

Arsenic Sulfide Nanowires

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Introduction

Arsenic trisulfide (As_2S_3) is a technologically significant material because it is optically transparent from the visible to the IR (e.g. $\sim 700\text{nm} - 11.5\mu\text{m}$), semi-conducting, amorphous, optically non-linear, and many of its properties are photo-modifiable. It is a member of the chalcogenide family (e.g. containing S, Se, or Te) which all have similar properties.

Key Results

1. Amorphous arsenic sulfide nanowires were deposited on an un-catalyzed, amorphous, fused silica substrate via a sublimation-condensation process (Fig. 1).
2. The morphology of the deposition varied from a continuous thin film, to faceted crystals, to discontinuous micron-sized droplets, to nanowires along the length of the ampoule wall, which was subjected to a temperature gradient. (Fig. 1).
3. The composition and amorphous nature of the nanowires was characterized using scanning electron microscopy (SEM), transmission electron microscopy (TEM), selected area diffraction (SAD), energy dispersive spectroscopy (EDS), x-ray diffraction (XRD), and Raman spectroscopy. (Fig. 2, 3, 4).
4. Microstructural control of the deposition process was achieved by variation of initial ampoule pressure, substrate temperature, and substrate surface treatment. (Fig. 5, 6, 7).
5. Detailed scanning electron microscopy (SEM) was used to construct a pseudo-phase diagram that illustrates the variation of deposition microstructure with initial reaction vessel pressure and substrate temperature.

Methods

As_2S_3 was manufactured in an evacuated and sealed glass ampoule, which was heated in a rocking furnace to induce passive mixing. A small amount of this glass ($\sim 1\text{-}2\text{g}$) was then placed into an evacuated quartz ampoule, which was inserted into a top-loading furnace at $\sim 465^\circ\text{C}$. The top half of the ampoule remained outside of the furnace and was therefore subjected to a low-temperature gradient with the cold end near 50°C . Utilizing the large temperature gradient inside of the ampoule, a sublimation/condensation process was observed and the As_2S_3 deposited on the cooler, top region of the ampoule in a brilliant red to yellow color gradient from the hot to cold regions, respectively, as seen in Fig. 1. In this cooling temperature gradient, a wide array of micro and nanostructures were observed (see Fig. 1, 2, 5-7).

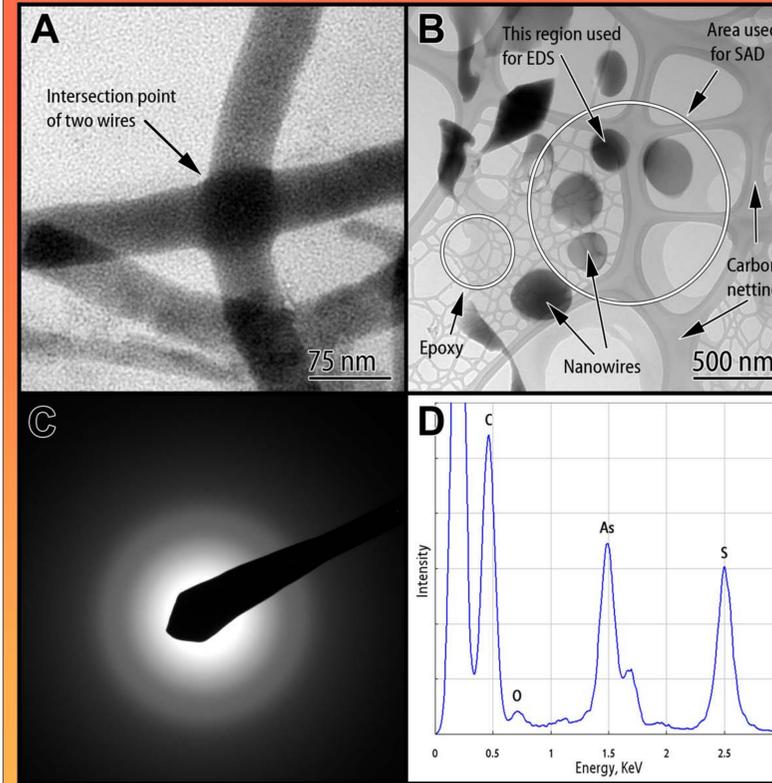


Figure 2. TEM bright field micrographs of arsenic trisulfide (As_2S_3) nanowires. "B" is a cross-section of the wires as well as a view of the TEM sample preparation material. "C" is an SAD pattern from the circled region in "B" indicating that the material was amorphous. "D" shows EDS analysis as performed on spot as seen in "B" indicating that the nanowire is composed of As and S.

