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Production and characterization of $MoSe_2$ nanotubes by electron irradiation

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ABSTRACT

In this work, we report the production of $MoSe_2$ (molybdenum diselenide) nanotubes formed by irradiating the samples with high doses of electron irradiation. The irradiation was performed on a 2 MeV Van de Graaff accelerator at the following conditions: voltage 1.3 MeV, current 5μA current, dose rate 25 kGy/min, and total dosage 1 MGy. We observed well-defined nanotubes of several nm long and few nm wide, which suppose to be hollow and capped at one end. As the level of irradiation was increased to 1 MGy, elongated onion-like structures were observed.

INTRODUCTION

Carbon nanotubes were discovered by Iijima [1] in 1991, which prompted numerous studies because of their superior mechanical property [2] and unique electronic behavior [3]. For example nanotubes are expected to have a high strength-to-weight ratio [4] which is advantageous in advanced composites to be used in high performance materials such as aircraft frames. The small dimensions of the tubes shows promise for use as gas adsorption medium [5,6], a field emitter for use in flat-panel display [7], nanoscale electronic devices [8], and lately they have been used as microscope probes [9].
In 1992, Ugarte [10] reported that electron irradiation of carbon soot produced onion-like structures. Ugarte obtained such structures under a high flux of electrons in a transmission electron microscope (TEM).
The graphite structure is also showed by metal dichalcogenides, represented by MX_2 (M = Mo, W, Nb, etc., whilst X = S, Se, Te, etc.). In our case $MoSe_2$, which its layered structure is depicted in Fig. 1. Still all the members of these family might present similar behavior and possible formation of nanotubes and onion-like structures, only MoS_2 and WS_2 compounds and few variation of them, have been extensively synthesized up to now. Lately, Galván et el. reported nanotube formation on $MoTe_2$ [11], WSe_2 [12] and $NbSe_2$ [13].

EXPERIMENTAL METHOD

In this work we report the formation of nanotube structures in $MoSe_2$ (molybdenum selenide) also known as Drysdallite. The initial samples were obtained from commercial

powders (Alfa-Aesar 99.9 % pure) of $MoSe_2$. Afterwards, the material was irradiated with electrons on a 2 MeV Van de Graff accelerator (High Voltage Engineering Corporation). The irradiation conditions were the following: 1.3 MeV voltage, 5 μA current, dose rate 25 kGy/min, total dosage 1 Mgy. The dosimeter used was radiochromic films (FWT-60) from Far West Technology. X-ray analyses were performed in a Philips XRD/X'PERT system using Cu Kα radiation at 40 KV voltage and 45 mA current.
For Transmision Electron Microscopy (TEM) observations, samples were ground in an agate mortar and placed on carbon coated copper grids. The microscope used was a JEOL JEM-2010 with a point to point resolution better than 0.19 nm.

RESULTS AND DISCUSSION

Figure 1 depicts the layered structure for $MoSe_2$. The repeated motif in $MoSe_2$ is made of a sandwich composed by three layers of Se-Mo-Se with covalent bonds between the atoms of each plane and weak Van der Waals bonds holding adjacent selenium sheets together.

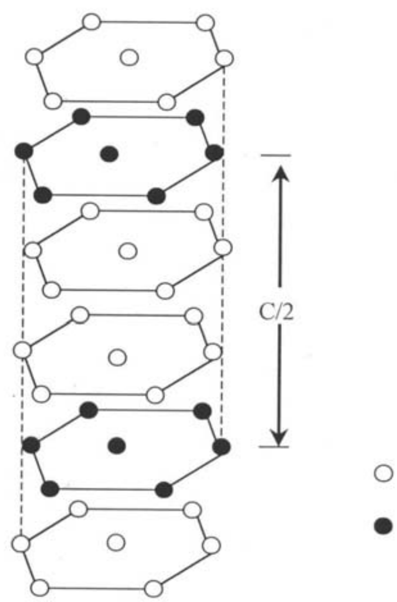

Figure 1 Layered structure for $MoSe_2$. Open circles represent Se atoms while close circles represent Mo atoms respectively.

Figure 2 shows an electron micrograph of a huge nanotube produced by irradiation. The distance in between planes has been measured and is in the order of 0.6240 nm, approximately c/2 value (0.646 nm) corresponding to [0 0 2] orientation corresponding to the characteristic spacing of the reflecting planes for $MoSe_2$ (JCPDS X-ray diffraction card No. 29-0914 [14]).

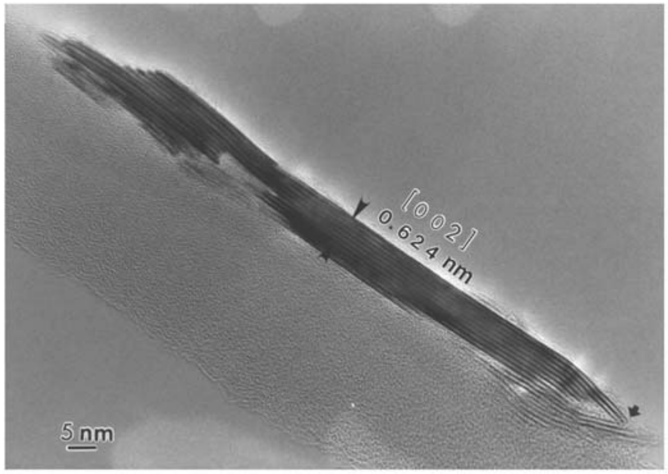

Figure 2 Electron micrograph showing a tubular crystal of $MoSe_2$.

