ACCURATE, RELIABLE CONTROL OF PROCESS GASES
BY MASS FLOW CONTROLLERS

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ACCURATE, RELIABLE CONTROL OF PROCESS GASES BY MASS FLOW CONTROLLERS

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Abstract

The thermal mass flow controller, or MFC, has become an instrument of choice for the monitoring and controlling of process gas flow throughout the materials processing industry. These MFCs are used on CVD processes, etching tools, and furnaces and, within the semiconductor industry, are used on 70% of the processing tools. Reliability and accuracy are major concerns for the users of the MFCs. Calibration and characterization technologies for the development and implementation of mass flow devices are described. A test facility is available to industry and universities to test and develop gas flow sensors and controllers and evaluate their performance related to environmental effects, reliability, reproducibility, and accuracy.

Additional work has been conducted in the area of accuracy. A gravimetric calibrator was invented that allows flow sensors to be calibrated in corrosive, reactive gases to an accuracy of 0.3% of reading, at least an order of magnitude better than previously possible. Although MFCs are typically specified with accuracies of 1% of full scale, MFCs may often be implemented with unwarranted confidence due to the conventional use of surrogate gas factors. Surrogate gas factors are corrections applied to process flow indications when an MFC has been calibrated on a laboratory-safe “surrogate” gas, but is actually used on a toxic, or corrosive process gas. Previous studies have indicated that the use of these factors may cause process flow errors of typically 10%, but possibly as great as 40% of full scale. This paper will present possible sources of error in MFC process gas flow monitoring and control, and will present an overview of corrective measures which may be implemented with MFC use to significantly reduce these sources of error.
Introduction

Materials processing is at the heart of semiconductor manufacturing. To make today's advanced integrated circuits, silicon wafers are subjected to over 200 process steps. Most of these steps involve processes such as etching, implanting, diffusion, annealing, deposition, and lithography (resist deposition and hardening). Often one or more process gases are used to complete the process step. To achieve the quality and performance required in the micro devices, accurate measurement and control of these gases is essential. The Oak Ridge National Laboratory, in conjunction with the semiconductor industry, has been working to improve process gas measurement and control accuracy and reliability. Standardized test methods have been developed to characterize and benchmark the performance of mass flow controllers (MFCs), the method of choice for monitoring and controlling gas delivery. Testbeds have been designed and fabricated to produce repeatable and accurate data using these standards. Baseline testing was performed along with development of a unique mass flow instrument, the gravimetric calibrator.

In addition to the MFC test methods and testbeds, the overall program included developing a training program for end-users and a portable flow standard developed by the National Institute of Standards and Technology (NIST) to benchmark calibration laboratories around the country. A round-robin test series was completed that included 22 flow calibration facilities.

Standardized Test Methods

The purposes of the standardized test methods were twofold. The first was to provide technically excellent and consistent methodologies to benchmark performance of MFCs. The second purpose was to provide an industry-accepted methodology for objectively comparing MFCs against each other and for giving a basis to judge improved performance over time. A task force representing expertise throughout the country, consisting of semiconductor producers, equipment suppliers, MFC manufacturers, the National Institute of Standards, and ORNL, surveyed the semiconductor industry to determine the highest priority performance parameters. Test methods were then developed to accurately and consistently measure these parameters. The highest priority parameters were reproducibility, accuracy, surrogate gas factor, reliability, particle generation, temperature effects, and pressure effects. ORNL was tasked to construct a facility to validate the test methods and perform baseline testing.

Facility Development and Design

To accommodate the variety of tests to be conducted, three separate testbeds were fabricated. One testbed was constructed to perform reliability testing of MFCs in an accelerated life testing mode. The testbed is shown in Fig. 1. Reliability of an MFC was characterized by repeated, continuous cycling of the control valve with performance parameters monitored as a function of time. The performance parameters monitored were reproducibility, particle generation, step response, and leakage. Over a 6 to 9 month period, MFCs were continuously cycled through setpoints of 0, 100, 25, and 75% of full scale with nitrogen gas. At discrete readpoints, typically every 100,000 cycles, the parameters were measured to determine degradation or failure of the MFC performance. The cycling normally continued for a total of 1,000,000 cycles. The reliability test stand was designed to allow for parallel and simultaneous testing of up to 28 MFCs of various full scale flow ranges. Control of the valve cycling, data collection, and setpoints was performed automatically with a dedicated data acquisition and control system. A universal electrical interface was designed to allow connection of any manufacturer's MFC. The reliability testbed was contained in a class 100 cleanroom to reduce ambient particle levels.

