REACTIVE MAGNETRON SPUTTER TECHNOLOGIES FOR PRECISION OPTICAL COATINGS

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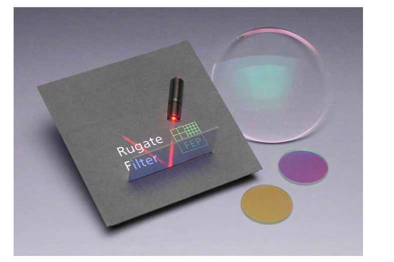
INTRODUCTION



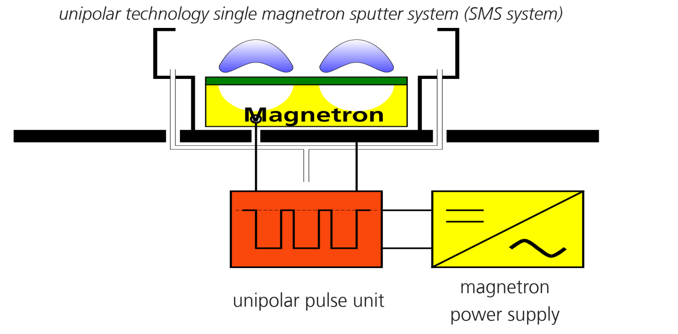
Reactive magnetron sputtering becomes increasingly important for the deposition of optical coatings. Due to energetic activation of film growth, sputtered films are dense and show a good environmental stability. Films like Al_2O_3, SiO_2, Ta_2O_5 can be produced with very little absorption and scattering losses. Therefore they are well suited for laser optics applications. Further advantages of reactive magnetron sputtering are the comparatively short deposition time, the adaptability to substrate shape and size as well as established methods of fully automatic substrate handling resulting in a very versatile coating technique. In stationary mode there is the possibility of depositing coatings with freely defined refractive index by reactive sputtering of different reactive gases or by reactive co-sputttering of different target materials. Changing the reactive gas composition or the power ratio during deposition allows gradient layers, giving new design possibilities. It is also the basis for the fast and cost efficient deposition of a variety of coatings in one coating step at a single deposition station. In the paper the antireflective coating of ophthalmic lenses is presented as an example of this technology.

In-line coating is suited for the uniform coating of large substrates with up to 1 m size. Similarly it also allows the cost efficient coating of large numbers of smaller substrates.

CHARACTERISTICS OF PULSE MAGNETRON SPUTTER PROCESS

- current standard technology for optical coatings: evaporation (thermal and electron beam)
- additional plasma activation ⟶ dense coatings with very good properties
- IBS and RF-sputtering technologies: very good properties, high reproducibility; low deposition rate ⟶ drawback for thick layer stacks

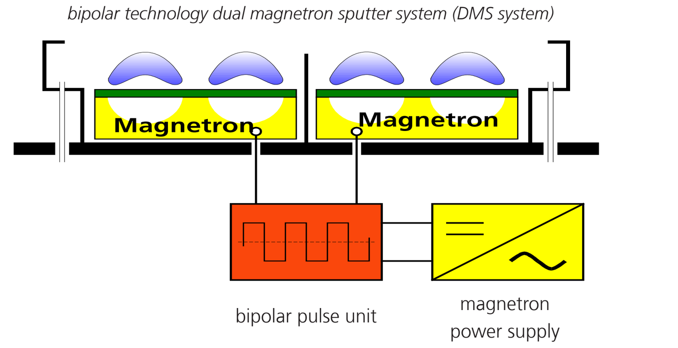
unipolar technology single magnetron sputter system (SMS system)



Reactive pulse magnetron sputtering and features

- unipolar and bipolar mode with adjustable duty cycle and arc handling:
  ⟶ solution of former problems in reactive sputtering and new features that generate advantages regarding accuracy, reproducibility, film properties, cost and productivity
  ⟶ technology becomes more and more interesting for different applications in optical coatings
- excellent process stability and reproducibility
- very high deposition rates (e.g. SiO_2: 4 nm/sec or 80 nm × m/min)
- uniform coating of »large« substrates: stationary coating: ±0.5 % on 8" substrates using Double Ring Magnetron; in-line coating: ±0.1 % on h = 500 mm
- new degrees of freedom to optimize film properties
- by matched energetic substrate bombardment during deposition:
  - dense climatically stable films, low roughness and low (≈zero«) stress coatings, thin closed layers, …
  - coating of temperature sensitive substrates
  ⟶ PMS Technology is suited for precision optical coatings, also for laser applications with high power density

bipolar technology dual magnetron sputter system (DMS system)



