RF GD-OES ANALYSES OF OPTICAL MULTILAYERS

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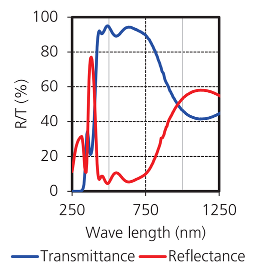
INTRODUCTION

Optical multilayers with antireflective or highly reflective properties in the visible range of spectrum or solar-control layers with low transmittance in the near-infrared range of spectrum are used on different substrates like glass or polymer films.
The antireflective or highly reflective coatings consist of alternating layers of high and low refractive metal oxides. The necessary number of the layers depends mainly on the requirements of the specific application. We present results for an antireflective system with 5 layers of silicon oxide and titanium oxide and for of a broad-band highly reflective Rugate filter with 46 periods of silicon oxide and tantalum oxide gradient layers. The solar-control system on a PET substrate consists of five silver layers which are surrounded by high refractive zinc-tin oxide layers.

In the frame of our work we have tested the possibilities and limitations for the analyses of the depth-profiles of these different optical multilayers by rf glow discharge optical emission spectrometry (RF GD-OES). For this purpose the sputter parameters sputtering pressure, power and duty cycle are optimized for high depth resolutions. The influence of the parameters on the shape of the sputter crater is investigated by optical microscopy and by profilometry. Additionally the effect of changes in spectral reflectance on measured intensities of optical emission lines was studied. For the solar control system on PET film the optimization of sputter parameters is challenging because of the very different material properties of silver, zinc-tin oxide and polymer and the additional necessary preparation of the flexible PET substrate for the analyses.

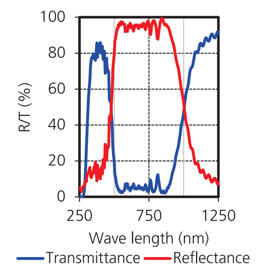
Spectral transmittance and reflectance of the investigated optical multilayers



Antireflective system with UV protection on soda-lime glass

5 layers of silicon oxide and titanium oxide

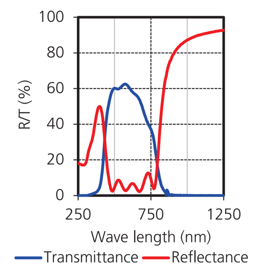
(total thickness: 670 nm)



Broad-band highly reflective Rugate filter on soda-lime glass

46 periods of silicon oxide and tantalum oxide gradient layers

(total thickness: 9650 nm)



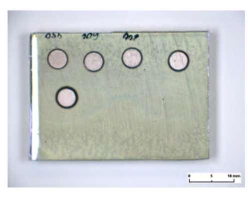
Solar control system on PET polymer film

5 silver layers with a thickness of 8 nm embedded in zinc-tin oxide layers

(total thickness: 450 nm)

EXPERIMENTAL

GD-Profiler 2 (HORIBA Jobin Yvon)



