Assessing critical imperfections of high-end optical coatings

Sven Schröder
Optical systems department
Optical coatings department
Fraunhofer Institute for Applied Optics and Precision Engineering (IOF)
Jena, Germany

sven.schroeder@iof.fraunhofer.de
Fraunhofer Institute IOF: Development of optical systems

Ultra-precision machining

Optical and functional coatings

Optical and functional coatings

Measurement and modeling: Roughness, scattering, functional properties

Micro- and nano structuring
## Types of imperfections in coatings

<table>
<thead>
<tr>
<th>Type of Defect</th>
<th>Causes</th>
</tr>
</thead>
<tbody>
<tr>
<td>Interface roughness</td>
<td>Substrate roughness, material, polishing process, intrinsic film roughness, deposition process, adatom mobility / growth process, ...</td>
</tr>
<tr>
<td>Subsurface defects</td>
<td>Polishing process, materials, Bilby layer, cleaning, ...</td>
</tr>
<tr>
<td>Surface defects</td>
<td>Scratch, dig, particle contaminations, cleaning, handling, pre-treatment, ...</td>
</tr>
<tr>
<td>Coating defects</td>
<td>Deposition process, machine type, maintenance, cleanliness, ...</td>
</tr>
</tbody>
</table>

+ molecular contaminations, stoichiometric defects, bulk inhomogeneities, ...

**Most imperfections can be assessed by light scattering methods**
Impact of imperfections onto performance

Impact of imperfections onto optical and functional properties depends on:
- Density and size of imperfections
- Dielectric properties of imperfections
- Position in film system
- Local field strength / interference conditions

Light scattering can be made sensitive to all these factors

Yet, detecting imperfections does not necessarily allow conclusions about their impact onto certain performance parameters

→ Comprehensive pool of characterization methods and expertise
Characterization methods

- **Optical properties**
  - Spectral reflectance, transmittance
  - Optical losses: CRD, absorption, light scattering
  - LIDT: ns, fs, spectral

- **Structural properties**
  - **Roughness**: AFM, WLI, light scattering
  - Morphology: SEM, TEM, ...
  - Molecular / atomar: XPS, TOF-SIMS, ...

- **Functional properties**
  - Scratch resistance / tribology
  - Environmental stability
  - Thermal stability
  - Contact angle analysis

+ Understanding to link optical, structural, and functional properties

**Strong expertise in some fields and cooperation with partners for other methods.**
Light scattering ARS vs TS

Angle Resolved Scattering (ASTM E2387, ISO/WD 19986)

\[ ARS(\theta_s) = \frac{\Delta P_s(\theta_s)}{\Delta \Omega_s P_i} = \text{BSDF} \cos \theta_s \]

\( \theta_s \) ... polar scatter angle
\( P_s \) ... scattered power
\( P_i \) ... incident power
\( \Delta \Omega_s \) ... detector solid angle

Total Scattering (ISO 13696)

\[ TS = \frac{P_s}{P_i} = \int ARS \, d\Omega_s \]
(ISO: 2° - 85°)

→ Goniometric scatterometers
  + more flexibility
  + more information
  - more challenging

→ Coblentz-/integrating spheres
  + fast
  + single number
  - limited information
  - only small and plane samples

\( \theta_s \) ... polar scatter angle
\( P_s \) ... scattered power
\( P_i \) ... incident power
\( \Delta \Omega_s \) ... detector solid angle

BSDF... bidirectional scattering distribution function

Surface roughness

Surface Power Spectral Density function

\[ PSD(f_x, f_y) = \lim_{L \to \infty} \frac{1}{L^2} |FT\{z(x, y)\}|^2 \]

- Power of different roughness components
- Fourier Transform of Autocovariance Function
- Isotropic roughness: PSD(f_x, f_y) \to PSD(f)

Rms roughness

\[ \sigma = \sqrt{2\pi \int_{f_{min}}^{f_{max}} PSD(f) \cdot f \, df} \]

- Standard deviation of surface topography
- Band-limited / relevant roughness

Roughness evolution models: Qualitative model for thin film growth

Physical vapor deposition process

- Replication of low-frequency roughness (substrate)
- Roughening at higher spatial frequencies (shot noise)
- Smoothing at cut-off (surface diffusion)
- Analytic models involving particle size, energy, diffusion length

