Feature Article

Building a Primary Mass Flow Standard, “PMFS”

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This paper recounts the evolution of a design for a primary mass flow standard (PMFS) targeted at measuring flows down to the 0.0012 grams per minute[*1] level of gas with a target accuracy of 0.1% of reading. It describes the basic concept of direct mass-flow measurement, identifies external factors that can corrupt the mass measurement and recounts system-design revisions intended to reduce the corrupting influences. A formal uncertainty analysis of the PMFS is not presented as the project is still evolving. However the core task of achieving the needed PMFS stability and noise levels at low-flow levels is discussed for a series of four evolving prototypes, each showing continuing performance improvements.

*1: 0.0012 Grams per minute is equals 1 standard cubic centimeter, 1 sccm, of N_2. A sccm is a mass flow term equal to one cubic centimeter per minute of a gas at 0 °C and 1 atm.

Introduction

Why Mass Flow Measurement?

Mass flow measurement is an enabling technology at the base of many industries from solar cell fabrication to process control in refineries. Critical industrial processes often involve chemical and physical reactions that require specific and repeatable amounts and/or ratios of different gas species to be present for proper reactions to occur.

HORIBA’s Reno facility is the U.S. flow metrology center for measuring the mass flows of gases used in semiconductor fabrication. These mass-flow measurements are used to characterize and validate product designs and calibration practices of the Mass Flow Controller products of HORIBA’s Semiconductor Division. The PMFS provides a primary standard for these mass flow measurements, augmenting Reno’s existing transfer standards[*2]. The target mass-flow measurement range for this first PMFS is 1 standard cubic centimeter per minute, “1 sccm”, to 3,000 sccm on N_2.

Basic Concept of the PMFS

The basic approach to direct mass-flow measurement is illustrated in Zone #1 of Figure 1 below. An electronic balance, “a scale”, is used to measure the force of the pull of gravity on the mass of a cylinder containing a gas. As gas is removed from the cylinder, the force on the scale reduces. By recording the scale readings over time, one can directly measure the mass flow rate out of the cylinder.

Corrupting Forces

This direct mass flow rate measurement approach is simple theoretically; however, successful implementation of the simple theory requires isolating the changing weight signal of gas leaving the scale from other corrupting forces and complicating factors that can mask the flow-induced changing weight signal. Specific details on these factors are described below.

- Corrupting factor #1: Helium balloons are not the only things that try to float.

  The buoyancy force on an object roughly equals the weight of the air an object displaces. A closed 1 liter

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*2: The main transfer standard used is a rate of pressure rise system, dP/dt, based on the ideal gas law, modified for non-ideal gas behaviors. This system has been successfully running process gases for 15 years.

cylinder has a nominal 1.2 grams buoyancy force.
At the target 1 sccm N_2 flow, the PMFS will need a stability of 0.0000012 grams per minute. Buoyancy force fluctuations from natural atmospheric changes dwarf this need.

- Complicating factor #2: Scale Resolution.

Compared to the cylinders and tubing needed to contain a gas, 1 scc of gas does not weigh much. Scale resolution becomes increasingly coarser, as a percent of full scale, as the size of the scale increases to accommodate gas-containment hardware.

- Complicating factor #3: Gravity is not the same magnitude at all locations.

Scales measure force, not mass. The force measured by a scale is the product of the local gravity and the mass on the scale. The gravity is a function of location and elevation. Reno's elevation, the location of the PFMS, is at 1500 meter above sea level and gravity is roughly 0.03% less than most locations.

- Corrupting factor #4: Electrostatic forces.

Initially, a non-electrically conductive cover was used with a prototype PMFS system. If rubbed, a static charge would build up and exert forces on the items on the scale. Over time, this force diminished as the charge dissipated.

Off-Scale Volumes

In addition to addressing the complicating factors that can corrupt the weight measurement from the scale, one must also realize that some portion of the plumbing delivering the gas from the on-scale cylinder to a device being calibrated will be off of the scale. This "off-scale" volume can induce flow errors unless properly addressed.

