Copyright 2005 by the SPIE. This paper will be published in the proceedings of the OSD, Jena 2005 Vol. 5963-13.

Substantial progress in optical monitoring by intermittent measurement technique

A. Zoeller, M. Boos, R. Goetzelmann, H. Hagedorn, W. Klug
Leybold Optics, D-63755 Alzenau, Siemensstrasse 88

ABSTRACT

The production capabilities for optical multilayer coatings were improved significantly in the last decade. So called "shift free" coatings have become a standard in the coating production. Direct optical monitoring plays a key role to improve the layer thickness accuracy and takes advantage of error compensation effects. For the production of DWDM filters direct monitoring was introduced in the last decade. Continuous measurement is applied on relatively small substrate areas. (Ø < 200 mm).

The paper reports substantial progress which has been achieved for coating systems with large area substrate holders (up to Ø 1050 mm). The stationary light spot of a single wavelength optical monitor is far out of centre of the rotating substrate holder. Intermittent monitoring on a substrate or a witness is applied. This technique enables rapid prototyping with tight specifications and high yields in large area batch coaters. Application results of challenging optical multilayer systems are demonstrating clearly the potential of this powerful monitoring technique. The monitoring capability was investigated for a lot of different layer systems such as dielectric mirrors, anti-reflection coatings, sophisticated edge filters, polarizer coatings, beam splitters and multiple cavity band-pass filters. Strong coincidence of theory with experiment was achieved with PIAD and magnetron sputtering. Reproducibility experiments have clearly shown the benefits of this monitoring technique.

Keywords: Direct optical monitoring, intermittent monitoring, plasma-IAD, magnetron sputtering, optical coatings, laser coatings, band-pass filters, fluorescence microscopy

1. INTRODUCTION

An interesting overview about the history of optical monitoring we can extract from Angus Mac Leod's article "Evolution of the measurement of Thin Films" [1]. In the 18th century, Isaac Newton studied the colours of light reflected from thin films. He found the relation between the thicknesses of thin air films between a plano convex lens of accurately known radius of curvature and a flat surface. This was the well known Newton's Rings experiment which he described in his book "Opticks" published in 1704. This was two and a half century before the same colours were used for the control of thin film thickness. Single layer and multilayer coatings were controlled by examining their reflected colour[2]. The human eye was used as the detector like in Newton's experiment. From this time the development of thin film measurement cannot be separated from the development of thin-film technology.

Probably the first publication of a single wavelength optical monitor was given by Dufour 1948[3]. He described an apparatus in which the thickness of each layer was controlled by measuring with a photo-cell and galvanometer either the transmitted or reflected intensity of a beam of monochromatic light from a monitor plate. This technique is still used today for the production of a wide range of optical coatings. L. Holland described some apparatus designs in his book "Vacuum Deposition of Thin films" [4]. He cited a lot of publications up to the early 1950's. The test-glass changer was introduced. Compensation effects obtained with direct monitoring were already discussed. Important contributions were given from Polster[5] and Giacomo[6]. The pros and contras of direct monitoring on substrate and indirect monitoring with a test-glass changer were discussed in Holland's book. A lot of methods to determine turning-points for accurate end point detection were published. Optical monitoring was automized in the early 1980's[7]. Since this time the production capabilities for optical multilayer coatings were improved dramatically. So called "shift free" coatings have become a standard in the coating production. The need for accurate in-situ film thickness monitoring increased with this development.

Besides single wavelength monitoring broad-band monitoring is another approach which was investigated since the 1970's [8]. With silicon array detectors 256, 512 or 1024 wavelengths can be monitored at the same time. Today fast CCD array detectors are commercial available. In reference 9 a direct measuring broad-band monitor system in an ion beam sputter process is described [9]. In broad-band monitoring the cut-off condition is linked to a merit function which describes an error function between theory and actual measurement in a selected wavelength range. Based on the measured spectra reverse engineering and online re-optimiazion of the subsequent layers is an option and may be required for complex layer systems. Single wavelength and broad-band monitors are basically distinguished in the number of wavelengths (up to 1024 for broad-band, 1 for single wavelength) and the amplitude resolution (> 20bit for single wavelength; ≤ 16 bit for broad-band). Furthermore array detectors are available in a more limited spectral range than single detectors. And also the algorithms to determine the cut-off condition are basically different.

