Deposition of Optical Coatings with Real Time Control by the Spectroellipsometry

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Abstract

In-situ spectroscopic ellipsometry is known to be very sensitive, non-invasive technique for monitoring and control of thin film growth. In the production of optical interference coatings the refractive index of the material is usually assumed to remain constant within a single layer. Under such assumption only the optical thickness of the layer can be efficiently controlled. For modern complex structures, however, even insignificant deviation from the design, due to shift in the refractive index, can be very detrimental to the resulting performance of the coating. Simultaneous real-time determination of refractive index and growth rate is necessary in order to comply with strict specifications. If the index departs from the target value, one has to adjust process parameters and, ultimately, perform re-calculation of the filter structure to compensate for an error. We will review the work performed in our laboratory during last few years. Development of several different control strategies will be discussed and their application to the control of PECVD of optical interference coatings demonstrated. Latest work is concerned with advances in the closed-loop control of the fabrication of optical thin films by in-situ multi-wavelength phase-modulated kinetic ellipsometry using both, direct numerical inversion algorithm for the real-time reconstruction of refractive index and layer thickness and real-time least-squire fitting-based approach. These techniques have been tested on quarter-wave index optical filters as well as on inhomogeneous refractive index profiles and demonstrate efficiency and robustness.

1 Introduction

With optical structures becoming more and more complex, the cost of possible process failures could rise to unaffordable levels. The complex multilayer designs of modern filters require high fidelity of the fabrication process and reliable control system. Therefore, demand is growing for reliable in-situ monitoring and control techniques for rapid real-time diagnostics of the film and of the process. In-situ transmission and reflection spectroscopy, though very valuable techniques, together with quartz crystal monitoring, are not sufficient anymore. It is necessary to be able to probe the fabricated structures during the process on any type of the substrates (not only on transparent). Sometimes, only optical ports at oblique angles of incidence may be available on the deposition system. Another problem is highlighted by the rising interest of optical coatings community to plasma enhanced chemical vapor deposition (PECVD) with its relatively high uncertainty for refractive index of material.

Due to its phase sensitivity, in-situ spectroscopic ellipsometry has been found to be one of the most valuable tools for performing the monitoring and control of the deposition of multilayer and gradient optical structures [1]-[4]. Another great advantage of ellipsometry is its insensitivity to the variations of intensity of light source. The control of the etching, where all indices and thickness are known beforehand is rather straightforward (especially on non-structured surfaces) and shown to be quite accurate [5]. As for the deposition, a tremendous amount of work was done on epitaxy (particularly of III-V compounds) and it is currently technology most developed in the sense of ellipsometric control of the process [6]. On the other hand, optical thin films are less studied area despite obvious advantages that ellipsometry can bring into it.

We would like to summarize the development and applications of different control algorithms and strategies for optical thin films growth developed in our laboratory during the last years.

2 Deposition Reactor and Control System

The control experiments reported here were performed in the high-density plasma reactor based on electron cyclotron resonance (ECR) effect. It is a further development of the distributed ECR line [7],[8], matrix distributed electron cyclotron resonance plasma system, thus abbreviated as MDECR. The deposition system in full will be presented elsewhere [9].

The MDECR system shown in Fig.1 consists of a plasma reactor, microwave system, pumping station, gas panel and control system. The reactor is a stainless steel chamber fitted with optical and diagnostics ports and movable substrate holder with a 40 × 40 cm^2 area. System equipped with 8 gas lines with computer-controlled mass flow controllers (MFCs). Argon, O_2 and N_2, are supplied through a grid located behind the most intense plasma region. Silane is injected in the front of the substrate.

A phase-modulated spectroscopic ellipsometer (PME) UVISEL from Jobin Yvon (JY) / HORIBA is installed on the system. The ellipsometer is mounted in-situ at an angle of 73.4°. Spectroscopic measurements from 1.5 to 5 eV can be performed as well as kinetic measurements on 32 wavelengths spaced from 1.45 to 5.5 eV. Multi-channel acquisition unit allows measurements to be taken in parallel with typical integration times of 200 ms.

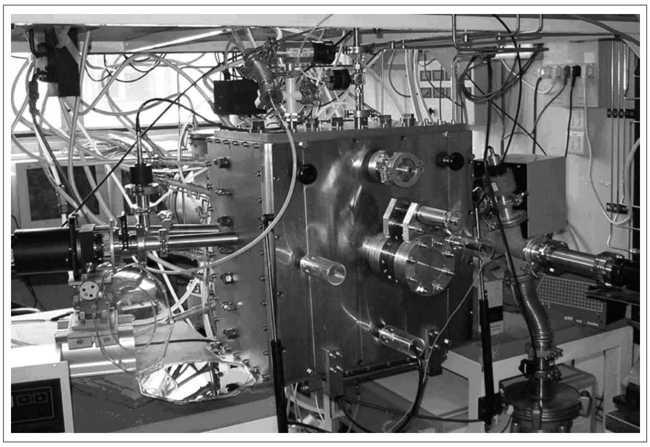

Fig. 1 MDECR-PECVD Deposition Chamber Equipped with JY HORIBA UVISEL In-situ Ellipsometer
(view from the front loading door side)

The ellipsometer is linked via a TCP/IP connection over local network to the control PC running proprietary software for treatment of ellipsometric data, adjustment of acquisition parameters and growth control, which, in its turn, communicate with system controller, responsible for vacuum, microwave and gas flows management via Siemens PROFIBUS network. Such task sharing allows easily upgrade PC involved in calculations each time the complexity of the algorithms increases and demand more computing power.

