

NIST Special Publication 400-101

**Workshop on
Mass flow measurement and control
for the semiconductor industry**

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Process Measurements Division*

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Executive summary

On 15-16 May 2000 at NIST, 45 scientists and engineers met to identify research and standards that will benefit users and manufacturers of mass flow controllers (MFCs) and related equipment. Most attendees represented companies closely associated with the semiconductor industry, including manufacturers of MFCs, of process tools, and of semiconductor devices. They were asked to:

1. Identify the technical problems limiting the productivity of the U.S. semiconductor manufacturing industry.
2. Prioritize the ways these problems can be resolved using NIST's assistance.

Brief presentations were given and lengthy discussions were held on the following topics:

1. Flow meter performance.
2. Standards and calibration.
3. Gas properties.
4. Alternatives to thermal mass flow controllers.

The attendees proposed 21 tasks directed at the identified problems. A subsequent vote identified the seven strongly recommended tasks listed below.

Strongly recommended task	Institution
1 Devise a technique to verify MFC performance that is independent of the process chamber.	none specified
2 Characterize the performance of each new MFC with nitrogen as well as with its nameplate gas.	MFC manufacturers
3 Increase the range of transfer standards for conducting round-robin tests (0.01 sccm to 1000 slm).	NIST
4 Improve the primary (0.025%) and transfer (0.1%) standards for gas flow.	NIST
5 Expand and reprioritize the list of gases to be studied. Schedule and conduct property measurements.	NIST
6 Establish and maintain a public, Web-based database of gas properties.	NIST
7 Develop metrology to characterize liquid flow controllers.	NIST

These recommendations will help NIST guide its research on gas properties, flow standards, and flow measurement techniques.

Introduction

Flow measurements are central to the manufacture of semiconductor devices, especially in chemical vapor deposition and plasma etch processes. A mass flow controller's performance affects production costs in at least two ways. Irreproducibility of the MFC increases the product's defect rate, and inaccuracy of the MFC increases the time required to copy a process recipe from one process tool to another. As shown in the table below, five of the technology working groups for the 1999 International Technology Roadmap for Semiconductors raised issues related to MFC performance.

Working group	Issues related to mass flow control
Design	<ul style="list-style-type: none">• Uncertainty due to manufacturing variability
Front end processes	<ul style="list-style-type: none">• Control boron penetration• Achievement of lateral and depth abruptness• Etch CD control and selectivity• Sidewall etch control
Interconnect	<ul style="list-style-type: none">• Combinations of materials...• Low plasma damage...• As features shrink, etching and filling high aspect ratio structures will be challenging...
Factory integration	<ul style="list-style-type: none">• Control production equipment and factory processes to reduce parametric variation• Minimize waste and scrap and reduce the number of nonproduct wafers
Defect reduction	<ul style="list-style-type: none">• Advanced modeling (chemistry/contamination), materials technology, software and sensors are required to provide robust, defect-free process tools ...

All MFCs require both a *model* and a *calibration*. The model, which depends on the MFC's design, relates the raw output, which might be in volts, to the final output, which is in units of flow. The model cannot account for differences between MFCs caused by manufacturing variations. The calibration, which is done for every MFC, accounts for these differences.

The great variety of fluids used in semiconductor processing challenges the model. More than 30 gases are in routine use, and the continual introduction of new processes is adding liquids as well as gases to this list. Not only must the model be sufficiently

general to accommodate different fluids, but also it must have accurate thermodynamic and transport property data for each fluid.

The variety of fluids challenges the calibration also. Calibrations frequently use a benign surrogate gas instead of the hazardous process gas for which the MFC is intended. With the use of a surrogate gas, an error in the MFC model or in the process gas's properties leads to an error in the calibration.

In addition to accurate property data, good models and calibrations require accurate flow standards. An MFC model can be tested only to the accuracy of the standard, and a calibration requires a flow standard whose accuracy exceeds that required of the MFC.

NIST has a long history of providing accurate, unbiased measurement standards and property data. In response to the semiconductor industry, NIST recently established programs to measure the properties of semiconductor gases and to extend gas flow standards to lower flow rates. The workshop's recommendations will help NIST guide its research on gas properties, flow standards, and flow measurement techniques.

Acknowledgements

Gil Yetter of International Sematech introduced us to engineers concerned with flow measurement in Austin, provided a large list of other contacts, and gave us constant encouragement. We thank him also for the encouragement he gave to the workshop attendees. We thank Lori Phillips Buckland of the NIST Public and Business Affairs Division for handling many essential administrative tasks. Planning for the workshop began under the direction of the recently retired chief of the Process Measurements Division, Greg Rosasco. We thank him and the present division chief, James Whetstone, for their support. Most of all, we thank the speakers, the session leaders, and the other participants, for the time, expense, and effort they generously gave.

