# Hyperspectral Imaging for in-line thin film characterization in large area roll to roll processing

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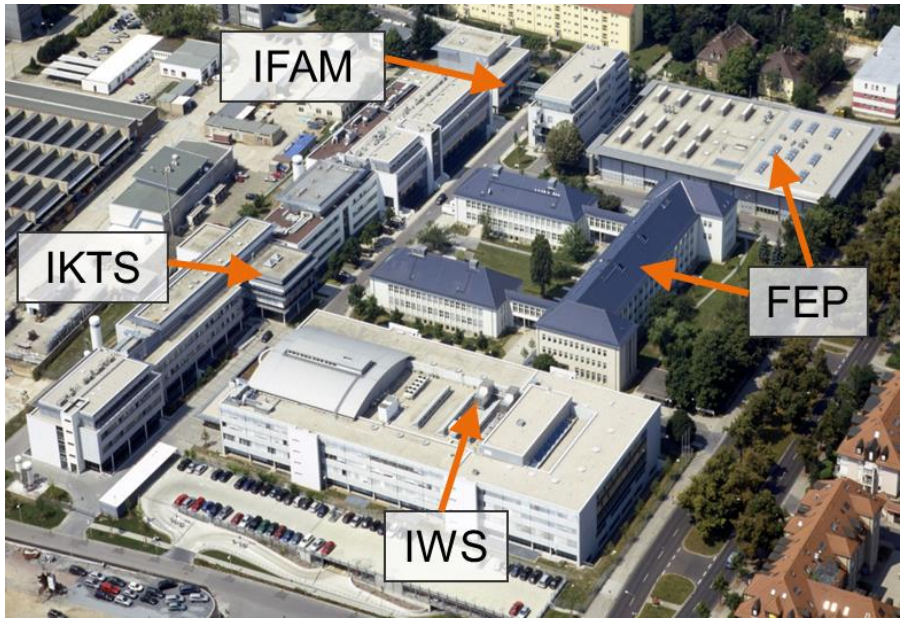


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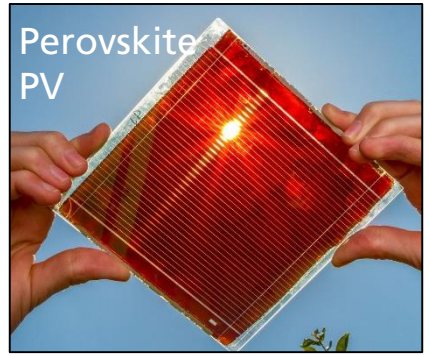
# The challenge

- **We need** precise *(< ± 1%)* control of
  - Thin film thickness
  - chemical composition
  - Solid-state phase / crystallinity
  - Surface roughness / morphology

  in thin film processing on large area in multilayer stacks

- **We miss** *fast, efficient and accurate* methods to measure
  - thickness of ultrathin transparent layers (≤ 100 nm)
  - individual layer properties in multilayers
  - „in-situ“ access to solid state phase
  - access to nano-roughness / density
  - inline access to functional properties


source: EControl-Glas, GmbH, Plauen

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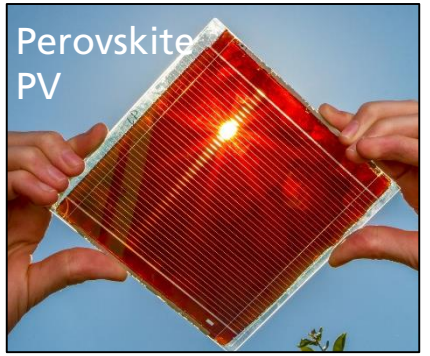
**Question:**
How can we map/image the (in)homogeneity of relevant thin film properties on large areas (up to 100m²) at high speed?

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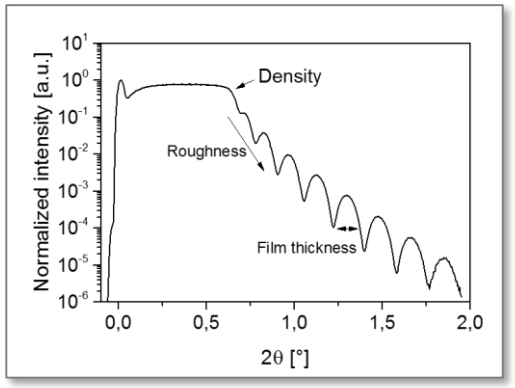
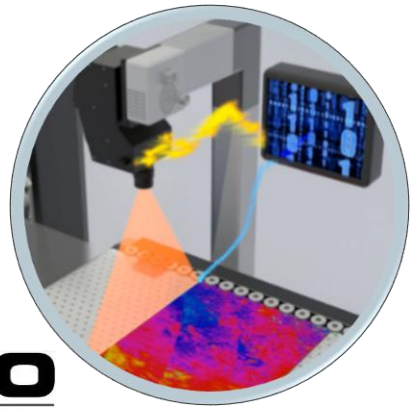
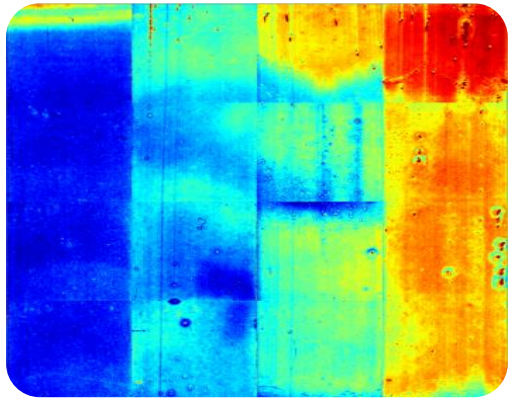
# NanoQI concept

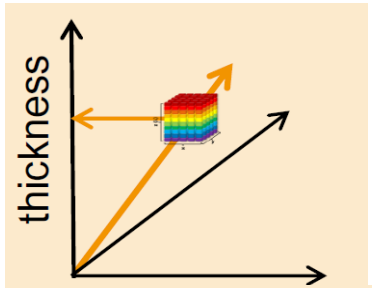
high-speed at-line / in-line
**XRR / XRD**



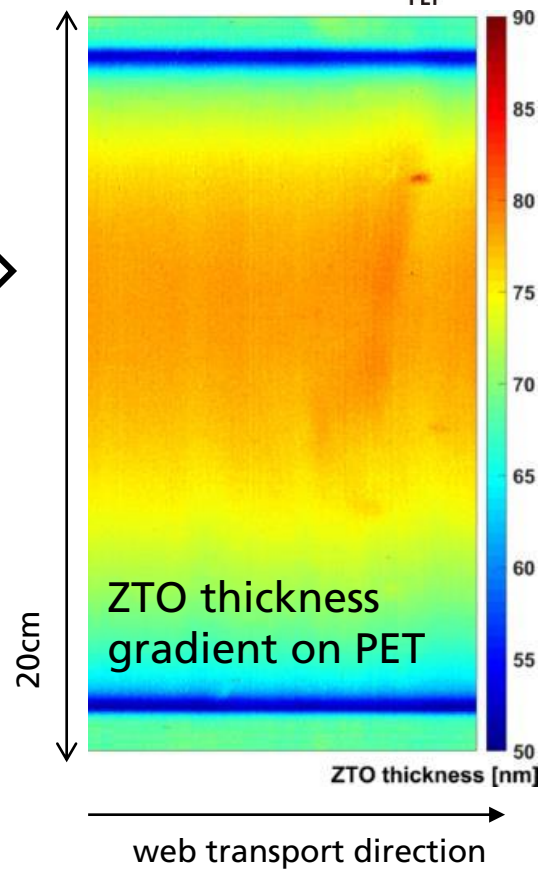
real-time in-line
**hyperspectral imaging**






**machine learning & modelling**

- fast large area surface inspection
- nano material quality control
- real-time process feedback



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# NanoQI use cases -> technology demonstration

