in-situ characterization of 2D materials growth

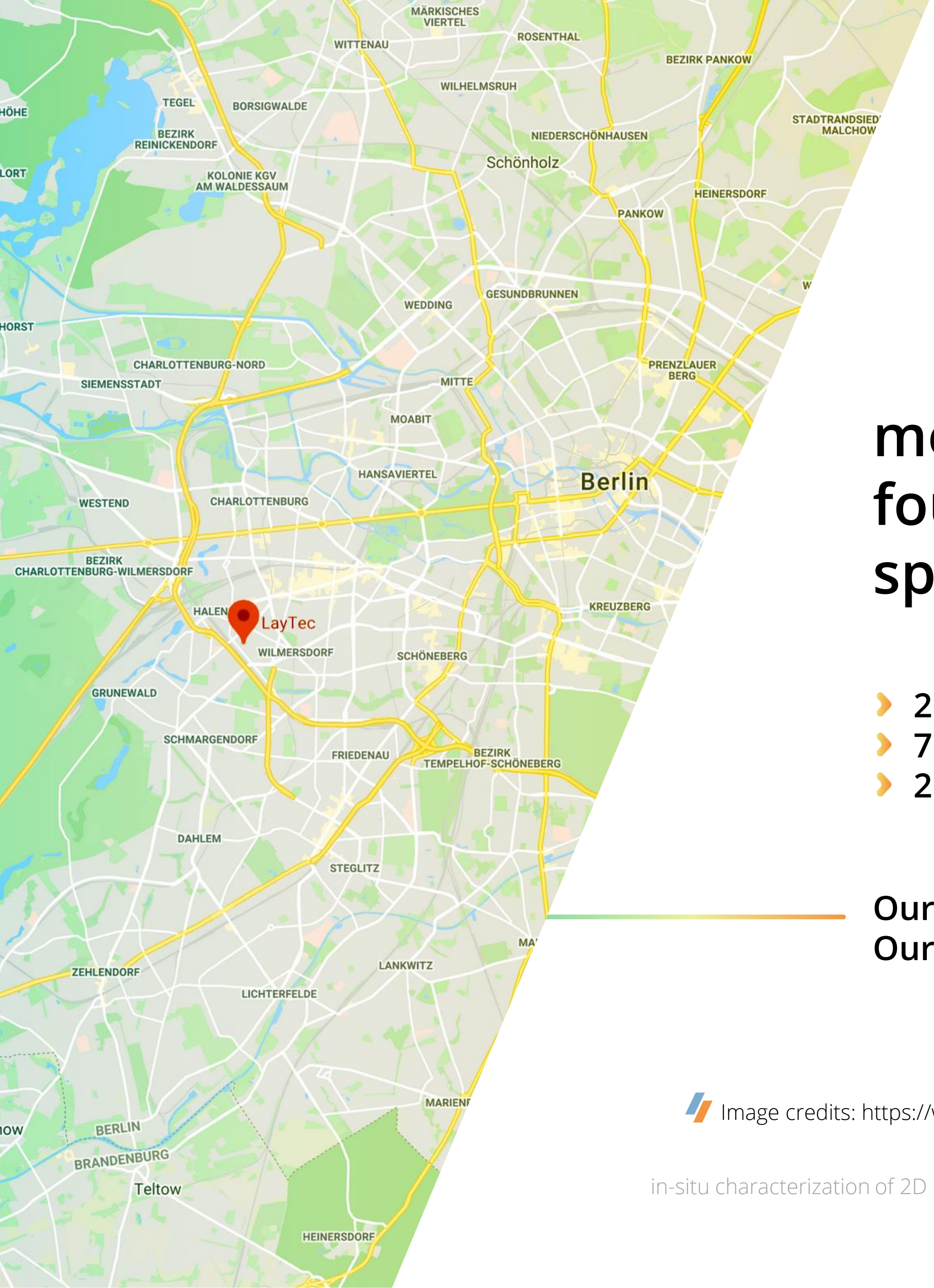
Dr. Marcello Binetti
sr. scientist novel applications

Graphene 2021
October 26-29
Grenoble (France)

AIXTRON

GIMMIK '19 – '21

LAYTEC
MEMBER OF THE NYNOMIC GROUP



metrology company
founded 1999 in Berlin
spin-off of TU Berlin

- 22 years old
- 70 employees
- 2500 systems sold
- operating worldwide
- member of Nynomic group

Our business: Process-integrated optical metrology

Our markets: Semiconductor and thin-film industry & academia incl. lighting, laser, PV, glass coating …

Image credits: https://www.google.de/maps

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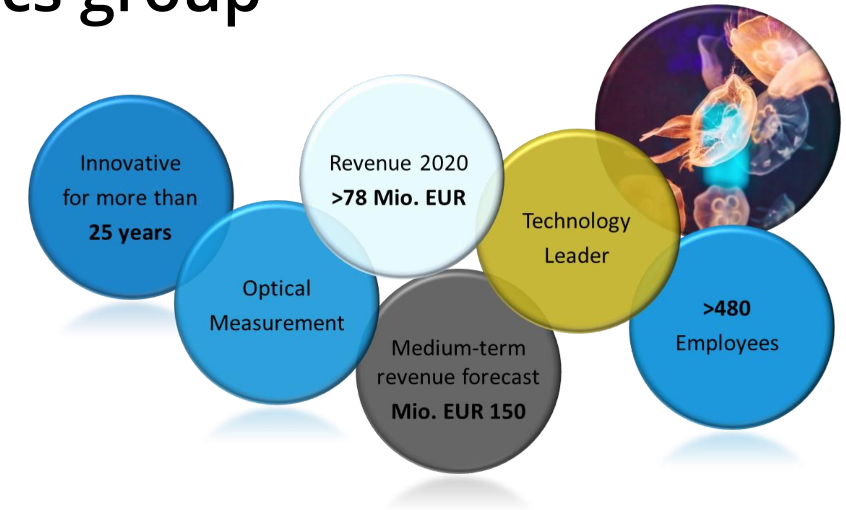
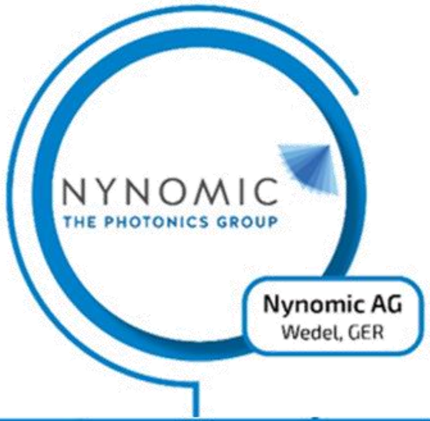
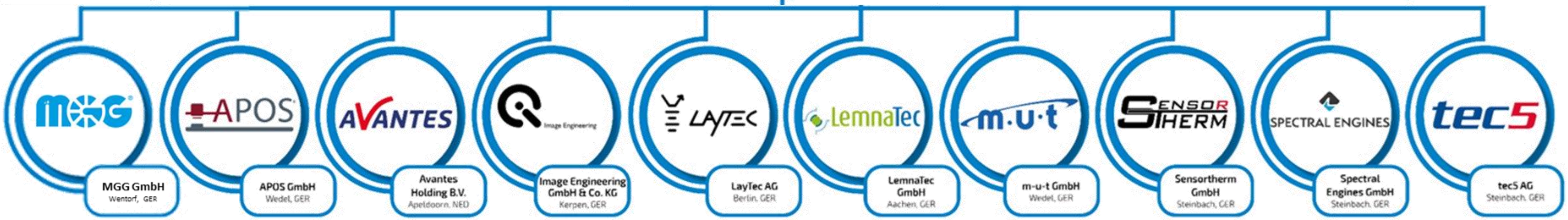
LayTec is a member of Nynomic AG – the photonics group