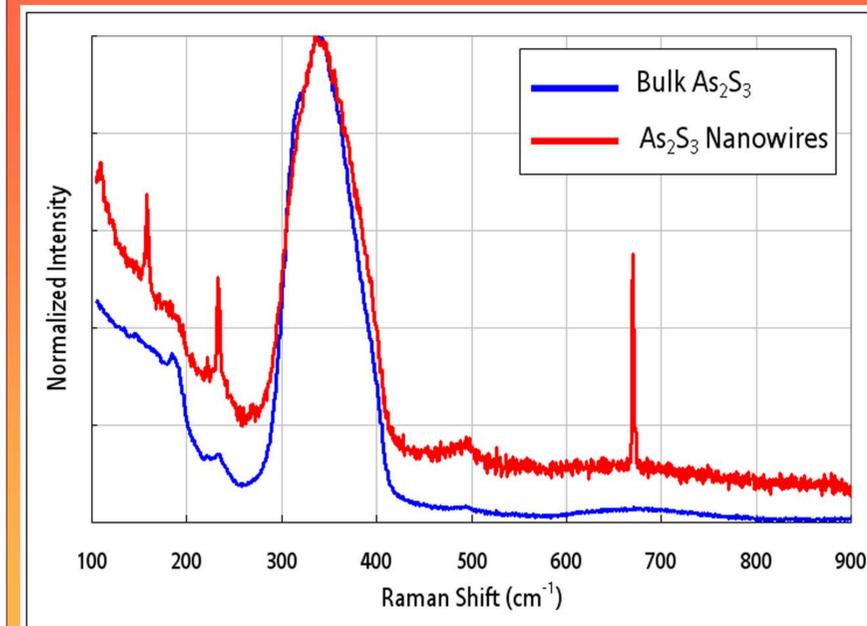


Figure 3. Raman spectroscopy performed on both bulk and nanowire As_2S_3 .

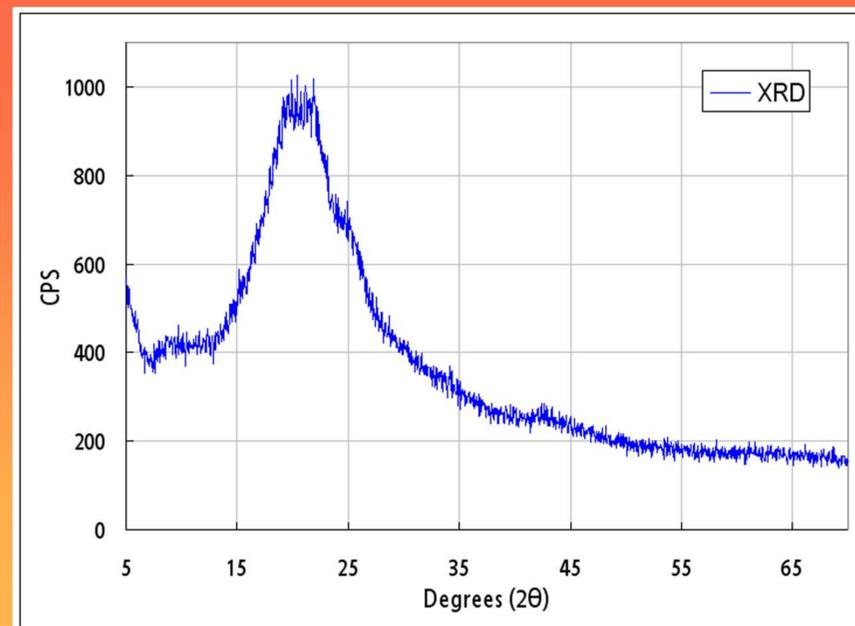


Figure 4. X-ray diffraction on As_2S_3 nanowires showing that they are amorphous.

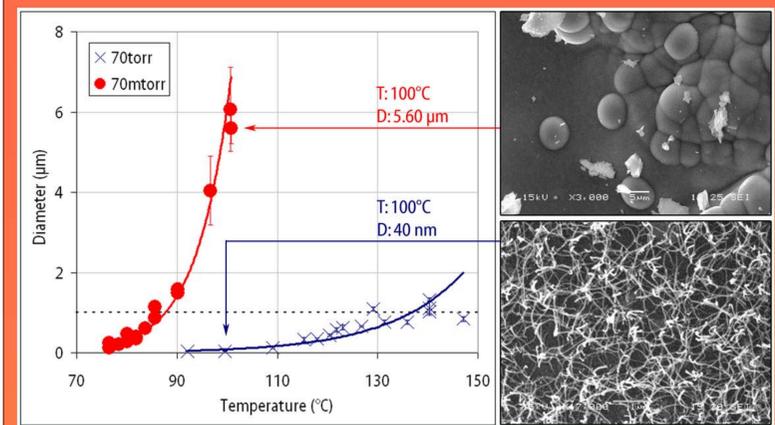


Figure 5. Demonstration of micro-nano structural control with changes in initial pressure of the ampoule (i.e., 70 torr, 70 mtorr).

