

# A model-based technique for real-time estimation of absolute fluorine concentration in a CF<sub>4</sub>/Ar plasma

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A technique for quantitative interpretation of actinometric data to deduce bulk plasma fluorine concentration in a CF<sub>4</sub>/Ar plasma has been developed and tested on a commercial reactive ion etcher. This static, *in situ* measurement is useful for monitoring fluorine in a manufacturing environment and, in particular, for application of real-time feedback control to plasma etching. Based upon a model of CF<sub>4</sub> chemistry reaction pathways and products, it improves upon current fluorine estimation techniques by accounting for varying levels of argon dilution resulting from CF<sub>4</sub> dissociation. A simple experiment was also developed in order to obtain an estimate of the actinometric scaling factor without an independent measurement of fluorine. Performance of this model-based fluorine estimation technique was compared to that of a standard technique by using time resolved etch rate measurements as an independent indicator of fluorine concentration, while a feedback control scheme decoupled the effects of physical etching by stabilizing the induced dc bias. The model-based estimator significantly reduced perturbations in the etch compared to those seen when using the standard estimator. © 1995 American Vacuum Society.

## I. INTRODUCTION

In order to realize continued advances in semiconductor manufacturing, accurate *in situ* sensors and diagnostics must be developed. Optical emission spectroscopy is one of the most widespread plasma processing diagnostics. It is a non-intrusive, *in situ* technique which can, therefore, be implemented in a manufacturing environment to provide real-time process data. Unfortunately, optical emission spectroscopy data is often quite difficult to interpret.

Actinometry<sup>4</sup> is a technique which has been developed to determine relative species' concentration from optical emission spectroscopy data and has been independently validated for fluorine with argon as the gas tracer.<sup>2,10,13</sup> By adding a small amount of a tracer gas, an actinometer, which is optically excited by the same set of electrons as the active gas of interest, electron energy dependence of the emission may be eliminated by ratioing the active and the tracer gases' emissions. Actinometry, however, only gives a relative measure of species' concentrations, while a relative or absolute measurement of the active species' concentration is often a more useful measurement. In most instances, one assumes that the inert tracer's molar fraction remains constant in order to obtain a relative measurement of the active species.<sup>13,18,19,24</sup> Dalvie and Jensen,<sup>6</sup> however, found that, with 1% argon mixed with their CF<sub>4</sub> feed gas, the tracer was diluted by as much as 25%. This sort of dilution induces errors in the interpretation of actinometry as a species concentration measurement.<sup>19,24</sup> Jenq and coworkers<sup>12</sup> proposed a combi-

nation of quadrupole mass spectroscopy, to measure the actinometer's concentration, and actinometry as a potential solution to the dilution problem. However, this solution required additional diagnostic equipment.

In this article, we propose and evaluate a method of interpreting actinometric data to obtain an absolute measurement of fluorine concentration in a CF<sub>4</sub>/Ar plasma. This method uses knowledge of the reaction pathways and dominant products to derive a static fluorine estimator which accounts for argon dilution and requires only optical emission spectroscopy and total pressure measurements.

This work is a portion of a larger effort in the University of Michigan Electronics Manufacturing and Control Systems Laboratory to apply real-time control to semiconductor manufacturing.<sup>8,9,18,28</sup> Because actinometry is an *in situ*, non-intrusive diagnostic, it is well suited for use with real-time control. The model-based estimator has been implemented within a feedback control system on a commercial reactive ion etcher; the controller regulates the estimated bulk fluorine concentration and induced direct current (dc) bias. Using etch rate reproducibility with fixed dc bias as the performance metric, this model-based fluorine estimator is shown to improve etch rate reproducibility over that achieved when using a standard fluorine estimator.

The estimator developed requires knowledge of the actinometric constant for fluorine and argon in a CF<sub>4</sub> plasma. We created an experiment to estimate the value of this constant for fluorine and argon in a CF<sub>4</sub> plasma. The results of this experiment are also presented and discussed.

