Real-Time X-Ray Texture Study of Langmuir Few-Layer MoS$_2$ Film During Compression

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The liquid exfoliation opened a new avenue for cost-effective and large-scale production of 2D materials. In contrast to the thin film CVD growth technology, the fabrication of thin films from solution-based fractions of 2D nanomaterials requires a careful reconsideration of available deposition technologies. The formation of Langmuir films of mono- or few-layer 2D materials provides high-quality layers in thermodynamic equilibrium which can be further transferred onto arbitrary substrates. Here we present a systematic study of the Langmuir film formation of few-layer MoS$_2$ on water subphase. A fraction of the few-layer MoS$_2$ flakes with a controlled size dispersion in ethanol/water solution was spread onto the water subphase. A dedicated laboratory X-ray experimental setup was employed to track in real-time the texture evolution of MoS$_2$ Langmuir film during continuous compression. The isotherm of surface pressure versus film area along with the texture analysis using 002 diffraction are shown in Fig. 1. A sketch showing different packings of MoS$_2$ flakes and the respective changes in reciprocal space are depicted in Fig. 2. At the low surface pressure below 3 mN/m, the Langmuir film consists of randomly scattered isolated islands of MoS$_2$ flakes. The width $\chi_{002}$ (FWHM) of the 002 diffraction streak at the constant $q_{002}$ value in reciprocal space is directly proportional to the mean angular spread of the flakes tilt from horizontal (water subphase surface). The surface pressure range from 3 to 4 mN/m can be associated with a liquid-condensed (Lc) phase similar to molecular Langmuir films. Here, the gradually growing surface pressure is accompanied by a minor 0.5° increase of the flakes tilt. Further increase of the surface pressure (equivalent to solid phase) up to a critical value of $\Pi_c \approx 15$ mN/m is manifested by a significant increase of the orientation misalignment by 7°. The compression above $\Pi_c$ results in an irreversible collapse of the MoS$_2$ monolayer.

References


Figures

Figure 1: $\Pi$-A isotherm and corresponding MoS$_2$ texture based on 002 diffraction.

Figure 2: Scheme of different phases and their representation in reciprocal space during compression of MoS$_2$ Langmuir film.