

Topical Review

Atomic layer deposition: an enabling technology for the growth of functional nanoscale semiconductors

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Abstract

In this paper, we present the progress in the growth of nanoscale semiconductors grown via atomic layer deposition (ALD). After the adoption by semiconductor chip industry, ALD became a widespread tool to grow functional films and conformal ultra-thin coatings for various applications. Based on self-limiting and ligand-exchange-based surface reactions, ALD enabled the low-temperature growth of nanoscale dielectric, metal, and semiconductor materials. Being able to deposit wafer-scale uniform semiconductor films at relatively low-temperatures, with sub-monolayer thickness control and ultimate conformality, makes ALD attractive for semiconductor device applications. Towards this end, precursors and low-temperature growth recipes are developed to deposit crystalline thin films for compound and elemental semiconductors. Conventional thermal ALD as well as plasma-assisted and radical-enhanced techniques have been exploited to achieve device-compatible film quality. Metal-oxides, III-nitrides, sulfides, and selenides are among the most popular semiconductor material families studied via ALD technology. Besides thin films, ALD can grow nanostructured semiconductors as well using either template-assisted growth methods or bottom-up controlled nucleation mechanisms. Among the demonstrated semiconductor nanostructures are nanoparticles, nano/quantum-dots, nanowires, nanotubes, nanofibers, nanopillars, hollow and core-shell versions of the afore-mentioned nanostructures, and 2D materials including transition metal dichalcogenides and graphene. ALD-grown nanoscale semiconductor materials find applications in a vast amount of applications including functional coatings, catalysis and photocatalysis, renewable energy conversion and storage, chemical sensing, opto-electronics, and flexible electronics. In this review, we give an overview of the current state-of-the-art in ALD-based nanoscale semiconductor research including the already demonstrated and future applications.

Keywords: atomic layer deposition, semiconductor, nanoscale, nanostructured, metal-oxide, III-nitride, self-limiting

(Some figures may appear in colour only in the online journal)

1. Introduction

Information age, digital revolution, age of computers... No matter how we name it, the latest technology revolution which still continues and deeply impacts our daily life was triggered by a key enabling device technology, i.e., the transistor. Germanium (Ge) was the semiconductor used throughout the early development phase, which later was replaced by another group-IV elemental semiconductor, silicon (Si), mainly due to its superior native oxide. Si-based integrated circuit (IC) technology (CMOS) began its journey in 1959 with only a single transistor/chip and 30 μm linewidth. Nearly 60 years of continuous and successful miniaturization effort led to more than three orders of magnitude shrinkage of the transistor gate lengths and about ten orders of magnitude increase in transistor density. Atomic layer deposition (ALD) played a crucial role in keeping the pace of Moore's law, particularly for the sub-65 nm technology nodes by providing novel materials and processing solutions including but not limited to ALD-grown high-k gate dielectrics and spacer-defined double patterning.

ALD is a chemical vapor deposition (CVD) technique where conventional gas-phase reactions are eliminated either by temporally or spatially separated half-cycles. Instead, within the ALD growth window, film deposition proceeds via self-saturating ligand-exchange surface reactions only. The unique surface-chemistry driven self-limiting growth character of ALD enables precision atomic-scale thickness control, ultimate three-dimensional conformality, and large-area uniformity, all being highly critical features for current and future atomic-scale precision device engineering and nano-manufacturing needs.

Originally developed and named as atomic layer epitaxy and molecular layering, initial efforts focused on sulfide and oxide semiconductor thin-film synthesis for electro-luminescent displays, sensors, catalytic materials, and thin-film solar cells. Early history details of ALD research can be found in the recently completed 'Virtual Project on the History of ALD' work and publications pioneered by Puurunen *et al* [1, 2]. Since then, both materials and applications list of ALD has been enriched tremendously: oxide, metal, nitride, sulfide, selenide, carbide, fluoride, polymer, and biomaterial growth recipes have been developed for applications including moisture barriers for light emitting devices/displays, surface passivation of solar cells, diffusion barriers for Li-ion batteries, anti-tarnishing jewelry coatings, anti-corrosion coatings of electronic circuit-boards/LEDs/sensors, gate insulators and nano-patterning of CMOS transistors, flexible/wearable electronics, surface functionalization for implants, nano-catalysts for remediation, hydrogen generation, solid-oxide fuel-cells, and self-cleaning surfaces.

