In-Situ Optical Monitoring and Rate Control of Thin-Film Deposition Using Index Dispersion Enhanced Monitoring (IDEM)

Running title: Thin-Film Deposition Rate Control Using Index Dispersion Enhanced Monitoring

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A new Index Dispersion Enhanced Monitoring (IDEM) technique has been developed for controlling the rate of material deposition during thin-film vacuum deposition. It provides a highly accurate method for measuring thin-film optical properties and for executing coating layer cut-off. During deposition the dispersion monitoring system provides real time determination of each film layer's optical thickness to better than 1nm resolution. Its accuracy and repeatability are described here along with secondary benefits including process drift notification, process interruption recovery, deposition rate optimization, and elimination of crystal rate monitoring. IDEM In-Situ full spectrum optical dispersion monitoring and deposition rate control has been tested in both research and commercial use at several institutions. Dispersion Enhanced monitoring of 6 different repetitions of a multiple layer per witness chip coating and of a complex 37 layer coating are explained and presented.

I. INTRODUCTION

Index dispersion is the variation of refractive index of a material as a function of wavelength. An Index Dispersion Enhanced Monitoring system (IDEM) measures a thin film's optical properties as part of the deposition process control in the same way that a final spectroscopic examination of a finished multilayer coating is done with a spectrophotometric scan. Limitations common to other monitoring techniques due to stress, density, material variations, temperature, etc.[1] can be avoided by including index dispersion when monitoring the optical thickness of a coating layer.

IDEM has a number of advantages over traditional intensity of reflection or transmission measurements. Since the shape of the spectrum is being measured in addition to the intensity the system is immune to small fluctuations in light source intensity. Calculation of the optimum monitor wavelength as used in single wavelength monitored systems is no longer necessary because the measurements are done over the entire range of wavelengths. The accuracy of IDEM allows multiple layers to be deposited for each witness chip and makes it possible to recover from process interruptions without losing the coating run. IDEM also provides real time process monitoring. Changes to the system that affect dispersion are immediately visible during coating as a mismatch between the predicted spectral shape and intensity and the actual spectrum monitored.

In situ dispersion monitoring has been applied to a number of deposition techniques over the last several years with excellent results. Techniques used to date include E-Beam, Sputtering, and Ion Beam Deposition of many different materials (SiO_2, Al_2O_3, TiO_2, MgF_2, Y_2O_3, HfO_2, ZrO_2, Ta_2O_5, YF_3, and ThF_4).

The difference between the IDEM technique and previous coating techniques is that the final optical properties of the film are measured in real time rather than post coating. This is accomplished by calibrating each coating process for the machine that will be depositing it. Experience has shown that once calibrated, the coating properties of a machine remain fairly constant. Any changes that do occur are highlighted by the IDEM system during the deposition, appearing as deviations between the deposited coating spectrum and intensity and that predicted from the calibration. This provides the system operator with the information necessary to identify sources of machine coating variability.

II. IDEM Technology

Several technical advances contribute to IDEM's enhanced functionality and are detailed below.

A. Photodetector Linearization

An important part of the technology enabling the IDEM system is a proprietary three stage optical filtering system that flattens the photodetector array's spectral response. It is desirable to have a low noise photodetector array to obtain an accurate and consistent curve fit for index dispersion during calibration and also later during coating. Due to the variation of photodetector response with wavelength however, measurements at shorter and longer wavelengths (400nm and 900nm, respectively) are up to 20 times noisier than those at peak sensitivity wavelengths.

The SpectraLock IDEM system circumvents this limitation by optically linearizing the spectrum from 400nm to 900nm. Instead of a 20 to 1 variance across the spectrum the intensity is within +/- 10% for all wavelengths (Figure 1). Signals from the photodetector array are integrated over a short time interval to collect sufficient signal for high quality readings. The result is a signal to noise ratio in excess of 1000 to 1 for each point on the measured spectrum, captured at more than 5 samples per second.

