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Research Article

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Practical optimization of a coating deposition process by application of hybrid monitoring strategies in an industrial production environment

Abstract: The applicability of broadband optical monitoring to an industrial optical coating production environment is investigated. Different monitoring strategies have been applied to the deposition of beamsplitters and longpass edge filters by electron beam evaporation without any plasma or ion assistance. While the shift and reproducibility in the optical constants were poorly specified, pure optical broadband monitoring of the coatings did not prove superior to traditional quartz crystal monitoring. On the contrary, the introduction of a hybrid monitoring strategy that combines quartz crystal and broadband optical monitoring led to significant improvements in the coating performance reproducibility compared to a pure quartz crystal monitoring approach. Accompanying computational manufacturing experiments performed for the beamsplitter design reveal the same trend.

Keywords: beamsplitter; electron beam evaporation; longpass filter; optical monitoring; quartz crystal monitoring.

OCIS codes: 310.1860; 310.6860; 310.3840; 310.4165; 310.1620.

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1 Introduction

Optical monitoring of coatings has been introduced in the 1970s [1–3]. In the last years, significant improvements in optical coating specification excellence and reproducibility have been achieved by optimizing *in situ* monitoring techniques of coating growth [4]. Principally, three directions of development can be identified here [5], which have been perfected to practical relevance and resulted in significant progress in coating technology:

– The development of realistic computational manufacturing algorithms that allow identifying the theoretical design with the highest expected production yield, at the same time identifying the most suitable monitoring strategy for its deposition [6, 7].
– The perfection of broadband optical monitoring systems and adapted real-time re-engineering routines, which combine accurate optical thickness monitoring with error self-compensation mechanisms [8], but also allow the immediate identification of deposition errors if they have occurred [9, 10].
– The development of reoptimization routines that allow compensating deposition errors by reoptimizing the thicknesses of the layers not yet deposited [11].

As it has been shown in different studies, the combined application of computational manufacturing, optical broadband monitoring, and reoptimization is expected to result in superior coating performance and reproducibility, no matter which particular monitoring system has been applied [12–14]. It should, however, be mentioned that the minimization of systematic measurement errors is of highest priority, as shown by corresponding theoretical studies [15, 16].

The present study reports on improvements in the industrial manufacture of beamsplitter and longpass edge filter coatings, when the traditionally applied (indirect) quartz crystal monitoring technique is combined with broadband optical monitoring [17]. The advantages of combining single wavelength monitoring and quartz crystal monitoring have been demonstrated for several specifications [18–20].

It seems to be clear that an accurate direct optical monitoring system must result in a superior coating performance, but nevertheless, industrially relevant production conditions may differ in several regards from the laboratory conditions where the monitoring system has been initially developed and tested. Therefore, the industrial coating company Beschichtungstechnik Elsoff (Bte) and the Fraunhofer Institute for Applied Optics and Precision Engineering (IOF) have performed a joint experimental study concerning the transfer of an OptiMon broadband optical monitoring system (Fraunhofer IOF, Jena, Germany) from the laboratory to industrial production conditions. As it became clear at the very beginning of the experiment planning, the testing conditions at IOF and the intended operation conditions at Bte are indeed very different from each other (Table 1).

In view of this couple of complications, it seemed reasonable to us to test the applicability of the OptiMon system in these industrial conditions. As the first model system, we decided to perform deposition runs of a seven-layer beamsplitter design (details will be outlined in section 3) from the production line of Bte. TiO_2 and SiO_2 have been used as the high- and low-index film materials, respectively. The lack of assistance during the deposition in a Leybold A1100 gives rise to disturbing effects caused by film porosity, such as strong thermal and vacuum shifts as well as maybe refractive index gradients [21].

The study is organized as follows: The film production conditions and the coating characterization are described in section 2. In section 3, the design and assumptions as well as the results for an accompanying (simplified) computational manufacturing are presented. In section 4, the measured performance obtained from several deposition runs is reported and discussed for different monitoring strategies.

