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Material Total Mass Loss in Vacuum Obtained from Various Outgassing Systems

John Scialdone, Peggy Isaac, Carroll Clatterbuck, and Ronald Hunkeler

National Aeronautics and Space Administration
Goddard Space Flight Center
Greenbelt, Maryland 20771

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*NASA's Goddard Space Flight Center
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ABSTRACT

Several instruments including the Cahn Microbalance, the Knudsen Cell, the micro-CVCM, and the vacuum Thermogravimetric Analyzer (TGA) were used in the testing of a graphite/epoxy (GR/EP) composite that is proposed for use as a rigidizing element of an inflatable deployment system. This GR/EP will be cured in situ. The purpose of this testing is to estimate the gaseous production resulting from the curing of the GR/EP composite, to predict the resulting pressure, and to calculate the required venting. Every test was conducted under vacuum at 125 degrees C for 24 hours. Upon comparison of the results, the ASTM E-595 was noted to have given readings that were consistently lower than those obtained using the other instruments, which otherwise provided similar results. The GR/EP was tested using several different geometric arrangements.

This paper describes the analysis evaluating the molecular and continuum flow of the outgassing products issuing from the exit port of the ASTM E-595 system. The effective flow conductance provided by the physical dimensions of the vent passage of the ASTM E-595 system and that of the material sample were investigated, among other factors, to explain the reduced amount of outgassing released during the 24-hour test period.

1.0 INTRODUCTION

Materials selected for space applications must maintain structural and chemical integrity when exposed to the environmental conditions of space. They should have low outgassing rates at normal operating temperatures and the outgassing should consist of a minimum of materials condensable at the temperatures of nearby surfaces.

The observation of distant sources of radiation or the measurement of the space medium is affected minimally with the spacecrafts having low outgassing. In addition, voltage breakdowns and undesirable heat transfer regimes at certain critical regions of the spacecraft are minimized by limiting material outgassing. The requirement on condensables reflects the potentially degrading effect of materials deposits on radiating surfaces and on elements of optical instruments. Deposits may change the thermal properties of a surface and attenuate, reflect, and disperse the incoming radiations to be measured.

Materials with the desirable characteristics have been screened by a test developed in [1] and used at various organizations including Goddard Space Flight Center (GSFC), European Space Research Organization (ESRO), and Jet Propulsion Lab (JPL). The test, described by [1-3], consists of maintaining samples of about 200 mg of the materials under consideration at a temperature of 398 K (125°C) for 24 hours in a vacuum of about 10^{-6} torr. The sample mass loss and the amount of that mass which condenses on a collector plate held at 298 K (25°C) and located near the sample are used as some of the criteria for the selection of these materials for space use. The test temperatures were purposely designed to be extreme; these temperatures can only be encountered in exceptional applications. The pressure of 10^{-6} torr is sufficiently low to prevent collisions between molecules leaving a surface. In general, the material is considered acceptable if the total mass loss (TML) is less than 1% and the volatile condensable mass (VCM) is less than 0.1% of the initial sample mass. The criteria arise from the observation that certain materials show mechanical and physical degradation when they have lost 3 to 5% of their mass. For others, such as elastomers, a 1% TML is usually detrimental to their mechanical properties. A more definite interpretation can be given to the 0.1% VCM criteria. The 0.1% condensate of a 1-kg material would cover 100m² of surface with a uniform 10^{-6} g/cm² layer corresponding to about 20 monolayers and to a thickness of about 10^{-6} cm.

Material coatings of these dimensions can be detrimental for optical and thermal applications. Regardless of these considerations, the selection criteria appear to provide sufficient protection against the use of objectionable

materials. However, the test and its selection criteria do not provide direct data that a designer requires to ensure against contamination and the performance of certain systems. The designer must know the materials' outgassing rates as a function of temperature, time, and surface area and the condensation of this outgassing at temperatures other than 298 K (25°C). With this data, the designer can select the materials to use and can estimate the pressure versus time in compartments and the deposits and contamination which may occur on critical surfaces [4-6].