Funding for the workshop's planning came from the Process Measurements Division (PMD) and the National Semiconductor Metrology Program (NSMP). The PMD develops and provides measurement standards and services, measurement techniques, recommended practices, sensing technology, instrumentation, and mathematical models required for analysis, control, and optimization of industrial processes. Work supporting flow measurements takes place in the PMD's Fluid Flow, Fluid Science, and Pressure & Vacuum Groups. The NSMP is a NIST-wide effort designed to meet the highest priority measurement needs of the semiconductor manufacturing industry and its supporting industries. The National Semiconductor Metrology Program supports a broad portfolio comprising 40 semiconductor metrology development projects at NIST.

Further information

National Institute of Standards and Technology
Process Measurements Division
National Semiconductor Metrology Program

<http://www.nist.gov>
<http://www.cstl.nist.gov/div836/>
<http://www.eeel.nist.gov/omp>

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Workshop objective and format

The objective of this two-day workshop was to identify research and standards that will benefit users and manufacturers of mass flow controllers and related equipment. The attendees were asked to:

1. Identify the technical problems limiting the productivity of the U.S. semiconductor manufacturing industry.
2. Prioritize the ways these problems can be resolved using NIST's assistance.

As shown in the table below, most of the workshop attendees represented companies closely associated with the semiconductor industry. The NIST attendees were from the Process Measurements Division or the Office of Microelectronic Programs. Appendix E lists the names and addresses of the attendees.

Type of institution	Attendees
MFC manufacturers	13
semiconductor tool manufacturers	3
MFC users in semiconductor industry	4
MFC users in other industries	2
other semiconductor flow measurement companies	8
semiconductor consortia (International Sematech)	2
independent consultants	4
NIST	7
other federal laboratories (Oak Ridge National Lab)	2
TOTAL	45

Appendix D gives the workshop schedule. On the morning of the first day, brief talks set the context for the workshop's four topics:

1. Flow meter performance.
2. Standards and calibration.
3. Gas properties.
4. Alternatives to thermal mass flow controllers.

That afternoon, the attendees divided into four groups corresponding to these topics. In each group, the attendees addressed the following questions:

- What are the present requirements and how well are they realized?
- How will the requirements change over the next ten years?
- How can national laboratories such as NIST best assist industry?

On the morning of the second day, the attendees met to discuss the results from each group. They then divided for a second, brief breakout session. That afternoon, each breakout group presented up to six proposed tasks. Proposals presented by more than one group were combined. The attendees then voted on the importance of the tasks.

Presentation abstracts

Mass flow controller performance and characterization

Gary Allen
Applied Materials

The presentation focuses on the characteristics, requirements, gases, and types of mass flow controllers utilized currently in the semiconductor industry. The importance of flow control in semiconductor equipment processes is paramount to the capability, repeatability, and manufacturing of integrated circuits. MFCs (mass flow controllers) are controlled by both analog and digital connections, some digital MFCs via a standard protocol.

The transient characteristics of gas flow into sub-atmospheric pressure chambers are important in the overall understanding of semiconductor processes. Some of these characteristics are dead time, step response time, settling (control) time, overshoot, repeatability and valve leak by. Other characteristics of accuracy, linearity, reproducibility, and zero offset are also necessary in understanding the behavior of MFCs.

In today's semiconductor industry different types of MFCs are becoming prevalent. The most common variety in the industry are thermal-based MFCs. Pressure-based MFCs are finding applications in semiconductor processes. Two types of flow sensors which are utilized in other industries are Coriolis and MEMS-based sensors. Although not fully developed, these types of sensors may find semiconductor applications in the future.

Since performance requirements had not been developed for the MFCs in the semiconductor industry, Applied Materials set forth a commodity specification to define and test to those requirements. Utilizing a rate of rise measurement system Applied was the first organization to characterize the transient behavior of MFC flow into a sub-atmospheric pressure chamber. This technique best replicates the behavior of gas entering into a wafer process chamber.

Calibration gases, referred to as surrogate gas(s) are utilized to best replicate the nameplate gas. The nameplate gas is the actual process gas which the MFC is calibrated for. The relationship of surrogate to name plate gas is paramount in understanding how to calibrate an MFC. Knowing that these relationships are non-linear, polynomial equations can be generated to best fit the function of this relationship. Additionally the relationships back to nitrogen, for all gases, are important so that testing of MFCs integrated into semiconductor equipment can be tested, prior to shipment and installation in the fab.

