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Template-Assisted Electrochemical Synthesis of Semiconductor Nanowires

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1. Introduction

Semiconductor nanostructures occupy the center of scientific interest because of their unique electronic nature. Among them, nanowires (NWs), also known as “nanorods” or “nanowhiskers,” are especially attractive for nanoscience studies. While their diameters are in the nanometer range, they can have lengths in the tens of micrometers. Because of their unique density of electronic states, NWs in the limit of small diameters are expected to exhibit significantly different optical, electrical, and magnetic properties from their corresponding bulk materials. Consequently, many physical properties of semiconductors are significantly altered within the confines of the NW surfaces. On the other hand, their large surface-to-volume ratio allows for distinct structural and chemical behavior. For example, they can emit laser light, act like optical fibers, and change conductance when bound to different molecules [1].

The synthesis of NWs has been studied intensively worldwide for a wide spectrum of materials. Several chemical and physical methods are commonly used to produce NWs. Among them, electrochemical synthesis or electrodeposition to produce NWs has a number of advantages. Since electrochemical synthesis is usually controllable in the direction normal to the substrate surface, this technique can be readily extended to fabricate NWs. The length of the deposited NWs can be controlled by varying the duration of the electrodeposition process. In addition, electrochemical methods are inexpensive; operate at ambient temperatures and pressure. This chapter describes the some methods to semiconductor NWs production via electrochemical synthesis. Several electrochemical approaches have been reported to synthesize NWs including anodic aluminum oxide (AAO) template-assisted electrodeposition, lithographically patterned nanowire electrodeposition, surfactant-assisted electrodeposition, and template-free electrodeposition, etc. A great number of nanomaterials with diverse morphologies such as nanodots, nanoparticles, NWs, nanorods, nanobelts, nanotubes, nanospheres, nanoring, and nanoarrays, etc. have been synthesized based on template synthesis. All of these nanostructures were synthesized by deposition or growth of materials either inside the pores of the template or on the surface of the template. Among them the AAO template can stand high temperatures, is insoluble in organic solvents and geometrical parameters can be easily tuned by changing the synthesis conditions. This chapter is by no means comprehensive in covering all the relevant

literatures. In the first part of this chapter, the AAO template-assisted electrochemical synthesis to make semiconductor NWs is presented and discussed. In the later parts, other template-assisted electrochemical synthesis methods are presented. For quick reference, examples of typical NWs that have been electrodeposited in the templates are listed in Table 1.

2. AAO template-assisted electrodeposition of semiconductor nanowires

In fact, electrodeposition techniques have been used to grow semiconductor thin films on conductive surfaces. Among the electrochemical approaches, semiconductor thin films can be formed by electrochemical surface-limited reactions. Electrochemical surface-limited reactions are generally referred to as underpotential deposition (UPD) [2]. As a result of the UPD process, which takes place at more positive potentials than the equilibrium (Nernst) potential, the electrode surface is partially or completely (up to an atomic layer) covered by a deposit. However, the overpotential deposition (OPD or bulk deposition) process is determined by electrode potential, deposit growth kinetics and mechanism, electroactive species concentration, and deposit-substrate and deposit-deposit interactions. OPD takes place at more negative potentials than the Nernst potential. Briefly, UPD may involve deposition onto substrate while OPD would involve deposition onto a substrate surface modified by an atomic layer, which was formed during the UPD process. Generally, deposits reach more than one atomic layer in the OPD regions [3].

Semiconductor	Band gap (eV)	Electrodeposition method	Reference
CdS	2.42	AAO, ESED	[12, 27–32, 34, 39]
CdSe	1.7	AAO, ESED, LPNE	[45–47, 49, 73]
CdTe	1.5	AAO	[52, 53]
ZnO	3.37	AAO	[57, 58, 60]
Cu ₂ O	2.1	AAO	[62–64]
In ₂ O ₃	3.7	AAO	[65]
Bi ₂ Te ₃	0.15	AAO, ESED, “track-etch”	[68, 72, 76]
PbTe	0.33	LPNE	[78]
PbSe	0.29	LPNE	[79]

Table 1. Selected electrodeposition methods for the synthesis of semiconductor NWs.

Traditionally, semiconductor thin films have been electrodeposited by the so-called induced codeposition method, where both elements are deposited at the same time from the single solution [4]. Stoichiometry is maintained by having the more noble element as the limiting reagent, and poisoning the potential where the less noble element will underpotentially deposit only on the more noble element. Codeposition holds great promise if greater control can be achieved. At present, the main points of control are solution composition and the deposition potential. Recently, we have reported electrodeposition of PbS, CdS, Bi₂Te_{3-y}Se_y, Bi_{1-x}Sb_x, Bi_{2-x}Sb_xTe₃, Bi_{2-x}Sb_xSe₃, SnSe, and CdTe thin films by codeposition method [5–11]. For semiconductor NWs, the electrodeposition technique was used in 1996 for fabricating arrays of CdS NWs in nanoporous AAO membranes [12]. Template-assisted electrodeposition is one of the important methods for synthesis of semiconductor NWs with controlled shape and size. The template contains very small cylindrical pores within the host material, and the empty spaces are filled with the chosen material to form nanowires. Such

host materials include nanoporous (AAO) membranes [13], “track-etch” membranes [14], polymer membranes [15], diblock copolymers [16], nanochannel array glasses [17] and SiO₂ nanocapillary arrays [18].