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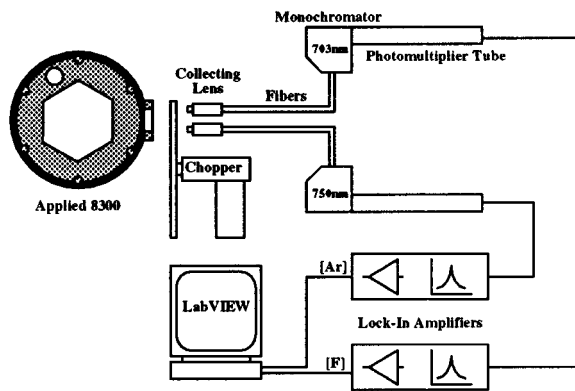


FIG. 1. Schematic of the actinometry equipment.

## II. EQUIPMENT AND EXPERIMENTAL SETUP

A detailed description of the equipment is available in Ref. 9, and the control strategy is described in Ref. 8. Only a brief overview will be included here.

The experimental portion of this work was performed on an Applied Materials 8300 reactive ion etcher (RIE). Typical operating pressures range from 10 to 70 mTorr. The powered electrode, driven at 13.56 MHz, is a hexode. The chamber is not equipped with a load lock. The feedstock gas,  $\text{CF}_4$  premixed with 5% Ar, is fed into the system through a showerhead arrangement at each of the six corners. Feed flow was fixed at 30 sccm, while exhaust flow was controlled through a butterfly valve.

Actinometry measurements were taken using monochromators aligned to the 703.7 nm fluorine and the 750.4 nm argon lines. The plasma emissions were mechanically modulated to 1 kHz and collected into two fused silica optical fiber bundles. The fluorine line was separated by a 125 mm Oriol Multispec monochromator, while the argon line was separated by a Spex 270M monochromator. Light was then detected using photomultiplier tubes and amplified via phase-sensitive amplifiers. The actinometry equipment is illustrated in Fig. 1. Since the optical fibers collect light from a conic section, looking from the unpowered electrode towards the powered hexode, the measurements represent an averaged bulk value without any spatial resolution.

*In situ* etch rate measurements were made using reflectometry. A HeNe laser with a wavelength of 6328 Å was shone onto the surface of the unmasked poly-Si/SiO<sub>2</sub>/Si wafer at nearly normal angles of incidence and reflected into an optical fiber. The peaks and valleys in the reflected light's interference pattern indicate the amount of time required to etch 417 Å of polysilicon, thereby providing an *in situ* etch rate measurement.

## III. DEVELOPMENT OF THE ESTIMATION TECHNIQUE

The model-based fluorine estimation technique is founded upon an interpretation of actinometry data. It is derived through some simple chemical equations and an assumption regarding chemical reaction products in a  $\text{CF}_4/\text{Ar}$  plasma.

## A. Background

Actinometry is an application of optical emission spectroscopy in which a small amount of an inert tracer, or actinometer, gas is added to the plasma feed. When certain stringent conditions are met,<sup>4</sup> spectral emission lines corresponding to the gas of interest and the actinometer may be ratioed to give a measurement of relative gas concentrations without any dependence upon electron excitation levels. The ratio technique, applied to fluorine and argon is reflected in the following equation:

$$\frac{[\text{F}]}{[\text{Ar}]} = K \frac{I_{\text{F}}}{I_{\text{Ar}}}, \quad (3.1)$$

where  $[X]$  is the concentration of species  $X$ ,  $I_x$  is the intensity of an appropriately selected spectral emission line corresponding to species  $X$ , and  $K$  is a constant which depends upon physical parameters as well as optical system losses.

