In-Situ Metrology: the Path to Real-Time Advanced Process Control

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Abstract. While real-time and in-situ process sensors have been effectively applied to fault detection, process control through course correction has been mainly focused on in-line metrologies to drive run-to-run feedback and feedforward control. We have developed in-situ metrologies based on mass spectrometry, acoustic sensing, and FTIR techniques which enable real-time thickness metrology and control in CVD processes at a level of about 1% accuracy. These developments open the door to real-time sensors as the basis for both fault management and course correction, i.e., for real-time advanced process control. We have also employed in-situ metrology to develop robust control schemes for CVD precursor delivery from solid sources, and we are exploring a new spatially programmable reactor design paradigm for which real-time, in-situ sensing, metrology, and control of across-wafer uniformity is fundamental.

ADVANCED PROCESS CONTROL

Metrology is a mainstay of advancement and practice in semiconductor technology development and manufacturing. [1, 2] Advanced process control (APC) [3, 4] is aimed at exploiting metrology to increase equipment productivity and lower manufacturing costs. Early efforts toward APC were in evidence from the late 1980's, including TI's MMST program [5-8], Sematech's J-88 program [9], and the inclusion of a metrology and process control roadmap as a supplement to the first NTRS in 1994. [10-12] Today, much progress has been made in developing and implementing APC in the industry, and APC has become a mainline component of the ITRS (Metrology section) [13] and the subject of professional meetings [14, 15], consortia [16], and publications [17, 18].

In both conceptual and operational senses, APC is perhaps best regarded in terms of two components, course correction and fault management.

Course correction (CC) is aimed at adjusting parameters in a process or process sequence such that targets metrics are maintained in the presence of process drift and short-term variability. In principle, CC may be accomplished as run-to-run (or wafer-to-wafer) feedback and/or feedforward control, or as real-time control within a unit process.

Fault management (FM) is intended to detect equipment and process problems as soon as possible (hopefully in real time), and then initiating corrective action, ranging from immediate shut-down and repair of equipment to a more extensive exercise in fault classification and prognosis that leads to rescheduling of tool maintenance.

As indicated in Table 1, the goals of CC and FM are quite different: CC is aimed at keeping product quality on track, while FM is intended to monitor and maintain equipment so that product is not wasted and equipment repair is timely. The other characteristic which differentiates APC approaches is a distinction between run-to-run and real-time control: run-to-run control can provide benefits from in-line or off-line measurements, while real-time control can deliver a different set of benefits if real-time/in-situ metrology is available. [19]

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<th>Control</th>
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Table 1. Goals and modes for run-to-run and real-time advanced process control.
An early, major success for APC was IBM’s application [20] of in-situ residual gas analysis (mass spectroscopy) to detect faults in process equipment, terminate the process, and alert the operator and/or engineer, thereby avoiding further yield loss. Since a number of important equipment failure modes are well understood and readily distinguishable this way (e.g., gas source impurities from inadequate purging upon gas bottle change), this real-time fault management mode has provided substantial return and has been broadly implemented in manufacturing, as indicated by “mainline” in Table 1. More sophisticated fault detection algorithms have been reported. [21]

Expanding fault management to more complex fault mechanisms, equipment health prognosis, and optimized maintenance scheduling is more challenging: in the general case, multiple sensor signals, system modeling, and probabilistic risk assessment may be required to classify faults, specify likely origins, and predict when they will demand repair. On the other hand, this more general FM goal can benefit from in-line and off-line as well as real-time/in-situ metrologies, since they deliver trend information helpful to both classification and prognosis. A variety of interfacing and software tools are currently in use for data collection, integration and algorithm application [22] to develop these emerging strategies to the point that they can define equipment repair actions needed and appropriate schedules for them.

The primary thrust of course correction APC has been in run-to-run control. [23-26]. In this case in-line or off-line measurements are used to guide process recipe changes for succeeding wafer steps – either subsequent wafers in the same tool (feedback) or subsequent steps for the same wafer (feedforward). Such approaches can detect and compensate for long-term drift of process/equipment behavior, where long-term signifies variation evident over multiple wafers, but not detectable within a single wafer run. Indeed, the Sematech J-88 program was aimed specifically at run-to-run control, and since then run-to-run control become mainline, with substantial success and adoption in manufacturing for feedback, feedforward, and multi-step applications. [27-29] This is due in significant part to the availability of in-line and off-line metrology technology, which is a prerequisite for development and manufacturing even without explicit APC intentions. Platforms for APC implementation – emphasizing run-to-run control – have been developed to meet industry needs. [30]