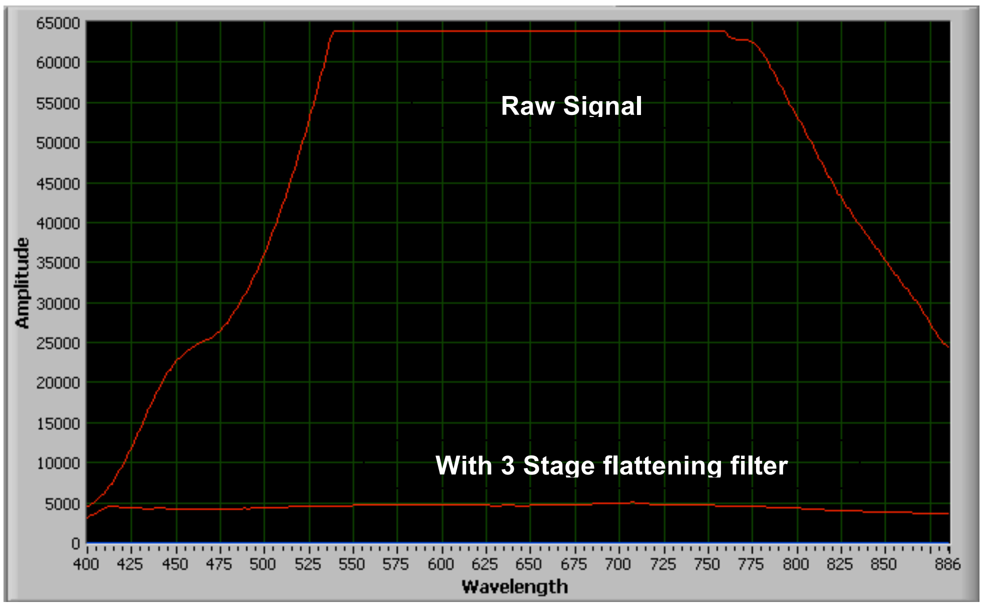


Figure 1 Photodetector Signal with and without Linearization Filter

B. Index Dispersion

Index Dispersion is the intrinsic change in refractive index, and hence reflectivity, of a material across the spectrum[2]. Because of this property the 1/4 wave reflectance peak of a coating being deposited differs in intensity depending on what wavelength it is measured at.

The shape of the index dispersion curve (Figure 2) is unique and repeatable for each material deposited with a specified set of process parameters (coating rate, oxygen level, and substrate temperature) in a particular coating system.

The ¼ λ peaks progress from left to right as the coating is deposited, repeatedly forming the Index Dispersion Curve shown in white near the top of Figure 2. The ¼ λ troughs follow the Index Dispersion curve for the substrate material shown in red near the bottom of Figure 2.

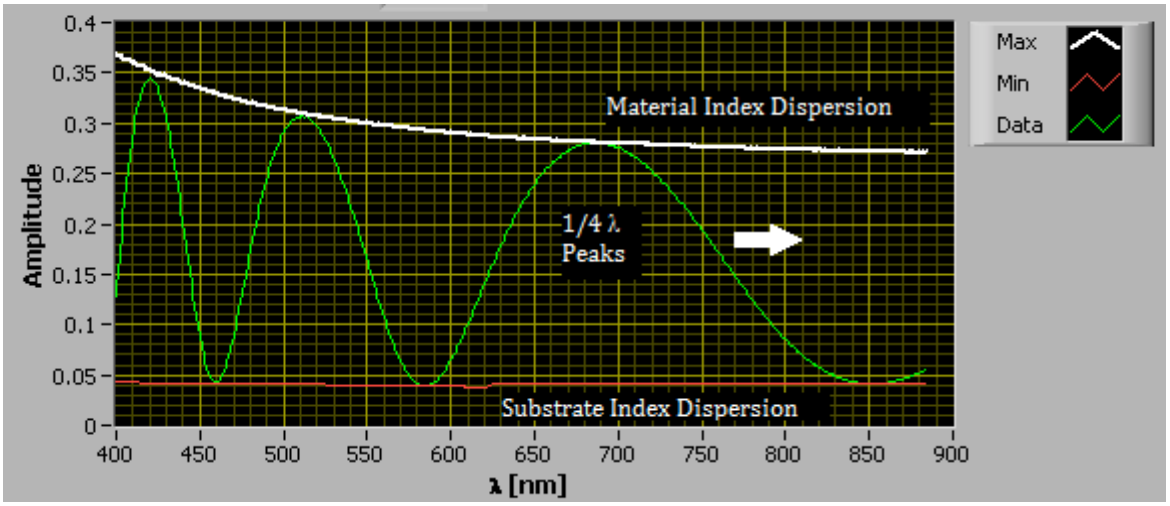


Figure 2 Index dispersion curve obtained during an IDEM calibration run

C. Calibration

In order to obtain the unique Index Dispersion curve for a coating process the IDEM system requires an initial calibration run. A typical calibration run involves the deposition of 2000 to 3000nm of material that provides multiple 1/4 wave transitions across the monitored spectrum. The Index Dispersion curve for a given process is determined during this run and then is fit to a Sellmeier equation.

Graphs of the reflectance vs. wavelength provided at the conclusion of the calibration allow for verification of the quality of the fit between the calibration derived curve and the actual measured intensity (Figure 3).