3 Control by Optics

Thin film filter is an optical device that changes properties of reflected or transmitted (or both) light in pre-determined fashion. Logically, the best way to control the optical response is with optics.

Historically, however, first control techniques utilized quartz crystal monitoring [10]. It proved to be very reliable, but, since it does not provide for the measurements directly on the substrate, required perfectly known flux distribution inside the deposition system. This is rather straightforward for thermal evaporation, but less so many other deposition techniques, including PECVD. Another weak point is that precisely known density of material is assumed, which is not always the case.

Transmission spectroscopy, especially broadband monitoring, allowed attaining remarkable success in the accuracy of the deposition and in a widespread use currently [11],[12]. Its main disadvantage is the fact that it can only be used on transparent substrates and films (at least in the part of wavelength range) of appreciable thickness. Also, stable light sources are necessary in order to control the growth, since transmission is intensity-only sensitive.

Ideal technique shall be reflection-based, and preferably have both, amplitude and phase sensitivity. Such technique is an ellipsometry. Ellipsometry is based on measuring the ratio ρ of the complex Fresnel reflection coefficients for an *s*- (r_s) and *p*- (r_p) polarized light [13]:

$$\rho = \frac{r_p}{r_s} = \tan\Psi e^{i\Delta} \text{ ------ (1)}$$

Phase-modulated ellipsometer (PME) determines ellipsometric angles Δ and Ψ (and r_p and r_s coefficients afterwards) from the parameters I_S and I_C, that are the first and the second harmonics of base modulation frequency of the polarized light registered by the detector after reflection from the sample surface:

$$I(t) = I_1\,[I_0 + I_s \sin\delta(t) + I_c \cos\delta(t)] \text{ ----- (2)}$$

Those parameters (I_S and I_C) are obtained directly from the digital signal processing unit of the ellipsometer, while all subsequent calculations related to the sample are done by external computer assuming certain structure of it (note, that as usually in optics, all the results in the ellipsometry are obtained via modeling and, thus, choice of correct model is a crucial step in the data interpretation process).

When modulator and analyzer angles are 0 and 45 degrees (most convenient for real-time studies configuration of PME), respectively, those I_S and I_C parameters can be related to complex Fresnel reflection coefficients via relations (3).

$$\begin{aligned} I_s &= 2\,\mathrm{Im}\left(\frac{r_s r_p^*}{r_s r_s^* + r_p r_p^*}\right) = \sin 2\Psi \sin\Delta \\ & \qquad\qquad\qquad\qquad\qquad\qquad \text{----- (3)} \\ I_s &= 2\,\mathrm{Re}\left(\frac{r_s r_p^*}{r_s r_s^* + r_p r_p^*}\right) = \sin 2\Psi \cos\Delta \end{aligned}$$

Those r_s and r_p coefficients represent material properties (dielectric function) unambiguously only for semi-infinite substrate with no roughness.

In all other cases modeling based on some preliminary knowledge about the sample is required in order to extract the optical constants of the materials and thickness of the layers.

The complex Fresnel coefficients for the multilayer system can be calculated, for instance, by the standard Abeles transfer matrix formalism, when each layer is characterized by a matrix **M** which depends on the type of linear polarization (-s or -p), the incidence angle, the thickness and complex refractive index of the layer and the wavelength. The Fresnel coefficients of the whole structure are consequently calculated by multiplying different **M** matrices [14].

After obtaining raw data one has a choice: to perform calculations of growth rate (thickness) and refractive indices, immediately by non-linear least-squire fitting or some numerical inversion procedure, or make control of the growth using the comparison of real-time ellipsometric trajectories with pre-computed using target thickness and refractive indices.

In the case of PECVD, the index of material can be a function of the several process parameters, such as gas composition, microwave power, process pressure, substrate temperature, etc. Fortunately, gas composition is by far the most important factor influencing material being deposited. Thus, for PECVD problem of refractive index control becoming problem of gas flows control. While ellipsometer is a sensor, providing precise information on what is happening on the substrate surface, mass flow regulation devices (typically MFCs) are the tools to close control loop. In order to interpret the ellipsometric signal and transform it into the set of commands to gas flow control system algorithms are needed that will transform classical gas distribution panel into “smart” gas panel. Consequently, future of PECVD control, in certain sense, is in developing intelligent flow control techniques.

3.1 Control using Re-computed Ellipsometric Trajectories

We define the ellipsometric trajectory as a curve given by measured ellipsometric parameters or calculated ellipsometric quantities plotted versus time, thickness or versus each other. Parameters I_S and I_C (or others, depending on the ellipsometer principle) are time-dependent functions during the deposition, and rate of the change of those parameters is related to growth rate, complex refractive index of the material being grown and initial values of I_S and I_C.

Using trajectories in I_S - I_C space one can eliminate time from the problem, as shown in Fig. 2, so prevent all errors associated with instabilities of growth rate (index stability requirement, however remains very important). Control techniques thus developed we call DISTANCE CONTROL and LENGTH CONTROL. Both of them are based on the assumption of stable and known refractive index of the growing film.