→ Coating roughness = substrate roughness + intrinsic thin film roughness

Example: Aluminum coating on superpolished fused silica

Which part of the roughness spectrum is relevant for the application?
Relationship light scattering – roughness (single surfaces)

\[
ARS(\theta_s) = \frac{16\pi^2}{\lambda^4} \cos \theta_i \cos^2 \theta_s \ Q \ PSD(f)
\]

- Optical factor
  - optical constants
  - geometry
  - polarization

- Roughness factor
  = surface PSD

→ For a given wavelength and geometry, scattering into a certain direction corresponds to the power of a corresponding roughness component with spatial frequency \( f \).

Scattering from thin film coatings

Vector perturbation theory ($\sigma << \lambda$)

\[ ARS(\theta_s) \sim \frac{1}{\lambda^4} \sum_{i=0}^{N} \sum_{j=0}^{N} F_i F_j^* \text{PSD}_{ij}(f) \]

**Optical factors**
- Multilayer design
- Optical constants
- Polarization

**Roughness factors**
- PSDs of individual surfaces
- Cross-correlation properties ($i \neq j$)

- Multilayer scatter influenced by roughness and interference effects
- $N^2$ parameters $\rightarrow$ roughness and correlation models required
- Verification through scatter measurements

System for spectral angle resolved scatter measurements

**MLS 1600**

- 3D Angle resolved light scattering (BSDF), transmittance, reflectance
- UV-VIS-NIR (193 nm – 2700 nm, bandwidth < 0.1 nm)
- High sensitivity (<10^{-6} sr^{-1}), high angular resolution (<0.1°) large samples (up to 700 mm)
- Applications: surfaces, coatings, filters, gratings, materials, ...

Instruments for angle resolved scatter measurements developed at Fraunhofer IOF

Laboratory scatterometers (EUV-DUV-VIS-IR) → Compact tools → Customized scatterometers developed for partners

- **Big UV-VIS-IR system** (325 nm – 10.6 μm)
- **TS** (193 nm – 10.6 μm)
- **Table-top 3D ARS system (VIS-NIR)**
- **Portable scatter sensor (VIS)**
- **New IR scatterometer (in-plane)** 532 nm, 4.6 μm, 10.6 μm, motorized sample stage

→ Scatterometer development based on R&D on high-end optics

Surface roughness and defects
Telescope mirror
Diamond turned and polished Al

 Scatter map 532 nm
(200x160 mm², fixed scatter angle of 25°)

\[ \sigma = 1.9 \text{ nm} \]

\[ \sigma = 3.0 \text{ nm} \]

\[ \theta_s (\degree) \]

Local BRDF

Full area characterization of BRDF, homogeneity, roughness, defects

Further development to characterize EUV mirrors before coating...

Characterization of EUV mirrors

→ Unique / superior technique for roughness characterization of high-end optical surfaces

Robotic scatter sensor

- Automatic mapping of freeform surfaces based on CAD data
- Fast and robust CMOS-based scatter sensor for rapid BRDF imaging
- Roughness measurement and defect classification from scatter data

→ Full area inspection of optical surfaces

Roughness vs. defects

Defect-induced scatter does depend on beam size

Example: Scatter map of Al mirror at 650 nm

Prediction of scattering at application beam diameter $D$ from scattering measured at smaller beam diameters $d$ (single defect):

$$ARS(D) = ARS_r(d) + ARS_d(d) \left( \frac{d}{D} \right)^2$$
Thin film coatings
HR coating at 193 nm

HR 193 nm, (AlF₃/LaF₃)²₀ on fused silica (thermal boat evaporation)

Surface roughness
(AFM 1x1 μm²)

- Coating: \( \sigma = 2.2 \text{ nm} \)
- Uncoated substrate: \( \sigma = 0.2 \text{ nm} \)

ARS measurement and initial modeling

TS increases from 0.11% (substrate) to 2.8% (coating)

Reasons: a) Increased \( \sigma \), b) enhanced \( R \), c) ???

