

Transparent composite coatings with adapted mechanical stress prepared by ion beam sputtering

Melanie Gauch*, Henrik Ehlers*, Detlev Ristau*

*Laser Zentrum Hannover e.V.

<mailto:m.gauch@lzh.de>

Ion beam sputtering (IBS) was used for the production of nearly stress free organic-inorganic composite materials. This study is dedicated to the optical and mechanical properties of composite materials, consisting of sputtered PTFE and oxides and the compatibility of these coatings to PMMA foils.

1 Introduction

Oxide materials are often employed for protective and optical coatings, because of their high transparency and their mechanical and chemical stability. However, thin and flexible PMMA foils as carrier substrates with different functions need adapted coating materials with properties beyond the quality of oxide coatings. Especially, high coating stress leads to deformation of the polymer foils and to coating failure due to flaking, folding and cracking. Contrary, organic layers reveal high flexibility and low stress, but the mechanical stability has to be improved. Therefore, a coating material which combines the stability of oxides with the flexibility of the polymers is needed.

2 Experimental

The composite coatings were produced by ion beam sputtering from a zone target consisting of PTFE and Al_2O_3 (s. Fig. 1). The composition of the mixed coating is determined by the position of the target in relation to the ion beam. During the deposition process the temperature of the substrates does not exceed 80°C , therefore polymer foils can be used as substrates. In order to avoid stoichiometric deficiencies, additional oxygen gas was let into the chamber.

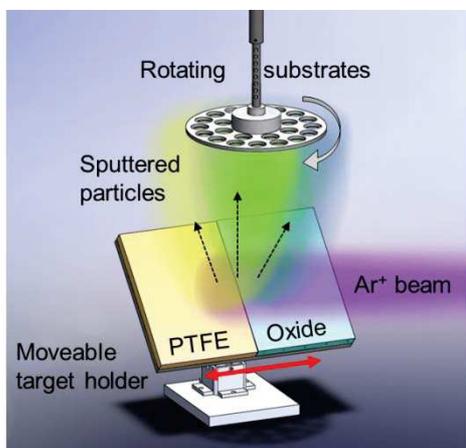


Fig. 1 : Principle of ion beam sputtering with a zone target.

The refractive index and film thickness was calculated from transmission spectra. For the determination of the coating stress, the radius curvature of the substrates before and the corresponding radius after the deposition process were measured with a commercial interferometer (Zygo). On this basis the stress can be calculated with the Stoney formula [1].

3 Composition

During the sputtering process the PTFE is cracked and the carbon reacts with the oxygen to carbon monoxide. Therefore, the composite film consists mainly of aluminum, oxygen, and fluorine with a small amount ($<10\%$) of carbon. Bonds between carbon and fluorine are only present for coatings with low aluminum content. For this reason the PTFE content is notated as the sum of the atomic percentage of fluorine and carbon. Coatings with a C+F content larger than 60 % have a high water content and degrade after a few weeks. More details about the composition can be found in ref. [2]. Therefore, in this study only coatings with a C+F content smaller than 60 % are investigated.

4 Optical properties

Since PTFE and Al_2O_3 are both materials with a low refractive index (1.38 and 1.63, respectively) the composite materials should have a refractive index between both values. In Fig. 2 the refractive index calculated from transmission measurements is shown for different compositions. As expected, the refractive index decreases nearly linearly from 1.63 to 1.36 with increasing C+F content. However, the refractive index of PTFE is already reached at a C+F content of 65 %. The composite with a C+F content of around 50 % is mainly an aluminum fluoride and therefore, a refractive index of 1.40, which is typical for aluminum fluoride [3], is expected.

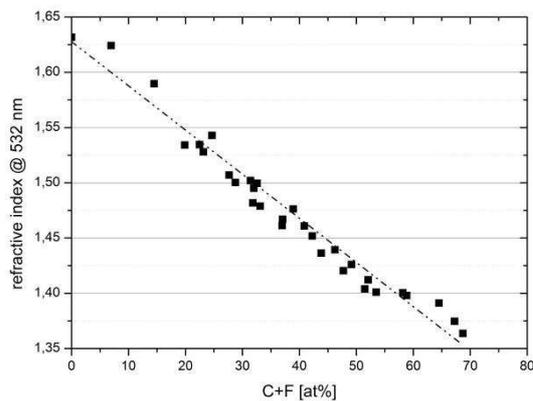


Fig. 2 : Refractive index at a wavelength of 532 nm of $AlO_xC_yF_z$ in dependence of the carbon and fluorine content.