The principle of single wavelength monitoring is well described in Mac Leod's book "Thin-Film Optical Filters"[10]. With state of the art single monitoring systems the variation of the transmission or reflectance at a selected single wavelength is measured with high accuracy on stationary test-glasses. The layer cut-off condition is coupled to turning-points and therefore linked to the optical thickness. Variations of the refractive index are compensated which makes this monitoring technique very robust in a production environment. However the layer thickness on a stationary test-glass differs from that of the substrates which are normally located on a rotating substrate holder. This is described by the so called tooling factor which varies more or less during the coating process and from run to run.

In magnetron sputtering systems intermittent monitoring was applied in the late 1980's [11]. Direct monitoring for multi-cavity band-pass filters is successfully used in production for DWDM filters. Turning-point monitoring is applied. With this method errors made during deposition of a layer are compensated in a subsequent layer. This so called auto-compensation is very effective especially for the production of narrow band-pass filters. Simulation and experimental confirmation is described in reference [12]. For DWDM filters continuous measurement more or less close to the center of a fast rotating single substrate is applied. Due to the extreme high sensitivity on thickness errors (< 0,02 %) the useful substrate area of DWDM coating processes is limited (<0,01 m^2). Also for standard applications with less error sensitivity (0,1 % - 1%) and larger substrate areas (0,1 m^2 - >1 m^2) optical monitoring plays a key role to improve production yields. Indirect monitoring using a stationary test glass is still the most common technique. The use of direct monitoring is a need for high precision applications (thickness errors <1%) with reasonable production yield.

2. EXPERIMENTAL SET-UP

Single wavelength optical monitoring in intermittent mode was developed in a box coater with plasma-IAD (PIAD) and in a magnetron sputtering system. The features and the capabilities of the used optical monitoring system are described in reference [13].

2.1 PIAD box coater

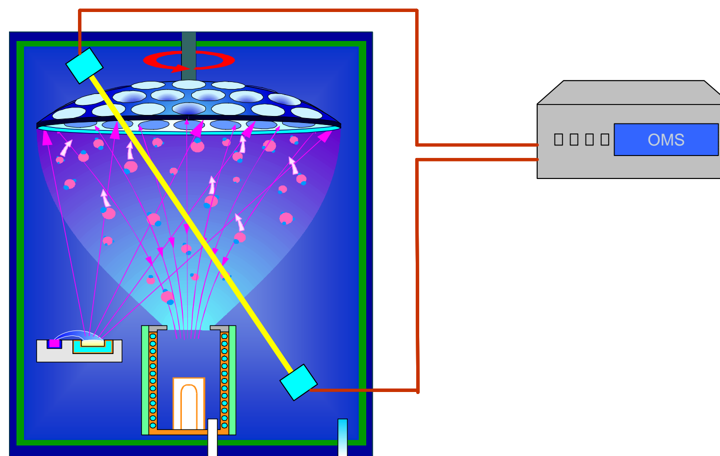


Figure 1: Schematic diagram of a PIAD process with intermittent monitoring on dome.

The experiments were done in standard Leybold Optics Syrus*pro* 1100 coating systems equipped with 2 electron beam evaporators, type HPE 10, a 1050 mm diameter dome shaped substrate holder with single rotation and the advanced plasma source (APS). Plasma ion assisted deposition (PIAD) with the APS was described in various papers. A review was given in reference [14].

Figure 1 shows the schematic of an intermittent measuring arrangement in a PIAD box coater. The standard dome shaped substrate holder rotates with approx. 30 rpm. A substrate or a witness which is located 330 mm out of center acts as the monitor glass. The control electronics of the optical monitor is synchronized with the substrate drive. At each rotation the transmittance is measured for a few milliseconds while the monitor glass is crossing two fiber collimators. Then the measured transmittance value is stored and refreshed at each rotation which takes approx. two seconds.

A quartz halogen lamp is used as light source. Either a fiber optic with collimator is used for illumination of the monitor glass or the light source is assembled directly on the chamber. The transmittance is measured under normal incidence. The transmitted light is guided into the entrance slit of a grating monochromator by use of a fiber optic. A photo detector is attached to the exit slit of the monochromator. For the majority of experiments a silicon detector was used. With this arrangement we were able to keep the noise to signal ratio below 0,02 %. During the film deposition the monochromator wavelength is kept constant. The selection of the monitor wavelength depends on the applied monitor strategy. The optical monitor is fully integrated in the automatic control of the coating system. More details about the features and the capabilities of the optical monitor were described in reference [13].