Sample preparation for coated PET polymer films

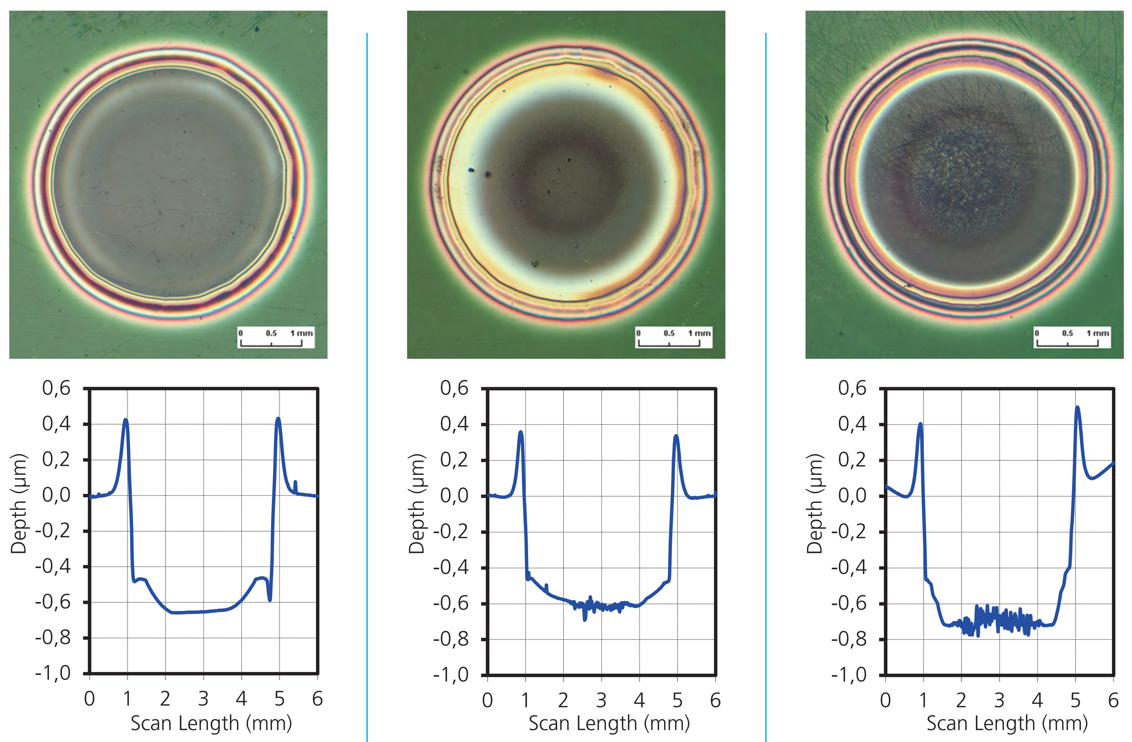
For rf GD-OES analysis the GD-PROFILER 2 (HORIBA Jobin Yvon) with a MARCUS GD-source and anode diameter of 4 mm was used. The instrument features a polychromator (110 ... 620 nm) with 45 installed optical emission lines and an additional high resolution monochromator (160-500 nm). The new designed 13.56 MHz rf sputtering source enables analyses in non-pulsed or in pulsed mode with a variation of duty cycle.

For rf GD-OES analyses the 50 µm thin coated PET polymer film must be glued on a rigid substrate. We have used therefor 3 mm thick aluminium sheets. By the additional use of pulsed rf power, relative low sputtering powers and additional sample cooling smooth craters can be obtained without thermal degradation of the samples.

RESULTS

ANTIREFLECTIVE (AR) SYSTEM WITH UV PROTECTION ON SODA-LIME GLASS

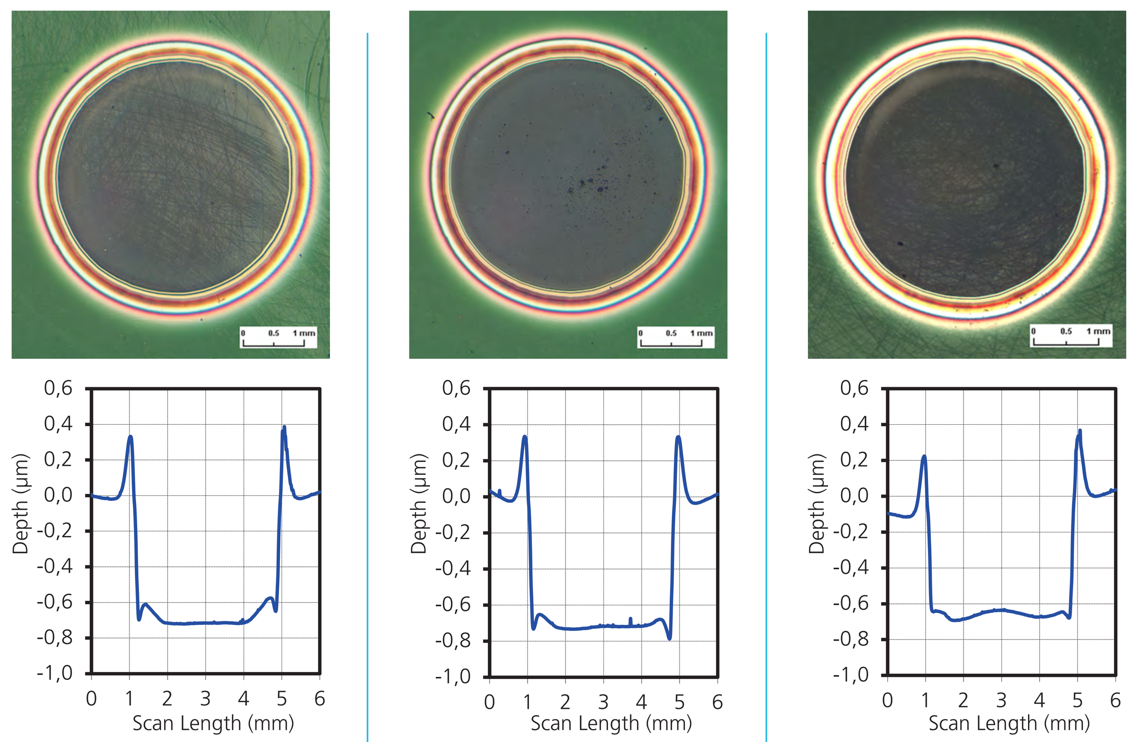
Variation of sputtering pressure using constant sputtering power of 35 W and duty cycle of 0.5; sputtering time about 100 s