Performance evaluation is a necessary evil for understanding which MFCs are best for a specific semiconductor process. The testing requirements allow for ranking of suppliers, and for interactive development of MFCs with the manufacturers of these instruments. Additionally, comparative analysis, such as: the analog vs. digital; along with thermal vs. pressure based and the like can be reviewed.

The improvements in calibrations, diagnostics, and digital communication protocols have enhanced the capabilities of MFCs and allow for statistical process control methods to be applied. This should allow for process repeatability improvements, necessary in the development of semiconductor processes.

Issues which need to be overcome are: Cross-talk, pressure regulator interaction, and gas bursting; these phenomenon and behaviors are evident in the issues which semiconductor manufacturers face on a daily basis. Also the behavior of various types of MFCs need to be studied and understood. Are various types of MFCs affected by the same phenomenon? Liquid and subatmospheric delivery regimes also require testing, understanding, and evaluation in order to develop and improve semiconductor processes.

I hope that this presentation stirs interest in the terminology, issues, behavior, performance and understanding of how MFCs are manufactured and applied in today's semiconductor industry.

Gas flow standards and calibration

John D. Wright
National Institute of Standards and Technology

The Fluid Flow Group at the National Institute of Standards and Technology in Gaithersburg, Maryland offers calibration services for flow meters used in gas, water, and liquid hydrocarbon. Gas flow meters are calibrated with piston provers, bell provers, or PVTt systems for flows between 0.04 L/min and 78000 L/min. Further details of these calibration services are documented, including the principle of operation and measurement uncertainties. The definition of traceability (direct and indirect) and the importance of proficiency tests that include inter-laboratory comparisons are discussed.

Consistent ± 3 sigma calibration

Bill Valentine
Kinetics Fluid Systems

MFC manufacturers have been claiming an accuracy of $\pm 1\%$ FS since the invention of the MFC. Several years ago, Unit Instruments set out to create a metrology system capable of delivering product such that 99.7% (± 3 sigma) of all product shipped would meet an accuracy of $\pm 1\%$ FS. Unit Instruments' strategy consisted of a three-tier attack.

First step was to understand our capabilities in metrology. We developed a system called CrossCheck, where we compare various primary standards against each other. The primary calibration techniques utilized in our system are constant volume (bell prover), constant pressure (rate-of-rise) and gravimetric. These calibration methods do not share common modes of error. Consequently, comparing primaries against each other is the most effective method to determine if one of your calibration techniques has degraded. In addition to comparing primaries internally, we participate in round robin comparisons with NIST. Critical flow nozzles are used to check metrology between service centers and our main metrology center, and laminar flow elements are used to transfer metrology to the production floor.

Next, we set out to determine if we had a capable process. 1092 MFCs were screened over a period of 14 months. The MFCs were selected to cover a wide range of gases and ranges. Calibration was verified on two different calibration stations. The measurements statistically demonstrated our process was capable to \pm three sigma limits.

Finally, we needed to show our solid metrology and production process would translate into superior on tool performance. Accuracy on nitrogen does not insure a MFC will perform on tool with the process gas. Using our onsite gravimetric facility and a gravimetric facility at Oak National Laboratory, we validated our product was linear and thus its surrogate gas calibration would not be compromised by the application of conversion factors. Several tests were performed on traditional problem gases. Results presented include Cl_2 , BCl_3 , HBr , and WF_6 .

The impact of various gas properties on the operation of an MFC

Dan Mudd
Mass Flow Associates of Texas

Gas properties directly influence the operation of an MFC. Specific MFC components are influenced by specific gas properties and determine if the component is operating within its linear region. Problems can arise with the use of surrogate gases as substitutes for "nasty" nameplate gases if any MFC component is operated outside its linear region when flowing either a surrogate calibration gas, the nameplate gas or a surrogate transient-response gas. An evaluation of the gas properties and foot printing of the individual components can suggest surrogate calibration practices and procedures to avoid miscalibrations seen in the industry associated with the use of surrogate gases by MFC manufacturers. A review of the key gas properties affecting an MFC and their effect on the individual MFC components is made.

