CHARACTERISATION OF RUTILE TiO_2 LAYERS FOR HIGH PRECISION OPTICAL APPLICATIONS

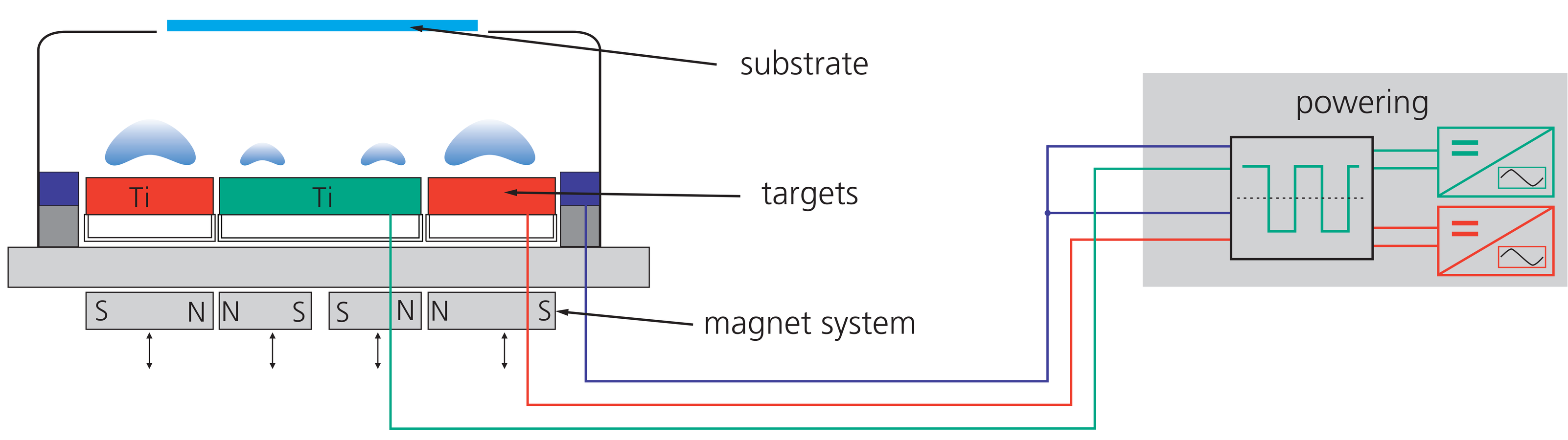
T. MODES, W. SCHÖNBERGER

FRAUNHOFER INSTITUTE FOR ELECTRON BEAM AND PLASMA TECHNOLOGY FEP

Fraunhofer FEP

MOTIVATION

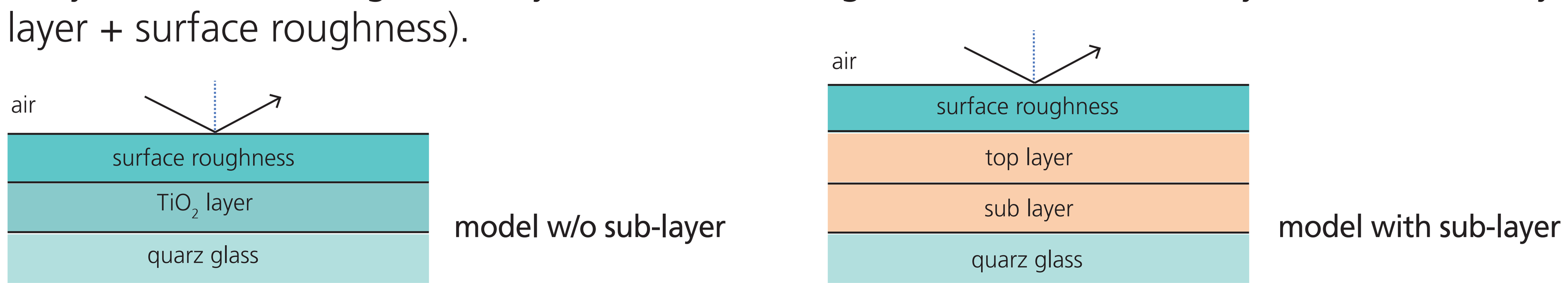
Titanium dioxide in the rutile modification is the material with the highest refractive index and transparent in visible wavelength range. The application of such a high refractive material would be desirable for high precision optical layer systems. Further requirements for these applications are homogenous layers with low roughness.
Sputtering technology allows deposition of amorphous TiO_2 layers with high precision and high productivity on large scale. But these amorphous layers have a lower refractive index than the crystalline TiO_2 layer. When depositing crystalline TiO_2 layers at elevated substrate temperatures and by increased ion bombardment the layer growth often starts with lower refractive sub layer and the surface roughness is higher due to crystallites. For the optimisation of the process parameters a precise method is required to detect these features.