The prototypes described in this paper use an on-scale cylinder to feed gas to the off-scale plumbing*3 associated with a Device-Under-Test, "DUT" via a flexible 1/32" I.D. tube as shown in Figure 1. With this configuration, the scale directly senses the mass (M1) in the cylinder (Zone 1), partially senses the mass (M2) in the 1/32" ID tubing (Zone 2) and does not sense the mass (M3) in the off-scale volume (Zone 3). Accordingly, the scale can be used to directly measure the mass flow ($\dot{m}_1$) leaving the cylinder; however the mass flow through the DUT ($\dot{m}_3$) will be different than m_1 if the mass in volumes 2 and 3 change with time. The following describes the mechanisms that can cause the mass in these volumes to change and discusses countermeasures to minimize their effect on the accuracy of the DUT measurement.

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*3: Fittings, pressure transducers, valving and the DUT's own internal volume.

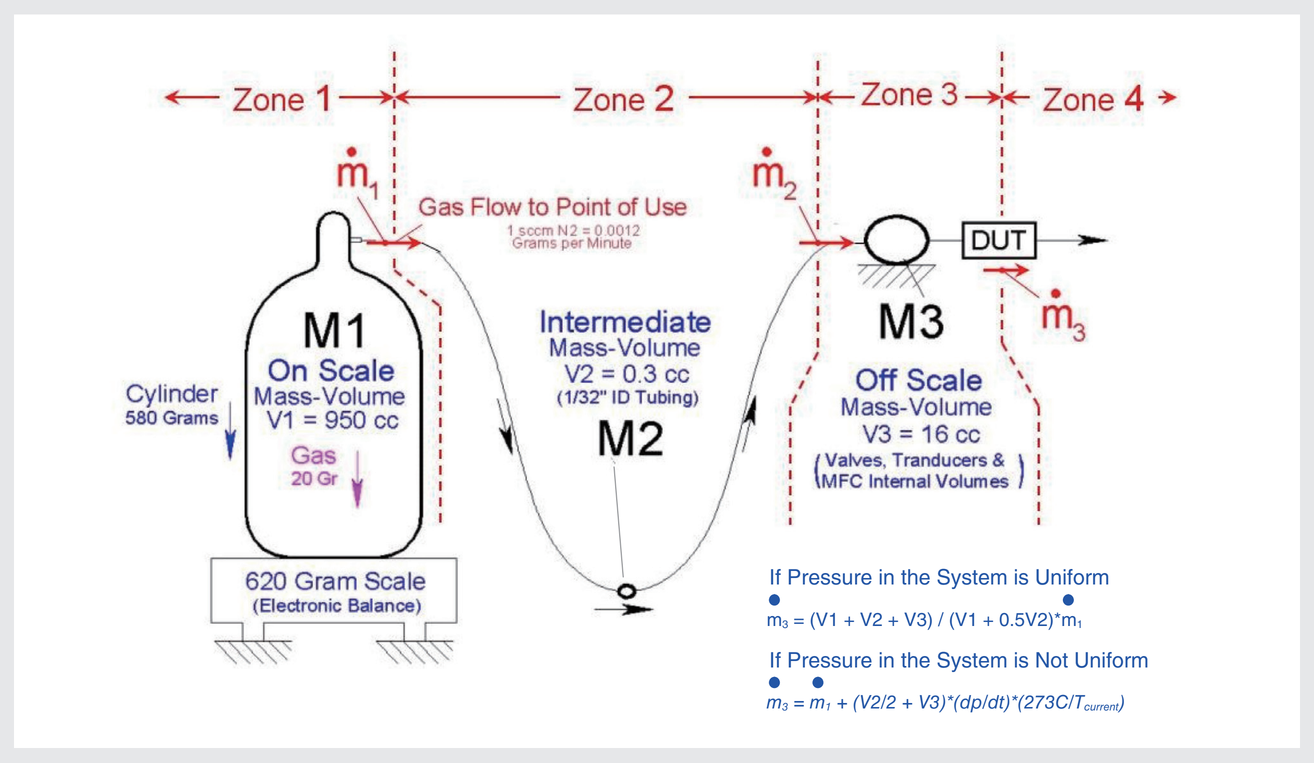

Figure 1 The Basic PMFS Concept and the Effect of Off-Scale Volumes

If one makes the simplifying assumption that pressure in the total system, V1, V2 and V3 from Figure 1 is uniform[*4], then the percentage of the total system mass that is in each volume is proportional to the volume of each V1, V2 and V3 and is independent of the system pressure. If so, the flow through the DUT ($\dot{m}_3$) will draw mass evenly from both the on-scale and off-scale volumes. Accordingly, the flow based solely on the readings from the scale ($\dot{m}_1$) will understate the flow through the DUT ($\dot{m}_3$) by the ratio of the on-scale volume to the total system volume. The relationship is roughly:

