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In-situ Transmission Electron Microscopy Study of 2D Transition Metal Oxide Nanosheets Formation Inside the Liquid Sandwiched Between Graphene Layers

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Growth of the metal oxide film on graphene layer and other metal's surfaces is an important synthesis methods of hybrid materials for different applications such as catalysis, electrochemical devices, and biomedical materials.[1], [2] Manganese oxides and oxyhydroxides are of considerable interest in many technological applications, for example, electrochemical reaction, batteries, catalysis and magnetic materials due to their outstanding structural diversity combined with novel chemical and physical properties.[3], [4] A clear understanding of the crystallization pathway of manages oxide film controlled by graphene bilayers is essential for the synthesis of such hybrid materials. Formation of 2D crystal between graphene layers is observed before. Monolayer MgO and CaO crystallization from $MgCl_2$ and $Ca(OH)_2$ is reported due to the effect of high van der Waals pressure on trapped interlayer molecules[5]. These monolayer metal oxides are formed with their cubic structure in four-fold symmetry between twisted graphene bilayers with 12-fold symmetry. In this work using $MnCl_2$ solution in graphene liquid cell, formation of manganese oxide 2D nanosheet with specific growth direction controlled by hexagonal symmetry of graphene layers is observed (Figure 1).

Single layer graphene substrate is used as the epitaxial growth template for mono/few layer crystallin film of different materials such as MoS_2, Bi_2Se_3 and InAs with specific synthesis temperature and conditions.[6]–[8] However, in this work, the 2D crystal growth is happening due to the high pressure of van-der Waals interlayer forces and confinement of extra thin water layers and its dissolved ions at room temperature. HRTEM of the 2D crystal nanosheets and the fast Fourier transform (FFT) diffraction pattern shows that formation of [110] plane of the cubic FCC crystal structure of manganese oxide is favored inside the graphene liquid cell (Figure 2). This specific growth of hexagonal shape plane of the cubic structure of metal oxide indicates the epitaxial growth effect of graphene layers. The 6-fold symmetry of graphene layers confining the metal ions solution, not only results in formation of 2D structure but also favors a selective growth of the crystal plane with better lattice match [9].

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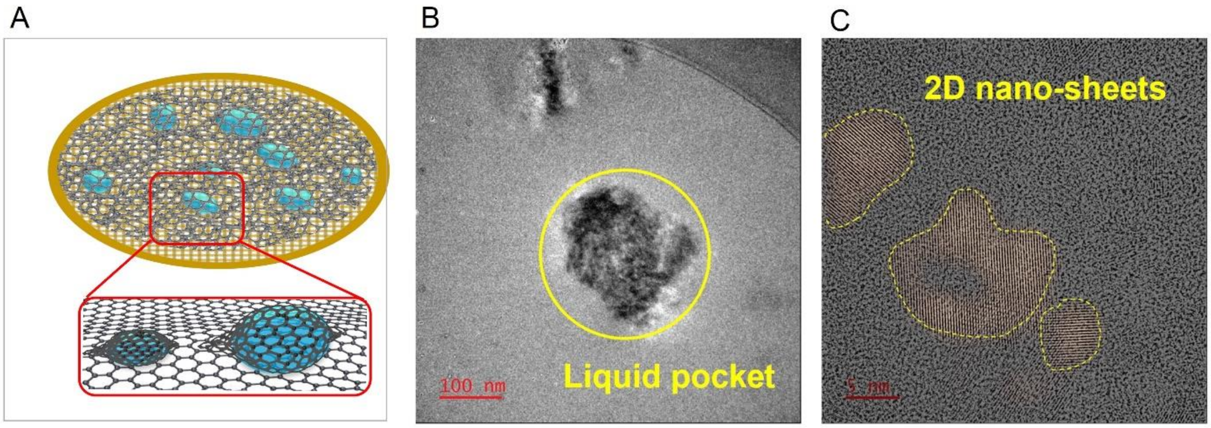

Figure 1. (A) Schematic of the graphene liquid cells. (B) TEM micrograph of liquid pockets between graphene layers on TEM grid. (C) TEM micrograph of 2D crystalline nanosheets formed inside the liquid trapped between graphene layers.

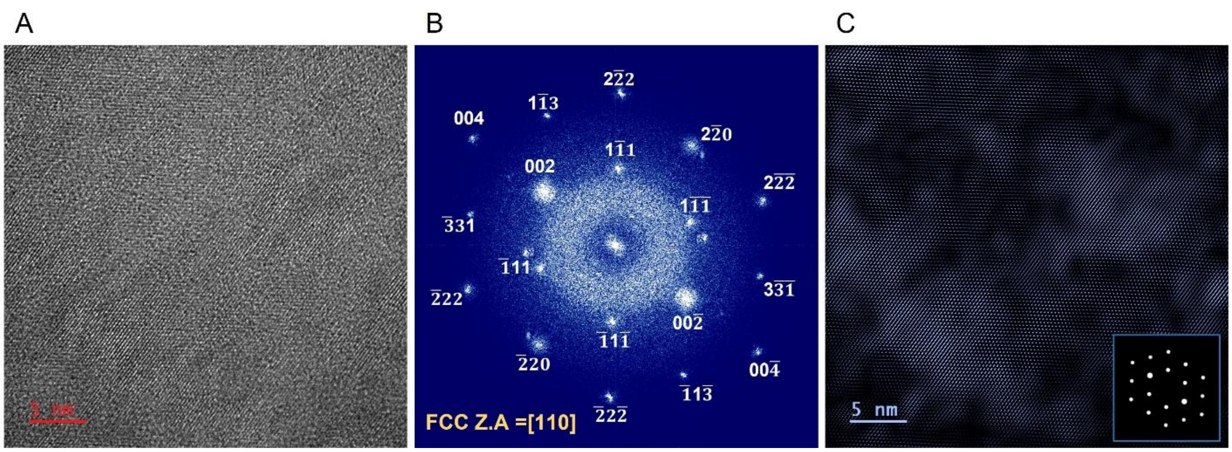

Figure 2. (A) HRTEM image of 2D metal oxide sheets and (B) corresponding FFT of (A) showing the [110]surface of FCC cubic metal oxide is formed. (C) Simulated Inverse FFT(IFFT) of (A) and inset shows the selected masked FFT diffraction spots to generate the IFFT.

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