NIST's program to measure the thermophysical properties of semiconductor process gases

John J. Hurly and Michael R. Moldover
Process Measurements Division, National Institute of Standards and Technology

NIST has developed a facility to safely study the toxic, corrosive, and hazardous gases that are used in the processing of semiconductors. We have completed measurements of the speed of sound in the process gases Cl_2 , HBr , BCl_3 , WF_6 , and $(\text{CH}_2)_2\text{O}$, and in the surrogate gases SF_6 , CF_4 , and C_2F_6 . The data span the temperature range from 200 K to 475 K and the pressure range from 25 kPa to the lesser of 1500 kPa or 80% of the sample's vapor pressure. The measurements are made along isotherms. Each isotherm is individually analyzed, and from the zero-pressure intercept the ideal-gas heat capacities $C_p(T)$ are obtained with uncertainties of $0.001 \times C_p(T)$. The slope and curvature of each isotherm provides information about the gas's virial equation of state. The density virial coefficients are obtained by simultaneously fitting all the sound speed measurements to model pair and three-body intermolecular potentials. From the potentials, we can estimate the viscosity $\eta(T)$ and the thermal conductivity $\lambda(T)$. The calculations extrapolate well and extend to temperatures in excess of 800 K, well above the range of the measurements. For gases where other data exist, we find the uncertainties in the calculated properties are less than $0.001 \times \rho$, $0.1 \times \eta$, and $0.1 \times \lambda$. We plan to measure $\eta(T)$ and $\lambda(T)$, thereby reducing their uncertainties under 1 %. We plan to measure the properties of the other gases that the semiconductor processing community identifies as having the highest priority. We have posted a trial version of a user-friendly database to

disseminate the properties of process gases and carrier gases. This database can be found at <http://properties.nist.gov/SemiProp/>. Please send comments concerning this database to john.hurly@nist.gov.

Requirements for the next generation gas mass flow controllers

Kaveh Zarkar
Millipore Corporation

Continued advancement and improvements in the era of 0.25 μm and finer feature sizes in semiconductor chip manufacturing have seen the advent of newer, faster and smaller fluid handling components. Also, shifts in the industry trend from batch process to single wafers has impacted the traditional gas system components. Future semiconductor process capabilities, particularly the emerging demand for CVD and plasma etch, eventually will affect the gas delivery systems and components, specifically the mass flow controllers, which are important gas delivery components directly affecting the film integrity and quality. Industry will require new and continuously improving generations of MFCs that are superior in performance, more versatile in handling multiple gases, as well more reliable with reduced cost of ownership. To achieve the best results, gas delivery component selection is going to play a vital role in achieving the tighter and more demanding process requirements. This paper examines the specifics of each critical process as it relates to the MFC selection and functionality.

Participants in breakout sessions

Performance of flow meters

Gary Allen (leader)	Applied Materials
Jeff Anastas	MKS Instruments
Robert Berg	NIST
Daniel Coffman	Applied Materials
Joel Derk	Lucent
France D'Spain	SW Research Institute
Ed Francis	National Semiconductor
Tim Kipley	Aera Corporation
Thomas Maginnis	University of Massachusetts at Lowell
George Mattingly	NIST
Mike Munson	Dominion Semiconductor
Thomas Naughton	Dresser Equipment Group
Jeff Rose (assistant)	Motorola
Greg Secord	DH Instruments
William White	W3

Standards and calibration

Michael Bair	DH Instruments
Trace Beck	International SEMATECH
Brian Dickson	Lucas Labs
Gary Frank	Unit Instruments
Bill Johnson	Eastman Kodak
William Kosh	Dresser Equipment Group
James Long	Aera Corporation
Balarabe Mohammed	Applied Materials
Daniel Mudd	Mass Flow Associates of Texas
George Porter	Porter Instrument Co.
William Valentine (leader)	Unit Instruments
Bob Williams	Coastal Instruments
John Wright (assistant)	NIST

Gas properties

William Alvesteffer	Teledyne Hastings
Wang Chiun	Unit Instruments
James Hardy (assistant)	Oak Ridge National Laboratory
John Hurly (leader)	NIST
Jim Hylton	Bechtel Jacobs Company LLC
Max Klein	Scitefair International, Inc.
Jack Martinez	NIST
Gil Yetter	International SEMATECH

Alternatives to thermal mass flow controllers

Chris Davis	FuGacity
Joe Dille	Brooks Instrument
David Green	NIST
Bin Han	MKS Instruments
Albert Henning	Redwood Microsystems

Michael Moldover (assistant)

Maceo Ward

Kaveh Zarkar (leader)

Jay Zemel

NIST

Millipore Corporation

Millipore Corporation

Scitefair International, Inc.

Discussions in the full workshop

The next four subsections summarize the discussions held in the full workshop. The context for these discussions consisted of six presentations, whose slides are reproduced in Appendix F, and the discussions in the breakout groups. Many discussions led to the proposal of a specific task. The list of tasks in Appendix A gives a summary of these topics. This section explains why some of the tasks were proposed, and it provides brief accounts of discussions that did not lead to proposed tasks.