The criteria and the testing methods employed to qualify a material for space application have worked well and continue to do so. A large number of materials have been tested according to those criteria. Goddard Space Flight Center provides a website with the outgassing data for various materials [7]. ESTEC, located in the Netherlands, also reports data on materials outgassing according to the ASTM E-595 [8]. Other important characteristics of materials, such as flammability, toxicity, and thermo-optical properties are reported in other documents. This paper discusses the use of the TML and CVCM obtained from the ASTM E-595 tests for purposes other than general material selection for space applications. These standard test data have been used to get results about distribution, amount of contaminant deposits, and other data at temperatures and pressures other than those of the ASTM E-595 test.

The TML and CVCM have been employed to obtain the outgassing activation energy, the vapor pressures, and the outgassing rates of that material at different temperatures and as a function of time. These extrapolations have been used to compensate for the absence of difficult and expensive tests to obtain kinetic data on the materials. The purpose of this paper is to ward against the indiscriminate use of those data. As an example, the author was recently requested to estimate the gaseous release from a graphite/epoxy composite (see fig. 1) when heated at 121°C in a vacuum and to indicate the vent(s) needed to prevent excessive pressures within the confined volume containing the material. The available data on the material for the calculations were the TML from the E-595 test at 125°C.

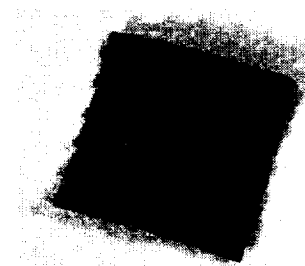


Figure 1. Graphite/Epoxy Weave Sample

No data were available on the rate, the temperature, and the length of time during which the major release of gas occurred. The TML from the many standard tests carried out on this material at 125°C for the 24 hours at 10^{-6} torr averaged about 1.8%. The author employed that value for the requested evaluation. Meanwhile, a fully operational Cahn microbalance that includes a residual gas analyzer and a quartz crystal microbalance became available at the author's laboratory. That facility was employed to test the composite material. The facility, which shows the real-time mass loss and the rate of mass loss as a function of time and temperature as seen in fig. 2, indicated that at 121°C for 24 hours, the %TML was in excess of 13% (see fig. 3). It also indicated that practically all of the mass loss occurred early

in the test and lasted for about 23 minutes. This TML result confirmed by many tests of the same material, indicated a much larger production of gas than that obtained using the E-595 result. As a consequence, a study was initiated to verify and compare the TML results given by the ASTM E-595 facility, the Cahn Microbalance, the Vacuum TGA, and the Knudsen Cell.

The analysis that follows is an attempt to obtain a better understanding of the parameters affecting the micro-CVCM tests. The analysis points to some of the reasons for the different results obtained for the same materials by different instruments. Most importantly, it gives some insight on the utilization of the test results for the use of the designer.