Actinometry is often used to provide an estimate of relative fluorine concentration. Under the assumptions that argon's inert nature prevents its molar fraction within the plasma from changing significantly<sup>21</sup> and that the plasma's temperature doesn't vary, the argon concentration is proportional to total pressure. Therefore,

$$[\text{F}] \begin{cases} = K \frac{I_{\text{F}}}{I_{\text{Ar}}} [\text{Ar}], & (3.2) \\ = \frac{I_{\text{F}}}{I_{\text{Ar}}} K \gamma P r s \sigma, & (3.3) \end{cases}$$

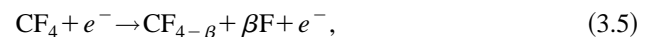
where  $\gamma$  is the fraction of argon to the total feed mixture and  $\sigma$ , the proportionality constant, represents a factor to account for the degree of argon dilution from its feedstock fraction. Since  $\sigma$  and  $K$  are unknown, only relative measurements of fluorine are made with the standard estimator<sup>19,24</sup>

$$[\text{F}] \propto \frac{I_{\text{F}}}{I_{\text{Ar}}} P r s. \quad (3.4)$$

Unfortunately,  $\sigma$  varies with changing plasma conditions, whereas (3.4) inherently assumes that it is constant— $\sigma$  is often assumed equal to 1.

## B. Model-based estimator

A more accurate method of interpreting actinometry data may be derived by incorporating a simple model of the chemical reaction pathways. In order to reflect the fraction of argon in the plasma, we define a variable  $\beta$  which represents the number of free fluorine radicals in the plasma per  $\text{CF}_4$  molecule dissociated. In a pure  $\text{CF}_4/\text{Ar}$  plasma,  $\beta$  would represent an effective stoichiometric coefficient in the dissociative reaction



where  $e^-$  represents an energized electron. The effective branching ratio of  $\text{CF}_4$  dissociation has not been accurately determined in the literature.<sup>7,16,17</sup>

Since the feedstock consists of a mixture of  $\text{CF}_4$  and argon gases, the mixing fraction is represented by

$$\gamma = \frac{[\text{Ar}]}{[\text{CF}_4] + [\text{F}]/\beta + [\text{Ar}]} \quad (3.6)$$

Because  $[\text{CF}_4]$  is the concentration after dissociation has taken place and  $[\text{F}]/\beta$  is the concentration of  $\text{CF}_4$  that has dissociated, the sum  $[\text{CF}_4] + [\text{F}]/\beta$  is the amount of feed-stock  $\text{CF}_4$ .

Through the ideal gas law, total pressure is proportional to the total number of particles in the plasma. By assuming that  $C_m F_n$ 's,  $m \geq 2$  and  $n \geq 1$ , are negligible in comparison to  $\text{CF}_n$ 's,  $n \geq 1$ —this is expected in a pure  $\text{CF}_4/\text{Ar}$  plasma<sup>17</sup> at the low pressures used in RIE processing<sup>29</sup>—we recognize that

$$\beta \begin{cases} \equiv \frac{[\text{F}]}{\# \text{ of dissociated } \text{CF}_4 \text{ molecules}} \\ \cong \frac{[\text{F}]}{[\mathcal{N}] - [\text{Ar}] - [\text{CF}_4] - [\mathcal{O}]} \end{cases}, \quad (3.7)$$

where  $[\mathcal{N}]$  represents the total number of particles in the plasma and  $[\mathcal{O}]$  is the number of other particles that are not fluorine or argon, or that do not contain a single carbon atom. Then we may write that

$$Prs \cong RT \left[ [\text{CF}_4] + \left( \frac{1}{\beta} + 1 \right) [\text{F}] + [\text{Ar}] + [\mathcal{O}] \right], \quad (3.8)$$

where  $R$  is the ideal gas constant and  $T$  represents the plasma temperature.

Combining Eq. (3.6) with Eq. (3.8) and using Eq. (3.1) to solve for  $[\text{F}]$  results in

$$[\text{F}] \cong \frac{I_F}{I_{\text{Ar}}} K \gamma \left( \frac{Prs}{RT} - [\mathcal{O}] \right) \frac{1}{(I_F/I_{\text{Ar}}) K \gamma + 1}. \quad (3.9)$$

The model-based estimator used here is formed by neglecting  $[\mathcal{O}]$  in (3.9) to obtain

$$[\text{F}] \cong \frac{I_F}{I_{\text{Ar}}} K \gamma \frac{Prs}{RT} \frac{1}{(I_F/I_{\text{Ar}}) K \gamma + 1}. \quad (3.10)$$