Fraunhofer IAP

**Large area ALD**

- Barrier coatings
- Functional coatings

**S2S thermal curing and sintering**

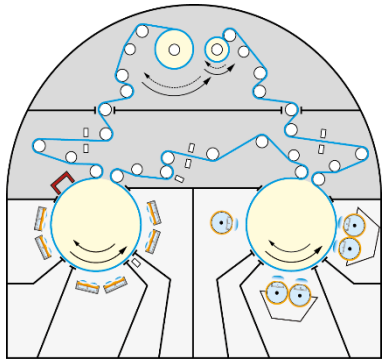
- perovskite solar cells

TNO innovation for life

SMIT THERMAL SOLUTIONS

nordmeccanica group

Fraunhofer FEP



**R2R vacuum web coating** (magnetron sputtering & evaporation)

- Barrier coatings, packaging
- Transparent electrodes
- Low-e coatings



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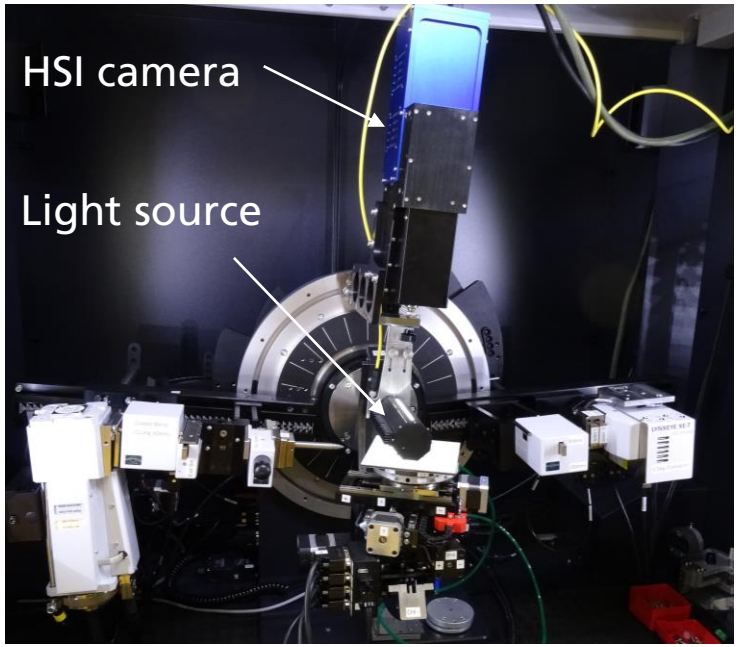
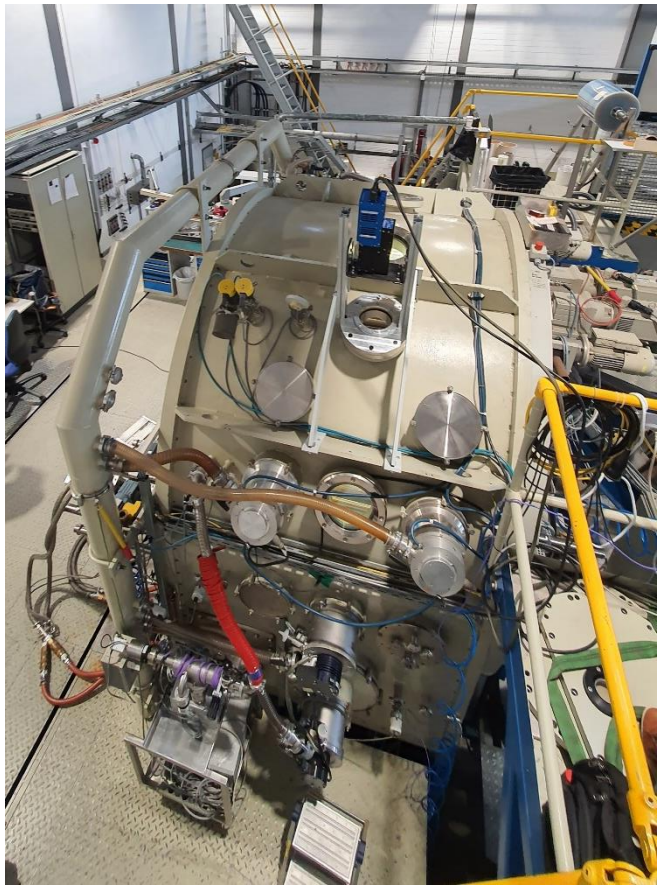
# Hyperspectral Imaging capabilities at FEP

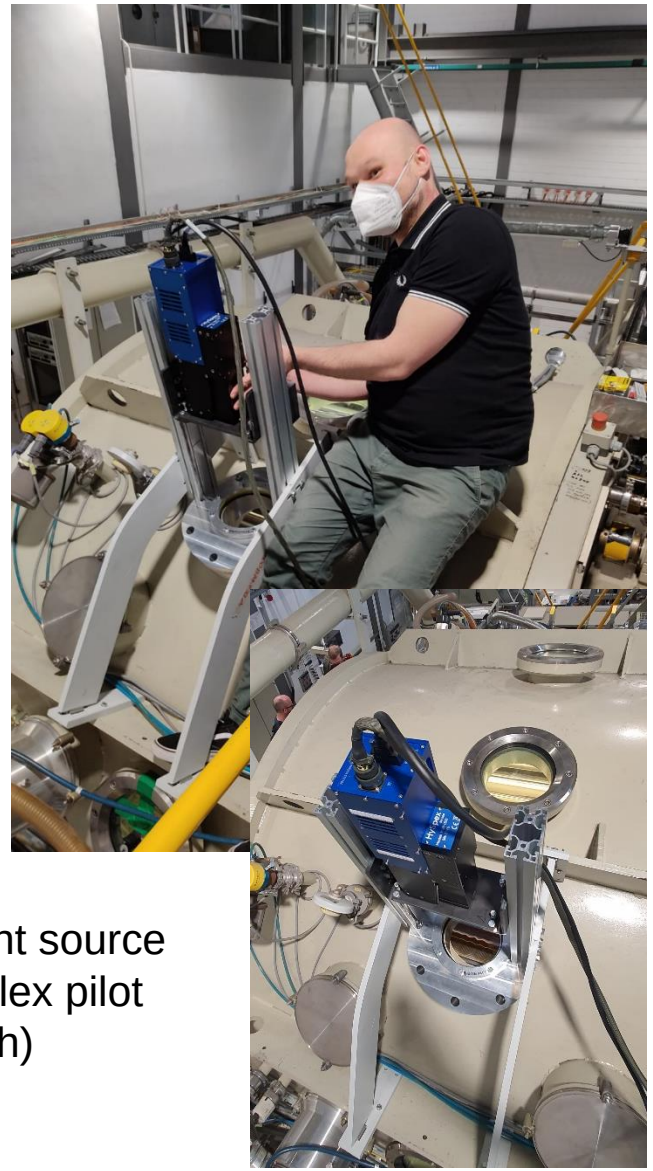



Multimodal combination of Bruker XRD/XRR D8 Discover diffractometer with NEO HSI camera

- at-line XRR measurements & data analysis
- HSI reference measurements up to 10x10cm² sheets

⇨ training of HSI ML models





Integration of diffuse broadband light source (IWS) & NEO HSI camera into CoFlex pilot web coater (up to 600mm web width)