In contrast, real-time course correction has been limited, primarily by the fact that real-time/in-situ metrologies with sufficient quantitative accuracy have not been available. It is important to note that real-time/in-situ sensors for fault detection do not demand the quantitative accuracy that is needed for CC applications. A primary advantage of real-time CC would be that unit process control could compensate for short-term random variability within a process step, as well as for long-term process drift, thus achieving a higher degree of overall course correction APC. In addition, sensors suitable to drive real-time metrology may be capable of serving both real-time course correction and fault detection goals simultaneously.

REAL-TIME APC

Now that important elements of APC have seen wide acceptance, one can envision a next-generation, i.e., real-time APC. In this scenario, quantitative real-time metrologies would drive real-time course correction to achieve enhanced controllability, effective for both short-term random variability and long-term drift, and these metrologies would be compatible with (if not identical to) those employed for real-time fault detection. With both real-time course correction and fault detection controlling control responses at the unit process level (rather like regulatory controllers), the existing benefits of feedback and feedforward run-to-run control could be retained at a more supervisory level of control, and future advances in fault classification and prognosis would ultimately add to this picture.

Over the past years our research has pursued the development of chemical analysis techniques as real-time, in-situ process metrologies, as well as the demonstration of these in control applications, with primary emphasis on mass spectrometry, acoustic sensing, and infrared (FTIR) sensors applied to chemical vapor deposition (CVD) processes and rate/thickness control. Currently, these methods – with three different sensors – have demonstrated better than 1% film thickness metrology and 1.5-2% real-time thickness control, and they have broad application (e.g., etching, heterostructures, etc.). Opportunities for sensing and controlling spatial uniformity – though longer-term – are also in view. And similar approaches to real-time sensor-based control have been shown, e.g., in the control of MOCVD source delivery for difficult materials (solids with low vapor pressure). This paper provides examples of such progress toward real-time APC in the hope that the next steps of technology transfer to manufacturing can be taken.

MASS SPEC METROLOGY

Initial investigations using in-situ mass spectrometry (mass spec) clearly revealed time-dependent behavior of reactive gases in CVD [31] and plasma [32, 33] processes. For example, RTCVD polySi and oxide results showed generation of reaction products and depletion of reactants to an extent dependent on reaction rate, and they indicated the possibility of using these for deposition rate metrology [34]. More recently, we have evaluated the metrology accuracy obtainable using downstream mass spec for several W CVD processes, both H2 reduction [35, 36] and SiH4 reduction [37], as carried out in a Ulvac ERA-1000 W CVD cluster tool. With reasonable reactant conversion rates (~20%) in the SiH4 reduction
process, thickness metrology of 1-1.5% accuracy was demonstrated and exploited to achieve real-time end point control consistent with this metrology. [37]

The experimental setup is indicated in Fig. 1, showing differentially pumped mass spectrometry (Inficon Transpector™) arranged to sample reactant and product species in the CVD reactor. As indicated by the time-dependent HF product signal in Fig. 2 for the H₂ reduction reaction, the sensor system actively process gases to determine dynamics through the process cycle. These in turn are consistent with dynamic simulations of equipment, process, and sensor systems. [38]

The SiH₄ reduction process for W CVD, 3 SiH₄ + 2 WF₆ ➝ 6 H₂ (g) + 2 W (s) + 3 SiF₄ (g), produces both H₂ and SiF₄ as reaction products. For a low pressure (0.1 torr) process, a thickness metrology of 1-1.5% accuracy was obtained using the H₂ product generation signal. [37] By tracking the integrated product signal and terminating the process when the target value was reached, run-to-run variability was reduced from ~4% to ~1.5%. Figure 3a shows the improvement for the mass spec product signal upon imposing real-time end point control, while Fig. 3b shows the corresponding improvement in control of the deposited W film thickness, as determined post-process.

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By weight measurements. In these experiments, “no control (experiment)” corresponded to a sequence of wafers where no end point control was imposed; “no control (estimate)” reflects an estimate of what the values would have been if end point control had not been imposed, judging from the signal levels observed.