The aim of this chapter is not to give a complete overview of all the host materials; the focus is on AAO template-assisted electrodeposition of semiconductor NWs. Hand made or commercial AAO membrane is one of the most popular templates in material science studies. Although AAO membranes can be brittle, they are used as templates for NWs. AAO membranes have many properties that make them especially valuable as templates. These features including [19]: (a) the pore sizes can be controlled; (b) highly transparent in the visible region of the optical spectrum that allows the investigation of the optical properties of the nanomaterials deposited within the nanopores; (c) alumina is amphoteric in nature and can be easily dissolved in an acidic or basic solution, by which the pure arrays of NWs can be obtained. Hand made porous AAO templates are produced by mild anodizing pure aluminum films in various acids [20, 21]. Aluminum anodization has been shown to be an easy and inexpensive way of template fabrication. The resulting oxide film possesses a regular hexagonal array of parallel and cylindrical channels under anodization conditions. The pores form with uniform diameters because of a delicate balance between electric-field-enhanced diffusion, which determines the growth rate of the alumina, and dissolution of the alumina into the acidic electrolyte. Depending on the anodization conditions, the pore diameter can be systematically varied from < 10 nm up to 200 nm [20, 22]. However, commercial porous AAO templates are produced by hard anodizing pure aluminum films. This method is faster but it produces films with disordered pore structures. In addition, commercially available anodic alumina offers only a limited number of pore diameters and therefore for experimental purposes requiring very thin pore sizes one has to synthesize his own template. Fig. 1 shows a representative SEM image of the top surface of porous AAO membrane (Whatman International Ltd.) with an average pore diameter of 100 nm. On the other hand, hand made template synthesis process involves handling of very corrosive chemicals and cryogenic liquids, making it a very dangerous procedure.

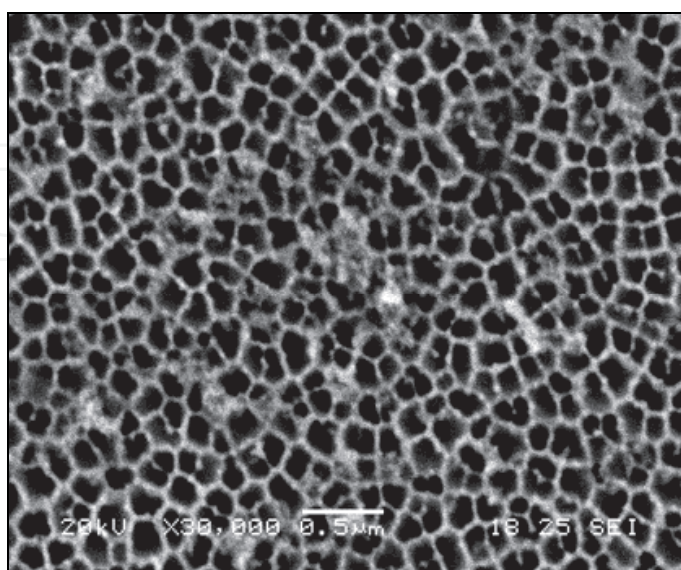


Fig. 1. A typical SEM image of the top surface of commercial AAO membrane. Marker is 0.5 μm.

In the electrodeposition processes, a thin conducting metal film, such as gold, is first evaporated on one side of the porous AAO membrane to serve as the cathode. The template is attached to the cathode, which is subsequently brought into contact with the deposition solution. When a current or potential is applied, cations and anions from the deposition solution diffuse towards the pore mouth, then inside towards the bottom of pores where electrochemical reactions of the species occur on gold surface. Then NWs grow along the pores to the top. The length of the NWs can be tuned by the time of electrodeposition process. After pore filling, the pure arrays of NWs can be obtained by dissolution of the template membrane in a basic solution. NW arrays are produced by the AAO template-assisted electrodeposition method as schematically shown in Fig. 2. In this fashion, semiconductor NWs can be deposited into the pores of AAO templates.