Performance of flow meters

The breakout group used a recent article to estimate future MFC requirements (Kaveh Zarkar, “Requirements for next-generation gas-flow components”, Solid State Technology, March 2000, pp. 27-32). Table 1 of this article listed improvement factors expected for MFC requirements by 2004. The table below applies Zarkar’s improvement factors to Applied Materials’ present MFC requirements. Four of the requirements in 2000 are listed on slide 10 of Gary Allen’s presentation (Appendix F). The other rows (turndown ratio, overshoot, and settling time) were written down after discussions in the breakout group.

Characteristic	Requirement in 2000	Requirement in 2004
accuracy	1% of full scale	0.5% of full scale
repeatability	0.25% of full scale	0.13% of full scale
valve leak	1% of full scale	0.3% of full scale
turndown ratio	20	80
overshoot	10% of set point	2% of set point
step response	1.5 s	0.3 s
settling time	2 s	1 s

The attendees generally agreed with this table. However, one representative of an MFC manufacturer asked if the desired requirements were driven more by measurement feasibility than by the needs of the manufacturing processes. Several examples of process needs were given in response, one of which was tungsten deposition requiring a 1-second step response with no overshoot. One participant made the general point that improved MFCs will enable new processes.

The attendees characterized the most important MFC requirement as *interchangeability*, which means that replacing one MFC with another MFC designed for the same flow rate has negligible effect on the manufactured product. Interchangeability comprises the requirements of accuracy, linearity, and reproducibility. Several participants emphasized that the MFCs must be interchangeable for transient as well as for steady flows. One participant pointed out that the interchangeability of MFCs from the same manufacturer is easier to achieve than interchangeability of MFCs from different manufacturers. Due to

differences in design and calibration, the interchangeability of two MFCs from different manufacturers seems unlikely unless both MFCs have accuracies better than the required interchangeability.

One representative of an MFC manufacturer stated that there is little demand for MFCs that operate at low flow rates. Others disagreed, saying that demand is increasing, or saying that demand would be greater if the MFCs were more reliable at low flow rates. Problems common at low flow rates, such as long gas delivery lines and poorly controlled valve sequencing, make it difficult to verify such reliability. Several participants stated that standards at low flow rates would be helpful here. Another comment was that accurate flow control is needed for recipes requiring stoichiometry ratios exceeding 100:1.

A benign surrogate gas, such as SF₆, is frequently used to calibrate an MFC intended for a difficult process gas, such as WF₆. Different MFC manufacturers use different sets of surrogate gases, which can complicate the comparison of MFCs from different manufacturers. Many participants advocated that every MFC manufacturer characterize each MFC's performance with nitrogen, even if nitrogen was not the calibration gas. This would allow a simple verification that the MFC was working properly, both at the tool manufacturer as well as at the semiconductor fabrication plant, even if the MFC was not intended for use with nitrogen. The cost of such characterization was not clear.

Other issues considered included the following.

- Better techniques to measure gas flows at subatmospheric pressures are needed.
- Characterization of an MFC for the process gas is best done by a function of flow rate instead of by a flow-independent "gas correction factor".
- Frustration exists with MFC zeros that are set either incorrectly or inconsistently.

Standards and calibration

The breakout group called for the following new or improved tests and standards.

- Transient flows and crosstalk due to pressure variations.
- An "*in situ*" standard for process gases accurate to 1 % between 0.01 sccm and 1000 sccm.
- Liquid flows below 15 ml/minute.
- Transfer standards between 0.01 sccm and 1000 slm for "round robin" (interlaboratory) tests.

The group also called for clarification in two areas.

- The phrase "NIST traceable" needs to be made more meaningful. This was motivated by John Wright's distinction between *direct* and *indirect traceability*. Several participants pointed out that "NIST traceable" is widely abused.

- Documents are needed on the “best practice” for various primary standards, similar to those produced by the National Conference of Standards Laboratories. The existence of SEMI standards for MFC testing needs to be publicized better.

Many participants emphasized the desirability of calibrating an MFC with the intended process gas instead of a benign surrogate gas. While more expensive, such “live gas” calibration improves the MFC’s accuracy, thereby reducing the cost of “tweaking in” a new process on the semiconductor manufacturing tool. The participants identified only four facilities for live gas testing. The first, at a government laboratory (Oak Ridge National Laboratory), has been little used in recent years. The others are at a commercial testing laboratory (W3 Corporation) and at two MFC manufacturers (Kinetics and Millipore). One process engineer suggested approaching end users such as himself for help. A widely accepted cost-benefit analysis of live gas calibration does not exist.

