6. Development of Quantitative Measurements for Vacuum Process Control

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Objective: Develop quantitative measurement capability to enable a real-time, *in-situ* semiconductor process-control scheme, building upon competence in optical diagnostics and flow calibration techniques.

Problem: The increasing volume and complexity of vacuum processing, most notably in the semiconductor industry, requires real-time monitoring and control of process gases, reaction products, and gaseous contaminants. Our previous work demonstrated that residual gas analyzers (RGAs) could be made quantitative for *in-situ* monitoring of reaction products, but the $\pm$5-10% imprecision (from analyte generation in the ionizer) was too large for process control, as is variability among mass flow controllers. Optical techniques are promising for real-time monitoring, but realizing their potential requires a better understanding of the factors limiting their performance. For mass flow controllers (MFCs), we need the industry to help identify key measurement and operational challenges to more consistent performance.

Approach: In FY00 we began developing an advanced chemical process monitor based on cavity ring-down spectroscopy (CRDS) to quantify the HF generated in the thermal chemical vapor deposition (CVD) of tungsten metal films (WF_6 in an H_2-rich environment). Our approach is to establish compact, low-cost and robust optical diagnostic hardware at NIST, then transition it to Prof. Gary Rubloff's CVD facility at the University of Maryland for process control trials. With CRDS, we expect to achieve the desired measurement precision, and hope to use it to improve other detection methods such as RGAs. For MFCs, we convened industry leaders to help develop programmatic targets for NIST.

Results and Future Plans: Our initial focus was to develop a measurement strategy utilizing low-cost continuous wave (CW) diode lasers. Given the HF partial pressures expected in the CVD reactor (1 Pa -10 Pa) and the weak (0-4) overtone band of HF, we chose the 670nm -700 nm region as the target band as measurements here are vastly easier than in the mid-infrared where fundamental-band transitions exist. Calculations were completed to redict the line strengths and CRD absorption characteristics for this band. Absorption lines of H_2O, a potential contaminant, were also identified in this region. Next, diode lasers at 670 and 685 nm were acquired and re-engineered to interface with current and temperature controllers. Problems persist in the temperature control and tuning capabilities of these diodes, and new strategies are being implemented to overcome them. A 50-cm transfer cavity and electronics were built for frequency stabilization of the diode laser. Electronics and initial tests of locking the transfer cavity to a frequency-stable HeNe laser were completed. Partially-reflective custom optics and special glass tubes were made for this cavity. Initial trials were conducted to test various optical configurations, including a fast (100 ns rise-time) acousto-optic deflector to rapidly switch the diode laser. The acousto-optic modulator and switching circuit have been tested and optimized. Work is in progress to lock the diode laser to the same transfer cavity and implement an acousto-optic modulator in a double-passed configuration for continuous laser tuning. Robust laboratory realization of this CW-CRDS approach is expected in FY01.

In order to promote more accurate and precise measurements with MFCs, we convened a workshop that identified five major recommendations for NIST. These were to 1) increase the range of transfer standards for round-robin tests (0.01 sccm to 1000 slm), 2) reduce uncertainty of primary (0.025%) and transfer (0.1%) standards for gas flow, 3) expand, reprioritize and conduct thermophysical measurements for the priority list of electronic gases, 4) establish and maintain a public, Web-based database of gas properties, and 5) develop metrology to characterize liquid flow controllers. These recommendations now form the basis of new and ongoing projects in the Division.