450 Pa; 35 W; 0.5 — **600 Pa**; 35 W; 0.5 — **750 Pa**; 35 W; 0.5

At a low sputtering pressure of 450 Pa the sputter crater is relative flat in the centre. Unfortunately the sputtering rate near to the edges is significant lower, which restricts the depth resolution. At higher pressures of 600 Pa the sputter crater becomes more concave, whereas at 750 Pa a significant increase of roughness is detected.

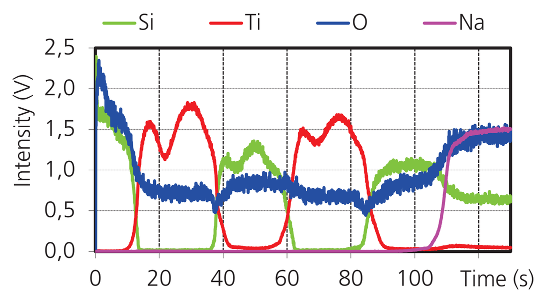
Variation of sputtering power and duty cycle using co nstant sputtering pressure of 450 Pa; sputtering time about 100 s

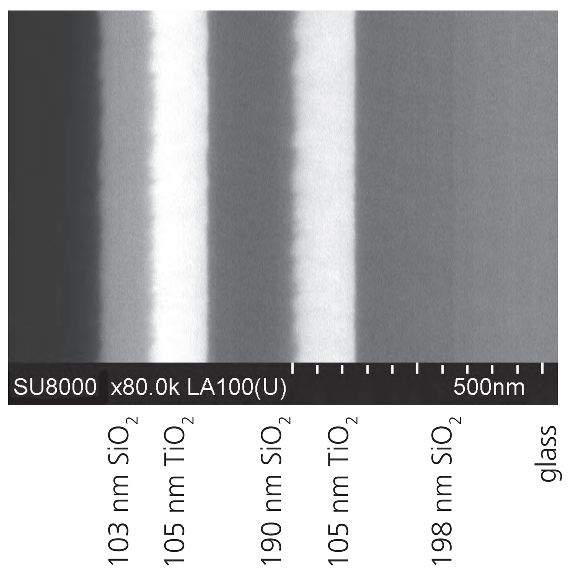


450 Pa; **50 W**; **0.3125** — 450 Pa; **65 W**; **0.25** — 450 Pa; **80 W**; **0.1875**

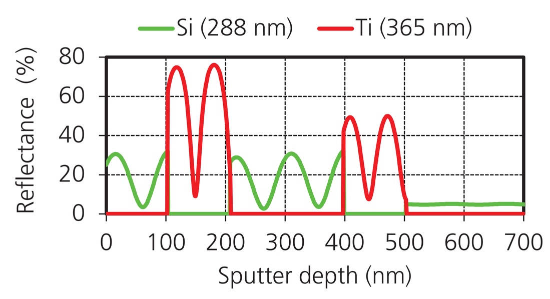
For further improvement of the crater shape the sputtering pressure was kept constant at 450 Pa and the sputtering power was increased. Simultaneously the duty cycle was decreased to avoid a thermal overload of the sample. It can be shown that for 65 W sputtering power and 0.25 duty cycle the flatness between centre and edges is significant enhanced.

