Plasma ion assisted deposition with radio frequency powered plasma sources

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ABSTRACT

The deposition of oxide coatings with excellent optical and mechanical properties requires powerful plasma or ion sources. We investigated the layer performance of oxide coatings using a large aperture radio frequency powered plasma source for plasma ion assisted application and related the achieved coating performance to beam parameter of the source. The coatings exhibit low compressive stress values, high refractive indices and low absorption values. Using the new type of source in combination with direct optical monitoring on the dome for the production of challenging interference filters shows a huge potential in terms of stability, running costs and easy maintenance. Examples of application in the visible spectral region will be given.

Keywords: SiO_2, TiO_2, PIAD, thin films

1. INTRODUCTION

For the production of interference filters in the visible spectral region SiO_2 and TiO_2 as coating materials are widely used. These materials are inexpensive, delivering a high refractive index contrast and high resistive coatings with excellent optical properties when deposited with the appropriate technology. Box coaters with e-beam evaporation are the commonly used deposition technique for producing interference filters using these materials. Combining the thermal evaporation with plasma or an ion assisted deposition process allows the production of dense-shift-free coatings. In combination with direct optical monitoring challenging interference filters can be produced. However, to deposit these materials with high rates and to achieve the combination of excellent optical and mechanical properties is still a challenge. Dense-shift-free optical coatings can be produced with high bombardment or by sputtering methods. But these coating often show high amounts of compressive stress and/or having higher absorption values than acceptable. We therefore investigated the potential of a large radio frequency plasma source in the production of interference filters for the visible spectral region in respect to achievable optical and mechanical properties.

2. EXPERIMENTAL

Figure 1 shows the large radio frequency source installed in the chamber door the SYRUSpro 1510. Details of the working principle and the chamber set up are described in [1, 2]. The distance between source and dome center was 1300mm. For the evaporation of the material we used two large HPE 12 electron beam evaporators. The system was equipped with a direct optical monitoring system OMS 5000 to control the film thickness of the evaporated layers and layer stacks on the dome. Details of the monitoring set up are described in reference [3, 4]. Spectral measurements were performed in vacuum immediately after deposition. By comparison with subsequent ex-situ measurements we were able to verify dense-shift-free coatings. As substrates we used B270 float glass and 3” silicon wafer for evaluating the intrinsic stress values and the surface roughness of the layers. Transmittance and reflection were characterized by spectro-photometrical measurements with a PE λ 900. The integral haze of the coatings measured with BYK-Gardner haze meter plus. Stress values were evaluated by means of the bending of the silicon wafer prior and past the deposition of the layers.
For characterization of the ion energy distribution and the achievable ion current density we used a faraday cup measurement tool with retarding grids. For measuring of beam profiles it could be mounted at any high above the source and translated horizontal via a linear feed trough while the source was working. For measuring of the ion current densities and ion energy distribution on the dome, the cup could be mounted in any substrate position.

3. RESULTS

3.1 Source characterization

The radio frequency plasma source can be operated with inert gases like Argon, reactive gases like oxygen or nitrogen or mixtures of these. The permanent magnetic field created by to two induction coils allows the use of the electron wave resonance principle [5]. Thereby large amounts of radio frequency power can be coupled into the plasma. We were able to use powers up to 5kW, which was the limit of the available power supply. The power can be either coupled inductively via an induction coil or capacitivly through a coupling capacitor into the discharge region. The matching network allows switching continuously between both excitation modes. In this way we were able to control the ion energy in a wide range.

Figure 2 shows the ion energy distribution as obtained from the faraday cup measurements by varying the retarding grid voltage. The source was operated with pure oxygen and the measurement was done at distance of 1200mm above the source. Three energy regions can be distinguished: a narrow low energy peak at approximately 80eV, a relatively broad peak at approximately 600eV and a weak peak at approximately half the energy of the high energy value. We characterize the curves by the high energy peak position as shown in figure 2. By increasing the value of the coupling capacitor we were able to shift the high energy peak position towards lower or higher values. The nearly linear behavior is shown in figure 3. Using the inductively coupling only allows running the source with ion energies below 100eV. Increasing the total radio frequency power increased the measured ion current density significantly, as shown in figure 4. To operate the source in a fully automatic way, parameter sets are created and stored in the control software of the SYRUSpro. The source can be switched at any time during the deposition process to another parameter set. The ion energy is mainly determined by the value of the coupling capacitor, the ion current density by the chosen power level.