COATING HARDWARE AND TECHNOLOGY

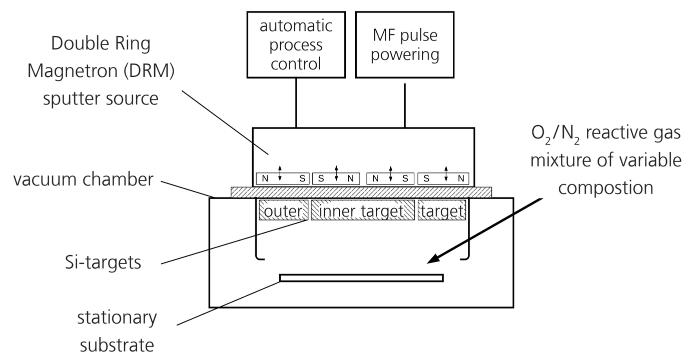
Stationary deposition of single and multilayer, ternary compounds and gradient layer with Double Ring Magnetron (DRM)

Double Ring Magnetron DRM 400 of Fraunhofer FEP for uniform coating of substrates up to Ø 200 mm (8")

- two electrically seperated conductive targets
- moveable magnet system for each target

time variable gas mixing ratio:
- deposition of gradient layer
- deposition of multilayer

at a single deposition station



Sputter equipment for stationary coating with DRM 400 of Fraunhofer FEP (I)
- cluster type sputter equipment
- simultaneous deposition processes at different coating stations

VON ARDENNE Anlagentechnik GmbH

Sputter equipment for stationary coating with DRM 400 of Fraunhofer FEP (II)
- rotary table equipment with 3 or 4 DRM stations in one chamber
- multilayer deposition at different coating stations
- one or two process chambers, load/lock and handling system

FHR Anlagenbau GmbH

Unipolar/Bipolar Pulse Magnetron Sputter System PMS 1000 of Fraunhofer FEP for precision in-line coating

self-sufficient system

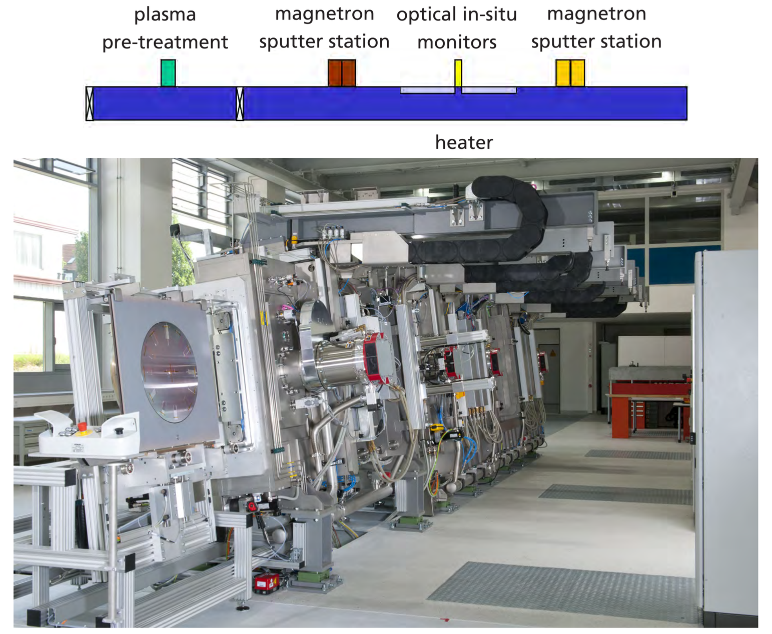
process control:
complete automatically with process management computer

RM-type magnetrons:
movable magnet system
target length: up to 1 m

pulse powering:
unipolar/bipolar switching unit UBS-C2 for two independant channels 20 kW each

variable frequency range: 1 … 50 kHz
duty cycle: 0.3 % … 99.7 % (at 1 kHz), 15.0 % … 85.0 % (at 50 kHz)