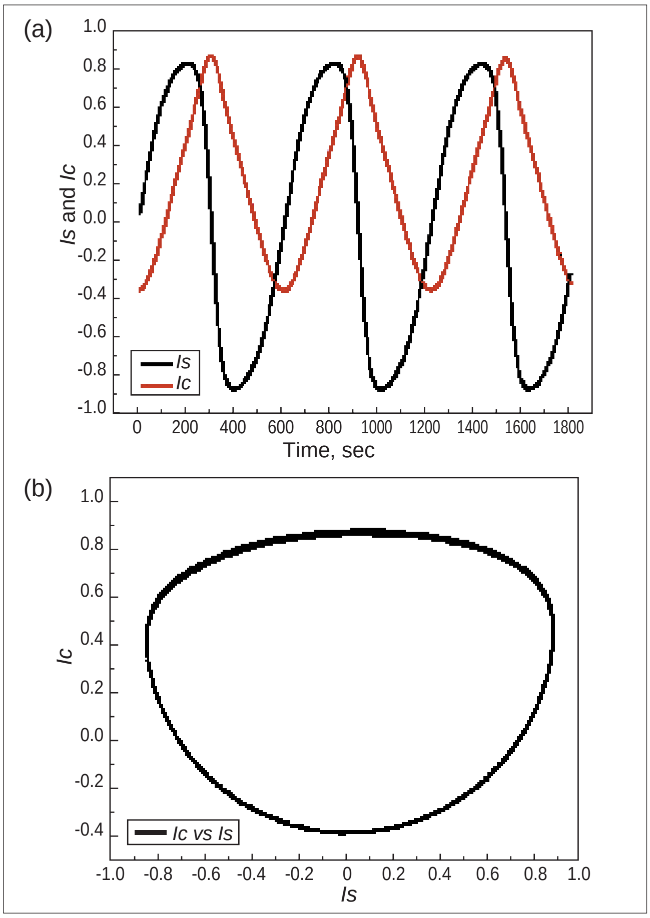

Fig. 2 Ellipsometric Trajectories
(a) I_s *and* I_c Shown Versus Time
(b) I_s *and* I_c Shown Versus One Another

3.1.1 Distance Control

For ellipsometric distance control[15], one defines a merit function in the form:

$$\chi^2 = \sum_{i=1}^{N} (I_S(t, \lambda_i) + I_{s,end}(\lambda_i))^2 + (I_C(t, \lambda_i) - I_{c,end}(\lambda_i))^2 \quad \text{----- (4)}$$

where the summation is over the various wavelength λ_i used for control (number of channels), $I_S(t, (\lambda_i))$ is the experimental value at the time t , and $I_{S,end}(\lambda_i)$ stands for the target value at the end of the layer.

During the deposition of each layer χ^2 is calculated in real time, and as soon as the minimum of this merit function for currently grown layer is reached an end of the layer is pronounced, the deposition of the next layer is starting. That is illustrated in Fig. 3. For practical reason the command to stop the current layer may not be executed for sometime after the start of the layer growth. This is done to prevent false stop if the end point is closely spaced with start point of the same layer.

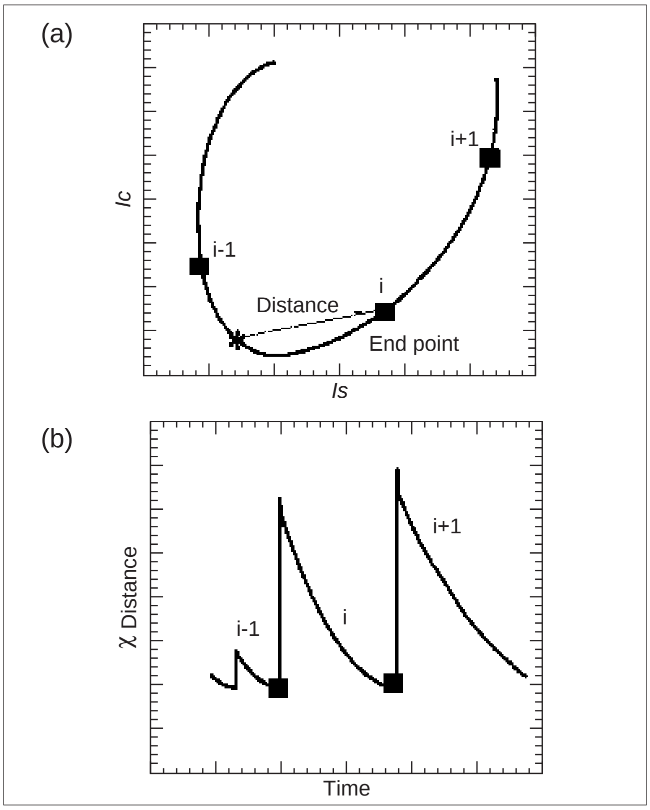

Fig. 3 Explanation of Distance Control Algorithm using I_S-I_C Trajectories
(a) *Is* - *Ic* Trajectory
(b) χ Distance vs Time

Such control technique proved to be reasonably accurate and efficient as far as stability of refractive index was preserved and the growth rate did not exceed several nanometers per minute due to the fact that stop can only be activated after passing the minimum of χ^2. That first condition for any silane-based PECVD process can only be satisfied with large excess of oxidant gases, which may demand too powerful pumping system to achieve high growth rate. The second condition may somewhat be relaxed if growth rate can be slowed down shortly before reaching the minimum. Note that the layers should remain transparent, otherwise trajectory degenerates to a point.

Since points of stop are pre-calculated in I_S - I_C space and actual stops need to be as close as possible to the predicted. Any errors in initial calculations will introduce errors in every subsequent layer and add to each other and cause detrimental changes of the filter performance. Thus, accurate characterization of the substrate is a must. However, not all the substrates can be perfectly characterized, float glass is one example. Nevertheless, this control technique was applied successfully to the growth of quarter-wave optical filters on glass and plastic [2],[7] and routinely achieved accuracy of between 1 and 2 per cent in terms of optical thickness as shown in Fig. 4. Target and real-time recorded trajectories are shown together with resulting performance of the optical filter. Discrepancy in the bandwidth is attributed to small deviation from the target value of high refractive index material (index of SiN_x is lower than expected), difference between actual and target evolution of control parameter is only due to different x-axis variables. In fact, the plots are good demonstration of insensitivity of the technique to growth rate variations.