- in-line HSI process monitoring



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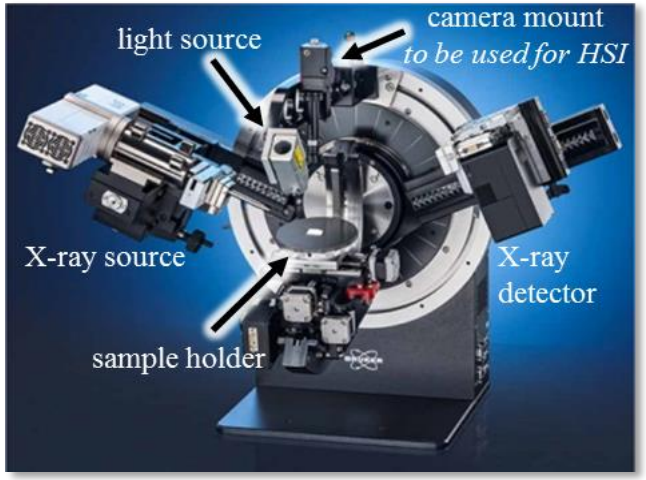
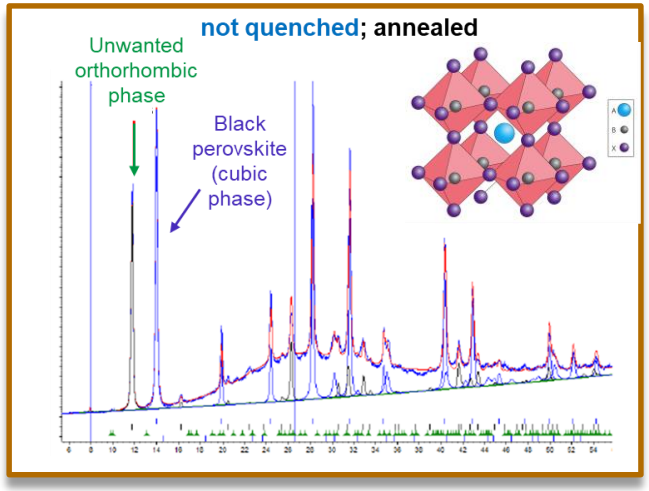
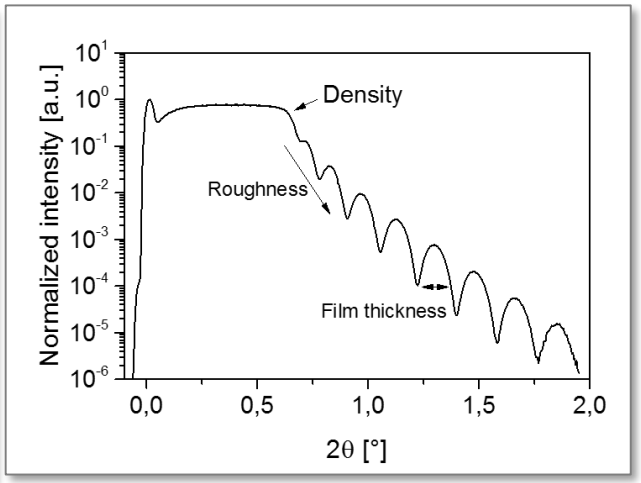
# NanoQI methods

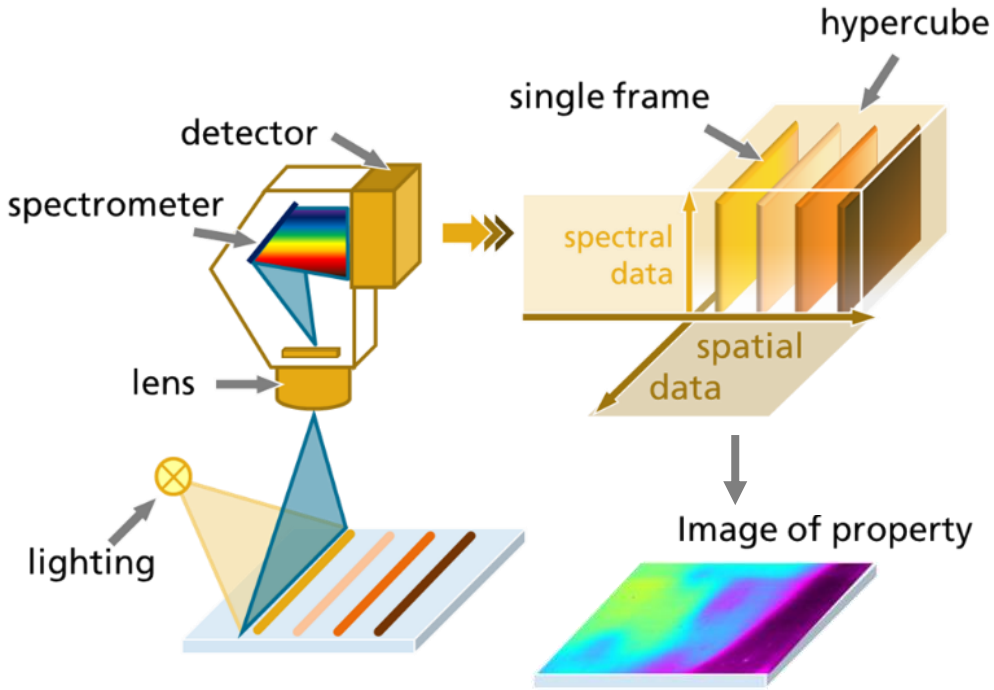



- **X-ray diffraction analysis**
  - Phase analysis
  - Chemical composition
- **X-ray reflectometry**
  - Thickness / Multilayers
  - Roughness
  - (electron) density




- High-speed & high accuracy semi-automated sample evaluation for quality control & HSI model training (calibration)




- **Hyperspectral Imaging**
  - 2D Spatially resolved optical transmission/ reflection spectrometry
  - Detect defects, gradients, property drifts
  - Large area imaging of functional properties
  - Inline integration to thin film processing



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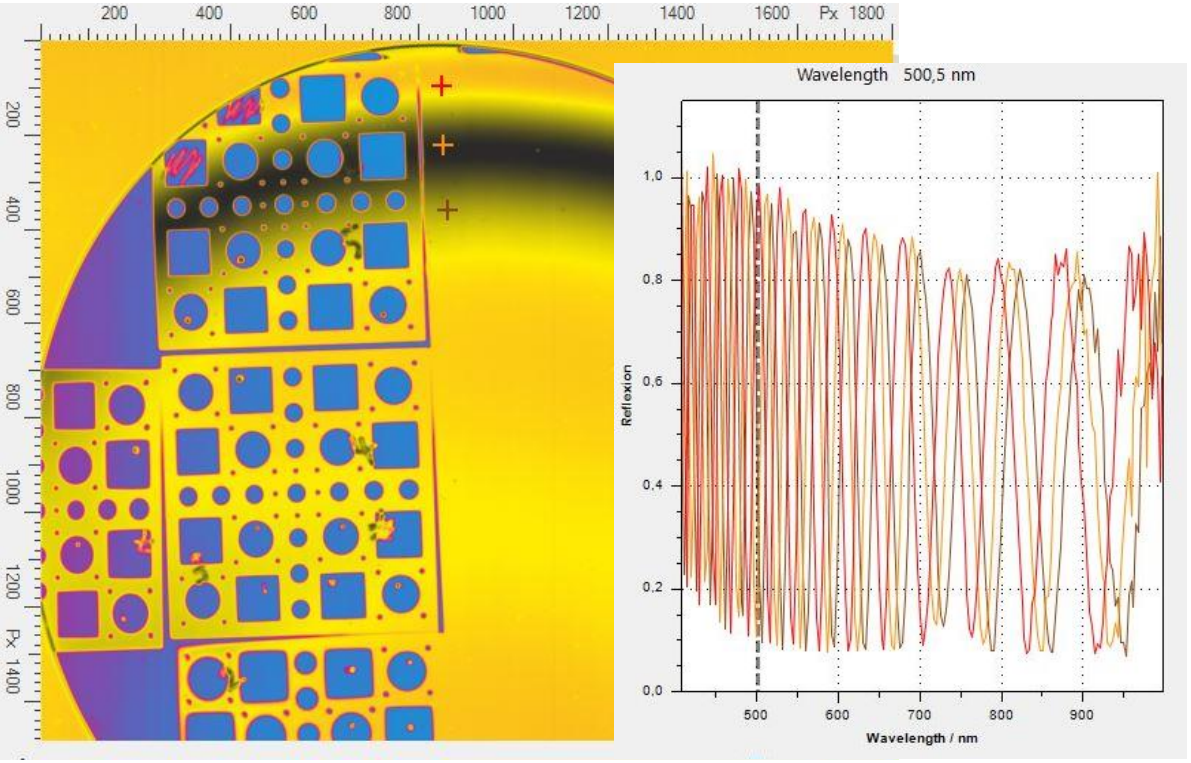
# HSI example: Thickness gradient modelling of AlN on Si wafer