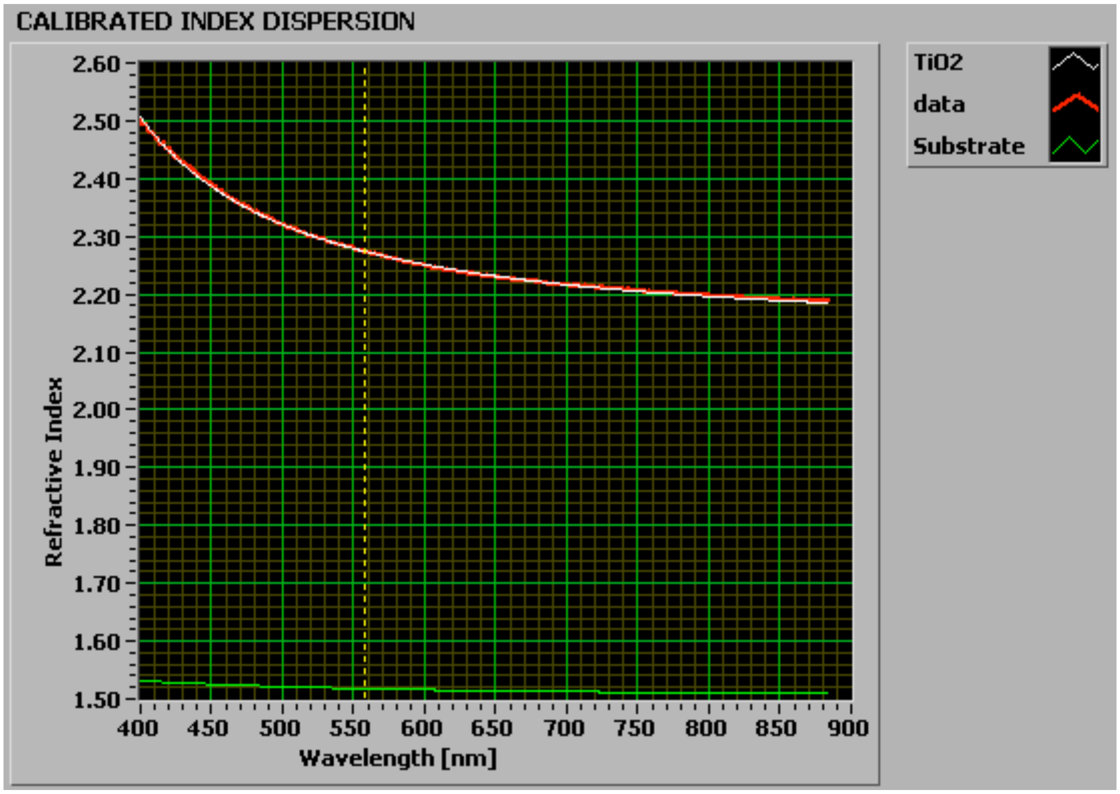


Figure 3 Index calibration curve compared to measured index dispersion

A poor fit may occur if a process parameter changed during the calibration run or if the coating process itself is unstable due to, for example, an excessive deposition rate. In extreme cases a poor fit can cause the curve fit calculations to fail entirely. If a failure to fit occurs the process and equipment should be evaluated for issues affecting stability before attempting another calibration.

A 3-D graph of the Reflectance vs. Wavelength vs. Time (Fig. 4) is also produced to provide a visual representation of the entire calibration run's results. The smooth ripples shown illustrate the number of ¼ wave transitions as they accumulate with increasing optical thickness.