A growing number of companies, acquired strategically and all dedicated to permanent, non-contact and non-destructive optical measurement technology

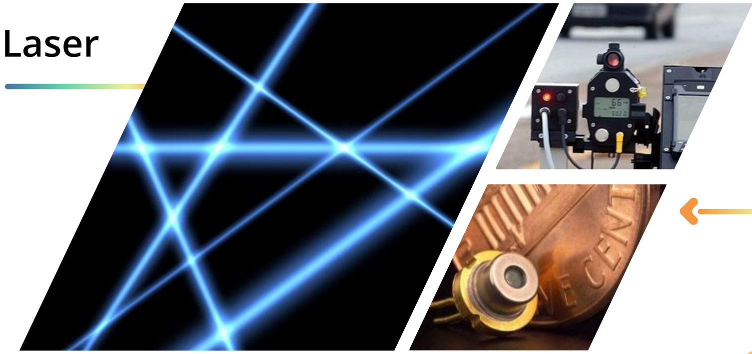






Integrated metrology for various industries and markets

Laser



LED

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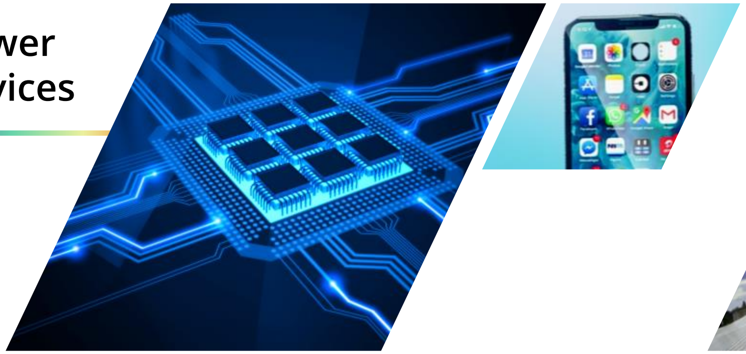
Functional glass

Power devices



Photovoltaics

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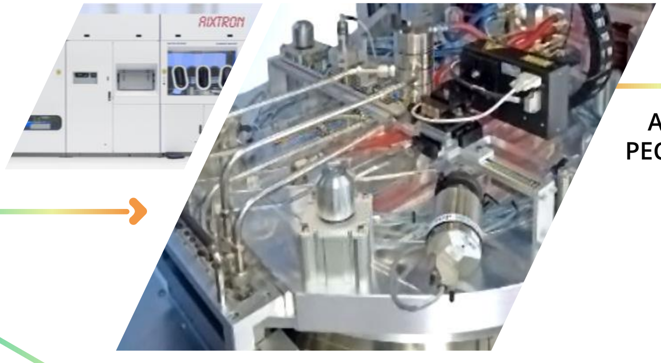
Integrated metrology for various industries and markets

In-line

Conveyor,
Roll-to-Roll,
in chamber



In-situ

ALD, Sputtering,
PECVD, MOCVD, …

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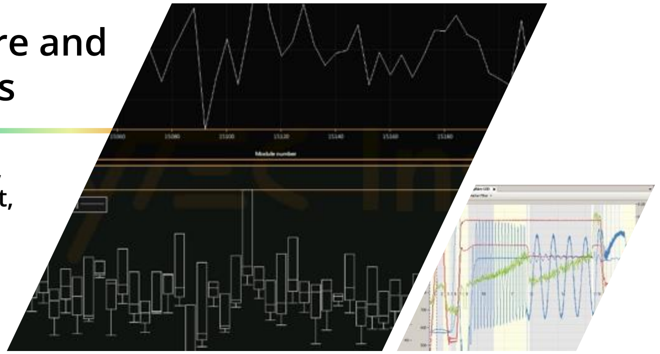
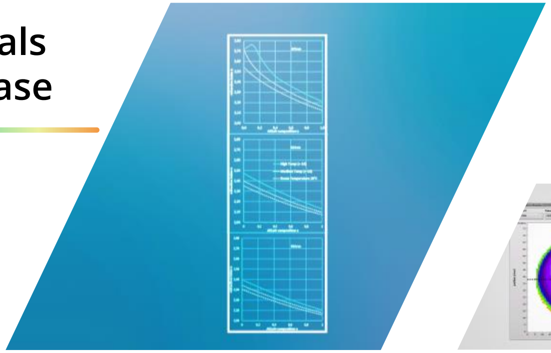
Software and Analysis

Simulation,
Analysis, Fit,
Integration



Materials data base



Mapping

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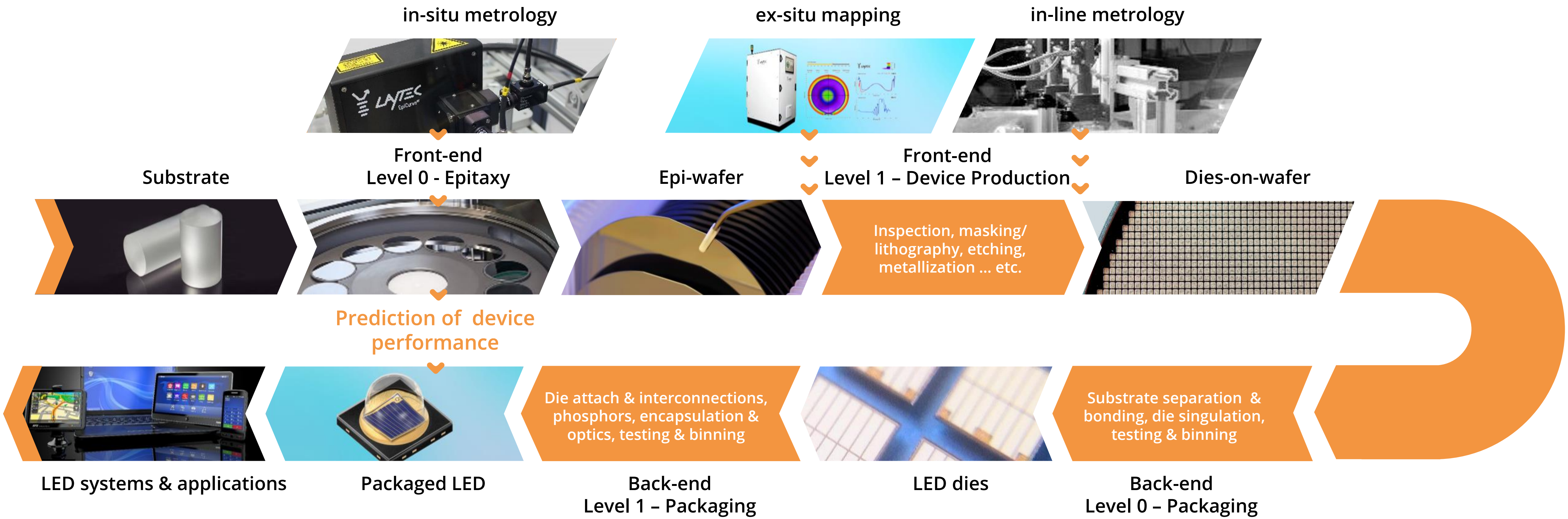
In-situ metrology within manufacturing chain, e.g. LED