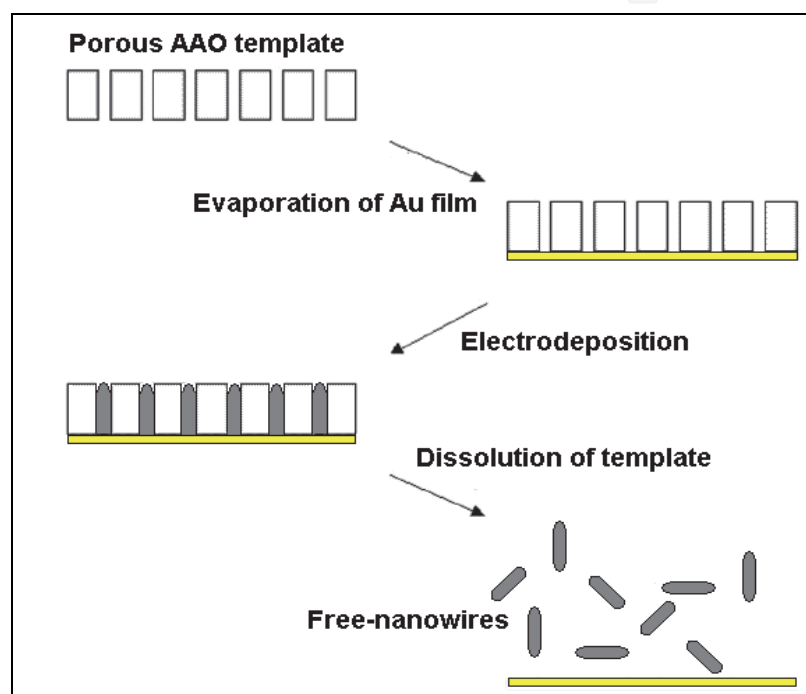
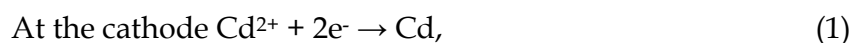


Fig. 2. Schematic diagram illustrating the growth of NWs by the AAO template-assisted electrodeposition method.

2.1 Cadmium chalcogenide NWs

Among the semiconductors explored in NW growth, cadmium chalcogenides (CdS, CdSe, and CdTe), being important direct band gap II-VI semiconductors with quantum-confined size-tunable properties, have found ample utilization opportunities in nanophotonics [23-25]. The AAO template-assisted electrodeposition technique is used for synthesizing cadmium chalcogenide (CdE = CdS, CdSe, and CdTe) NWs after the pioneering work by Routkevitch and co-workers [12] on ac electrodeposition of CdS from nonaqueous solution containing CdCl_2 and elemental S. But high temperature and expensive organic reagent (dimethylsulfoxide, DMSO) are necessary in order to dissolve elemental S in this method. Furthermore, a high density of defects such as stacking faults and twinned segments have been observed in those nanowires and the structure appears to be mainly the hexagonal form interleaved with domains of cubic structure [12, 26]. In a similar way, many researchers [27-31] deposited CdS NWs onto AAO membranes from nonaqueous solution

containing CdCl_2 , elemental S, and DMSO at high temperatures (100-120 °C). The formation of CdS NWs in the AAO template in DMSO solution containing CdCl_2 and elemental S may be written in the following equations [12]:



When a current or potential is applied between the cathode and anode, Cd^{2+} ions enter inside the cylindrical pores. On the other hand, elemental S enters into the pores by the diffusion process and forms CdS with the reaction of Cd atoms and they nucleate at the pore walls. As the reaction occurs inside the cylindrical pores, the wire diameter is controlled by the pore size, which grows from the surface of the electrode to the top of the template. However, Xu et al. [32] used an aqueous solution containing CdCl_2 and thioacetamide to produce CdS NWs onto AAO membranes at 70 °C. The overall reaction for the CdS NWs growth is written as eq. (3) [33].



Recently, we have reported the synthesis of CdS NWs onto AAO membranes by an electrodeposition method from aqueous solution containing CdSO_4 , Na_2S , and ethylenediamine tetraacetic acid (EDTA) at room temperature (25 °C) [34]. EDTA is used to form a CdEDTA^{2-} complex in order to prevent the chemical precipitation of Cd^{2+} and S^{2-} as CdS. The method starts with the investigation of electrochemical behaviors of each element in the presence of EDTA. A representative cyclic voltammogram for S^{2-} on the Au(111) working electrode in 0.1 M EDTA and 2 mM Na_2S solutions is shown in Fig. 3a.

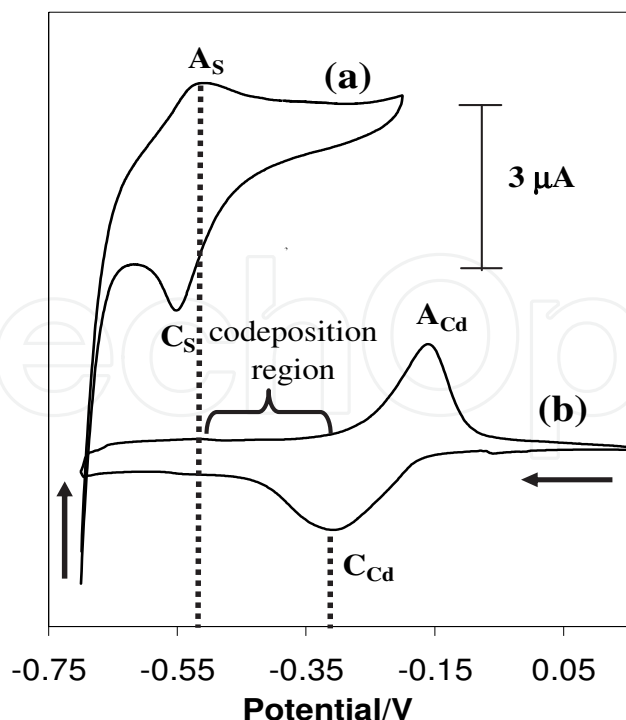


Fig. 3. Overlapped cyclic voltammograms of Au(111) electrode in 0.1 M EDTA solution containing: (a) 2 mM Na_2S and (b) 10 mM CdSO_4 at 100 mV/s.

