In-situ Characterization of 2D-Materials MOCVD Growth

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sr. scientist novel applications

WOCSDICE EXMATEC 2026 GDANSK POLAND

LAYTEC
MEMBER OF THE NYNOMIC GROUP

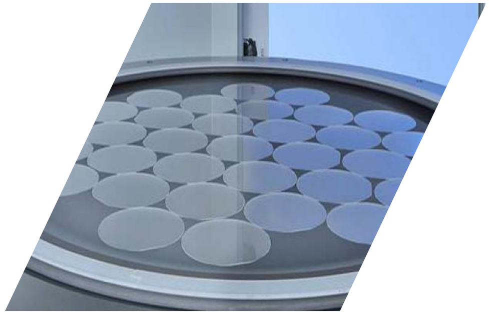
Goal: use in-situ metrology to control and optimize MOCVD of 2D-Materials

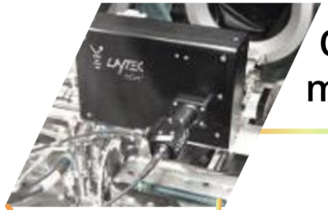
- (MO)CVD is a multi-step process:
  - precursor adsorption and dissociation
  - surface diffusion
  - island nucleation and growth
- All steps are thermally activated
  - Wafer Temperature control improves Reproducibility and Yield
- with Emissivity Corrected Pyrometry → monitor and control wafer Temp., enabling tight process control

- Grow fully coalesced monolayers (MLs) at wafer-scale
  - (differential) Reflectometry at selected λ → measuring surface coverage (Θ), ML $\leq$ 1
- Some properties depend on number of stacked MLs – e.g.: bandgap transition direct (1^{st} ML) → indirect (2^{nd} ML) in Transition Metal Dichalcogenides (TMDs)
  - spectral Reflectometry: follow nucleation and evolution of further MLs (ML $\geq$ 2^{nd})

In-situ metrology for compound semiconductor and 2D-Materials epitaxy

Substrate wafers + recipe for layer stack



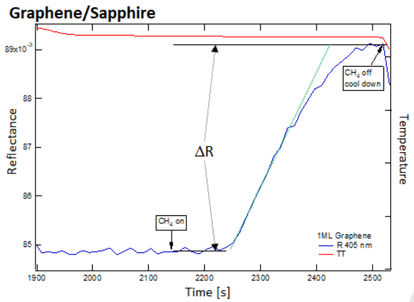


Optical in-situ metrology tool

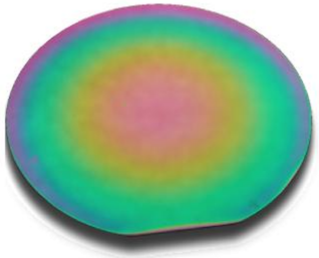
Optical techniques

- Pyrometry (emissivity corrected)
- Reflectometry
- Deflectometry

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



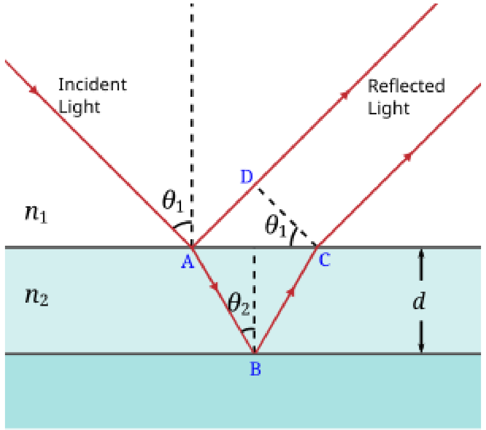
MOCVD equipment



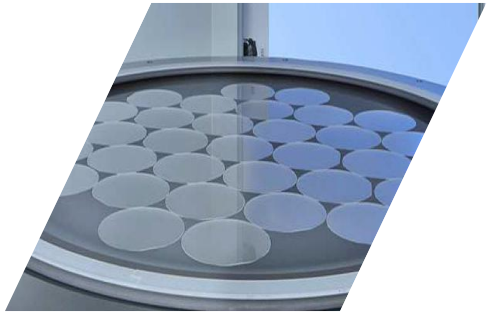
Characterized epi wafer

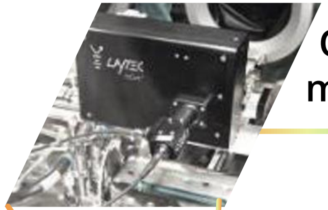
In-situ metrology for compound semiconductor and 2D-Materials epitaxy

