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In-situ TEM Analyses over FIB Lamellae - Investigating High Temperature Conversion of Solution Processed Mo-precursor to MoS_2 Semiconductor Films.

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Currently, single layered MoS_2 nanosheets can be obtained by top-down mechanical[1] or chemical exfoliation[2] and bottom-up chemical vapour deposition[3] process. These are difficult for making viable devices either due to their limited lateral size or being expensive respectively. The current state-of-the-art chemical vapour deposition (CVD) used to synthesize MoS_2 requires vacuum and high-purity precursors rendering it expensive and substrate also plays an important role in determining the film quality. Other methods including chemical bath deposition and hydrothermal synthesis also have limitations and requires improved processing to obtain layered nanosheets.

Here, novel liquid phase synthesis of MoS_2 thin films has been achieved at high temperature. Chemical conversion of the deposited Mo-precursor films on silicon/silicon dioxide substrate to MoS_2 were obtained by annealing in presence of sulfur. To increase the crystallinity of the deposited films, higher processing temperature was required. Annealing temperatures above 750 °C are necessary for the formation of films. This novel wet-chemical synthesis approach for 2D materials allows upscaling, controllable film thickness and can be used for fabrication of various electronic devices i.e. TFTs, photovoltaic cells, sensors, etc.

Uniformity of the layers was proven by FIB (Focussed Ion Beam)) shown in figure 1 (a and b). It is clearly observed that sample annealed at 900°C has uniform layers compared to lower temperatures. The planar view as could be seen in figure 1(b) showed sulfur cluster formation at lower temperatures (was proved via EDX analyses). Larger grain boundaries were formed compared to sulphur cluster formation at higher temperatures (Figure 1-e). To investigate this further in-situ heating was performed in TEM using the DENS heating holder (Figure 2). It is not trivial to observe the transition towards uniform layers in real time. Performing such in-situ heating over lamellae requires sticking the electron transparent lamellae over transparent window (on a MEMS chip – Figure 2). This would be dealt in more details during the presentation [4].

References:

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[3] K. Kang *et al*, Nature 520 (2015), p. 656.

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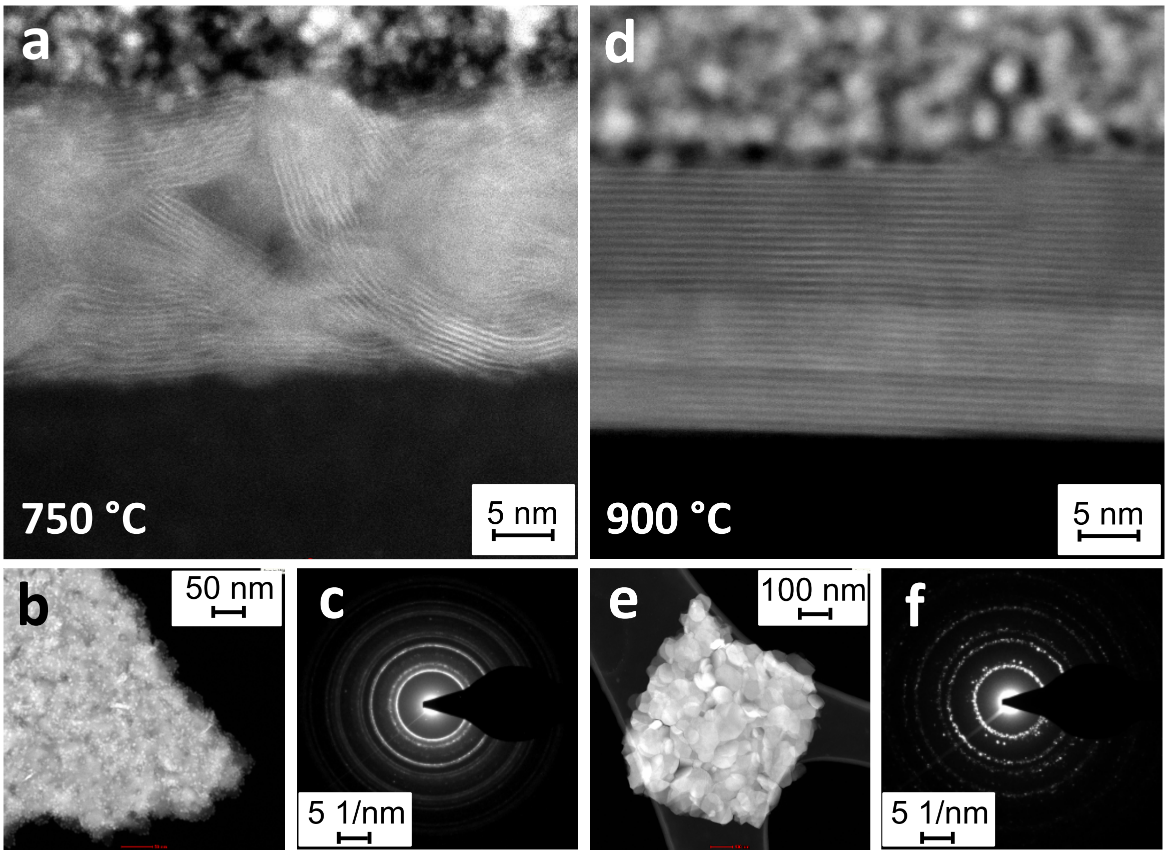

Figure 1. TEM analyses of fabricated MoS_2 films annealed at 750 °C: (a) the cross-section STEM image, (b) planar view reveals no distinct grain boundaries and (c) shows its diffraction pattern. MoS_2 films annealed at 900 °C revealed (d) uniformity in the interlayer spacing with (e) distinct grain boundaries and (f) the diffraction pattern taken from the planar view (e).

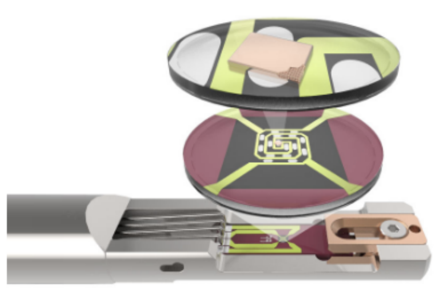

Figure 2. Schematic of the heating DENS Wildfire holder used for in-situ heating.

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