

Electron Microscopy Study of Molybdenum Disulfide Nanowires

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Molybdenum disulfide, a typical layered semiconductor, has been widely used in lubricant, batteries, and catalysis industries. Since the discovery of MoS₂ by wrapping MoS₂ sheets [1], research of fabricating MoS₂ nanotubes/nanowires has attracted many researchers [2,3] due to its unique structure and potential applications. This paper presents the morphological and internal structure study of the MoS₂ nanowires synthesized by sulfurization of MoO_x nanowires.

The fabrication of MoO_x nanowire using an electrochemical step edge decoration method has recently been developed [4]. An overpotential was applied to the highly oriented pyrolytic graphite (HOPG) in aqueous 1mM Na₂MoO₄ solution. Due to the preferred deposition of MoO_x along the step edges of HOPG, long and uniform MoO_x nanowire arrays were formed. These MoO_x nanowires were placed in H₂S atmosphere at elevated temperature for 7hrs and annealed in N₂ or H₂S for 10-40hrs. MoS₂ nanowires on HOPG were directly examined by SEM. After sulfurization, part of the graphite was peeled off, leaving only part of the graphite surface directly contacted with MoS₂ nanowires. A TEM grid was then gently pressed onto the graphite/MoS₂ nanowires boundary region. Some of the nanowires along the boundary were transferred onto the TEM grid for TEM imaging.

The SEM image (Fig.1) shows the synthesized MoS₂ nanowire arrays on HOPG. The nanowires have diameters ranging from 30 to 300nm with lengths up to 500 μm. Higher magnification images show that the wires are uniform along their entire length and continuous after the chemical conversion (Fig. 2 and 3). The nanowire morphology remains unchanged at relatively low annealing temperature (500 – 700 °C in N₂). However, they begin to break into nanoparticles at higher temperature (>800 °C in N₂).

The TEM results show that the internal structure of the nanowires displayed an ordered transition upon different sulfurization conditions. The low magnification TEM image (Fig. 4) shows the overall morphology of a nanowire, and is quite similar to that revealed by higher magnification SEM image (Fig. 3). At higher magnification as shown in Fig. 5, the nanowire appeared to consist of ribbon-like crystals after sulfurizing in H₂S at 500°C for 7hrs and annealed in N₂ for additional 40hrs. The ribbons usually have less than 10 MoS₂ atomic layers (002 plane), and are randomly interweaved with each other. The crystallinity was greatly improved after annealing at 700°C in H₂S. More than 100nm long and rather preferred oriented MoS₂ ribbons/grains can be clearly seen around the edge of nanowires in the high resolution TEM image (Fig. 6). In general, individual ribbon inside the wire has 20-40 atomic layers, which is much larger than those annealed at 500°C in N₂. Although the orientation of the ribbons was still randomly interweaved inside the wires, initial TEM results suggest that layered nanowire with preferred orientation along the long axis could be formed by changing the annealing conditions. More research is in progress.

Reference

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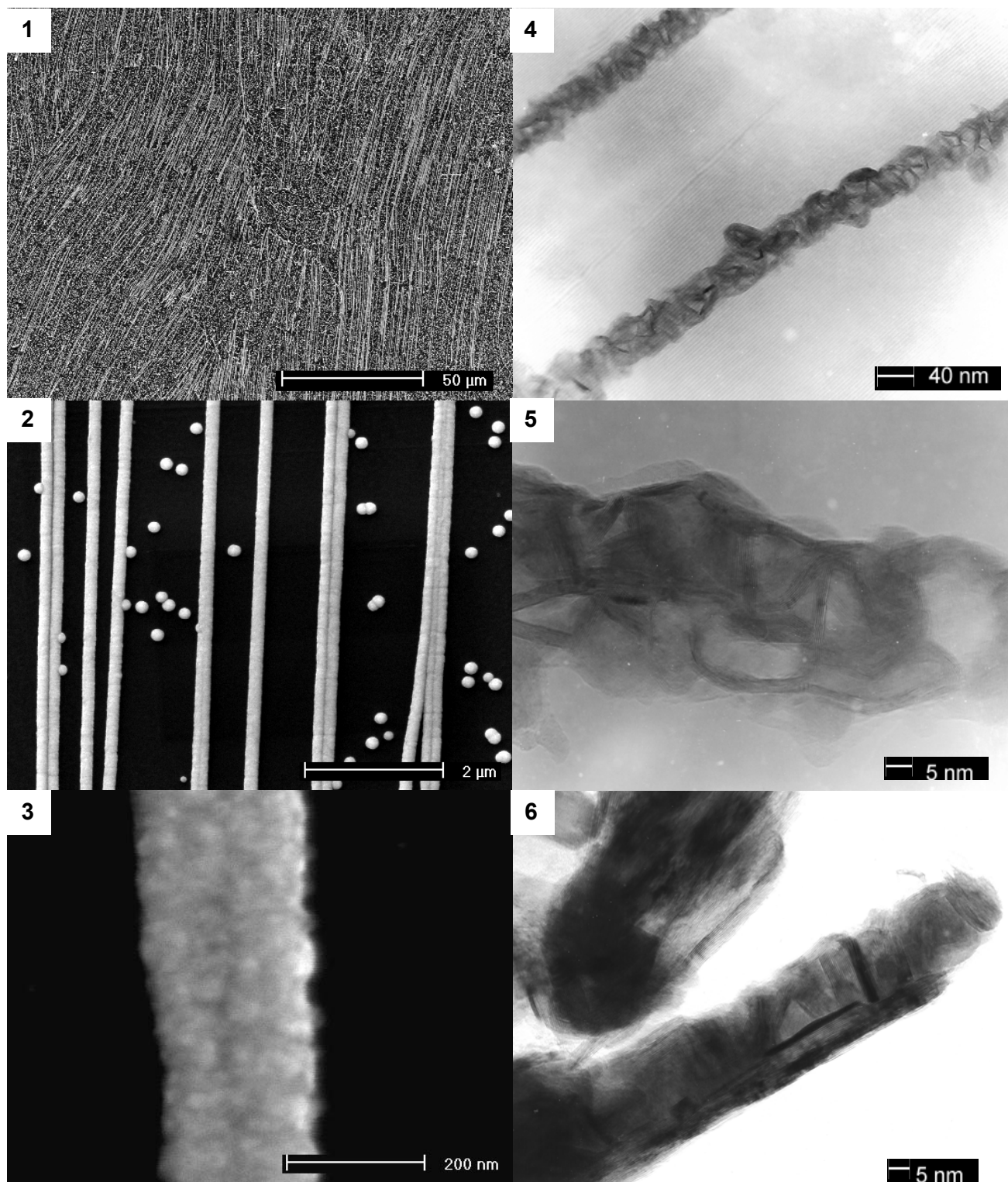


Fig. 1. Low magnification SEM image showing synthesized MoS₂ nanowire arrays on HOPG.

Fig. 2. SEM close-up view of MoS₂ nanowires.

Fig. 3. Higher magnification SEM view depicting nanowire with a rough surface.

Fig. 4. TEM micrograph of a MoS₂ nanowire.

Fig. 5. HREM image showing fiber-like MoS₂ randomly interweaved each other.

Fig. 6. HREM image depicting lattice fringes of somewhat lath-like MoS₂ crystals.