2 Experimental setup and measurement of the optical sample properties

All deposition experiments were performed in a Leybold A1100 deposition plant (Leybold Heraeus GmbH, Cologne, Germany) at Bte without any ion assistance at a substrate temperature of 300°C. In this deposition plant, the layer thickness is usually controlled by the originally delivered quartz crystal monitoring system. For this study, the in situ broadband optical monitoring system OptiMon has been implemented as a second monitoring system and integrated into the control software of the deposition system. A set of three hall sensors has been mounted close to the rotating substrate holder to generate trigger signals to initiate spectrometer measurements when the substrate holder is in the relevant position.

It has frequently been shown that broadband optical monitoring is extremely useful for high-performance coating deposition, when (environmentally) stable and reproducible optical film constants are guaranteed by the choice of a suitable deposition technique such as ion beam sputtering (IBS), magnetron sputtering (MS), ion plating, or (plasma) ion-assisted electron beam evaporation ((P)IAD) [22, 23]. Particularly, the suitability of the OptiMon system for monitoring PIAD processes using a Leybold APSpro plasma source [24] has frequently been demonstrated for PIAD processes at IOF. In the deposition plant used for the present study, the operation conditions are far from those

Subject	Lab at IOF	Production at Bte	Possible complication
Deposition temperature	100°C	300°C	Not crucial for the OptiMon system in terms of the thermal load, but stronger intrinsic thermal shift
Sample size	1 inch (2.54 cm) diameter	Larger	Different impacts of sample on measurement beam geometry
Database on optical constants reproducibility	Available	Not available	Lack in input data for realistic computational manufacturing
Deposition rate	≈0.2 nm/s	≈0.4 nm/s (TiO_2)≈1.2 nm/s (SiO_2)	Less accurate determination of termination points
Rotation speed of substrate holder	40 rpm	20 rpm	Increased impact of deposition rate variations to interpolated termination time
Assistance	With APS pro	No assistance	Thermal and vacuum shifts result in differences between *in situ* and *ex situ* optical constants
Oxygen flow	Only during deposition	Additional oxygen flush after each layer termination	Further change in film optical constants after each layer termination

Table 1 Comparison of different operation conditions in laboratory and production environment and possible resulting complications.

where the previous studies have been performed, so that a successful implementation of optical monitoring appeared to be rather challenging. Therefore, in Table 1, the main differences between the rather laboratory conditions at IOF and the mass production environment at Bte and possible resulting complications are listed.

In this study, the dependence of optical constants and layer thicknesses from environment conditions have been taken into account only by an empirically determined correction of target thicknesses to fit the spectral position of the beam splitter in the atmosphere. Thus, the difference between *in situ* and *ex situ* optical constants has not been taken into account for monitoring issues, in contrast to the treatment described in the study [25]. Instead, merely optical thicknesses have been corrected.

We refrain from a detailed description of the applied optical monitoring system OptiMon. The reader is referred to ref. [17].

The *ex situ* layer characterization has been performed by transmission/reflection measurements at nearly normal incidence as well as an incidence angle of 45° (p and s polarization) using a Perkin Elmer Lambda 950 scanning spectrophotometer (Waltham, MA, USA) equipped with a VN-measurement attachment (Fraunhofer IOF, Jena, Germany) for absolute measurements [26]. The measured spectra have been reengineered in terms of a multioscillator model [27] using the LCalc software (Steffen Wilbrandt, Jena, Germany) [26].

3 Beamsplitter: design and computational manufacturing

Beamsplitter coatings are widely used in optical systems. In Table 2, the standard design of a beamsplitter from the production line of Bte for the following specification: R:T=50:50 with ±5% tolerance in the spectral range from 420 up to 680 nm for an angle of incidence of 45°, assuming unpolarized light, is summarized. Layer one is next to the used planar borofloat substrate.

Layer	Material	Thickness (nm)
1	TiO_2	11.5
2	SiO_2	38.7
3	TiO_2	111.5
4	SiO_2	181.0
5	TiO_2	76.6
6	SiO_2	54.4
7	TiO_2	62.8

Table 2 Coating design of the beamsplitter.

In general, a large variety of alternative design solutions may be found, and for each design, a large variety of different monitoring strategies may be investigated by computational manufacturing [9]. Here, only the standard design and a small subset of different monitoring strategies have been investigated. The impact of an automated redesign (as demonstrated, for example, in the studies [12, 14]) has been excluded from this study because no reliable data of *in situ* relevant optical constants have been available, so that the reliability of online recorrection routines appeared questionable.