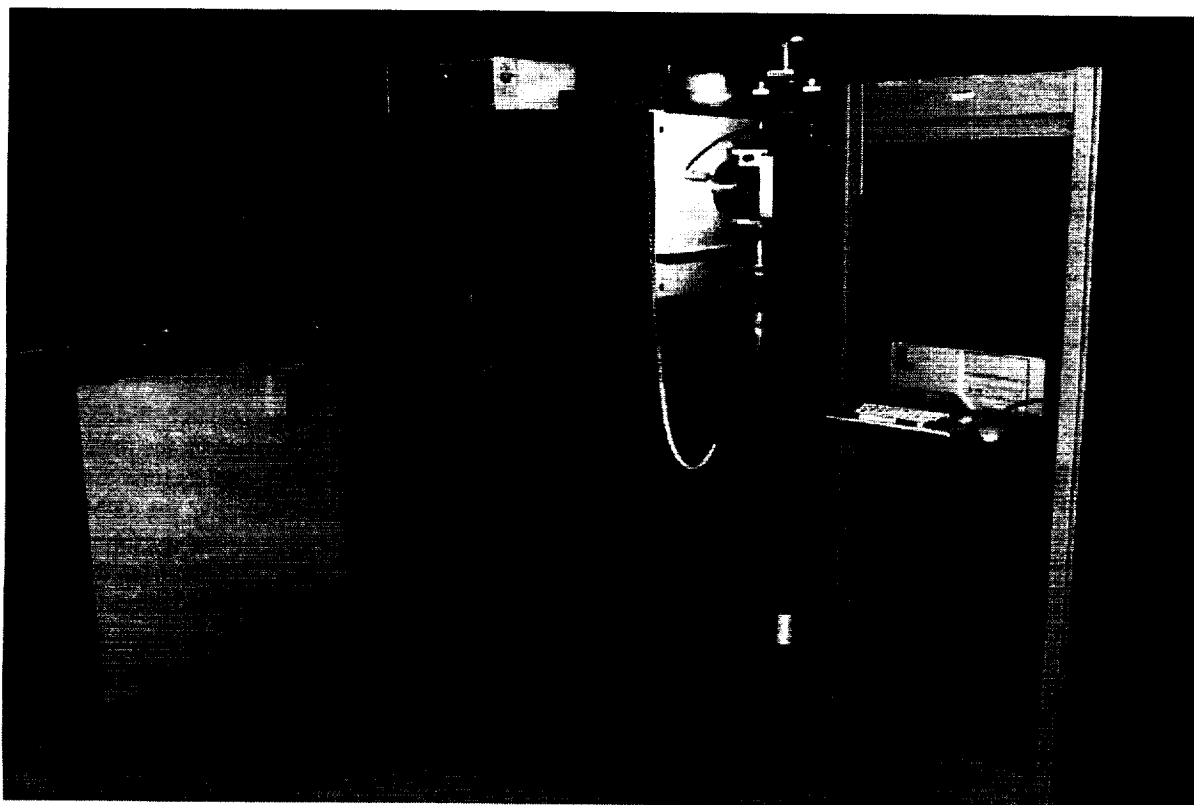


Figure 2. Cahn/QCM/RGA Outgassing Facility

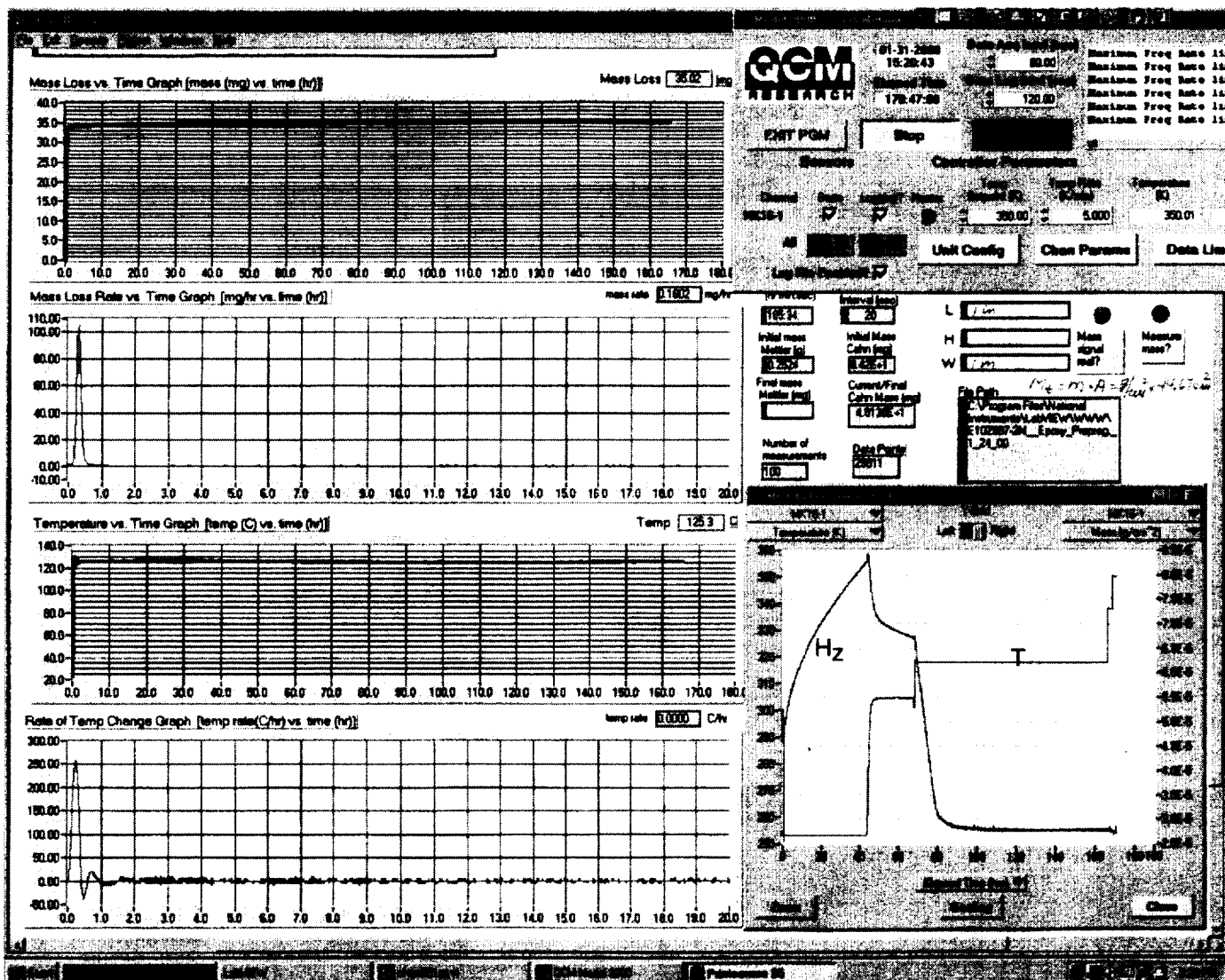


Figure 3. Graphite/Epoxy Outgassing Test

