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ORIGINAL ARTICLE

Synthesis of WS_2 and WSe_2 nanowires on stainless steel coupon by reaction under autogenic pressure at elevated temperature method

R. Yuvasravan[2] · G. Apsana[2] · P. P. George[1,2] · I. Genish[1] · Shirly ben-david Maklouf[1] · Y. Koltypin[1] · A. Gedanken[1]

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Abstract The novel flower-like WE_2 (E = S or Se) nanoflakes are synthesized and the growth of WS_2 and WSe_2 nanowires on stain less steel coupons (SSC) is observed by reaction under autogenic pressure at elevated temperature technique between the metallic tungsten and chalcogen powders at 750 °C for 3 h. Powder X-ray diffraction, scanning electron microscopy and transmission electron microscopy are used to characterize all reaction products, viz., neat WS_2, WSe_2 powder, WS_2/SSC and WSe_2/SSC (stainless steel coupon). The photoluminescence spectrum of WS_2 and WSe_2 samples are also reported. In addition, the direct use of metals as precursors will devoid the harmful effects of organometallic precursor.

Keywords Deposition · Nanoparticles · WS_2 + WSe_2

Introduction

Transition metal dichalcogenides (TMDCS) have been explained by scientists to exhibit excellent electronic, magnetic and electrochemical properties which have generated interest for energy-associated device applications for example solar cell and lithium batteries.(Shi et al. 2015) One-dimensional nanostructures are receiving increasing attention because of their potential applications in electronics and photonics (Zhang et al. 2007). Fabrication of nanoflakes, nanorods, and nanoribbons has been demonstrated for elemental semiconductors, such as silicon and germanium compounds (Wang et al. 2000; Liang et al. 2001). Tungsten chalcogenides, WE_2 (E = S or Se) are very useful as a high-efficient solid lubricant (Erdemir and Bhusan 2001; Voevodin et al. 1999; Voevodin and Zabinski 2000) and catalyst (Wu et al. 2004; Breysse et al. 1984). In addition tungsten chalcogenides also have wide range of applications such as photoelectrochemical cells (PEC), photovoltaic (PV) solar cells (Tributsch 1977, 1978; Tributsch and Bernnett 1977; Jäger-Waldau et al. 1994; Niu et al. 2014; Matthaus et al. 1997; Srivastava and Avasthi 1985), tribology and also as electrode material in lithium ion batteries (Martin-Litas et al. 1999, 2002). The current methods used to prepare the WS_2 and WSe_2 as thin films on a variety of substrates are DC and RF sputtering method (Regula et al. 1996; Ellmer et al. 1997), sulfurization of ion beam sputtered WO_3 thin films (Genut et al. 1992; Ennaoui et al. 1997), pulse laser deposition (Zabinski et al. 1994), vapor deposition method(Huang et al. 2014), electrodeposition (Devadasan et al. 2001), chemical bath deposition (Chatzitheodorou et al. 1988). Pol and his coworkers had demonstrated the synthesis of WS_2 breeds embedded in carbon and WSe_2/C nanocomposite by employing the RAPET technique (Pol et al. 2007, 2008). In the current article, the synthesis of WS_2 and WSe_2 nanocrystals with novel flower-like pattern of radially aligned nanoflakes via a RAPET method is demonstrated.

The synthesis of WS_2 and WSe_2 nanowires on the SSC is also reported in this paper. There is no literature about the synthesis of WS_2 and WSe_2 nanowires on the SSC. Intense

✉ P. P. George
georgepanikulangara@yahoo.in; georgepp@mits.ac.in

✉ A. Gedanken
gedanken@mail.biu.ac.il

[1] Department of Chemistry and Kanbar Laboratory for Materials, Center for Advanced Materials and Technology, Bar-Ilan University, Ramat-Gan, Israel