$\dot{m}_3 = (V1 + V2 + V3) / (V1 + 0.5V2)*\dot{m}_1$

For the Figure 1 Configuration, this represents a 1.7% understatement.

$\dot{m}_3 = (950 + 0.3 + 16) / (950 + 0.3/2)*\dot{m}_1 = 1.017\ \dot{m}_1$

Countermeasures and discussion

1. Minimize the off-scale volume by component selection and placement of the DUT.
   - This directly reduces the magnitude of the needed correction to the $\dot{m}_1$ flow.
2. Determine the on-scale and off-scale volumes and apply a fixed correction to $\dot{m}_1$ to determine $\dot{m}_3$ as shown above.
   - This fixed correction induces errors as the pressure drop in the tube becomes sufficient to affect the uniform pressure in the system assumption.
3. Measure the pressure in the off-scale volume and utilize the ideal gas law to calculate the mass being drawn from the off-scale volume and add it to the on scale flow, $\dot{m}_1$. An application of this correction is summarized below.

i.e.: $\dot{m}_3 = \dot{m}_1 + (V2/2+V3)*(dp/dt)*(273C/T_{current})$

The PMFS of this paper is instrumented to employ this correction. If the error of the correction is limited to 2% of reading[*5] when correcting the ~1.7% off-scale volume effect, the net induced total-system error would be 2.0%*1.7% = 0.034% roughly.

4. Install a regulator on the outlet of the on-scale cylinder to keep the pressure in the off-scale volume at a near constant pressure for steady state flows.
   - If the regulator performs ideally, and provided a constant outlet pressure, the off-scale volume effect would be totally eliminated. However, regulators are not perfect and the outlet pressure of regulators will creep up as the supply pressure to the regulator decreases as gas is removed from the on-scale cylinder source. This effect is illustrated in Figure 2. The addition of the regulator reduces the off-scale volume effect by a factor of 70. Figure 2A below illustrates the real world performance of the regulator on the prototype.
     - After flow begins, the outlet pressure from the regulator to the DUT creeps up 1 psi for every 70 PSI the cylinder pressure supplying the regulator drops.
     - When the cylinder pressure reaches the regulator outlet setting, the regulator can no longer control its outlet pressure and the pressure to the DUT drops rapidly with the cylinder pressure.
   - The improvement of adding a regulator can be combined with the correction method of item #3, to further reduce the magnitude of the effect. Figure 2B shows the scale flow, $\dot{m}_1$, and the DUT flow, $\dot{m}_3$ ($\dot{m}_1$ + ideal gas law correction) over the period when the on-scale cylinder pressure starts to drop to the regulator outlet pressure setting, time = 212 minutes, and the regulator looses it’s capability to maintain a constant pressure in the off scale volumes. As can be seen the constant flow through the DUT is reflected by the m1 flow + the dP/dt correction.

[*4] This assumption is practical for small flows. For larger flows, pressure drop through the tube becomes significant and the assumption becomes invalid.

[*5] This 2%R error estimate is 4 times larger than our experience with our current rate of rise systems currently operate at the Reno facility.