### C. Determining the actinometric constant

The constant  $K$  is still unknown. It is defined by

$$K \equiv \frac{\Gamma_F K_F(\epsilon) L_F}{\Gamma_{\text{Ar}} K_{\text{Ar}}(\epsilon) L_{\text{Ar}}}, \quad (3.11)$$

where  $\Gamma_x$  is the branching ratio for emissive de-excitation of species  $X$ ,  $K_x(\epsilon)$  represents the excitation efficiency of species  $X$ ,  $\epsilon$  is electron energy, and  $L_x$  is the equipment optical path loss for the spectral line corresponding to species  $X$ . Several attempts have been made in the literature to estimate a value for  $K$  through modeling. Jenq and coworkers<sup>12</sup> used published estimates and semi-classical theory to estimate that  $K \approx 0.56$ , within a factor of two, while neglecting optical path losses. Dalvie and Jensen<sup>6</sup> estimated a value of 26 for  $K$  on their equipment by matching their experimental data to absolute values calculated from a chemical model. Similarly, Singh *et al.*<sup>24</sup> compared absolute concentration predictions from a model to experimental results by using a value of 0.04 for  $K$ . However, it's difficult to compare these experimental numbers with one another, because the optical path

losses vary between different sets of equipment. In fact, care must be taken to track or minimize non-proportional drifts between the fluorine and argon paths after a value for  $K$  is determined on any given equipment set. The purpose here is to develop a practical methodology for determining  $K$  for a particular equipment set.

Ideally,  $K$  should be determined through a direct, simultaneous measurement of both  $[\text{F}]/[\text{Ar}]$  and  $I_F/I_{\text{Ar}}$ , possibly through mass spectroscopy. In the absence of such equipment, we devised an experiment to calculate a rough estimate of  $K$  that only requires actinometric and pressure measurements. This is important for commercial applications since, in a fabrication facility, most reactive ion etchers are not equipped with expensive and intrusive diagnostic equipment. The experimental procedure makes use of the same concepts employed in deriving the model-based estimator.

Conceptually, the experiment is intended to isolate a known quantity of undissociated  $\text{CF}_4/\text{Ar}$  gas by flushing the chamber without any applied power and then closing off the feed and exhaust flows. Applying power causes dissociation, an increase in temperature, and a subsequent pressure increase, since the chamber is isolated. The pressure increase can then be related to the amount of fluorine released in dissociation.  $K$  may be then calculated through

$$K \cong \frac{1}{(I_F/I_{\text{Ar}})} \frac{1}{\gamma} \frac{Prs_p T_{np} - Prs_{np} T_p}{Prs_{np} T_p}, \quad (3.12)$$

where the subscripts p and np denote the values when power is applied and when no power is applied, respectively.

The pressure data resulting from the experiment described above is shown in Fig. 2. The moment at which power was applied is easily identified by the sharp jump in pressure at 160 seconds. The pressure reached a sharp peak and then quickly decreased to eventually stabilize near 25 mTorr. It is believed that this peak was the result of a fast dissociation process overlapping with slower recombination and polymerization processes which are able to occur in the infinite residence time situation created inside the isolated chamber. Since  $\text{CF}_4$  dissociation occurs first, and then recombination and other more complex reactions follow, the assumption that non- $\text{CF}_n$  molecules are negligible is more valid immediately after the application of power than later; therefore, the moment when pressure reached its peak was used to calculate  $K$ . Temperature changes upon striking the plasma also contributed to the increase in pressure. In order to isolate the effects of temperature on the pressure by eliminating dissociation, the same experiment was repeated with argon gas. A modest pressure increase of only 3.33% occurred in this case, as can be seen in Fig. 2. The fact that the argon experiment saw the pressure jump and then remain nearly constant until the applied power was removed lends credence to the hypothesis that recombination processes cause the slow pressure decay seen during the  $\text{CF}_4/\text{Ar}$  experiment. The calculated value of  $K$  was 29.1.