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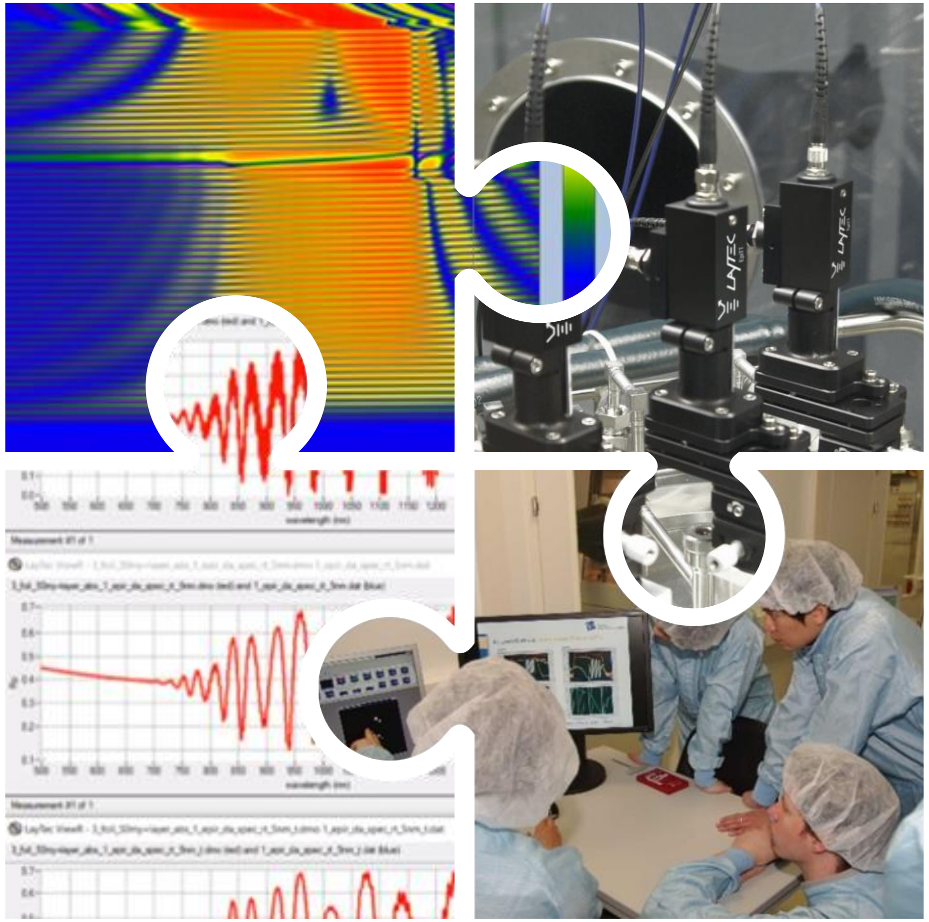
The one-stop-shop for integrated metrology

- Layer and stack analysis & algorithm development
- System, software & hardware development
- Data analysis
- Application Engineering
- Service & support



Customer’s benefits:

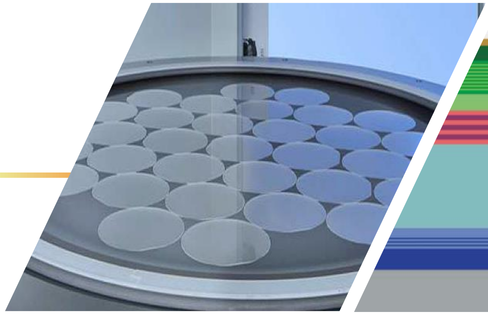
- Advanced process optimization
- Fast failure analysis
- Variance reduction
- Improved quality control
- Short R&D cycles

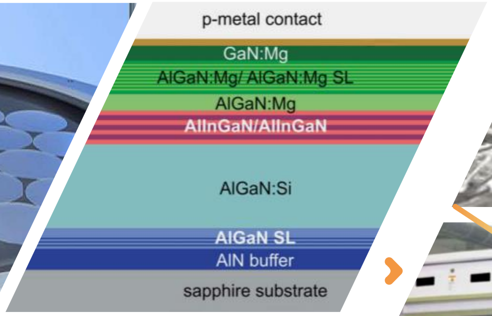
- Yield improvement
- Cost reduction
- Better efficiency
- High quality
- Better competitiveness
- Time to market

We make the related global industries more effective, more productive and less consuming of energy and resources!