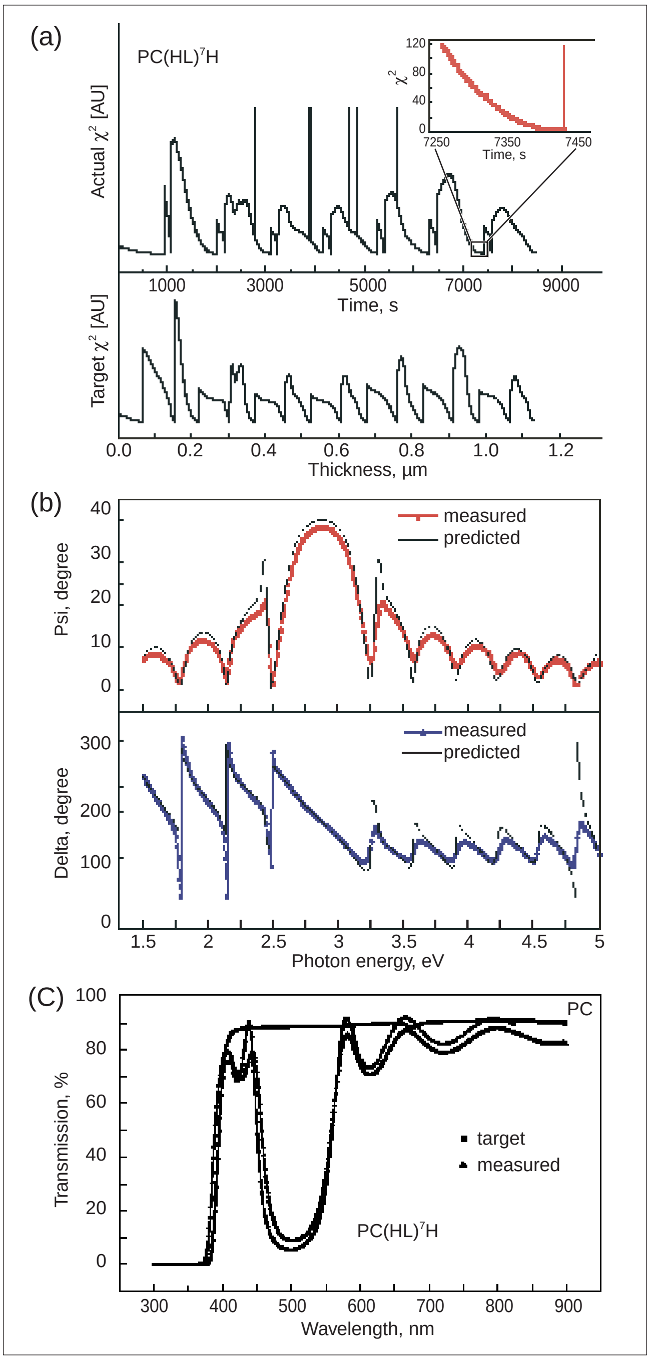

Fig. 4 Target and Recorded Evolution of Optical Filter on Polycarbonate Substrate

15 layers are grown using distance control algorithm.

(a) Control Parameter (b) Ellipsometric Spectra
(c) Transmission Spectra

3.1.2 Length Control

The use of the length of an ellipsometric trajectory for control purpose first was introduced in [15] only to activate the distance control algorithm in the neighborhood of expected stop-point to avoid the detection of false minima. The procedure used in a length control is based on the real-time comparison of the length of the experimental trajectory to the length of the pre-calculated target trajectory as shown in Fig. 5. The control parameter Λ for the length control, is defined as next equation [16].

$$\Lambda = \sum_{i=1}^{N} [L(t, \lambda_i) + L_{end}(\lambda_i)] \text{ ------ } (5)$$

where $L(t,\lambda_i)$ is length of the experimental target, and $L_{end}(t,\lambda_i)$, the length of the pre-calculated target trajectory, and summation is over all wavelength channels.