Gas properties

The thermophysical properties of process gases have a direct effect on the design, calibration, and operation of MFCs. The large uncertainties associated with the gas properties of many process gases make the improvement of MFC models more difficult. The accuracy required of a property depends on how the MFC’s performance is affected by that property. For example, for a thermal MFC the most important property is the heat capacity at constant pressure, but for a sonic nozzle MFC it is the speed of sound. Three improvements were discussed.

- Direct experimental measurements of properties. The Fluid Science Group at NIST is characterizing four to ten semiconductor process gases per year with accuracies sufficient for thermal MFCs (for example, 0.1% in heat capacity and 0.5% in viscosity).
- Development and application of techniques to estimate properties. This will provide property values much faster than the measurements at the cost of worse accuracy. The associated uncertainties are expected to be approximately 20 times larger than for direct measurements, and the techniques require at least a few measurements for their validation and improvement.
- Compilation of existing property values, both measured and estimated, in an easily accessible database.

The importance of mixture properties was unclear. MFCs that prepare a mixture by controlling the flow of pure gases do not require the properties of the mixture created downstream. MFCs that control the flow of a dilute mixture (for example, a small amount of O₂ in He) may require the mixture’s properties, but they are easily estimated from the properties of the pure components because the mixture is dilute. NIST is not aware of any process that requires the flow control of a concentrated mixture. The identification of such processes would be extremely valuable.

The breakout group recommended development of a generic MFC model, starting with components such as the flow divider. This recommendation, which was discussed twice earlier in the contexts of gas correction factors and of surrogate gases, was controversial. Attendees representing MFC manufacturers noted that MFC designs are proprietary.

Alternatives to thermal mass flow controllers

The breakout group used a matrix approach to think about competing flow measurement techniques. One side of the matrix listed measurement techniques, including thermal MFCs. The other side listed manufacturing processes, examples of which can be found in Kaveh Zarkar's presentation (Appendix F). In principle, each cell of the matrix could be filled with an assessment of the suitability of a particular technique for a particular process. In practice, this could not be done during the workshop because it would have required detailed knowledge of the processes and their fluids as well as the techniques. Examples of such details include the following.

- process
 - operating pressure
 - flow requirements
 - flow dynamics
 - step time requirement
- fluid
 - precursor phase (solid, liquid, gas, vapor)
 - chemical compatibility
 - density
 - specific heat
 - vapor pressure

Predicting the future suitability of the techniques was even more difficult. Alternatives to thermal MFCs have capabilities that are still being developed, and new manufacturing processes continue to immerge. The panel recommended that SEMI, NIST, and the semiconductor industry work together to characterize the new processes and fluids.

The breakout group concluded that NIST can help the development of new flow measurement techniques in the following ways.

- Provide flow standards suitable for new techniques.
- Provide property data for new process gases.
- Use scientific understanding to improve existing techniques. A recent example is the identification of molecular relaxation effects in sonic nozzles.
- Develop new techniques.

Final recommendations

Of the 21 proposed tasks listed in the Appendix A, seven received a vote from at least 40 % of the non-NIST attendees. These strongly recommended tasks are listed in the table on the next page. Each of the other 14 tasks received a vote from less than 25% of the attendees.

Five of the seven strongly recommended tasks require action by NIST. As part of NIST's Chemical Science and Technology Laboratory (CSTL), the Process Measurements Division uses six guiding criteria to set program priorities. The workshop's recommendations are discussed below in relation to these criteria.

1. The magnitude and immediacy of the industrial need.

The industrial interest in this workshop showed that the industrial need for gas property values and flow standards is immediate and at least moderate. The rapid introduction of new processes by the semiconductor industry may make the need more urgent.

2. The degree of correspondence between a particular industrial need and CSTL's mission.

The degree of correspondence is high. Providing reference standards for flow and property values for pure, industrially important fluids will fulfill CSTL's mission by enhancing the productivity of U.S. industry.

3. The opportunity for CSTL participation to make a major difference.

CSTL's participation will make a major difference for two reasons. First, CSTL is the premier source for the thermophysical properties of gases. Second, NIST's reputation as an unbiased, reliable provider of reference standards for flow and other quantities makes it likely that the proposed flow standards will be used by industry.

4. The nature and size of the anticipated impact resulting from CSTL's participation.

CSTL has the capability to match most of the industrial needs. See criteria 1 and 2.

5. CSTL's capability to respond in a timely fashion with a high-quality solution.

CSTL's capability to respond is large because many of the needs match existing programs or expertise in CSTL. Tasks 4, 5, and 6 correspond to programs in the Process Measurements Division. Tasks 1 and 3 correspond to recent work done in the Division.