RF GD-OES intensity-time-profile for optimized sputter parameters and **FE-SEM micrograph** of cross-section





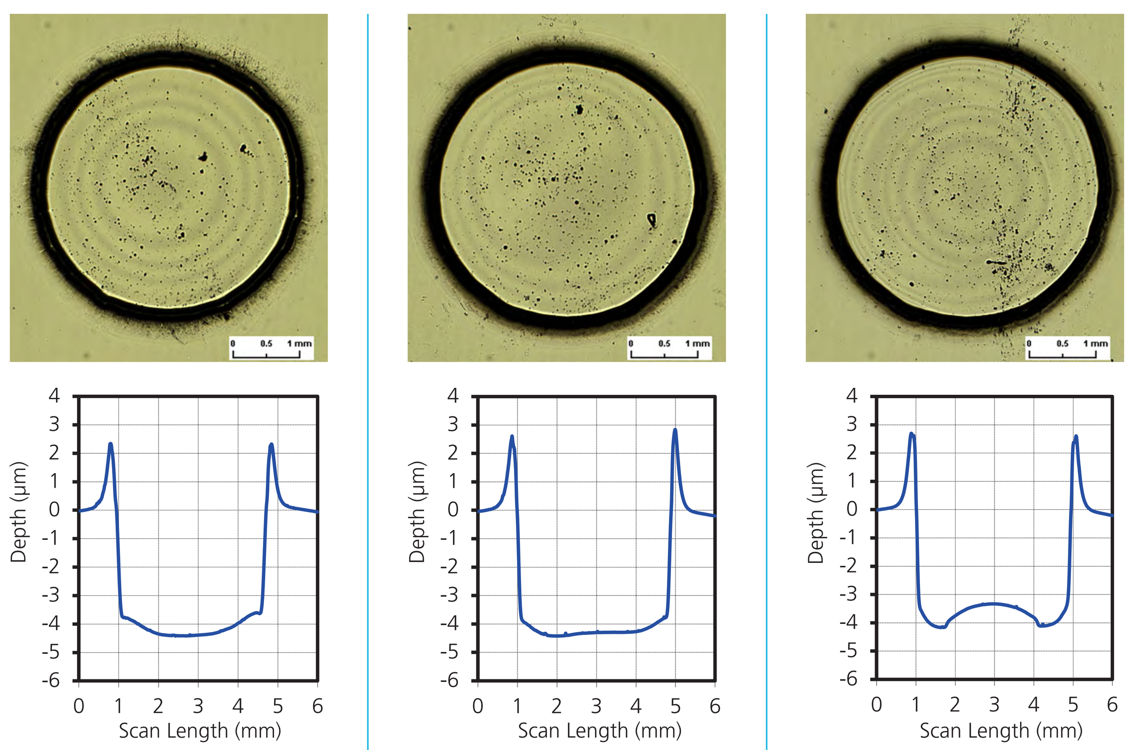
Simulated Reflectance versus sputter depth for the used optical emission lines of silicon and titanium



For optimized sputter conditions (450 Pa; 65 W; 0.25) all SiO_2 and TiO_2 layer of the antireflective system can be clearly resolved.
The results show that measured intensities of the optical emission lines 288.158 nm for silion and 365.35 nm for titanium are drastically inflenced by the spectral reflectivity. The mesured intensity-time profil correlates very well to the values of simulated reflectance versus sputter depth. For quantification of the results this effect has to be taken into acount.