In-line sputter equipment PreSensLine for precision coatings at Fraunhofer FEP

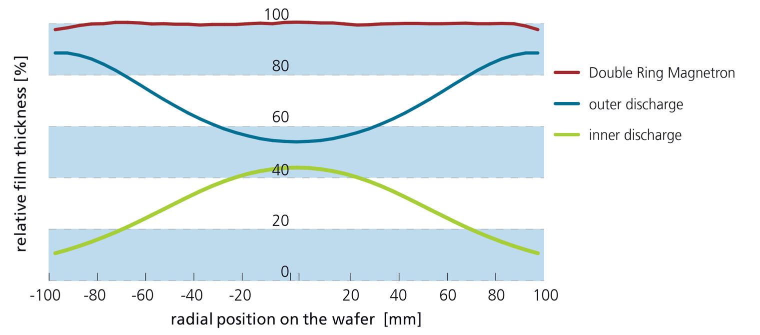


New level of precision at high productivity
- high reproducibility by excellent stability of sputter process
- control of film structure and interface by PMS process
- precision control of substrate movement during coating (±0.025 % at linear speed up to 0.5 m/s)
- substrate rotation up to 120 rpm
- long term film thickness uniformity by in-situ correction using adjustable shieldings
- stabilized substrate temperature for constant film growth conditions up to 400 °C
- absolute precision of film thickness and optical function by in-situ monitoring of every half-layer and in-situ correction of substrate movement or of film design (in situ ellipsometer)
- RM 800 magnetrons of Fraunhofer FEP with 800 mm target length
- substrate size up to 600 mm × 750 mm; thickness: up to 90 mm
- substrate bias (rf, rf-pulse, dc)

Vacuum equipment: VON ARDENNE Anlagentechnik GmbH
Process equipment: Fraunhofer FEP

RESULTS AND EXAMPLES OF APPLICATION

Uniform coating of Ø 200 mm (8") substrates using Double Ring Magnetron DRM 400



- superposition of film thickness distributions of two concentric discharges
- film thickness uniformity: up to ± 0.5 % on 8" substrate by stationary coating
- adjustment for coating of curved substrates

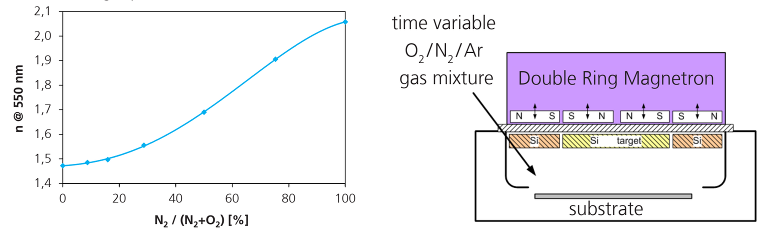
material	deposition rate [nm/sec]	refractive index n		extinction coefficient k		
		193 nm	500 nm	157 nm	193 nm	500 nm
SiO_2	4	1.58	1.46	-	7×10^{-4}	$< 10^{-5}$
Si_3N_4	2		2.05			4×10^{-4}
Al_2O_3	2.5	1.83	1.67	-	5×10^{-3}	$< 10^{-5}$
AlF_3	0.1 … 1	1.46	1.38	2×10^{-3}	1×10^{-3}	$< 10^{-5}$
Ta_2O_5	3		2.20			2×10^{-4}

- deposition of dense films due to energetic bombardment
- films show high refractive index and very low roughness
- Al_2O_3, AlF_3 and SiO_2 suited for UV applications at 193 nm

Methods for depositing gradient layers using stationary coating

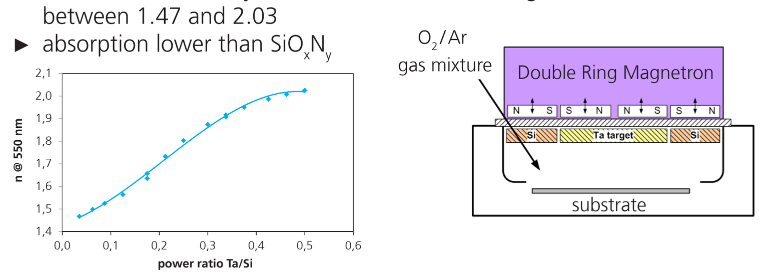
refractive index of SiO_xN_y films
- freely adjustable between SiO_2 and Si_3N_4