HSI reflection „image“
@ 600nm

z-axis = intensity (raw data)

optical reflection spectra
at selected pixels:
interference pattern
-> thickness information



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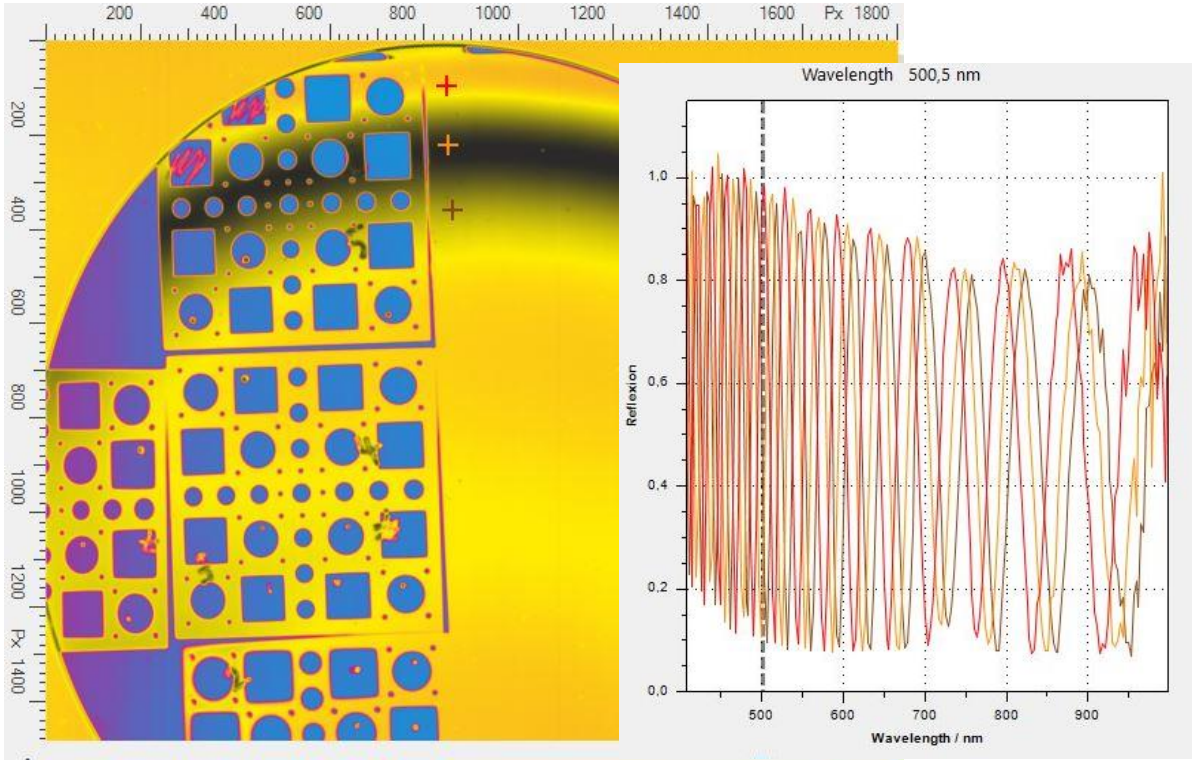



HSI reflection „image“
@ 600nm

z-axis = intensity (raw data)

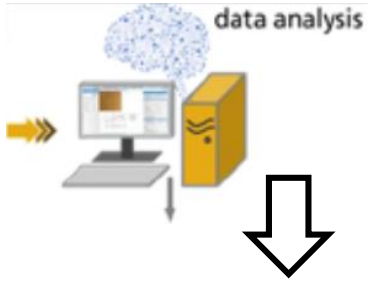
optical reflection spectra
at selected pixels:
interference pattern
-> thickness information

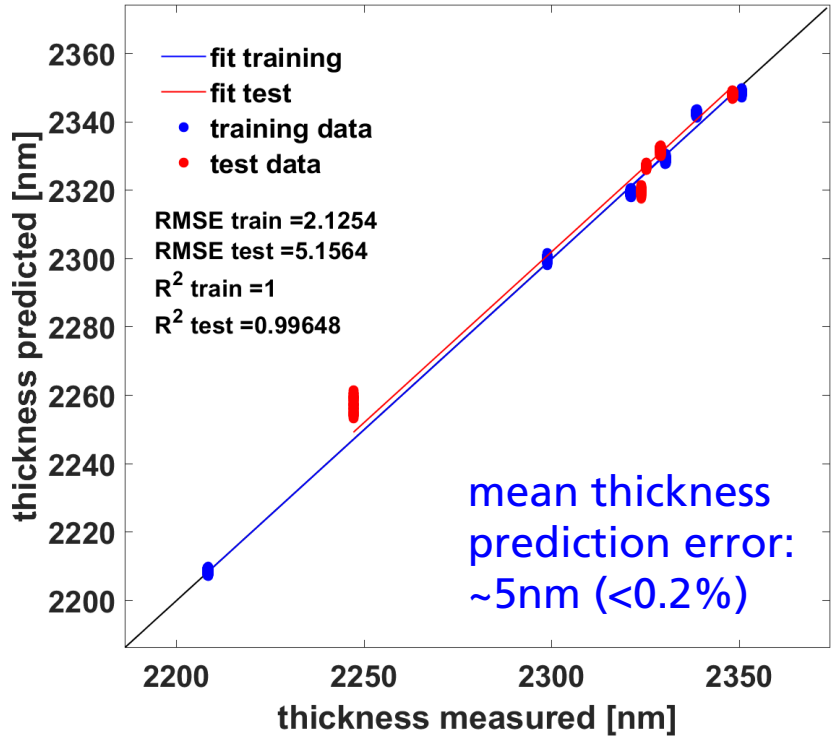
+ 10 thickness values from locations across wafer by external method (spectroscopic ellipsometry)

Vector normalization

Principle Component Analysis

ML model training & verification



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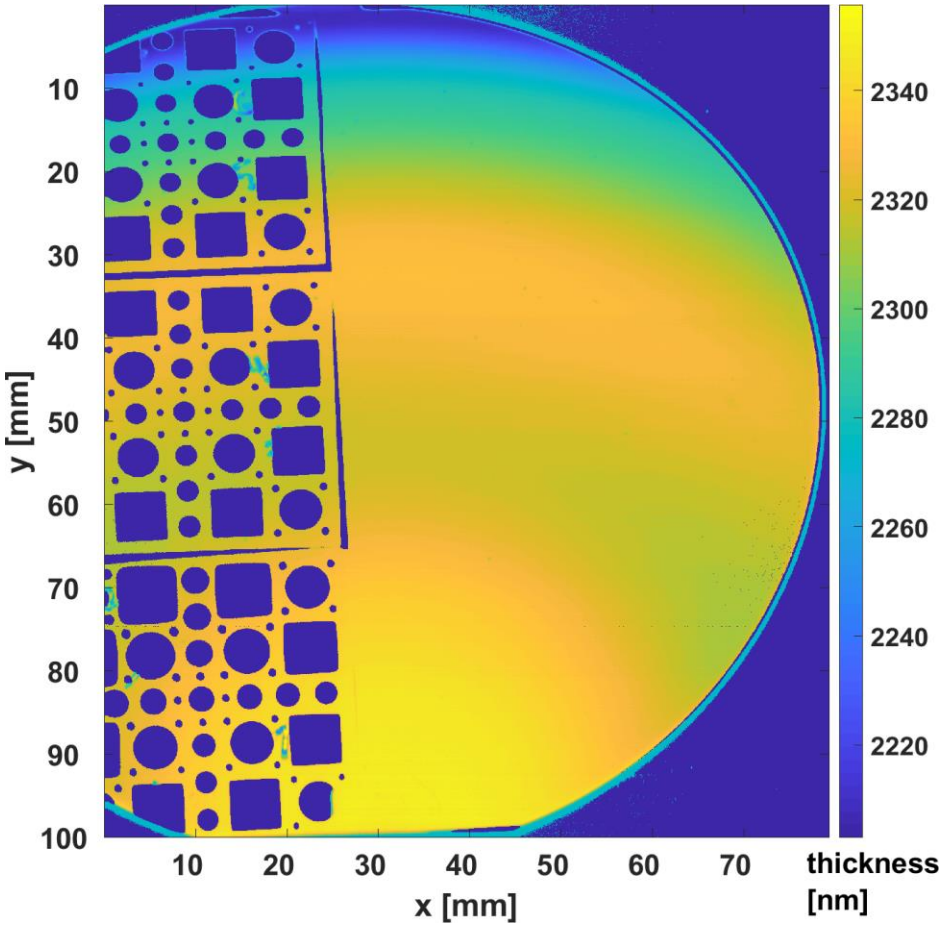