When compared with ALD-grown semiconductors, dielectric and metallic ALD-materials constitute the majority of ALD literature, mainly due to the intense research efforts in high-k dielectrics, passivation/protection coatings, and relatively easy forming metallic nanocatalysts. Semiconductors, on the other hand, suffer mainly from low-temperature self-limiting growth windows, resulting in poly-crystalline or

amorphous-like films with high levels of impurities which are incompatible with high-performance devices. In this respect, ALD-grown semiconductors can hardly compete with the material quality of mainstream epitaxial films grown via high-temperature metal-organic chemical vapor deposition (MOCVD) or molecular beam epitaxy (MBE). Instead, ALD-grown semiconductors might find significant use in precision coating of low-temperature compatible and highly porous/high surface-area substrates/templates, as well as in fabricating highly controlled functional nanostructures. Already demonstrated device applications of such low-temperature grown nanoscale semiconductors include solar cells, catalysis, energy storage, photocatalysis, flexible electronics, and chemical gas sensors.

Although excellent review articles on general ALD overview [3–8], specific ALD methods [9–11], nano-materials [12–15], and application areas [16–19] exist in the literature, an effort focusing on ALD-grown nanoscale semiconductors and their applications is yet missing. The aim of this review is to present the status and summary of the ALD semiconductor research activity by covering critical findings and contributions in the field. First, thin-film semiconductor growth reports are reviewed, followed by the efforts for the ALD synthesis of 0D, 1D, 2D, and core-shell nanostructured semiconductors. Finally, reported device applications are summarized along with a future perspective on ALD semiconductor research outlining the important challenges and opportunities.

2. Semiconductor thin-film synthesis via ALD

Among the semiconductor thin films grown via ALD, oxides, nitrides, and sulfides constitute the vast majority of reported literature. In addition to these three compound semiconductor material families, ALD of selenides, tellurides, and arsenides have been studied as well to a lesser extent. Low-temperature ALD efforts of group-IV elemental semiconductors, Si and Ge, are rather scarce with very limited studies available. An overview of the literature on semiconductor thin films grown via ALD is summarized in table 1.

2.1. Oxides

ALD of ZnO has been studied quite extensively [12, 20–26]. While H_2O has been the predominant choice for oxygen precursor, the first Zn precursor used for ALD of ZnO was zinc acetate, however it required high substrate temperatures (lowest reported is 280 °C) [27]. The most common Zn precursor used for ZnO ALD growth is diethylzinc (DEZn), which significantly lowered the growth temperatures (typically 100 °C–200 °C) due to its high reactivity and exothermic reaction for the formation of ZnO [21–24, 26, 28–68]. Dimethylzinc is another precursor used for ZnO growth which produces slightly higher ZnO growth per cycle (GPC) values when compared with diethylzinc [69]. Besides these common precursors, elemental zinc and ZnCl_2 have also been used as Zn source for ZnO ALD, they however required

Table 1. (Continued.)