Thin film interference



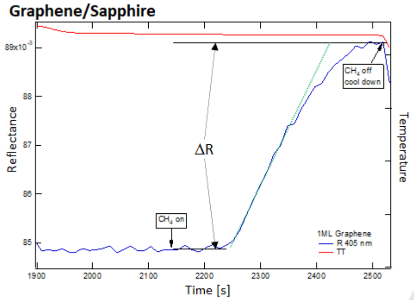
Substrate wafers + recipe for layer stack



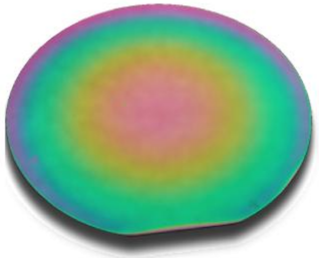


Optical in-situ metrology tool

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



MOCVD equipment



Characterized epi wafer

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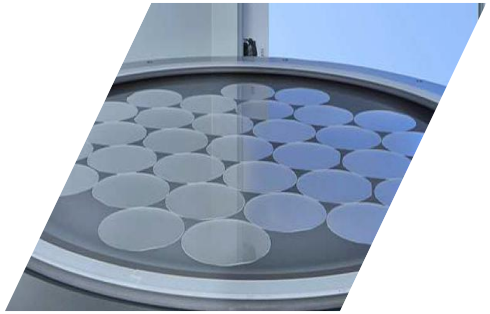
In-situ metrology for compound semiconductor and 2D-Materials epitaxy

Substrate wafers + recipe for layer stack

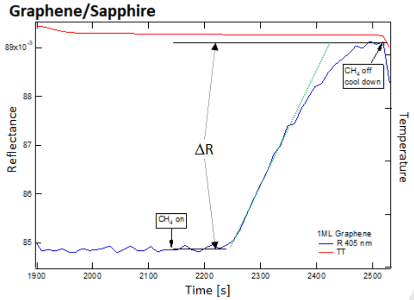


Optical in-situ metrology tool

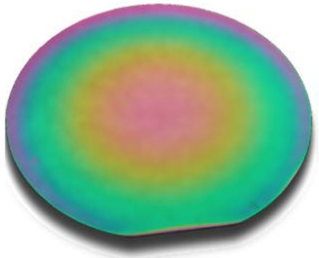
Measurement of

- wafer temperature
- surface coverage (Θ)
- growth rate
- monolayer number (ML)
- on-wafer uniformity
- ...

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



MOCVD equipment

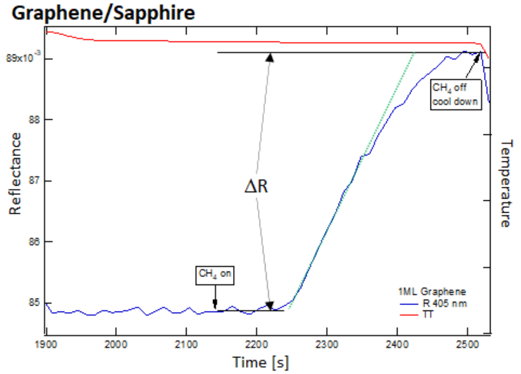


Characterized epi wafer

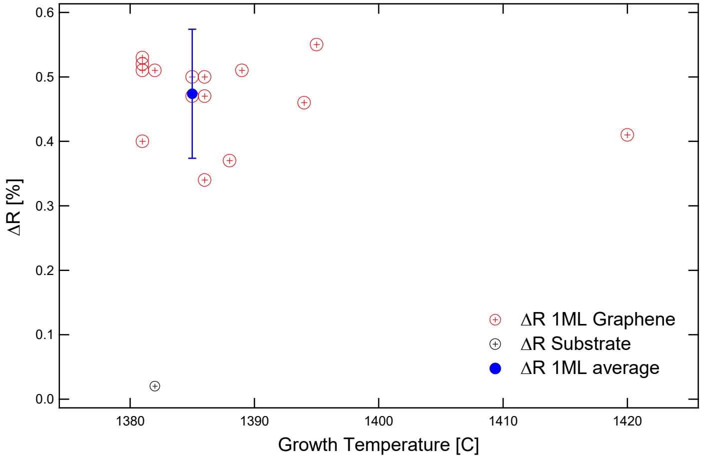
Graphene – CVD deposition: 1^{st} ML

in-situ

R_{405nm}:
coverage (Θ) measurement
$\frac{\delta R}{\delta t} \rightarrow 0$ for $\Theta(1^{st} ML) \rightarrow 1$