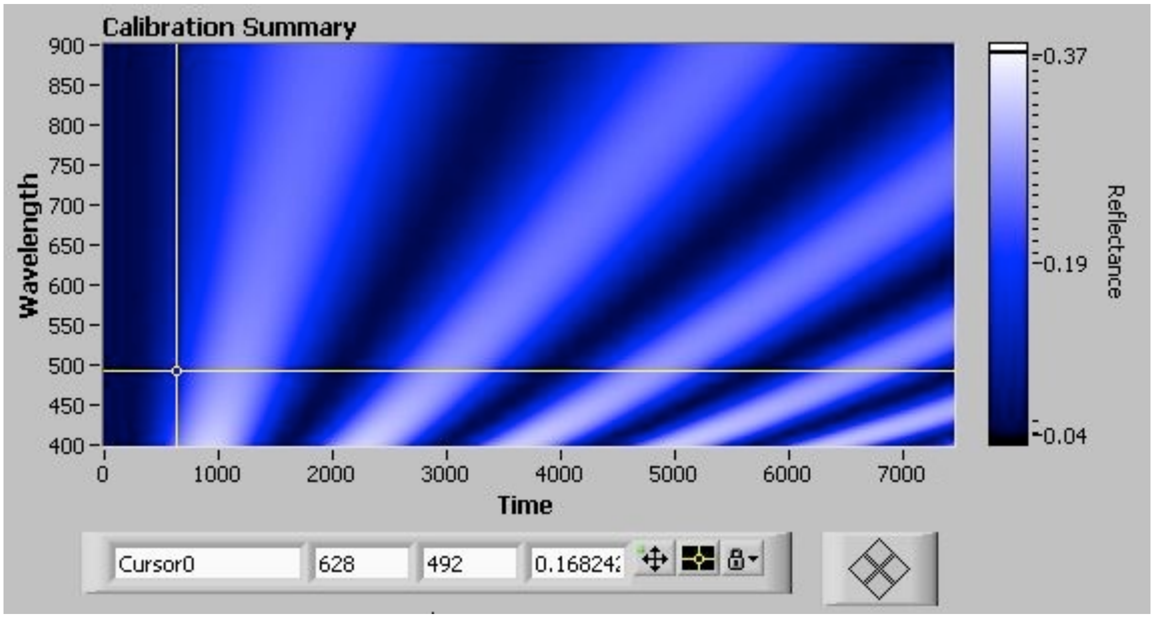


Figure 4 Full Calibration Reflectance vs. Wavelength vs. Time (Thickness)

D. Coating Thickness Control

An accurate measurement of the index dispersion allows coating thickness control to be achieved by fitting the modeled reflectance curve to the entire measured spectrum. Coating cutoff is controlled by an estimate of the optical thickness based on this curve fit.

The unique Index Dispersion properties of a specific material coated in a particular machine will not change unless the something in the system does. This can occur as a slow drift, which can be addressed by periodic recalibration without significant loss of interim coating quality.

E. Deposition Rate Control

By fitting the modeled reflectance curve to the entire measured spectrum and performing continuous measurements of optical thickness the IDEM system is also able to control deposition rate. A continuous calculation of optical thickness and its rate of change allow the IDEM monitor to provide a rate control signal to the deposition source system whether it be E-Beam, sputtering, ion beam, or other. A crystal monitor is therefore no longer needed for deposition rate control of transparent materials. Crystal monitor related problems such as crystal failure, increased noise with thickness, and fluctuations due to crystal temperature and stress are eliminated.

III. EXPERIMENTAL

In order to measure the accuracy of optical coatings deposited using IDEM rate and thickness control a series of films were deposited without using a crystal rate monitor.

A. Experimental Setup and Methodology

1. Equipment

- 0.91 meter (36") diameter optical coating system (Eddy Company, Apple Valley, CA)
- SL-2010C SpectraLock IDEM Optical Monitoring System (Eddy Company, Apple Valley, CA)
- CC-60 Chip Changer (Eddy Company, Apple Valley, CA)
- XYC-20 Electron Beam Gun Sweep control (Eddy Company, Apple Valley, CA)
- Model 271 Electron Beam gun, 4 pockets, (Telemark, Inc.)
- V670 Spectrophotometer, (Jasco, Inc.)

The equipment setup is shown in Figures 5, 6, and 7.

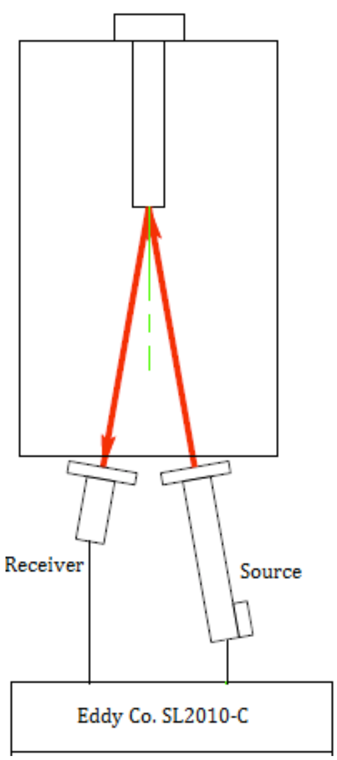


Figure 5 IDEM schematic diagram

Figure 6 IDEM Installed Source & Receiver photo

Figure 7 Coating System with SpectraLock IDEM installed

2. Coating Materials

- Silicon Dioxide (SiO_2), 3-6.5mm granules, EM Industries
- Titanium Dioxide (TiO_2), 2gm pellets, EM Industries
- BK7 Witness chips 21.59mm x 21.59mm x 1.57mm
- BK7 Substrate slides 21.59mm x 21.59mm x 1.57mm

3. Coating Methods

All substrates and witness chips were allowed to stabilize in the coating chamber at 260ºC and 3.0×10^{-6} pressure for 1.5 hours before each coating run. SiO_2 was coated at 0.6 nm/second while TiO_2 was coated at 0.2 nm/second. Coating was performed by E-beam evaporation using the equipment listed.

