Atomic imaging of dynamic behaviour at 2D material solid-solid and solid-liquid interfaces

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Transmission electron microscopy (TEM) is used for understanding the local structure of nanomaterials. Although, we are frequently concerned about understanding behaviour during chemical reactions or while undergoing physical processes, most TEM is performed with the sample exposed to high vacuum, which can change the atomic structure of surfaces and interfaces. Unfortunately, commercial in-situ liquid, gas or electrochemical cell TEM imaging holders often severely limit atomic resolution imaging and chemical analysis.

For investigating the chemical reactivity and degradation of 2D materials without exposing them to the TEM vacuum, an effective approach is to encapsulate the sample between two inert and impermeable few-layer 2D sheets (such as graphene or hexagonal boron nitride). This also enables transfer of air sensitive specimens from an inert vacuum or glove environment to the TEM without exposing the material to atmospheric conditions. We have applied this approach to study air sensitive 2D materials, including local point defects, doping and edge structures in a wide range of structures (e.g. CrBr3, GaSe, black phosphorus) (Fig. 1c)[1,2]. We also find that encapsulation with inert 2D materials is an effective route to preserve the delicate surface structure of hydrated 2D materials, enabling the visualization of exchangeable surface cations on few layer clays and micas [3] and a route to understand the changes in atom/ion motion at interfaces where the 2D materials are twisted with respect to each other (Fig. 1d).[3,4]

This 2D heterostructure approach can also be used to investigate solid-liquid interfaces. Building on nanochannel technology developed by the group of Andre Geim (Fig. 1b)[5] we have developed in-situ liquid phase TEM imaging using 2D heterostructure nanochannels. The in-situ 2D heterostructure liquid cell approach provides atomic resolution imaging and analysis and makes it possible to study the earliest stage of chemical synthesis [6]. It also reveals the large differences in adatom adsorption sites on 2D surfaces in vacuum compared to hydrated environments and allows study of dynamic adatom motion at solid liquid interfaces [7]; something that was not previously possible by any technique (Fig. 1a).

[1] Hopkinson, et al. ACS Nano, (2019), 13, 5, 5112; [2] Hamer et al. Nano Lett. (2020), 20, 9, 6582 [3] Zou et al, Nature Materials (2021), 20, (12) 1677; [4] Weston et al, Nature Nanotechnology (2020), 15 (7), 592; [5] A Keerthi et al, Nature, (2018), 558 (7710), 420; [6] Kelly et al, Advanced Materials, (2021) 33, 29, 2100668; [7] Clark et al. https://arxiv.org/abs/2203.04906 (2022);

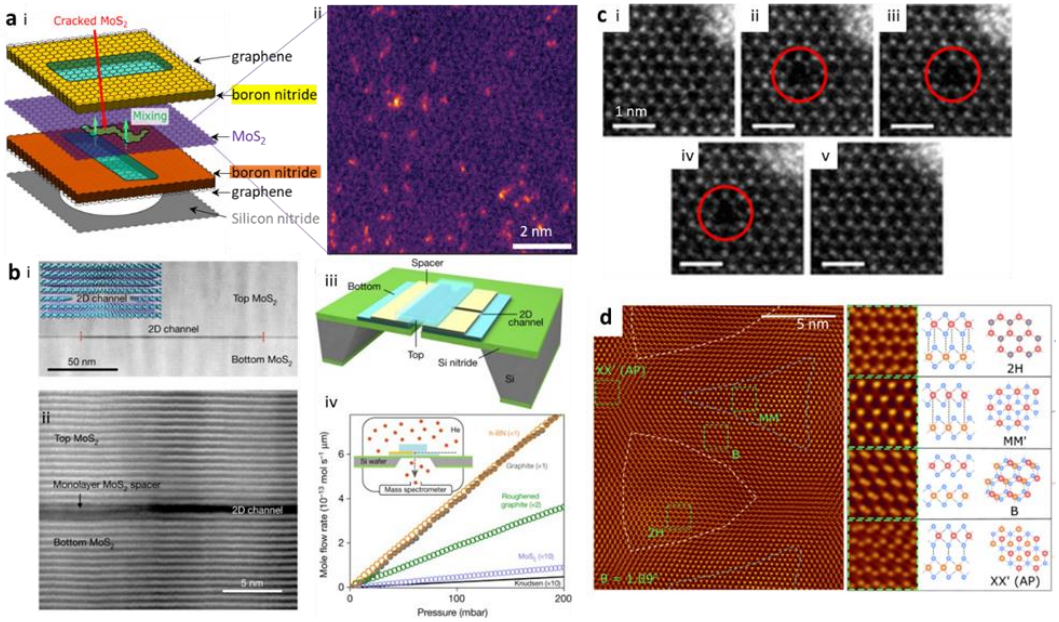

Figure 1 (a) i) 2D heterostructure TEM mixing cell platform [6] (ii) TEM movie imaging dynamics of Pt atoms in liquid [7]. (b) (i-iv) 2D nanochannels and their flow behaviour [5], (c) i-v) Formation and healing of single Se vacancies in InSe [1]. (d) TEM imaging of local lattice reconstruction in a twisted WS_2-WS_2 bilayer [4].