Operando Monitoring of Epitaxial Graphene Growth using High Temperature Neutron Reflectometry

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Synthesis of epitaxial graphene (EG) on cubic silicon carbide (3C-SiC) on silicon offers an attractive route towards novel wafer-scale electronics and photonics integrated on silicon [1]. We have pioneered a unique way to achieve a controlled graphene growth on this challenging substrate by using a thin Ni/Cu alloy [2]. A detailed understanding of the epitaxial graphene growth mechanism, via operando characterization is critical to further optimize the process. In this work we report on the real-time epitaxial graphene growth mechanism on 3C-SiC/Si substrates using a unique setup of neutron reflectometry (NR) (up to 1100 °C, below 5×10^{-5} mbar and characterization [3,4], refer to Fig 1a) and b). We have conducted NR measurements during a controlled annealing on the Spatz time-of-flight neutron reflectometer at the 20 MW OPAL Multipurpose Reactor at the Australian Nuclear Science and Technology Organization (ANSTO, Lucas Heights, Australia). Spatz is a time-of-flight neutron reflectometer, which uses cold neutrons with wavelengths in the range of 2-20 Å. [5]

The advantages of using neutron beam are that it can penetrate through the walls of vacuum furnace to monitor the graphene formation at 1100 °C. In addition, due to the larger and distinct neutron scattering length of carbon, oxygen and silicon, these elements can be observed and differentiated under neutrons [6]. The use of standard X-ray reflectometry cannot achieve this due to two reasons: first, the X-rays cannot penetrate through the vacuum furnace walls and second, the X-ray scattering length for carbon, oxygen and silicon are relatively similar and smaller, making them less sensitive under X-rays.

The observations confirm formation of the metal silicide, which is the key player enabling the release of atomic carbon from SiC. Furthermore, the experiments also confirm the liquid phase nature of the epitaxial growth, which is the key reason for the uniform coverage of graphene on the substrate. It also illustrates formation of graphene layer as well as a thin silicate layer of ~20 Å between graphene and SiC. [3].

Through this work we unlock new avenues to use high temperature neutron reflectometry for the observation and optimization of epitaxial graphene growth and other 2D materials.

References:
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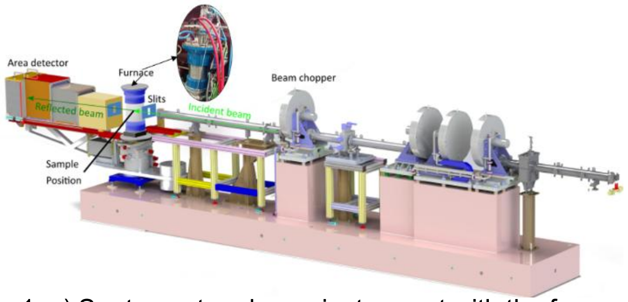
a)



b)

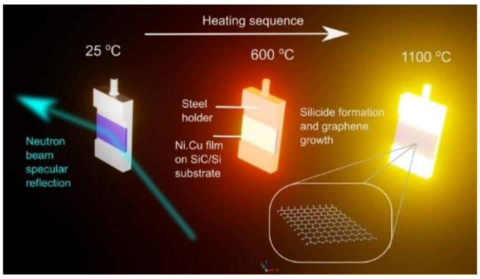


Fig. 1: a) Spatz neutron beam instrument with the furnace installed, located within the Australian Nuclear Science and Technology Organization (ANSTO), recreated from ref. 4 under creative commons license CC BY 4.0 DEED. b) Operando monitoring of EG synthesis using neutron reflectometry