EXPERIMENTAL

Titanium dioxide layers have been deposited using reactive pulse magnetron sputtering (PMS) and high pulse power magnetron sputtering (HPPMS). The film deposition was carried out by stationary sputtering using the Double Ring Magnetron DRM400. Pulsed powering is carried out by using the UBS-C2 two-channel pulse generator (Fraunhofer FEP) with Pinnacle DC generator (Advanced Energy) in case of PMS and by TruPlasma Highpulse 4006 Generator (TRUMPF Hüttinger). The powering, process pressure, gas composition, substrate temperature and HF-Bias on substrate holder were varied to obtain homogenous TiO_2 layers with high refractive index.
Transmission and reflectivity measurements have been carried out by Lambda 950 (Perkin Elmer) spectrophotometer with the VN accessory. The T/R spectra have been fitted by CODE (W. Theiss) optical simulation software using a single layer or a sub layer model.

The ellipsometric measurements were done with a spectroscopy ellipsometer SE 850 (SENTECH) in the wave length range 280 – 830 nm at different angles. The measured data were fitted with a 2 layer model (homogenous layers + surface roughness) and with a 3 layer model (sub layer + top layer + surface roughness).

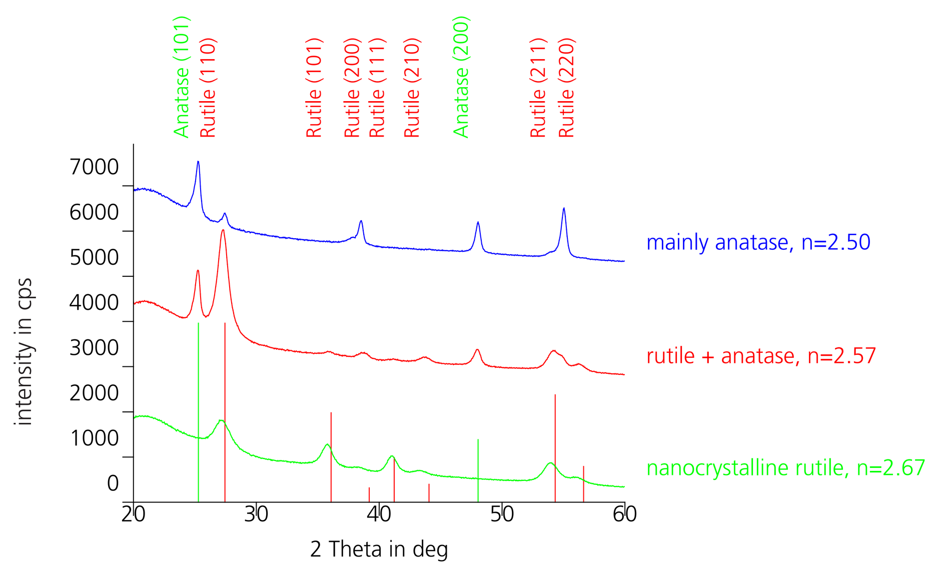


RESULTS

OCCURRING PHASES

Amorphous TiO_2 has a maximum refractive index of 2.45. The crystalline phases anatase and rutile have refractive indexes of 2.52 - 2.59 and 2.65 - 2.95 (depending on the crystallographic direction).

The refractive index of the top layers was determined between 2.5 and 2.72. Therefore these high values could be assigned to the crystalline TiO_2 phases. For layers with a refractive index in the range from 2.5 to 2.6 rutile-anatase mixtures were assumed and for values above 2.6 a predominantly rutile phase.
These assumption from the refractive index were confirmed by X-ray diffraction.
For TiO_2 it is therefore possible to correlate the occurring phases with the refractive index determined by spectroscopic ellipsometry.