4. Coating Design

For IDEM demonstration purposes a 5 layer coating 572nm bandpass coating was designed. The coating sequence was high index, low index, double high index, low index, high index using SiO_2 and TiO_2.

5. IDEM Calibration

Prior to fabrication of the designed coating, a calibration layer of 2000nm-3000nm was deposited on a witness chip and a sample substrate for each coating process to be used on this machine at the rates listed in the Coating Methods. Following each calibration run the IDEM system software calculates the refractive index and index dispersion of the material and stores the curves for future use. A substrate sample produced during each calibration run was used to determine the monitor to work ratio for each coating process. The monitor to work ratio is a standard optical coating system parameter used to compensate for the difference in coating path for the witness chip and the sample substrate.

B. Experimental Design

Single layer films of SiO_2 and TiO_2 were deposited at previously calibrated rates on BK7 substrates. It is important for IDEM to use glasses such as BK7 that display very consistent refractive indices and dispersion properties throughout the spectrum[3]. Monitor chip properties are an important factor in calculating optical thickness. Index Dispersion Enhanced Monitoring makes it possible to do true "optical rate" monitoring from fractions of a nanometer/second up to multiple nanometers/second. Its pre-set parameters (determined at calibration) are similar to those of a quartz crystal monitor except that corrections for density, tooling factor, acoustic impedance, cooling water flow, and temperature fluctuations are unnecessary. All coatings were deposited using only SpectraLock IDEM rate control.

The 5 layer coating described in the Coating Design section was deposited 6 times on new substrates in separate runs in the following order:

Run # **Coating Description**

1. 5 monitor chips were used, one for each layer deposited
2. 3 monitor chip were used, one for layers 1 & 2, one for layers 3 & 4, and one for layer 5
3. 2 monitor chips were used, one for layers 1, 2, & 3 and one for layers 4 & 5
4. 1 monitor chip was used for all 5 layers
5. 1 monitor chip was used for all 5 layers. The coating was interrupted in the middle of layer 3 by pulling a USB connecting cable out of the SpectraLock causing it to reboot.
6. 1 monitor chip was used for all 5 layers. The coating was interrupted during the first layer by an operator error.

The reflectance spectrum for each completed coating was measured within 20 minutes of removal from the machine and again at 3 days. The results are shown in Figures 8, 9, & 10 and Table 1.

IV. RESULTS AND DISCUSSION

A. Spectroscopy of 5 layer coatings

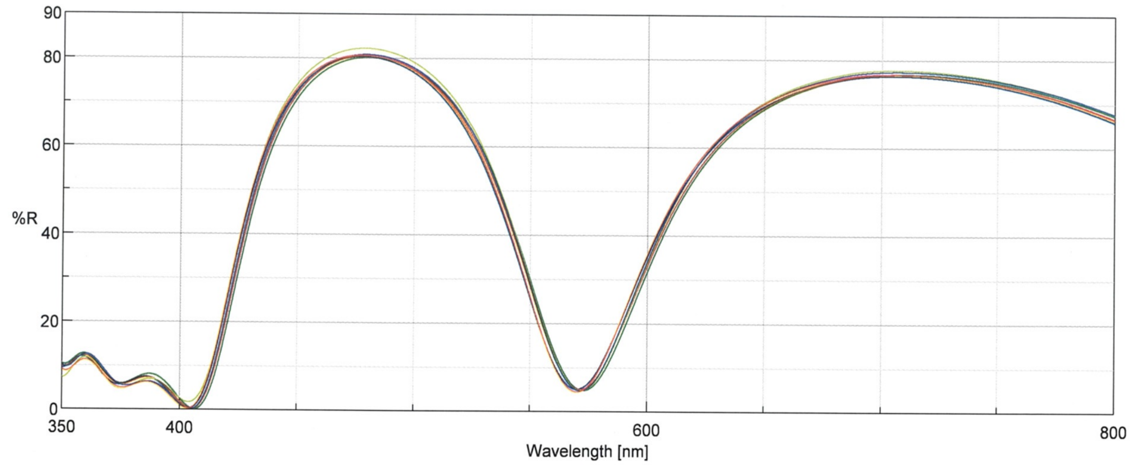
The reflectance spectra for each of the six 5 layer coatings was measured with the spectrophotometer and overlaid in the chart of Figure 8. Additional detail zoomed in on the 572nm reflectance trough for each spectrum is shown in Figure 9.

These results are summarized in the Table 1. The standard deviation of the trough wavelength was within 0.2% or approximately 1nm.