[2] Department of Chemistry, Madanapalle Institute of Technology and Science, (Affliated to JNTUA, Ananthapur and Approved by AICTE, New Delhi), Post-Box No.14, Angallu, Madanapalle 517325, AndhraPradesh, India

research has been focused on the deposition of materials on variety of substrates due to application in various fields such as antireflection coatings, optical filters, solar cells, photoconductors, sensors, etc. (Pawar et al. 2011). In this paper, RAPET method is used to deposit the WS_2 and WSe_2 nanowires on the SSC. This hybrid material cannot only combine the uses of tungsten sulfide or tungsten selenide nanoparticles and SSC but also may result in new properties which might have potential applications in the nanoscale electronic devices and catalysis. The SSC coated with the WS_2 and WSe_2 nanocrystals have properties different from that of neat WS_2 and WSe_2 nanocrystals. In addition, these inorganic coatings of WS_2 and WSe_2 nanocrystals saved SSC from corrosion. The main aim in this paper is the production of WS_2 and WSe_2 nanomaterials without carbon as well as on flat surface like SSC via a RAPET reaction of metallic tungsten and S or Se. We also include the characterization and PL studies of WE_2 (E = S or Se) nanoflakes.

Characterization

The XRD patterns of pristine WS_2 and WSe_2 nanoflakes are recorded using a Bruker D8 diffractometer with Cu Kα radiation. The morphologies of the WS_2/SSC, WSe_2/SSC, WS_2 and WSe_2 are studied by a scanning electron microscope (SEM). Transmission electron microscopy (TEM) studies are carried out on a JEOL 2000 electron microscope. High-resolution TEM (HRTEM) images are taken using a JEOL 2010 with a 200 kV accelerating voltage. Samples for the TEM and HRTEM measurements are obtained by placing a drop of the suspension from the as-sonicated reaction product in ethanol onto a carbon-coated copper grid, followed by drying under air to remove the solvent. The photoluminescence measurements are performed on a Perkin Elmer Luminescence spectrometer L550B at room temperature.

Results and discussion

Powder X-ray diffraction (PXRD), elemental (C and H) analysis, SEM and HRSEM analysis

The XRD pattern of the product obtained by the thermal decomposition of the reaction mixture of tungsten and sulfur at 750 °C in a closed Letlok cell filled with N_2 atmosphere is presented in Fig. 1a. The X-ray diffraction peaks indexed to the WS_2 polycrystalline structure with hexagonal space group *P63/mmc* with lattice constants $a = 3.1800$ Å and $c = 12.5000$ Å [powder diffraction file (PDF) no. 01-084-1398]. The diffraction peaks at $2\theta = 29.0$, 32.7, 33.5, 39.6 and 49.8 are assigned to (004), (100), (101), (103) and (105) planes of WS_2, respectively. From the (100) diffraction peak, the average interlayer spacing was calculated as 2.73 Å. The average crystallite size for WS_2 was calculated as ca 14 ± 3.5 nm using the Debye–Scherrer equation.