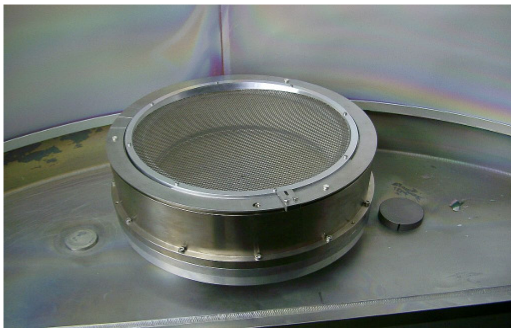

Fig.1 30cm prototype rf-plasma source mounted in SYRUSpro 1510

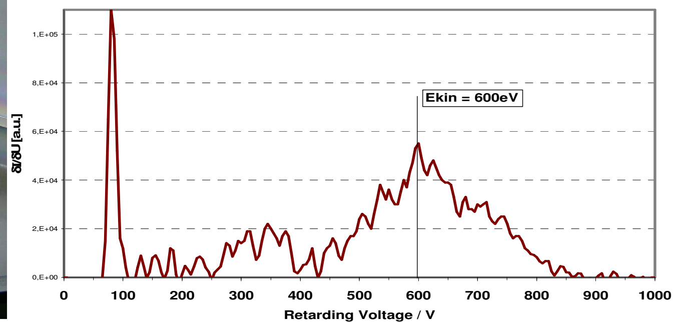

Fig.2 Ion energy distribution of an oxygen plasma

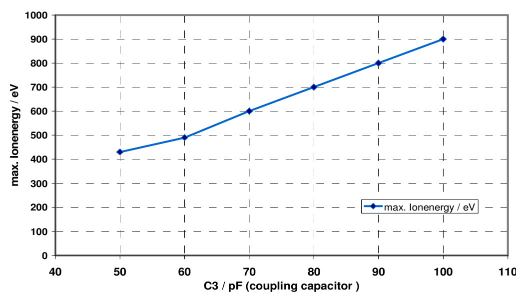

Fig.3 Ion energy dependence by changing the capacitive coupling

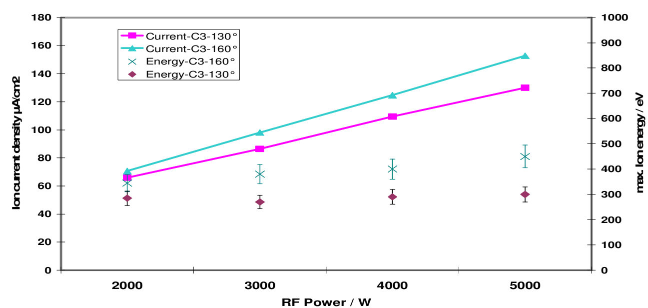

Fig.4 Ion current density and energy dependence by changing the radio frequency power input at a distance of 1200mm

3.2 Single layer

Single layers of SiO_2 and TiO_2 were investigated with different parameter sets of the plasma source, using ion energies between 400eV and 900eV. At deposition rates of 0,6nm/s we were able to achieve dense-shift-free silica layer with moderate compressive stress values of -245MPa and excellent refractive index uniformity over the whole dome as determined by the reflection curves over the 6 radial position of the 1400mm dome (fig.5). The coatings were very smooth. We measured by scanning atomic force microscope a surface rms roughness of 0,345 nm at a 430 nm thick layer, when deposited on a silicon wafer.
Excellent optical properties could also be obtained when depositing TiO_2. With the same high rate of 0,6nm/s we were able to get dense, non absorbing single layers with high refractive index and low surface roughness. The transmittance measurement over the 6 radial positions shows excellent refractive index uniformity as well (fig.6). The measured rms surface roughness of 0.45 for a 253 nm thick layer is slightly higher then the value obtained with SiO_2, but still far below 1nm. The TiO_2 coating exhibited low compress stress. We measured a value of -54MPa. The cross section of the layer was investigated be scanning electron micoscopy. From the picture (fig.7) one can assume that the layer consist of small microcrystallines of approximately 10nm diameter. Table 1 summarizes the results.

