In-situ monitoring of 2D materials epitaxy during chemical vapor deposition

Graphene and other sp^2-bonded 2D monolayer materials like hexagonal boron nitride (hBN) and tungsten disulfide (WS_2) are opening up exciting new device and material possibilities. One of the most promising methods to synthesize them is thermal chemical vapor deposition (CVD). CVD process steps-precursor dissociation, adsorption, surface diffusion, island nucleation and growth - are usually thermally activated. Therefore, a reproducible process temperature measurement is of paramount importance for the control and repeatability of the deposition. The process control frequently takes place after deposition by well-established ex-situ techniques like Raman spectroscopy, atomic force microscopy, etc. In contrast, LayTec's in-situ metrology systems EpiTT and EpiCurve® TT can be integrated into a deposition reactor and thus enable close control of key parameters during epitaxial deposition of 2D materials. For materials being transparent at 950 nm like sapphire LayTec's EpiTT measures the temperature on the top side of the carrier. Reflectance is measured at 950 nm, 633 nm and 405 nm. If the optical contrast between substrate and epi-layer is sufficiently high, reflectance variations can also be measured during the growth of 2D materials with sub-monolayer coverage. In this case, the reflectance measures the surface coverage within the measurement spot, and its variation describes the different growth stages. At the 12th Graphene and 2D Materials Conference in Aachen in July 2022, LayTec's Marcello Binetti presented the latest results on characterization of 2D material growth, in particular in-situ reflectance, temperature and wafer bow of graphene on sapphire during deposition. In this newsletter we highlight some of the results achieved by monitoring graphene CVD on a single side polished (ssp) sapphire in AIXTRON's CCS reactor (Fig. 1) and demonstrate how LayTec's metrology systems can help increasing reproducibility and yield in CVD production of 2D materials.

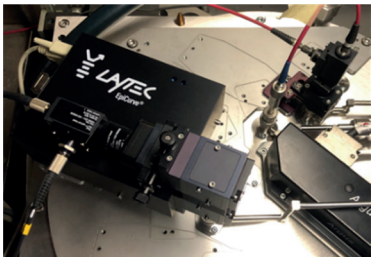

Fig. 1 LayTec's EpiCurve® TT mounted on an AIXTRON CCS UVC reactor customized for 2D-material depositions (courtesy of AIXTRON Ltd.)

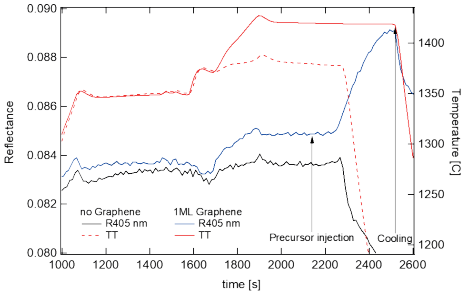

Fig. 2 Data collected during two similar runs: "no Graphene" and 1ML-Graphene.

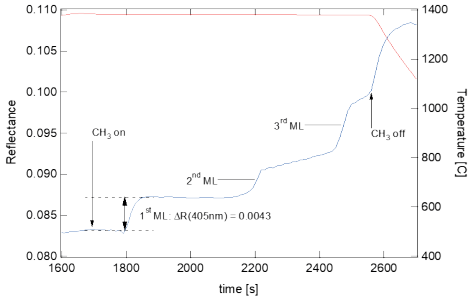

Fig. 3 Evolution of ΔR_{405} with graphene coverage Θ between Θ=0 and Θ=3

Fig. 2 shows two runs of graphene growth on sapphire. In the first run ("no Graphene") the substrate only underwent the thermal cycle with temperature steps at 1347°C and 1370°C but no precursor was injected into the chamber. In the run "1ML Graphene" the sapphire substrate was heated up to 1347°C and then to 1420°C when the deposition of 1ML graphene took place while admitting the precursor for 380s into the chamber. Then the precursor flow was stopped, and the reactor was cooled down. The 405 nm reflectance tends toward a maximum suggesting the existence of an energetic barrier for the nucleation of further monolayers. Fig. 3 illustrates the deposition of up to 3ML graphene. After the saturation of the first monolayer, between 1900s and 2100s, the reflectance remains constant despite the availability of the precursor because an energetic barrier prevents the nucleation of further monolayers. Then, the precursor partial pressure was increased allowing the controlled growth of the second and third monolayers. This process is mirrored by the increase in R_{405}.

To verify the results, ex-situ Raman spectroscopy measurements were performed which confirmed the presence of 1-2ML of graphene and 3ML of graphene on the two samples. Consequently, wafer temperature and surface coverage can be directly deduced right during deposition which ensures accurate process control. Moreover, measuring the variation of the surface reflectance during the growth reveals details on the deposition kinetics and enables a tighter control of the number of deposited monolayers already during the process. LayTec's EpiTT and EpiCurve® TT allow for in-situ monitoring of 2D materials deposition and offer new means for controlling these complex processes thereby accelerating the research on 2D materials and the scale up for industrial production.

To learn more about in-situ monitoring of 2D materials, please download our application note.

To download the conference talk, click here.

Meet us next at ICMOVPE XX in Stuttgart, Germany and join LayTec´s 26th In-situ Seminar.

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