In-situ metrology for compound semiconductor deposition processes

substrate
wafers + recipe
for layer stack



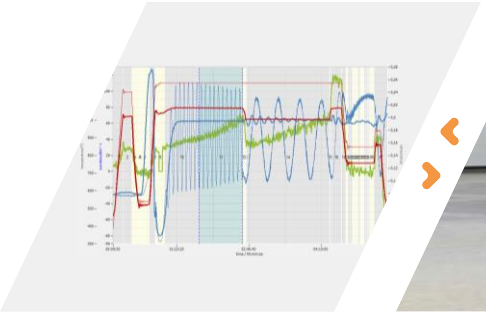


optical in-situ
metrology tool

Measurement of

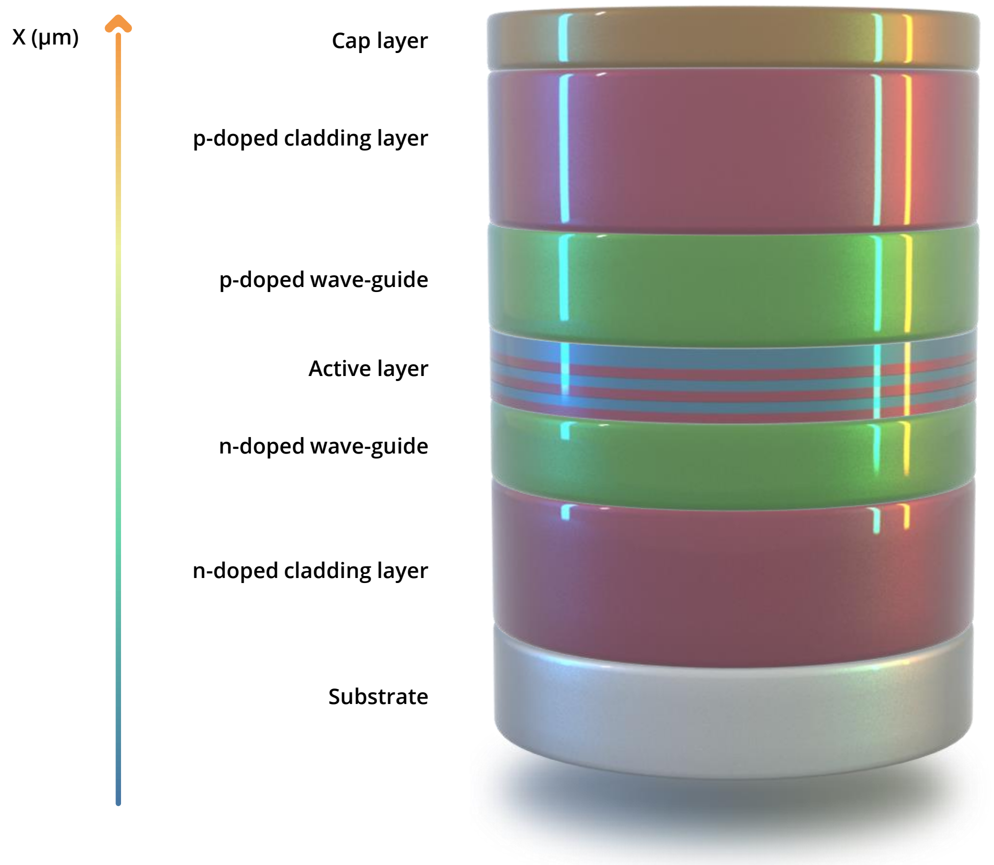
- wafer and pocket temperature
- layer thickness
- growth rate
- wafer curvature
- ternary composition
- surface morphology
- on-wafer uniformity
- ...

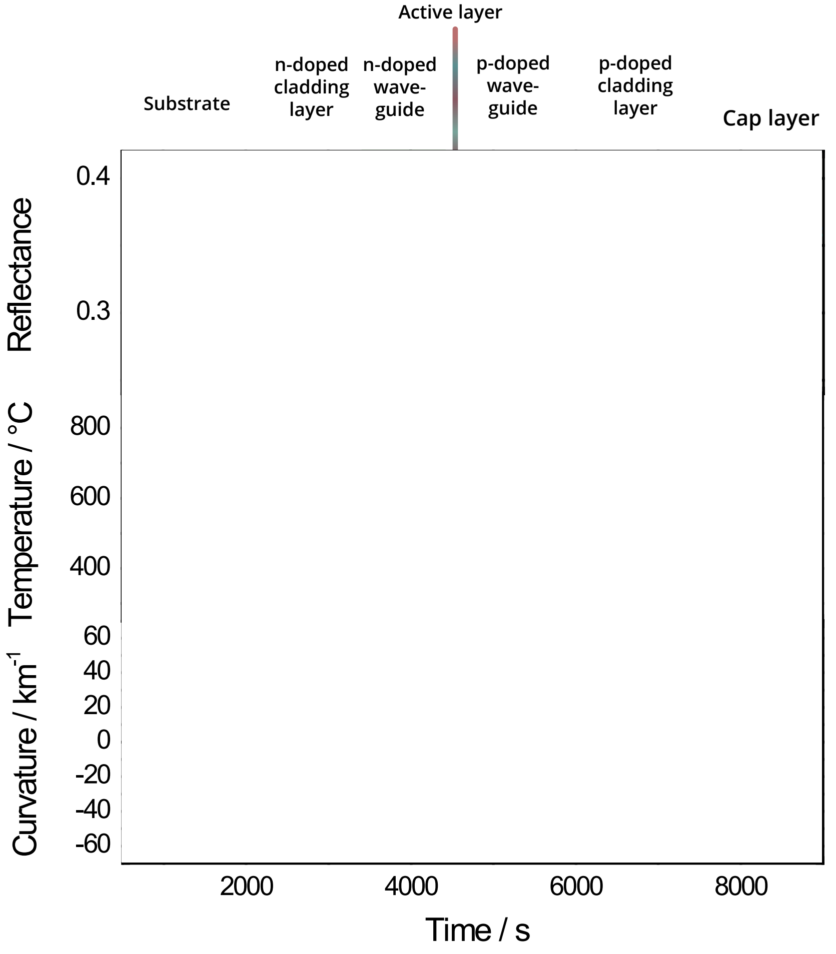
In-situ data for
process control
= monitoring and
control of key growth
parameters and layer
properties



semiconductor device
(e.g. laser)

Edge emitting laser diode: in-situ data and epi steps





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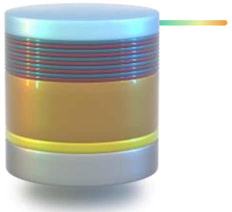
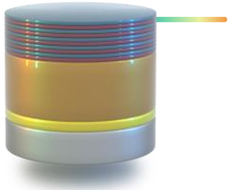
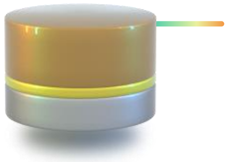
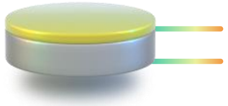
Optimization of device properties: Optoelectronics

Epi growth, e.g. LEDs & lasers	LayTec's monitoring	Prediction of final device properties
Step 3  Cap layer	**Cap layer** › Thickness › Morphology › Final bow	Outcoupling efficiency
Step 2  Active layer (MQW)	**Multiple quantum wells (MQW):** › Thickness › Ternary homogeneity › Growth rate homogeneity	Emission wavelength Emission intensity
Step 1  Buffer	**GaN buffer quality:** › Surface morphology › Thickness, growth rate › Thickness homogeneity › Film strain	Emission intensity
 Nucleation layer Substrate	**Nucleation layer quality:** › Recovery time › Surface morphology › Growth rate homogeneity	Low defect density

