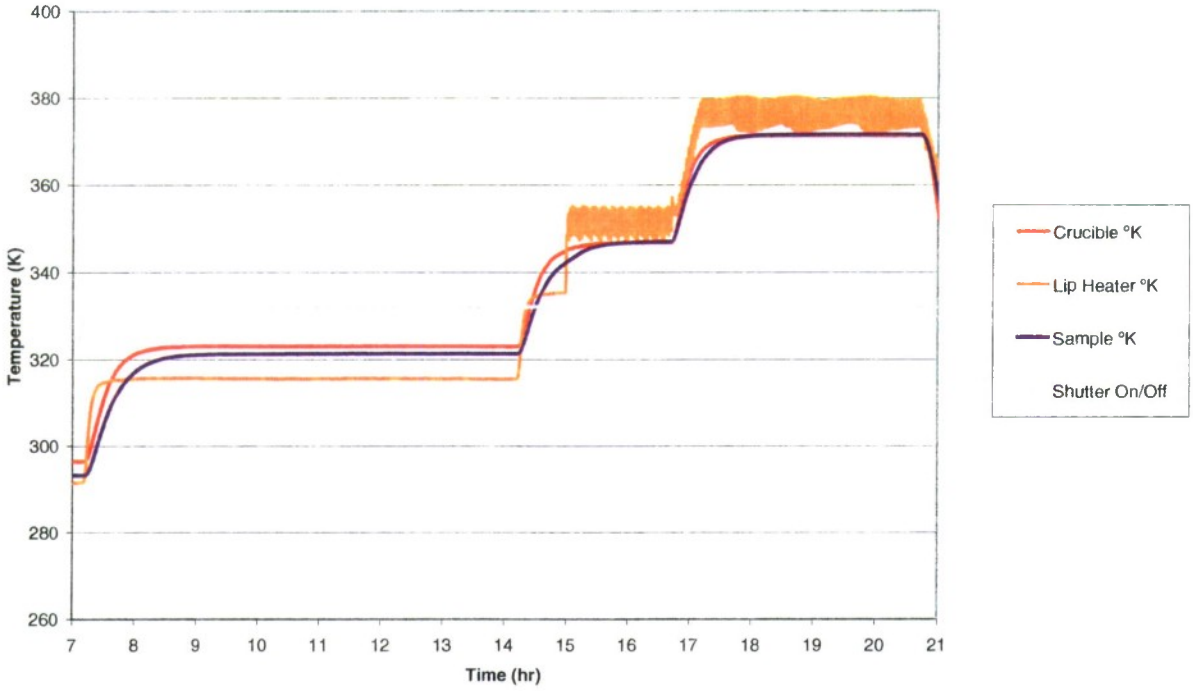

Figure B2. Effusion cell temperature calibration profile.