Based on previous reports [6, 35, 36], the A_s and C_s peaks correspond to the S UPD and dissolution of S UPD, respectively. Fig. 3b shows a cyclic voltammetric curve of Cd on Au(111) in aqueous solution containing $CdEDTA^{2-}$ ions. The broad cathodic peak (C_{Cd}) corresponds to Cd UPD. On the reverse scan, the stripping peak (A_{Cd}) corresponds to dissolution of Cd UPD. The electrochemical behaviors of Cd in this work are similar to that reported by previous authors [6, 37, 38].

Based on the above results, for electrodeposition potential of Cd and S in UPD range can be determined from overlapped cyclic voltammograms of Cd and S. If the potential of the working electrode was kept constant at a potential between the reductive UPD wave of $CdEDTA^{2-}$ and the oxidative UPD wave of S^{2-} (codeposition region), Cd and S would deposit underpotentially at the electrode surface. These underpotentially deposited Cd and S atoms react to form the CdS. Since the potential values of the co-deposition region are not enough for the bulk deposition (OPD) of Cd and S, deposition of $CdEDTA^{2-}$ on Cd or S^{2-} on S will not occur. As a consequence, it should promote the electrochemical atom-by-atom growth of CdS compound. This concept was introduced and analyzed by Öznülür et al. [5] and is a key concept to explain the success of the deposition of PbS. The electrodeposition starts with the preparation of AAO membrane as a template, with one side of the membrane coated with Au film as an electrode. After inserting the coated membrane into a solution of $CdSO_4 + Na_2S + EDTA$, CdS NWs can be obtained by applying a suitable potential value. The electrochemical quantification of the deposits was investigated by stripping voltammetry. The coulometric stripping of the resulting CdS indicated that the coverages of S and Cd were nearly 1:1 under most of the deposition conditions studied. This confirms the CdS nature of the deposited film. In addition, the deposition current during CdS nanowire arrays electrodeposition into the AAO template at -0.50 V was investigated. When the electrodeposition was performed for 6 h, it was observed that the current values dropped from 60 to 15 μA slowly. This decrease suggests the formation of a poorly conducting structure in the template. Fig. 4 shows TEM image of CdS NWs obtained -0.50 V for 2 h after etching the template. The diameters of the NWs within this array are about 100 nm and the lengths are up to 1 μm .

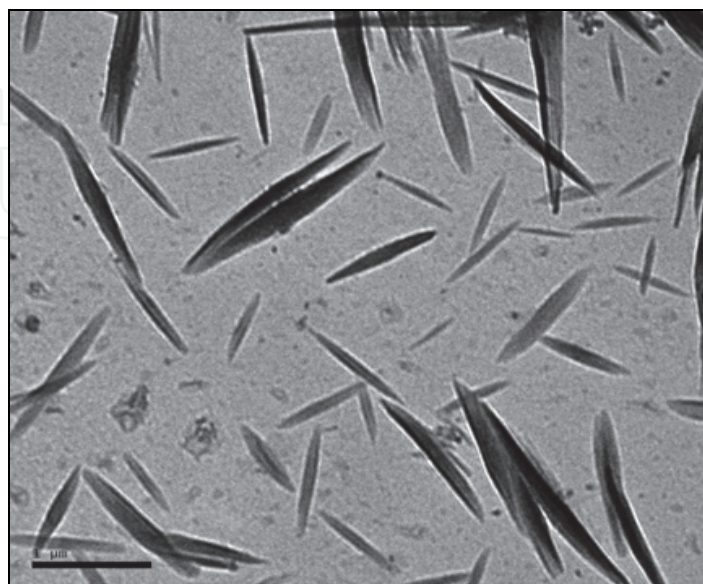


Fig. 4. TEM image of CdS NWs with a diameter of about 100 nm. Marker is 1 μm .