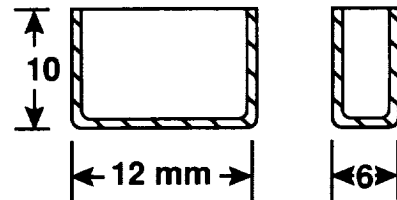
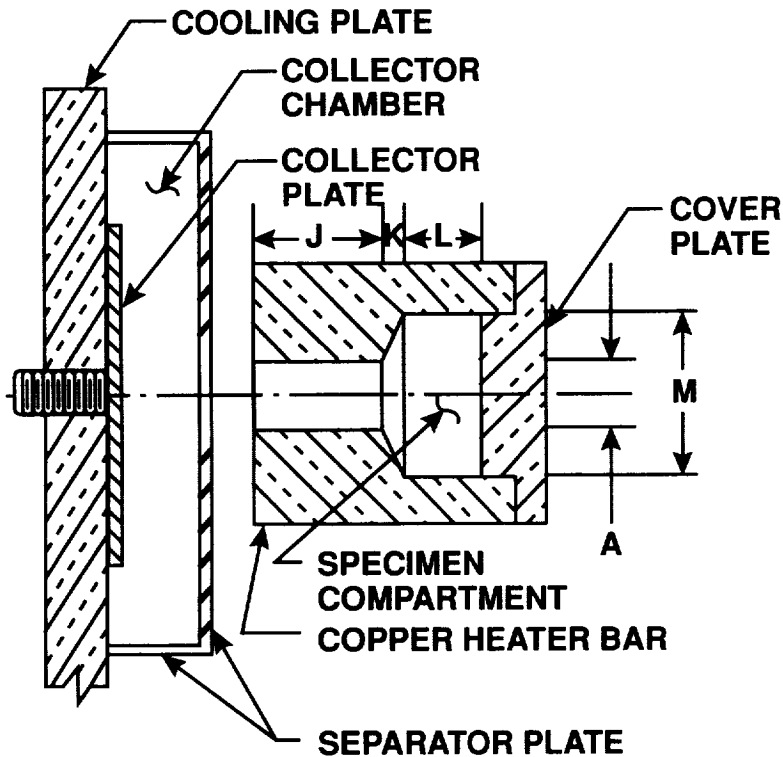
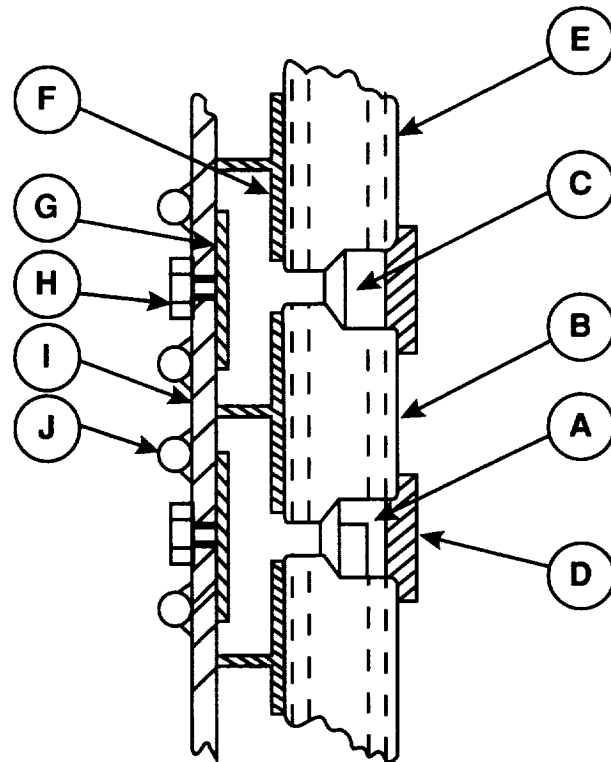
2.0 EXPERIMENTAL