reactive sputtering in a time variable mixture of different reactive gases



refractive index of $Si_xTa_yO_z$ films
- refractive index n adjustable between 1.47 and 2.03
- absorption lower than SiO_xN_y

reactive co-sputtering of two different target materials



Environmental stability of dielectric optical filters

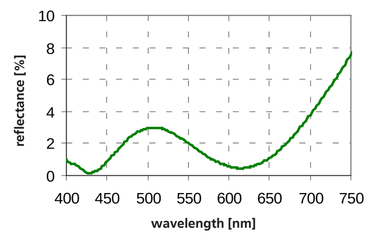
optical filter	process technology	number of layers	total thickness [µm]	wavelength shift [%] between … 30 % rH and 90 % rH	90 % rH at air pressure and vacuum
SiO_2/Al_2O_3 multilayer	Reactive Pulse Magnetron Sputtering (Fraunhofer FEP)	81	5.6	0	0
$Si_xTa_yO_z$ rugate filter		-	16.5	0	0.04
SiO_2/Si_3N_4 multilayer		251	42	0	0.12
SiO_2/TiO_2 multilayer	Plasma-IAD (METI)	30	2.8	0.24	1.4

transmission measurements in mini-atmosphere control box (standardization project ISO/TC206 of METI Japan) at 25 °C with 0.2 nm resolution:
- shift free optical filters by Reactive Pulse Magnetron Sputtering technology

result of this investigation:
- excellent environmental stability of films deposited with reactive PMS-technology

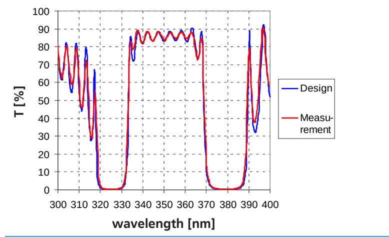
Antireflection coatings for ophthalmic lenses

double sided $Si_xO_yN_z$ antireflective (AR)-coatings
- homogeneous $Si_xO_yN_z$ AR coating of strongly curved lens (surface radius 84 mm) with uniform green reflection color by power matching of Double Ring Magnetron discharges
- fast and cost efficient deposition: deposition time < 2 min without interruption of plasma process at one coating station
- low thermal substrate load for coating temperature sensitive substrates (ΔT_{Coat} < 30 K)
- good adhesion and environmental stability on plastic substrates, e.g. PC (passed: cross cut tape test, boiling test, climatic test, scratch test)



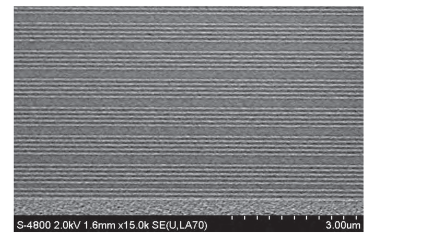
AR-coated (with special green reflectance) and uncoated ophthalmic lenses (right side)

Precision optical filters: example bandpass filter



- design: (HL)^2 H 2L (HL)^2 [(HL)^2 H 4L (HL)^2]^6 (HL)^2 H 2L (HL)^2 H
- H: Al_2O_3, L: SiO_2
- 81 layer, total thickness 5.6 µm

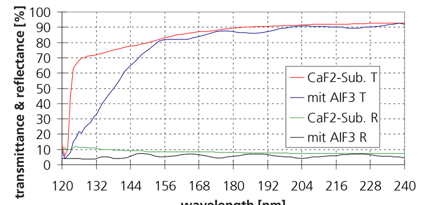
Structure and roughness of the filter



- average roughness R_a = 0.2 nm

SEM of bandpass filter

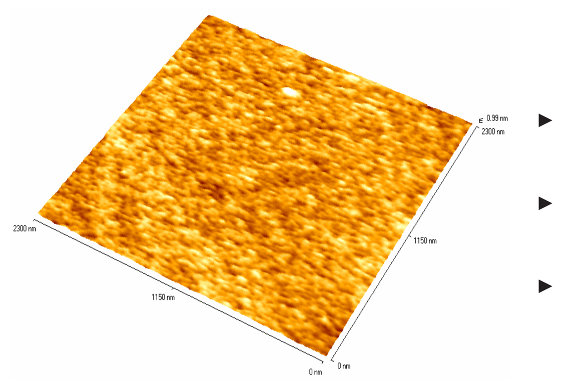
Fluoride coatings
- reactive pulse sputtering of Al target in Ar/F_2 mixture
- deposition rate 5 … 40 nm/min
- spectral T and R measurement of 500 nm AlF_3-layer on CaF_2-substrate at incident angle 0°, with back side reflection, without scattered light



