

Article ID 1004-924X(2005)04-0403-10

# Optical broadband monitoring of thin film growth

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**Abstract:** This contribution is focused on applications of spectroscopic methods for the precise control of deposition processes. In this context, the present study gives a review on selected combinations of conventional and ion deposition techniques with different broadband online spectrophotometric systems. Besides two systems operating in the VIS- and NIR-spectral range in combination with ion processes, also a monochromator system developed for conventional deposition processes in the DUV/VUV-spectral range will be discussed. The considerations will be concluded by a comparison of the major advantages of the specific combinations of processes with online monitoring concepts and by a brief outlook concerning future challenges.

**Key words:** optical coating; optical broadband monitoring; ion assisted deposition; ion beam sputtering; DUV; VUV

## 1 Introduction

Recent advancements in laser technology, lighting, communication and other fields of modern optics call for a new generation of optical coatings with spectral characteristics of extreme complexity and high optical quality. In contrast to this, the present production processes in optical thin film technology are still dominated by optimisation cycles involving several test runs to achieve the desired performance of the coating product. Considering economical aspects and flexibility, the ideal production concept would allow for the realisation of even extremely complicated coating designs on the basis of a precise and stable process in a linear chain without iteration steps. On the way to this "rapid manufacturing" of optical thin films, the combination of appropriate deposition processes with a precise monitoring of the layer thickness is considered as a key position of major importance.

Even though the ultimate production technique could not be achieved until now, the related research work of the last 3 decades furnished enormous progresses, especially in the field of online spectroscopy in deposition processes. The first approaches towards optical broadband monitoring date back to the 1970-es, when automatic monochromators were coupled to the deposition plant for measuring the spectral characteristics of a test glass in the centre of the cathode<sup>[1-2]</sup>. Besides the pioneering work of the group at the Ecole Nationale Supérieure de Physique Marseille in France, several other research groups in Canada<sup>[3]</sup>, the United States<sup>[4]</sup>, Australia<sup>[5]</sup> and Italy<sup>[6]</sup> implemented spectrophotometric systems for optical online monitoring of the growing layer systems during the 1980-es. As a consequence of the insufficient performance of the computer systems available in these times, the measured spectra could be evaluated only on the basis of selected data, and the deposition processes had to be interrupted for an ex-

tended analysis of the growing layer system. In the next decade, direct spectral monitoring found the first applications in industrial production environments<sup>[7]</sup>, especially in conjunction with low rate processes, as for example ion beam sputtering<sup>[8]</sup>. Also the first control algorithms were developed for a switching of the layers on the basis of the measured spectrophotometric data, and the research work concentrated as well on a deeper understanding of the error budgets for a direct spectrophotometric deposition control<sup>[9]</sup>. In the course of the development of advanced spectrophotometers with sensitive CCD cameras and the rapid growth of computer power, sophisticated online spectrometers, which acquire spectra directly from the products within some milliseconds, can be integrated in the deposition system today. However, the spectrophotometric system is only one component of a precise production concept and has to be combined with the appropriate stable deposition process and furthermore, an optimised evaluation algorithm for the determination of the actual layer thickness. In this context, the present study gives a review on selected combinations of conventional and ion deposition techniques with different online spectrophotometric systems. Besides two systems operating in the VIS-and NIR-spectral range<sup>[10-11]</sup> in combination with ion processes, also a monochromator system developed for conventional deposition processes in the DUV/VUV-spectral range<sup>[12]</sup> will be discussed. The considerations will be concluded by a discussion of the major advantages of the specific combinations of processes with online monitoring concepts and by an outlook concerning future challenges in optical thin film technology.

