Reducing Process Variation through Multiple Point Crystal Sensor Monitoring

J. Kushneir and C. Gogol, Leybold Inficon, Inc. Syracuse, NY; and
J. Blaise, OFC Corporation, Natick, MA

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ABSTRACT

Optical coating materials often sublime and consequently have time-dependent evaporant flux distributions. This affects surface coverage uniformity and often leads to inconsistent film thickness. Aggregate rate, which represents a survey of the evaporant's flux distribution, can be determined by placing multiple crystal sensors, each at a strategically selected location. Compared to measuring at only one point, this aggregate rate better represents the flux distribution of the deposition process. This knowledge of the evaporant's flux distribution can be used to maximize the number of substrates receiving the desired evaporation thickness. It also improves the reproducibility of the thickness termination. Combining the aggregate rate with data from an optical monitor leads to improved reproducibility in the film's optical thickness.

Two examples which illustrate how multiple point crystal sensor monitoring reduces process variation are presented. In the first, a metal was deposited onto static substrates, and the film thickness reproducibility improved by over 400%, yielding absolute reproducibility of ±0.6% in extremes. In the second, dielectrics deposited onto a rotating dome, absolute reproducibility improved to ±1.1% in batch extremes.

INTRODUCTION

In physical vapor deposition, the variation of the deposited film's optical thickness can be traced back to a change in one of the process's parameters that was not detected nor controlled. A methodology for reducing process variation, due to evaporant flow distribution (EFD) changes, is presented here. EFD is the time dependent geometric flux density map of the evaporant as it is transported from the source to the substrate.

The quartz crystal monitor (QCM) has a successful history[1] as an in situ deposition rate monitor. Generally, the QCM is installed in a vacuum system using one crystal sensor for evaporant flow measurement. The process engineer establishes a scaling factor which is a ratio between the material collected on the substrates and the material collected by the sensor. This factor forms the QCM parameter "Sensor Tooling." This single sensor QCM process control technique provides excellent production yields for many deposition processes.

However, the performance requirements for optical coatings are steadily increasing, dictating reduced variation for each film's optical thickness. As a consequence, the development of improved instrumentation and sophisticated control techniques is a necessity. And, because dielectric materials often sublime, evaporation occurs from their irregular surface instead of from the smooth puddle that commonly forms with metals. During the deposition of a film, the EFD for dielectrics depend on the source's shape and level. Any EFD variation that can change the QCM's Sensor Tooling will add to the process variation.

Simple AR coatings often require 1/4 wavelength films. For visible products, the required films are 1000 to 2000 Å thick. If 5% thickness reproducibility is required to meet performance goals, film thickness variations of 50 to 100 Å are acceptable. These limits are easily within the measurement precision of the QCM. However, high performance coatings often require a thickness reproducibility of 1%. This is especially true for IR coatings. A QCM can easily terminate layers to within 5 to 10 Å. But during deposition, any variation of the EFD alters the Sensor Tooling's connection between the substrate and the sensor. Therefore, thickness reproducibility variations of 3 to 5 % are common.

High performance coatings require a skilled process engineer to minimize the numerous variations affecting the layer's growth and to maintain reproducibility of all of the deposition parameters. Unfortunately, not all parameters can be held constant; source material is consumed in the deposition process. This consumption changes the EFD, frequently resulting in a focusing, or an increase of the evaporant's flow into a particular direction in the deposition system. Mechanisms that cause focusing are: the electron beam gun drilling into the source material, a resistive boat's decreasing source height, and changes in shape of the exposed surface of the source material. These mechanisms will cause the EFD to have a consumption driven time dependency. The crystal sensor and control loop maintains a constant rate of material arrival only at the location of the sensor. Under less than ideal conditions,

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the substrates in the chamber located far from the crystal sensor may accumulate either more or less than the desired film thickness.

If source consumption affects the EFD, the premise that, "A single sensor monitoring only one point in the evaporation chamber adequately controls a deposition" must be questioned. This premise is reasonable only if the deposition source provides a long term *constant* EFD. If a long term constant EFD can be provided, then a constant value for the single crystal sensor's Sensor Tooling can be successfully used. Without a long term constant EFD, Sensor Tooling looses it's connection between the sensor and substrate, causing the deposition rate and film thickness at the substrates to be increased or reduced.