A realistic computational manufacturing approach would principally require knowledge on the impact of all relevant error sources in a quantitative manner [7]. In our standard treatment, each relevant parameter p is characterized by its mean value $\langle p \rangle$ and its standard deviation Δp. The careful experimental determination of all these parameters is a prerequisite for a relevant computational manufacturing run. This is a matter of fact, but on the other hand, in a production environment designed to combine mass production requirements with cost restrictions and the side condition of short delivery times, it is unrealistic to expect the corresponding database to be available. Nevertheless, the present study is focused on the use of optical monitoring in such a particular situation. Therefore, we were forced to perform computational manufacturing experiments assuming somewhat simplified conditions. Moreover, keeping the additional complications as listed in Table 1 (the *systematic* errors, which may result from the sixth and particularly the seventh row) in mind, at present, we are far from having a model available (or even implemented into a ready-to-use software tool) that allows us to reliably connect *ex situ* and *in situ* optical constants. With regard to these complications, we still may expect a more or less reliable prediction of the success of deposition experiments performed with quartz crystal monitoring, while computational manufacturing of optical monitoring will be, in our case, only a rather coarse orientation. Nevertheless, we strongly believe that the relative degree of suitability of modern tools like computational manufacturing and optical broadband monitoring to non-assisted deposition processes is of certain interest for the scientific community, so that we present these results here.

Keeping the mentioned restrictions in mind, the following input data have been assumed for the computational manufacturing runs. In terms of the models and calculation methods described in ref. [7], *stochastic* variations in the Lorentzian oscillator model parameters (only the oscillator strength in our case) have been allowed at a level that corresponds to an arbitrarily postulated standard deviation in the reproducibility of refractive indices of the coating materials for about 1.3% for both silica and titania (at a wavelength of 550 nm). These values are rather guessed than experimentally established, but seem reasonable to us. Quartz crystal monitoring has been modeled according to [7, 28] assuming a superposition of a statistic thickness offset error (with 0.5-nm standard deviation for silica, and 0.2 nm for titania) and a stochastic variation of the tooling factor (0.2% for silica and 0.2% for titania). All other parameters required for the simulations of broadband optical monitoring are assumed to be identical to the well-quantified laboratory conditions [7].

Keeping in mind the rather arbitrarily assumed *stochastic* errors, and particularly, the mentioned *systematic* model deviations, the commonly estimated production yield Y will have only a limited relevance in our calculations. Therefore, in addition to production yield estimations, we focused on the reproducibility of the reflection characteristic of the beamsplitter in terms of a spectrally averaged standard deviation of the reflectance as obtained from a certain number of (simulated or real) deposition runs. We define it according to Eq. (1):

$$\langle \Delta R(\lambda) \rangle_\lambda = \frac{1}{261}\sum_{j=0}^{260}\left(\frac{1}{M-1}\left\{\sum_{m=1}^{M} R_m^2(\lambda_j) - \frac{1}{M}\left[\sum_{m=1}^{M} R_m(\lambda_j)\right]^2\right\}\right)^{\frac{1}{2}}$$
$$\text{with } \lambda_j = (420 + j)\,\text{nm} \quad (1)$$

Thereby, the integer m indicates the number of an individual deposition run, and M gives the total number of deposition runs. This averaged standard deviation has been determined by simulations as well as from real deposition experiments with different monitoring techniques (pure quartz crystal monitoring, pure optical monitoring, and mixed (hybrid) strategies). It is rather a measure of the impact of *stochastic* deposition errors and, therefore, not so sensitive to the *systematic* deficiencies of our model.

In this study, each computational manufacturing experiment will be composed from a total of 200 virtual deposition runs. This is again a low number for a reliable calculation of the production yield (compare [7, 29]), but as already mentioned, we focus on reproducibility and use the calculated production yields only as a coarse orientation.

In Figure 1, the results of the thus performed simplified computational manufacturing runs of the beamsplitter design are summarized. The virtual deposition runs have been performed assuming quartz crystal monitoring XMS and optical broadband monitoring OM. For reasons that will become clear from the next section, two hybrid strategies have also been tested, namely, terminating the first two layers by quartz crystal monitoring and the remaining five by optical monitoring (Hyb.2+5), as well as terminating the first three layers by quartz crystal monitoring and the remaining four by optical monitoring (Hyb.3+4). These virtual deposition experiments favor, with all care, the mixed strategy Hyb.3+4; it reaches the lowest standard deviation as well as the highest formally calculated production yield.