- low absorption for λ > 150 nm

AlF_3: transmission and reflection

Very low roughness by adapted process and pulse parameters



- Tantalum oxide Ta_2O_5 unipolar PMS on Si
- film thickness 200 nm
- 2.3 µm scan
- R_a = 0.08 nm

Layer properties

roughness

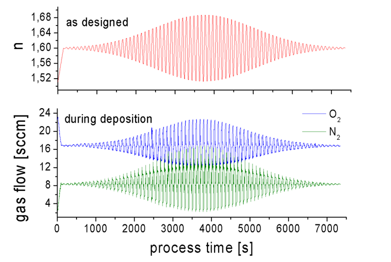
	SiO_2	Si_3N_4	Ta_2O_5	Nb_2O_5
Ra [nm] (film thickness)	0.1 (1000 nm)	0.2 (400 nm)	0.08 (200 nm)	0.3 (870 nm)

internal stress

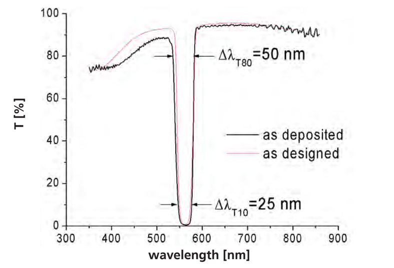
	SiO_2	Si_3N_4	Ta_2O_5
σ [MPa]	-340 … -30	-1000 … -50	-360 … -50

Internal stress can be adjusted using process parameters

Single and multi band rugate filters

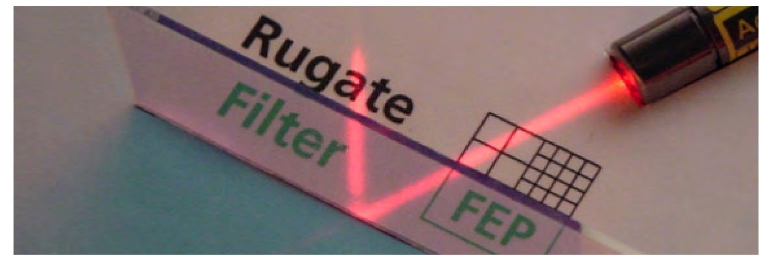


deposition of SiO_xN_y rugate filters
- rugate filter with a gaussian apodization function
- variation of the refractive index between 1.5 and 1.7
- time controlled variation of the refractive index by changing the reactive gas composition
- deposition time for a total thickness of 14 µm: 2 hours



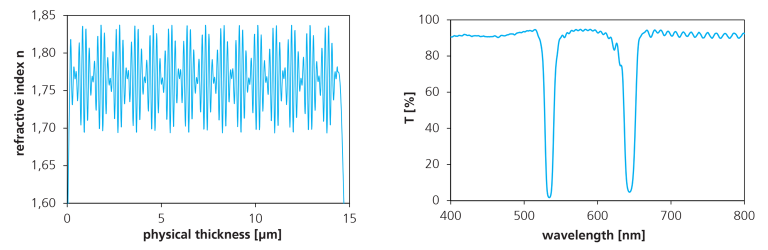
transmission of SiO_xN_y rugate filter

- narrow stop band width: T < 10 %: 25 nm; T < 80 %: 50 nm
- transition from rugate filter to ambient medium by equivalent layers (not imaged in design graph)
- transmission at 500 nm is higher than 85 % (backside-reflection of the glass substrate included); consistent with extinction of 2.5×10^{-4}



- SiO_xN_y rugate filter with transmission-stopband at 650 nm used as laser mirror
- application: laser optics, fluorescence and raman spectroscopy