## 2 Experimental set-ups

The data of the considered deposition processes and online spectrophotometers are compiled in Tab. 1. The combinations had been

selected in respect to special applications of laser technology in emerging fields of modern optics. For example, applications in lithography and material processing impose ever increasing demands on coatings with low losses and high stability in the UV/VUV-spectral range. Conventional thermal deposition processes of fluorides and a few oxide materials are still preferred for these coating types. Therefore, a conventional deposition plant was equipped with an online UV/VUV-spectrometer which records the spectra of the test glass in the centre of the substrate holder<sup>[12-13]</sup>. For the production and confinement of the necessary measurement radiation in the DUV/VUV-spectral range, an illumination unit containing a deuterium discharge lamp and an adjustable mirror is flanged to the bottom of the deposition plant. After passing the test glass, the measurement radiation is analysed by a monochromator system, which is evacuated through its coupling flange on top of the plant. The functioning of the system is based on a monochromator with a holographic grating in Seya-Namioka configuration which has to be tuned mechanically for recording of the spectra (see Fig. 1). As a consequence of the inertia, which can not be totally avoided for the mechanical grating drive, the acquisition speed of this single channel system is limited to approximately 5 spectra per second. For detection of the spectrally resolved radiation, a photomultiplier tube with a specially coated cathode is employed. The UV/VUV-online spectrophotometer is controlled by a computer which is also used for data acquisition as well as for the visualisation of the spectra and the derived online data. The computer program includes an algorithm for indication of the layer switching points, but no direct link exists between the plant and the monitoring system for automatic process control on the basis of the UV/VUV-online spectrometer. For an assessment of the error budget, a variety of limitations attributed to the mechanical system and

the available radiation power have to be taken into account. Typical relative errors in the transmittance can be estimated to around 1.5 % in

the spectral range between 120~240 nm covered by the online spectrometer with a spectral resolution of better than 2 nm.

**Tab. 1** Considered combinations of deposition processes and online spectrophotometers

Process	Spectral range	Type	Resolution	Error $T_r$
Thermal PVD	120~240 nm	photomultiplier, test glass	<2 nm	$\pm 1.5$ %
IAD	350~1 050 nm	CCD, synchronous, product	2~7 nm	$\pm 1$ %
IBS	500~1 000 nm	CCD, synchronous, product	<1 nm	$\pm 0.4$ %

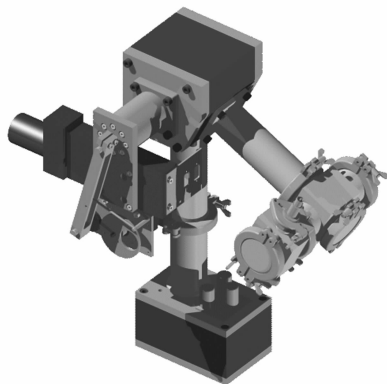


Fig. 1 Online UV/VUV-spectrometer: mechanically tuned monochromator with a holographic grating in Seya-Namioka configuration.

Advanced coatings with improved stability for laser applications in the VIS- and NIR-spectral range, including fs-pulse systems, laser medicine, or high power diode lasers, are often produced with ion assisted deposition (IAD), magnetron sputtering (MS)<sup>[14]</sup>, or ion beam sputtering (IBS). To cover a broad spectral range, one concept of the present study is based on a broadband spectrometer (BBM)<sup>[11, 15]</sup> which is combined of three discrete CCD-spectrometers for a wavelength range 350 ~ 1 050 nm. This broad band optical monitor had been tested initially in conjunction with an IAD-process implemented in a BAK 760 plant (Balzers). In further steps the system had been developed to a commercial product for a widespread application in a variety of processes and deposition plants. The major functional units comprise the measurement channel, the spectrophotometer system, and the control computer with a synchronization link to the drive unit of the sub-

strate holder. To achieve a direct measurement of products mounted at a certain radial position of the calotte, the light source is installed directly in the plant near the calotte. When the selected product passes the optical measurement channel, the light is transmitted to a fibre collimator which is mounted at a corresponding position at the top of the plant. The measurement light is then transmitted via a fibre to the multi channel monochromator system, which consists of a wavelength demultiplexer and three commercial spectrometers. Each spectrometer is assigned to a certain wavelength range (see. Fig. 2) specified by the filter arrangement in the demultiplexer system. A computer system is employed for recording of the spectral raw data and for harmonisation of the wavelength channels. For a direct monitoring of the products, the data acquisition is synchronised to the rotation of the calotte and can be gated to one or several pre-selected substrate positions. In addition, this mode of operation enables a calibration of the spectrometer during each revolution of the substrate holder, by recording an empty and an opaque position on the calotte, respectively. The corresponding lamp spectrum and the zero line are processed by the computer to calibrate the measured product spectra for each cycle of the substrate holder. In the tested arrangement, the broadband optical monitor was equipped with a data reduction algorithm, which calculates the actual deposition rate on the basis of least squares fits of the layer thickness to the measured spectrum of the actual layer. Also, the