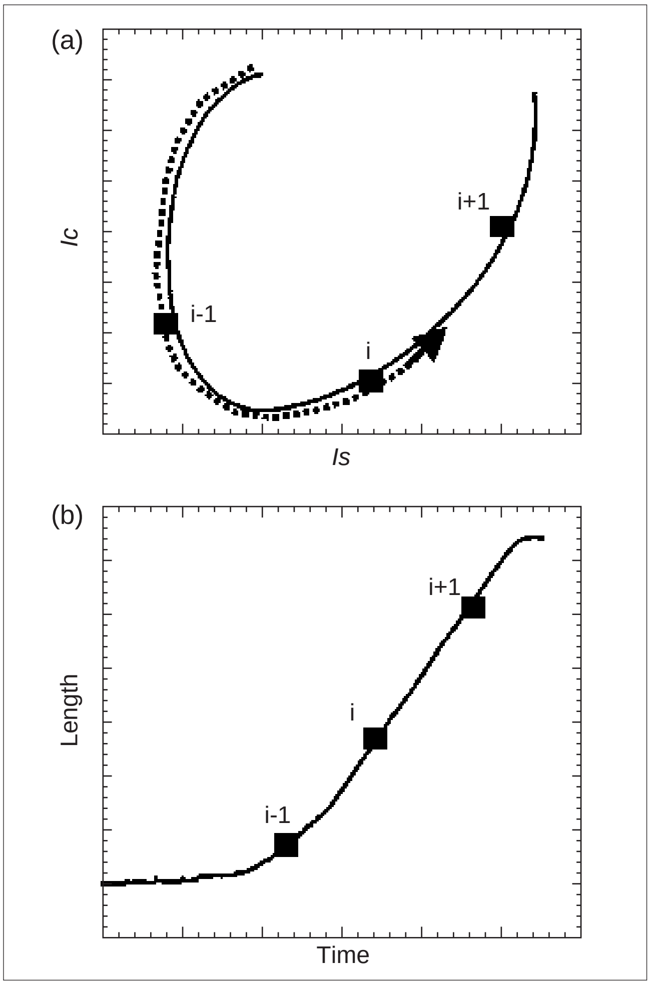

Fig. 5 Explanation of Length of Trajectory Control Algorithm using I_S - I_C Trajectories

(a) I_S - I_C Trajectory (b) Length vs Time

During deposition Λ is calculated after each acquisition step and as soon as it becomes positive, the deposition of the current layer is stopped, and the deposition of the next layer is started. It has been shown that use of length of trajectory helps to overcome problems associated with small deviation of refractive index, since it is optical thickness that mainly influences the length of the curve, and errors in the determination of substrate parameters, influence of which manifests itself primarily as shifts of the whole trajectory.

For correctly described substrate structures this control procedure is equivalent to the distance control, but the length control has the advantage of being considerably less sensitive to imperfections in the substrate modeling [16]. This is especially true in case of samples, whose precise optical structure is unknown. Sometimes it may even be found that the optical structure defined by the above methods fits well the measurements for the bare substrate but does not allow to model thin film growth on the top of the structure. A typical example for such a substrate is commercially available float glass.

However, using pseudo-dielectric function of the substrate one can still calculate trajectory with sufficient accuracy because for transparent isotropic materials, the optical response of any multilayer structure at a given wavelength can be equivalently modeled by a two-layer thin film system [14]. This was used to control the deposition of a gradient silicon oxynitride-based broadband antireflection coating (ARC). The gradient structure was split into 20 layers with a total thickness of 0.26 microns. The thickness of the individual layers varies between 1 and 25 nm, the refractive indices (measured at 632.8 nm) varies between 1.46 (SiO_2) and 1.91 (SiN_x). The filter is designed to provide an increase of 4 % (single side deposition) and 7 % (double side) of the transmission coefficient in the spectral range (300 - 800 nm).

For the purpose of process calibration 8 different depositions with oxygen to nitrogen ratios between 0 and 2.5 have been performed. The gas flows for the layers with intermediate refractive indices have been interpolated from these values. Fig. 6 shows the refractive index profile and compares the transmission measurements for deposited structures. We observe a transmission increase of about 4 % (from 91 to 95 %) between 400 and 700 nm, which proves the efficiency and the successful control of the filter.

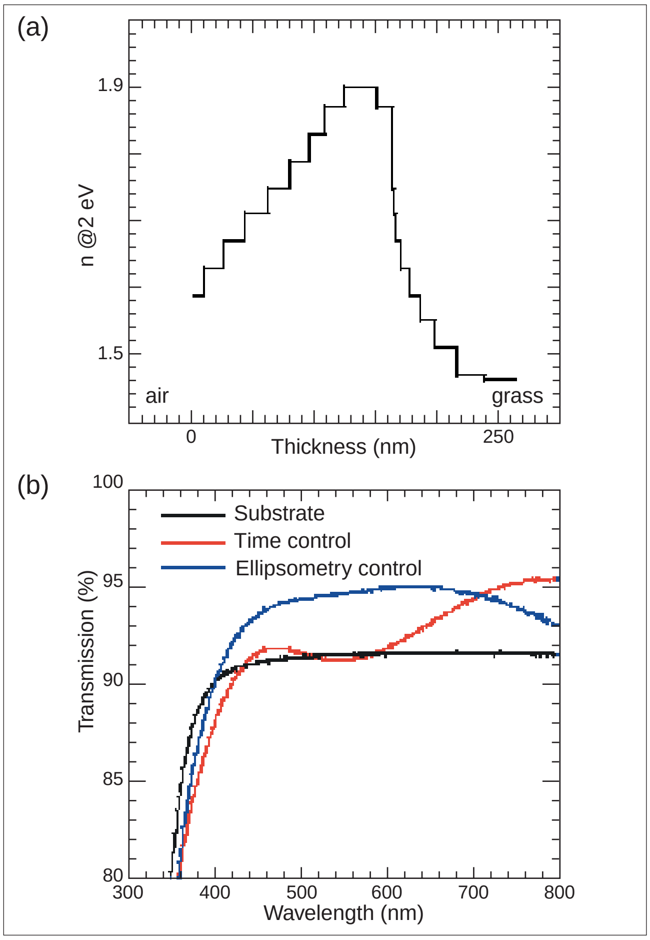

Fig. 6 Refractive Index Profile and Transmission
(a) Refractive Index Profile of 0.26 μm ARC
(b) Transmission of the Float Glass Sheet

This technique showed high precision and number of complex optical structures (from antireflection coatings to heat-reflection filters) was manufactured with its aid. However, main problem still remained – in order to effectively control the deposition, knowledge of index and deposition rate is required.

3.2 Control using Determination of the Refractive Index and Deposition Rate

Control based on pre-calculated target trajectories can be simple and efficient, but as was said above has limited practical value. The limitation of the assumption of stable refractive index does not fit well with real processes, where fluctuations of the index are inevitable, and sometimes even part of the design.