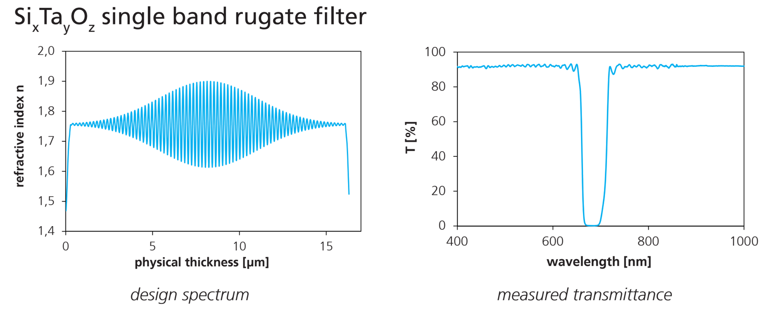
$Si_xTa_yO_z$ multi band rugate filter



design spectrum — measured transmittance

- multiband design by superposition of two sine wave frequencies

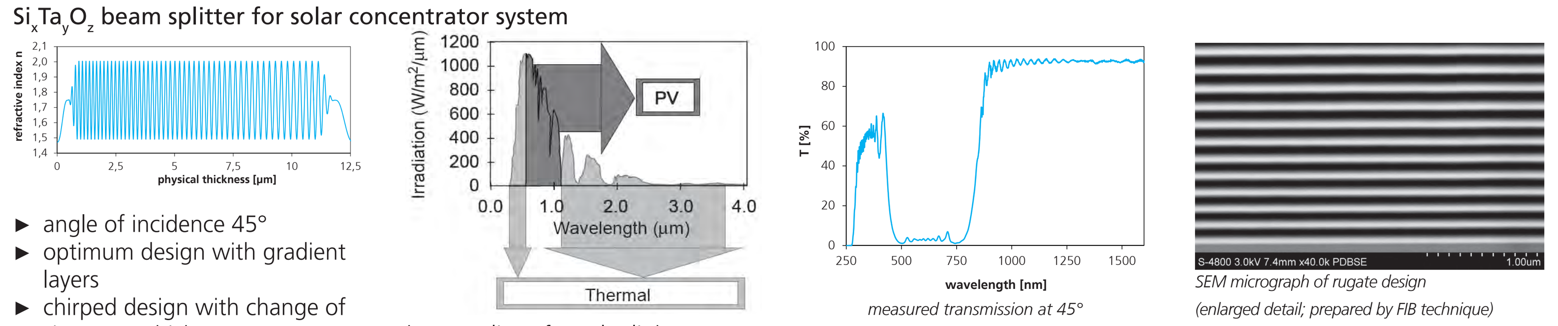
$Si_xTa_yO_z$ single band rugate filter



design spectrum — measured transmittance

- no absorption in the VIS
- refractive index profile of single-band rugate design (apodized and with matching transition to ambient media)

$Si_xTa_yO_z$ beam splitter for solar concentrator system



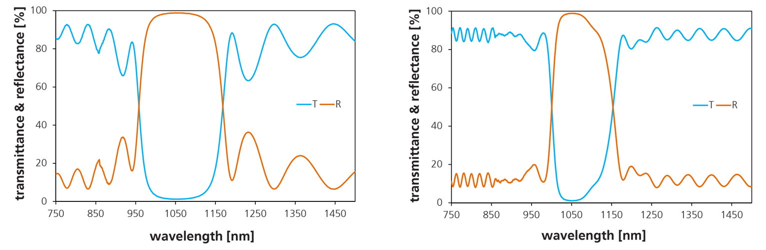
- angle of incidence 45°
- optimum design with gradient layers
- chirped design with change of sine wave thicknesses

beam splitter for solar light:
⟶ ultraviolet and infrared for solar thermal energy generation
⟶ VIS for photovoltaic energy generation

measured transmission at 45°

SEM micrograph of rugate design (enlarged detail, prepared by FIB technique) smooth and homogeneous film structure

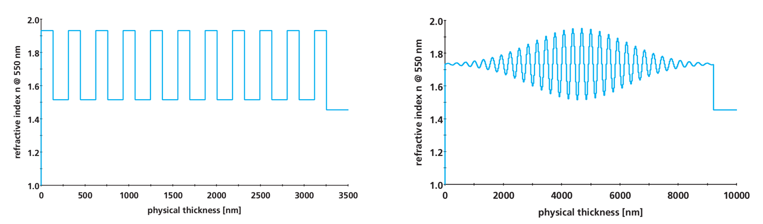
Comparison of laser induced demage threshold (LIDT) of HR 1064 nm $Si_xHf_yO_z$ rugate and SiO_2/HfO_2 quarterwave mirrors