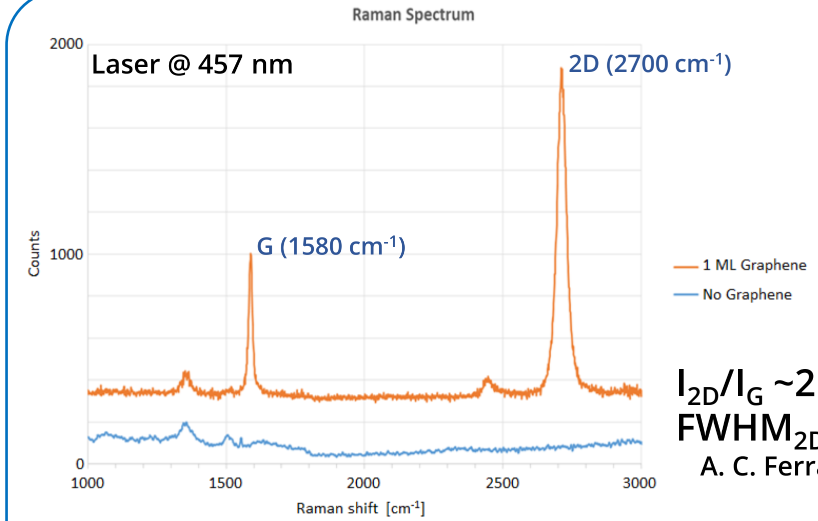


Exp. result:
$\Delta R(1ML)_{405nm}$ =
= 0.48% ± 0.11%



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)

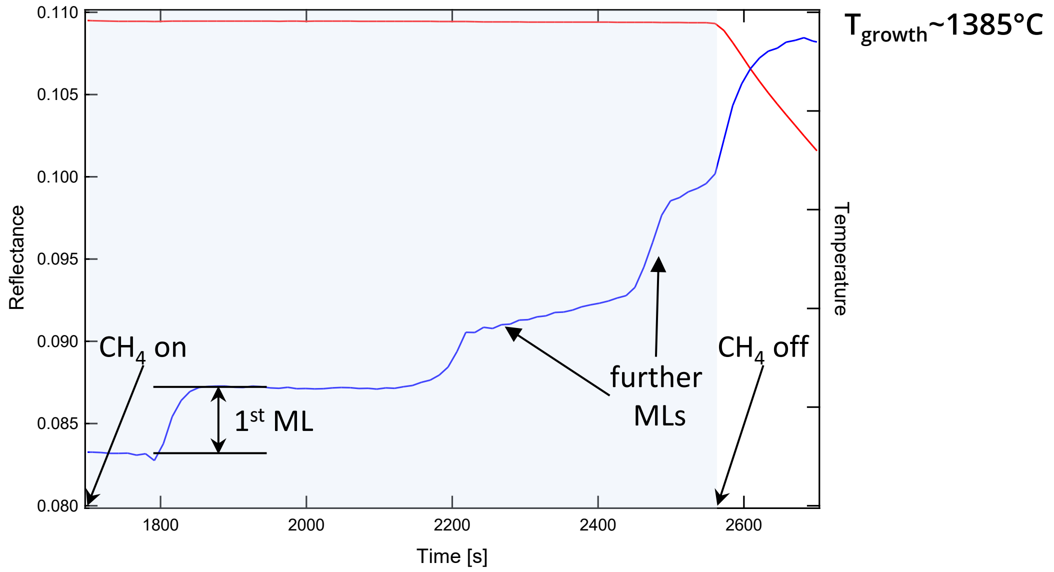
Analysis (RT): $\Delta R(1ML)_{405nm}$ =0.63%

- 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: Reflectance measures coverage degree (Θ) up to 1^{st} ML

Graphene – CVD deposition: further monolayers

in-situ analysis, during deposition:

