PbS sub-micron structures with anisotropic shape: Ribbons, Wires, Octa-pods and Hollowed Cubes

M. Bashouti and E. Lifshitz

Department of Chemistry and Solid state, Technion Institute, Israel institute of Technology, Haifa, 32000

Preparing the uni-precursor, \([\text{Pb(S}_2\text{CNEt}_2)_2]\)_2, molecules: The uni-precursor molecules were prepared by mixing 3.321 grams of lead nitrate [Pb(NO\,3)] (dissolved in 50 mL of water) with 2.2531 grams of sodium diethyldithiocarbamate hydrate [Et_2NCS_2Na*3H_2O] (dissolved in 50 mL of water). The chemical product was filtered, dried by a warm carbon disulfide at 40\(^0\)C, and was left in vacuum for 24 hours. NMR analysis of the uni-precursor, shown in the Figure below, confirmed a high purity product. NMR spectra were recorded on Avance 300 spectrometer. Chemical shifts for \(^1\text{H-NMR}\) are referenced to internal solvent resonances and are reported relative to tetramethylsilane. \(^1\text{H NMR}\) (300 MHz, CDCl_3): \(\delta\) 3.74 (q, J = 7 Hz, 4H, NC\,H_2); 1.28 (t, J = 7 Hz, 6H, CH_3). This NMR found to fit the literature NMR\(^2\).

X-ray analyses: The crystallographic properties of the various PbS sub-micron structures were characterized by the use of an X-ray diffraction. Representative examples are shown in Figure 1, suggesting crystallographic rock salt structure symmetry with a unit cell dimension of 6.12Å relevant to a rock salt structure, with space group of Fm\(\overline{3}\)m. This means that the produced shapes initiate from the same nuclei, however, they develop differently, according to the growth external conditions. The XRD of the cubes concides solely with the conventional X-ray data card 00-005-0592, while the XRD of the other shapes are similar to the cubic structure, but, show some deviation, that may be associated with the existence of excess sulphur or excess lead at different facets.

---

(1) To whom correspondence should be address, Department of Chemistry and Solid State Institute, Technion, Haifa 32000, Israel. E-mail: (E. Lifshitz, ssefrat@tx.technion.ac.il).

Figure 1: X-ray diffraction patterns of PbS ribbons (A), Cubes (B), Octa-pods (C) and wires (D).

HR-SEM measurements of the PbS wires

Figure 2 show representative HR-SEM images of PbS wires, obtained in ethylenediamine solution, at low temperatures (80°C), after a duration of about 40 minutes. The XRD of those wires are shown in Figure 1D.
Figure 2: HR-SEM images of PbS wires

**Energy dispersive analysis of X-ray (EDAX):** Atomic percentages of the Pb and Se elements in the PbS crystals were derived from the EDAX, suggesting a consistence appearance of a Pb:S molar ratio of 1:1 in the wires, ribbons, octa-pods and cube structures. Representative example of the EDAX is shown in Figure 3 and in Table 1 of this supporting information.

![EDAX spectrum of PbS sub-micron structure](image)

Figure 3: EDAX spectrum of PbS sub-micron structure

<table>
<thead>
<tr>
<th>Element</th>
<th>Atom</th>
<th>Element weight</th>
</tr>
</thead>
<tbody>
<tr>
<td>S-K</td>
<td>50.90</td>
<td>15.32</td>
</tr>
<tr>
<td>Pb-M</td>
<td>49.10</td>
<td>84.68</td>
</tr>
</tbody>
</table>

Table 1: Atomic percentages of Pb, S constituents in PbS sub-micron crystals, as derived from the EDAX data.