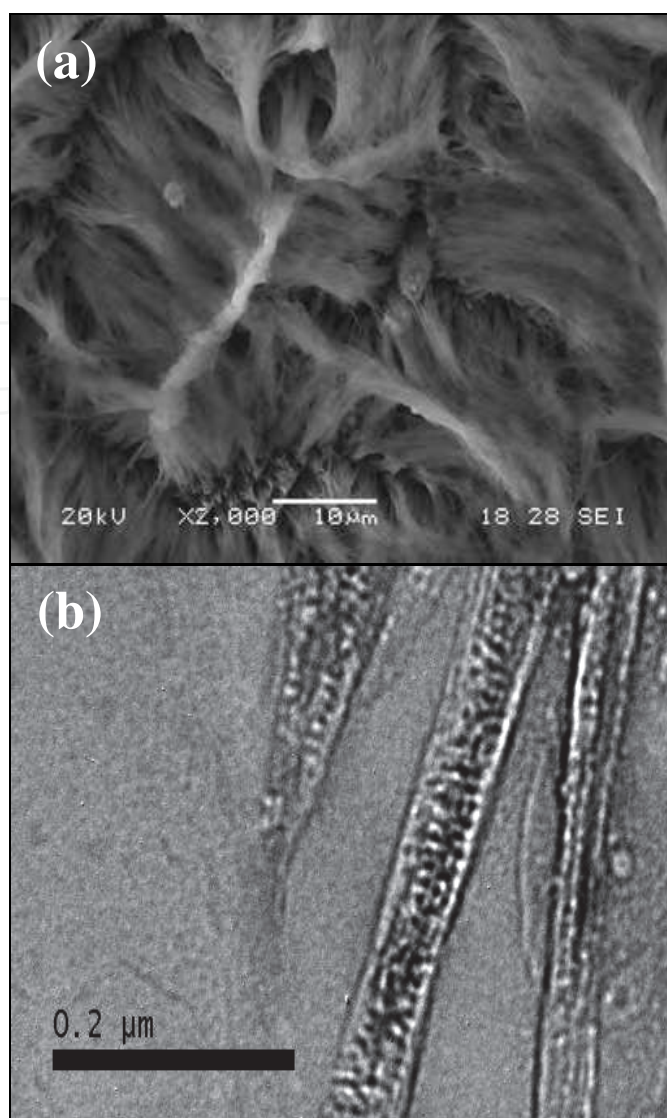
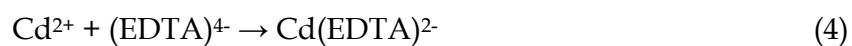


Fig. 5. (a) A typical SEM image of CdS NW arrays on the AAO template after etching times of 10 min. (b) TEM image of the a few CdS NWs electrodeposited in the template shown after the complete dissolution of AAO in 1 M NaOH.

SEM and TEM images of the CdS NWs electrodeposited on AAO template at -0.50 V for 6 h are shown in Fig. 5. The diameters of the NWs within this array are about 100 nm and the lengths are up to 10 μm . It is clear that the length of the CdS NWs increases with an increase in the deposition time.

Consequently, formation of CdS NWs in the AAO template in EDTA solution containing Cd^{2+} [39, 40] and S^{2-} at an acidic medium may be written in the following equations:



According to this sequence, CdS formation inside of AAO involves firstly diffusion of $\text{Cd}(\text{EDTA})^{2-}$ and H_2S species from solution towards the pore mouth, then inside towards the bottom of pores where electrochemical reactions of the species occur on gold surface. Then CdS nanowires grow along the pores to the top.

XRD pattern of the CdS NWs electrodeposited on amorphous AAO is shown in Fig. 6. XRD analysis reveals that the electrosynthesized NWs have a preferential orientation along (110) direction for hexagonal crystal (JCPDS, 41-1049). The broad XRD peak is ascribed to the small grain size.

Fig. 7 shows the UV-vis spectra of CdS NWs inside the deionized water after complete dissolution of template. The blue shifted absorption at 489 nm as compared to the ~ 515 nm absorption for bulk CdS crystal may be attributed to the quantum confinement effect of CdS NWs [41].

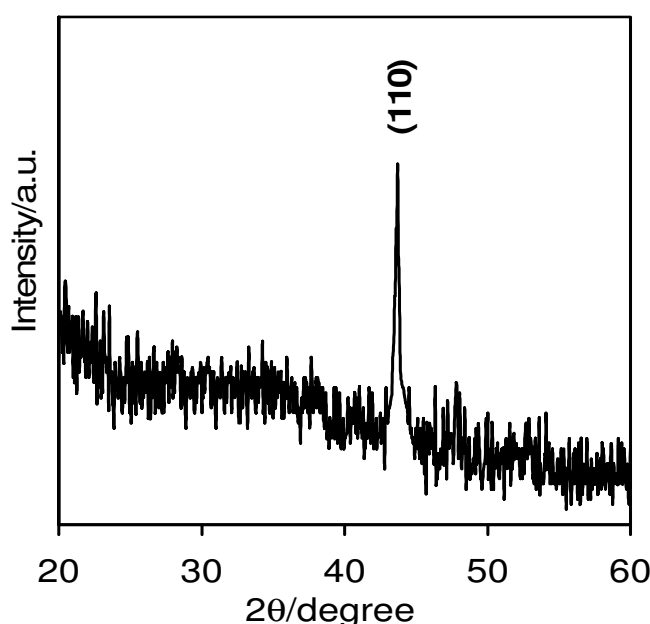


Fig. 6. A representative XRD pattern of the CdS NWs embedded in the AAO template.