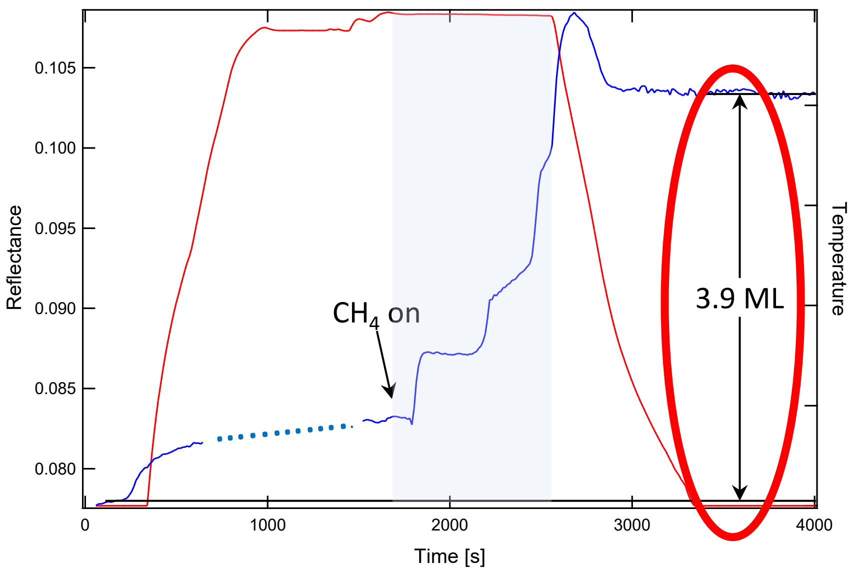


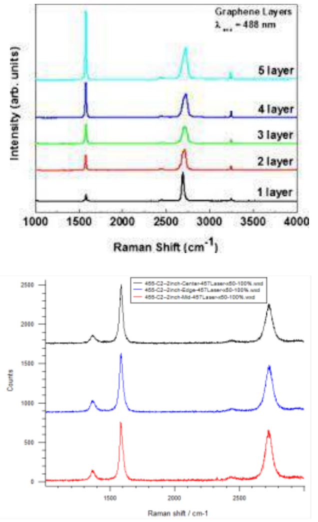
CH_4 pressure: stepwise increase to deposit 3 ML

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

Graphene: Reflectance measures further MLs (≥ 1)

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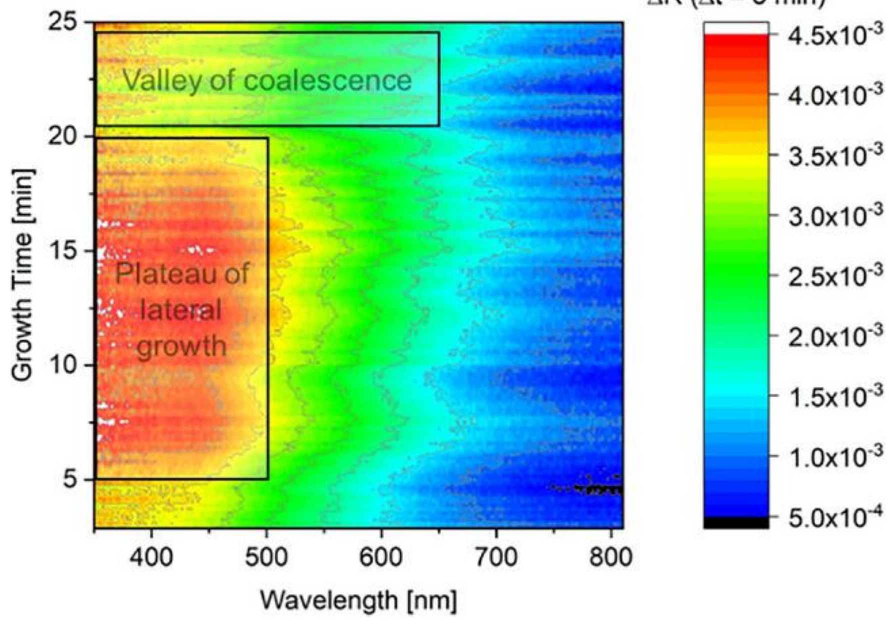
WSe_2 MOCVD – WSe_2 on sapphire: In Situ Spectroscopic Reflectance

- Differential spectra $\Delta R(\lambda,t)_{\Delta t} = R(\lambda,t_i) - R(\lambda,t_i-\Delta t)$
  - Δt is optimized to improve sensitivity to R variations, allowing sub-ML Θ resolution
- Precursors: tungsten hexacarbonyl (WCO) and di-iso-propyl selenide (DiPSe)
- Substrate: ssp sapphire
- T_{dep} = 700°C
- Spectral acquisition rate: 15 spectra/minute
- *S.Tang, et al. Adv. Mater. Interfaces12, no. 22 (2025): e00349* https://doi.org/10.1002/admi.202500349