2.2 Magnetron sputtering

A Leybold Optics high precision magnetron sputtering system, type Helios was used[15]. Figure 2 shows the schematic of intermittent monitoring in a Helios sputtering system. A high speed plane substrate holder directs the substrates on the sputtering cathode. One of the substrates or a witness is used as the monitor glass. At each rotation a small thickness increment is deposited. Like the monitoring the deposition is also intermittent.

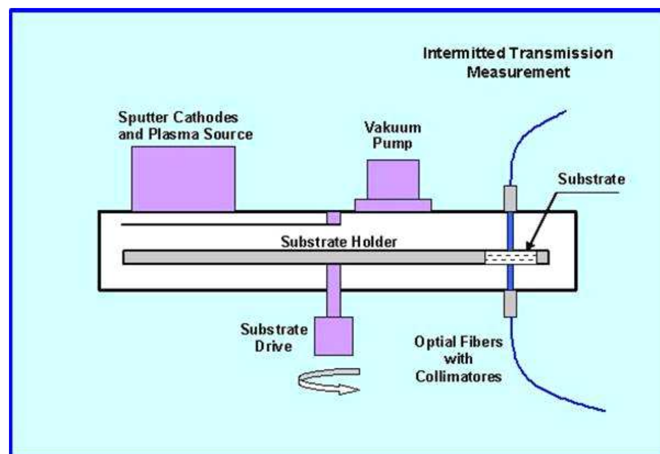


Figure 2: Schematic diagram of a sputtering system, type Helios with intermittent monitoring.

A fibre optic with collimator is used to guide the light from a quartz halogen lamp to the vacuum chamber. Another fibre with collimator transmits the light from the chamber to the optical monitor. The monitoring was performed in the same way as described in 2.1 except the rotation speed and the substrate location were different. For the sputtering experiments the substrates were located 300 mm out of centre while the rotation speed was 240 rpm. This allows only a few milliseconds to measure the substrate transmittance.

The Helios system used for the experiments was equipped with two sputtering cathodes. For the majority of experiments multilayer systems with alternating layers of Nb_2O_5 and SiO_2 were applied.

3. APPLICATION RESULTS

The above described coating systems were used to investigate the monitoring capability of a lot of different layer systems such as dielectric mirrors with a simple λ/4 design, anti-reflection coatings for the VIS, NIR and mid IR spectral range, sophisticated edge filters, polarizer coatings, beam splitters and multiple cavity band-pass filters. The monitor system was equipped with silicon and an indium gallium arsenide detector which allows to use monitor wavelengths between 400 nm and 1700 nm. As described above the noise to signal ratio of the measured transmittance was kept below 0,02% which supports a very accurate turning-point and trigger-point detection. Extensive software algorithms were used for online correction of trigger-points. The optical constants of the coating materials, the refractive index and the extinction coefficient if necessary were determined on the basis of single layer coatings. For each layer system an individual monitor strategy was developed. Depending on the monitor strategy turning-point or trigger-point cut-offs were applied. Besides the thickness monitoring at a single wavelength the optical monitor is also able to measure spectra by scanning the monochromator. This function may be useful for comparison of the optical performance between vacuum and air. For R&D purposes it may also useful to measure spectra between the depositions of individual layers.

3.1 Mirror coating

A laser mirror @1064nm with a simple λ/4 design was deposited in the SYRUS*pro* box coater. HfO_2/SiO_2 were used as the coating materials. The monitor wavelength was 965 nm for all layers. Figure 3 shows the theoretical monitoring curves. Trigger-point cut-off was used for all layers. Figure 4 shows the measured transmittance curve of the monitor glass in comparison to the theoretical layer design. The coincidence in a wide spectral range is impressive. The deviations in the range of 520 nm to 560 nm and of the mirror bandwidth are obviously caused by a small deviation of the refractive index. This result was achieved just with the first coating run. As preparation only the optical constants of the coating materials were determined on the basis of single layers. The original layer design was downloaded into the coating machine without any corrections. Since the tooling factor of the monitor glass is always one no calibration or test runs were required.

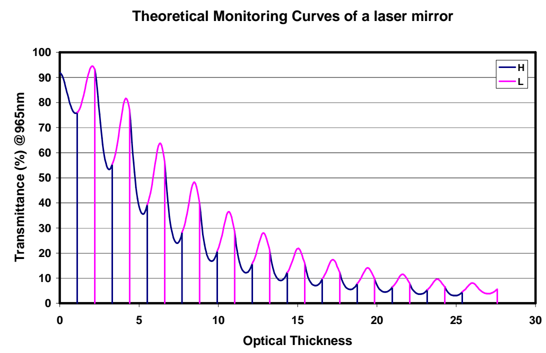


Figure 3: Theoretical monitoring curves of a laser mirror at one single wavelength.