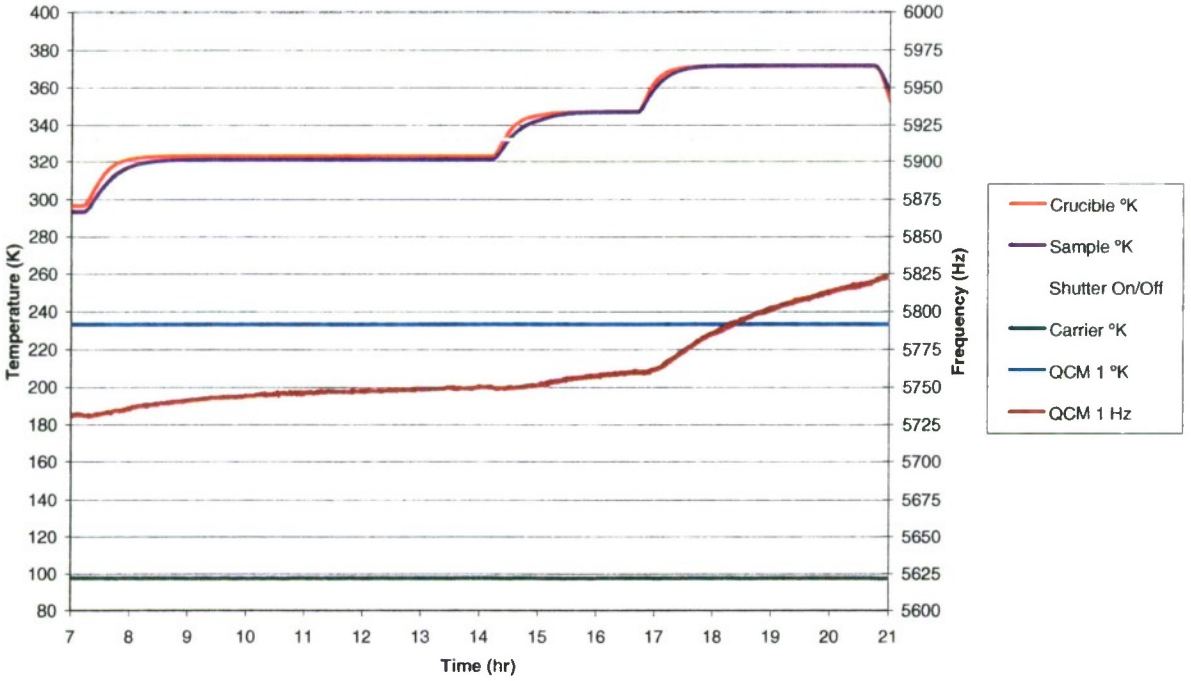

Figure B3. QCM 1 (233 K) data from effusion cell temperature calibration.

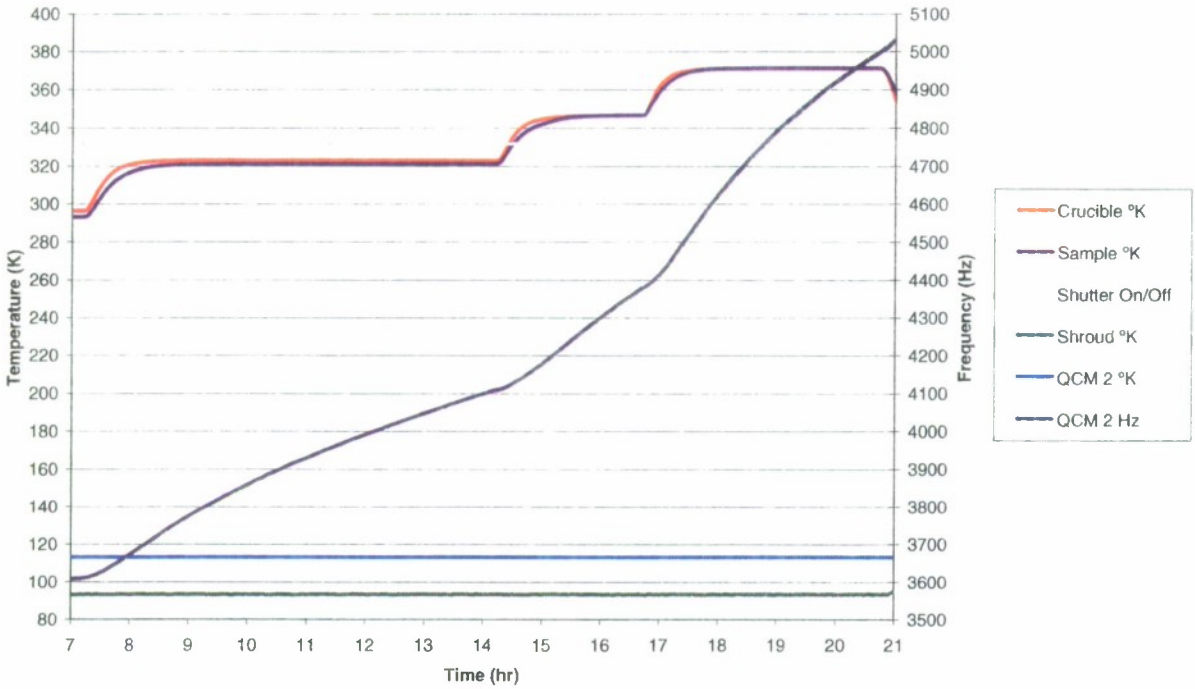

Figure B4. QCM 2 (113 K) data from effusion cell temperature calibration.

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