These results indicate that highly crystalline CdS NWs can be grown by this method. By fixing the deposition potential range, stoichiometric CdS NWs can be produced in this method. Furthermore, the length of the NWs can be tuned by the controlling the deposition or etching time. As a result, this method is different than other AAO template-assisted electrodeposition approaches.

AAO membranes are frequently employed as templates for fabricating other cadmium chalcogenides (CdSe and CdTe). CdSe has been investigated over the past decade for applications in optoelectronics [42], luminescent materials [43], lasing materials [44], and biomedical imaging. CdSe NWs have been produced by direct current electrodeposition from both aqueous and nonaqueous electrolytes [45-47]. On the other hand, metal-CdSe-metal NWs have been synthesized by the electrochemical replication of porous AAO and polycarbonate membranes with pore diameters of 350 and 700 nm respectively [48]. The overall reaction for the CdSe NW growth is written as eq. (8) [49].



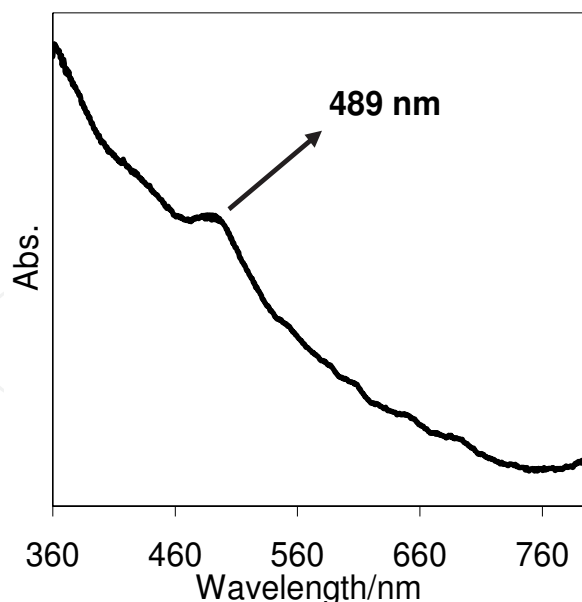
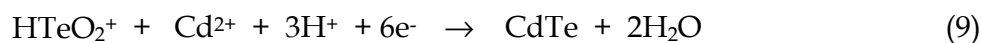


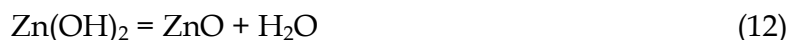
Fig. 7. Typical UV-vis absorption spectrum of CdS NWs.

CdTe NWs can also be produced by this method. Cadmium telluride (CdTe) is one of the most important II-VI group compound semiconductors due to its interesting photoelectric properties. It has a direct energy band gap [50], a relatively high optical absorption coefficient, and advanced photovoltaic properties, which make it suitable for fabricating photoelectron devices and solar cells [51]. It is expected that CdTe NWs would be of interest in technological application for nanodevices. However, few studies of the electrosynthesis of CdTe NWs on AAO membranes are reported [52, 53]. In that case the deposition proceeds directly by the global reaction [54]:



2.2 Oxide NWs

Most of the oxides are semiconductors. Among these, ZnO NWs are especially attractive due to their tunable electronic and optoelectronic properties, and the potential applications in the nanoscale electronic and micro-optoelectronic devices [1, 55, 56]. Numerous reports on the preparations of ZnO films and nanoparticles have been reported. In contract, very little work has been focused on ZnO NWs. Ordered ZnO NWs embedded in AAO templates have already been fabricated by electrodepositing Zn into nanopores of AAO to form metallic nanowire arrays and then to oxidize them [57]. But the long time thermal oxidation at 300 °C needed in this method limits its application. ZnO NWs have also been synthesized by one-step electrodeposition [58] based on AAO templates conducted in aqueous Zn^{2+} solutions. The mechanism of electrodeposition of ZnO NWs from oxygenated aqueous Zn^{2+} solution could be summarized as followings [59]:

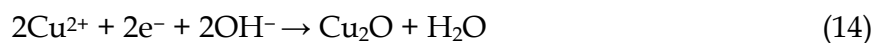


The total reaction may be written as:



It can be seen that the aqueous solution induces the deposition of $\text{Zn}(\text{OH})_2$ competing with the formation of ZnO . Wang et al. [60] reported a different approach to prepare the ZnO NWs into AAO templates involving nonaqueous DMSO. It is important that the formation of $\text{Zn}(\text{OH})_2$ is prevented in a nonaqueous media.