process tracing algorithm compares the spectra of the actually deposited layer to its target spectrum, which indicates the switching point of the layer. As a result of this comparison, the time to switching of the layer is continuously determined on the basis of the deposition rate history stored during the process. If the calculated time to switching enters a predefined time window at the end of the layer deposition, the program waits for the lastly calculated time interval and executes the switching. Thus, besides visualisation of the measured process data and spectra, the program allows for an automatic control of the deposition process. The measurement accuracy of the broadband monitoring system is constrained by the resolution of the installed spectrometers and by the signal to noise ratio of the measurement channel. Typical relative errors in transmittance below 1 % can be achieved for a spectral range between 350 nm and 1 050 nm.

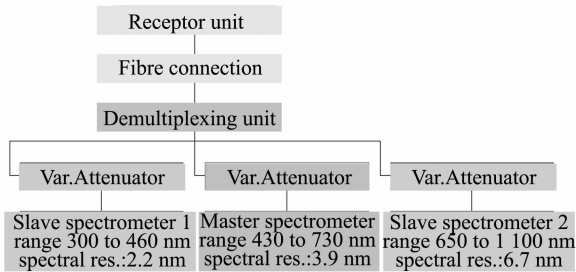


Fig. 2 Assignment of the spectrophotometers to the wavelength channels in the optical broadband monitoring system. For an adaptation of the individual signal levels, additional attenuators are installed in each wavelength channel.

The synchronisation technique described before is also employed for monitoring of an IBS-process with a high resolution CCD-camera spectrometer (HRS) operating in a typical spectral region between 500 nm and 1 000 nm. In contrast to the measurement channel of the BBM with a radiation source located directly near the substrate holder in the plant, the light source of the HRS is located outside the plant and is con-

nected by an optical fibre to the optical measuring section at the substrate holder. The compartment for the generation of the measurement radiation contains a tungsten halogen lamp as light source, second order filters, gain flatteners and a lens for coupling the radiation into the input fibre. After passing the plant wall by means of a vacuum feed through, the input fibre is coupled to the optical measuring section, which collimates the light, directs it through the substrate, and finally couples the light into the output fibre. The output fibre is connected to an imaging spectrograph equipped with a CCD-detector which covers the entire spectral region with a resolution of  $\sim 1$  nm. For a precise wavelength calibration, about 30 spectral lines of a Hg(Ar)-lamp are used. The signal-to-noise ratio is improved by averaging several rows of the CCD-array. A mechanical shutter, which is synchronised to the rotation of the substrate holder, is employed for controlling the exposure time (typical: 66 ms). On the basis of these options, the relative error of the monitoring system could be optimised to less than 0.4 % for the transmittance measurements. The process tracing algorithm contains similar routines as the broadband monitoring system and is adapted to the ion beam sputtering process with the typically long deposition times. Once started, the algorithm controls the deposition until the end of the coating system fully automatically over time periods extending over several days of continuous operation.