Material	Reactant A	Reactant B	Growth temperature	Substrate	Type of ALD	References
	TiCl ₄	O ₂ , H ₂ O	30 °C–180 °C	Si (111)	Plasma assisted	[235]
	Tetrakis dimethyl-amidotitanium	H ₂ O	200 °C–250 °C	Si	Thermal	[104]
	Tetrakis dimethyl-amidotitanium	O ₃	75 °C–400 °C	Si	Ozone assisted	[236]
	Titanium tetraisopropoxide	O ₂	70 °C–100 °C	Si (100) and fused silica	Plasma assisted	[237]
	TiCl ₄	H ₂ O	150 °C–400 °C	Fluorine doped tin oxide glass (FTO) and Si (100)	Thermal	[238]
	Tris(dimethylamido)-(dimethylamino-2-propanolato)titanium(IV) (TDMADT).	O ₂	60 °C	Polyethylene-terephthalate (PET) and Si	Plasma assisted	[239]
Ga ₂ O ₃	Ga(acac), (acac = pentane-2,4-dionate)	H ₂ O, O ₃	350 °C–400 °C	Si (100), soda lime glass, and corning glass	Thermal	[105]
	Ga ₂ (NMe ₂) ₆	H ₂ O	150 °C–300 °C	Si (100)	Thermal	[106]
	[(CH ₃) ₂ GaNH ₂] ₃	O ₂	200 °C	Si (100) and sapphire (001)	Plasma assisted	[240]
	[(CH ₃) ₂ GaNH ₂] ₃	O ₂	200 °C	Si (100)	Plasma assisted	[241]
	[(CH ₃) ₂ GaNH ₂] ₃	O ₂	50 °C, 150 °C, 250 °C	Si (100)	Plasma assisted	[242]
	Ga(CH ₃) ₃	O ₃	200 °C–450 °C	Si (100) and SiO ₂	Ozone assisted	[243]
	Ga ₂ (NMe ₂) ₆	H ₂ O	—	TiO ₂	Thermal	[244]
	Gallium tri-isopropoxide	H ₂ O	150 °C–250 °C	Si, glass, and carbon	Thermal	[107]
	Ga(CH ₃) ₃	O ₂	100 °C–400 °C	Si (111)	Plasma assisted	[108]
	Ga(CH ₃) ₃	O ₃	150 °C–400 °C	Si (100)	Ozone assisted	[245]
	Tris (2,2,6,6-tetramethyl-3,5-heptanedionato) gallium(III)	O ₂	100 °C–400 °C	SiO ₂ /Si	Plasma assisted	[246]
	Ga(CH ₃) ₃	O ₂	250 °C	Si (111)	Plasma assisted	[247]
	Ga(CH ₃) ₃	O ₂	50 °C–150 °C	Si (100)	Plasma assisted	[109]
	Ga(CH ₃) ₃	H ₂ O	550 °C	Sapphire, GaN, and (111)- and (001)-oriented 3C–SiC	Thermal	[248]
	Ga(CH ₃) ₃	O ₂	250 °C	SiC	Plasma assisted	[249]
In ₂ O ₃	InCl ₃	H ₂ O	500 °C	Corning glass	Thermal	[250]
	Cyclopentadienyl indium	O ₃	200 °C–450 °C	Si (100) and glass	Ozone assisted	[111]
	In (acac) ₃ (acac = acetylacetonate, pentane-2,4-dione)	H ₂ O, O ₃	160 °C–300 °C	Si (100), fused silica, and soda lime glass	Ozone assisted and thermal	[112]
