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Structural Analysis of MoS_2 Layers in a 3D Assembly

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Two-dimensional transition metal dichalcogenides (TMDs) are a versatile family of nanomaterials composed by more than forty different layered composites. They have the general formula MX_2 where M is a transition metal of the groups 4-10 and X represents the respective chalcogenide; molybdenum (IV) disulfide (MoS_2), titanium disulfide (TiS_2) and tungsten sulfide (WS_2) are examples of such compounds [1]. These 2D materials have attracted research interest due to their novel layer-dependent electronic and optical properties. For instance, MoS_2 monolayers exhibit a semiconductor behavior with a direct band gap of 1.8 eV, compared with its band gap in bulk, of about 1.29 eV[2]. MoS_2 has been extensively studied as an electrocatalyst for hydrogen production and has been considered as a potential alternative to Pt. Effective production of MoS_2 with specific arrangement at the nanoscale is of particular interest for its applications in catalysis. In this work, the formation of MoS_2 microstructures in pyramidal form was studied using electronic microscopy techniques, such as SEM, FIB-SEM, and aberration-corrected STEM, to understand the organization and growth of these microstructures.

The synthesis process was carried out in a chemical vapor deposition (CVD) reactor; molybdenum trioxide (MoO_3) powder was placed on a SiO_2 substrate and then introduced in an alumina boat. This boat was placed in the center of the reactor. Sulfur powder was placed on a second alumina boat and positioned in the inlet of the furnace, 15 cm away from the center. The full system was heated at 900ºC for 20 minutes and then cooled down to room temperature.

Optical microscopy and scanning electron microscopy images show the formation of MoS_2 pyramidal microstructures with a size of 3.8 micrometers per side (Figure 1b). Raman spectroscopy revealed the characteristic signals of MoS_2, peaks centered at 383 cm^{-1} (E^1_{2g}) and 407 cm^{-1} (A_{1g}). FIB-SEM analysis reveals the layers of the pyramidal microstructures (Figure 1b), the results show a combination of triangular and hexagonal arrangement, due to a complex growth that includes two dislocations that share a common core[3]. High-resolution TEM (Figure 1a) and SADP (Figure 1c) confirm the hexagonal structure of MoS_2.

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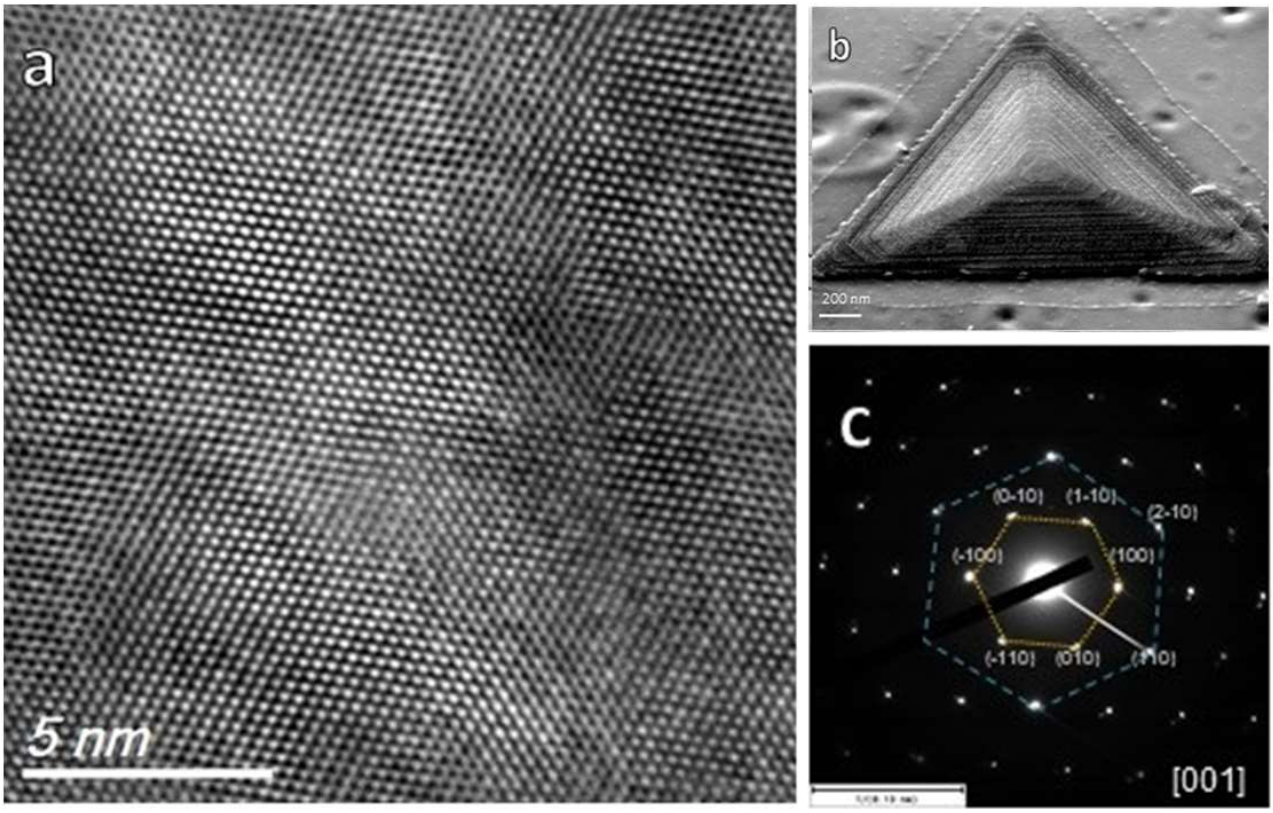

Figure 1. a) High-resolution TEM micrograph of the MoS_2 layers organized in pyramidal microstructures (b). c) Selected area electron diffraction of MoS2 layers show hexagonal structure of MoS_2 in [001] direction.

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