By fitting a double exponential function (a linear combination of two exponential functions) to the decaying portion of the pressure curve and extrapolating this function backwards to the moment at which power was applied, an upper bound on  $K$  was calculated. This is based upon the idea that

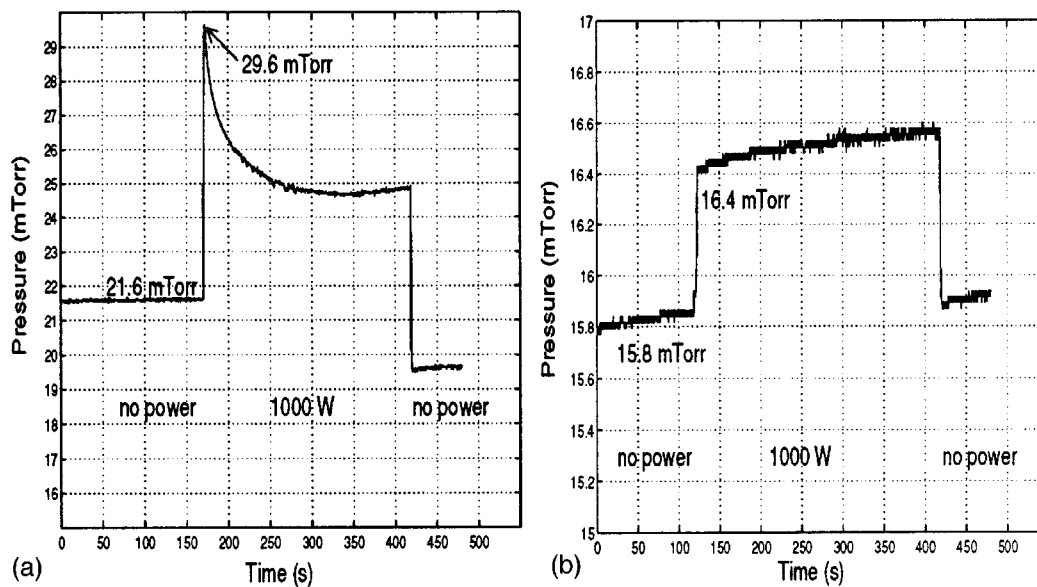


FIG. 2. Pressure variations during the closed chamber experiments. (a) The experiment when a mixture of  $\text{CF}_4$  and Ar are used to fill the chamber, while (b) shows the experiments repeated with only Ar. Both (a) and (b) indicate the pressure immediately before and after the application of rf power.

dissociation begins only when power is applied, quickly reaching a steady state, and is then followed by slower recombination and polymerization reactions. The effect appears to be a fast time constant expansion process overlapping with secondary, slow time constant recombination processes. Some classical calculations predict the temperature changes to require only about 1 ms, and, indeed, the pressure changes were seen to be nearly instantaneous in the argon-only experiment. Thus, temperature effects are much faster than the chemical processes. Calculated in the manner described above, the upper bound on  $K$  was 32.06.

The closed chamber experiment proved to be extremely sensitive to outgassing from the chamber walls. Bouchoule and Ranson<sup>3</sup> showed that the amount of gas that desorbs from chamber walls is a function of the plasma cleaning of the chamber preceding the experiment. Some of their plasma cleans lasted for up to 30 minutes. In order to obtain reproducible results in the  $\text{CF}_4$  closed chamber experiment and to minimize baseline pressure drift during the argon closed chamber experiment, the walls needed to be passivated by running a plasma of the chemical composition and pressure used in the experiment for several hours. Although the cleaning procedure could probably be optimized to reduce the amount of time required in this experiment, that was not the focus of this study. It was hoped that this cleaning procedure would help the experimental results to reflect the chemical scenario described above instead of being dominated by the chamber seasoning history. After maintaining a plasma for several hours, applied power was removed and, after a short pause, the feed and exhaust flows simultaneously closed. Then, pressure fluctuations were allowed to settle for several minutes and the experiment proceeded as described earlier.