Material tests according to the E-595 method are carried out by placing chips or thin slices of the material with thicknesses of less than 3.1 mm (1/8 in) in a 10x12x6-mm container (fig. 4). This container boat is placed inside a receptacle within a temperature controlling heated copper bar. The receptacle has a venting port facing a collector. For the test, the copper bar and the materials under test are held at 125°C and the collector at 25°C. The assembly is located within a vacuum chamber providing pressures of about 10^{-6} torr. The TML and CVCM are measured after the 24-hour test. Other facilities not of the E-595 design can provide direct measurements of the TML and CVCM for a 125°C/25°C, 24-hour test. These facilities provide results that are not affected nor controlled by the fixture design. Those

used here are a Vacuum Thermogravimetric Analyzer (TGA), a Cahn microbalance, and a Knudsen cell. The TGA provides an isothermal change of the mass of a small sample of material and records it as a function of time. The Knudsen cell holds a sample of material at a desired temperature. A collector surface facing the cell exit collects the mass lost from the sample at a desired temperature. The TML is obtained by measuring the weight of the sample before and after the test and then subtracting the final weight from the initial weight. The Cahn microbalance records the mass loss of the sample and the rate of change at the chosen temperature as a function of time. These three facilities were employed to obtain the TMLs of the sample materials at 125°C or at 121°C for 24 hours in vacuum and to compare the results.

NOMENCLATURE

- A - SAMPLE BOAT
- B - HEATER BAR
- C - SAMPLE COMPARTMENT
- D - COVER PLATE
- E - HEATER ELEMENT
- F - SEPARATOR PLATE
- G - COLLECTOR DISK
- H - RETAINING NUT
- I - COOLING PLATE
- J - COOLING COIL



SAMPLE BOAT

$$A_{\text{BOAT}} = 0.72 \text{ cm}^2$$

$$A_{\text{TUBE}} = \frac{4 D}{3 L} \cdot 0.31 = 0.2 \text{ cm}^2$$

$$A_e = \frac{A_{\text{TUBE}} \cdot A_{\text{BOAT}}}{A_{\text{TUBE}} + A_{\text{BOAT}}} = 0.15 \text{ cm}^2$$

A = 6.3 mm dia	J = 12.7 mm
M = 16 mm dia	K = 1.6 mm
L = 8 mm	

Figure 4. Micro-CVCM Apparatus

The results of the many tests done on the same material using three different facilities are shown in Table 1. The TML test results obtained with the E-595 averaged 1.67% with the exception of one test. The Cahn system results averaged 15.86% for a 6.45 cm² (1 in²) sample. Staking samples of the material resulted in smaller average TMLs: five-staked samples produced an average TML of 5.9%; four-staked samples produced an average of 7.23%; three-staked samples produced an average of 8.6%; a single sheet of material fitting the E-595 boat produced a 18.63% average TML. The results obtained from the Knudsen cell indicated a 16.94% average for a 6.45 cm² (1 in²) sample and a 6.1% TML for a single test consisting of 6 sheets. The TGA indicated a 15.7% TML for a single 1.5 cm diameter sample and 11.8% for a stack of three of the same samples. The table shows the

temperature, the initial mass m_o , the mass loss Δm for the 24-hour tests, and the resulting TMLs. A small symbol shows the sample arrangement.

The samples used in these tests came from the same batch of materials, but it may have been possible that some differences may have existed among them due to the non-uniformity of the epoxy coatings on the graphite filaments. With regards to the sizes and dimensions, those for the E-595 were in accord with the recommendations in the standard for the E-595 tests. For the Cahn and Knudsen tests, the samples were 1 in² (6.45 cm²). They were made of stacked layers of the same dimensions. The samples for the TGA tests were of round shapes made to fit the small instrument pan.