After coating the optical performance of the monitor glass was measured by use of the spectrophotometer function of the optical monitor. In figure 5 the in-chamber spectra measurement with the optical monitor is compared with a measurement of a Varian standard spectrophotometer. The excellent coincidence of the two measurements demonstrates the high wavelength and amplitude accuracy of the used optical monitor.

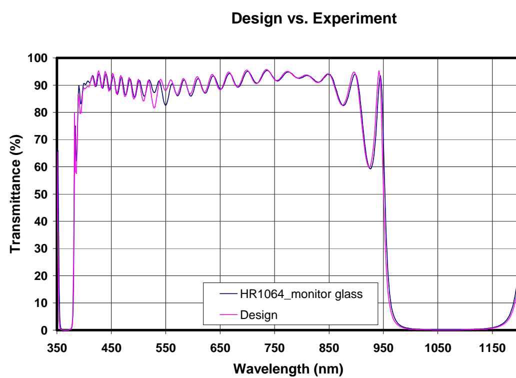


Figure 4: Measured optical performance of a laser mirror in comparison to the theory.

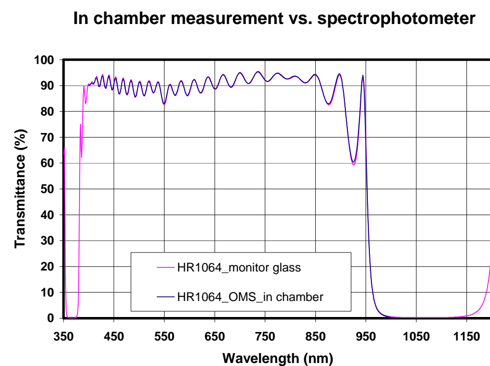


Figure 5: Comparison of the measurements: OMS in-chamber vs standard spectrophotometer Varian, type Cary 500

3.2 Anti-reflection coatings

The monitoring capability of anti-reflection coatings was investigated in the VIS, NIR and mid IR spectral range. An anti-reflection coating for two laser wavelengths was deposited in the SYRUS*pro* box coater. HfO_2/SiO_2 were used as the coating materials. Figure 6 shows the comparison of the first run with the theory. The spectra measurement was performed with a PE lambda 9 spectrophotometer. To avoid backside reflectance the rear side of the glass was frosted. The laser wavelengths were met very accurate. The residual reflectance of approx. 0,17% in the VIS and <0,1% @1064nm is probably caused by the backside which is not perfectly frosted. Figure 7 shows the theoretical monitor curve at one single wavelength.

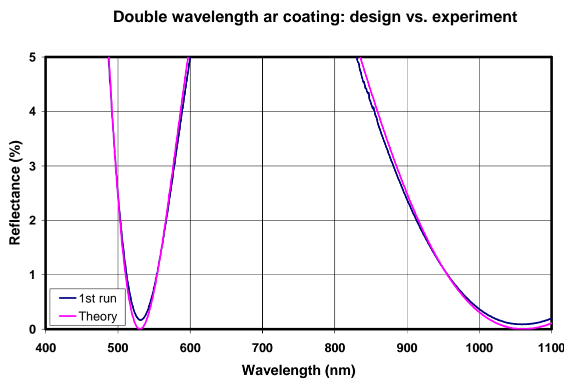


Figure 6: Design vs. first experiment of a double wavelength anti-reflection coating with HfO_2/SiO_2.

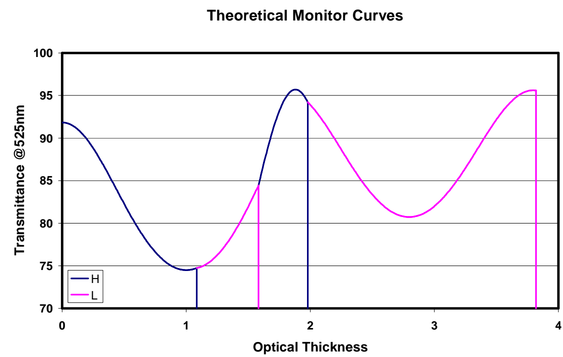


Figure 7: Theoretical monitor curve @ 525 nm of a double wavelength anti-reflection coating.