6. The nature of opportunities afforded by recent advances in science and technology.

The opportunities are significant and numerous. The gas property measurements rely on acoustic techniques recently developed and under constant improvement at NIST. The

existing transfer standard for low flow rates of gases relies on recent advances at NIST in modeling laminar flow elements.

Tasks strongly recommended by the workshop

Task	Institution	Relation of task to work at NIST
1 Devise a technique to verify MFC performance that is independent of the process chamber.	none specified	NIST's Process Measurements has a history of solving fluid measurement problems. An example is the acoustic flow meter recently developed in NIST's Fluid Science and Pressure & Vacuum groups.
2 Characterize the performance of each new MFC with nitrogen as well as with its nameplate gas.	MFC manufacturers	Not applicable.
3 Increase the range of transfer standards for conducting round-robin tests (0.01 sccm to 1000 slm).	NIST	NIST's Pressure and Vacuum has developed a laminar flow meter that is suitable as a transfer standard for flows between 1 and 1000 sccm. NIST's Fluid Flow Group has expertise with sonic nozzles, which are suitable for flows up to 1000 slm. In 1993, the Fluid Flow Group built and coordinated round-robin tests with a sonic nozzle artifact for flows at 300 sccm and 800 sccm. These efforts could be extended to the recommended range of flow rates.
4 Improve the primary (0.025%) and transfer (0.1%) standards for gas flow.	NIST	NIST's Pressure and Vacuum Group recently demonstrated the operation of a new primary standard with 0.1% uncertainty. In the near future, an improvement to 0.05% is expected. The existing transfer standard was modeled with 0.2% uncertainty, and improvement in the near future to 0.1% is expected.
5 Expand and reprioritize the list of gases to be studied. Schedule and conduct property measurements.	NIST	Workshop attendees are being queried about gas priorities. Gas property measurements are under way in the Fluid Science Group.
6 Establish and maintain a public, Web-based database of gas properties.	NIST	NIST's Fluid Science Group has posted a trial Web database of gas properties.
7 Develop metrology to characterize liquid flow controllers.	NIST	NIST's Fluid Flow group has expertise in developing liquid flow standards.

Appendices

A. Prioritization of the proposed tasks

The following tables show the tasks proposed by the breakout groups and their prioritization by the attendees. Most proposals also specify the institution that would accomplish the task. The tasks are reworded here to improve the descriptions written on flipcharts during the workshop.

Each attendee was allowed to vote for six tasks without voting more than once per task. *The votes of NIST attendees are excluded from the tables.*

We emphasize that all of the tasks were proposed only after discussion in the full workshop as well as in the breakout groups. Thus, even those tasks with few votes deserve serious consideration.

Performance of flow meters

Task	Institution	Votes
Write standard on procedure for adjusting MFC zero.	SEMI	6
Devise a technique to verify MFC performance that is independent of the process chamber.	none specified	14
Characterize the performance of each new MFC with nitrogen as well as with its nameplate gas.	MFC manufacturers	23
Develop techniques to characterize delivery of gas below atmospheric pressure.	NIST	4

Standards and calibration

Write document on best practices for primary standards.	NIST	6
Develop a facility and methods for testing transient performance.	NIST	3
Increase the range of transfer standards for conducting round-robin tests (0.01 sccm to 1000 slm).	NIST	24
Improve the primary (0.025%) and transfer (0.1%) standards for gas flow.	NIST	27

Develop a test facility for corrosive and toxic gases.	none specified	4
Develop primary standards for liquid flows below 15 ml/min. (TEOS, TMB, etc.)	NIST	8

Gas properties

Task	Institution	Votes
Expand and reprioritize the list of gases to be studied. Schedule and conduct property measurements.	NIST	20
Supplement experimental measurements by estimating, with uncertainties, the properties of pure gases.	NIST	6
Establish and maintain a public, Web-based database of gas properties.	NIST	20
Create an industry advisory board to guide NIST.	MFC manufacturers, tool manufacturers, MFC users	5
Develop a generic MFC model. Suggested first submodels: sensor, flow restrictor, transient response.	MFC manufacturers, NIST	6
Identify important gas mixtures. Estimate, with uncertainties, their properties. (Industry survey, literature search, measurements.)	NIST	5

Alternatives to thermal mass flow controllers

Identify processes likely to be important.	SEMI	2
Identify chemical precursors likely to be important.	SEMI	1
Create a database of precursor properties.	NIST	1
Identify the flow ranges likely to be important.	SEMI	0
Develop metrology to characterize liquid flow controllers	NIST	19

B. Suggested topics for breakout sessions

1. Flow meter performance

1.1. Industries

- 1.1.1. semiconductor device manufacturing
- 1.1.2. others: air pollution, pharmaceuticals, leak testing, ...