Table 1: Test Results of Mass Losses & TMLs of Graphite/Epoxy Weave

Test	T (°C)	A (cm ²)	m_o (mg)	Δm (mg)	TML (percent)	Sample Configuration
<u>ASTM E-595</u>	125	-	121.77	2.90	2.38	///
	125	-	118.90	1.97	1.65	///
	125	-	145.14	2.63	1.81	///
	125	-	140.25	2.16	1.54	///
	125	-	149.34	2.16	1.44	///
	125	-	266.73	3.25	1.22	///
	Average:		157.02	2.51	1.67	
<u>Cahn Microbalance</u>	121.0	6.45	243.00	32.58	13.40	□
	121.9	6.45	240.70	40.70	16.90	□
	122.5	6.45	240.90	39.54	16.40	□
	125.0	-	254.00	14.97	5.89	≡ : 5 pieces 0.95cm x 0.95cm
	125.0	-	126.90	9.70	7.64	≡ : 3 pieces 0.95cm x 0.95cm
	126.4	6.45	230.80	38.80	16.80	□
	125.0	-	243.80	41.39	16.98	: 3 pieces 2.54cm x 0.83cm
	125.0	-	256.00	24.47	9.56	≡ : 3 Pieces 0.95cm x 0.95cm
	125.0	-	103.36	7.47	7.23	≡ : 4 pieces 0.95cm x 0.95cm
	125.0	-	38.10	7.10	18.63	≡ : 1 piece 0.95cm x 0.95cm
	125.0	-	39.40	6.26	15.90	: 1 piece 0.95cm x 0.95cm
	Average (1cm ²):		238.85	37.89	15.86	
<u>Knudsen Cell</u>	125	6.45	251.20	41.03	16.33	□
	125	6.45	257.70	44.90	17.42	□
	125	-	218.50	13.32	6.10	
	Average (1cm ²):		254.45	42.96	16.87	
<u>Vacuum TGA</u>	125	1.53	16.90	2.66	15.70	○
	125	1.77	53.53	6.34	11.86	≡

3.0 ANALYSIS

Table 1 shows the average test results from the Cahn Microbalance, the Knudsen Cell, and the TGA, and compares them to the average TML result of 1.67% from the E-595 test. The average results from the Cahn Microbalance, the Knudsen Cell, and the TGA are 9.49, 10.10, and 8.25% higher, respectively, than those of the ASTM E-595. This indicates that the results from the non-E-595 facilities were on the average about 9.28% higher than those of the E-595. The justification for the difference could be found on the sample-exposed area and the mass released during the 24 hours. In the case of the E-595, the sample is within a boat and the outgassing leaves the cavity via a tubular passage. The flow must proceed through a series of passages from the boat and through the tube. The second consideration must be the nature of the flow, either molecular or viscous flow. The molecular density created in the cavity by the outgassing of the E-595 test dictates the nature of the flow. The mass flow from the other facilities is not restricted by the geometry or by the gas mean free path conditions above the sample. The samples are in an environment where the pressure is less than 10^{-6} torr and the flow of escaping gas is molecular. In both types of testing facilities, the quantity of released gas is affected by the staking and the percentages by the sample masses as shown by the results produced by the restrictions among layers.

On the basis of the above, two flow analyses have been considered for the E-595 system. First, the flow may be molecular. If so, the flow conductance is either a series conductance made up of the boat passage area and the tubular passage or the conductance is that of the tubular passage alone. Alternatively, the assumption that the flow is a viscous flow controlled by the tubular passage or by the boat open area. These alternatives have been compared to the losses indicated by the other facilities operating in the molecular region without area restrictions. The ratios of the mass losses from the two systems, E-595 and the others, are then compared to the TML ratios obtained from the tests.

In the systems other than the micro-CVCM, the outgassed molecules leave the surface of the sample according to the cosine distribution law. The mass loss in a time t from the material at temperature T is given according to the kinetic theory as

$$m = \frac{1}{4} \rho v A t \quad (1)$$

where ρ (g/cm³) is the mass density of the molecules moving with velocity $v = (3kT/m_m)^{1/2}$ where k (erg/K) is Boltzman's constant, T is the surface temperature in K and m_m (g/molec)

is the molecular mass [9-10]. The outgassing area A (cm²) is the exposed surface of the sample.