In Tab. 2, the technical features, the materials, and some specific parameters of the employed processes are listed. The deposition plants are either equipped with a cryo-pump or operated with diffusion pumps in conjunction with LN<sub>2</sub>-baffles. On the one hand, the IAD-process is implemented in a conventional deposition chamber of a Balzers BAK 760 system by integration of a Denton CC-105 cold cathode ion source<sup>[16]</sup>. On the other hand, a second IAD-

configuration is based on a Leybold SyrusPro 1100 plant, equipped with a Leybold APSpro advanced plasma source. A radio frequency ion source<sup>[17]</sup> with a three grating system is operated for the reactive IBS-process with metallic targets, which is optimised in respect to process stability and reproducibility of the layer disper-

sion behaviour. Besides  $\text{MgF}_2$  and  $\text{LaF}_3$  as dominant materials for the UV/VUV-spectral range, the high refracting materials  $\text{TiO}_2$ ,  $\text{Nb}_2\text{O}_5$ , and  $\text{Ta}_2\text{O}_5$  were used in conjunction with  $\text{SiO}_2$  for the production of coating systems in the VIS- and NIR-range.

**Tab. 2 Technical features and deposition materials of the employed processes.**

Process	Plant	Pumping system	Ion source	Materials
Thermal PVD	BAK 600	Cryo pump	-	$\text{MgF}_2/\text{LaF}_3$
IAD	BAK 760	Diffusion pump	Denton CC-105	$\text{TiO}_2/\text{Nb}_2\text{O}_5/\text{Ta}_2\text{O}_5/\text{SiO}_2$
IAD	SyrusPro 1100	Cryo pump	Leybold APSpro	$\text{TiO}_2/\text{Nb}_2\text{O}_5/\text{Ta}_2\text{O}_5/\text{SiO}_2$
IBS	Varian	Diffusion pump	rf-source RIM 10	$\text{TiO}_2/\text{Ta}_2\text{O}_5/\text{SiO}_2$

The process tracing software for the ion processes is embedded in a software environment which contains also different interfaces to ex situ measurement devices, reverse engineering algorithms and a design tool for optical thin films<sup>[15,18]</sup>. To model and evaluate deposition runs controlled by online spectrophotometry, the software environment is also equipped for a simulation of production cycles under control of a monitoring system with a defined error budget.

### 3 Results

The described combinations of the online monitoring systems with the deposition processes were practically tested for the production of single layers and coating systems for applications in laser technology and precision optics. In the following, the major practical aspects found for the different combination are summarised.

#### 3.1 UV/VUV-online spectrometer

The material combination  $\text{MgF}_2/\text{LaF}_3$  is often used for the production of coatings in the DUV/VUV spectral range. Both materials exhibit a low band gap wavelength below 150 nm, and the optical losses in terms of absorption and scattering can be reduced to extinction coeffi-

cients well below  $10^{-3}$ , for example at the wavelength 193 nm of the ArF-excimer laser. These materials were considered for an assessment of the single channel UV/VUV-spectrophotometer which was employed for the operator assisted deposition of high reflecting and antireflective coatings, predominantly for the excimer laser wavelengths 193 nm and 157 nm, respectively. During the deposition of the layer systems, the recorded data render possible a direct assessment of the layer quality on the basis of the band gaps in the short wavelength region and the transmittance levels in the pass bands of the coating systems. Also, a direct control of the spectral position can be continuously performed to identify deposition errors or malfunctions of the plant instantaneously. Besides these advantages for online monitoring of the growing layers, the spectrometer offers an analysis of the layer systems during the cool down and venting cycle after the production. For the cool down cycle, which starts at relatively high substrate temperatures of more than 300 °C for the fluoride materials, a small negative spectral shift, typically below 1 nm, was observed for most layer systems (see Fig. 3).

The observed shift towards shorter wavelengths can be explained by the thermal contrac-

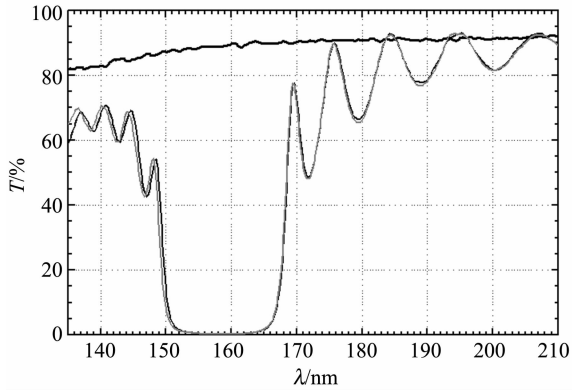


Fig. 3 In situ spectra of a high reflecting stack for the wavelength 157 nm. The solid black line in the upper part of the diagram depicts the substrate spectrum before deposition. A slight shift can be observed for the spectrum of the mirror after the cooling down cycle (grey line) in comparison to the spectrum directly after deposition (black line).