1.2. Process conditions

- 1.2.1. flow rate
- 1.2.2. fluid composition
- 1.2.3. pressure (including transients)
- 1.2.4. temperature
- 1.2.5. corrosion

1.3. Requirements

- 1.3.1. accuracy
- 1.3.2. stability (repeatability)
- 1.3.3. dynamic range
- 1.3.4. interchangeability

1.4. Challenges from new processes

- 1.4.1. lower flow rates
- 1.4.2. pressures below one atmosphere
- 1.4.3. new fluids (gas mixtures, high temperature vapors, liquids)

2. Standards and calibration

2.1. Requirements

- 2.1.1. flow rate
- 2.1.2. uncertainty
- 2.1.3. traceability
- 2.1.4. relation of surrogate gas to process gas
- 2.1.5. location (standards lab, MFC manufacturer, process tool)

2.2. Primary flow standards

- 2.2.1. gravimetric (weighing)
- 2.2.2. constant volume (pressure rate-of-rise)
- 2.2.3. constant pressure (piston prover)

2.3. Transfer flow standards

- 2.3.1. pressure drop across a laminar flow impedance
- 2.3.2. thermal MFC

2.4. Research at national laboratories

- 2.4.1. improved flow standards
- 2.4.2. new standards (transient flow, mixtures)
- 2.4.3. validation of MFC models by comparison of process and surrogate gases
- 2.4.4. MFC corrosion and reliability testing

2.5. SEMI guidelines and test methods

- 2.5.1. practical implementation
- 2.5.2. validation

3. Gas properties

- 3.1. Influence of properties on MFC models
- 3.2. Property measurements
 - 3.2.1. thermodynamic (heat capacity, compressibility, virial coefficients)
 - 3.2.2. transport (viscosity, thermal conductivity)
 - 3.2.3. other (speed of sound, Prandtl number)
- 3.3. Property models
 - 3.3.1. prediction from molecular structure
 - 3.3.2. mixture properties
 - 3.3.3. sources of reliable data and correlations
- 3.4. Generic modeling of dynamics
 - 3.4.1. hydrodynamics
 - 3.4.2. slip
 - 3.4.3. thermal diffusion
 - 3.4.4. molecular relaxation rates

4. Alternatives to thermal mass flow controllers

- 4.1. Micro-electrical-mechanical systems (MEMS)
- 4.2. Pressure drop across a laminar flow impedance
- 4.3. Sonic nozzle
- 4.4. Coriolis effect
- 4.5. Acoustic

C. Abbreviations and jargon

CD	critical dimension
CSTL	Chemical Science and Technology Laboratory
MFC	mass flow controller
nameplate gas	The process gas named on the body of the MFC.
NIST	National Institute of Standards and Technology
round robin	A scheme to compare laboratory measurement capabilities in which a test artifact is circulated among the laboratories.
sccm	standard cubic centimeter per minute ($\approx 1.34 \mu\text{mol/s}$)

SEMI	Semiconductor Equipment and Materials International
semiconductor tool	Work station for deposition on and etching of semiconductor wafers, including a process chamber and a gas handling system.
slm	standard cubic liter per minute (1000 sccm)

D. Workshop schedule

Monday, May 15

8:30 Introductions

9:00 **Talks to outline the issues**

- *Performance of flow meters*
Gary Allen, Applied Materials
- *Standards and calibration*
John Wright, NIST
Bill Valentine, Kinetics

10:15 Coffee

- *Gas properties*
Dan Mudd, Mass Flow Associates of Texas
John Hurly, NIST
- *Alternatives to thermal mass flow controllers*
Kaveh Zarkar, Millipore

12:00 **Guidelines for breakout sessions**

12:15 Lunch

1:15 **Breakout sessions**

3:00 Coffee

5:00 Adjournment

6:00 Social hour

7:00 Dinner

Tuesday, May 16

8:30 **Reports from breakout sessions**

10:15 Coffee

11:00 **Final breakout sessions**

12:15 Lunch

1:15 **Prioritization of recommendations**

3:00 Adjournment

3:30 Tours of NIST flow facilities

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F. Presentation viewgraphs

Mass flow controller performance and characterization
Gary Allen, Applied Materials

Gas flow standards and calibration
John Wright, NIST

Consistent ± 3 sigma calibration
Bill Valentine, Kinetics Fluid Systems

The impact of various gas properties on the operation of an MFC
Dan Mudd, Mass Flow Associates of Texas

*NIST's program to measure the thermophysical properties of
semiconductor process gases*
John Hurly, NIST

Flow controller for semiconductor industry
Kaveh Zarkar, Millipore