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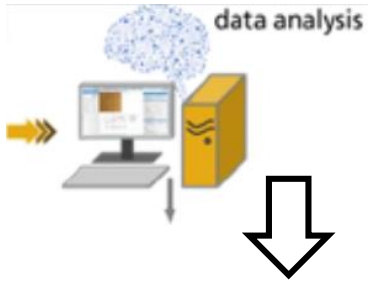
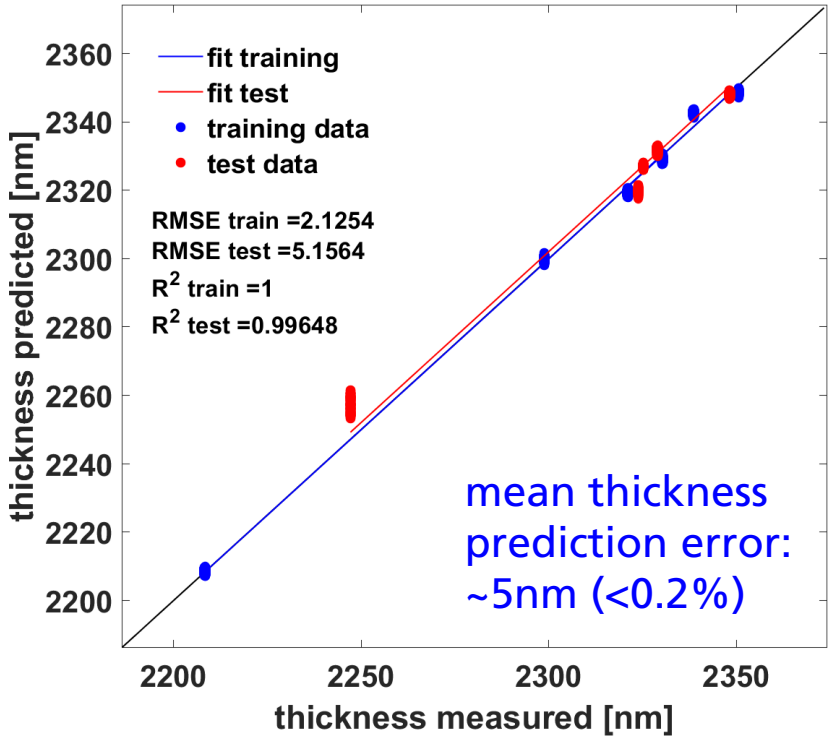



data transformation




Result

- thickness map of complete wafer with sub-mm spatial resolution
- HSI model can now be used for any unknown AlN/Si sample (without external data)



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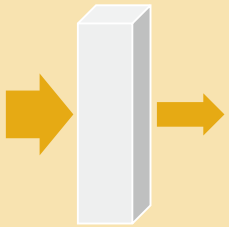
# Data evaluation options for HSI data

## *Hard modeling*

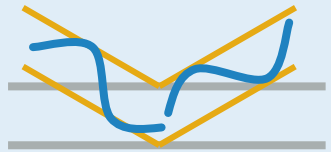
Physical description of received data set

→ Could be slow

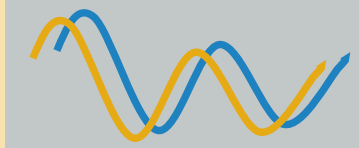
→ No external ground truth needed



Lambert-Beer's law
$E = \varepsilon * c * d$



Bragg's law
$n * \lambda = 2 * d * \sin \theta$



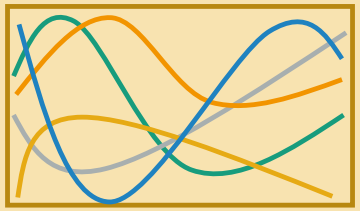
Dielectric function
$\varepsilon(\omega) = \varepsilon_\infty - [\varepsilon_0 \rho(\omega^2 \tau + i\omega)]^{-1}$

## *Soft modeling*

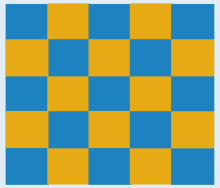
Pattern recognition by means of un-/supervised data evaluation algorithms

→ Could be fast

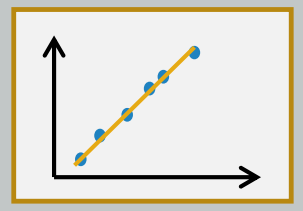
→ Prediction model must be trained



Spectral pattern recognition, chemometrics
(i. e. PCA, LDA, SIMCA)



graphical pattern recognition
(i. e. wavelet analysis)



multivariate correlation algorithms (i. e. PLS)



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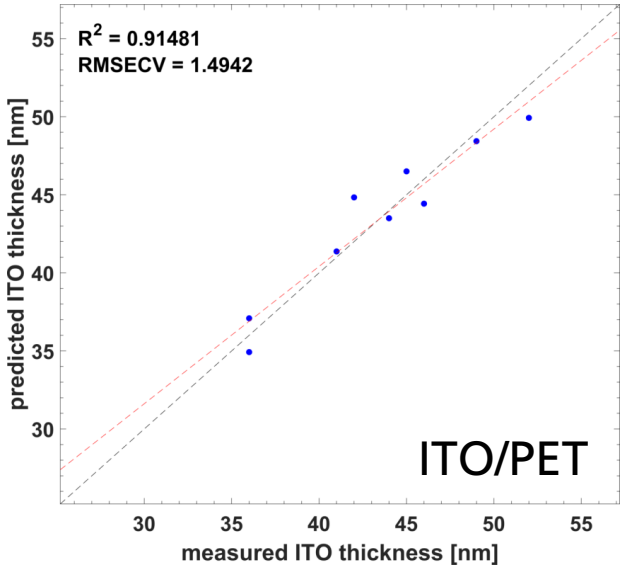
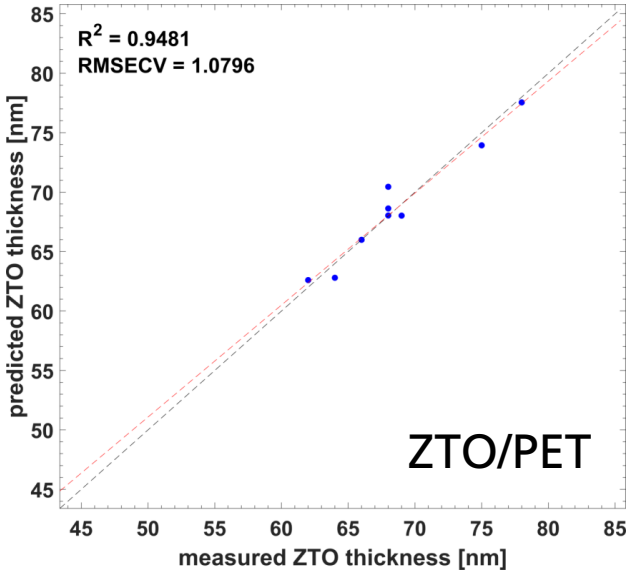
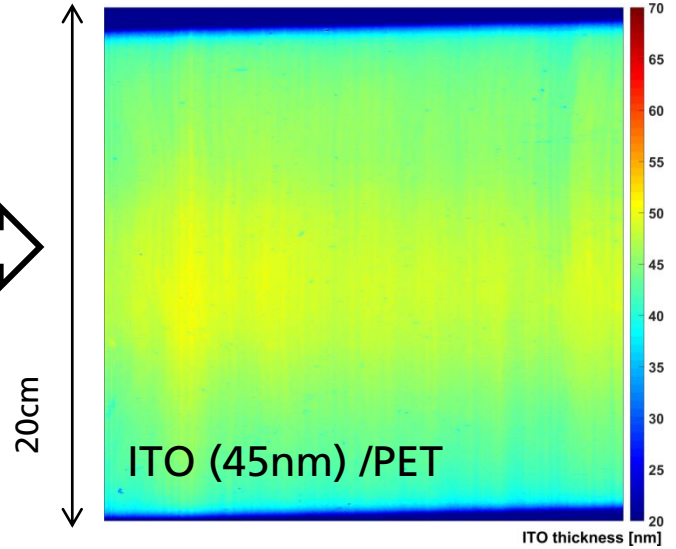
# OK, but what about HSI modelling of thin layers? -> ZTO/ITO layers on PET