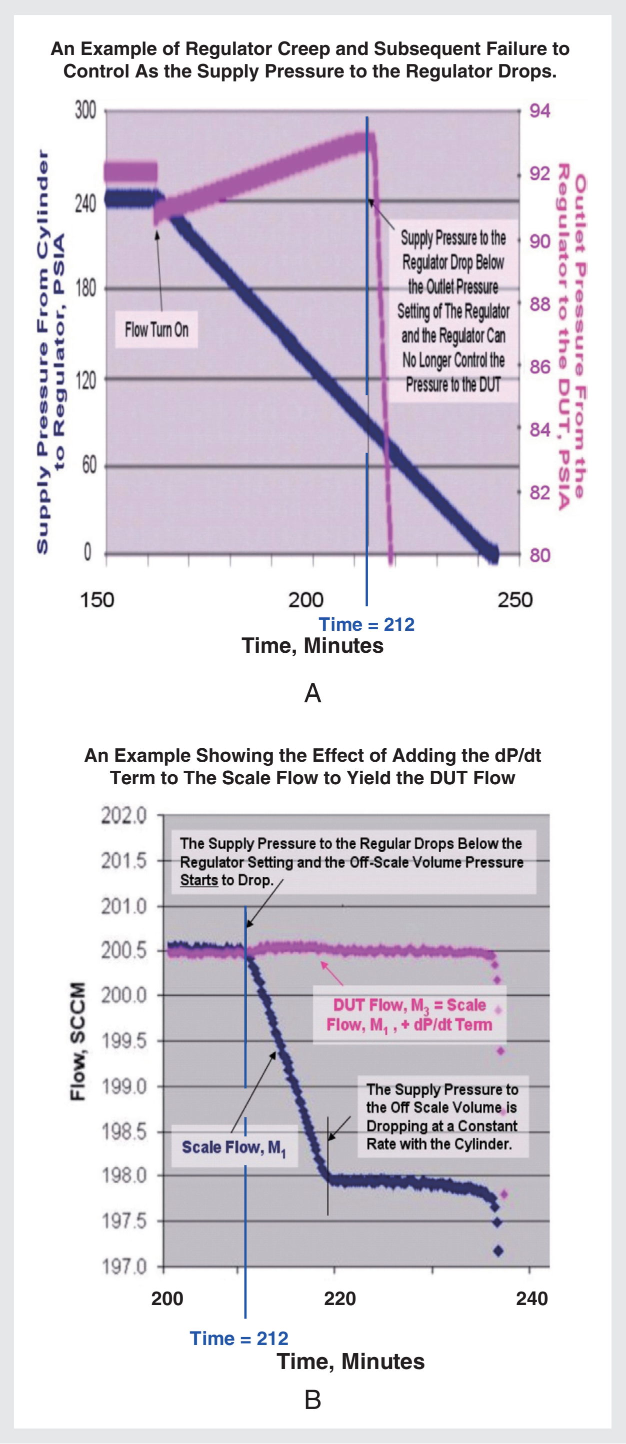

Figure 2 Regulator Creep, Its Effect On the Flow Calculation and a Correction Method

Prior Art

The authors are aware of three previous PMFS systems and two related mass measurement devices. The first was a Unit Instruments effort in the 90's. This system was a simple cylinder on scale system that addressed the corrupting force issues discussed above by taking the required time needed for the totalized gas mass that leaves the scale to grow sufficiently large, relative to the corrupting forces, to allow the system attain the desired accuracy. Unfortunately this approach requires long time periods to achieve accuracy for low flows. The Unit system reportedly required 6 months for a flow measurement at 1 sccm N_2.

In the late 90's a PMFS was built at the Oak Ridge National Laboratory, ORNL. It consisted of a ridged large chamber, suspended from a wire attached to a scale, while floating at slightly negative buoyancy in a large water bath. Gas from a DUT would flow into the chamber increasing the weight sensed by the scale. This "submarine" approach addressed the effects of changing air density and allowed a small scale with higher resolution to be used.

In the last 4 years the authors became aware of a PMFS commercially offered[*6] by DH Instruments. Like the previous Unit Instrument effort, it also consisted of a cylinder-on-scale configuration, but it utilized lighter components and took measurements to determine air density and numerically compensated for the sensed buoyancy effect. The speed of this system at lower flow was markedly faster than it predecessor. Customers indicate a 10 sccm flow reading can be taken in 10 hours.

During the early phase of this Horiba effort, the authors searched for the highest resolution electronic balance capable of a 1 kg payload. They found a class of scales called mass comparators. One version commercially offered[*7] by Mettler Corporation weighs an object placed in a vacuum chamber to negate the effect of changing buoyancy forces corrupting the weight measurement.