BROAD-BAND HIGHLY REFLECTIVE RUGATE FILTER ON SODA-LIME GLASS

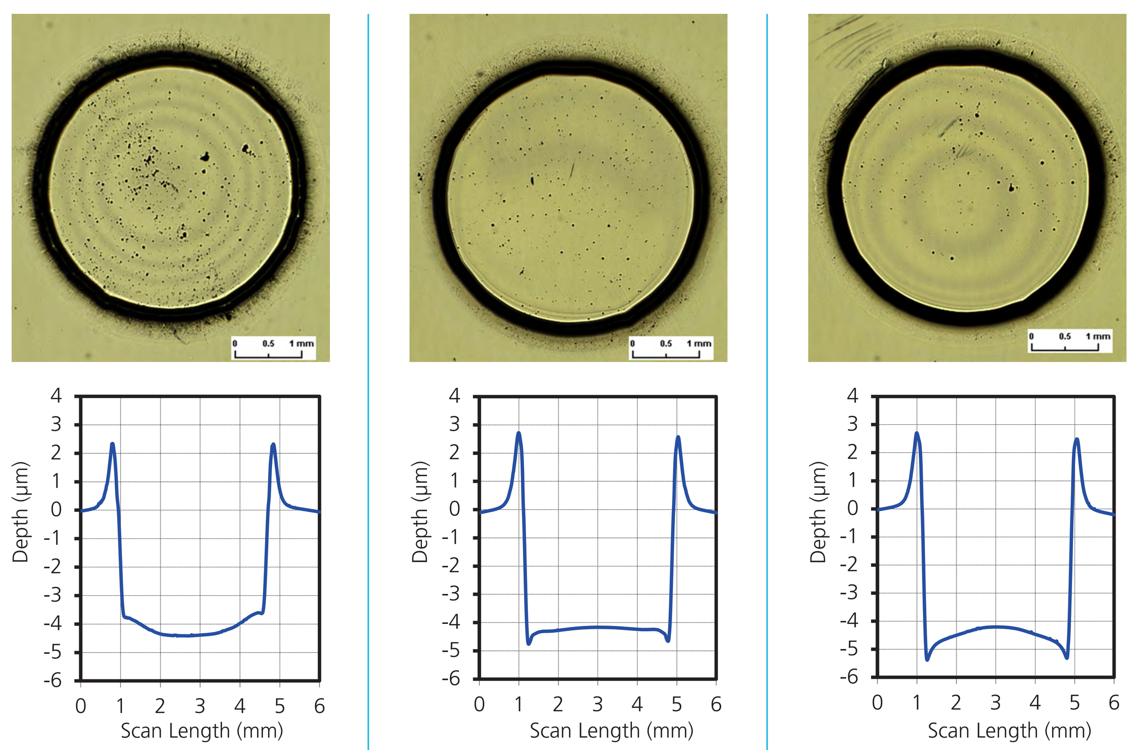
Variation of sputtering pressure using constant sputtering power of 35 W and duty cycle of 0.5; sputtering time 300 s



450 Pa; 35 W; 0.5 — **600 Pa**; 35 W; 0.5 — **750 Pa**; 35 W; 0.5

For lowest investigated sputtering pressure of 450 Pa the centre of the crater is flat whereas the edges are less sputtered. By increasing sputtering pressure to 750 Pa the bottom becomes more and more concave.

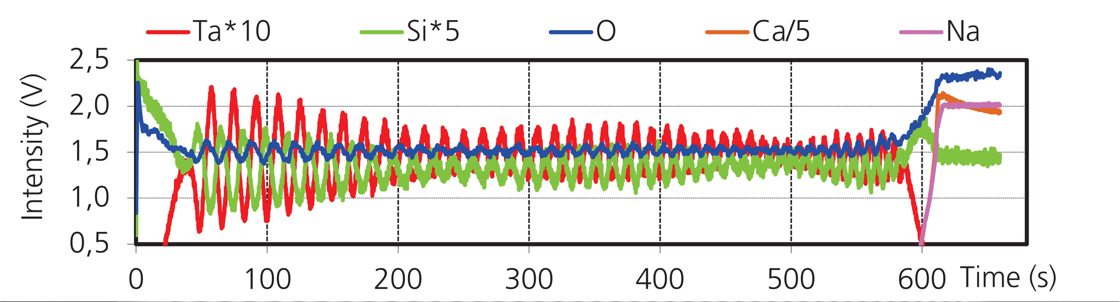
Variation of sputtering power and duty cycle using constant sputtering pressure of 450 Pa; sputtering time 300 s