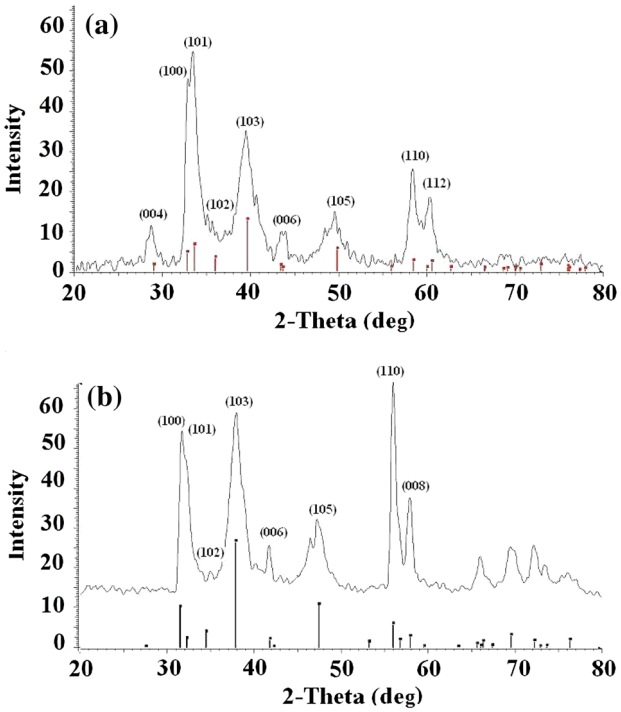

Fig. 1 a–b PXRD pattern of (*top*) WS_2 and (*bottom*) WSe_2 nanoflakes

Figure 1b demonstrates the representative XRD pattern for our as-synthesized WSe_2 nanoparticles. The WSe_2 polycrystalline structure has hexagonal space group *P63/mmc* with lattice parameters of $a = 3.2820$ Å and $c = 12.9600$ Å [(PDF) no. 01-071-0600]. The diffraction peaks at $2\theta = 31.5$, 32.2, 37.8, and 47.4 are assigned to (100), (101), (103) and (105) planes of WSe_2 respectively. From the (100) diffraction peak, the average interlayer spacing was calculated as 2.84 Å. The average crystallite size for WSe_2 was calculated as ca 15 ± 3.5 nm using the Debye–Scherrer equation.

The absence of the impurities from the PXRD Fig. 1a, b indicates that the starting materials introduced in the Letlok cell are used for the formation of crystalline WS_2 and WSe_2, respectively. We had carried out the PXRD measurements of the powderic samples from the Letlok cell after the completion of RAPET reaction.

Morphology

HRSEM measurements

The RAPET reaction between the W and S for 3 h at 750 °C generates the WS_2 powder and their SEM images are shown in Fig. 2a, b. Figure 2a reveals the large-scale

unusual flower-like pattern composed of radially aligned WS_2 nanoflakes. The flower-like pattern usually has the diameters range from 0.66 to 4 μm. Figure 2b shows the interesting feature that these flower-like WS_2 nanocrystals (Fig. 2b) compose of a large quantity of small WS_2 nanoflakes with smooth surface and uniform thickness of about 20 nm. The length of nanoflakes is in the range of 100–400 nm. This type of patterns may find uses in a variety of areas such as the fabrication of advanced electronic and optoelectronic nanodevices.

Figure 3a, b demonstrates the SEM images of WSe_2 powder obtained after the thermal treatment of W and Se under RAPET reaction conditions. Figure 3a shows the flower-like arrangement and is composed of the several nanoflakes of length in the range of 60–200 nm and the mean thickness of 20 nm. Figure 3b is the high-magnification SEM image of one such flower and the flower-like pattern comprising several nanoflakes with the diameter 1.2 μm approximately. Du et al. had synthesized the ZnSe nanoflakes of similar pattern by using the solvothermal method and the thickness of nanoflakes is 40 nm (Du et al. 2007). The length of nanoflakes is in the range of 400–600 nm and the average thickness of nanoflake is in the range of 20–30 nm. Our approach involves only the metallic tungsten, S or Se powders. Although Pol et al. had demonstrated the synthesis of WS_2 breeds embedded in carbon and WSe_2/C nanocomposite by the reaction of $W(CO)_6$ with S or Se under RAPET conditions at 750 °C for 3 h.(Pol et al. 2007, 2008). Our approach yields WS_2 and WSe_2 product without carbon.

TEM analysis of an anisotropic WS_2 and WSe_2 sample

The representative TEM images of the prepared samples of WS_2 and WSe_2 nanoflakes are presented in Fig. 4a, b. From Fig. 4a, the sample appears to have micro-pattern, which consists of tiny WS_2 nanoflakes with an average length of 250 nm. The micro-pattern is having a diameter of 900 nm.

The RAPET technique had afforded the nanoflakes of WS_2. The nanoflake morphology of the current WS_2 nanoparticles differs from the WS_2 nanotubes obtained by hydrothermal route and was carried out by Tremel and his coworkers(Therese et al. 2005). Tremel's synthetic strategy is a complicated procedure and was carried out in two steps. In the first step, synthesizing the WO_3 nanorods and it requires approximately 8 days and then converting the WO_3 nanorods into WS_2 nanotubes. The current RAPET synthetic approach needs very less time for the formation of WS_2 nanoflakes and involves only metallic tungsten powder and sulfur and does not require solvents, or toxic chemicals like H_2S unlike in the Tremel's experimental procedure of WS_2 nanotubes. The WS_2 nanotubes prepared by Tremel's synthetic method have diameter in the range of 150–250 nm and are comparable to those of the WS_2 nanoflakes (250 nm) produced under Letlok reaction conditions.