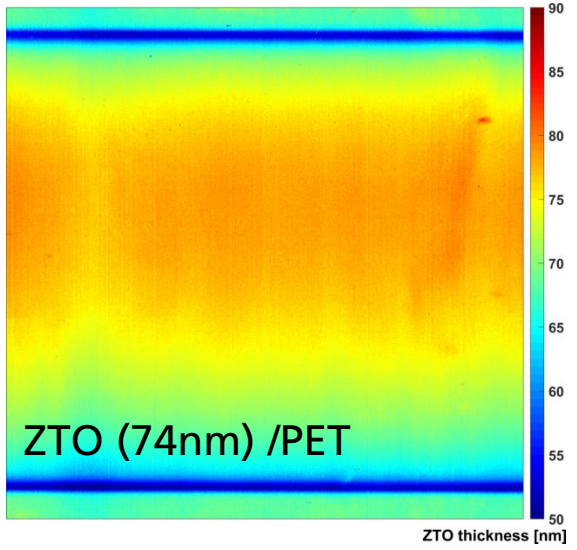
HSI raw data

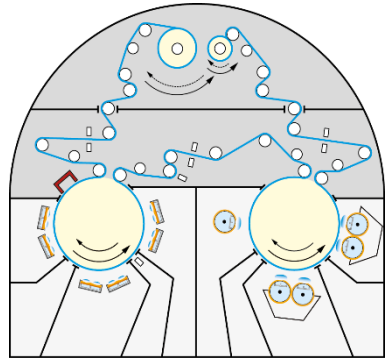
external reference thickness values (XRF)

web transport direction



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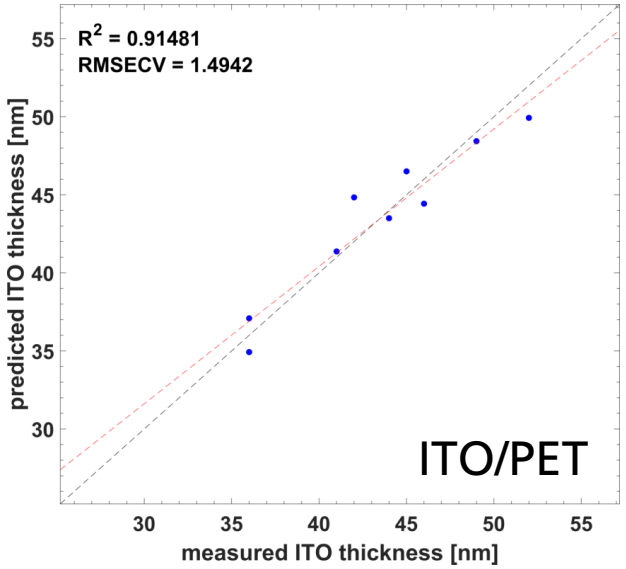
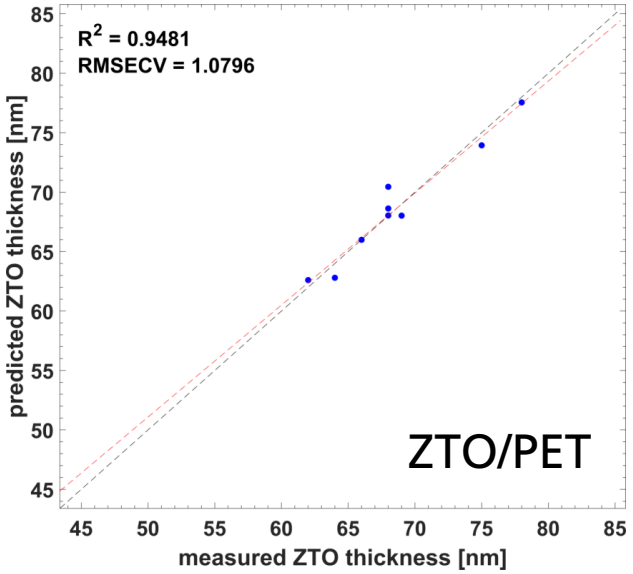
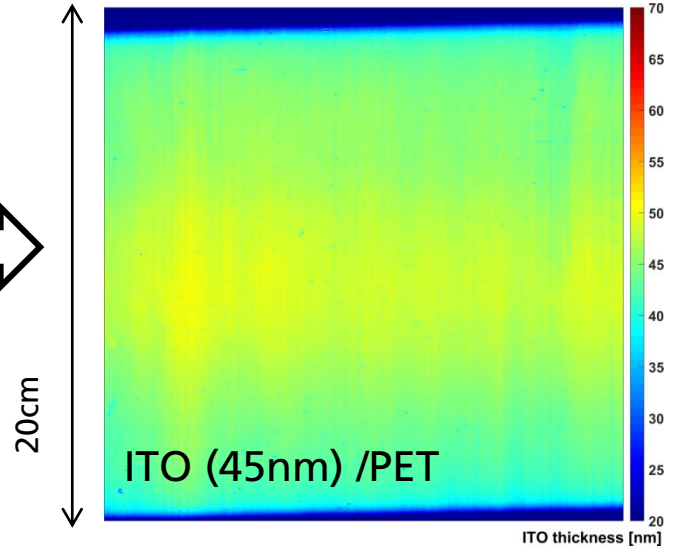
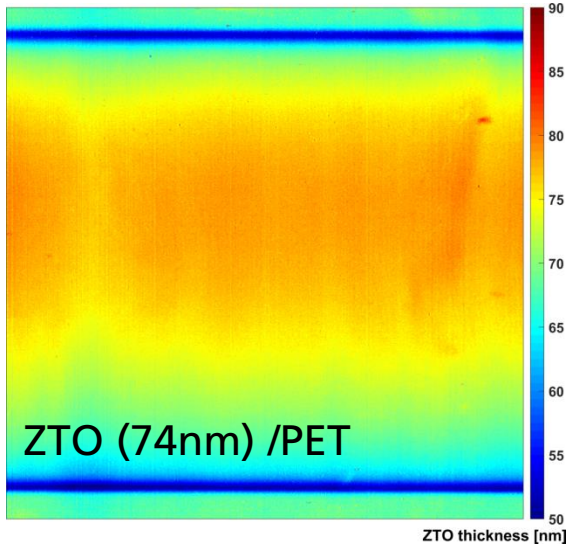
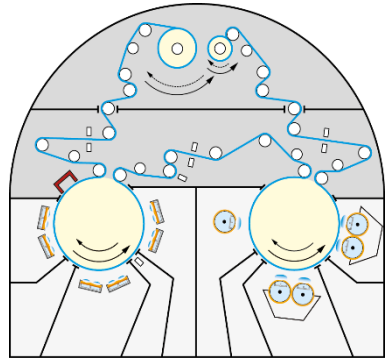