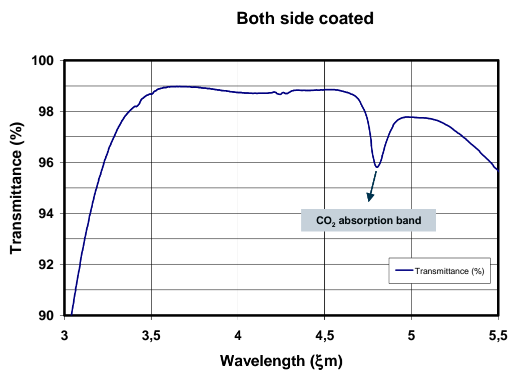


Figure 8: Transmittance of a both side anti-reflection coated germanium substrate.

An anti-reflection coating for 3,5 µm to 5 µm on germanium substrates was done in the SYRUS*pro* box coater. Silicon and a metal oxide were used as coating materials. In this case the monitoring was also direct in terms of the measurement location but indirect in terms of the monitoring wavelengths. An InGaAs detector which is sensitive up to 1700 nm was used. The selected monitoring wavelengths were between the absorption edge of silicon (1100 nm) and the upper limit of the detector (1700 nm). A standard glass substrate was used for the monitoring. Figure 8 shows the measured transmittance of a both side coated germanium substrate. Considering that the monitor wavelengths were far away from the specified wavelength range for the coating this result is remarkable.

3.3 Performance and reproducibility of UV-IR cut filters

The reproducibility and uniformity of a UV-IR cut filter with 42 alternating TiO_2/SiO_2 layers was investigated in a SYRUS*pro* 1100 box coater. Two different monitor wavelengths in the range of the pass-band of the filter were used. The deposition rates were 0,5 nm/s for the TiO_2 and 0,8 nm/s for the SiO_2. Five consecutive production runs were done. Ar each run five substrates were distributed over the Ø 1050 mm dome. Figure 9 shows the optical performance of the twenty five substrates. The excellent reproducibility and uniformity is obvious. The wavelength of the transmittance edge is within +/- 2nm which corresponds to +/- 0,3 %. This is a remarkable result in particular if we consider that the useful substrate area of the dome shaped substrate holder is approx. 1 m^2.

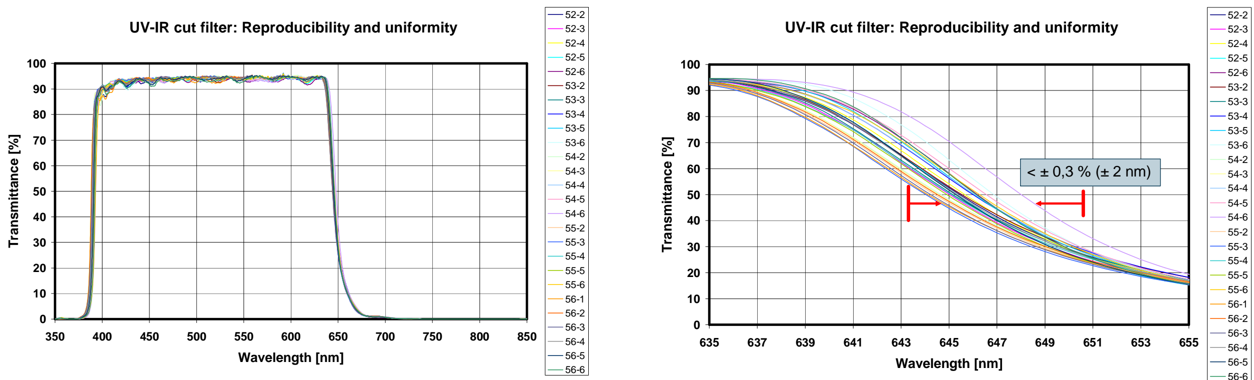


Figure 9: Reproducibility and uniformity of 5 consecutive runs of a UV-IR cut filter produced in a SYRUS*pro* 1100 box coater.

3.4 Polarizer

A polarizer coating for a 45° cube polarizer was performed with the Helios sputtering system. Nb_2O_5/SiO_2 coating materials were used. Five different monitor wavelengths in the range between 500 nm and 900 nm were used. The majority of layers were terminated by online corrected trigger-points. Figure 10 shows the comparison of the theoretical design with the measured curve of the first and second coating run under normal incidence. The coincidence with the theory and the repeatability in a very broad spectral range is amazing. This demonstrates both, the advantage of the monitoring and the high stability of the coating process.

