Preparation of the graphene layer

Chemical vapour deposition (CVD) using a vertical quartz tube was used to synthesized large-area single layer graphene on a copper foil. The foil was first smoothened in acetic acid for a minimum of 30 min to reduce the nucleation sites, allowing to minimize defects during the graphene growth. The foil was then cleaned in acetone and isopropanol, before being placed in a quartz tube and annealed during 90 minutes in a 10 sccm H₂ flow at a temperature of 1050°C and at a pressure of 120 Torr. A 0.15 sccm flow of CH₄ was then introduced for 30 minutes while maintaining the H₂ flow. The tube was then taken off the oven and rapidly cooled down to room temperature while maintaining the H₂ flow. The graphene was characterized by Raman spectroscopy, confirming the full monolayer coverage. Poly(methyl methacrylate) was then spin coated on the graphene before etching the copper in ammonium persulfate. Remaining copper residues were then removed by submerging the graphene in a 10% HCl solution for 10 minutes. Graphene was then rinsed in DI water before being transferred onto the Si substrate. The sample was dried overnight before removing the PMMA with acetone.

Fig. 1 Atomic force microscopy cross section of the 85 nm thick P3HT film on silicon.
In-situ cooling of the films

Both P3HT films were spun on silicon and on graphene, and their thickness was controlled using atomic force microscopy. The cross-section of one of the films is shown in Fig. 1. Both films were placed in a chamber filled with helium and heated up to 240°C before being cooled down stepwise to room temperature. At specific intervals, grazing incidence X-ray diffraction measurements were done at an incidence angle of 0.13° and a X-ray wavelength of \( \approx 0.974 \) Å. Example of the obtained scans are shown in Fig. 2.

Cross-sections of the observed in-plane and out-of plane peaks have been obtained for every measured step, in order to determine the crystalline properties of the films during the cooling process. Such cross-sections are shown in Fig. 3 for the in-plane and out-of-plane 100 peaks at a high temperature close to the onset of crystallization and at room temperature at the end of the cooling process, showing both the increase in intensity as well as the shifting in position of the peaks due to the cooling of the films.

References


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Fig. 2 Grazing incidence X-ray diffraction (GIXD) snapshots of both films on silicon (Si) and graphene (G) at different temperatures during cooling.

Fig. 3 Cross sections of both the in-plane and out-of-plane 100 peaks close to the onset of crystallization and at the end of the cooling process showing the shift in q due to evolution of the crystalline structure in the films during cooling.