3.2.1 Numerical Inversion

The direct numerical inversion method in details is explained in two our subsequent articles [3],[4]. It is based on the expansion of the changes of the measured signal $dI_{c,s}$. as a polynomial function of the dielectric constant ε, and the thickness d_x of the newly grown layer. Since mathematical background of the technique is rather elaborate, we refer the reader to those articles and will concentrate here of results and limitations of the technique.

After calibration of the refractive index range we tested inversion algorithm by the depositing a linear gradient layer and an index-matching layer between a PC substrate and a silicon oxide scratch-resistant coating. The parameters obtained during calibration were used in the deposition of the layer of silicon oxynitride with linear index gradient on glass substrate. The gradient design consisted of a 2100 Å layer whose index was varied linearly from silicon oxide to silicon nitride. At both ends of the linear part a 500 Å thick low and high index silicon oxynitride was grown, in order to clearly define the onset of the linear gradient evolution. We observe that the total reconstructed thickness of the stack is with 3287 Å, about 6 % higher than the target value. The perfect linear shape of the index profile indicates however that the global tendency of the growth rate behavior is very well reproduced as can be seen in Fig. 7(a). Fig. 7(b) shows the complete ellipsometric spectra between 1.5 and 5 eV recorded after deposition with a step of 0.025 eV.

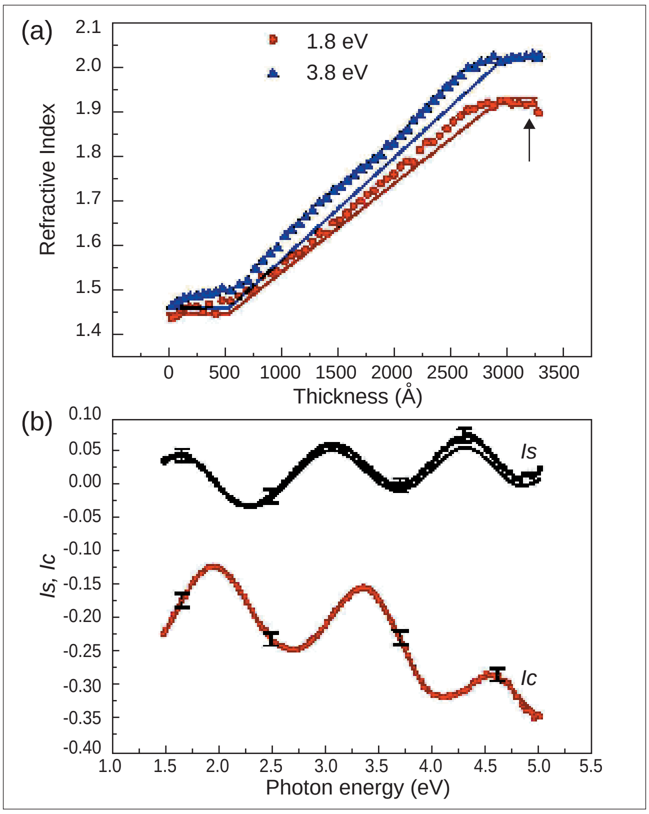

Fig. 7 Reconstruction of Refractive Index Gradient with Inversion Algorithm in Real Time
Thickness of gradient layer reconstructed in real time is 3287 Å, whereas thickness determined from spectroscopic measurements is 3253 Å
(a) Real-time Kinetic (b) Spectroscopic Modeling

The stack has been modeled by a linear gradient sandwiched between two constant index layers. The dispersion laws of these two layers, as well as the thickness of the individual layers have been chosen as the fitting parameters. With a χ^2 of 0.46 the fit can be considered as excellent, especially for the ellipsometric intensity I_c, which is in this case particularly sensitive to the slope of the refractive index profile. The total thickness (3253 Å) and the values of refractive index found by the spectroscopic fit is in very good agreement with the result of the reconstruction algorithm. The thickness of this last layer, as determined by the spectroscopic fit is however smaller than the thickness found by the reconstruction algorithm. It must however be noted that the difference between the result of the spectroscopic fit and the reconstruction profile remain within the experimental uncertainties.

Finally, the data obtained for the process calibration were used to deposit an index-matching interlayer between a polycarbonate substrate and a PECVD deposited anti-scratch silica coating. The reduced interference effects in such a coating lead to several advantages compared to a substrate, coated with a single homogeneous layer. For instance, the color effect, which occurs when the sample is tilted, is considerably reduced. Several design methods for such index-matching layers are known from the literature.

In our case we used a simple gaussian shape. The thickness of the matching layer has been fixed at 500 nm, and the continuous index profile presented in Fig. 8(a) has been split into 50 individual layers. The refractive index and the growth rate have been calculated using the calibration parameters previously derived. Fig. 8(b) compares the transmission of a polycarbonate substrate coated with a 5 μm thick scratch-resistant coating with and without an index-matching layer. The amplitude of the interference fringes drops from more than 2 percent in absolute transmission values, to less than 0.3 percent in the whole visible part of the electromagnetic spectrum. This indicates that the refractive index of the high index silicon oxynitrides matches very well the refractive index of the polycarbonate substrate, and that the gaussian shape of the index-matching layer has been very well reproduced.

Furthermore, it validates the use of our inversion method as a tool, that allows reducing significantly the time spent for process calibration, as a whole process window can be explored in one single run.