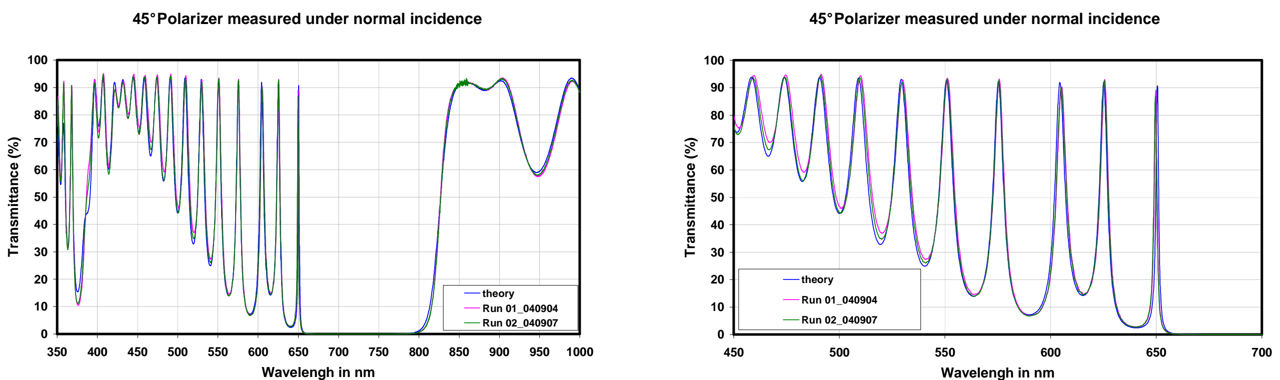


Figure 10: Theory and repeatability of a polarizer coating performed in a Helios sputtering system.

A laser polarizer coating with HfO_2/SiO_2 was performed with the SYRUS*pro* coating system. Figure 11 shows the transmittance measurement of the monitoring glass of the first run in comparison to the theoretical design. The coincidence in a broad spectral range is excellent and demonstrates the superior monitoring capabilities. As expected from the measurement under normal incidence the desired p-pol and s-pol separation under the specified AOI was achieved with the first coating run (see figure 12). The measurement was performed with a PE Lambda 9 spectrophotometer. This work was done in collaboration with SAGEM-REOSC.

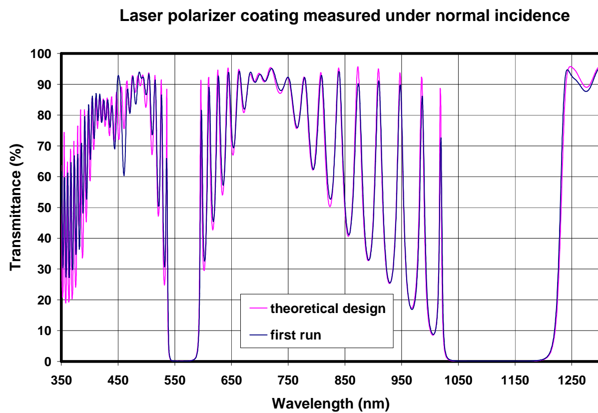


Figure 11: Theory vs. experiment of the optical performance of a laser polarizer coating with HfO_2/SiO_2.

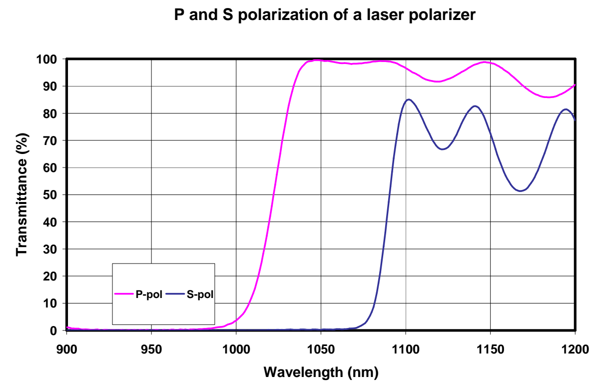


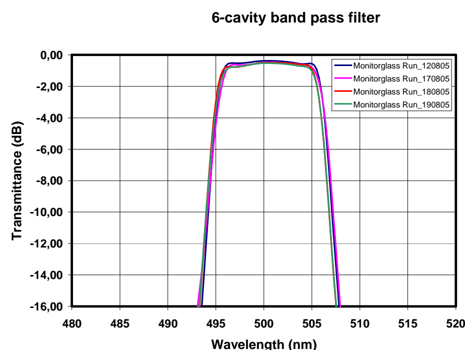
Figure 12: Polarized transmittance measurement of an HfO_2/SiO_2 laser polarizer.