#### IV. RESULTS AND DISCUSSION

An independent measurement of fluorine concentration is necessary in order to compare the accuracy of the standard

estimator to the model-based estimator. As our independent measure, we have used time-resolved etch rate measurements during the course of etches in which a real-time controller varies applied power and throttle valve angle to maintain induced dc bias and an estimated fluorine concentration at constant levels. RIE etching consists of two components: a physical component and a chemical component. The physical component is realized through ions accelerating across the sheath voltage and colliding with the wafer. The ion energy is related linearly to the induced dc bias.<sup>15</sup> Free fluorine radicals reacting with silicon constitute the chemical etching component. The physical and chemical etch mechanisms interact at the surface of the wafer where the ions not only sputter silicon, but also remove reaction inhibiting polymer and create activated sites for the fluorine. Therefore, if two etches have identical physical components but varying chemical components, the etch rate will give an indication of the level of fluorine present in the plasma.

#### A. Experimental conditions

Each repetition of the experiment consisted of a set of three etches. The first etch was performed following standard industrial practice: the throttle valve was controlled by a proportional-integral-derivative (PID) controller to maintain a constant pressure of 20 mTorr and applied power was held constant at 1000 W. The other two etches were performed with a multivariable real-time controller<sup>8,9,18</sup> which maintained the induced dc bias and either of the two fluorine estimates at a constant level. Figure 3 illustrates the control scheme through a block diagram.

We would like for the setpoint levels to reproduce the chamber conditions found during an undisturbed, steady state etch. Recall, however, that the chamber is not equipped with a load lock. Therefore, the chamber's interior was exposed to the atmosphere during wafer loading. After exposure to the atmosphere, water vapor outgassing from the

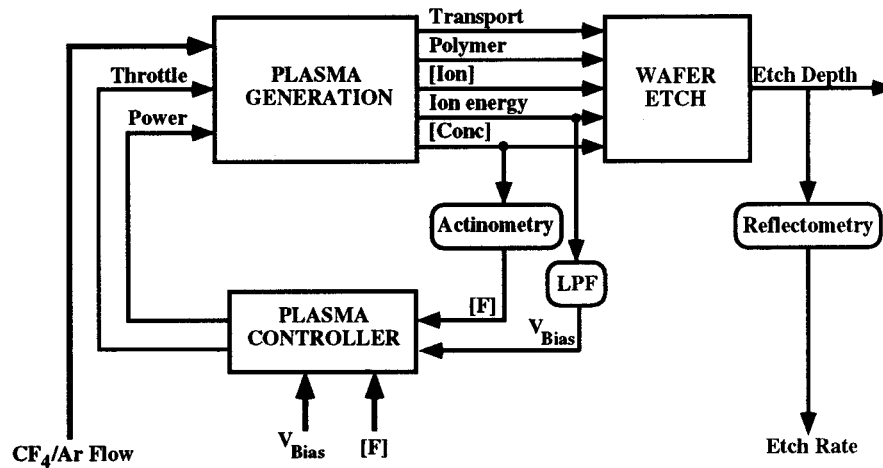


FIG. 3. Block diagram illustrating the conceptual breakdown of RIE etching into subsystems and the control scheme used during etches.

chamber walls constituted a significant disturbance to plasma parameters during etches. The chamber was pumped to a base pressure of  $4e-5$  Torr before beginning the etches. After 20 minutes of the standard practice etch, however, the perturbation caused by the water vapor had largely decayed, and the etch parameters represented an undisturbed system. Therefore, the bias voltage and fluorine measurements taken at that point were used as setpoints for the two closed loop etches. Ideally, a closed loop etch would maintain a constant etch rate that matched the rate measured at the twenty minute point of the standard practice etch.