At higher pressures (10 torr) the quality of the thickness metrology is even better, which is important because W CVD processes in manufacturing operate at 100-500 torr. [36] Using the HF product signal from the H₂ reduction reaction, a linear regression fit to the weight (thickness) vs. integrated HF product signal yielded a standard deviation of 0.67% uncertainty, as indicated in Fig. 4. These results indicate that mass spectrometry is a viable sensor-based metrology for real-time APC.
### MASS SPEC FOR ULTRATHIN LAYERS

There is an increasing need for thickness of ultrathin layers (in the 10 nm range), driven by specific applications (e.g., barrier and seed layers, gate dielectrics). Mass spectrometry based metrology shows promise for such applications. Figure 5 shows mass spec signals for the initial stages of H₂/WF₆ CVD reaction (H₂ reduction) on the Si surface. In this case, the H₂ reduction reaction is considerably slower than direct reaction of the WF₆ reactant with the Si surface (the Si reduction reaction), which generates a SiF₄ volatile product.[39] The Si reduction reaction continues until the initial Si surface is completely covered by W, at about 30 nm thickness.

The sharp SiF₄ peak in Fig. 5 demonstrates that real-time mass spec has sensitivity in the 10 nm range, suggesting a thickness metrology for such ultrathin layer applications of order 5% precision or better. This suggests at least two key areas of application. First, it may mean that mass spec metrology will be useful for ultrathin barrier, seed, or gate dielectric applications. Second, it shows that the approach can certainly reveal faults, such as inadequately cleaned contacts where the shape and intensity of the resulting peak changes considerably if the CVD chemistry is reasonably selective (e.g., reduced deposition rate on oxide cf. Si).

### ACOUSTIC METROLOGY

Acoustic sensors enable the determination of gas composition from the measured velocity of sound in the gas. Given the characteristic product generation and reactant depletion which occur in processes like CVD, this implies that acoustic sensors should also be capable of reaction rate and thickness metrology. In fact, real-time in-situ metrology for deposition thickness has been demonstrated at a comparable level of accuracy using acoustic sensors. [40, 41]
FTIR METROLOGY

Finally, optical sensing, carried out using Fourier transform infrared spectroscopy (FTIR) has been used to monitor reactant depletion and product generation in these CVD processes. [42] In this case the depletion of WF$_6$ is more sensitive than the generation of HF reaction product. As shown in Fig. 8, the integrated FTIR absorption peak of WF$_6$ provides an in-situ thickness metrology accurate to about 0.5%, i.e., comparable to the mass spec and acoustic sensor results.

WAFER STATE METROLOGY FOR REAL-TIME APC

The three sensors described above are, strictly speaking, process state sensors, yet by suitably monitoring the dynamic state of the process and recognizing that the process conditions reflect wafer state conditions, all three generate a wafer state thickness metrology of order 1% accuracy or better, seemingly sufficient to deliver value in manufacturing. In addition, at least one of them – mass spectrometry – is already in widespread use for real-time fault detection, indicating that it could provide dual use to real-time APC. Indeed, we have exercised mass spectrometry for fault detection purposes in concert with its role in thickness metrology for real-time course correction.

While these techniques have achieved the metrology accuracy originally sought, they must be used carefully. For example, they reflect the total material deposited, though some of this could be on other internal surfaces of the reactor (e.g., wafer chuck, chamber walls). In general, they require pressure
transduction systems to transform the sampled gas from the process pressure used to the pressure regime needed by the sensor, e.g., lower pressure for mass spectrometry and higher pressure for acoustic sensing. And of course they do not indicate across-wafer uniformity, an equally important metric for manufacturing productivity, but probably less sensitive to sources of process variability.

Nevertheless, these results are very promising in providing a base for expansion of APC to real-time APC. The problems of implementing sophisticated sensors like mass spectrometry have already been surmounted for real-time fault detection, and important issues of sensor conditioning and reliability have been dealt with.

In some sense, there remain two key issues for transfer of these real-time metrologies to manufacturing. First, in the context of pervasive run-to-run course correction applications and existing real-time fault detection, how will real-time course correction be integrated into the larger APC context? This may be a significant architecture issue, though not a fundamental obstacle given that real-time quantitative metrologies like these would primarily drive real-time feedback control at the unit process level, thereby functioning more like regulatory controllers within a larger supervisory control system which executes feedforward as well as feedback control involving multiple process steps and tools.