3.5 Band-pass filters

With indirect monitoring a reliable production of multi cavity band-pass filters is not useful. The above described intermittent monitoring was investigated with several layer designs. Turning-point monitoring was applied except for the couple layers between the individual cavities.

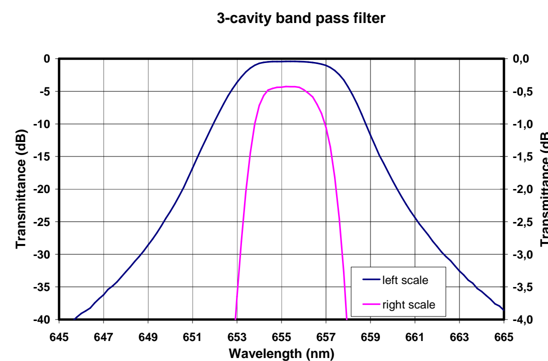
Four consecutive runs of a 6-cavity band-pass filter @500 nm with 10,5 nm bandwidth were deposited with the SYRUS*pro* box coater with TiO_2/SiO_2 coating materials. Figure 13 shows the results on the monitor glasses without backside coating. The reproducibility of the centre wavelength was +/- 0,055%. Other substrates which were located on the dome at a similar radial position as the monitor glasses showed an adequate performance too.

Figure 14 shows the performance of a 3-cavity filter @655 nm with 4,8 nm bandwidth which was also deposited in the SYRUS*pro* box coater. For this filter $Nb2O_5/SiO_2$ were used as coating materials. Based on the measured peak transmittance of 91% one can expect losses less than 5%.



3dB (50%) BW – 10,5 nm
10dB (10%) BW – 13,2 nm
CWL – 500,5 nm ± 0,28 nm (0,055 %)

Figure 13: Reproducibility of a 6-cavity band-pass filter produced with PIAD without backside coating.



0,5dB (89%) BW – 3,2 nm
3dB (50%) BW – 4,8 nm
10dB (10%) BW – 6,8 nm
Highest peak – 0,4 dB

Figure 14: Optical performance of a 3-cavity narrow band-pass filter produced with PIAD without backside coating.

Fluorescence microscopy for life sciences applications requires steep edge band-pass filters in the visible spectral range[16]. Multiple cavity filters for such applications were performed with PIAD and magnetron sputtering. Figure 15 shows the optical performance of a 7-cavity band-pass filter produced with TiO_2/SiO_2 in the SYRUS*pro* coating system. The measurement was performed with a PE lambda 9 spectrophotometer. The dynamic range of the spectrophotometer was too low to measure the blocking of -60dB over the stop band. The performance indicates that there is no significant mismatch of the individual cavities. The maximum peak transmittance of 94% indicates losses of <2%. The steepness of the edges amounts approx. 5dB/nm.

With the Helios sputtering system a 13 cavity filter with an edge steepness >5dB/nm was produced. The layer system consists of more than 80 $Nb2O_5/SiO_2$ alternating layers. Figure 16 shows the optical performance of 2 consecutive runs. The ripple over the pass-band is < 0,5dB. Considering that the deposition and the monitoring was 300mm out of centre of the substrate holder this is a very remarkable result. In production a reasonable yield would be anticipated.

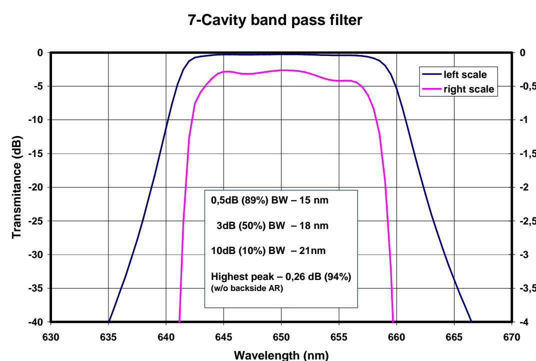


Figure 15: Optical performance of band-pass filter for life science applications produced with PIAD

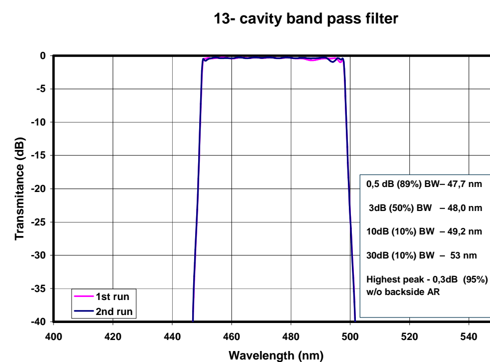


Figure 16: Optical performance of band-pass filter for life science applications produced with magnetron sputtering.