HR coating at 193 nm - modeling

Simplified scatter model

\[ ARS(\theta_s) \sim \sum_{i=0}^{N} \sum_{j=0}^{N} F_i F_j^* \text{PSD}_{ij}(f) \]

\[ \beta \]

roughness evolution

\[ \sigma_i \sim i^\beta \]

→ Rapid roughening

Underlying interfaces are much smoother than top-surface

HR coating at 193 nm - modeling

Simplified scatter model

\[ \text{ARS}(\theta_s) \sim \sum_{i=0}^{N} \sum_{j=0}^{N} F_i F_j^* \text{PSD}_{ij}(f) \]

Variation of \( \delta \)

\[ \delta \]
thickness deviations

\[ OT' = (1 + \delta)OT \]

\( \rightarrow \) Enhanced scatter caused by film thickness deviations

\( \rightarrow \) Indicates enhanced fields, linked to laser stability


Light scattering depends on roughness and field distribution.

Spectral angle resolved scattering (modeling)

Spectral field distribution (modeling)

→ Resonant scatter indicates enhanced fields
→ Also correlated with laser stability
Spectro-Scat: Rugate filter (continuous index profile)
Sample: HR 532 nm Rugate, Fraunhofer FEP

→ Scatter loss increases from 0.1% at 532 nm to 2.2% at 505 nm!
→ Critical effect for spectral filters, spectral shifts, ...
New: Spectral laser-induced damage testing

- Implementation of procedure for laser-induced damage testing into OPO-based scatterometer
- Spectral LIDT testing (ISO 21254), 5 ns pulses, Spectral range: 192 nm - 2600 nm, Max. fluence: $\sim 10^3$ J/cm²
- On- and offline monitoring of structural and material properties using angle-resolved scattering (ARS)
- Combination of (pre-damage) scatter/defect mapping with damage testing

Examples of spectral LIDT measurements

Interference coating

- NIR edge filter (Tongji University, Shanghai)
- Strong variation of LIDT, not necessarily according to simple scaling laws
- LIDT closely related to field distribution and defect locations

Selective laser damage test

Scatter mapping (532 nm, AOI 0°, scatter angle 10°) before damage test

161 defect-free positions

S-on-1 damage test at 532 nm ($S = 3000, 20 \text{ Hz}, 5\text{ns}, 170 \mu\text{m}$)

→ 33% lower LIDT on defect locations

→ Additional influence caused by nanodefects not detected by light scattering

Sample: HR 1064 nm + 532 nm (Optics Balzers Jena)

78 defect positions
Loss analysis using scatterometry

Angle Resolved Scatter measured at 640 nm of HR coating designed for 630 nm - 640 nm

\[ 1 = R + T + TS_b + TS_f + A \]

\[
\begin{align*}
R &= 0.99\ldots \pm 0.006 \\
T &= (1.5\pm0.1) \cdot 10^{-6} \\
TS_b &= (5.9\pm0.6) \cdot 10^{-6} \\
TS_f &= (5.6\pm0.6) \cdot 10^{-7}
\end{align*}
\]

→ Less layers for higher R!

A known → R with ppm precision

R known → A with ppm precision (at low fluence)
Summary

- Manufacturing of high-end optical coatings requires controlling imperfections (roughness, substrate and coating defects, ...)
- Collaboration with partners and own expertise
- Angle resolved light scattering to characterize imperfections:
  - Of substrates before coating (roughness, defects, contaminations, ...)
  - Of coatings (roughness evolution, thickness errors, ...)
  - Of Materials (inhomogeneities, defects, ...)
- Significant impact of defects onto LIDT shown by selective laser damage testing using combination of scattering and damage test
- Highly sensitive loss analysis using angle resolved scatterometry
Thanks to colleagues at IOF: Marcus Trost, Matthias Hauptvogel, Tobias Herffurth, Alexander von Finck, Méabh Garrick, Luisa Coriand, Nadja Felde, Angela Duparré, Andy Tänzer, Ronald Schmidt and mechanical workshop, Thomas Ganz, Beate Wendt, ...

As well as partners: Optixfab, Opcrown, Lenstec, Optics Balzers Jena, Lasos, IPHT, LZH, ...

Financial support: EU / TAB project SpectroScat, BMBF ZIM project LOSASS

Thank you!
Merci – Grazie – Danke ;)

Fraunhofer IOF