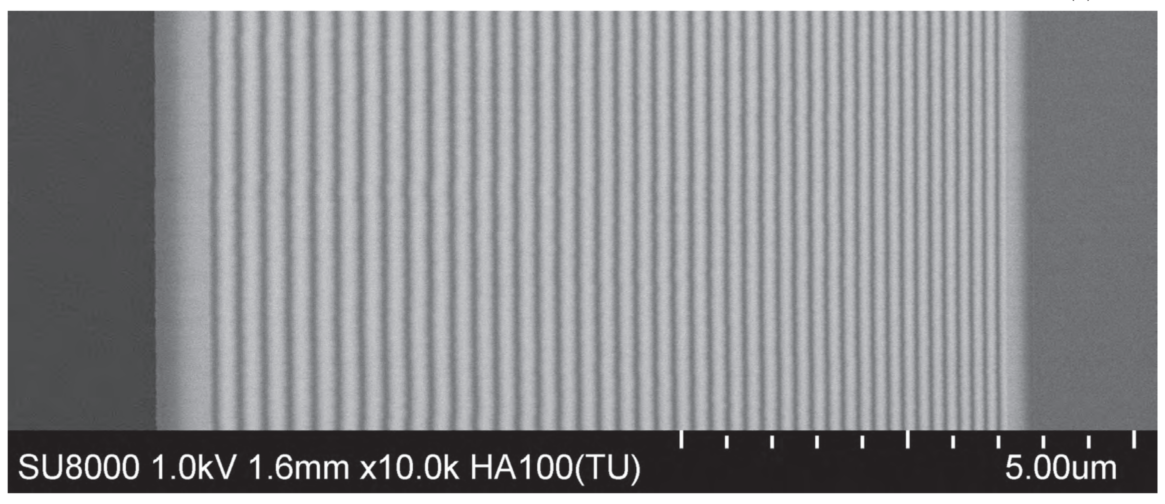


450 Pa; **35 W**; **0.5** — 450 Pa; **50 W**; **0.3125** — 450 Pa; **65 W**; **0.25**

By increasing sputtering power and simultaneous decreasing of duty cycle an optimum for the sputter crater shape could be achieved for 450 Pa sputtering pressure, 50 W sputtering power and 0.3125 duty cycle. The crater shows the typical development from a concave over flat to a convex bottom.

RF GD-OES intensity-time-profile for optimized sputter parameters and **FE-SEM micrograph** of cross-section