The HORIBA Effort

Four prototypes were built during HORIBA's PMFS effort. Each subsequent prototype benefited from the performance lessons learned from its predecessors. Figure 3 illustrates the four prototypes along with their concept and performance results.

The authors' first prototype was chosen based on the perceived successes of the Oak Ridge PMFS. A prototype, built utilizing the "submarine" approach, consisted of a cylinder suspended in water by a wire hung from a scale. The intent of the prototype was take a preliminary look at the external forces influencing the

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*6: The price for the DHI system plus associated instrumentation approaches $250,000.

*7: The price for the Mettler system plus associated instrumentation was $500,000.

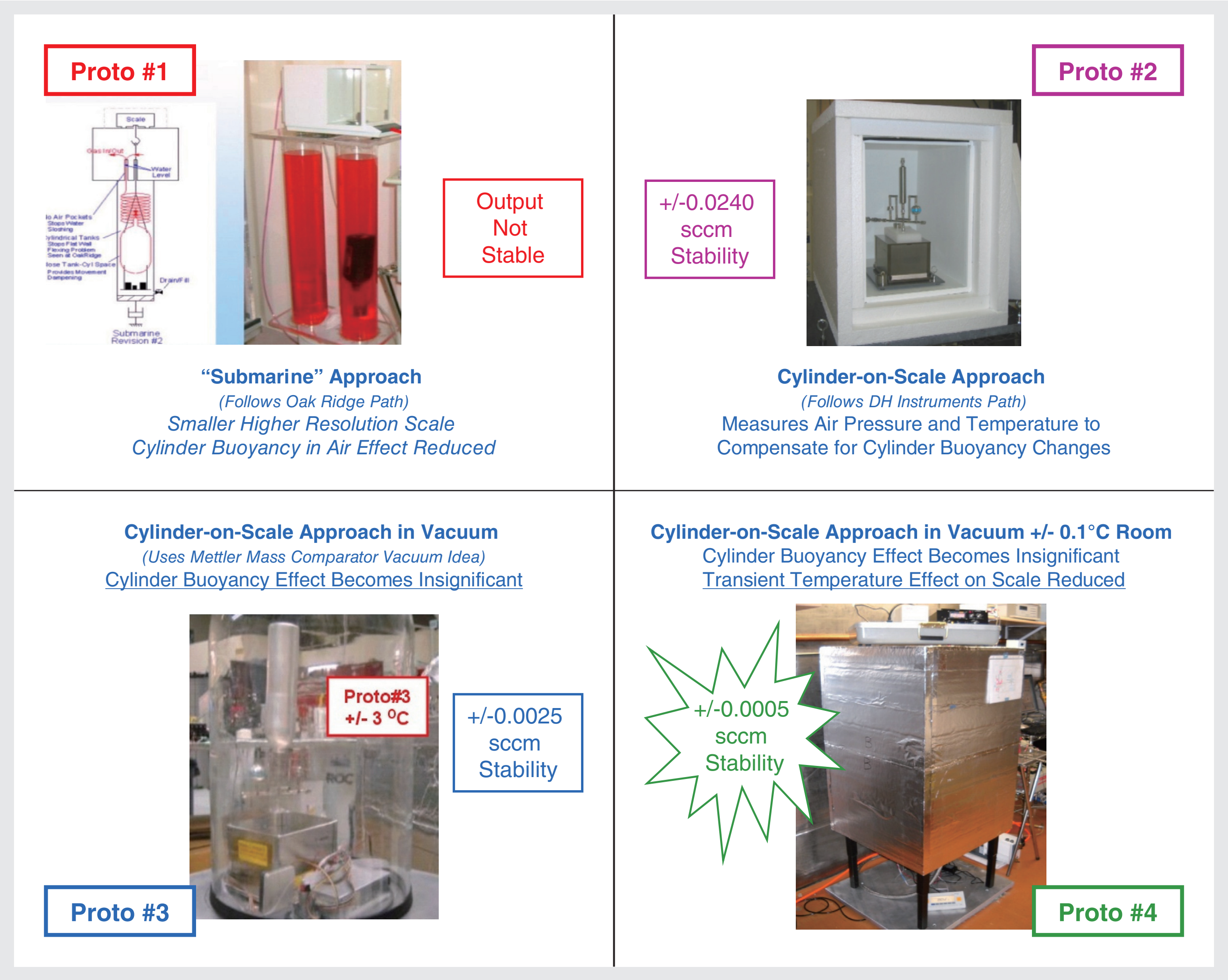

Figure 3

scales zero reading under a no-flow condition rather than to actual measure flow.