Table 1 Six Repetitions of a 5 Layer Coating – Trough Wavelength

Coating Number	**Description**	**Wavelength**
1	1 layer/chip	572.8
2	2 layer/chip	570.1
3	3 layer/chip	571.7
4	5 layer/chip	571.9
5	Interrupted	571.7
6	Interrupted	570.1
Mean		571.383
% Deviation		0.188

The trough wavelengths were also measured 3 days later to compare the initial values with those after stabilization. The mean was found to have stabilized at 573.23nm or 0.323% higher.



[Comments]
Sample name
Comment
User
Division
Company Eddy Co

[Detailed Information]
Creation date 2/14/2013 2:41 PM

Data array type Linear data array
Horizontal axis Wavelength [nm]
Vertical axis %R
Start 800 nm
End 350 nm
Data interval 0.1 nm
Data points 4501

[Measurement Information]
Instrument name Jasco V670
Model name V-670
Serial No. A041061154

Photometric mode %R
Measurement range 800 - 350 nm
Data pitch 0.1 nm
Band width(UV/Vis) 2.0 nm
Band width(NIR) 8.0 nm
Response Slow
Scanning speed 40 nm/min
Source change 340 nm
Grating change 850 nm
Light source WI
Filter exchange Step
Correction Baseline

1 - 5 Chips.jws
2 - 3 Chips.jws
3 - 2 Chips.jws
4 - 1 Chip.jws
5 - 1 Chip B.jws
6 - 1 Chip C.jws

Figure 8 Coating Spectroscopy

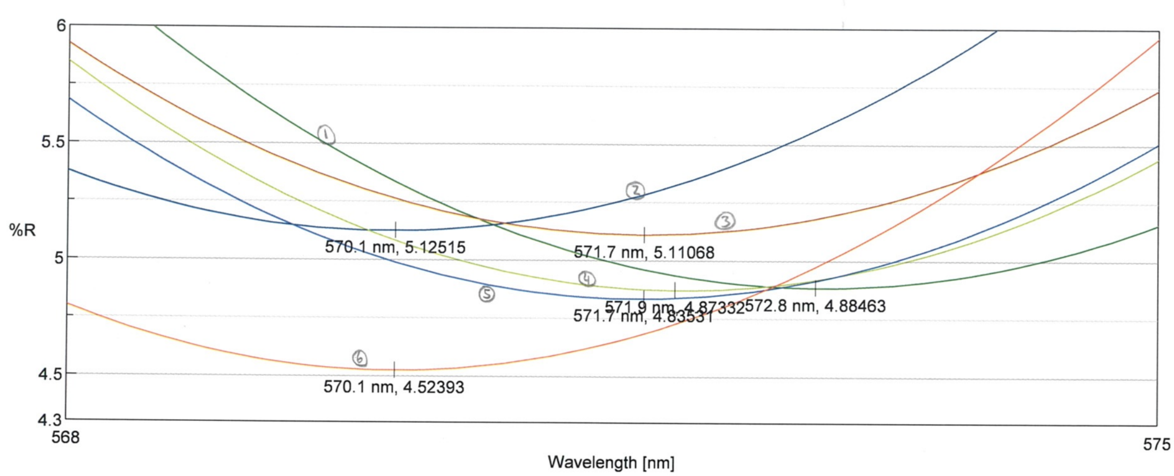


Figure 9 Trough Details (568nm - 575nm)

B. IDEM Control Display

Figure 10 shows the SpectraLock display at the conclusion of Coating Run #4; performed by applying 5 layers to the first monitor chip. The blue line shows the calculated spectrum derived from the coating design and calibrated deposition rate. The red line shows the measured spectrum of the witness chip. These lines deviate from each other when a variable in the process has changed. Figure 11 illustrates an unrelated coating run that experienced deviations due to a change in raw material lot. Subsequent recalibration restored the accuracy of coating control for this system.