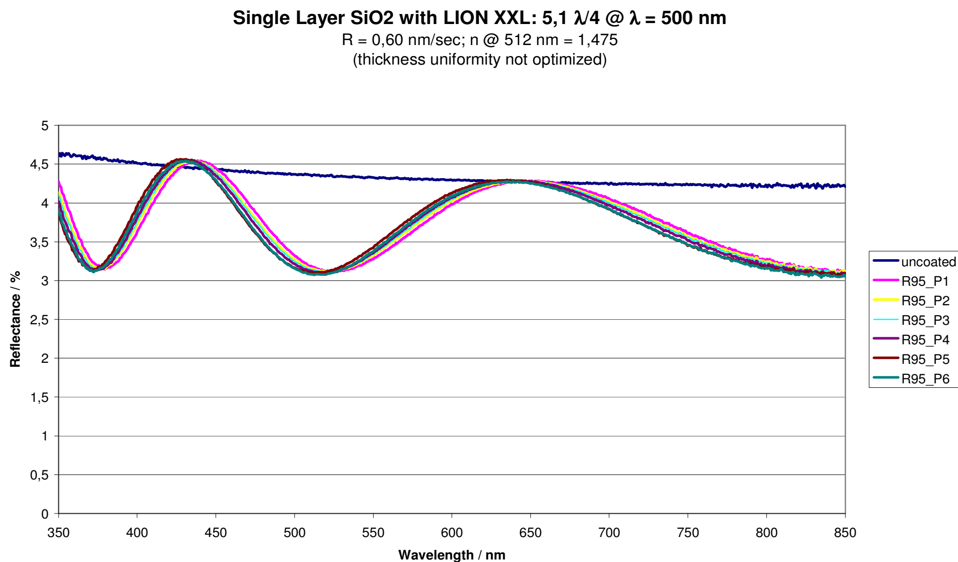

Fig. 5

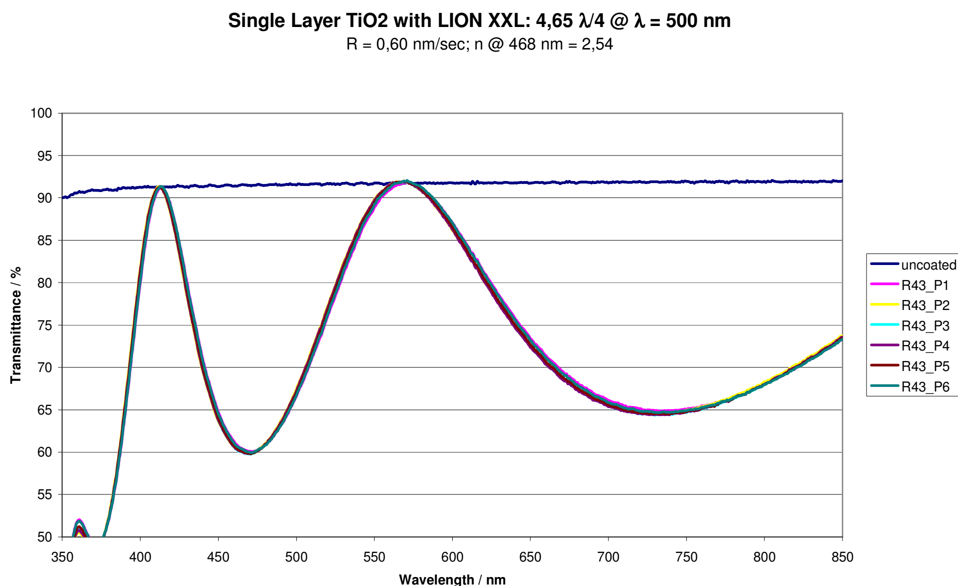

Fig. 6

Table 1	Thickness	Refractive index	Intrinsic Stress	Surface roughness @ 10µm X 10µm	
	nm	n@λ	MPa	Rms (nm)	Ra (nm)
SiO_2 @ 0,6nm/s	430	1,475 @512nm	-245	0,345	0,263
TiO_2 @ 0,6nm/s	253	2,54 @468nm	-54	0,360	0,192
UV-IR-Cut @ 0,5nm/s	5415	-	-153	0,487	0,363

3.3 Multilayer

As an example of a challenging multilayer we deposited a 42 layer UV-IR-Cut filter with edge wavelengths of 400 and 690 nm and a broad blocking range up to 1200nm. The thickness of the layers was controlled by direct monitoring on the dome with the OMS 5000. The dispersion data necessary for an optimum use of the multi-wavelength monitoring strategy were calculated from the presented single layer data. The transmittance curve was measured in situ by the OMS 5000 and ex-situ immediately after the deposition process. No measurable wavelength shift could be detected. Measurement after six month storage under ambient condition confirmed the dense structure of the coating by the absence of any detectable shift. (Fig 8).