Figure 4b depicts the part of a WSe_2 particle with many non-separable flakes protruding out. The neat WSe_2 nanoflakes have diameters between 13 and 20 nm, and lengths between 50–100 nm, respectively, and these values are consistent with the SEM measurements. The current RAPET synthetic approach involves a low-cost starting materials, metallic tungsten powder and selenium. The WSe_2 nanoflakes prepared by the current RAPET synthetic strategy have lengths between 50 and 100 nm and are smaller than those of the WSe_2 nanoflakes.(Zhang et al. 2015).

Optical properties of WS_2 and WSe_2 nanoflakes

Transition metal disulfides and diselenide (MS_2 and MSe_2) are of prominent interest for many technological applications. Parkin et al. had synthesized the WS_2 thin film by

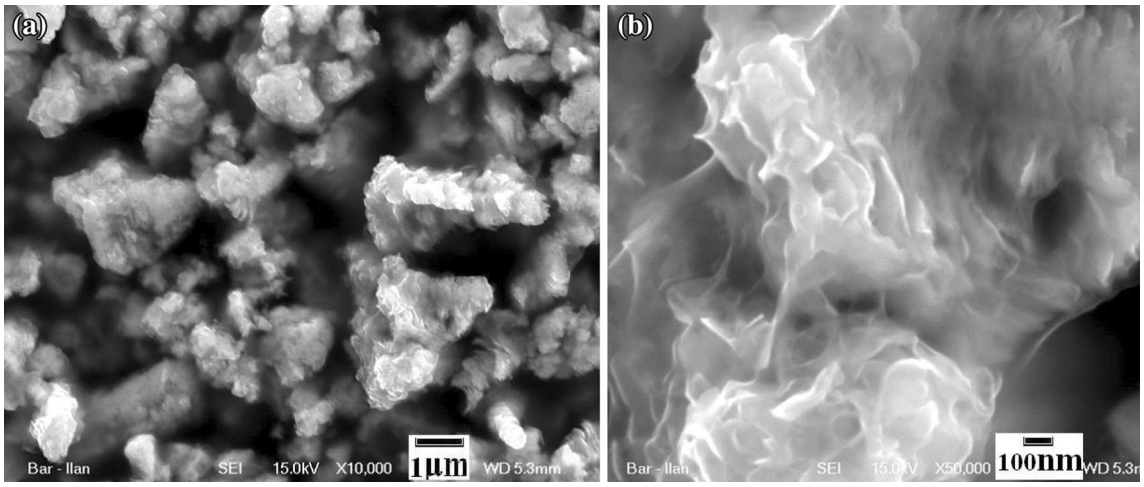

Fig. 2 **a** SEM image of WS_2 at 1000 magnification, **b** SEM image of WS_2 at 5000 magnification