From prototype #1 the following was observed.

- Ambient temperature greatly influenced the zero of an unloaded scale.
- Placing the scale in a +/- 0.1 °C temperature-controlled room helped but did not reduce the noise to an acceptable level. It never achieved stability in a multi-day run.
- Corrosion products*8 developed on the aluminum tank prototype, changing its effective volume and changing the buoyancy force.
- Antifreeze for aluminum engines was added to the water as a counter measure for the corrosion issue:
- The density of the fluid increased requiring additional ballast to be added to tare the increased buoyancy. As the water in the antifreeze mix evaporated, the density of the remaining fluid increased changing the buoyancy force.

These initial difficulties, combined with an aversion to the task of maintaining a wet system, pushed the authors into investigating the next approach before pushing further to refine the submarine approach.

The second HORIBA prototype followed the DH Instrument’s cylinder-on-scale configuration. The PMFS operated with two different cylinder sizes, a 950 cc and a 50 cc cylinder.

From prototype #2 the following was observed.

- The system with the 1 liter cylinder displayed a +/- 0.0240

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*8: Or some other undefined phenomenon.

sccm equivalent flow window over the 6 day test.

- The smaller 50[*9] cc cylinder displayed a +/- 0.0025 equivalent flow window over the same period. Unfortunately, using the smaller 50 cc cylinder reduced the higher flow capability more than the authors' desired.
- Numerical corrections for the external forces using pressure, temperature and relative humidity measurements removed roughly half the effect of the external forces.
- Numeric efforts which adding the time-based derivative of the temperature signal were more effective.

Prototype #2's early results, +/-0.0240 sccm N2 equivalent, while promising, were still markedly above the evolved target of 0.0005 effective sccm N_2 flow noise. The authors choose to abandon the DH Instrument's approach of sensing and numerically compensating for changing air density affecting the buoyancy force. Rather for Prototype #3, the authors followed the Mettler mass comparator's direct, brute force method of removing the atmosphere around the system to eliminate corrupting buoyancy effect.

Prototype #3 placed the hardware of prototype #2 in a bell jar equipped with a turbo-molecular pump to maintain a 1 Pa vacuum around the system. A metal skinned foam housing was added to provide insulation, electrical grounding and IR shielding to buffer the system from ambient temperature changes and prevent electrostatic charge build up.

From prototype #3 the following was observed.

- The system with the 1 liter cylinder displayed a +/- 0.0025 sccm flow window over the 8 day test, a 10X improvement over the prototype #2 performance.
- The remaining flow variation correlated roughly with laboratory temperature.

The rough correlation between the flow variation and temperature can bee seen in Figure 4; however a numeric correction for a short data run did not seem practical. In addition, another 5X reduction in flow variation was needed to meet the initial target of 0.1% accuracy at 1 sccm N2 flow. To reduce the temperature induced effect, prototype #4 was placed in a used transoceanic shipping container modified to control temperature to +/- 0.1 degree C.

Prototype #4 was operated in two different configurations. In the first configuration, the PMFS has no cylinder installed and there was no mass flow, just a 10 gram weight. In the second case the 1 liter cylinder is installed and a flow rate of roughly 0.11 sccm is being consumed by a DUT.