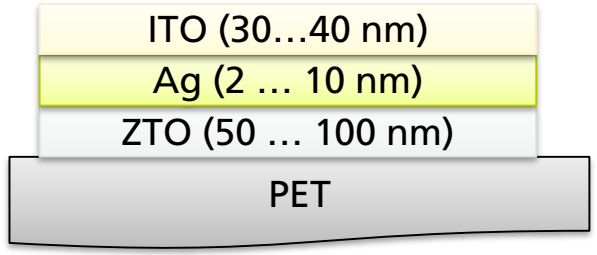



Goal:
multilayers on flexible substrates (IMI coatings, barriers)




web transport direction



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# In-line HSI at CoFlex web coater: performance parameters

**Configuration (stage 1)** (final stage)

- HSI camera working distance: 100 cm (~80 cm)
- HSI camera FoV angle 16° (40°)

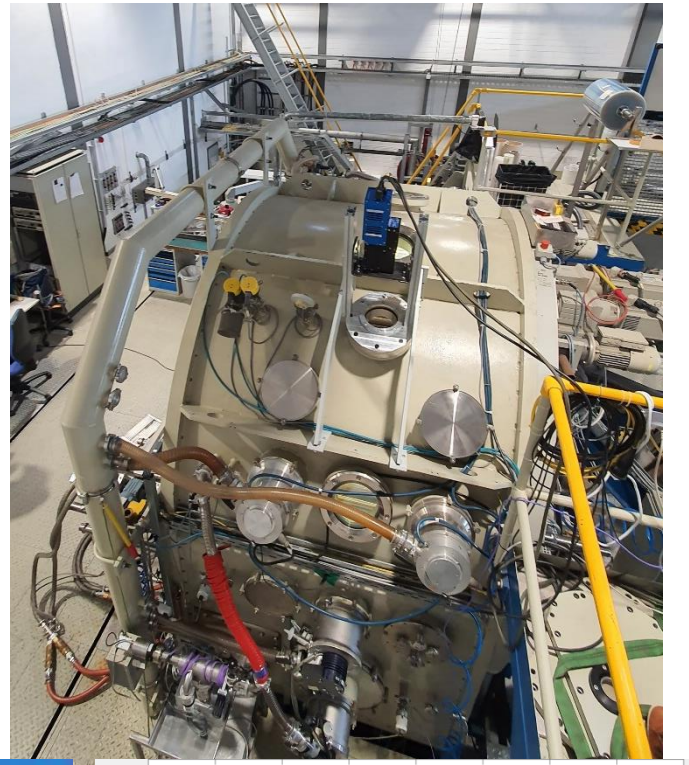
- Web speed* <1.5 m/min (~2.5 m/min)

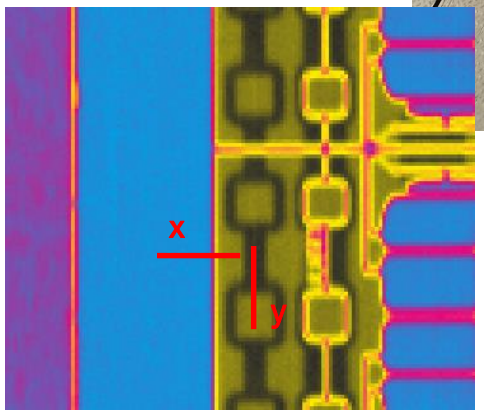
(at full HSI resolution, w/o distortions)

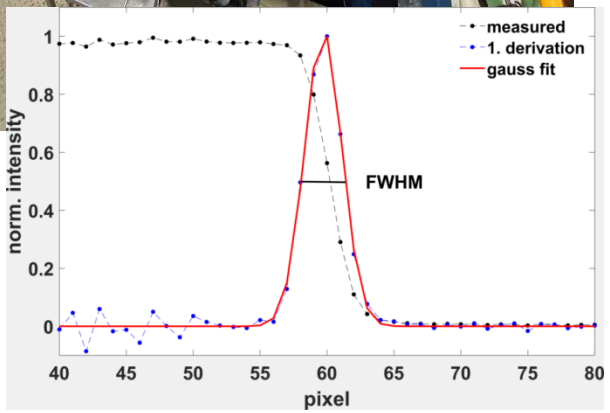
**Experimental parameters:**

| | | |
|---|---|---|
| Field of View (FoV): | 292 mm | (500mm) |
| Pixel size: | 0.162 mm/px | (~0.3mm/px) |
| Spatial Resolution | ~0.25 mm (x) | (~0.4mm) |
| (using FWHM of 1st derivative) | ~0.70 mm (y) | (~1.1mm) |

*) much higher web speed possible via
- accepting some image spatial distortion (in coating direction)
- intelligent „online“ HSI data reduction
- increasing HSI data bandwidth





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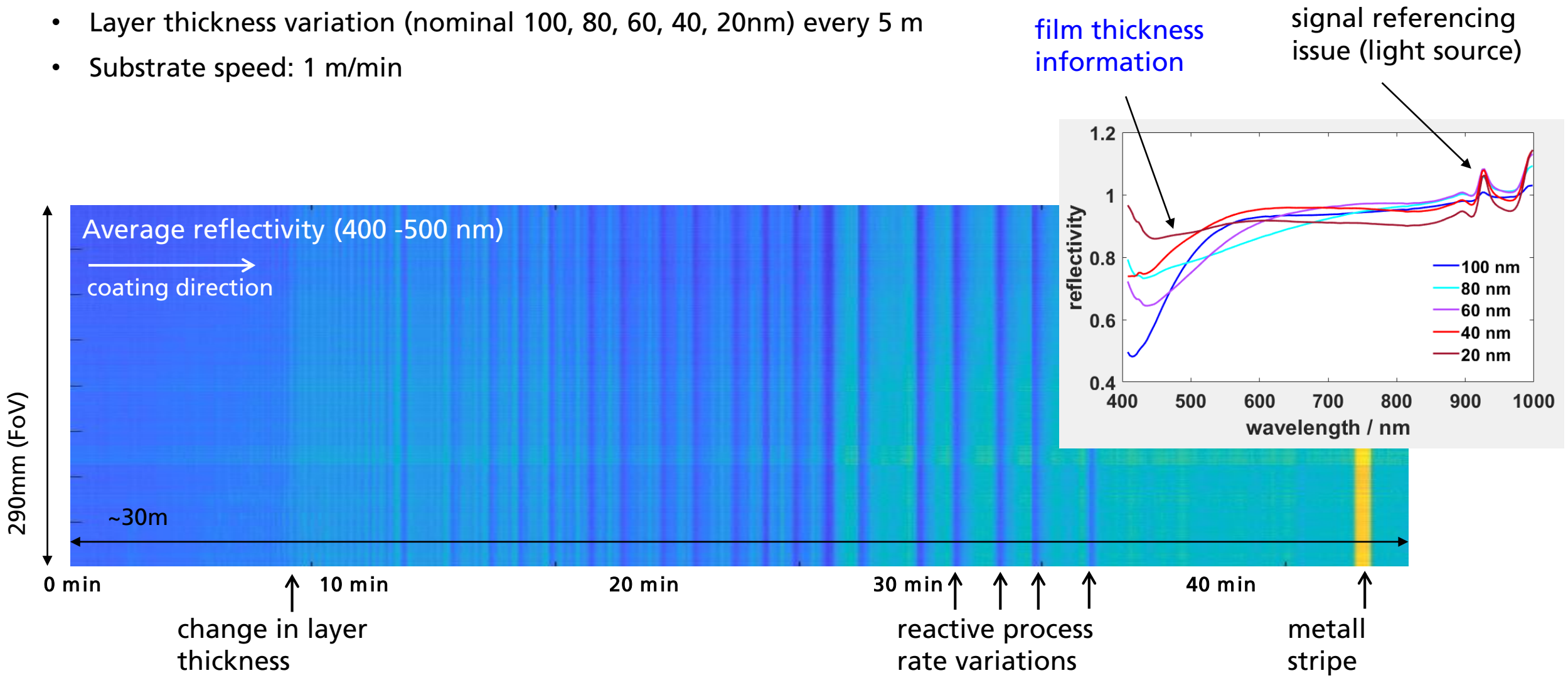
# In-line HSI process monitoring at CoFlex web coater: sputtered $SnO_2$ / PET

- Layer thickness variation (nominal 100, 80, 60, 40, 20nm) every 5 m
- Substrate speed: 1 m/min



