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# Origin and compensation of deposition errors in a broadband antireflection coating prepared using quartz crystal monitoring

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## ABSTRACT

To improve the performance of TiO<sub>2</sub>/SiO<sub>2</sub> broadband antireflection (BBAR) coating prepared using ion assisted deposition, a hybrid approach was proposed to probe, deduce, and compensate for the thickness errors resulting from quartz crystal monitoring. Using transmission electron microscopy (TEM), a thickness-dependent relative error was found in the prepared BBAR coating. Thinner layer had larger relative error and this was the main reason for the degradation of the performance of the BBAR coating. By fitting the thickness errors via a linear model, the origin of the thickness-dependent relative error is determined to be a constant thickness offset of the quartz crystal monitor. The thickness offsets are 1.5 nm and 2.7 nm for TiO<sub>2</sub> and SiO<sub>2</sub>, respectively. Finally, an approach through reverse engineering of a 4-layer coating was proposed to obtain the offsets for both materials. The results achieved are comparable to that obtained using TEM, but this approach is much more convenient. After compensating these offsets, a BBAR coating was prepared with transmittance higher than 99% from 450 to 900 nm.

## 1. Introduction

The rapid development of modern optical technologies imposes ever increasing demands on the performance of optical coatings. Optical coatings with more complex and broader spectral characteristics are the current focus of researches [1]. Especially, intensive studies including design, fabrication and characterization have been devoted to improving the performance of broadband antireflection (BBAR) coatings that the key components in optical systems [2]. They have refined the understanding of the behavior of BBAR coatings [3–5] and provided further guidance for achieving the optimal design [6,7] and fabrication strategy [8,9]. To fulfill the desired spectral requirements of BBAR coating, more layers with thickness varying from very thin to thick are necessary [10,11]. Their complex layer stack makes the precise fabrication of BBAR coating quite challenging. Deposition errors in thickness resulting from monitoring technology can cause a significant difference between design and fabrication results. The correction of deposition errors is essential to achieving the desired spectral performance of BBAR coating. Deposition errors can be classified into systematic errors and random errors, and both of them could have significant influence on the prepared spectrum. The knowledge of the origin of deposition

errors is very important, which is a precondition to find a simple and feasible approach to correct the deposition errors.

Typical methods for monitoring BBAR coatings include time monitoring, broadband monitoring and quartz crystal monitoring [12]. Time monitoring is adaptable to the sputtering process whose deposition rate is stable, and the systematic errors are the main origin of errors. If the systematic errors can be corrected, it is possible to obtain a satisfactory production result of BBAR coating by time monitoring. Broadband monitoring can not only be applied in the sputtering process, but also in other processes having unstable deposition rate, such as electron-beam evaporation (EBE) and ion-beam assisted deposition (IAD) [13–15]. For the broadband monitoring, the real-time reverse engineering with online re-optimization can efficiently detect the occurred errors during deposition [16,17] and then optimize the remaining layers to compensate the identified errors [18,19]. Therefore, most research to date adopted this approach to prepare the BBAR coating [20–22]. Although both systematic errors and random errors can be compensated to ensure the desired spectrum by this way, this approach is rather complicated and the origins of errors are sometimes ignored. Quartz crystal monitoring is the most predominant monitoring method for preparing BBAR coatings in practice because of its ease of

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installation and operation with ready automation [23,24]. But its accuracy is usually not good enough to prepare high performance BBAR coating [12,25]. Some previous studies used a hybrid approach combining quartz crystal monitor and broadband monitor, where broadband monitoring was used to calibrate the quartz crystal monitor during the deposition process to achieve better spectral performance [25–27]. However, it is quite desirable to investigate how to prepare high performance BBAR coating only using quartz crystal monitoring for EBE or IAD process.

Quartz crystal monitoring could result in considerably large systematic errors and (or) random errors. But it has not been demonstrated through experimental results which type of error is the main reason for the degraded performance of BBAR coating prepared by advanced EBE or IAD process. Due to a lack of knowledge about the origins of errors in quartz crystal monitoring, it is quite challenging to find convenient approach to correct the deposition errors. Although it is possible to apply the reverse engineering to the off-line measured spectrum and to obtain and compensate the errors, the multi-solution problem may greatly affect the reliability of reverse engineering of complex BBAR coatings and it is hard to confirm the origin of monitoring errors [28]. So, it is practically desirable to know the origin and influence of errors in quartz crystal monitoring and then to find an efficient method to correct them. In this paper, we try to explore the following issues: 1) Is the systematic error or random error of quartz crystal monitoring the main reason for the degraded spectral performance of BBAR coating prepared by IAD? and 2) Is it possible to find an efficient and convenient approach to compensate for the deposition errors to improve the spectral performance of BBAR coating?

In this work, a systematic study was designed to correct the errors of the quartz crystal monitoring to prepare BBAR coating. The design of BBAR coating is described in Section 2. Section 3 presents how to probe, deduce and compensate the errors. A summary of our findings appears in Section 4.