4 Beamsplitter: results and discussion

4.1 Quartz crystal monitoring

To judge the relevance of the simulations, we start with the case of quartz crystal monitoring because the selected design is well known to be stable in Bte production practice using the conventional quartz crystal monitoring. The majority of the simulated reflectance spectra corresponding to 200 virtual deposition runs is in the specified range, but shows some variations, which seem mainly caused by uncorrelated errors in layer thicknesses. The formally calculated production yield Y is about 96.5%. We were unable to perform an identical number of real deposition

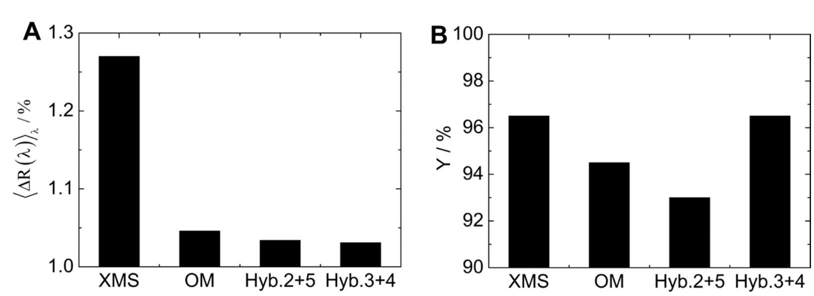

Figure 1 Results of simplified computational manufacturing runs: (A) Reproducibility of beamsplitter performance in terms of reflectance standard deviations $\langle \Delta R(\lambda) \rangle_\lambda$. (B) Estimated production yield Y of beamsplitter.

experiments, but the measured reflectance obtained from 11 corresponding deposition runs with production conditions (Figure 2) is always close to the specified limits, while a relevant fraction of deposition runs fails with respect to the defined tolerance. A calculation in terms of Eq. (1) leads to the result that $\langle \Delta R(\lambda) \rangle_\lambda = 1.27\%$ in the simulation, and as a rather happy coincidence, a value of $\langle \Delta R(\lambda) \rangle_\lambda = 1.27\%$ is also obtained in the real experiment. When eliminating the experimental curve with the worst performance (which might be regarded as a statistical outliner) from the calculation, the experimentally obtained standard deviation is still 1.08%. Anyway, simulation and experiment are in a good agreement when comparing standard deviations, which is really astonishing when keeping the arbitrarily assumed variations in optical constants in mind. Obviously, this beamsplitter design is the result of long-time selection process in manufacturing practice, where it has proven to be very stable with respect to variations in optical constants and thickness errors inherent to the quartz crystal monitoring. It is therefore a rather challenging task to find a better monitoring strategy for this design, keeping the real deposition conditions from Table 1 in mind.

4.2 *In situ* broadband optical monitoring

A second monitoring strategy of interest is the purely optical monitoring strategy. In this case, all layers are terminated using the broadband optical monitoring system. According to the simplified computational manufacturing, the application of optical monitoring is not expected to result in serious improvements. The simulated spectra are here corresponding to a standard deviation of 1.08%, while the estimated production yield is slightly decreased

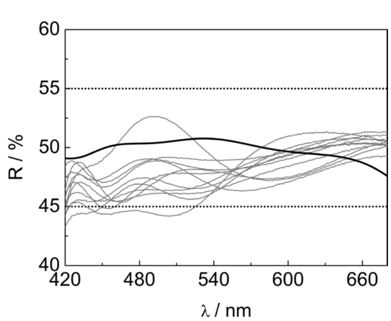

Figure 2 Measured reflectance (gray lines) for an angle of incident of about 45° and unpolarized light of the beamsplitter outlined in Table 2 obtained as a result of 11 real deposition runs using quartz crystal monitoring. The black curve corresponds to the design performance. Specification tolerances are shown by the dotted horizontal lines.

with respect to the quartz crystal monitoring, it is about 94.5%. Nevertheless, we must keep in mind that our assumption on *stochastic* refractive index errors is rather arbitrary in the given simulation experiment, and in particular, no quantitative model on the relevant *in situ* optical constants (see Table 1), which give rise to *systematic* errors, has been available. Therefore, these results have to be treated with care. Moreover, a detailed analysis of the simulation data shows a rather low impact of the layer thickness to the expected *in situ* transmittance spectra in the initial stage (the first three layers) of the deposition run. In this design, the initial layers are essential to match the refractive index of the substrate to the ambient, in full analogy to the well-known behavior in antireflection coatings [30]. As it has been shown in a previous study, this can result in an enhanced error sensitivity at the initial deposition stage and, consequently, to a complete failure of the layer termination with optical broadband monitoring in practice [14].