Another testbed included in the Mass Flow Development Laboratory is used to evaluate environmental effects on the performance of MFCs. This testbed is presented in Fig. 2. The two effects originally evaluated were temperature effects and pressure effects. Temperature effects included changes in the ambient conditions as well as changes in the gas temperature flowing through the MFC. Ambient temperature may be controlled from 30 deg C to 150 deg C. Gas temperatures may be varied by an upstream heat exchanger to temperatures exceeding 400 deg C. Pressure effects tests that may be performed include step-change transients in the inlet gas pressure and downstream gas pressure, slow ramp transients in the inlet gas pressure, and inlet gas pressure steady-state values. All tests can be conducted with benign gases such as nitrogen or argon. A data acquisition and control computer allow fully automated operation and data collection.
Figure 1. Twenty-Eight Parallel Runs of the Reliability Test Bed.

Figure 2. The Pressure and Temperature Effects Test Bed.
The third test stand was the accuracy/surrogate testbed and was constructed for calibrating MFCs with hazardous, toxic, corrosive, and flammable gases typically used in material processing by the semiconductor industry. The accuracy/surrogate testbed, pictured in Fig. 3, is used to evaluate MFC accuracy, linearity, repeatability, hysteresis, and deadband. This testbed was designed to safely handle a wide variety of gases including pyrophorics. The testbed is fully double-contained and all exhaust gases can be fully remediated with a combustion-decomposition oxidizer. The testbed was designed to be operated in a fully automatic mode to help ensure safety and data accuracy and consistency.

The accuracy testbed can be operated in two modes, one with benign gases where the MFC accuracy is evaluated against a laminar flow element and the second mode with hazardous gases. In order to operate with hazardous gases, an innovative new flow standard was required that could accurately measure these gases. A true gravimetric calibrator was designed and built to directly measure the mass flow of gases. The gravimetric calibrator is described in detail in the following section.

During the past two years the Mass Flow Controller Development Lab has been open to universities and industry for product development and testing. Over 20 users have used this DOE National Users Facility and its flow instrumentation experts to develop and improve the state-of-the-art in mass flow controllers. A major area of interest for these recent activities has been in the development of improved gas correction factors, which are conventionally accepted multiplicative factors for correcting the flow indication of MFCs when used for differing gases.
Gas correction factors are often applied when mass flow controllers are used for the metering and control of toxic and corrosive process gas flows. Their use arises from the inability to calibrate MFCs upon the actual gases for which they will be used. Such a calibration is typically not practical either through expense, resultant MFC contamination, or the inherent safety problems involved with process gas use. Instead, a benign, surrogate gas will be used for a factory calibration of the MFC, and a gas correction factor will be applied for specified process-gas/calibration-gas pairs. Traditionally, gas correction factors are analytically determined from a ratio of thermophysical properties of gas pairs, specifically density and thermal heat capacity. Experimentally, however, gas correction factors have not been well scrutinized, again due to the inherent difficulties involved with process gas use. Recent studies have demonstrated that gas correction factor use may result in accuracy errors as great as 40.0% of full scale flow. Manufacturers of MFCs will typically specify the inaccuracy of the MFC at ±1.0% of full scale. This specification, however, only applies to the calibration gas, i.e. the gas with which the MFC was calibrated at the factory. The flow indications of actual process gases must be corrected using correction factors to account for differing thermophysical and hydrodynamic properties with respect to the calibration gas.

The Gravimetric Calibrator

In an effort to improve the accuracy of process gas delivery, and reduce the errors involved with calibration/process gas corrections, a mass flow standard capable of safely handling toxic and corrosive gases was developed. This standard, referred to as the “gravimetric calibrator” and depicted in Fig. 4, measures mass flow by directly accounting for the change in mass of a control volume over a measured time period (1). It is comprised of a large, 20-liter collection vessel made of corrosion resistant stainless steel. The vessel is rigidly suspended from a load cell at the center of mass of the system. Gas enters and exits the collection vessel from a single torsional capillary tube at one end of the collection vessel. The large vessel “tare” weight is suspended via buoyancy by submerging the entire collection vessel in a water bath. By careful adjustment, the collection system may be made to be only slightly heavier than neutral buoyancy, such that the percentage change in mass of the system due to gas collection is a significant portion of the overall mass of the system. This allows accurate mass flow calibrations to be conducted virtually “on the fly”, requiring short collection times in order to collect measurable amounts of gas. Employing the gravimetric calibrator, calibrations may be performed on most hazardous process gases to better than ±0.3% of full scale. Using this information, comparisons to published calibration/process corrections may be made.

![Figure 4. The Gravimetric Calibrator.](image-url)
Manufacturer published data on correction factors indicates that, with an error bound of 10-15% of full scale flow, constant correction factors may be employed to correct process gas flow indications against calibration gas data. In actuality, the corrections are not merely multiplicative factors, but are actually non-linear flow-dependent functions. Using the gravimetric calibrator, improved calibration/process gas correction functions may be determined for a specific flow controller and for a specific calibration/process gas pair. Unfortunately, however, these values will be applicable only to the limited number of specimens which have been tested and those only for the gas pairs being studied. Although useful, it is much more desirable to develop a generic model of the MFC which can take into account flow geometries, gas properties, and flow conditions to predict correction functionalities. Such a model could be employed by MFC manufacturers and end-users to develop correction values without the expense and risk of hazardous gas use.