	Cyclopentadienyl indium	H ₂ O, O ₂	100 °C–250 °C	Si (100), fused silica, and quartz	Thermal	[114]
	In(CH ₃) ₃	H ₂ O	150 °C–325 °C	Si and SiO ₂ /Si	Thermal	[113]
	[In{(iPr) ₂ CNR ₂ }] ₃ where R = Et (1) and Me (2), namely tris-(N,N'-diisopropyl-2-diethylamido-guanidinato)-indium(III)(1) and tris-(N,N'-diisopropyl-2-dimethylamido-guanidinato)-indium(III)	H ₂ O	160 °C–320 °C	Si (100), Al ₂ O ₃ (0001), and glass	Thermal	[251]
	Et ₂ InN(SiMe ₃) ₂	H ₂ O	225 °C–250 °C	SiO ₂ /Si	Thermal	[115]
	Cyclopentadienyl indium	H ₂ O, O ₂	100 °C	SiO ₂ /Si	Thermal	[252]
	Cyclopentadienyl indium	H ₂ O, O ₂	100 °C	SiO ₂ /Si	Thermal	[253]
	[3-(dimethylamino)propyl] dimethyl indium	H ₂ O	275 °C	Si	Thermal	[254]
	Dimethylamino- dimethylindium	H ₂ O	300 °C	Si (100)	Thermal	[255]
	Dimethyl(N-ethoxy- 2,2-dimethylpropanamido)indium	O ₂	70 °C–250 °C	Si and glass	Plasma assisted	[256]
	In(CH ₃) ₃	O ₃ , O ₂ , H ₂ O, H ₂ O ₂	100 °C–250 °C	Si(100), fused quartz, and glass	Thermal, ozone assisted	[257]
	[1,1,1-trimethyl-N-(trimethylsilyl)silanaminato]- indium (InCA-1)	H ₂ O ₂	150 °C	Polyimide	Thermal	[116]
NiO	Ni(dmamp) ₂ (dmamp = 1-dimethylamino-2-methyl-2-propanolate)	H ₂ O	80 °C–240 °C	Si (001)	Thermal	[258]
	Ni(Cp) ₂ (Cp = cyclopentadienyl, C ₅ H ₅) or Ni(EtCp) ₂ [EtCp = ethylcyclopentadienyl, (C ₂ H ₅)(C ₃ H ₄)]	O ₃	150 °C–300 °C	Si (100)	Ozone assisted	[259]
	Bis(2,2,6,6-tetramethyl- 3,5-heptanedionato)Ni(II)	H ₂ O	205 °C–275 °C	MgO (100) and Al ₂ O ₃ (00 1)	Thermal	[260]
	bis(2,2,6,6-tetramethylheptane-3,5- dionato)nickel(II) (Ni(thd) ₂)	H ₂ O	205 °C–290 °C	SiO ₂	Thermal	[261]
	Ni(Cp) ₂	O ₃	230 °C	Si (100)	Ozone assisted	[262]
	Bis- methylcyclopentadienyl-nickel ([MeCp] ₂ Ni)	O ₂	150 °C–350 °C	Pt, Ru, and W	Plasma assisted	[263]
	Nickel amidinate	H ₂ O	175 °C	Fluorine-doped tin oxide-coated glass substrates (FTO)	Thermal	[264]
	Nickel bis(N,N'- di-tert-butylacetamidate) [Ni(tBu-MeAMD) ₂]	H ₂ O	200 °C	Si	Thermal	[265]
	Ni(Cp) ₂	O ₃	275 °C	FTO	Ozone assisted	[266]