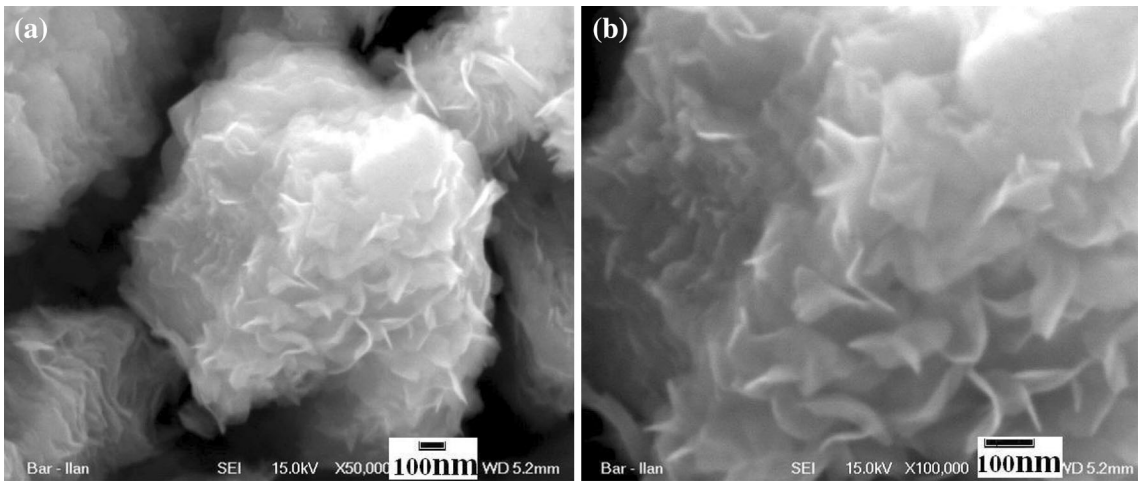

Fig. 3 **a** SEM image of WSe_2 at 1000 magnification, **b** SEM image of WSe_2 at 5000 magnification

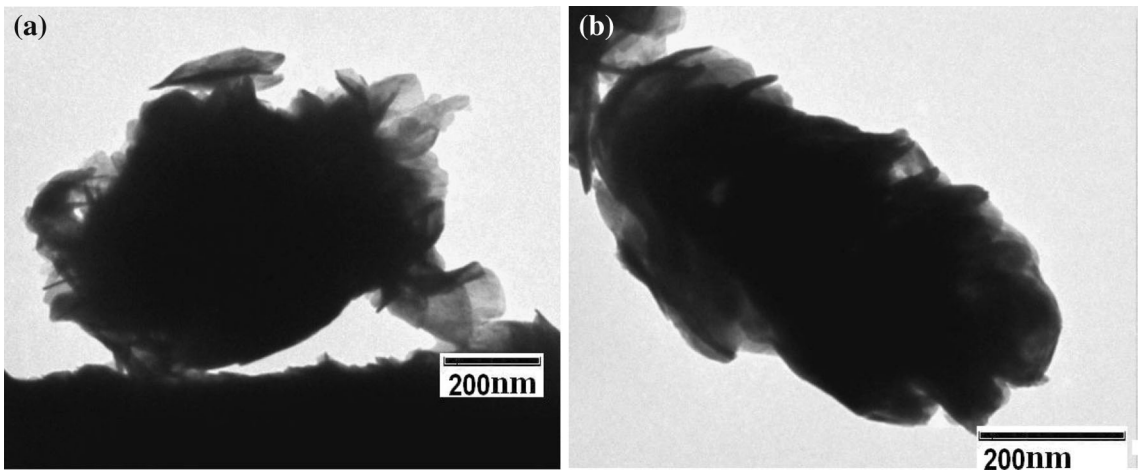

Fig. 4 **a** TEM image of WS_2, **b** TEM image of WSe_2

atmospheric pressure chemical vapor deposition (APCVD) and its optical band gap is 1.4 eV (Carmalt et al. 2003). Figure 5 shows the typical PL spectra of the WS_2 nanoflakes (1) and WSe_2 nanoflakes (2).

Figure 5 shows that the room temperature PL spectrum of the as-prepared WS_2 and WSe_2 nanoflakes is measured with the excitation at 300 nm. Excitonic absorption peaks A and B arise from direct gap transitions around 473 and 588 nm for WS_2 and around 471 and 592 nm for WSe_2, respectively. The energy difference between the A and B peaks is an indication of the strength of spin–orbit interaction.

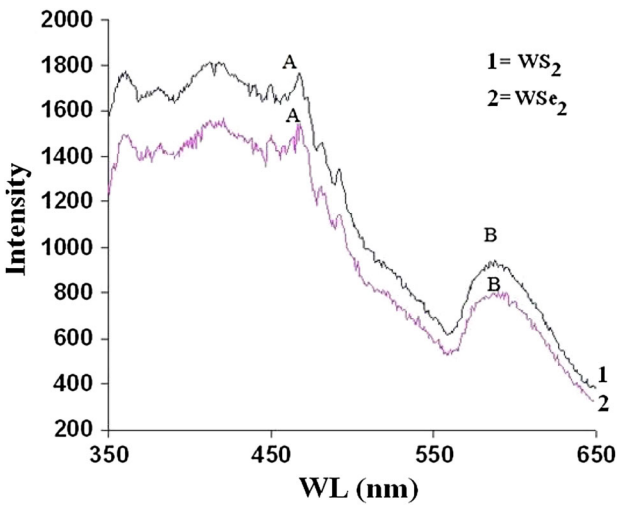

Fig. 5 The photoluminescence spectrum of a (*top*) WS_2 and (*bottom*) WSe_2 nanoflakes at room temperature excited at 300 nm

