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Synthesis of Monolayer Molybdenum Disulfide and ToF-SIMS Characterization

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Two dimensional (2D) materials have attracted much attention in the past decade. This family of materials includes metallic graphene and semiconducting transition metal dichalcogenides such as MoS_2. Due to the excellent mechanical flexibility, electron transportation property, and transparency, 2D materials have great potential applications in electronics, sensor, and supercapacitor. Mechanical exfoliation and chemical vapor deposition (CVD) are two common methods to prepare the monolayer and a few atomic layer films. Because of the high yield and good quality of the monolayer film, the CVD method becomes the main stream to prepare 2D materials.

We prepared the MoS_2 film with a similar method to reference 1 except for lower heating temperature and simpler cleaning procedure of the substrate. A 1 cm by 1 cm piece of silicon wafer with 500 nm SiO_2 was put in acetone and ultrasonicated for 5 min. Then the substrate was cleaned with oxygen plasma for 20 min. 125 mg of sulfur and 30 mg of MoO_3 were placed into separate ceramic boats. The sulfur was placed at the upstream and the MoO_3 was placed at the center of the tube furnace. The polished-side of the wafer was put down on top of boat containing MoO_3, using two Si wafers as spacers to provide excess ventilation for vapor to escape. Ar gas was flowed through CVD tube (~20 psig) for 15 min at room temperature. Then furnace was set to heat up to 650°C at a rate of 15°C per min and maintained for 15 min. Then it was turn off and the sample was allowed to cool down to room temperature.

The sample was observed with SEM and the image (Fig. 1) shows the MoS_2 grain size varies from a few micrometers to a hundred micrometers. The central area was covered with a continued film and the edge was decorated with triangular monolayer MoS_2 crystalline. The Raman and photoluminescence (PL) spectra were shown in Fig. 2 and Fig. 3 separately. The Raman peak frequency difference between E_{2g} and A_{1g} mode is 20.4 cm^{-1}. The PL peak is located at 668 nm and shows strong intensity. The results show that the film has atomic layer and high quality of crystalline. It indicates that this procedure is highly efficient to prepare high quality MoS_2 atomic film.

To further characterize the film, time of flight secondary ion mass spectroscopy (ToF-SIMS) was conducted on this sample. The ions of Mo^+, S^-, and Si^+ have been mapped and the ion mass images are illustrated in Fig. 4. The results show that molybdenum and sulfur have been uniformly distributed in the film and there is a clear sharp boundary between the MoS_2 crystalline and SiO_2 substrate. It further indicates the high quality of the atomic layer film.

In summary, a large scale of atomic layer MoS_2 film has been prepared with a simple CVD method. The Raman and PL results show the high quality of the crystalline. At the first time, we showed the ion distribution with ToF-SIMS. Depending on the applications, the doping of 2D materials is often conducted to alter the electron transport property. Due to the ppm level of sensitivity and high lateral resolution, ToF-SIMS will be proven to be an informative characterization tool for 2D materials, especially for doped film.

Reference:

[1] A van der Zande et al, Nature Materials **12** (2013), P554.

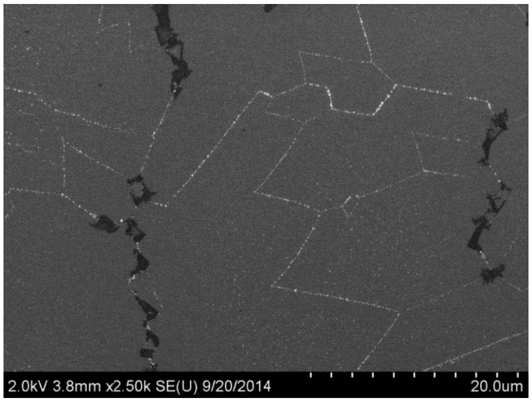

Figure 1. SEM image of CVD grown MoS_2 film

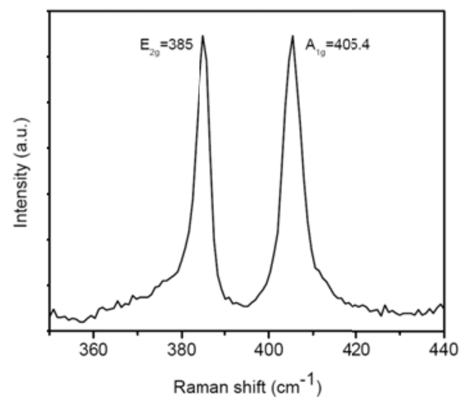

Figure 2. Raman spectrum with excitation of 514.5 nm laser

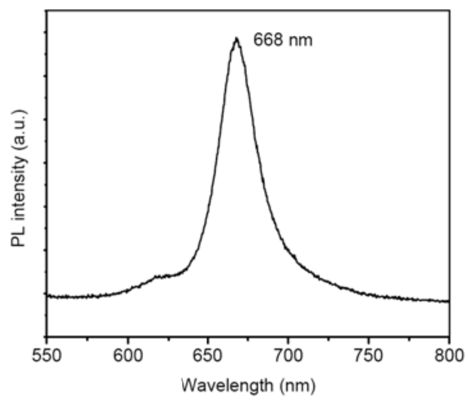

Figure 3. Photoluminescence spectrum with excitation of 514.5 nm laser

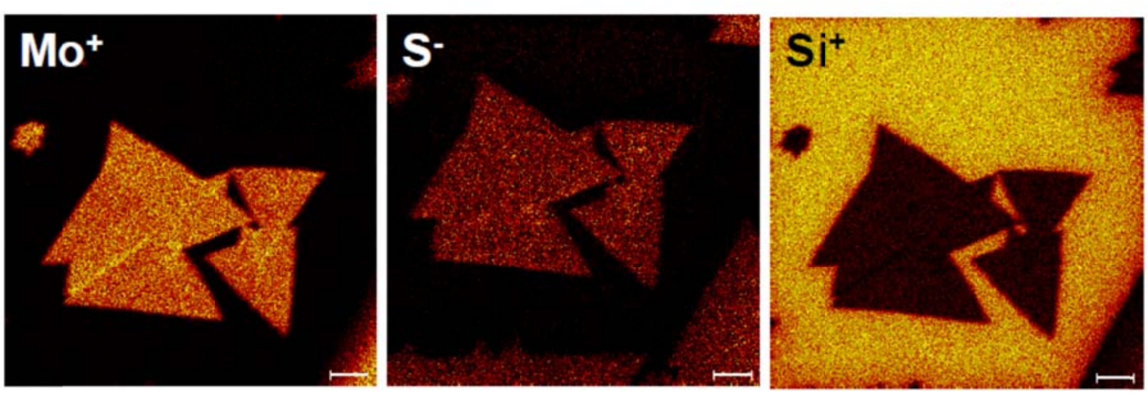

Figure 4. ToF-SIMS images of ion Mo^+, S^-, and Si^+ (scale bar = 10 um)

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