Unfortunately, that pessimistic conclusion could be validated in practice. We performed a total of five corresponding deposition experiments, and the broadband optical monitoring always failed already in the second or third layer, so that no successful deposition run could be recorded in practice. Therefore, it seemed reasonable to test monitoring strategies where the first two or three layers of the design are deposited using quartz crystal monitoring, while the rest of the design is monitored optically.

4.3 Hybrid optical monitoring

Based on the results of the previous sections, terminating the initial three layers by quartz crystal monitoring and all other layers by broadband optical monitoring could be the most promising monitoring strategy.

For this hybrid monitoring strategy, the simulated averaged standard deviation was obtained to be $\langle \Delta R(\lambda) \rangle_\lambda = 1.03\%$ in the simulation. It is, thus, lower than that predicted by the simulation for quartz crystal monitoring. Hence, the simplified computational manufacturing predicts an increase in reproducibility of beam splitter production when the quartz crystal monitoring strategy is replaced by the mentioned hybrid strategy.

In the corresponding deposition experiment, a lower variation in measured reflectance spectra has really been observed (Figure 3). Moreover, all deposition runs have resulted in a performance in the specified limits. The standard deviation in the experiments (10 deposition runs in this case) has been found to be $\langle \Delta R(\lambda) \rangle_\lambda = 0.80\%$ for

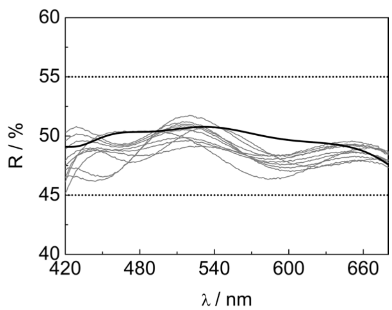

Figure 3 Measured reflectance (gray lines) for an angle of incident of about 45° and unpolarized light of the beamsplitter outlined in Table 2 obtained as a result of 10 real deposition runs using the hybrid monitoring strategy. The black curve corresponds to the design performance. Specification tolerances are shown by the dotted horizontal lines.

the mixed (hybrid) strategy. When again eliminating the worst deposition result, the standard deviation becomes 0.74%.

Let us finally emphasize, that the introduction of the hybrid monitoring ($\langle \Delta R(\lambda) \rangle_\lambda$=0.80%) has led to a decrease in the averaged standard deviation of the reflectance down to 63% of the value relevant for quartz crystal monitoring in the experiment ($\langle \Delta R(\lambda) \rangle_\lambda$=1.27%). When eliminating the worst samples from both experimental series, the decrease in standard deviation is down to 69%. So the improvement in experimental reproducibility is about one-third. On the other hand, basing on the simulation results, a decrease from $\langle \Delta R(\lambda) \rangle_\lambda$=1.27% as relevant for quartz crystal monitoring down to $\langle \Delta R(\lambda) \rangle_\lambda$=1.03% for the mixed strategy is predicted. This corresponds to a simulated decrease down to 81% when changing from quartz crystal monitoring to a hybrid monitoring strategy. Keeping in mind the low number of deposition experiments, the rather poor database available for the computational manufacturing, and the difficulties resulting from Table 1, the experimental and simulated trends (decrease in standard deviation, increase in specification adherence) are in a sufficient agreement, established here for the special case of the beamsplitter design.

5 Longpass edge filter

Encouraged by the results obtained from the beam splitter, corresponding deposition experiments have been performed with a longpass edge filter design with the edge centered at 610 nm. Table 3 summarizes the filter design and defines a hybrid monitoring strategy, which has been tested and compared to the corresponding experiments performed with pure quartz crystal monitoring.