Discussion

The current RAPET synthesis of WS_2 and WSe_2 nanoflakes by the thermal treatment of mixture of W and S or Se is a straightforward reaction. The synthesis of WE_2

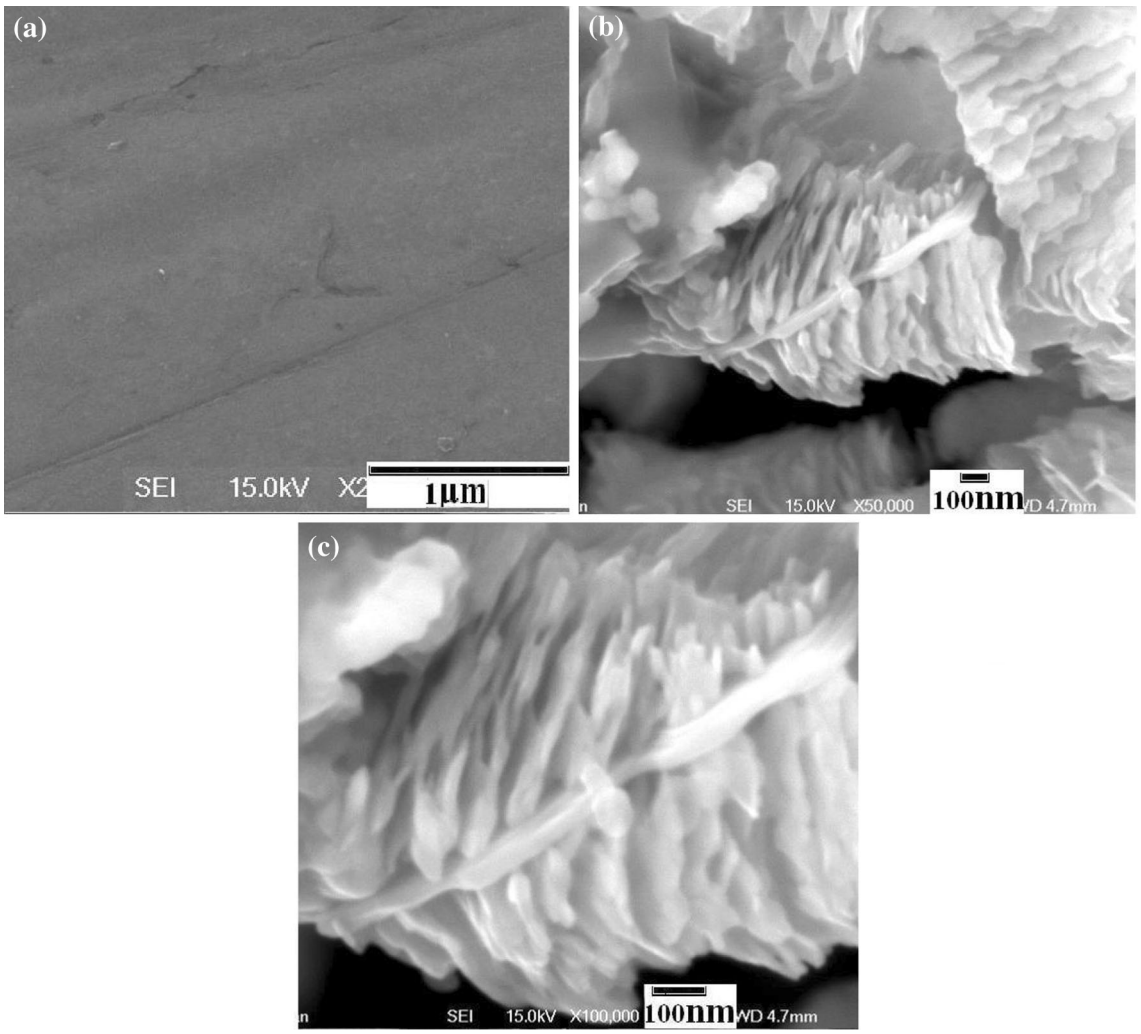

Fig. 6 **a** SEM image of SSC before coating. **b** SEM image of WS_2/SSC. **c** Represents the high-magnification image of WS_2/SSC nanowires