The outgassing releasing area for the sample in the E-595

$$m = \rho v A_e t \quad (2)$$

will be an equivalent area A_e resulting from the effective conductance of the passages. The mass loss for the cotinuum flow when the flow when sufficient gas is released is

For viscous conditions in the cavity, the tubular passage may control the flow. Its diameter d is 0.63 cm, providing an area 0.31 cm², and the length L is 1.27 cm. The open area of the boat is 0.72 cm², larger than that of the tube. A discharge coefficient can be applied to the tube passage. For an orifice, the discharge coefficient for viscous flow can be assumed to be 0.6. For the pipe, the Clausing factor α for molecular flow is approximately $4/3 D/L = 0.66$. The effective area A_e with $\alpha = 0.6$ can be taken as $0.6 \times 0.31 = 0.20$ cm². A possibility that the effective area for the flow out of the cavity is a series combination of the boat opening area and the tubular passage can be considered. In that case, the equivalent area of the exit is 0.31 cm², which modified by the Clausing factor or discharge coefficient, becomes $A_{tube} = 0.2$. The boat's equivalent area A_{boat} is 0.72 cm² and the series combination of the two can indicate an equivalent $A_e = A_{tube} A_{boat} / (A_{tube} + A_{boat}) = 0.157$ cm². Another alternative for the viscous flow is that the effective area is simply the open area of the boat (0.72 cm²).

In the following, the assumption has been made that for the micro-CVCM, the equivalent area A_e is 0.15 cm² and the flow is viscous. These assumptions have been verified by the calculations comparing mass losses and TML. The mass loss m_2 from the open systems tests will be expressed by the molecular flow leaving the exposed area A_2 of the sample during the time t_2 as

$$m_2 = \frac{1}{4} \rho v A_2 t_2 \quad (3)$$

and the TML when the initial mass of the sample is m_{20} by

$$TML_2 = \frac{m_2}{m_{20}} = \frac{1}{4} \cdot \frac{\rho v A_2 t_2}{m_{20}} \quad (4)$$

The results of the two types of tests have been compared as follows: for the same outgassing rates (ρv) released by the same material samples at the same temperature, the masses are equal when

$$A_e t_1 = \frac{1}{4} A_2 t_2 \quad (5)$$

The TML_1 and TML_2 are equal when

$$\frac{A_e t_1}{m_{10}} = \frac{1}{4} \cdot \frac{A_2 t_2}{m_{20}} \quad (6)$$

and the ratio of the TMLs is given by

$$\frac{TML_1}{TML_2} = \frac{4A_e t_1}{A_2 t_2} \cdot \frac{m_{20}}{m_{10}} \quad (7)$$

Using the value of 0.15 cm² for A_e, the ratio of the test times based on the TML value is

$$\frac{t_1}{t_2} = \frac{1}{4} \cdot \frac{A_2}{A_e} \cdot \frac{m_{10}}{m_{20}} = \frac{A_2}{0.6} \cdot \frac{m_{10}}{m_{20}} = 1.66 A_2 \frac{m_{10}}{m_{20}} \quad (8)$$

where the subscript 1 refers to the micro-CVCM and 2 to the open systems.

As an application, one may want to know when the mass losses are the same. For example, for testing a sample with an exposed surface A₂=6.45 cm² (1 in²) on the Cahn Microbalance for t₂=24 hours, the same mass loss will be produced with the micro-CVCM when t₁=1.66x6.45x24=256.9 hours and the TML will be the same when from equation 8, t₁=1.66A₂t₂(m₁₀/m₂₀)=256.9(m₁₀/m₂₀). Note that the times for the two tests to produce the same mass are nearly the same if A₂=0.72 (the area of the boat). Then for t₂=24 hours,

t₁=1.66x0.72x24 hours and if one makes the m₂₀=1.2m₁₀ the test times and the TML will be the same since (TML)₁/(TML)₂=4A_e/A(t₁/t₂)(m₂₀/m₁₀)=4x0.15/0.72x1x1.2=1.