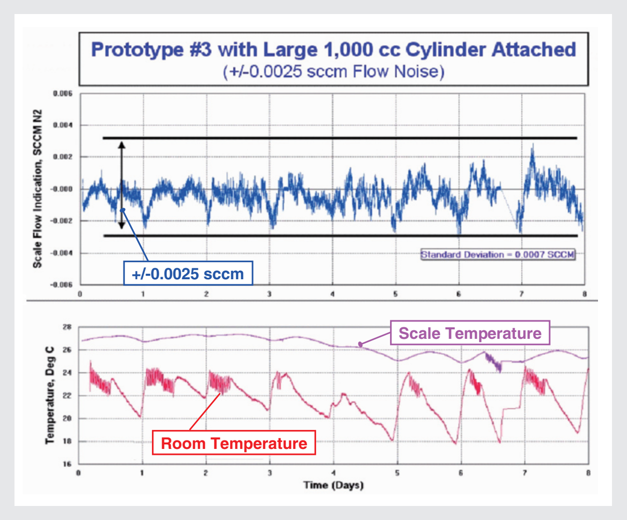

Figure 4

From prototype #4 the following was observed. See Figure 5.

- In both cases, (1) with a 1 liter tank and 0.11 sccm flow to a DUT and (2) without tank and no flow, the PMFS flow window was on the order of +/- 0.0005 sccm and no baseline trend was observed.
- In addition, the period of the flow indication's "wandering" in both cases appears similar.

The act of placing the PMFS in a vacuum and in a +/-0.1 C environment appears to have achieved the target 0.0005 sccm noise level[*10]. At this point the author's moved away from the task of reducing external noise and moved on to

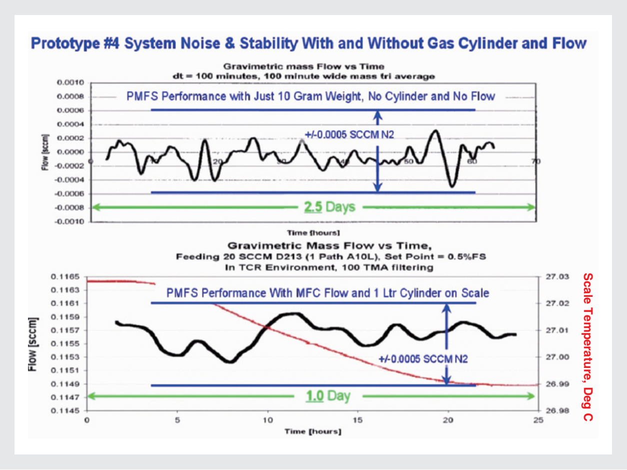

Figure 5

[*9] The better stability numbers for the smaller cylinder did suggest that buoyancy forces were still significant.

quantifying the off-scale volume’s performance at higher flow rates and NIST traceability.

*10: Based on the similar operating results, noise magnitude and period of wandering, with and without a cylinder attached to the scale, the authors concluded that that the residual noise was not buoyancy related and ended the effort to further reduce this external force.

NIST Traceability

The primary path back to NIST for this PMFS is through a 0.5 gram OIML class E1 and a 10 gram OIML class E 2 weight. These weights are serialized and traceable back to NIST. A second set of weights are installed on small robot arms in the vacuum chamber of prototype #4. These “working” weights can be remotely hung on and removed from the gas cylinder to periodically check the scale’s calibration. See Figure 6.

Information on the scale’s span and zero stability can be inferred from repeatedly placing a weight on and off the cylinder attached to the scale. Figure 8 below illustrates the scale output for the load-unload cycles continuously performed over 18 hours during system cool down after initial evacuation pumping*11.

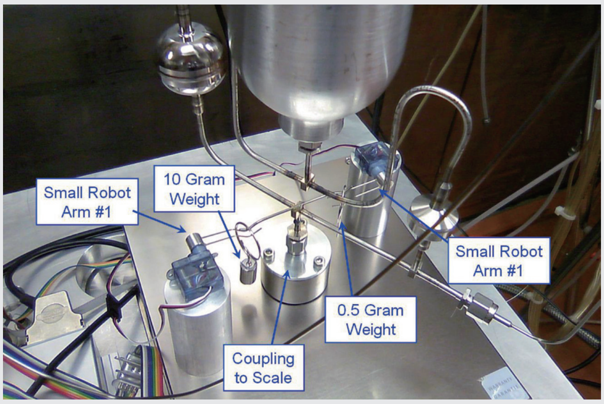

Figure 6

Selecting the cool-down period after initial system pump was intentionally chosen to be illustrative of the effect of temperature on the scale’s span and zero. It can be seen that the zero of the scale is affected by the temperature transient; however, the span of the scale is not so affected. During-steady state operation the scale’s temperature gradients is on the order of 0.01 °C per hour within a fixed band of 0.1 degree C maintained in the shipping container. This steady-state operation temperature transient, 0.01 °C per hour, is 70 times more stable that seen in Figure 7 and is sufficient to maintain a stable scale zero.