To successfully monitor a system with a changing EFD, it is ideal to place a crystal sensor at each individual substrate's location. When the average of these measurements reaches the desired substrate film thickness, the deposition is terminated. Unfortunately, this sensor placement is not realistic because the substrates must occupy these deposition system locations. Multiple crystal sensor measurement points that sample the substrate surface are the viable alternative.

METHOD OF MULTIPLE SENSOR MEASUREMENT

Using multiple sensor measurement points for statistically improving the reliability and accuracy of a process variable is common practice.

A small area deposition source in a "dome" configuration is displayed in Figure 1.

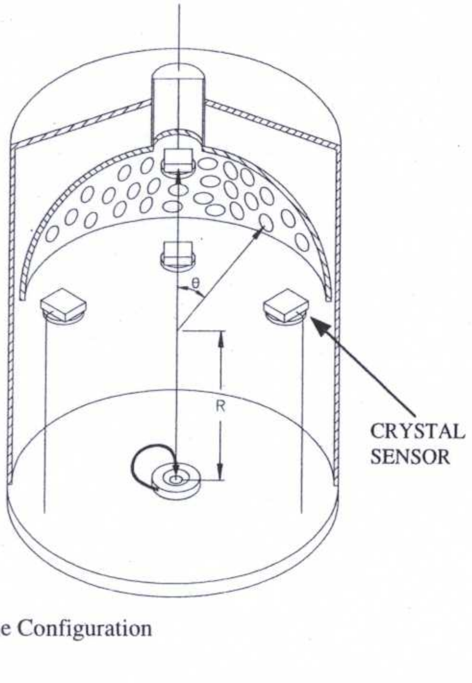


Figure 1. Dome Configuration

The variables that describe the EFD of an ideal small area source are:[2]

$m \equiv$ the mass of material evaporating from the source per unit time [g / sec].

$\rho \equiv$ the material density [g / cm3].

$R \equiv$ the Radius of the dome's spherical surface [cm].

Rate $\equiv$ the thickness condensed per unit time [cm / sec]. It is calculated as follows:

$$\text{Rate} = [\, m / (4 * \pi * \rho * R^2) \,] \qquad \text{[Eqn. 1]}$$

Two quantifiable benefits accrue when using multiple sensors: reduction of the statistical sensor tooling measurement error and an increase in the system's ability to obtain consistent, large high yields.

STATISTICAL SENSOR TOOLING ERROR REDUCTION

The Sensor Tooling parameter is a scaling factor that connects the deposition rate measured by the monitor crystal to the deposition rate arriving at the substrate. It is calculated by forming a ratio of the thickness condensed at the substrate ($t_{substrate}$) to that condensed at the sensor location (t_{sensor}). For ideal small area sources, the Sensor Tooling parameter corrects for the positional difference between the sensor and the substrate locations in the dome system. Real sources, which are usually non-ideal, have an EFD that can vary from deposition to deposition. If the deposition source's EFD is different from the EFD that existed during Sensor Tooling calibration, an error occurs. The relation between the thickness measured at the substrate to that at sensor$_1$ including this "error" term, is:

$$t_{substrate} = (\text{Sensor Tooling}_1 + \text{Error}_1) * t_{sensor1} \qquad \text{[Eqn. 2]}$$

The thickness calculated for the substrate is increased or reduced based on the sign and magnitude of Error$_1$. Suppose a second sensor is added to the deposition system which has an other calibration factor, Sensor Tooling$_2$. Using this second sensor to monitor substrate thickness a similar relationship is found:

$$t_{substrate} = (\text{Sensor Tooling}_2 + \text{Error}_2) * t_{sensor2} \qquad \text{[Eqn. 3]}$$

In general, the Error$_1$ and Error$_2$ values are not equal to each other and are different for each layer. If the sensor with the minimum error in it's measurement can be identified, it will give the closest correlation to the thickness at the substrate. However, without predestine information, the identification of this sensor is not possible. Consequently, averaging the information from multiple sensors to form an aggregate rate can reduce the substrate's thickness error from possibly being a maxima. In this manner, the process engineer is assured of a statistical opportunity for reducing process variation.