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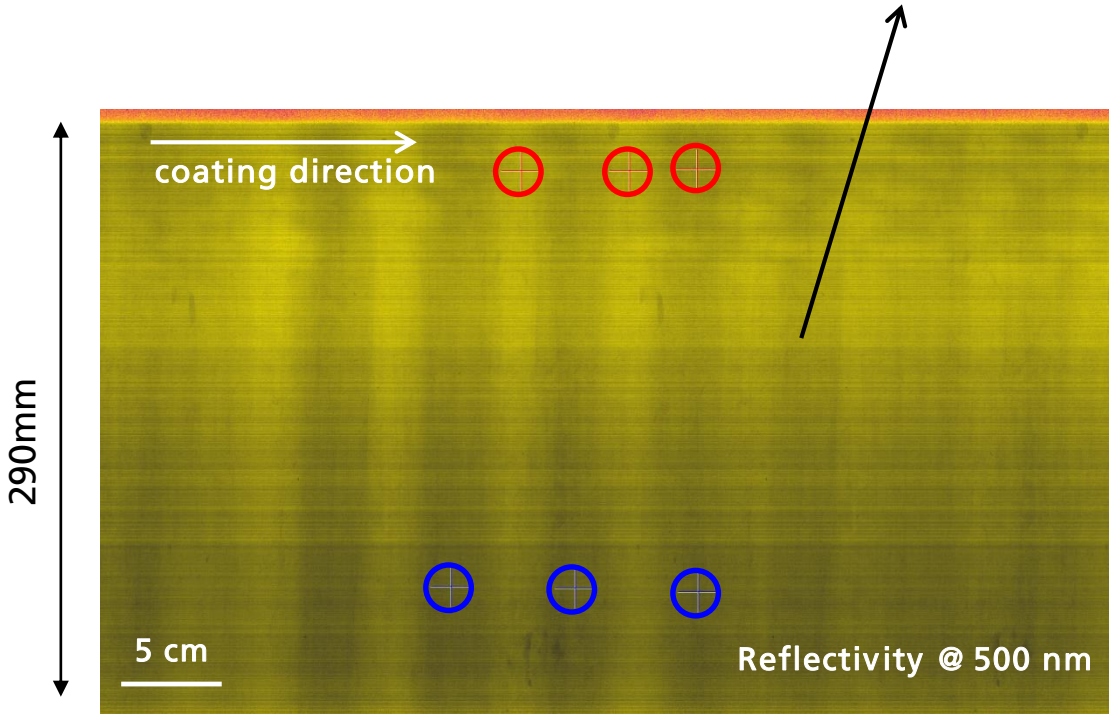
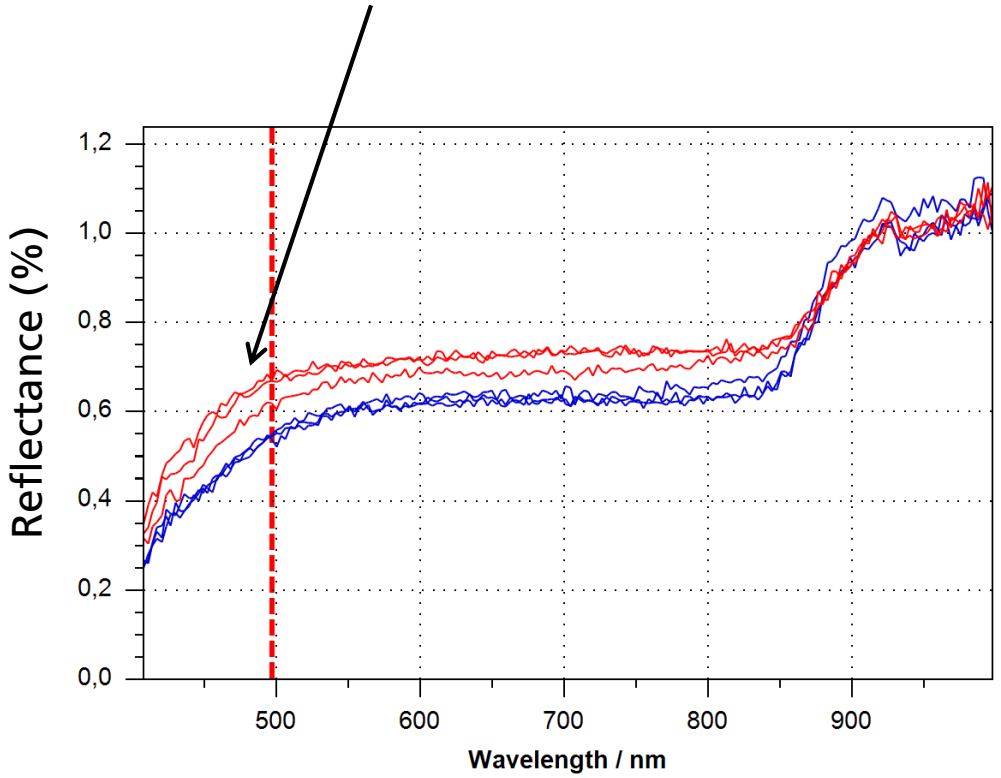
# In-line HSI process monitoring at CoFlex web coater

periodic oscillation of overall HSI signal -> disturbs thickness evaluation from spectral data




- Reason: small acentricity of transport roller/bearing (probably few 100um) at HSI measurement position
- Counter strategies:
  - pure software correction (record & remove the „roller pattern“ in frame blocks)
  - measure full uncoated web once before process -> use as new R% reference
  - …



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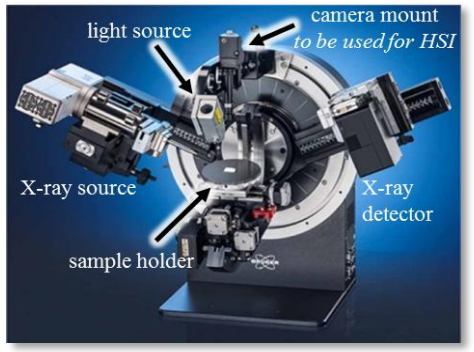
# Summary & Outlook

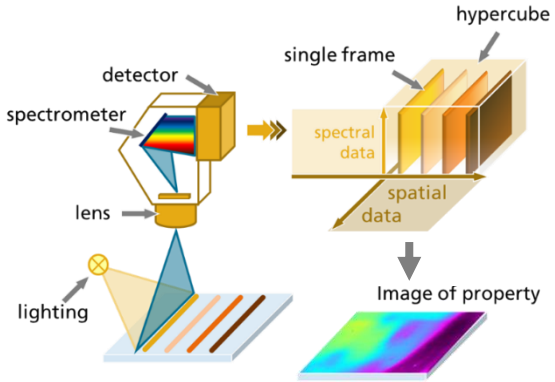
## Summary

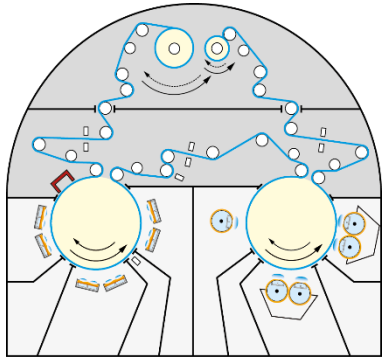
- Light sources, HSI cameras, optics, software developed and commissioned according to the technical requirements of several thin film processes (ALD, slot die coating, sputtering, thermal evaporation)
- Promising results obtained from HSI modelling for various material systems and measurement conditions

## Outlook

- Evaluate technical potential of XRR/XRD measurements in combination with HSI
  (high measurement speed, thickness accuracy, very thin films, robustness & reproducibility, … )
- validate novel NanoQI solutions in an industry relevant environment at several pilot facilities
  (software/process integration, automation, ease-of-use, …)
- cost-of-ownership and return of investment analysis of NanoQI solutions -> viability of commercialization ?





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# Thank you for your attention!

NANO QI

Multimodal X-ray and Hyperspectral
Thin-Film Nanomaterial Evaluation and Quality Imaging












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