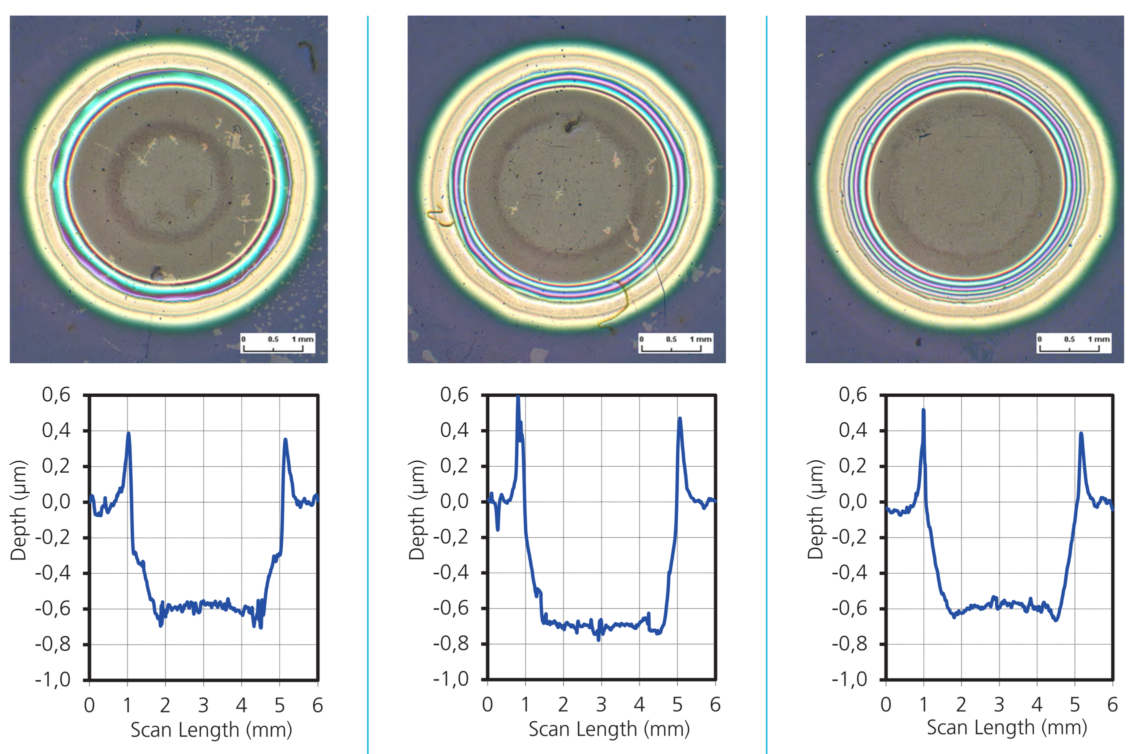




GD-OES intensity-time profile and corresponding FE-SEM micrograph of a Rugate filter with 46 periods of silicon oxide and tantalum oxide gradient layers with a total thickness of 9.65 µm. For optimized sputter parameters a depth resolution better than 50 nm could be achieved at sputter depth of about 9 µm!

SOLAR CONTROL SYSTEM ON PET POLYMER FILM

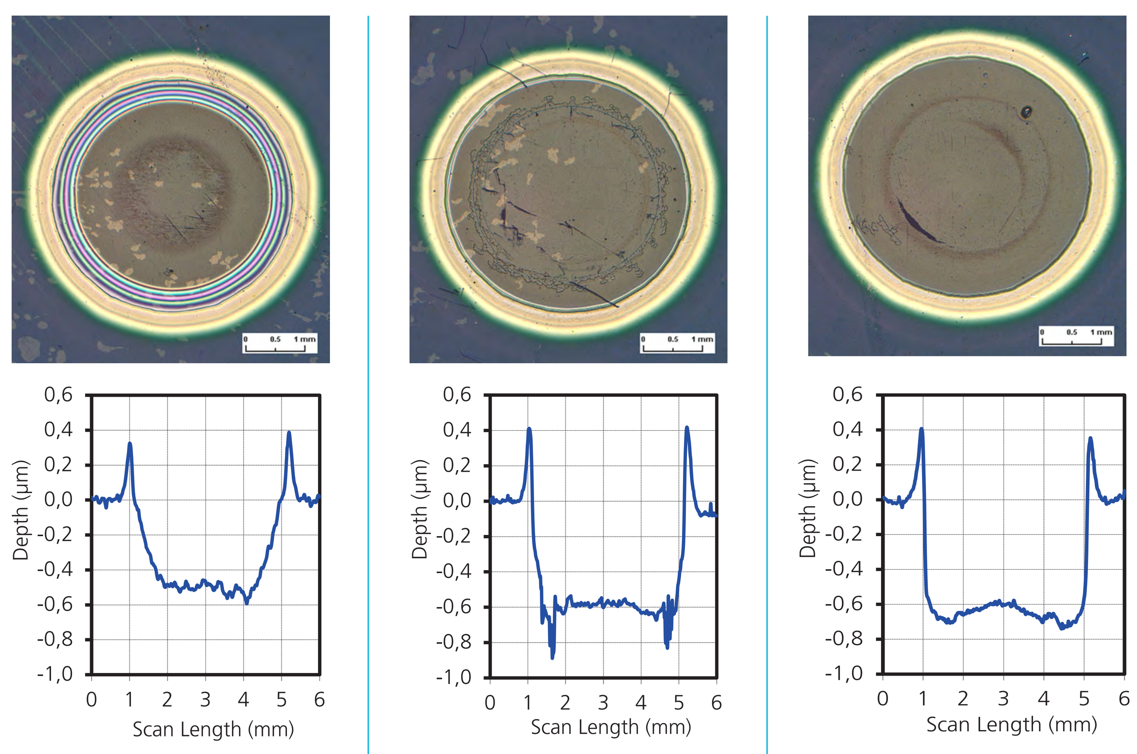
Variation of sputtering pressure using constant sputtering power of 15 W and duty cycle of 0.1875; sputtering time of about 100 s