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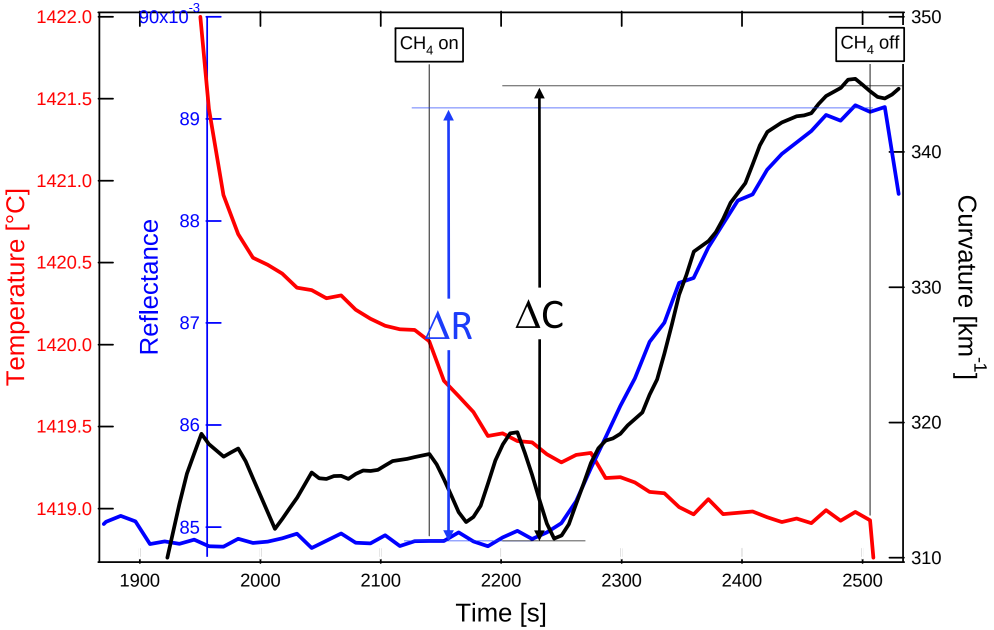
LAYTEC EpiCurve® TT on AIXTRON CCS MOCVD Reactor

Graphene/Sapphire



in-situ measurables:

Emissivity corrected IR pyrometry (ECP)	Substrate back-side temperature (T_{growth} ~1400°C)
Reflectance (R)	Precise process information: induction period (~100s, depending on deposition parameters), R is function of coverage (Θ)
Wafer bow (C)	Substrate front-side temperature

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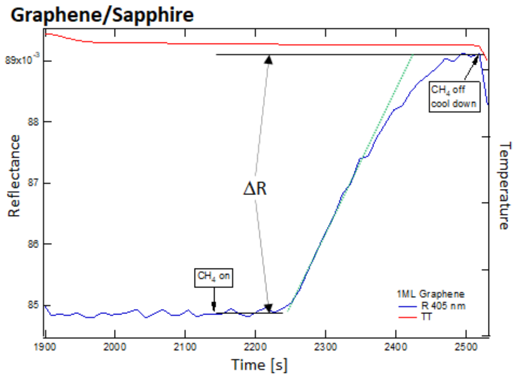
Graphene – CVD deposition: 1st ML (in-situ reflectance, R405nm)

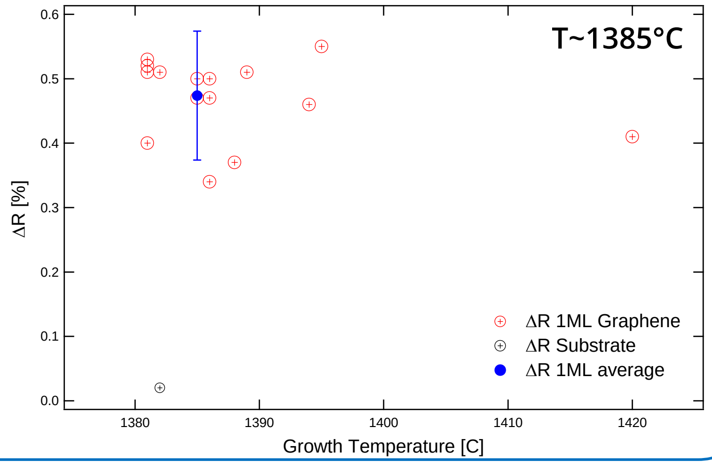
in-situ

R_{405nm} allows measuring coverage (Θ)

$\frac{\delta R}{\delta t} \rightarrow 0$ for $\Theta(1^{st} ML) \rightarrow 1$

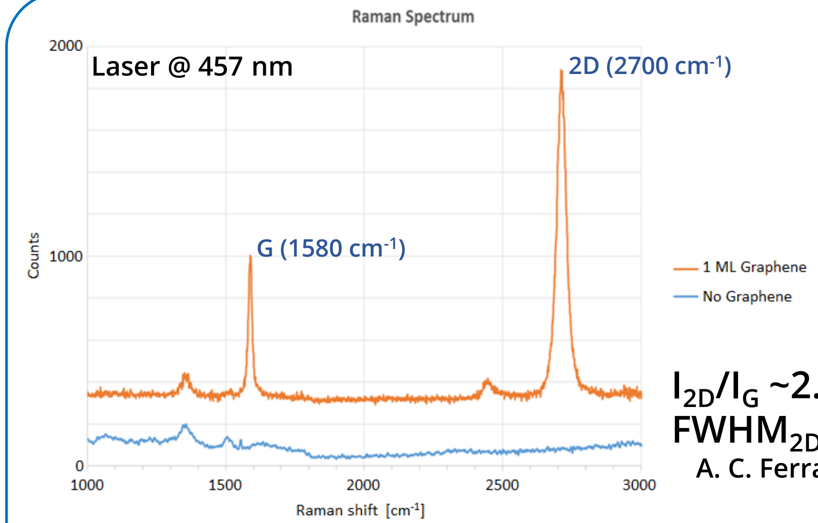


Exp. result:
$\Delta R(1ML)_{405nm}$ =
= 0.48% ± 0.11%



Exp. data by AIXTRON

Raman spectroscopy & simulation



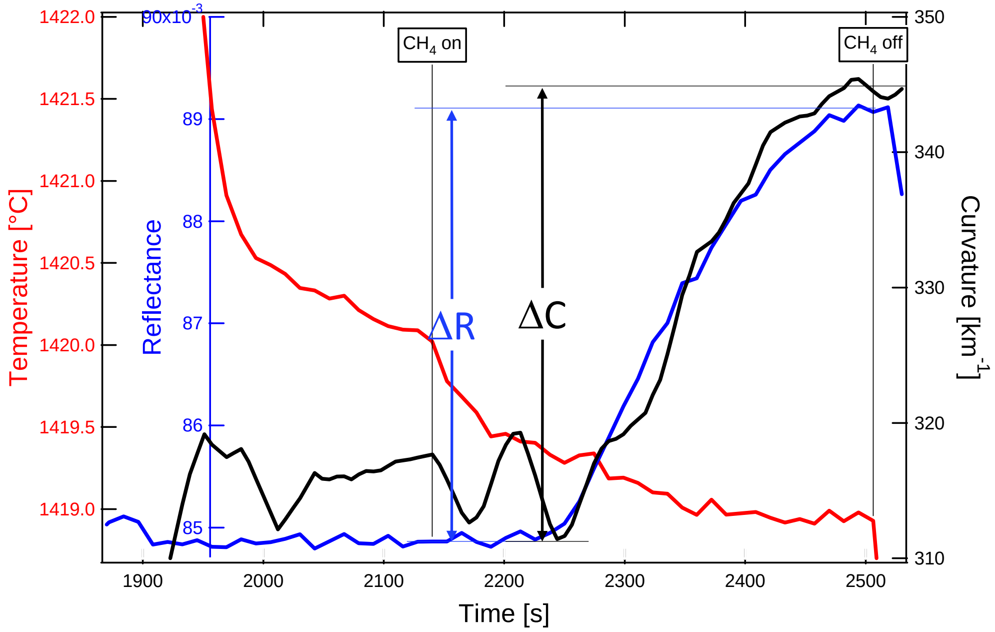
I_{2D}/I_G ~2.3 → Graphene
$FWHM_{2D}$ = 25cm^{-1} } 1ML