5 Stress

Coating stress can be either compressive or tensile (s. Fig. 3). In general, ion beam sputtered coatings have a compressive stress of several hundred MPa. Since coating stress leads to bending or cracking of the substrates and to cracking and folding of the coating a low coating stress is desirable.

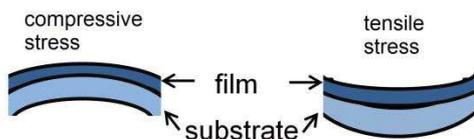


Fig. 3 : Bending of a substrate due to compressive and tensile coating stress.

Ion beam sputtered PTFE is a material with relatively low stress, whereas Al_2O_3 exhibits a compressive stress of around 400 MPa. In literature, tensile stress is reported for ion beam sputtered AIF [4]. For a C+F content of 0% to 50% the composite material changes from Al_2O_3 to AlF_x . Therefore, a change from compressive to tensile stress is expected.

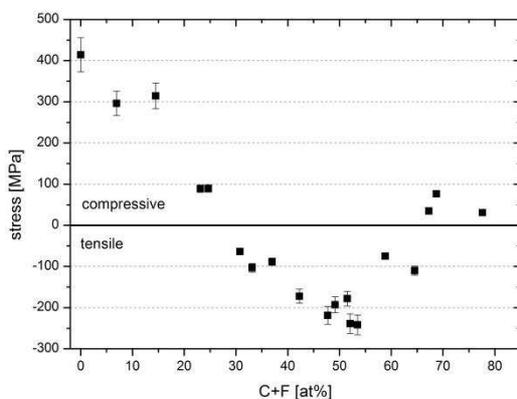


Fig. 4 : Coating stress of $AlO_xC_yF_z$ in dependence of the carbon and fluorine content. In this study, compressive stress is chosen as positive and tensile as negative.

In Fehler! Verweisquelle konnte nicht gefunden werden. the stress in dependence of the C+F content is shown. The coating thickness is 500 nm and fused silica was used as substrate material. Obviously for a C+F content between 0% and 50%, the coating stress changes from compressive to tensile. For higher C+F contents the stress changes back to a low compressive stress comparable to the stress of sputtered PTFE. In case of a C+F content of 30% the stress is near zero. Since the coating stress depends on the substrate material, the C+F content has to be adapted to the substrate in order to produce stress free coatings.

In case of PMMA foils as substrates, a coating with a C+F content of around 50% is suitable, because no stress induced defects like cracks or bending are observed (see Fig. 5 left side).



Fig. 5 : 500 nm thick $AlO_xC_yF_z$ (left side) and Al_2O_3 coating on PMMA foil. The $AlO_xC_yF_z$ is compatible to the foil, whereas the Al_2O_3 coating (right side) shows stress induced cracks and bends the foil.

Acknowledgements

The authors gratefully acknowledge the financial support by Deutsche Forschungsgesellschaft (DFG) within the collaborative research center "Transregio 123-Planar Optronic Systems".

References

- [1] V. Zaporozhchenko, V.S.K. Chakravadhanula, F. Faupel, S. Tamulevicius, M. Andrulevicius, A. Tamuleviciene, et al., "Residual stress in polytetrafluoroethylene-metal nanocomposite films prepared by magnetron sputtering," in *ThinSolidFilms* **518**:5944–5949 (2010)
- [2] M. Gauch, H. Ehlers, D. Ristau, "Properties of transparent organic-inorganic composite coatings prepared by ion beam sputtering of PTFE and Al_2O_3 ," in *Proc.SPIE.* 9627 (2015)
- [3] M. Mende, I. Balasa, H. Ehlers, D. Ristau, D. Douth, L. Gallais, et al., "Relation of optical properties and femtosecond laser damage resistance for Al_2O_3/AIF_3 and Al_2O_3/SiO_2 composite coatings," in *Appl.Opt.* **53**:A383–A391 (2014)
- [4] A. Ode, "Ion beam sputtering of fluoride thin films for 193 nm applications," in *Appl.Opt.* **53**:A330–A333 (2014)