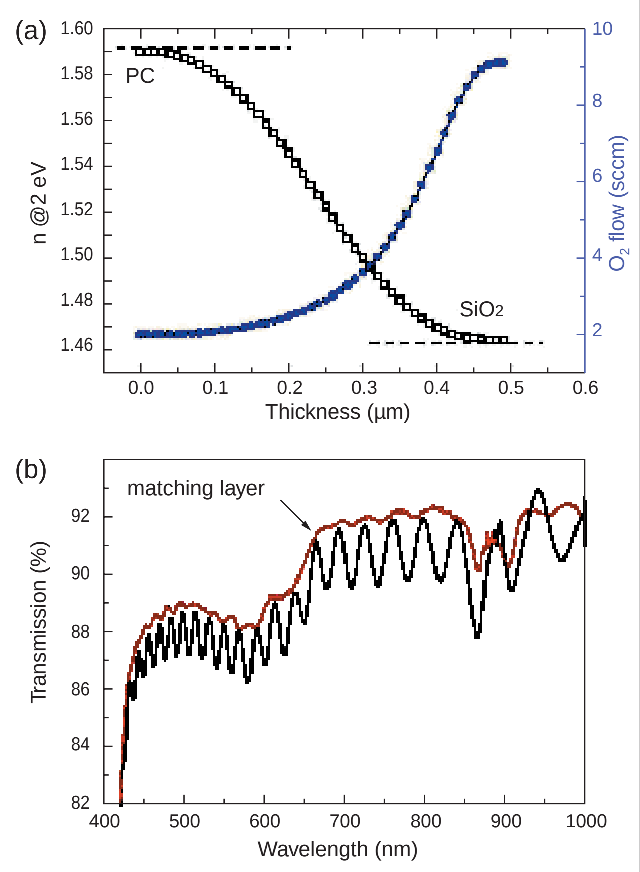

Fig. 8 Refractive Index Matching Layer for Color-effects Suppression

(a) Profile of the Gradient Refractive Index Matching Layer

(b) Comparison of Transmission Data
Polycarbonate window coated with a 5 μm thick scratch-protection SiO_2 coating with and without index matching layer

In conclusion, it can be anticipated, that due to the precision and the high speed of the algorithm (typically 150 ms for 16 wavelengths), the method will proof to be a new powerful tool for the monitoring and control of index profiles and will allow in near future the closed loop control of graded-index optical filters. One problem, however, that may affect stability of inversion algorithm is the abrupt interface. Thus it is very well adapted for graded index filters and not as much for standard multilayer coatings.

3.2.2 Real-time Least Square Fit

In order to develop technique, that will be practically useful for any arbitrary layer structure deposition, and to achieve rapid and precise real-time determination of the complex refractive index of the growing layer and its thickness, we finally focused on least-square fitting algorithm based on four following principles:

1) Raw Data Treatment

All parallel ellipsometric channels are treated independently for ***n*** and ***k*** determination without smoothing procedure and no assumption for dispersion relation is made for the fit of complex refractive index (with exception of initial guess).

2) Filtering of the Data

Points considered informative and worthy for treatment only when the change from previous point exceeds the noise level (threshold is set at double the noise level, which equals 0.01 for I_S and I_C values in typical conditions).

3) Minimal Computing Load

The thickness of the current layer is fitted with initial guess dispersion first (always same for the entire deposition) and only if the results are not satisfactory fit on ***n*** and ***k*** values is performed (assumed to be constant inside the layer – with time only thickness is changing).

4) Data Buffering

Moving window of adjustable size is used to stabilize observables.

More detailed description of the algorithm will be given elsewhere, here we will present only some results. We have tested both, rate (thickness) determination for material with known refractive index (SiO_2 in our case) and simultaneous rate and index determination for the stack of 4 subsequent layers of oxynitrides with 4 different indices. The results are presented in Fig. 9 and Fig. 10.

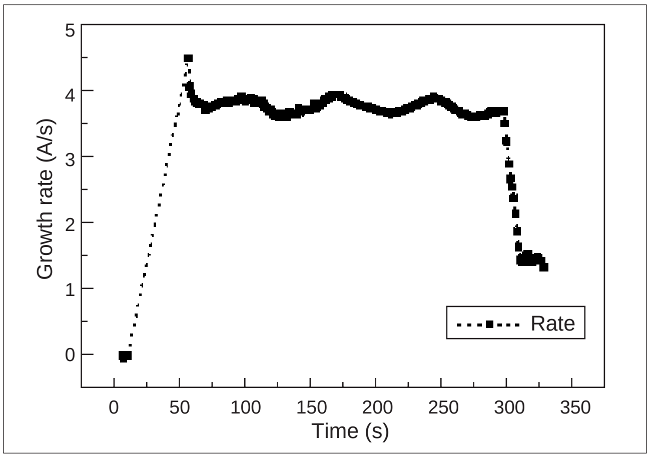

Fig. 9 Deposition of 100 nm SiO_2 on c-Si with Slowing Down the Growth Shortly before the End
Resulting accuracy in thickness is 0.1 % (100.1 nm instead of 100 nm) with full agreement between kinetic and post-deposition spectroscopic data

For the deposition of material with known index, the results are truly remarkable and show both the possibility to precisely control the thickness and handle high growth rates using gas flow control. Silane flow was automatically lowered at the end of the run in order not to overshoot the target thickness. Over 10 depositions made with slowing down the growth just before the process end the error in final thickness never exceeded 0.3 % (being usually about 0.1 %) both, for very thin (~30 nm) and very thick (~500 nm) layers.

For the deposition of the stack of 4 oxynitride layers, results are very encouraging too. The software not only very well handled the determination of the thickness and refractive index simultaneously in real time (corresponding errors fall well within 1 percent error margin if compared with spectroscopic post-deposition values), but did not suffer from significant noise that frequently hinders such index determination.