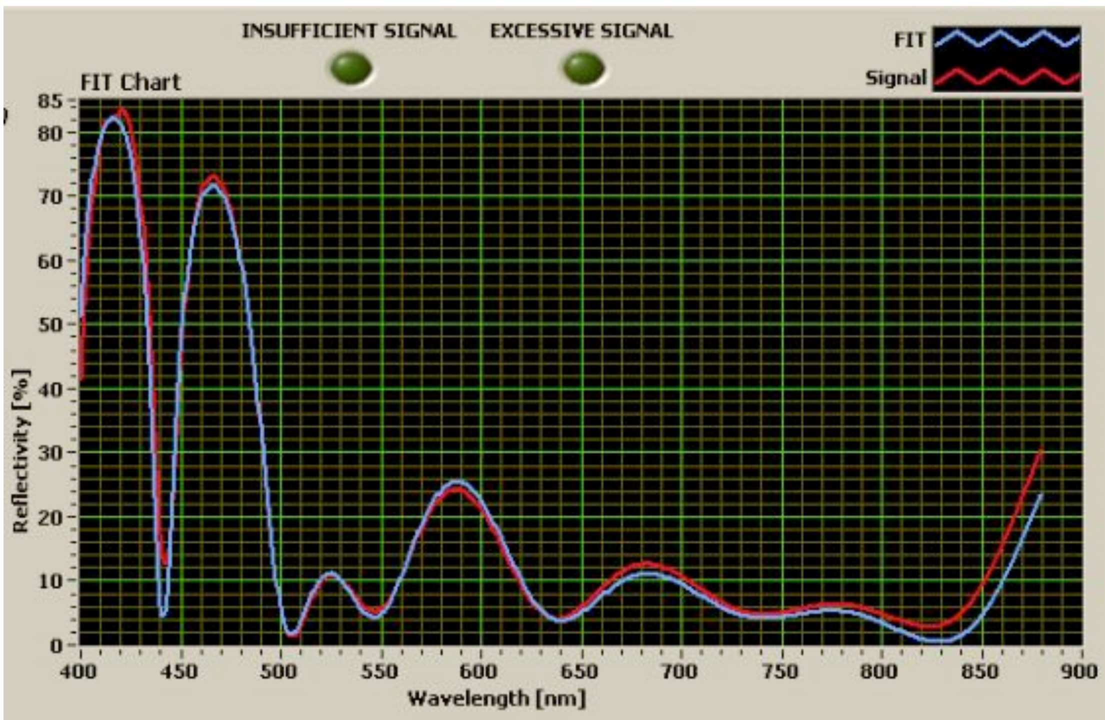


Figure 10 SpectraLock Fit display - Coating Run 4

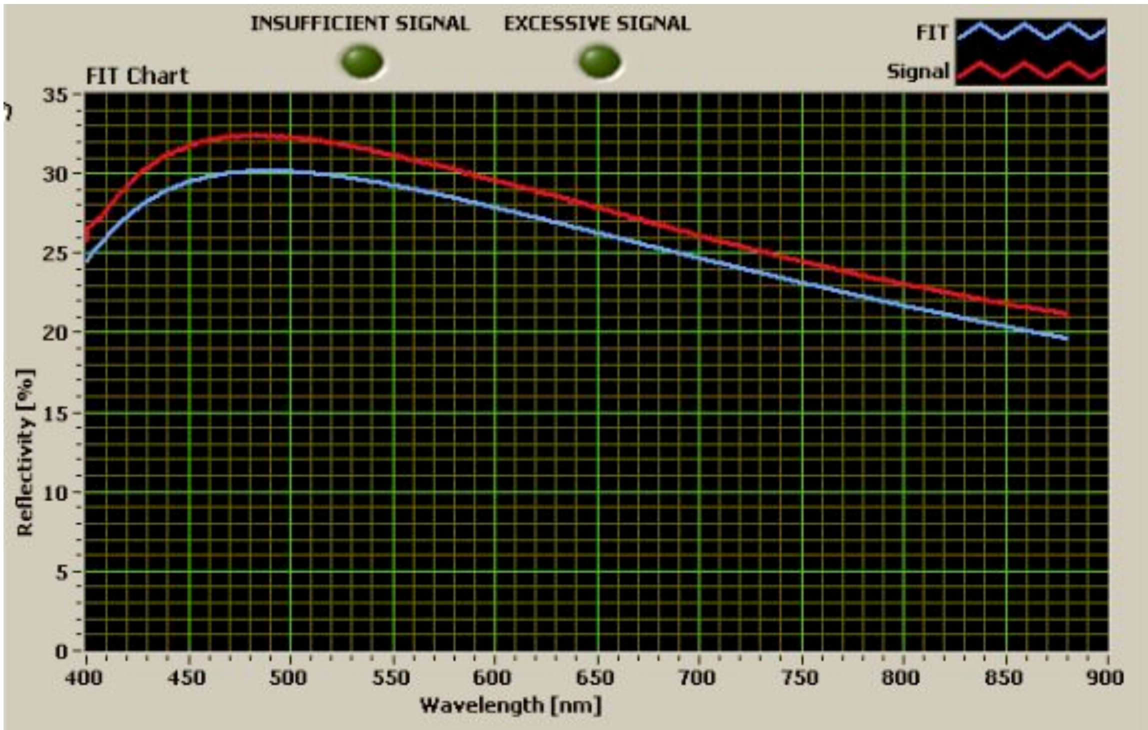


Figure 11 Unrelated Coating - Example of fit needing recalibration

C. Discussion

The IDEM system highlights discrepancies between the predicted coating properties and the actual coating properties in real time. This provides the system operator with information needed to identify changes and make corrections as needed. One benefit of this information is the ability to optimize coating speed. Attempts to coat too rapidly for the desired accuracy can result in unstable coating processes. The unrelated Fit chart in Figure 11 illustrates this case. The SpectraLock IDEM system has similarly been used to identify opportunities for greater coating speed. Calibrating at increasing deposition rates until a threshold of Index Dispersion Fit stability and acceptable optical transmission properties is reached can provide improved system throughput.