## B. Experimental results

The etch rate of the standard practice etch was very rapid during the early portions of the etch and asymptotically approaches a rate of  $5.6 \text{ \AA/s}$ , as illustrated in Fig. 4. While an accelerated etch rate may not be detrimental to desired etch characteristics, the accelerated etch rate varied considerably

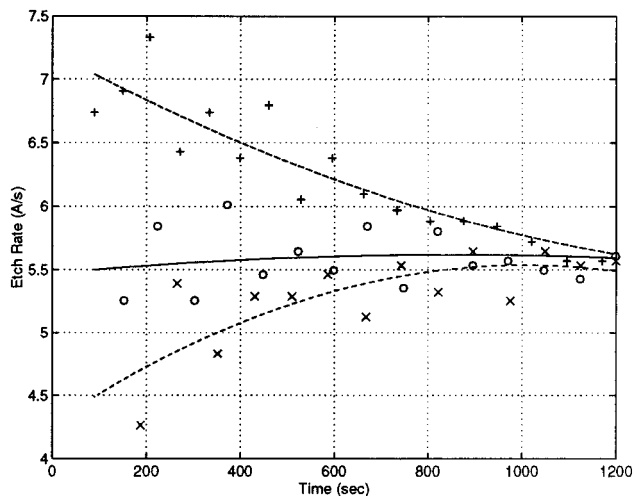


FIG. 4. Etchrate measurements and polynomial fits for (+-dashed) the standard practice etch, (O-solid) the controlled etch using the model-based fluorine estimator, and (X-dashed) the controlled etch using the standard fluorine estimator.

between consecutive standard practice etches. Hence, etch depth reproducibility suffered, reducing etch success. The etch rate was fast because of the chamber exposure to atmosphere and moisture during wafer loading. One reasonable explanation is that oxygen desorbed from the chamber walls and reacted to increase the amount of available fluorine. Also, since no control was exerted over induced dc bias, it was allowed to peak and then decay towards a final value, so that ion energy was also higher during the earlier segments of the etch. In Ref. 11 a control scheme which stabilized pressure and induced dc bias was examined in a similar manner. The etch rate which resulted under that control scheme closely resembled that seen during the standard practice etch, implying that the etches in this operating regime are dominated by the chemical etching component. Therefore, the higher ion energy isn't expected to have a large impact on the standard practice etch rate and etch rate variations are better reflections of fluorine concentration.

Using either fluorine estimation technique, the controlled etches consistently showed reduced sensitivity to the water vapor disturbance on a run-to-run basis; however, the disturbance did induce errors in both of the estimators. The standard estimator (3.4) significantly overestimated fluorine concentration. The desorbed oxygen and subsequent reactions diluted the fraction of argon in the plasma, and, therefore,  $\sigma$  from (3.3) became smaller than it was at the reference point of the standard practice etch. The estimator then overestimated the amount of fluorine in the plasma and caused the controller to compensate by reducing the fluorine level. As the oxygen disturbance decayed, the estimation error diminished, and the etch rate returned to the appropriate level. The model-based estimator more accurately accounted for the change in argon's molar fraction and, therefore, gave improved performance by more closely reproducing the desired etch rate. The model-based estimator's errors resulted from the assumption that  $[O]$  is negligible in (3.9) becoming less true due to the oxygen influx.

## V. CONCLUSIONS AND FUTURE WORK

In this article, we have derived and evaluated a fluorine estimator which uses optical emission spectroscopy and pres-

sure measurements. This estimator accounted for the varying levels of argon dilution that occur due to CF<sub>4</sub> dissociation. Using etch rate reproducibility in the presence of a real-time controller, this estimator was shown to perform better than a standard fluorine estimator. The experimental results affirm the important contribution that phenomenological models can make to process control of reactive ion etching.

We also described an original experiment which was intended to estimate the actinometric constant. This experiment did not require special diagnostic equipment, beyond optical emission spectroscopy, and could, therefore, be used with a variety of CF<sub>4</sub> etchers. Possible interpretations of the experimental results were discussed.

Future work will include the development of more sophisticated model-based diagnostics. Although this static estimator makes use of a simplified chemical model, it falls short of fully exploiting the available knowledge of the plasma chemistry. By more completely modeling the chemical dynamics, we will construct an observer or a Kalman filter. These will provide real-time estimates of additional key plasma parameters and allow better process control.

## ACKNOWLEDGMENT

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