nanoparticles is produced via the reaction between W vapor and the S or Se vapor. The sulfur or selenium has low BP's (445 °C or 685 °C respectively), and are relatively volatile, allowing them to get involved in this RAPET reaction relatively quick. The Letlok cell in our reaction is heated at the temperature of 750 °C is sufficiently hot to initiate the reaction between metallic W and S or Se and resulting in the formation of large number of WS_2 or WSe_2 nucleii in vapor state. After completion of reaction for 3 h, the letlok reactor, starts cooling and the WS_2 or WSe_2 powders are collected. Earlier, Pol et al. had demonstrated the synthesis of WS_2 breeds embedded in carbon and WSe_2/C nanocomposite. However, the structure and morphology of samples obtained in this work differ from the results of similar samples obtained previously in Pol et al. experiments. The difference between the Pol et al. reported results and those discussed here is the use of $W(CO)_6$ in the former and the metallic W in the latter for the preparation of WE_2 [$E = S$ or Se] nanoparticles. Moreover, in the previous results, there is a carbon coverage which requires further treatment to obtain the neat WSe_2 sample. This discrepancy exemplifies the importance of the use of starting materials.

In a controlled reaction, the inorganic coating of WS_2 and WSe_2 nanowires onto pieces of pristine stainless steel coupons are achieved by employing a RAPET method. The growth of WS_2 and WSe_2 nanowires on SSC is fabricated by the thermal treatment of mixture W, S or Se and SSC contained in the Letlok cell at elevated temperature of 750 °C under autogenic pressure for 3 h. The morphologies of the WS_2 and WSe_2 nanowires on SSC obtained are primarily investigated by SEM measurements. Figure 6a represents the neat SSC before the start of a reaction and indicating the clean and smooth surface. SEM images (Fig. 6b) demonstrate the bunch of WS_2 nanowires arranged in the leaf-like pattern. This nanowire leaf assembly

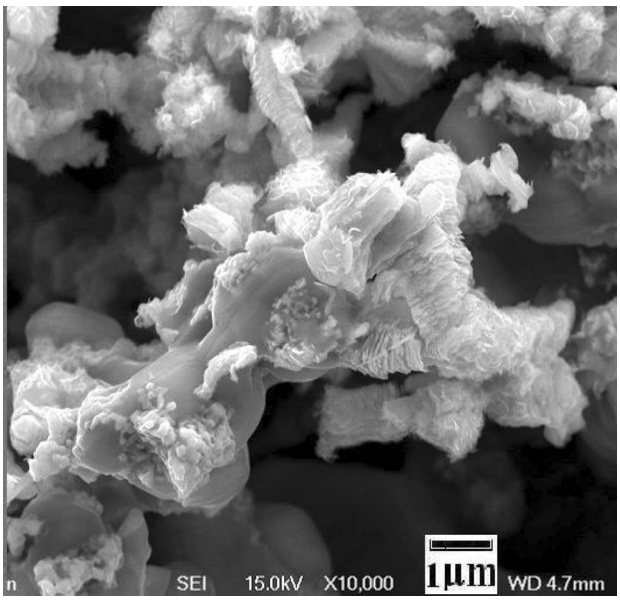

Fig. 7 SEM image of WSe_2/SSC nanowires

might be due to the sintering of the adjacent nanowires, which occurs upon heating in the Letlok cell at 750 °C for 3 h. The length of nanowires in each frame work of leaf is in the range of 200–400 nm and the thickness is in the range of 40–50 nm. Figure 4c is the high-magnification SEM image and represents the nanowires composed in leaf-like pattern. The two sides of the leaf are separated by the long nanowire of length 1.2 μm and the thickness is 50 nm.