600 Pa; 15 W; 0.1875 — **750 Pa**; 15 W; 0.1875 — **900 Pa**; 15 W; 0.1875

The solar control system is composed of 5 periods with alternating thin silver and zinc-tin-oxide layers deposited on PET substrate. For low sputtering power of 15 W the optimum flatness of the bottom of the sputter crater was achieved for a sputtering pressure of 750 Pa. Unfortunately the sidewalls of sputter crater are oblique and not perpendicular under these conditions.

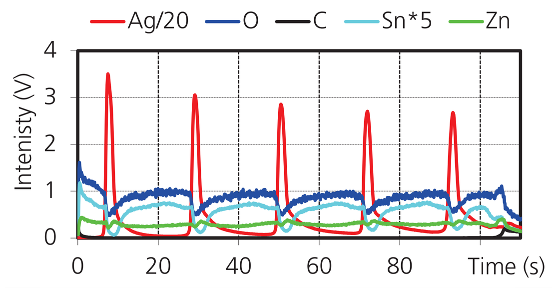
Variation of sputtering power using constant sputtering pressure of 750 Pa and duty cycle of 0.1875; sputtering time of about 100 s

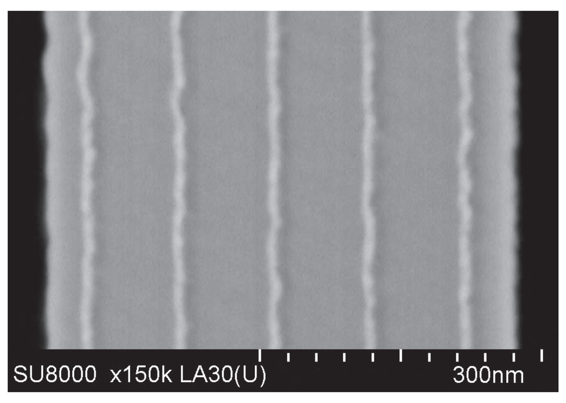


750 Pa; **10 W**; 0.1875 — 750 Pa; **20 W**; 0.1875 — 750 Pa; **25 W**; 0.1875

At lower sputtering power of 10 W the sidewalls becomes as expected more oblique. After increasing the sputtering power to 20 W the sputter craters show more perpendicular sidewalls, but also some first hints of thermal damage of PET film are visible. At 25 W power the sputter crater has additionally a convex bottom.

RF GD-OES intensity-time-profile for optimized sputter parameters and **FE-SEM micrograph** of cross-section





For analyses of the solar control system on 50 µm PET films the samples are glued on 3 mm thick aluminum sheets.
The solar control system with a total thickness of 450 nm is composed of 5 periods with alternating very thin silver layers and zinc-tin-oxide layers.

It can be shown that with optimized sputter parameters (750 Pa; 15 W; 0.1875) flat sputter craters are obtained. It is remarkable that all 5 silver layers with a thickness of only 8 nm can be clearly resolved.

SUMMARY AND CONCLUSIONS

The results show that optical multilayers on glass and polymer films can be successful analysed by rf GD-OES. Depth resolution in the nanometre range can be achieved with optimized sputter parameters and the additional use of pulsed rf power. For quantitative estimations the effect of changes in spectral reflectivity on measured intensity of optical emission lines has to be taken into account.

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