tion and a decrease of the refractive indices of the layer materials resulting in a small reduction of the optical thickness with falling temperature. In contrast to this, a positive spectral shift of a few nanometers and degradations of the transmittance can occur during the venting cycle. Depending on the quality of the coatings, this effect, which is attributed to the adsorption of water and other contaminants from the environment, persists even days after production of the coating systems. In many cases, a comparison of the recorded online spectra of the test glass to the spectra of the actual products indicates an additional deviation, which can be explained by the slightly different deposition conditions at the test glass position in the centre of the substrate holder. With the exception of the ageing processes in the layers, the described shift effects can be reasonably compensated by a wavelength offset added to the acquired spectral data. Further specific disadvantages of the implemented production process in respect to the stability and reproducibility have to be considered. As a consequence, the present combination of the UV/VUV-monitoring device with a conventional

thermal deposition process could not be qualified for a precise calculation of the deposition rate and an automatic switching between the layers. Nevertheless, the spectrophotometric systems is a versatile tool for the production of DUV/VUV-coatings which supports the operator during the production providing additional information on the band gaps and the optical quality of the deposited layers as well as a continuous control of the wavelength position. These features offer important advantages enabling a more efficient quality management and production of coatings for the DUV/VUV-spectral range.

### 3.2 Broad band optical monitor

A significant reduction of the spectral shift and ageing effects can be achieved with the IAD-processes, which enable the deposition of coatings with a compact microstructure and lower adsorption capacity. Studies of the shift behaviour with the optical broad band monitoring (BBM) system during the cool down and venting cycle as well as ex situ spectroscopy confirmed wavelengths shifts of less than  $10 \times 10^{-6}/^{\circ}\text{C}$  for the produced NIR-coatings. As a consequence, a good agreement between the spectral data recorded during deposition, the spectra acquired after the venting cycle in the plant, and the spectral characteristics measured ex-situ with a commercial spectrophotometer was observed for the oxide materials employed in the IAD-process on the basis of the Denton CC-105 ion source (see Fig. 4).

In this example, the optical broad band monitor was tuned to the wavelength range from 580 nm to 1 050 nm for the production of a broad band antireflective (BBAR) coating which is designed for low reflection in the wavelength region from 1 000 nm to 2 000 nm. Besides the good correspondence between the spectra, the desired performance of the BBAR coating system could be achieved even though the monitoring wavelength range was selected apart from the operation range. The BBAR system was pro-

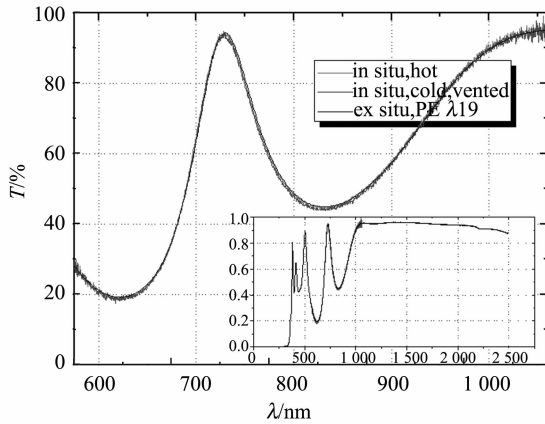


Fig. 4 Comparison of on-line spectra for a broad band antireflective coating BBAR 1000-2500 nm (8 layers of  $\text{TiO}_2/\text{SiO}_2$ , total thickness: 826 nm, Balzers BAK 760/ Denton CC-105) directly after production (in situ hot), after cool down and venting cycle (in situ cold) recorded by the broadband optical monitor between 580 nm and 1 050 nm, and ex situ measured with a commercial photometer (PE- $\lambda$ 19). The insert illustrates the spectral characteristics of the layer system for the entire spectral region of interest.