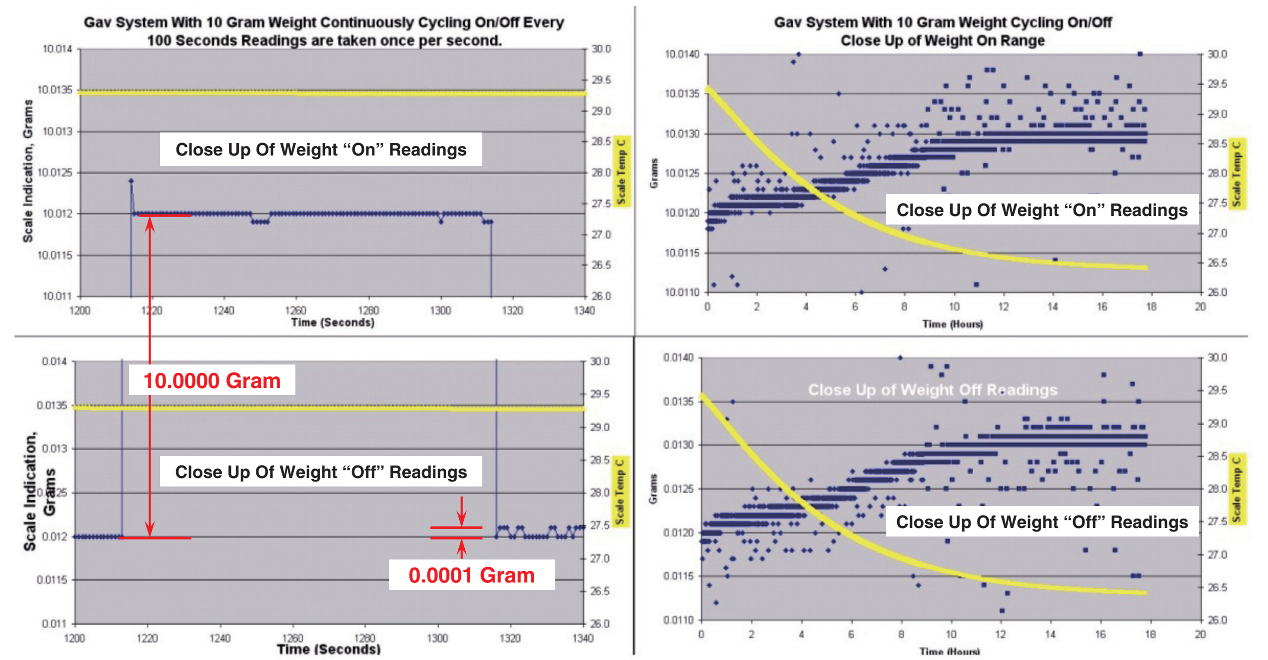

Figure 7

[11] During system evacuation a turbo-molecular pump mounted directly on the aluminum base plate of the system is powered adding heat to the system. After an acceptable vacuum is reached this pump is turned off and a remote pump is used to maintain the desired vacuum level. The system then cools to a lower steady-state temperature.

Conclusion

The final prototype Primary Mass-Flow Standard appear to be practical to measure flows near 1 sccm in a 100 minute data run. From our evolving series of PMFS prototypes, we found the insulated vacuum chamber placed in a temperature controlled shipping container allowed us to reduce external noise on the system to a window of +/- 0.0005 sccm. We believe this level is adequate to support the lowest flow levels currently used in the semiconductor and related industries.

After exploring and validating the system performance for the upper-flow limits[12] of this PMFS, estimated to be roughly 3,000 sccm, the authors will begin a 2nd PMFS targeted at measuring flows up to 200,000 sccm. It is anticipated that developing countermeasures to address momentum and thrust issues from the higher flow rates will be the primary technical challenge in the next device.

[12] Clock timing validation will become more important and quantification efforts will be required on time measurement.

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