AAO templates can be employed for electrosynthesis of Cu_2O NWs. Cu_2O is a relatively non-toxic p-type semiconductor with a band gap of 2.1 eV [61]. It can be used in solar energy converting devices. Cu_2O NWs have been produced by electrodeposition on AAO templates from alkaline aqueous solutions [62-64]. In alkaline solution, the reaction leading to the deposition of copper oxide is [64]



In addition, In_2O_3 NWs can also be produced by this method. However, to the best of our knowledge, there is only one report found on the electrodeposition of In_2O_3 NWs into AAO templates [65].

2.3 Bismuth-based NWs

The VA-VIA group semiconductors are commonly used for thermoelectric devices such as thermoelectric generators, coolers, radiation detectors, and so forth [66]. Among them, Bi_2Te_3 and its derivative compounds are considered to be the best materials used in thermoelectric refrigeration at room temperature [67]. High-quality Bi_2Te_3 , $\text{Bi}_2\text{Te}_{3-y}\text{Sey}$, $\text{Bi}_{1-x}\text{Sb}_x$, and $\text{Bi}_{2-x}\text{Sb}_x\text{Te}_3$ NWs have been prepared by electrodeposition in AAO membranes from both aqueous and nonaqueous electrolytes [68-71]. The obtained Bi_2Te_3 arrays are dense with a narrow distribution of NW diameters. On the other hand, by tuning the solution concentrations and controlling the growth rate by controlling the potential, the composition, crystallinity, and morphology of the $\text{Bi}_{1-x}\text{Sb}_x$ NWs can be tuned. Other bismuth-based NWs ($\text{Bi}_2\text{Te}_{3-y}\text{Sey}$ and $\text{Bi}_{2-x}\text{Sb}_x\text{Te}_3$) are single phase and crystalline.

3. Electrochemical Step Edge Decoration (ESED)

The method, developed by Penner et al. [39], is claimed to have the capability to synthesize CdS NWs which are long, uniform in diameter, and removable from the surface on which they are synthesized. The method starts with selective electrodeposition of metal from an aqueous solution as a precursor, along step edges present on the stepped surface, such as graphite, to form into NWs in the first step (Fig. 8), according to the reaction



In the second step, Cd NWs were converted to CdS hemicylindrical shell NWs by exposure to flowing H_2S at 280–300 °C according to the reaction



This method has been successfully applied to obtain NWs of Bi_2Te_3 [72] and CdSe [73].

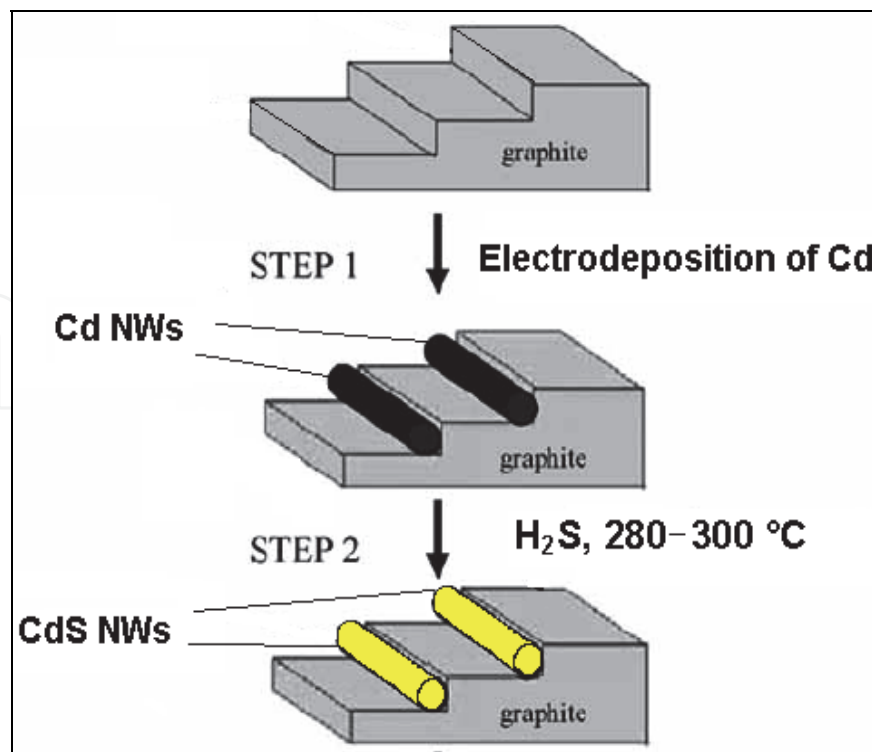


Fig. 8. A schematic diagram for the synthesis of CdS NWs through ESED method.