Table 2 shows the test results for the graphite/epoxy material for a scotchweld 2216 sample and a jonal rubber sample. The calculated results for the scotchweld and jonal rubber were obtained using the micro-CVCM equivalent area of A_e=0.15 cm². The result for the graphite/epoxy was obtained using a continuum viscous (pvA) flow originating at the sample surfaces (i.e., A₁=0.72 cm² for the micro-CVCM and 6.45 cm² for the Cahn system) for both the open systems and the micro-CVCM. These assumptions were needed to justify the large difference in TML results obtained for this material. The justification of continuum viscous flow in both tests may reside in the sudden, short (about 25 minutes) release of the outgassing from the weave, as shown in figure 3. A test attempting to justify the TML difference based on the test time proved erroneously. The weave was tested for 150 hours using the micro-CVCM system and the results showed again that its TML was again about 1.6% as obtained for the 24-hour tests.

Table 2: Test Results & Calculated TML

Outgassing Facility & Test Material	Test Results:				Calculated t _c =t _m =24h
	A (cm ²)	m ₀ (mg)	t (hr)	%TML	%TML
1. Graphite/Epoxy					
Cahn Test	6.45	238.85	24	15.80	16.20
MicroCVCM Test	0.72	157.02	24	1.60	2.67
2. Scotchweld 2216					
Cahn Test	0.72	297.68	24	1.15	1.15
MicroCVCM Test	0.15	221.13	24	1.34	1.54
3. Silicone Rub. Jonal					
Cahn Test	1.00	1633.70	24	0.65	0.65
MicroCVCM Test	0.15	1200.30	24	0.36	0.53

Notes: general case $\frac{TML_1}{TML_2} = \frac{4A_1 t_1}{A_2 t_2} \cdot \frac{m_{20}}{m_{100}}$, A₁=A_e=0.15 cm²

for Graph/Epoxy $\frac{TML_1}{TML_2} = \frac{A_1 t_1}{A_2 t} \cdot \frac{m_{20}}{m_{100}}$, A₁=0.72 cm² (boat area)

4.0 CONCLUSIONS

The micro-CVCM test has been appropriate and effective for the selection of acceptable materials for space applications. It has protected against excessive outgassing and deterioration of materials when they are exposed to vacuum, excessive temperatures, and radiation in the space environment. It has provided indications on material stability and on its volatility.

Those test results, however, should not be used to quantify the outgassing and its condensable content or to perform estimates with regard to pressure conditions in and about a system. These conclusions have been confirmed by tests on several sample materials showing the difference in the results for the TML and the mass losses obtained using the micro-CVCM in comparison with those from the other facilities. They show that the micro-CVCM results can be considerably less than those from the other tests.

The difference in results has been attributed to the narrow exhaust passage and the variable flow, either viscous or molecular, that may exist within the micro-CVCM sample cavity.

The equivalent exhaust vent area resulting from the series combination of the boat vent area and the tubular passage of the cavity has been calculated to provide an effective area for the outgassing of about 0.15 to 0.20 cm². This is the effective, limiting outgassing surface area of the sample.

Simple relationships have been proposed for the equivalence of the results of the micro-CVCM and those of the open systems. The equivalence for the mass losses and the total mass loss percentages (%TML) have been based on a set of considerations. First, the effective exposed outgassing area of the sample in the micro-CVCM is 0.15 cm². Next, the flux of outgassing per unit of area (p_v) is the same since the sample materials and their testing temperatures are the same. In addition, the flow out of the micro-CVCM system is viscous due to the small dimensions of the cavity and the resulting small molecular mean free path offered to a reasonable outgassing quantity. Last, the flow leaving the high vacuum exposed surface of the samples in the open tests is molecular and the molecules leave according to the cosine distribution.

An exception to the consideration of the flow being molecular in an open system has been found for the case of the graphite/weave outgassing. In that case, the sudden and large loss of mass of the material in a few minutes has indicated a condition of continuous viscous flow for both the open and the micro-CVCM systems.

In general, the equivalence for the mass losses for the two methods of tests should occur when $0.15t_1 = 1/4A_2t_2$ and the

equivalence for the TML when $0.15t_1/m_{10} = 1/4m_{20}(A_2t_2)$. In these equations, the subscript 1 refers to the micro-CVCM and 2 to the others and m_{10} and m_{20} refer to the initial masses of the samples. The dimensional units are the same and the outgassing fluxes (p_v) in the equations have been cancelled out based on the considerations given above. The difference in the results obtained using the equations and those obtained by the Cahn Microbalance and the micro-CVCM were indicated.

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