Unforeseen circumstances can sometimes cause interruptions in a coating run. The use of IDEM allows for recovery and resumption of coating from where they were paused. IDEM control starts immediately upon resumption due to full spectrum index fit matching.

D. Commercial Use Example

To investigate the performance of IDEM in commercial use an example spectrum was obtained for a complex production coating. The results for a commercial 37 layer Hot Mirror of SiO_2 and Ta_2O_5 coated using an IDEM system for both optical thickness and deposition rate control were reviewed. The comparison of the Theoretical and Actual transmission spectrums is charted in Figure 12.

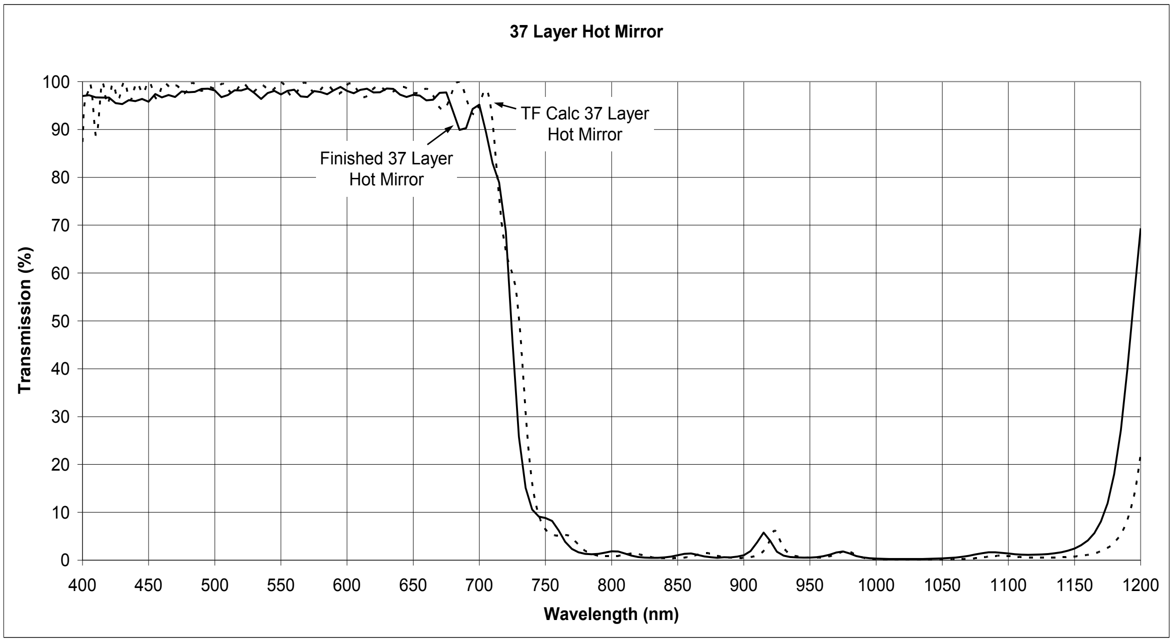


Figure 12 Complex 37 Layer Coating produced with IDEM control

While the conformance of the actual to the theoretical curve shown in Figure 12 is good, the remarkable aspect of this run was its ease of manufacture. No calibration was required prior to the run as one had been performed many days previously. No pre-run was needed to verify that the system was performing well that day. The coating design was simply loaded into the controller, the parts were put in the vacuum chamber, and the run was started knowing that the finished product would come out properly coated.

V. CONCLUSIONS

The results of this work demonstrate that IDEM systems provide accurate real time control of thin-film optical deposition. Multilayer coatings produced with IDEM control are precise and repeatable to better than 0.2% resolution. IDEM systems highlight discrepancies between predicted coating properties and actual coating properties in real time. This provides system operators with the information necessary to identify unanticipated changes and then to make corrections as warranted.

Substrates coated using IDEM optical monitoring systems consistently match their design models. IDEM makes it possible to provide rate control of transparent material deposition without a crystal monitor; to monitor the thickness of low index materials as accurately as for high index; and to optimize coating operations for more efficient production.

ACKNOWLEDGMENTS

[1] S. Grimshaw, “Quartz Crystal Thin-Film Monitoring Forges Ahead”, Photonics Spectra April 2003, p. 82

[2] S. Weber, Handbook of Optical Materials, CRC Press, 2003

[3] N-BK7 Datasheet, Schott Glass, 9/19/2007