Figure 3 depicts a transmission electron micrograph showing the formation of more tubules. White arrows identify multi wall nanotubes.

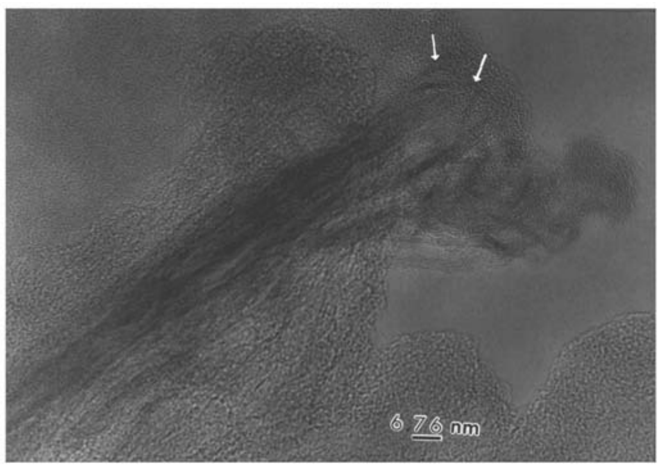

Figure 3 Electron micrograph showing the formation of more tubules.

Figure 4 depicts an electron micrograph showing the typical polyhedral forms reported in another samples like WS_2. The arrowheads indicate inter plane separation.

Figure 4 Electron micrograph showing the typical polyhedral forms encountered in WS_2.

Figures 5 shows an electron micrograph of another area of the sample. White arrows indicate nanotube formation.

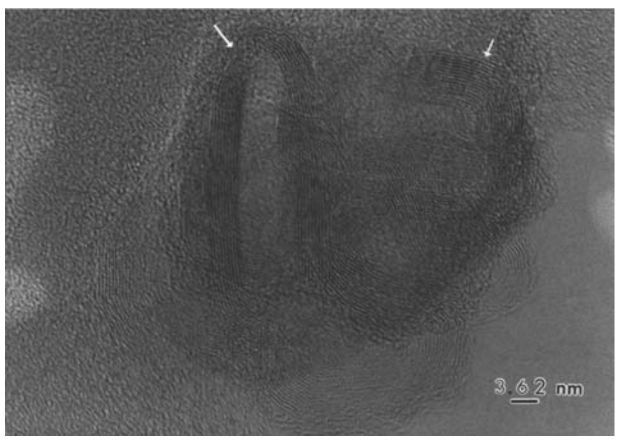

Figure 5 Electron micrograph showing nanotube formation.

Figure 6 shows an electron micrograph showing that electron irradiation form capricious structures. The black arrow indicates the bending of the straight nanotube. This bend nanotube could be considered a likely candidate for a ***Schottky barrier.***

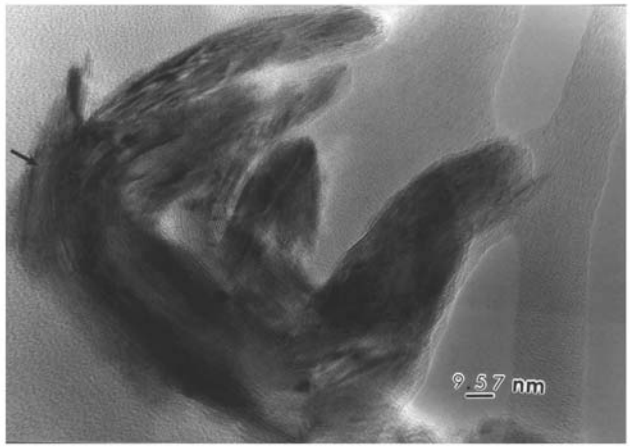

Figure 6 Electron micrograph showing that electron irradiation produces capricious structures.

Figure 7 shows an electron micrograph of a bigger area of the sample. Black arrows indicate capped nanotubes at one end. White arrow identifies an onion-like structure.

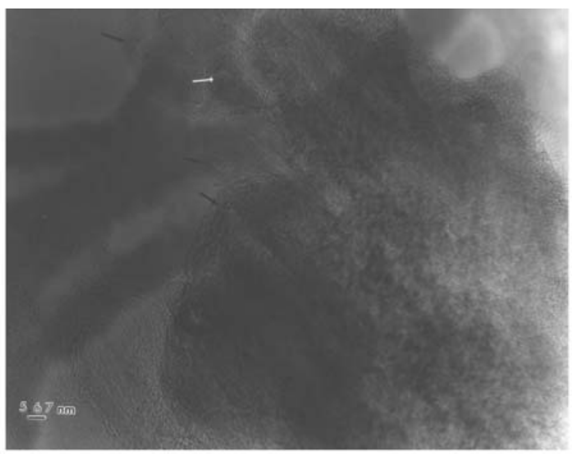

Figure 7 Electron micrograph showing a bigger area of the sample.

CONCLUSIONS

We have shown the production of nanotubes of different sizes, onion-like structures of $MoSe_2$ by electron irradiation. Although, our production method lacks of uniform distribution of these structures through out the matrix, the production method is not as expensive as other reported methods of production.

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