Figure 7 shows the SEM image of the growth of WSe_2 nanowires on SSC. From the Fig. 7, it can be seen that the SSC is composed of several nanowires, grown perpendicularly and the product is homogeneous on the SSC. The length of nanowires in leaf-like pattern is in the range of 1.2–3.5 μm and the thickness is in the range of 200–500 nm In addition to the nanowires, there is agglomeration of nanocrystals of mean length and average diameter of 50 and 200 nm, respectively. Zhang and his co workers had synthesized the growth of arrays of Cu nanowires on the SBA-15 template. In this method, the template requires the pre-treatment process of coating with the carbon and also requires the reduction of the organometallic precursor $Cu(acac)_2$ with hydrogen gas for the generation of Cu nanoparticles.(Zhang et al. 2007) WS_2 and WSe_2 nanowires had been successfully deposited on SS coupon via a simple route of RAPET technique. This approach is relatively low cost (George et al. 2007a, b; George and Gedanken 2008; Pol et al. 2004a, b) and requires only readily available reactants like W and S or Se powders. In addition, the method does not require any template or pre-treatment process.

Conclusions

In this work, we presented a simple and straightforward RAPET method to synthesize the novel flower-like patterns of radially aligned WS_2 or WSe_2 nanowires. In addition, we had fabricated the WS_2 and WSe_2 nanowires on SSC using a same RAPET process. With such an unusual and novel form, the structure of the material may have expectation of novel properties. The key point for the preparation of this novel flower-like WS_2 or WSe_2 nanowires is using the tungsten and S or Se powder and reacting under the Letlok reaction conditions. Our synthetic process is relatively simple and efficient and may provide a novel method for large-scale synthesis and studies of the potential physical and chemical properties of novel materials with special nanomorphologies. The present synthetic method is a convenient process with good reproducibility and high yield, which make it possible to scale up to industrial production.

Experimental

The synthesis of WS_2 nanoflakes, WSe_2 nanoflakes, WS_2 nanowires/SSC and WSe_2 nanowires/SSC is carried out by the thermal treatment of reaction mixture of W, S or Se and SSC. Before the start of the reaction, the SSC was washed with water, ethanol and acetone. The 3 mL closed-vessel cell was assembled from stainless steel Letlok parts (manufactured by the HAM-LET Co., Israel). $A^1/_2''$ union part was plugged from both sides by standard caps, for the synthesis of neat WS_2 nanoflakes and WS_2 nanowire/SSC, 0.2 g of W and 0.076 g of S are introduced along with one SS coupon of 0.5 × 0.5 cm into the cell at room temperature under nitrogen atmosphere. The filled cell was closed tightly by the other plug and then placed inside an iron pipe in the middle of the furnace. The temperature was raised at a heating rate of 10 °C/min, and the closed-vessel cell was heated at 750 °C for 3 h. The reaction took place under the autogenic pressure of the precursors. At the end of the reaction, the Letlok was gradually cooled (∼5 h) to room temperature, and after opening, a black powder (0.2 g) was obtained. The total yield of the product material was 72 % of the total weight of the materials introduced into the cell. The weight of the SSC is measured before and after the RAPET reaction and it was found that the amount of WS_2 material coated on the SSC is 50 mg.

For the synthesis of neat WSe_2 nanoflakes and WSe_2 nanowire/SSC, 0.2 g of W and 0.189 g of Se are introduced along with one SS coupon of 0.5 × 0.5 cm into the cell at room temperature under air. The filled cell was closed tightly by the other plug and then placed inside an

iron pipe in the middle of the furnace. The temperature was raised at a heating rate of 10 °C/min, and the closed-vessel cell was heated at 750 °C for 3 h. The reaction took place under the autogenic pressure of the precursor. At the end of the reaction, the Letlok was gradually cooled (~5 h) to room temperature, and after opening, a black powder (0.35 g) was obtained. The total yield of the product material was 90 % of the total weight of the materials introduced into the cell. The amount of WSe_2 material coated on the SSC is obtained by measuring the weights of SS coupons before and after the RAPET reaction and is found to be 60 mg.

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