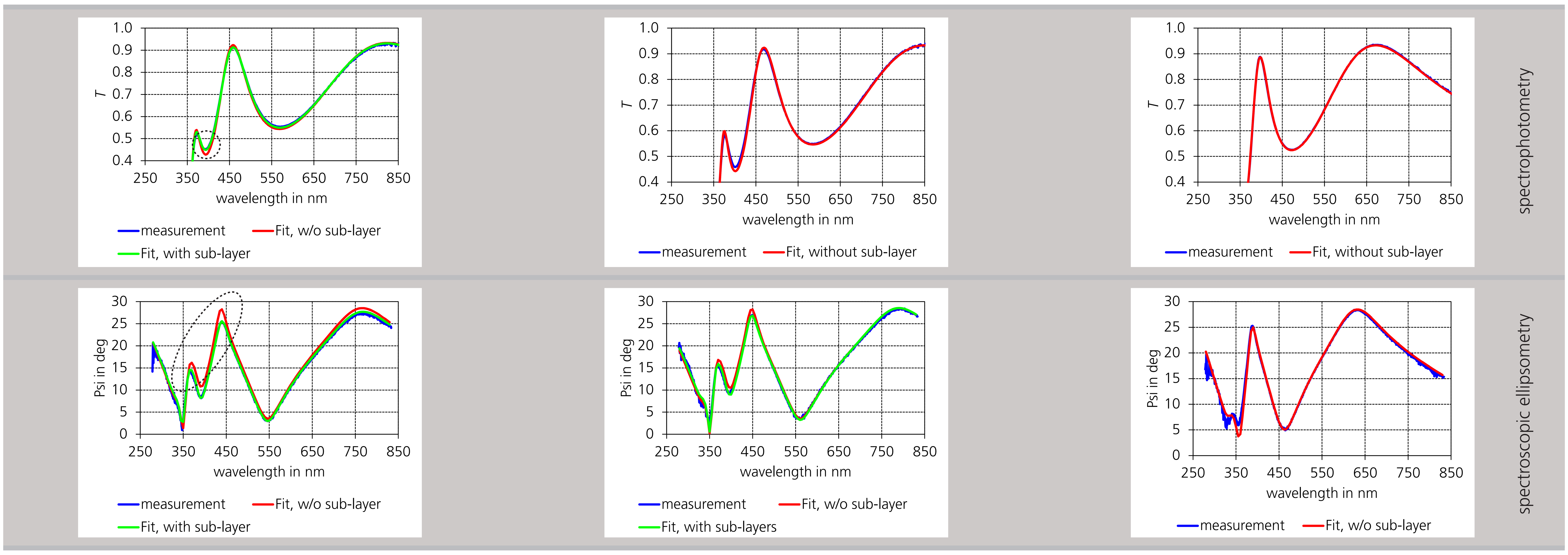


SUB LAYER

For un-optimized deposition parameters crystalline TiO_2 layers have a sub layer with a significant lower refractive index (Δn = n(sub layer) – n(top layer) = -0.33, thickness 20 – 50 nm). These sub layers can be detected with spectrophotometry and spectroscopic ellipsometry.

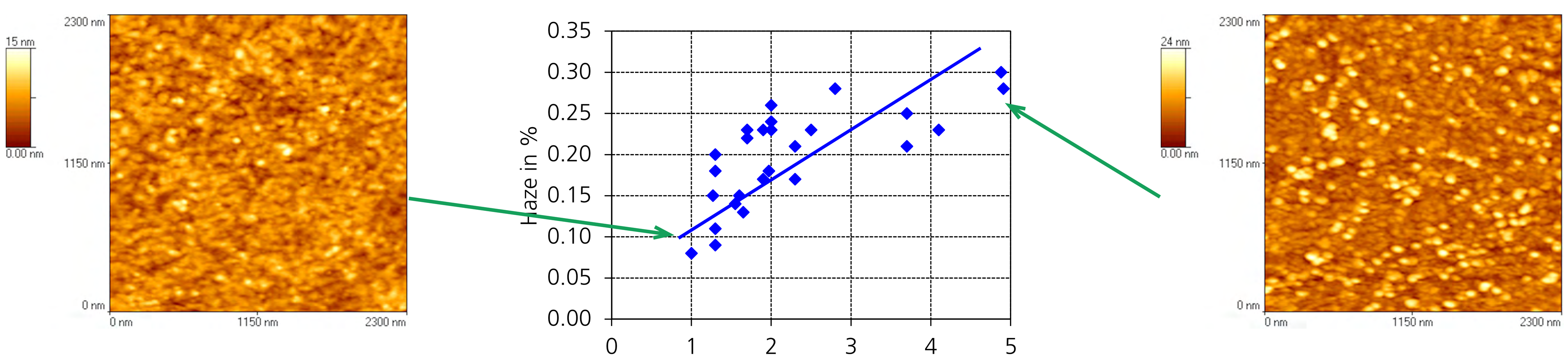
By optimizing the deposition parameters the difference in the refractive index could be decreased. Here: a 40 nm thick sub layer with Δn = -0.01
Spectrophotometry was not able to analyze this sub layer. But with spectroscopic ellipsometry this sub layer can be clearly detected.

With these results the process parameters could be further optimized to deposit high refractive layers (here: n=2.68) without a sub layer, which are required for high precision optical layer systems.



SURFACE ROUGHNESS

The surface roughness could also be modelled by spectroscopic ellipsometry. Depending on the process parameters surface roughness layer thicknesses between 1.0 and 5 nm were determined. The different roughness values correlate well with haze measurements (measure for light scattering).
These results could be approved by roughness investigations by AFM.



Sample with low roughness values *Sample with high roughness values*

CONCLUSIONS

- The occurring TiO_2 phases can be concluded from the refractive index. This correlation could be confirmed by x-ray diffraction measurements.
- The detection of relatively thin sub layers with very low refractive index differences (down to 0.01) is reliably possible by spectroscopic ellipsometry.
- Furthermore, the modelling of roughness by ellipsometry could be approved by further roughness investigations and correlated well with haze measurements.
- It could be shown, that the spectroscopic ellipsometry allows the fast and reliable characterization of sputtered rutile TiO_2 layers in respect to optical properties influenced by microstructure gradients and surface roughness.

CORRESPONDING CONTACT

Fraunhofer-Institut für Elektronenstrahl- und Plasmatechnik FEP

Winterbergstraße 28
01277 Dresden, Germany

www.fep.fraunhofer.de

Dr.-Ing. Thomas Modes
thomas.modes@fep.fraunhofer.de

Phone +49 351 2586 183
Fax +49 351 258655 183

Corresponding Contact

ACKNOWLEDGEMENTS

This work has been supported by the German Federal Ministry of Education and Research (BMBF) under promotional reference 13N12125-OptiOxid.

Federal Ministry of Education and Research

Poster online (PDF)