4. “Track-etch” membranes

Another type of porous template that is commonly used for NW synthesis is the template type fabricated by chemically etching particle tracks originating from ion bombardment, such as track-etched polymeric membranes [14]. These membranes contain cylindrical pores of uniform diameter. The most commonly used material to prepare membranes of this type is polycarbonate; however, a number of other materials are amenable to the track-etch process [14]. A number of companies sell nanoporous polymeric filtration membranes that have been prepared via the “track-etch” method. The method involves the irradiation of polycarbonate foils (PCF) using a gold-ion beam at the heavy-ion accelerator to damage the polymer structure. Then an ultraviolet treatment is applied to each face of the PCF. The pore density corresponds to the ion fluency, whereas the length of the pores is equal to the thickness of the membrane. The pores are randomly distributed across the membrane surface. The pore diameter is adjusted by a partial chemical dissolution. Membranes with a wide range of pore diameters (down to 10 nm) are available commercially. High-quality Bi_2Te_3 / $(\text{Bi}_{0.3}\text{Sb}_{0.7})_2\text{Te}_3$, $\text{Si}_x\text{Ge}_{1-x}$, and Bi_2Te_3 NWs have been prepared by electrodeposition in “track-etch” membranes from both aqueous and ionic liquid mediums [74-76]. These organic membranes may be preferred over anodized alumina membranes since they exhibit greater flexibility.

5. Lithographical Patterned Nanowire Electrodeposition (LPNE)

Lithographically patterned nanowire electrodeposition (LPNE), developed Menke et al. [77], is a new method for synthesizing semiconductor NWs on glass or oxidized silicon surfaces. The method involves the fabrication of a sacrificial, horizontal template using conventional

microfabrication methods. LPNE uses photolithography to prepare a three-sided "nanoform" into which a nanowire can be electrodeposited using the horizontal nickel edge that defines the back surface of this nanoform. The LPNE synthesis of PbTe NW arrays was carried out according to the twelve-step process flow: (Step 1) A 1-2 μm thick photoresist (PR) layer is spin-coated onto a glass or oxidized silicon surface, (Step 2) a nickel layer 20 - 100 nm in thickness is thermally deposited, (Step 3) a positive-tone PR is coated onto this nickel layer and, (Step 4) photopatterned using 365 nm illumination and a contact mask, (Step 5) the exposed PR is removed, (Step 6) now the remaining PR on the same is flood-exposed. A crucial point is that after this flood-exposure, no developing is carried out; (Step 7) the exposed nickel was etched using HNO_3 to produce a 500 nm deep undercut at the edges of the exposed regions. The horizontal trench formed by this undercut is the "nanoform" into which the PbTe NWs will be electrodeposited. The height of this nano-form equaled the thickness of the nickel layer which defines the vertical back of the trench. This nano-form follows the contour of the photopatterned region. (Step 8) The PbTe NW is electrodeposited into the trench, (Step 9) the previously exposed top-PR layer is removed using developer, (Step 10) the nickel layer is removed by etching in HNO_3 leaving free-standing PbTe NWs on top of an intact and unexposed layer of bottom-PR, (Step 11) this bottom PR layer is exposed as before, (Step 12) the exposed PR is removed leaving suspended PbTe NW segments up to 25 μm in length [78]. This method could also be used to grow NWs of CdSe [49] and PbSe [79].

6. Conclusion

In conclusion, we have presented a brief overview of the selected template-assisted electrodeposition methods for the synthesis of semiconductor NWs. Compared to conventional methods, the high degree of reproducibility makes template-assisted methods the most attractive way. In this chapter, four groups of template-assisted electrodeposition methods have been investigated. Among these templates the nanoporous AAO is the most promising. For example, the AAO template can stand high temperatures, is insoluble in organic solvents and geometrical parameters can be easily tuned by changing the synthesis conditions. The suggested electrodeposition mechanisms for some semiconductor NWs onto templates were presented and discussed. As an example of semiconductor NWs, we were focused on AAO template-assisted electrodeposition of CdS NWs under the conditions of UPD. Using this method, many material parameters such as dimensions and compositions can be tuned. The method may serve as a model for synthesis of other semiconductor NWs.

7. Acknowledgment

Sakarya University is gratefully acknowledged for the financial support of this work.

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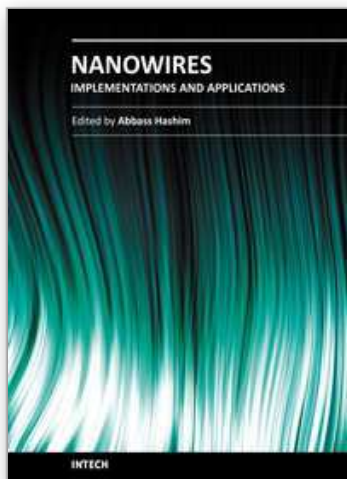
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Nanowires - Implementations and Applications

Edited by Dr. Abbass Hashim

ISBN 978-953-307-318-7

Hard cover, 538 pages

Publisher InTech

Published online 18, July, 2011

Published in print edition July, 2011

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Sisman (2011). Template-Assisted Electrochemical Synthesis of Semiconductor Nanowires, Nanowires - Implementations and Applications, Dr. Abbass Hashim (Ed.), ISBN: 978-953-307-318-7, InTech, Available from: <http://www.intechopen.com/books/nanowires-implementations-and-applications/template-assisted-electrochemical-synthesis-of-semiconductor-nanowires>

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