duced several times to assess the reproducibility of the process concept in conjunction with the BBM in the automatic layer switching mode. For all coating runs, the spectral positions of the points of extremum could be reproduced to a few nanometres in the monitoring region. In order to qualify the process concept further, a variety of practical coating systems was produced and evaluated. Also for complicated coating designs, a high reproducibility in the range of less than 1 % could be achieved in most cases on the basis of the automatic BBM layer switching algorithm. The strategy of monitoring coating systems designed for other wavelengths than accessible by the broad band spectrometer was established as a versatile tool for the production of coatings in the wavelength range from 1 000 nm to 3 000 nm. Limitations of the initially installed straight forward switching algorithm were observed for specific filter systems which show only small

spectral variations in the monitoring band. For a detailed evaluation of the influence of monitoring errors on the accuracy of the switching algorithm, a modelling software was developed which simulates the coating runs on the basis of pre-selected designs and error levels of the BBM. This simulation software is now routinely employed prior to the production step to identify general problems of the calculated layer designs and to indicate critical layers which have to be observed during the deposition process. In a next step towards "rapid production" of optical coatings, first approaches were made to determine the optical constants of the growing layers during deposition. By re-optimisation of the current dispersion curves in respect to the measured online spectra, the quality of the coatings can be continuously monitored, and the development of errors can be detected in an early stage. In order to demonstrate the present state of the BBM, a spectrum of a long pass filter, which had been produced in the Leybold SyrusPro 1100 deposition plant, is depicted in Fig. 5. Several runs of this coating resulted in a wavelength reproducibility of  $\pm 0.5\%$  for this filter design. As a consequence of its versatility for industrial production processes, the BBM has been improved further to a commercially available system, which can be operated in conjunction with a variety of deposition plants<sup>[19]</sup> and allows for automatic layer switching control.

### 3.3 Online monitoring in an IBS-process

In consideration of the high stability of the IBS-process and the optimised precision of the employed commercial spectrophotometric system, attempts to realise an automatic deposition concepts were made first with this concept in the framework of the present study on different on-line monitoring systems<sup>[7-8, 10]</sup>. In Fig. 6 an example of a coating produced with this deposition process is illustrated. The underlying design is based on  $\text{TiO}_2$  as high and  $\text{SiO}_2$  as low refractive index material deposited from the corresponding

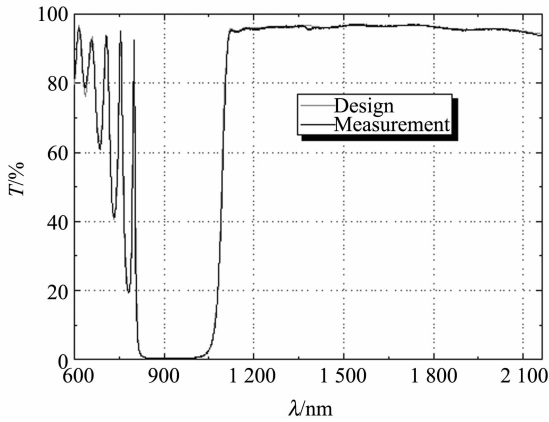


Fig. 5 Long pass filter of the material combination  $\text{TiO}_2/\text{SiO}_2$  produced on the basis of a plasma assisted process with the Advanced Plasma Source (APSpro) in a Leybold SyrusPro 1100 deposition plant. Comparison of the design curve (grey) to the spectrum (black) of the produced system measured ex situ in a commercial photometer (PE- $\lambda$ 19).