Layer	Material	Thickness (nm)	Monitoring strategy
1	TiO_2	31.0	OM
2	SiO_2	70.9	OM
3	TiO_2	61.8	OM
4	SiO_2	75.1	OM
5	TiO_2	54.4	OM
6	SiO_2	88.1	XMS
7	TiO_2	54.4	OM
8	SiO_2	88.1	XMS
9	TiO_2	54.4	OM
10	SiO_2	88.1	XMS
11	TiO_2	54.4	OM
12	SiO_2	88.1	XMS
13	TiO_2	54.4	OM
14	SiO_2	88.1	XMS
15	TiO_2	59.0	OM
16	SiO_2	88.1	XMS
17	TiO_2	45.9	OM
18	SiO_2	99.1	XMS
19	TiO_2	44.5	OM
20	SiO_2	171.1	OM

Table 3 Construction parameters and definition of a hybrid monitoring strategy for the longpass edge filter.

The results of 10 deposition runs per monitoring strategy are presented in Figures 4 and 5. The gray lines correspond to experimental curves, while the black line shows the theoretical performance according to the data from Table 3. From the first glance, it is clear that again, the hybrid monitoring strategy is superior to the quartz crystal monitoring strategy. The deposition success has here been quantified in terms of the standard deviations of the wavelength where the transmittance is 50%. Clearly, the hybrid strategy results in a lower standard deviation than the quartz crystal monitoring approach.

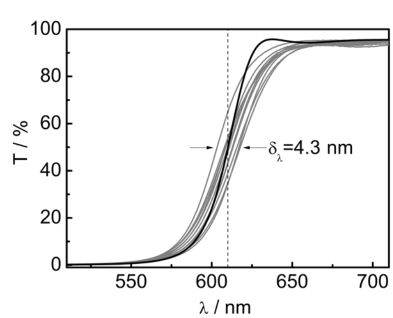

Figure 4 Measured transmittance (gray lines) of the longpass filter outlined in Table 3 obtained as a result of 10 real deposition runs using quartz crystal monitoring. Design performance given in black.

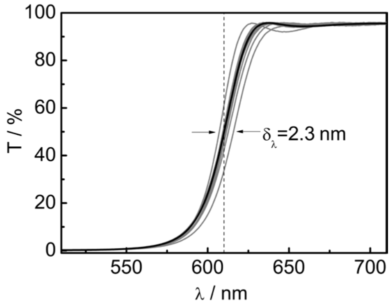

Figure 5 Measured transmittance (gray lines) of the longpass filter outlined in Table 3 obtained as a result of 10 real deposition runs using hybrid monitoring. Design performance given in black.

6 Conclusion and outlook

In this short study, we have demonstrated the applicability of the optical broadband monitoring system OptiMon in an industrial mass production environment. Thereby, we have used a 50:50 beamsplitter design for the VIS from the production line of Bte as a model system, as well as a 610-nm longpass edge filter.

It turned out that the application of optical broadband monitoring is beneficial even in a standard deposition process without any plasma assistance. Under these conditions, every layer will change its optical properties, and so the spectral performance of a stack will significantly change during venting, cooling down to room temperature, and long-time exposition to the atmosphere. Nevertheless, a combination of quartz crystal and optical monitoring was clearly superior to pure quartz crystal monitoring with respect to the reproducibility of the optical performance. The observed enhancement of reproducibility will decrease the number of waste charges and finally result in a higher sustainability caused by fewer cost and energy exhaust. In particular, the rather coarse correction procedure with respect to vacuum and thermal shift of the optical constants did not impede the stability of reengineering and thickness monitoring in a crucial manner, except the rather critical first three layers in the design.

As a side result, information has been generated about the relative use of computational manufacturing calculations in a situation where reliable data about the reproducibility in optical constants are not available. Under these circumstances, the quantitative results concerning reproducibility (and consequently also the production yield) are definitely not as reliable with respect to *absolute* values as in the case of reliably quantified error levels, but nevertheless, trends can be identified. As the main result of this study, an improved monitoring strategy for beamsplitter and longpass edge filter deposition combining features of quartz crystal as well as optical monitoring could be identified even in situations where the refractive indices are not particularly stable. This offers application fields for optical broadband monitoring operation in situations where coatings are prepared without assistance.

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