## 2. Experiments

### 2.1. Design of the BBAR coating

In this work, a BBAR coating working in the spectral region of 450–900 nm under normal incidence was designed on a quartz substrate using OptiLayer [29].  $\text{TiO}_2$  and  $\text{SiO}_2$  were used as the high and low refractive index materials because of their highly desirable properties in the visible and near-infrared ranges, such as big contrast in refractive indices, as well as low absorption and good mechanical properties [30–34]. The refractive indices of two materials were obtained through the characterization of single-layer coatings and are given in Fig. 1. The characterization was based on the transmittance spectra from 300 to 1200 nm, which were measured at normal incidence by a Cary5000 spectrophotometer from the company of Varian.

Fig. 2 presents the coating structure and spectral response of the BBAR design. It can be seen in Fig. 2(c) that the theoretical transmittance is higher than 99.7% from 450 to 900 nm (no backside reflections included). Fig. 2(a) shows the layer thickness profile of the design. The minimum and maximum layer thicknesses in the design are 5 nm and

130 nm, respectively. The layer structure of the design can be interpreted as two approximately similar arrangements of refractive index profile called clusters, as shown in Fig. 2(b). Cluster is the basic constitution of BBAR coating; in theory, the more clusters in design, the better spectral performance it can achieve [10,11]. However, increasing the number of cluster to three can bring only a small improvement on the final transmittance, but greatly increase the difficulty of coating fabrication. Therefore, a 14-layer designed BBAR coating with two clusters is selected as the final design in our study.

### 2.2. Preparation of the BBAR coating

The BBAR coating was prepared using an EBE plant OTFC1300 from the company of Oporun. During the deposition, an RF-type ion source is applied to densify the deposition layers with a working condition of 900 V and 1000 mA. The quartz crystal monitoring is utilized to control the thickness of deposited layers and the deposition rate, which are 0.2 nm/s and 0.5 nm/s for  $\text{TiO}_2$  and  $\text{SiO}_2$ , respectively. Since the quartz crystal monitoring is an indirect monitoring technique, the difference between the film thickness on the quartz crystal and the sample, which can be described by the tooling factor, should be considered. In our work, the tooling factor is also obtained from the thickness determination in the characterization of single-layer coatings for each material described in Section 2.1. They are calculated as the ratio of monitored crystal thickness to the deposited layer thickness on sample and equal 1.247 and 1.408 for  $\text{TiO}_2$  and  $\text{SiO}_2$ , respectively.  $\text{Ti}_3\text{O}_5$  is used as the starting material and is evaporated in a reactive atmosphere of oxygen for the correct stoichiometric composition. The deposition temperature was about 393 K and the chamber was pumped down to a base pressure of  $2.3 \times 10^{-4}$  Pa. To guarantee the reproducibility of deposition process, the chamber and samples were heated for 3 h.

## 3. Results and discussion

The transmittance spectrum of a double-side coated BBAR coating sample was measured at normal incidence by a Cary5000 spectrophotometer, as shown in Fig. 3(a). A large discrepancy can evidently be seen between experimental and theoretical transmittance curves, which reflects the presence of considerable deposition errors in layer thickness. To exclude the influence of the reproducibility of the deposition process on the double-side coated BBAR coating, we also compared the spectral curves of single-side coated BBAR coatings in two deposition runs in Fig. 3(b). Their spectral curves are quite similar and the small differences can be interpreted with the random errors in both the deposition and measurement. This confirms that the reproducibility of the deposition process in our experiment can be guaranteed from run to run.

Applying reverse engineering to the measured spectrum of BBAR coating resulted in multi-solutions, it is difficult to tell which is correct and to determine the origin of the errors. To understand the origin of errors resulting from quartz crystal monitoring, high-resolution transmission electron microscopy (TEM) was used in this work to obtain the deposition errors in a reliable way. Cross-sectional specimen of the BBAR coating for TEM analysis was prepared by a FEI Helios precision focused ion beam system with the standard procedures of mechanical polishing, dimpling and ion beam thinning at the milling angle of  $4^\circ$  and the ion energy of 5 keV. And the specimen was mounted in a double-tilt holder and investigated in a Tecnai G2 F20 S-TWIN TEM device from the company of FEI. The accelerating voltage of TEM is 200 kV and the image magnifications are in the range  $4 \times 10^5$ – $10^6$ . The images were taken using a CCD camera with  $1024 \times 1024$  pixels. Fig. 4(a) shows the cross-sectional image of the BBAR coating sample obtained using TEM. The light region at the bottom of the image represents the substrate, and the upper areas are for the thin film, where the dark layer is  $\text{TiO}_2$  and the light layer is  $\text{SiO}_2$ . After making a gray-scale processing on the image by an offline analysis algorithm, the gray value curve versus film

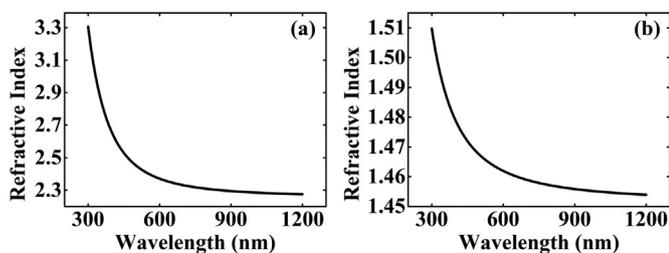


Fig. 1. The refractive index of the (a)  $\text{TiO}_2$  and (b)  $\text{SiO}_2$  coatings.

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