A model has been developed for a large subclass of MFCs which feature designs of type indicated in fig. 5. MFCs of this style employ a small capillary tube as a flow sensor. Fine wire wraps around the capillary tube are used to heat the flow, while simultaneously determining a temperature gradient axially with the flow. This temperature gradient is established due to convective heat transfer within the flow and, over a limited flow range, is linearly related to the flowrate. To maintain this linearity, but increase the overall range of use of an MFC, a laminar bypass flowpath is often employed in parallel to the sensor flowpath. This bypass routes a major portion of the overall flow, typically increasing the total flow range of the MFC by a factor of 100. This parallel configuration allows for the determination of total flow by measuring the flowrate in the sensor and assuming a constant flow splitting ratio between the bypass and sensor flowpaths.

Figure 5. A Typical Mass Flow Controller Featuring an Annular Bypass Flowpath.
The model developed for the evaluation of this style of MFC features a hybrid analytical and finite-element technique (2). The hybrid model employs a finite-element portion, which accounts for thermophysical and hydrodynamic phenomena within the capillary sensor portion of the MFC, and a analytical portion, which accounts for minor loss effects of the flow stream within the annular bypass. The latter effect, dominated by entrance and exit effects and the associated $\rho V^2$ pressure drop, promotes a significant portion of the flowrate functional dependence of the surrogate gas correction values. Figs. 6 and 7 present gas correction data for helium and argon respectively, for a typical annular bypass MFC design. Indicated on these graphs are gas correction values versus flowrate, where a correction value, or K-factor, of 1.45 would imply that a multiplication of indicated flow by 1.45 is necessary to obtain actual flow. Data is presented for 3 test MFCs, each of the same make and model, denoted 11166, 11168, and 11169. Manufacturer published data is presented as a constant K-factor and is indicated as “Mfg Published”. Model data using a non-hybrid exclusive finite-element model of the capillary sensor is indicated as “Model Data w/o ML”. This data does not incorporate minor losses within the annular bypass. Model data employing the hybrid analytical/finite-element scheme is denoted as “Model Data w/ML,” and demonstrates the improvement made by incorporating minor loss effects.

Figure 6. Gas Correction Data for Helium Gas.

Figure 7. Gas Correction Data for Argon Gas.
Model results as presented in these figures were based on dimensions and other parametric measurements specifically from device #11169. The correlation between the hybrid model and this device indicates a vast improvement over manufacturer published data, with a resulting reduction in error from 5.5% of full scale using the constant K-factor, to less than 1.0% using the hybrid model. It is interesting to note, however, that deviations exist from device to device, perhaps indicative of manufacturing tolerances between serial products of the same model type. Figs. 8 and 9 present data generated using the hybrid model to explore the effects of various parameter changes which may be experienced due to imprecision of control in the manufacturing process. Data is presented reflecting changes in the thermal conductivity of the capillary sensor insulation layer, which may be experienced due to the tightness of insulation wrap within this portion of the MFC. Data is also presented to indicate changes in the correction values due to dimensional variations of the inner diameter of the capillary sensor and the outer diameter of the annular bypass. It becomes evident from this data that variation in the outer diameter of the annular bypass may be responsible for the significant deviations of the correction function between test specimens.

Figure 8. The Effect of Manufacturing Tolerances on Helium Gas Correction Functions.

Figure 9. The Effect of Manufacturing Tolerances on Argon Gas Correction Functions.
The hybrid model data presented is very limited in scope, dealing only with argon and helium which can easily be evaluated using most gas flow calibration setups. It remains to be demonstrated that hybrid model data can be directly applied to other gases. Given this ability, however, calibration data may be obtained using several benign surrogate gases, i.e. nitrogen, argon, and helium, while device dimensional parameters may be adjusted by the hybrid model until a good match is obtained between model and experimental data. Once the match is made, parameters may be fixed and the model may then be employed to develop functional gas correction curves for other gases, simply by re-evaluating the model using differing gas parameters. A whole catalog of gas functions may then be determined using the hybrid model, simply by performing actual calibrations on a select few benign surrogates for each manufactured MFC.

Conclusion

Mass flow controllers are well-suited to the rigors of toxic and corrosive process gas handling, and have become the method of choice for process gas delivery in the semiconductor community. MFC users should be aware, however, that cautions should be employed when implementing MFCs. With regard to accuracy, MFCs will typically not be calibrated on the actual process gases for which the MFC will be used. In such instances, traditional gas correction factors may generate significant sources of error in process gas delivery. Methodologies exist which can substantially reduce the errors involved with process gas delivery, and facilities are available to characterize and validate these methodologies. These facilities are available, as a DOE User Facility, for academia and industry for the advancement and development of flow measurement and control.

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