If the Sensor Tooling errors happen to be of opposite sign, combining the sensor measurements will partially cancel the error. The averaging and canceling of error in the Sensor Tooling parameter is the motive for placing additional sensors in the system and calculating the aggregate rate. Placing one at the apex (in Figure 1: the apex is where theta = 0), allows the flux at the center of the substrate dome to be monitored. Additional sensors placed around the periphery of the substrates allow the entire substrate dome region to be surveyed (in Figure 1: where theta = max value). Aggregate rate, as computed by combining sensor data from both the apex and peripheral positions, allows the greatest region of the dome to be surveyed.

Aggregate rate, however, does not compensate for less than ideal sensor placement or the reliability of an individual sensor's measurements. A method for calculating a weighting factor that can be applied individually to each sensor must be developed. One method is to calculate the weighting factor on the ability of a sensor position to consistently measure the thickness of a film. At the end of a deposition, the sensor's thickness reading is recorded into a log. After some number of depositions, the standard deviation of the sensor's thickness reading is calculated from the data in the log. This standard deviation value gives an estimate of the amount of EFD change which occurs from deposition to deposition. The sensor's weighting factor might then be chosen to be inversely proportional to this estimate of the EFD's change. The subsequent aggregate rate calculation would weight more heavily those sensors that correlate more consistently with film thickness than would be applied to less consistent sensors.

Another method of calculating a weighting factor is to give additional emphasis ("n" times greater where "n" is the number of peripheral sensors) to the sensor at the apex. This would apply an equal weight to apex and peripheral sensors. Of course, other methodologies for determining a weighting factor can be developed to address particular process concerns.

INCREASING THE ABILITY TO OBTAIN CONSISTENTLY HIGH YIELDS

The dome configuration substrate holder takes advantage of an ideal small area source's EFD. If the actual behavior of the source's EFD was not nearly that of an ideal small area source, the surface of constant deposition rate would not take on the shape of a sphere and the domed substrate carrier would be of little use. Ideally, the deposition rate is uniform everywhere on the dome, implying that a crystal sensor placed anywhere on the surface of the dome will measure a constant deposition rate. Many dome configurations place the crystal sensor at the dome's apex.

Consider the source that becomes focused during the deposition process. In Figure 2, the surface of desired rate changes from matching the dome's surface to the surface labeled SINGLE. SINGLE indicates the surface of the desired deposition rate when measured and controlled by the single apex sensor for a focused EFD. The intersection of where the desired deposition rate surface meets the dome is the optimal region of the substrate holder. If additional sensors are placed around the outer circumference of the dome, they will measure a lower deposition rate. Combining the average rate information from the apex sensor (which is the highest rate) with the sensors at the periphery (which have the lower rate) yields an average, or aggregate, rate that is less than the deposition rate measured by only the apex sensor.

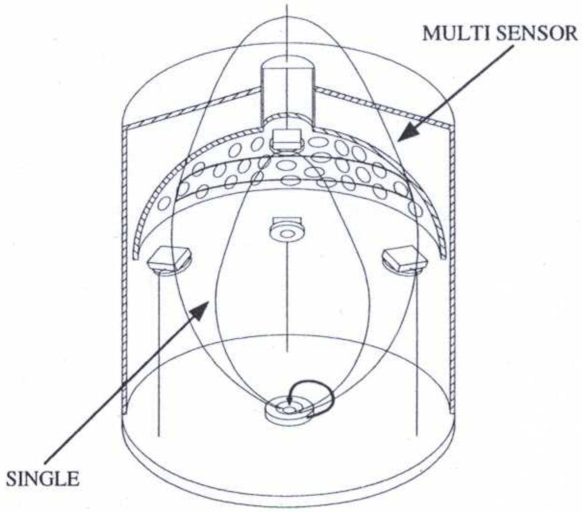


Figure 2. Sensor Location

The deposition controller employs this aggregate rate to effectively move the location of where the desired rate surface of the focused EFD intersects the dome. In Figure 2, the desired rate has been moved from occupying only the point intersecting the apex, to that of a circle where the MULTI SENSOR surface intersects the dome. This movement of the desired rate—from the SINGLE sensor point to the MULTI SENSOR surface—changes the area in the dome that receives the desired rate. The dome's area of desired rate is increased from only occupying the region near the apex's monitor crystal to the larger MULTI SENSOR region, thereby improving yield.