Final Θ : coalesced ML + ~30% BL



Courtesy of Compound Semiconductor Technology | RWTH AACHEN UNIVERSITY

WSe_2: differential Reflectometry shows the evolution of Θ for 1st ML

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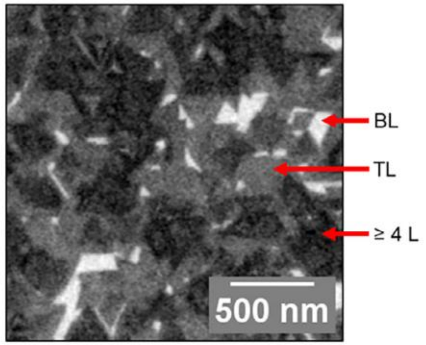
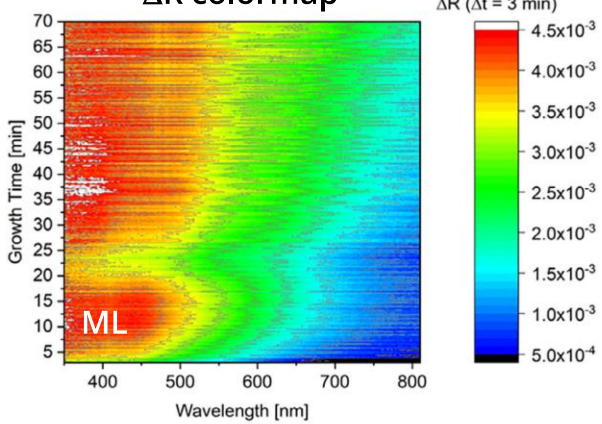
WSe_2 MOCVD – WSe_2 on sapphire: In Situ Spectroscopic Reflectance

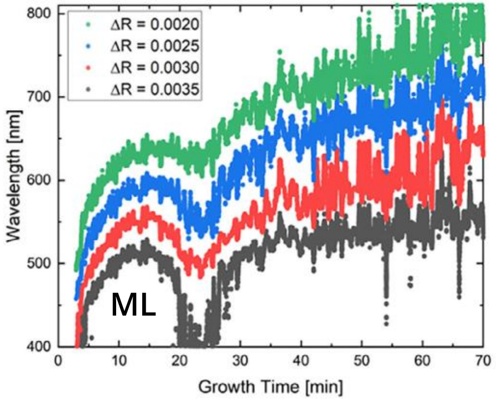
SEM image



ΔR colormap



Extracted contour lines in the ΔR colormap

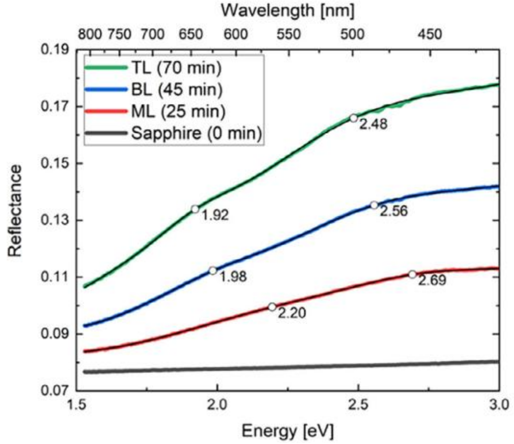


Trilayer (TL): ✗

Bilayer (BL): ✗

Monolayer (ML): ✓

- Comparison of R spectra recorded at 700 °C, on samples of different thicknesses:
  - The fitted position of high T excitonic peaks C and D depends on the number of layers
- Beyond 1st ML: deposited thickness can be deduced from fitted reflectance peak positions



Trilayer (TL): ✓

Bilayer (BL): ✓

Monolayer (ML): ✓

Courtesy of

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WSe_2: fitted peak position measures layer thickness

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Summary: LayTec in-situ metrology for 2D material epitaxy

- LayTec EpiCurve® TT measures wafer or pocket temperature, reflectance and wafer bow
  - monitor 2D material deposition
- Presented results:
  - Graphene/Sapphire
  - WSe_2/Sapphire (example for TMD)
- Measurement of Monolayers (ML) coverage (θ) and multilayer thickness:
  - Up to 1st ML: multi-λ Reflectometry, with sub-monolayer sensitivity
  - Beyond 1st ML: Spectral Reflectometry (shift of excitonic peak position)

LayTec metrology instruments enable:

- detailed process optimization
- control of 2D material epitaxy

Thanks to…

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LayTec

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

… and you all for your attention!

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Knowledge is key

www.laytec.de

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