Table 1. (Continued.)

Material	Reactant A	Reactant B	Growth temperature	Substrate	Type of ALD	References
Bi ₂ O ₃	Bi(thd) ₃ (thd: 2,2,6,6-tetramethyl-3,5- heptanedionato)	H ₂ O	200 °C–350 °C	Si	Thermal	[267]
SnO ₂	SnI ₄	O ₂	400 °C–750 °C	SiO ₂ /Si (100)	Thermal	[118]
	SnCl ₄	H ₂ O	400 °C–500 °C	Pyrex glass and sapphire	Thermal	[119]
	Dibutyl tin diacetate	O ₂	200 °C–400 °C	Si (100)	Plasma enhanced	[126]
	Dibutyl tin diacetate	O ₂	300 °C	(100), (110), and (111) yttria-stabilized zirconia (YSZ) substrates	Plasma enhanced	[125]
GaN	GaCl ₃	NH ₃	550 °C	(001) GaAs	Thermal	[268]
	Ga(CH ₃) ₃	NH ₃	550 °C	(0001) Sapphire	Thermal	[127]
	Ga(CH ₃) ₃	NH ₃	500 °C–650 °C	(001) GaAs	Thermal	[128]
	GaCl ₃	NH ₃	400 °C–750 °C	Si (100)	Thermal	[129]
	Ga(CH ₃) ₃	NH ₃	100 °C–500 °C	Si	Plasma enhanced	[130]
	Ga(C ₂ H ₅) ₃	N ₂ /H ₂	200 °C, 450 °C	Si (100) and quartz	Plasma enhanced	[133, 269]
	Ga(CH ₃) ₃ , Ga(C ₂ H ₅) ₃	N ₂ /H ₂	200 °C	Si (100) and quartz	Plasma enhanced	[69, 132, 133, 139–269]
	Ga(C ₂ H ₅) ₃	N ₂ /H ₂	200 °C	Si (100), Si (111), and c-plane sapphire	Plasma enhanced	[138]
	Ga(C ₂ H ₅) ₃	N ₂ /H ₂	200 °C	Si (100)	Plasma enhanced	[136]
	Ga(CH ₃) ₃ and H radicals	NH ₃	100 °C and room temperature	Si (111)	Electron enhanced	[139]
	Ga(CH ₃) ₃	N ₂ /H ₂ and NH ₃	200 °C	Si (100), Si (111), and c-plane sapphire substrates	Plasma enhanced	[131]
	Ga(C ₂ H ₅) ₃	N ₂ /H ₂ plasma	150 °C–425 °C	C-plane sapphire substrates	Plasma enhanced	[270]
	Ga(C ₂ H ₅) ₃	N ₂ /H ₂ plasma	275 °C	C-plane sapphire	Plasma enhanced	[137]
	Ga(C ₂ H ₅) ₃	NH ₃	200 °C–500 °C	Si (100)	Plasma enhanced	[271]
InN	In(CH ₃) ₃	N ₂ plasma	150 °C–350 °C	Si (100) and quartz	Plasma enhanced	[157]
	In(CH ₃) ₃	N ₂ plasma	160 °C–260 °C	a-plane sapphire, Si(111), and GaN/sapphire	Plasma enhanced	[158]
	In(CH ₃) ₃	N ₂ plasma	200 °C–260 °C	Si (100), Si (111), and sapphire (0001)	Plasma enhanced	[272]
	C ₂ H ₅ In and In(CH ₃) ₃	N ₂ /H ₂ plasma or N ₂ plasma	200 °C	Si (100), Si (111), and AlN/Si (100)	Plasma enhanced	[154]
BN	BCl ₃	NH ₃	630 °C	SiO ₂ /Si (100)	Thermal	[273]
	BBr ₃	NH ₃	400 °C and 750 °C	SiO ₂	Thermal	[159]
	BCl ₃	NH ₃	227 °C	ZrO ₂ particles	Thermal	[160]
	(C ₂ H ₅) ₃ B	NH ₃	500 °C–900 °C	sapphire and Si (001)	Thermal	[274]
	(C ₂ H ₅) ₃ B	N ₂ /H ₂ plasma	250 °C–450 °C	Si (100)and quartz	Plasma enhanced	[162]
	BBr ₃	NH ₃	250 °C–750 °C	SiO ₂	Laser assisted and thermal	[161]
BN	BBr ₃	NH ₃	327 °C	Ru (0001)	Thermal	[164]
	BCl ₃	NH ₃	327 °C	Co (0001)	Thermal	[163]
AlN	AlCl ₃	NH ₃ /H ₂	350 °C	Si (100)	Plasma enhanced	[146]
	Al(CH ₃) ₃	NH ₃	325 °C–470 °C	Glass	Thermal	[147]
	Al(CH ₃) ₃	NH ₃	240 °C–370 °C	Si (100)	UV-assisted	[148]
	Al(CH ₃) ₃	N ₂ /H ₂	350 °C	Si (111)	Thermal and plasma enhanced	[144]
	Al(CH ₃) ₃	N ₂ /H ₂	250 °C	Si (100), Si (111), and sapphire	Plasma enhanced	[152]
	Al(CH ₃) ₃	N ₂ /H ₂	210 °C and 250 °C	Si (100), Si (111), and sapphire (001)	Plasma enhanced	[153]
	Al(CH ₃) ₃	N ₂ /H ₂	150 °C–300 °C	Si (100) and SiO ₂	Plasma enhanced	[275]
Al(CH ₃) ₃	N ₂	500 °C	Pt/HfO ₂ /Si(100)	Plasma enhanced	[276]	

Table 1. (Continued.)