The aggregate rate value is based on the number of sensors used, their individual Sensor Tooling parameter (T_i), and their importance weight (W_i) and the individual rates ($Rate_i$). The automatic recalculation of these weights, should any sensor fail during a process, is through the renormalization of the weighting factors. The equation for aggregate rate is:

$$\sum_{i=1}^{\#\text{sensors}} T_i W_i \text{Rate}_i \Bigg/ \sum_{j=1}^{\#\text{sensors}} W_j \qquad \text{[Eqn. 4]}$$

The use of a multiple point measurement technique will reduce process variation from a source that has a changing EFD. The magnitude of benefit depends on the process's metrics or scales: current yield, measured EFD variation of source material, the cost of a deposition or run, etc. The actual benefits can only be measured by the specific process under evaluation.

EXPERIMENT

Metal and dielectric source materials that have time dependent EFDs were intentionally chosen. Zinc melts at a low temperature, but it forms a partial oxide skin on it's source surface. The oxide skin has a lower vapor pressure at the deposition temperature and it moves around, giving rise to an irregular EFD as the deposition proceeds.

For the oxide materials, TiO_2 and SiO_2 were selected. SiO_2 sublimates, therefore, it has an inconsistent EFD during deposition. During a SiO_2 deposition, the EFD may change dramatically from favoring one side of the deposition system to another.

Metal Film

0.550" diameter gold coated quartz crystals were used as substrates. They were coated with a 290 Å titanium (ρ = 4.50 g/cc) adhesion film, and then with a 3000 Å zinc (ρ = 7.04 g/cc) film. The adhesion film improved the initial Zn condensation coefficient at the start of the coating process. The Leybold Inficon IC/5 deposition process controller employed four sensors with equal weighting to monitor the titanium film and four sensors with equal weighting to monitor the zinc film. Ti & Zn was placed in a four pocket electron beam turret. The deposition system was monitored by crystal sensors which surrounded all four sides of the stationary substrates (19 cm x 19 cm). To measure each substrate's coating thickness, their fundamental frequencies were measured before pump down, and again after the deposition was vented. Using the impedance matching method, the thickness of the nominal 3000 Å Zn film (with a nominal 290 Å Ti film) applied to each substrate crystal was calculated.[3]

To compare the zinc film's reproducibility for all four sensors or any single sensor, examine the average substrates zinc thickness against the individual sensor thickness. In order to calculate the substrate thickness of the Zn layer, the nominal 290 Å Ti thickness was subtracted. In Table A, the column "Substrate Average" is the Zn layer's thickness average derived from ten substrates placed at the center and around the extrema of the plate. (The Ti layer's thickness variation from deposition to deposition, while small in impact, will add noise to the measurement of the substrate's Zn layer calculation.)

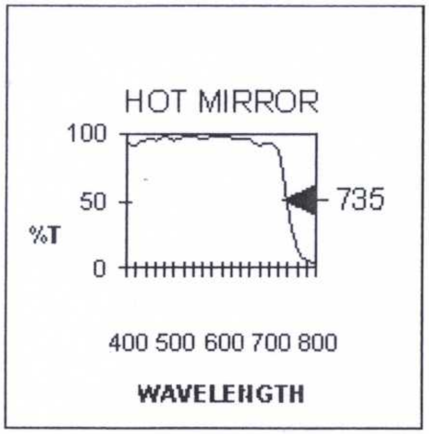
Table A.
Consecutive Ti Zn coatings (Å)

	Substrate Average	Aggregate	Sensor 1	Sensor 2	Sensor 3	Sensor 4
Run 1	3028	3003	3013	3286	2938	2777
Run 2	2996	3003	2836	3138	3103	2937
Run 3	2969	3003	3014	3064	2894	3040
Run 4	3014	3003	2899	2967	3076	3070
Run 5	3018	3003	2920	3029	3059	3004
Run 6	3004	3003	3030	3005	2956	3020
Run 7	3031	3003	2857	3134	3113	2908
Run 8	3024	3003	2747	3065	3234	2970
Run 9	2996	3003	2936	3076	3042	2958
Run 10	3017	3003	2886	3122	3073	2929
Average	3010	3003	2914	3089	3049	2961
StdDev	19	0	89	89	99	83