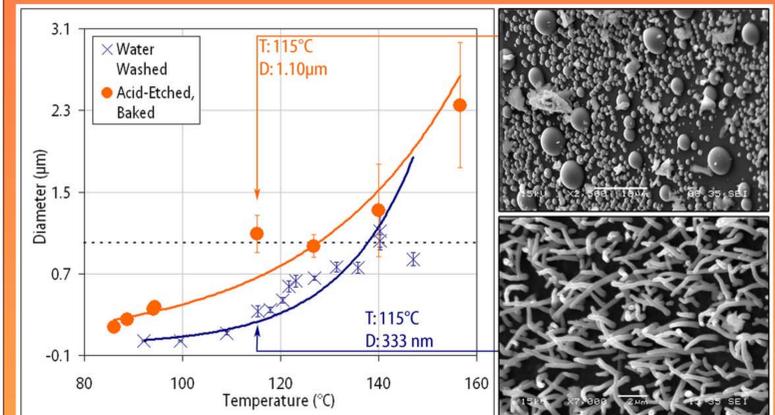


Figure 6. Demonstration of micro-nano structural control with substrate surface control using different cleaning methods (i.e., acid, water).

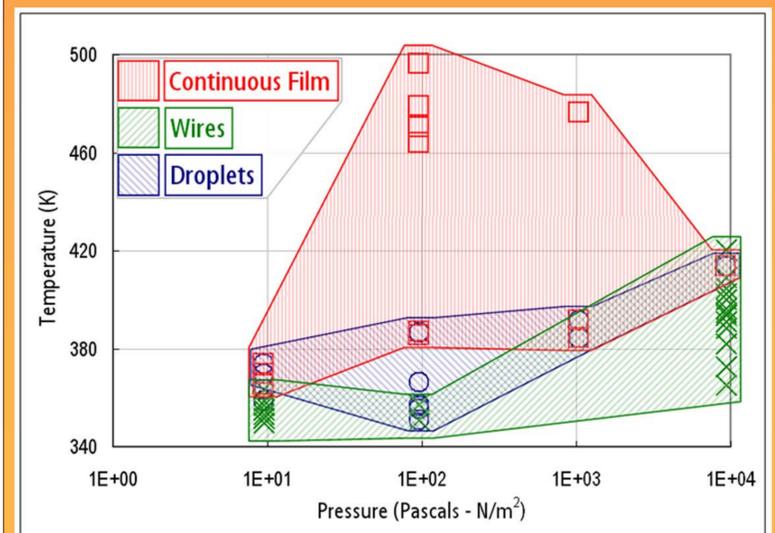


Figure 7. Pressure-temperature spaces in which certain structures were found over the course of various experiments.

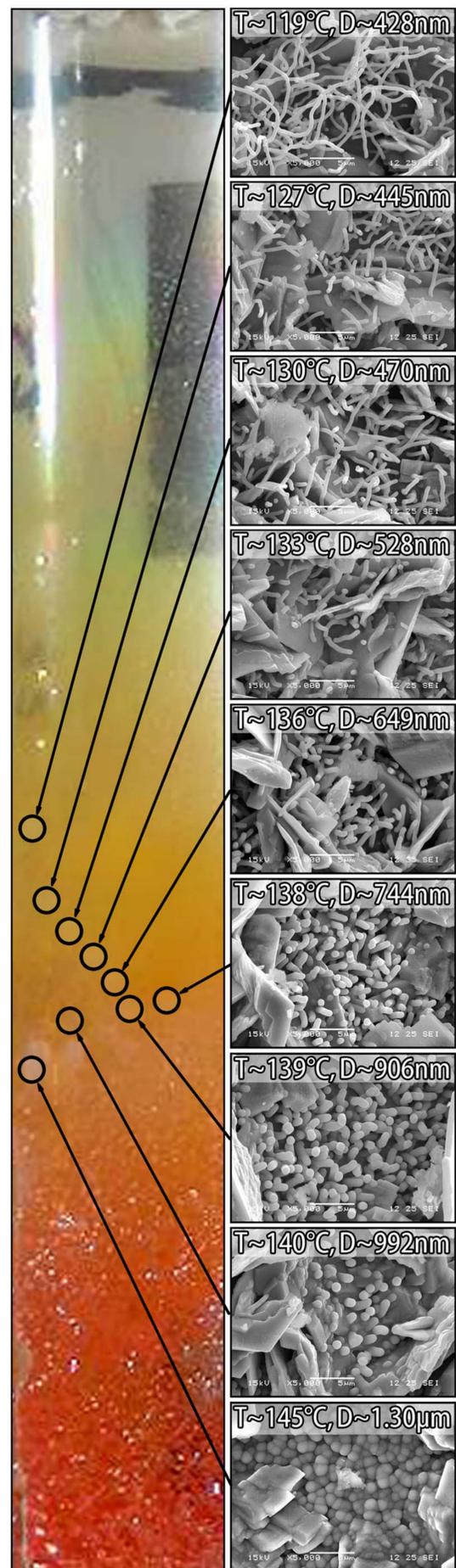


Figure 1. Optical picture of As_2S_3 deposition on glass tube and selected SEM micrographs showing variation of microstructure with substrate temperature.