A. C. Ferrari et al., PRL 97, 187401 (2006)

Analysis (RT): $\Delta R(1ML)_{405nm}$ =0.63%

- 1ML ~0.335nm *[Dresselhaus M. S. et al., Science of Fullerenes and Carbon Nanotubes; Academic Press: San Diego, CA, 1996; p 965]*
- Graphene n&k at room temperature *[Song, B et al., Appl. Surf. Sci. 439 (2018) 1079–1087, https://doi.org/10.1016/j.apsusc.2018.01.051]*
- Sapphire optical constants (@HT) *[LayTec database]*

Graphene – CVD deposition: 1st ML (wafer curvature → $T_{front\text{-}side}$)



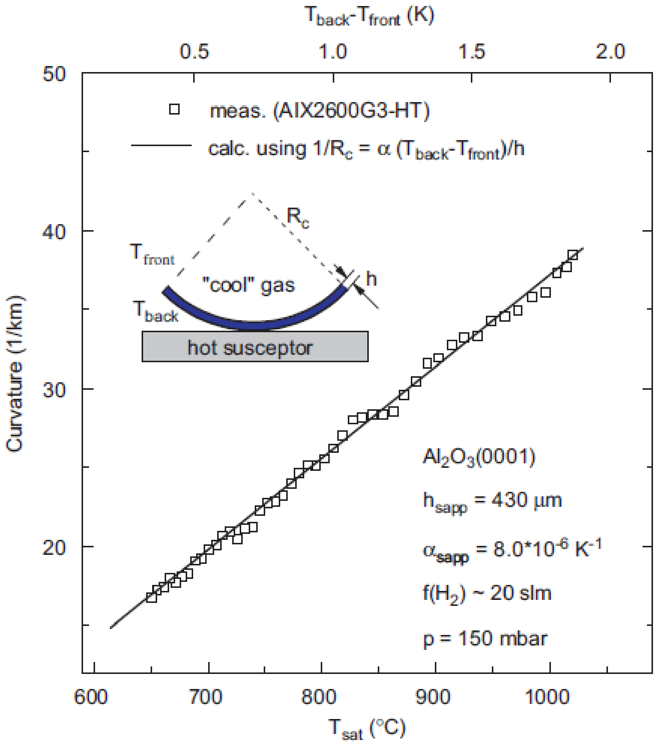
Increase of wafer curvature (ΔC) follows ΔR

Graphene layer increases:

- Wafer surface emissivity
- Vertical temperature gradient through wafer

Exp. data by AIXTRON

Similar to thermal cycling of bare sapphire wafers:



$T_{front\text{-}side}$ =

$= T_{ECP}^{pocket} + \Delta C * h / \alpha$

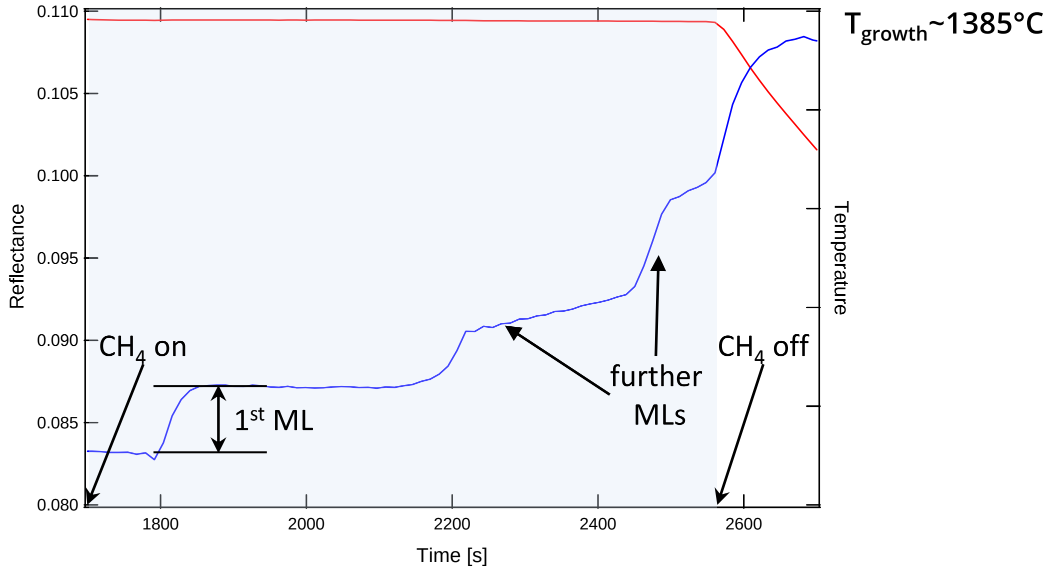
h = wafer thickness

α = sapphire thermal expansion coefficient

[F. Brunner et al., J. Crystal Growth 289 (2007) 202]

Graphene – CVD deposition: further monolayers

in-situ analysis, during deposition:

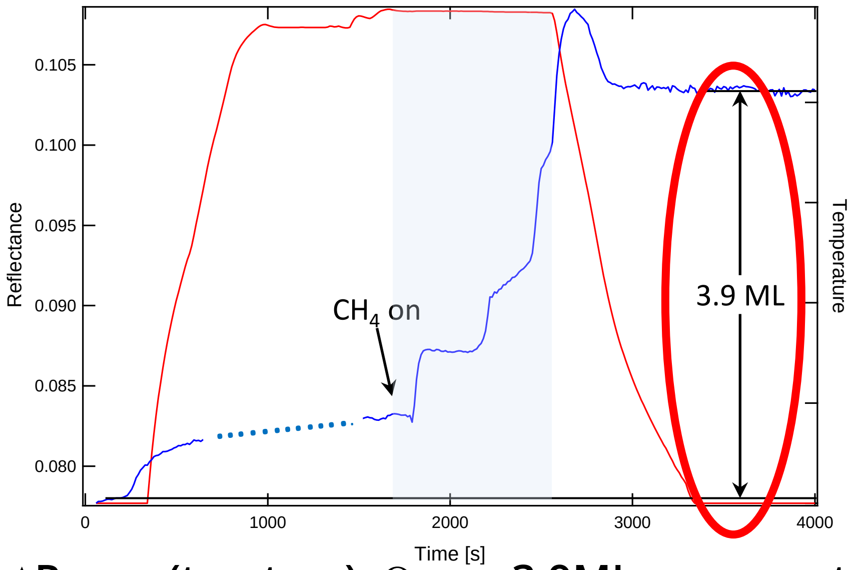


CH_4 pressure: stepwise increase to deposit 3 ML

ML≥2: difficult control for single ML deposition

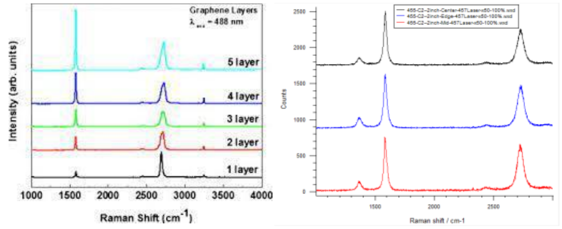
- reduced barrier for ML nucleation?
- different C-species?