By visual inspection the coated B270 substrates showed no signs of scattering losses even by illumination with a projector light. Measuring the integral haze by illumination with a D65 light source resulted in values off 0.09% compared to 0.08% of an uncoated glass slide. The absent of measurable scattering losses was confirmed by the low rms-surface roughness of 0,487nm as measured with an atomic force microscope. The scanning electron microscope picture of a cross section of the filter further confirmed the excellent layer quality. The layer structure of the single layer is present in the whole stack.

We determined the resulting compressive intrinsic layer stress of the filter and achieved a very low value of -153 MPa, keeping in mind the low surface roughness and the dense microstructure. The combined stress of the layer stack could be predicted by using the single layer stress values. Based on the net thickness of SiO_2 (3390nm) and TiO_2 (2025nm) in the stack, combining the stress using a linear approximation results an average compressive stress value of -175 MPa.

To estimate the potential for high performance UV-IR-cut filter we measured the reflection losses and plotted the T+R values in the pass band of the filter (fig.9). Since we found no scattering losses, this represents the achievable transmittance of an ideal AR coated filter. We compared this plot with computed curve from single layer material (fig.9). Within the measurement accuracy of the used equipment we found an excellent correlation.

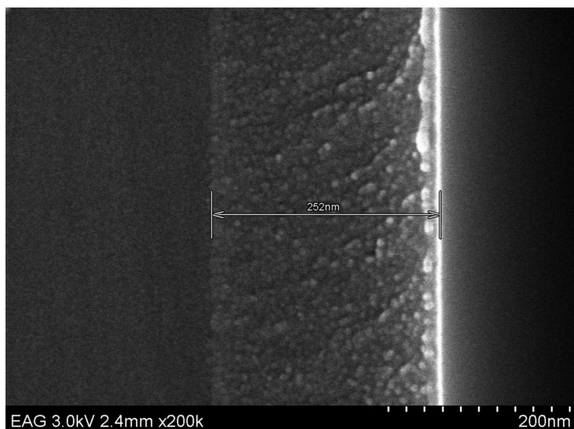

Fig.7 SEM cross section of a TiO_2 single layer

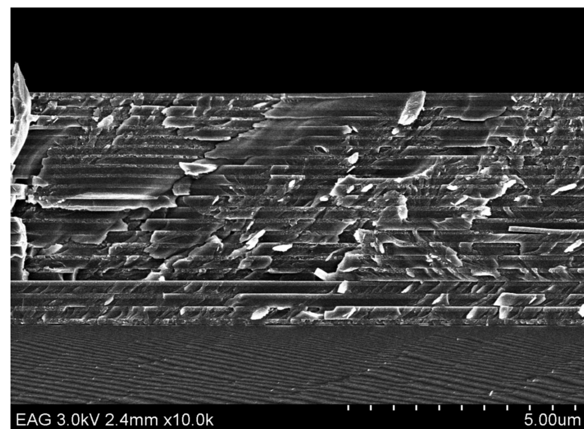

Fig.8 SEM cross section of a UV-IR-Cut filter

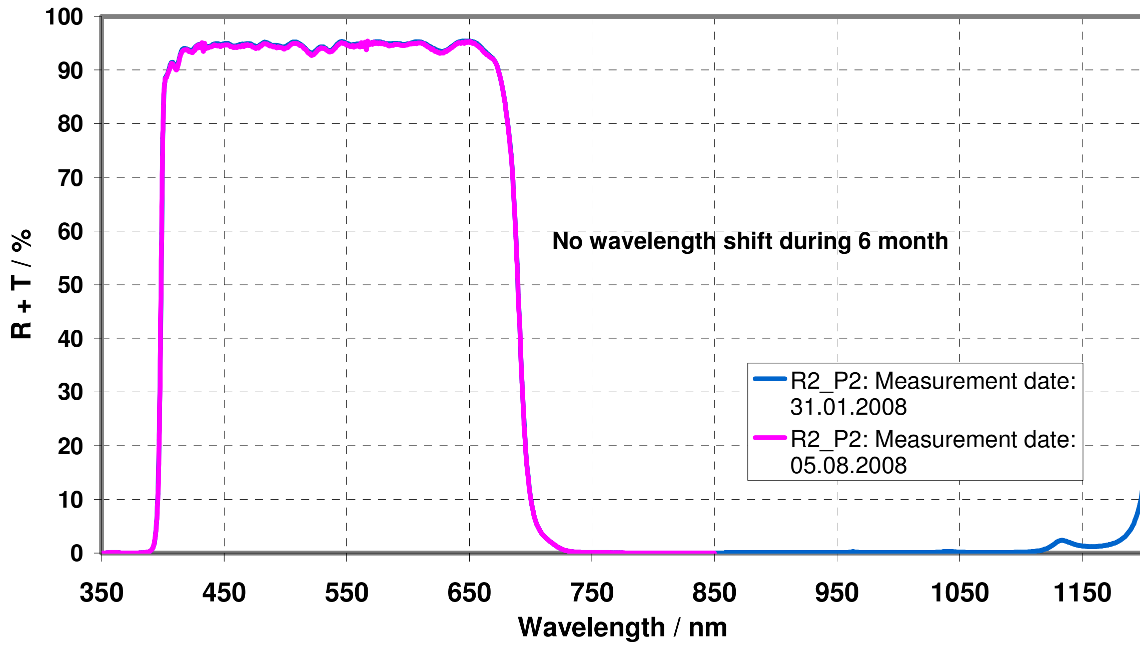

Fig.8 Spectral performance and stability of a 42-layer UV-IR-Cut filter without backside AR

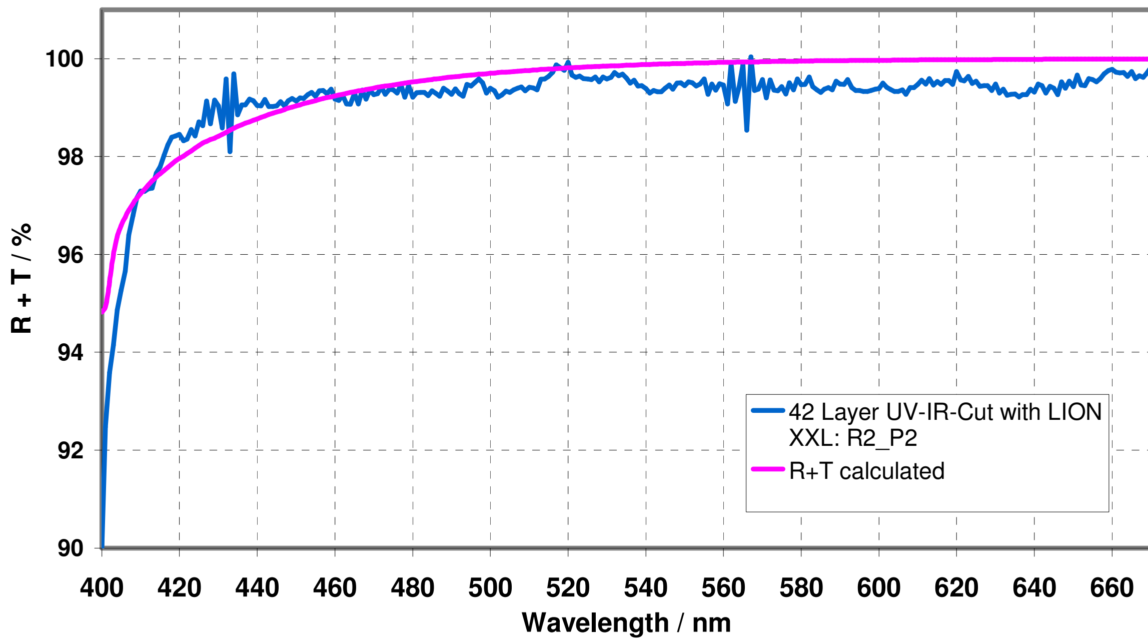

Fig.7 R+T plot of a 42-layer UV-IR-Cut filter produced by plasma ion assisted deposition in a SYRUSpro box coater and a comparison with calculated data from single layer deposition

4. CONCLUSIONS

From the results an average transmittance value in the pass band of an UV-IR-Cut Filter of more than 98% can be achieved with the coating, materials TiO_2 and SiO_2 in a large coating chamber like the SYRUSpro 1510. The prototype of a large single grid radio frequency plasma source proofed its outstanding capabilities in the production of demanding interference coatings. The use of a kinetic energy region between 500 and 900 eV in a plasma ion assisted deposition process for the bombardment of the growing films showed a huge potential in managing the layer properties for the needs of future optical interference filters. Low compressive stress values, dense-shift-free layer structure with excellent optical properties can be combined in an industrial application.

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