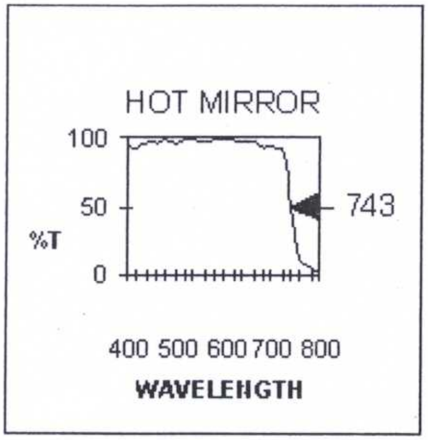
In Table A, the average and standard deviation (StdDev) results show that a Sensor Tooling adjustment applied to each sensor can move the average thickness for each sensor to the 3000 Å target. However, the standard deviation for each sensor can not be as easily adjusted (standard deviation is a measure of process repeatability). A single-sensor deposition controller using any one of the individual sensors for control will exhibit 430-520 % more variation in the layer thickness than would a deposition controller using four sensors.

Optical Materials

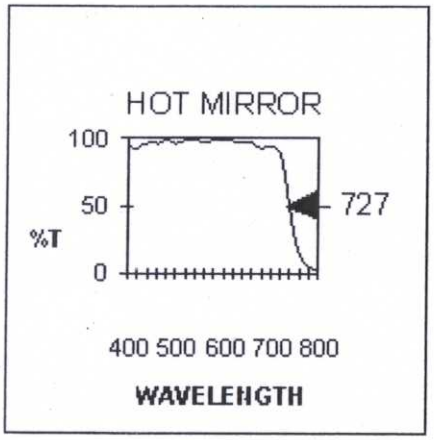
A 13 layer Hot Mirror process using TiO_2 and SiO_2 was evaluated. Ten consecutive batch runs were selected as a measure of the repeatability. The graphs below show the Hot Mirror's transmission cut off. The 50% cut off transmission measured in Run 2 is nominal and is at an extreme in Runs 4 and 10. These graphs show that Run 4 was 1.1% long (thick) and Run 10 was -1.1% short (thin) of nominal. All of the other runs fell in between these extremes. The standard deviation for the ten batches is 0.75% of the nominal cut off wavelength.



Run 2



Run 4



Run 10

When coating large batches of substrates in a 52" coating chamber, a multiple point survey during film growth yields the average, or aggregate, film thickness for the entire chamber. The crystal monitors were strategically placed below the substrate carriers where they will not interfere with the substrates and can survey the largest substrate region. An optical monitor is used to measure the refractive index. The IC/5 was used to control the aggregate deposition rate. A host computer divides the desired optical thickness by the index of refraction to obtain the desired aggregate layer thickness, and updates the IC/5's Layer Thickness parameter. The IC/5 deposition controller maintains the evaporation rate until the desired aggregate layer thickness is reached. This method, developed Jack Blaise at OFC Corporation, has enabled consistent (1% or better) results for large batches. This combination of multiple point crystal monitoring and optical monitoring provides the information critical to enhancing yield.

Ta_2O_3 and HaO_2 are other materials commonly used with this process. These materials are also commonly paired with SiO_2 in multi-layer stacks. OFC Corporation obtains approximately 25% better control with metals, fluorides and sulfides than with oxides.

SUMMARY

Tighter thickness tolerances require enhanced source monitoring techniques. The data presented shows that multiple point crystal sensor monitoring improves the deposition controller's ability to reach the desired process set points for deposition rate and final thickness. Stated simply, the multiple point technique provides a more thorough survey of the EFD in the deposition system. This wider coverage is used to increase the area that receives the desired rate during the deposition process and also provides a statistical advantage with regard to Sensor Tooling error reduction. These advantages are employed to either tighten process capability or introduce new film processes previously prohibited because of poor yields.

Using multiple point crystal sensor monitoring has other advantages during coating. Sensors placed in the deposition chamber can gather in situ information on how the evaporant flow distribution changes with time. This can be used to quickly characterize the source materials EFD, and changes can be made to the process to reduce the variation.

ACKNOWLEDGMENTS

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