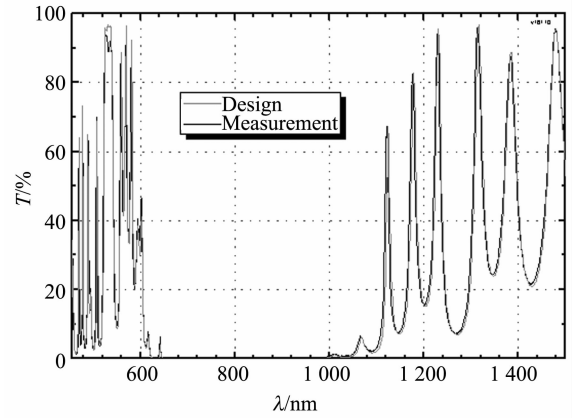


Fig. 6 Example for a layer system deposited with the described IBS-concept. Comparison of the design curve (grey) to the spectrum (black) of a double chirped mirror for 800 nm. For the depicted ex situ transmittance measurement a commercial spectrophotometer (PE- $\lambda$ 19) was employed.

metallic targets on fused-silica substrates in a reactive oxygen atmosphere. Investigations in the wavelength shift mechanisms of the produced IBS-coatings indicate a very high stability, for example ranging below  $5 \times 10^{-6}/^\circ\text{C}$  in thermal shift. In conjunction with the direct monitoring of the products on the substrate holder, an accurate and reproducible production of coating can be achieved. The example in Fig. 6 shows a spectrum for a double chirped mirror, which is employed in ultra short pulse laser systems for compensation of the group delay dispersion introduced by the numerous optical elements installed in such laser sources. The design consists of 54 layers with thickness values varying between 16 nm and 205 nm resulting in a total thickness of 6.4  $\mu\text{m}$ . The special challenge of such coatings is the realisation of the demanded spectral group delay dispersion, which is commonly not achieved if only the correct transmittance spectrum is attained. However, tests in the corresponding laser system indicated a good agreement with the demanded dispersion profile.

This result is representative for the IBS-process and was attained without any re-optimisation during the deposition run. In the present configuration, the online spectrophotometer is employed routinely for automatic ion beam sputtering of high quality coatings with complex spectral characteristics. After optimisation and simulation with the online modelling software, the designs are directly loaded into the control computer of the deposition plant, and the process is started. The stability of the IBS process allows for continuous operation of the plant over more than 70 h without intervention of the operator resulting in low process and maintenance costs. In most of the cases, the production cycle is completed without online-corrections enabling rapid prototyping of optical coatings, also for applications with a required wavelength accuracy well below 0.5 %. The further development of this process concept includes the deposition of rugate filters on the basis of targets with zones of different materials and the digitalisation of the rugate designs.

## 4 Conclusion and outlook

Three different approaches for spectrophotometric online monitoring systems were investigated in conjunction with conventional and ion processes for the reliable production of optical coatings. The described approach for the conventional deposition of UV/VUV-coatings is not suitable for a programmed production, but offers significant advantages for the quality management and efficiency in the industrial production environment. The combination of an optical broadband monitoring system with IAD-processes has recently passed the threshold to an automatic deposition. For the IBS-process, a reproducible and automatic production can be achieved with a wavelength accuracy of better than 0.5%.

Further steps towards an implementation of advanced online spectrometric control techniques will include the combination of the recorded spectra with other process parameters measured during deposition. For example, a direct link to the current rate values measured by a quartz crystal monitor would enable a higher accuracy for the deposition of very thin layers or the continuous comparison of the respective data from

the optical monitor and the quartz rate meter. The direct determination of the dispersion behaviour and the inhomogeneity of the growing layers are considered as advanced options for the process tracing algorithm. Additional improvements can be achieved by implementation of forward layer optimisation techniques which would extend the automatic layer switching to designs with critical layers and to the control of less stable deposition processes. Finally, the monitoring and automatic deposition control of rugate filter designs is a very interesting objective to expand the application area of optical thin film technology.

## 5 Acknowledgements

The authors thank the German Federal Ministry of Economics and Labour (BMWA) for the financial support of the research project "In-1on" under contract No. 16IN0088 within the framework of the "InnoNet" program. The support of the German Ministry for Science and Education (project "RAPIDOS") and the European Commission (TMR-network UV-coatings, contract-No. ERBFMRX-CT97-0101) is also acknowledged.

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