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In Situ X-ray and Optical Characterisation of Monolayer Graphene and h-BN Growth on Liquid Metal Catalysts

Two-dimensional materials (2DMs) such as graphene and hexagonal boron nitride (h-BN), are one of the most promising classes of materials for future technological advancement. However, nearly two decades since their discovery, effective mass production of defect-free 2DMs still remains a challenge.
The current state-of-the-art synthesis approach for 2DMs includes the dissociative adsorption of gas-phase precursors on a catalytic substrate. [1]
Although solid substrates are typically used, recent research has shown that liquid metal catalysts (LMCats), such as molten copper, provide a more efficient alternative. They offer an atomically flat and isotropic surface, high diffusion rates, and superior catalytic activity compared to their solid counterparts. [2]
The difficulty of effectively monitoring 2DM growth on LMCat substrates however, has resulted in limited research focus in this area.
Addressing this challenge, our team has dedicated efforts designing and developing a reactor specifically tailored to monitor and regulate growth on an LMCat substrate *in situ* and in real-time. [3]
Having successfully probed and perfected the growth of mm-size single-layer graphene [4,5], the LMCat reactor is now being utilized to synthesize h-BN.
Here we report the successful growth of h-BN, as well as possibly the first real-time *in situ* synchrotron X-ray diffraction (XRD) and measurements of single layer h-BN on liquid copper.
Our experiments yielded reflectivity curves (XRR) in line with what is expected of single-layer h-BN based on simulations and literature.
Further measurements using methods such as *in situ* grazing incidence diffraction (GID) and radiation mode optical microscopy (RMOM), as well as ex situ SEM, EDX and Raman spectroscopy were conducted in order to confirm the nature of our samples.
Using these results we plan to refine synthesis parameters such as reactor temperature, precursor gas flow ratios and reactor pressure so as to achieve optimal monolayer growth for h-BN.

[1] https://lmcat.eu
[2] D. Geng and G. Yu, 'Liquid Catalysts: An Innovative Solution to 2D Materials in CVD Processes." Materials Horizons', Royal Society of Chemistry, 2018.
[3] M. Saedi et al., 'Development of a reactor for the *in situ* monitoring of 2D materials growth on liquid metal catalysts, using synchrotron x-ray scattering, Raman spectroscopy, and optical microscopy', Rev. Sci. Instrum., vol. 91, no. 1, p. 013907, Jan. 2020.
[4] M. Jankowski et al., 'Real-time multiscale monitoring and tailoring of graphene growth on liquid copper', ACS Nano, vol. 15, no. 6, pp. 9638–9648, Jun. 2021.
[5] V. Rein et al., 'Operando characterization and molecular simulations reveal the growth kinetics of graphene on liquid copper during chemical vapor deposition', arXiv [cond-mat.mtrl-sci], 24-May-2023.

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