in-situ analysis, post deposition:



ΔR_{405nm} $(t_{end}-t_{start})$: Θ_{end} = 3.9ML expected: 3 ML

Excess reflectivity: Amorphous carbon (Raman)



Exp. data by AIXTRON

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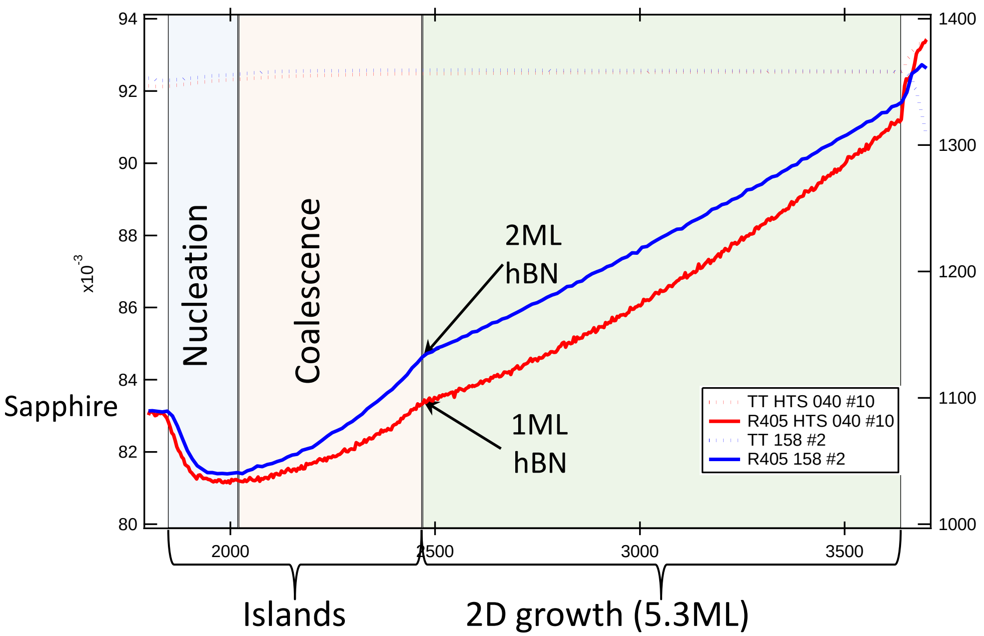
hBN CVD – Nucleation and growth



R405nm in-situ:

- **reveals details of early phase in hBN growth**
- **enables in-situ control of hBN surface quality**

Analysis:

- *1ML hBN/Sapphire (@T_{Room}) = 6.72Å*
  [Yang et al., JCG, 482 (2018)1-8; DOI: 10.1016/j.jcrysgro.2017.10.036]
- *hBN optical constants (@T_{Room})*
  [Lee et al., Phys. Stat. Sol. B 256 (2018) 1800417, https://doi.org/10.1002/pssb.201800417]
- *Sapphire optical constants (@T_{Growth}) [LayTec database]*

Measurement accuracy:

- *hBN birefringence ~-0.8 (@405nm)*
  [Segura A., Artus L., Cusco R., Taniguchi T., Cassabois G. and Gil B., 2018 Phys. Rev. Mat. 2, 024001]
- *n: depends on material crystallinity & porosity, growth stage: thickness accuracy by reflectometry < ±20%*
  [K. Pakula et al., https://arxiv.org/ftp/arxiv/papers/1906/1906.05319.pdf]

in-situ optical methods allow comparing main trends in growth rate changes as a function of basic process parameters.

Exp. data by AIXTRON

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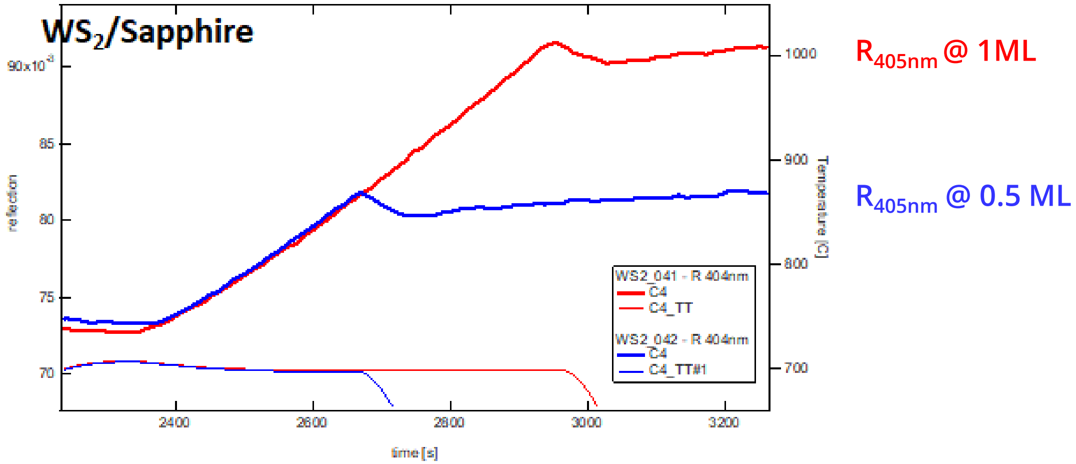
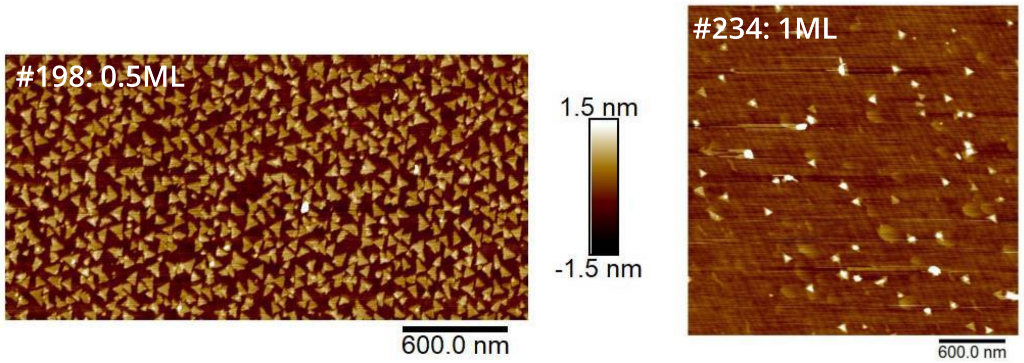
WS_2 CVD – WS_2 on 0.2 offcut sapphire: Nucleation and growth