Recent experiments show that even for highly absorbing materials, like amorphous hydrogenated silicon, the technique works well and produces highly accurate multilayers a-Si:H/SiO_2 for optical stacks. Results will be reported elsewhere [17]. We consider this as a significant breakthrough on the way to fully automatic optical thin film fabrication process under ellipsometric control.

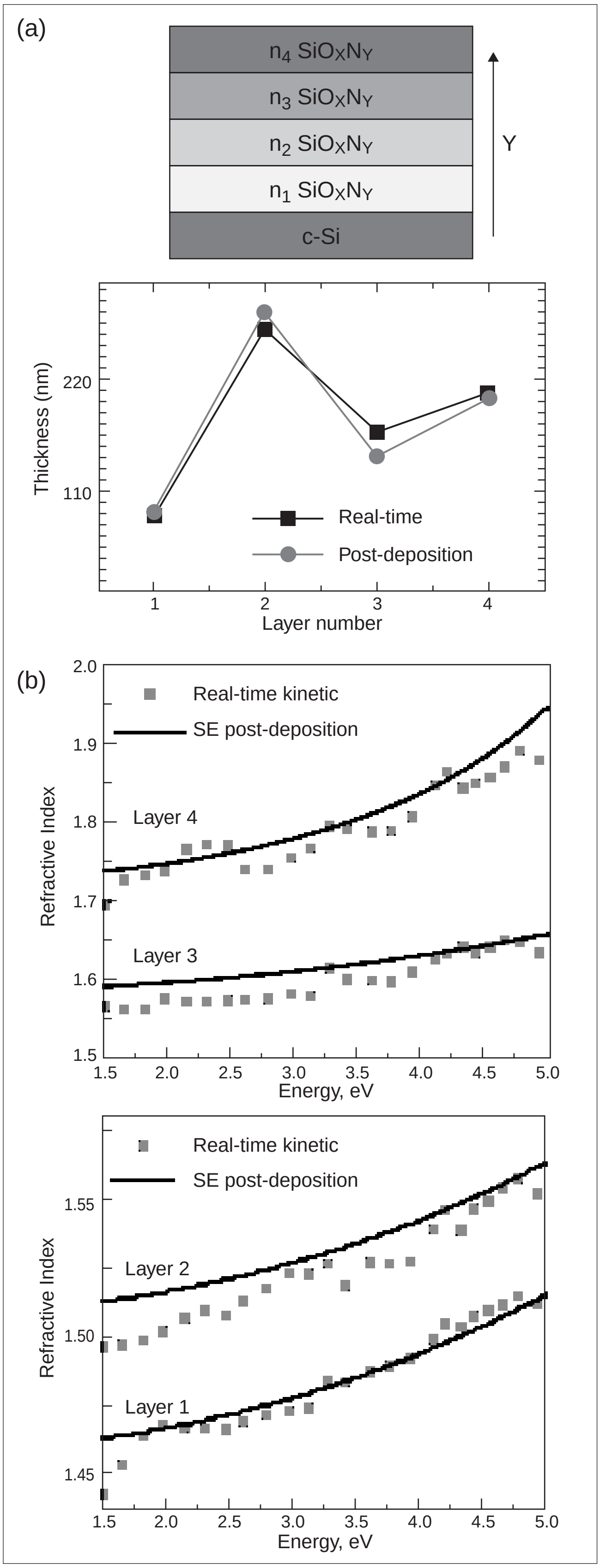

Fig. 10 Comparison of Post-deposition Spectroscopic and Real-time Kinetic Determination
Solid lines are results of the fitting of spectroscopic data with classical Lorentz oscillator dispersion function.
(a) Thickness of Each Layer
(b) Refractive Index vs Energy

4 Conclusion

In this article we reviewed our work on monitoring and control of optical filters and thin films with spectroscopic and multi-channel kinetic ellipsometry. Beside well-established thickness control techniques, which are based on the comparison of real-time ellipsometric trajectories with pre-computed ones, and do not involve index and rate determination, we have discussed two other approaches, one based on the direct numerical inversion method and the other – on real-time least square fitting of ellipsometic data.

They both provide good speed and precision necessary for accurate control of thin film growth, being best adapted to gradient and multilayer coatings, respectively. Both algorithms can be used on transparent finite (with contribution of incoherent reflections) and semi-infinite substrates for transparent and weakly absorbing films (with least-square fitting capable to cope with strongly absorbing films too).

Silicon-based films grown by PECVD were chosen for the study, although the techniques can be applied with much broader range of materials and deposition techniques. Application of a simple algorithm consisting in slowing down growth rate by reducing silane flow when the current thickness approaches the target value allowed us to improve the absolute and relative accuracy to 0.3 nm and 0.3 %, respectively.

Accuracy of index determination in all cases of the order of 1 per cent or better and precision of thickness end-point detection vary between 1 and 0.1 %, latter achieved routinely with least-square fitting algorithm for layers with known refractive index. Number of structures was grown in order to demonstrate the validity of the techniques and results are truly encouraging. Process control, based on multi-channel kinetic ellipsometry, has been implemented and tested using commercial phase-modulated ellipsometer (UVISEL from JY/HORIBA SA) allowing spectroscopic measurements, as well as real-time simultaneous data acquisition at up to 32 wavelengths.

In summary, modern multi-channel ellipsometers coupled with compact personal computers emerging not only as an efficient tools for optical materials characterization and process monitoring, but also for real-time thin film growth control.

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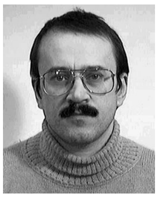

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