Second, what will be the accuracy of these metrologies when implemented in a manufacturing tool? Of course this can only be judged by experiment, and it should be emphasized that this must be the next step. While we have processed and tested 10-20 wafers per day in our experiments, it is doubtful that our academic research environment (or any other) can add much more in evaluating these techniques for manufacturing. Technology transfer to a development or manufacturing environment is the essential next step, where considerably larger wafer lots can be processed through real manufacturing tools.

Nevertheless, we can make some predictions based on our experience. A major hurdle in achieving the metrology accuracy reported here has been the presence of wall reactions (e.g., WFC adsorption and reaction with subsequent H2 to form HF), and the concomitant need to condition the reactor system (chamber, gas lines, etc.) in order to stabilize these factors, and when this is done the results are very favorable. In manufacturing tools, where wafers are processed at high rate over long periods of time, this conditioning is substantially guaranteed. Furthermore, when variations from continuous operation of manufacturing tools are made (e.g., preventive maintenance), the tools undergo conditioning procedures (e.g., monitor wafers for recalibration, recognition of first-wafer effects, etc.). Because the regularity of the equipment environment is much higher in manufacturing, we can only predict more accurate metrology for real-time APC than we have achieved in the research cited here.

CVD is a pervasive semiconductor manufacturing process and thickness control is important, so this example may serve as a useful entry point for development and integration of such sensors into a larger real-time APC system. However, semiconductor technology demands a considerably broader set of processes as well as different kinds of metrology. Therefore, one should consider the prognosis for expansion of a real-time APC capability.

Other chemical processes are certainly amenable to the same or similar techniques. Besides other thermal CVD processes, these methods can be applied to plasma-enhanced CVD and plasma etching, as already demonstrated. [32, 43] But the general notion of sensing chemical reaction signatures for chemical processes is much broader. For example, we have begun investigations of the curing process used to create nanoporous low-K dielectrics by spin-casting, using time-of-flight secondary ion mass spectrometry (ToF-SIMS) [44]. The properties of the material depend sensitively on the details of how the sacrificial porogen species are volatilized (they are the placeholders for the nanopores that will remain in the low-K matrix). Initial results clearly show the kinetics of porogen depletion with curing (annealing), and we have begun experiments to identify the volatile products directly through thermal desorption mass spectrometry.

**UNIFORMITY CONTROL**

While most in-situ sensors target the average behavior across the wafer, or a single point on the wafer, across-wafer uniformity is a key metric for manufacturability and an intense focus in process development. Clearly, it would be highly desirable to extend real-time process and wafer state metrology to encompass uniformity measurements for real-time APC.

Some sensor approaches are promising in this regard. Spatially resolved optical emission spectroscopy and multichannel laser interferometry have been used to achieve process and wafer state uniformity measurements in plasma etching processes [45], and the latter has led to development of real-time interferometric imaging instruments. [46] Ellipsometry has been employed using in-situ etch rates measured at a single point on the wafer together with offline multi-point measurements and response surface modeling for etch uniformity control. [47] Transient behavior at end point sensed by real-time mass spectroscopy has distinguished signatures of uniformity. [43]

We have been exploring a new concept in equipment design aimed at uniformity control, namely spatially programmable reactor design. The goal is reactor designs which ultimately enable spatial uniformity to be modified through arrays of sensors and actuators, so that the performance characteristics
of the reactor are software-programmable. Such a paradigm would provide for spatial arrays of process and wafer state sensors, or multiplexing of spatial sampling to such sensors, along with arrays of actuators and control systems, to accomplish spatial control of the process in run-to-run and real-time control modes.

Programmable reactor designs would enable three broad applications: (1) programmable uniformity, assuring that manufacturing requirements for uniformity can be achieved at any process design point which is sought for materials performance; (2) programmable nonuniformity, in which intentional nonuniformity is introduced to expedite experimental process optimization, essentially a “one-wafer design-of-experiments”; and (3) combinatorial materials discovery, in which gradients of stoichiometry are intentionally introduced to accelerate the development of complex new materials.

The initial testbed for this concept is the development of a spatially programmable CVD showerhead, applied initially to W CVD under NSF ITR sponsorship. [48] The current prototype is a three-zone showerhead [49] to test the approach, evaluate novel design concepts, and validate models [50]. Future embodiments will involve higher levels of integration and MEMS devices for sensors and actuators.