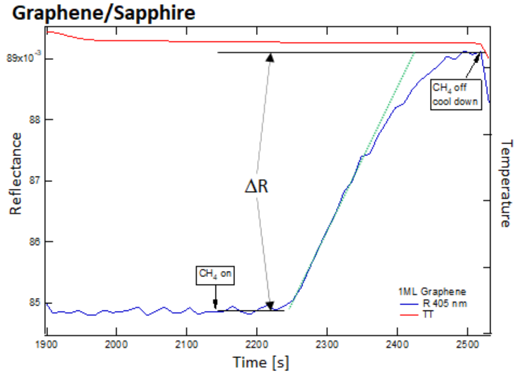


Analysis:

- *1ML WS_2 (@T_{Room}) = 6.18Å*
  [Yilei Li et al., PHYSICAL REVIEW B 90, 205422 (2014)]
- *WS_2 optical constants (@T_{Room})*
  [G.-H. Jung et al., Nanophotonics, 2018-09-29, DOI: https://doi.org/10.1515/nanoph-2018-0120]
- *Sapphire optical constants (@T_{Growth}) [LayTec database]*
- *1ML WS_2 → ΔR(405nm) =1.5%*

ϴ: R405nm (in-situ) consistent with AFM (ex-situ)

No energy barrier for 2nd ML nucleation and growth



Exp. data by AIXTRON

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LAYTEC in-situ metrology for 2D material epitaxy on AIXTRON reactors

- LayTec EpiCurve® TT measures pocket temperature, reflectance and wafer bow
  - monitor 2D material deposition on AIXTRON CCS MOCVD Reactors
- 2D materials and sample structures investigated:
  - Graphene/Sapphire
  - hBN/Sapphire
  - WS2/Sapphire
- Reflectometry: measures coverage (θ) and effective thickness with sub-monolayer sensitivity
  - Validation: comparison with ex-situ results (Raman spectroscopy, AFM), simulations (nk from literature)
- Pocket temperature + wafer bow:
  - substrate's surface temperature during 2D material growth

LayTec EpiCurve® TT enables:

- detailed process optimization
- control of 2D material epitaxy

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Thanks to ...

AIXTRON:

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Ben Conran, Xiaochen Wang

Oliver Whear

D: Simonas Krotkus, Sergej Pasko

Bundesministerium für Bildung und Forschung

German Federal Ministry of Education and Research partial funding through:

GIMMIK '19 – '21

GIMMIK – Grant 03XP0210F
Graphene processing on 200mm wafers for microelectronic applications

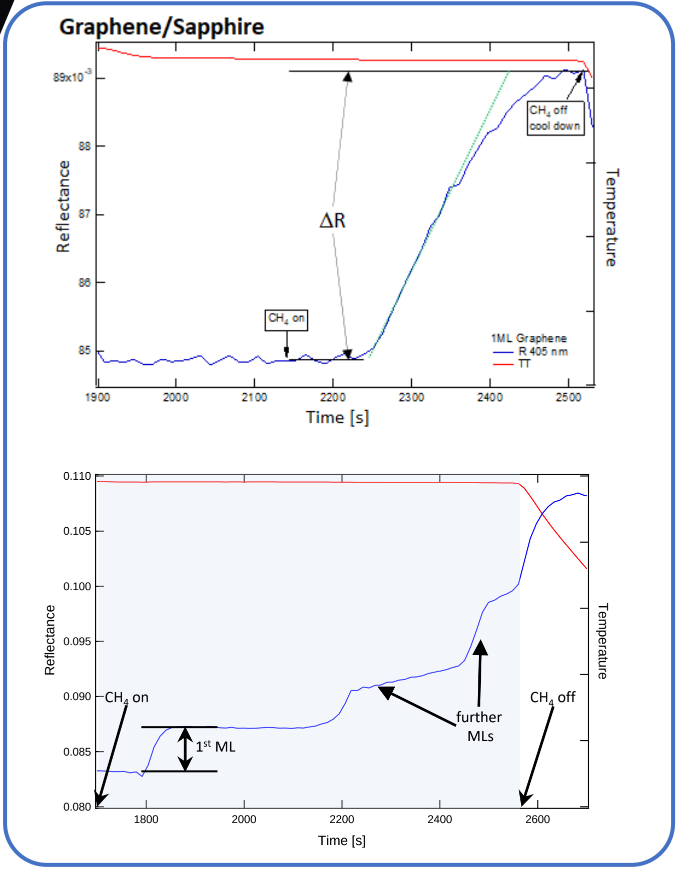
LayTec:

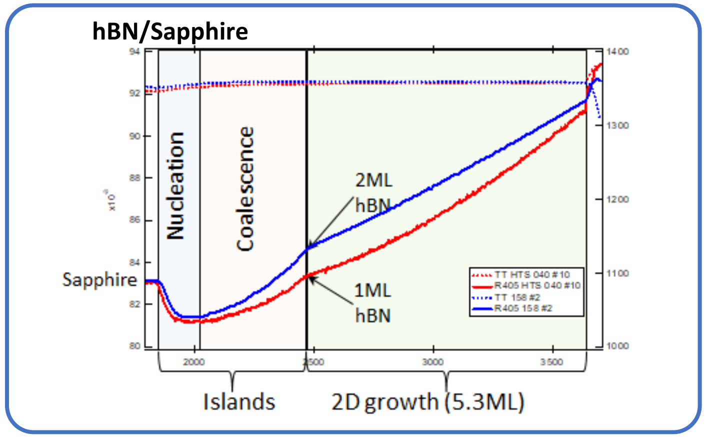
Eugen Speiser

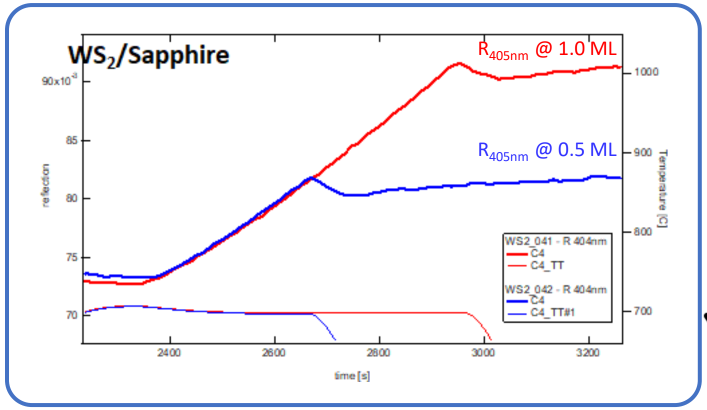
Kolja Haberland, Thomas Zettler

...all of you for your attention!

LayTec EpiCurve® TT enables comprehensive process optimization and control of 2D material epitaxy







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Knowledge is key

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