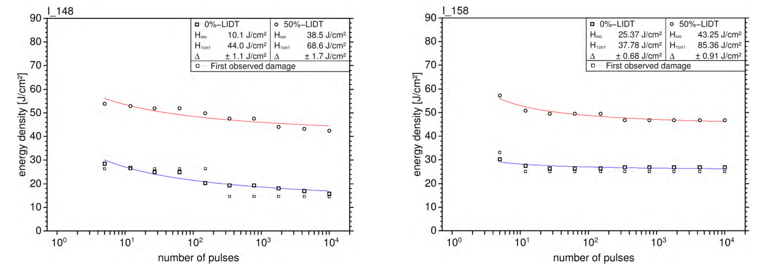
quarterwave design — rugate design

optical measured transmission and reflection of the HR 1064 nm filter

- LIDT measurements at Laser Zentrum Hannover (LZH) by H. Maedebach
- laser beam parameters: wavelength: 1064 nm, effective beam diameter: 226 µm, pulsed @ 100 Hz, pulse duration: 11 ns
- typical literature values of LIDT at 1064 nm with ion beam sputtering: quarterwave design 12 … 25 J/cm² (8 ns); Günster, LZH (conference V2011, Dresden): rugate-design about 100 J/cm²



quarterwave design — rugate design



quarterwave design — rugate design

- classical quarterwave design with discrete SiO_2 and HfO_2 layer
- rugate filter with apodized sinewave oscillation of refractive index ($Si_xHf_yO_z$)
- nearly identical optical function (transmission in blocking range <1 %)

- LIDT (0 %) of Fraunhofer FEP's HR 1064 nm mirrors: quarterwave 16 J/cm², rugate 27 J/cm²
- LIDT with rugate design about 70 % higher than quarterwave design
  ⟶ potential for significant inhancement of LIDT
- deposition rate using Fraunhofer FEP technology about 10 times of ion beam sputtering

SUMMARY

Reactive Pulse Magnetron Sputtering allows the deposition of dense, climatic stable films with low absorption and good integration of the film deposition into the complete production process of an optical component. In stationary mode there is the possibility of depositing coatings with freely defined refractive index by reactive sputtering of a silicon target in a mixture of oxygen and nitrogen gas. The new in-line sputter coater concept for precision optical coatings is the entry into scaling up the precision optic coating technology for higher throughput, larger substrates and significant cost reduction.

Summary: deposition in stationary mode

good adhesion on plastic substrates; low thermal substrate load increased LIDT with rugate compared to quarterwave designs	
applications	⟶ antireflection coatings ⟶ optical filter (Rugate, dichroic, cut, …) ⟶ coatings for UV: Al_2O_3, fluorides
high productivity due to	⟶ high deposition rate ⟶ uniform coating of substrates up to Ø 200 mm ⟶ deposition of complete filter at one coating station in one step
moderate investment cost due to compact equipment	
overall coating cost (calculated incl. deduction) for plant with one DRM 400 station:	⟶ double side AR coating of ophthalmic lens: ≈ 5 €/lens ⟶ single side coating of edge filter with 25 mm Ø (6 µm): ≈ 15 €/filter

Summary: deposition in in-line mode

PreSensLine: Multilayer and ultra-thin layer with accuracy < 1 nm	
applications:	⟶ optical precision components from UV to NIR, e.g.: cut, narrow band, DWDM, gain flattening filter, laser optical components also for high energy lasers, Bragg mirror for laser fluorescence analysis, Zero-reflection coatings (broad band antireflective coatings with neutral color), ⟶ EUV-, X-ray- and neutron mirror
PreSensLine concept is designated to scale up for mass production of precision optical elements and for precision coating of large substrates	⟶ high productivity, low coating cost
overall coating cost (calculated incl. deduction) for pilot plant with 800 mm target length:	⟶ single side coating of edge filter with 25 mm Ø (6 µm): ≈ 8 €/filter ⟶ DWDM with 1.4 mm Ø (200 layers, 32 µm): ≈ 10 €/filter

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