**EQUIPMENT SUBSYSTEMS CONTROL**

Major manufacturing tools are complex equipment systems, comprising not only the reactor environment in which processes modify the wafer, but also equipment subsystems for reactant delivery, exhaust management, wafer transport, and other functionalities. Delivery and control of reactive gas species has been a notorious problem, e.g., relating to mass flow controller reliability.

More recently, the management of delivery and control of liquid and solid sources has become more important due to the profusion of new and complex materials needed to support the ITRS roadmap. Delivery of liquid metallo-organic CVD (MOCVD) precursors has been accomplished by standard bubbler techniques, in which an inert carrier gas is bubbled through the liquid source to take up its saturation vapor pressure of the source material for delivery to the reactor. More recently, liquid delivery systems have become common, in which the liquid is delivered to the reactor and vaporized as it enters. While reproducible liquid delivery remains a challenge, perhaps the most difficult is the use of solid sources with low vapor pressure, where the precursor delivery rate is destabilized by variability e.g., in temperature or in the active area of the powder source as the precursor is consumed. Indeed, because of such difficulties suppliers have adopted the alternative of dissolving such sources in organic solvents and then using liquid delivery systems, but this raises new issues of contamination and chemical complexity.

We have applied acoustic sensing for real-time control of such problematic precursors, particularly one of the most challenging - solid Cp₂Mg, with a very low vapor pressure (0.04 torr at 25°C). This materials is important in optoelectronics materials growth. The real-time APC setup is shown schematically in Fig. 9. The inert H₂ carrier gas draws precursor from the solid source, which in turn is mixed with a H₂ diluent stream, after which the composition of the mixture is sensed by the acoustic sensor.

![FIGURE 9. System for real-time control of solid source delivery using acoustic sensor.](image)

Results in Fig. 10 show the performance of this real-time control system. With a low Cp₂Mg concentration (0.01 mol-%) as the target, the source temperature was varied from 40 to 32°C, causing a 50% decrease in vapor pressure, to simulate a major disruption of the precursor delivery rate. With the acoustic sensor providing a real-time composition metrology and driving a controller for both H₂ streams, precursor delivery rates can be controlled to within 1% of their targets.

This source delivery control strategy should be widely applicable, not only to the most difficult of precursors like solid Cp₂Mg, but to a broad range of liquid and solid precursors as well. Such sensors can accomplish both real-time course correction and fault detection. And the ability to control delivery of exotic chemical precursors is particularly timely in this era of
new multicomponent materials for semiconductor technology.

CONCLUSIONS

Advanced process control, only a vision a decade ago, has now become a mainstream thrust for semiconductor manufacturing. Its two areas of primary acceptance and success are in run-to-run control and real-time fault detection. Run-to-run control relies on continuing advances in in-line and off-line metrology tools, whose technology advancement is already required to meet the future nodes on the ITRS roadmap. In contrast, real-time fault detection demands real-time/in-situ sensors; however, high quantitative accuracy is not required, since the sensors are intended only to quickly identify known faults with serious consequences.

Improvements in quantitative accuracy of real-time, in-situ sensors opens the door to a full complement of real-time APC. High accuracy sensors which resolve chemical or spectroscopic information promise to resolve subtle changes in process and to enable course correction in real time. Amid the various available in-situ sensor technologies, we have demonstrated that chemical sensors of process state can reveal wafer state consequences associated with reactions occurring in the process module, leading to wafer state metrologies. For mass spectroscopy, acoustic sensing, and FTIR we have demonstrated thickness metrology with accuracy of 1% or better, certainly suitable for manufacturing requirements, and in the case of mass spectroscopy we have demonstrated its application in real-time end point control. The process spectrum available with these sensors is considerably broader than the thermal CVD example addressed here, and other in-situ approaches (e.g., optical [51, 52]) add to the available sensor portfolio for real-time APC.

With research to date indicating that in-situ metrology can support real-time course correction, it is time to move these efforts into a manufacturing environment. Our experience to date suggests that their performance will be even better because of the high volume conditioning which manufacturing equipment experiences.

New directions for research emphasis are necessary and evident for in-situ metrology and real-time APC. One is certainly in development of spatially resolved sensors which can determine across-wafer uniformity, or possibly in new concepts for spatially programmable reactor design. Another is in employing in-situ metrologies for real-time APC in equipment subsystems, where manufacturing variability and fault generation is often found. To this end, the application of acoustic sensing to a broad range of precursor delivery systems seems an excellent example of focusing attention on the larger equipment system, rather than solely around the wafer.

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