4. CONCLUSIONS

Substantial progress in optical monitoring by intermittent measurement technique was shown for PIAD and sputtering processes. The capability to monitor dielectric mirrors, anti-reflection coatings, sophisticated edge filters, polarizer coatings and multiple cavity band-pass filters was demonstrated. Especially for mirror, anti refection and polarizer coatings the coincidence of theory and first experiments is impressive. Reproducibility experiments with a UV-IR cut filter have clearly shown the benefits of this monitoring technique. The ability to monitor narrow band-pass filters was demonstrated with various filter designs. Multiple cavity filters for fluorescence microscopy applications were successfully produced in a SYRUS*pro* PIAD box coater and in a Helios sputtering system.

With intermittent direct monitoring calibration and test runs can be widely avoided. The coating machine is programmed with the original layer design without any corrections. The monitoring reproducibility is negligible in comparison to the uniformity over large area substrate holders.

5. ACKNOWLEDGEMENTS

The presented work was supported by several Leybold Optics colleagues who developed the monitor and machine control software, the layer designs, the monitoring strategies and performed the coatings and the measurements. The authors would like to thank them all. Parts of the presented work were funded by the Bundesministerium für Bildung und Forschung (BMBF), project no. 13 N 8297. Many thanks to Angus Mac Leod who contributed some important literature of the early history of optical monitoring.

REFERENCES

1. H.A. Mac Leod, “Evolution of the measurement of Thin Films”, Optics & Photonics News, Sept. 2001, 20-25
2. M. Banning, ”Practical methods of making and using multilayer filters”, J. Opt. Soc. Am., 42, 1947, 21-5
3. Ch. Dufour, Le Vide, 3 (1948), 480.
4. L. Holland, “Vacuum Deposition of Thin Films”, Chapman and Hall, London, 1952, 308-319
5. H. D. Polster, J. opt. Soc. Amer., 42 (1952), 21.
6. P. Giacomo, P. Jaquinot, J. Phys. Radium, 13 (1952), 59A.
7. R. Herrmann, A.Zoeller, “Automated control of optical layer fabrication processes”, SPIE Vol. 401,1983, 93-96
8. B. Vidal, A. Fornier, E. Pelletier, “Optical monitoring of nonquaterwave multilayer filters”, Appl. Optics, Vol. 17, 1038, 1978
9. K. Starke, T. Gross, M. Lappschies, D. Ristau, “Rapid Prototyping of Optical Thin Film Filters“,SPIE, Vol. 4094, 83, 2000
10. H. A. Mac Leod “Thin-Film Optical Filters”, IOP Publishing ISBN 0 7503 0688 2, 1986, 499-522
11. A. Zoeller et al., “Optical thickness monitoring of dielectric optical filters using a new in-situ photometer with high signal resolution and excellent long term stability”, SPIE Vol. 1019-14, 1988, 106-113
12. S. Larouche, A. Amassian, B. Baloukas, L. Martinu, “Turning-point monitoring is not simply optical thickness compensation”, OSA, Optical interference coatings 9th top. Meeting 2004, TuE8
13. A. Zoeller, M. Boos, H. Hagedorn, W. Klug, C. Schmitt, “ High Accurate In-situ Optical Thickness Monitoring for Multilayer Coatings”, SVC 47th ann. Conf. proc., 2004
14. A. Zoeller, R. Goetzelmann, W. Klug, K. Matl, “Plasma ion assisted deposition: a powerful technology for the production of optical coatings,” SPIE, Vol. 3133-23, 1997
15. M. Scherer, H. Hagedorn, , W. Lehnert , J. Pistner, “Innovative production of thin film laser components”, SPIE Vol.5963-45, 2005
16. H. Chui, “Advances in Optical Filters for Life Sciences”, Photonics Spectra, Jan. 2005, 63-66