Material	Reactant A	Reactant B	Growth temperature	Substrate	Type of ALD	References
	Al(CH ₃) ₃	NH ₃	100 °C–500 °C	Si (100), Si (111), <i>c</i> -plane sapphire, MOCVD-grown	Thermal and plasma enhanced	[141]
	Al(CH ₃) ₃	N ₂ /H ₂	200 °C	GaN on <i>c</i> -plane sapphire, and glass (Pyrex) Si (100)	Plasma enhanced	[150]
	AlCl ₃ or Al(CH ₃) ₃	NH ₃	80 °C–260 °C	Si (111)	Plasma enhanced	[143]
	Al(CH ₃) ₃	N ₂ /H ₂ or N ₂ or NH ₃	200 °C	Si (100), Si (111), and <i>c</i> -plane	Plasma enhanced	[149]
	Al(CH ₃) ₃	N ₂ /H ₂ or N ₂ or NH ₃	200 °C	Sapphire Si (111)	Plasma enhanced	[140]
	Al(CH ₃) ₃	NH ₃	280 °C	Ti/Si (100), Si (100), and glass	Plasma enhanced	[151]
	Al(CH ₃) ₃	NH ₃	200 °C	Polytetrafluoroethylene (PTFE)/SiO ₂	Plasma enhanced	[277]
	Al(CH ₃) ₃	N ₂ /H ₂	250 °C	Si (111)	Plasma enhanced	[142]
	Al(CH ₃) ₃	N ₂ /H ₂	200 °C	Si (100)	Plasma enhanced	[145]
ZnS	(C ₂ H ₅) ₂ Zn (C ₂ H ₅) ₂ Zn (C ₂ H ₅) ₂ Zn (C ₂ H ₅) ₂ Zn (C ₂ H ₅) ₂ Zn Bis(2,2,6,6-tetramethyl-3,5-heptanedionato)zinc (Zn(TMHD) ₂) (C ₂ H ₅) ₂ Zn Zn ZnI ₂ or ZnCl ₂ Zn(CH ₃) ₂ or (C ₂ H ₅) ₂ Zn (C ₂ H ₅) ₂ Zn	H ₂ S H ₂ S H ₂ S H ₂ S H ₂ S H ₂ S H ₂ S S H ₂ S H ₂ S H ₂ S/Ar	200 °C–350 °C 250 °C–400 °C 60 °C–400 °C 25 °C–160 °C 100 °C–300 °C 150 °C–375 °C 100 °C 100 °C–350 °C 300 °C–490 °C 100 °C–200 °C 60 °C–300 °C	Glass Al ₂ O ₃ Si (100) Au (111) Si (100) Quartz glass Soda lime glass and Si Si (100), Si (110), and Si (111) Al ₂ O ₃ /In ₂ O ₃ /Sn ₂ O ₃ /Al ₂ O ₃ covered soda lime glass Si (100) SiO ₂ /Si(100)	Thermal Thermal Thermal Thermal Thermal Thermal Thermal Thermal Thermal Thermal Plasma enhanced	[173] [172] [171] [168] [169] [175] [167] [278] [166] [69] [246]
CdS	Cd(CH ₃) ₂ Cd Cd(CH ₃) ₂	H ₂ S S H ₂ S	Room temperature 340 °C 100 °C–400 °C	ZnSe (100) (100)GaAs Si (100) and glass	Thermal Thermal Thermal	[177] [176] [178]
Cu _x S	Cu(thd) ₂ (thd = 2,2,6,6-tetramethyl-3,5-heptanedione) Cu(thd) ₂ Bis(N,N-di-sec-butylacetamidinato)dycopper(I) Cu ₂ (DBA) ₂ Cu ₂ (DBA) ₂ Cu(thd) ₂ Cu(acac) ₂ (acac = acetylacetonate = 2,4-pentanedionate)	H ₂ S H ₂ S H ₂ S H ₂ S H ₂ S H ₂ S	125 °C–250 °C 160 °C–260 °C 130 °C 80 °C or 135 °C 200 °C 130 °C–220 °C	Soda lime glass and Si(100) Corning 7059 glass, SnO ₂ :F, and TiO ₂ films on SnO ₂ :F glass. Si (100) and fused silica Fused quartz and silicon substrates Quartz glass Borosilicate glass and Si	Thermal Thermal Thermal Thermal Thermal Thermal	[184] [183] [182] [181] [180] [179]
PbS	Pb(tmhd) ₂ Pb(tmhd) ₂ Pb(tmhd) ₂	H ₂ S H ₂ S H ₂ S	140 °C–220 °C 160 °C 160 °C	Si (100) Si (100) Si/SiO ₂ substrates	Thermal Thermal Thermal	[186] [185] [187]
SnS	Tin(II) 2,4-pentanedionate (Sn(acac) ₂) Bis(N,N'-diisopropylacetamidinato)tin(II) [Sn(MeC(N-iPr) ₂) ₂] ₂ , Sn(amd) ₂ Sn(amd) ₂ Tetrakis(dimethylamino)tin (TDMASn, [(CH ₃) ₂ N] ₄ Sn)	H ₂ S H ₂ S H ₂ S H ₂ S	175 °C 100 °C–200 °C 200 °C 60 °C–180 °C	Si and Al ₂ O ₃ SiO ₂ and Au a-SiO ₂ SiO ₂ , Si, and glass	Thermal Thermal Thermal Thermal	[188] [189] [279] [190]
In ₂ S ₃	Indium acetylacetonate In(III) N,N'-Diisopropylacetamidinate [In-(amd) ₃]	H ₂ S H ₂ S	150 °C–160 °C 140 °C–240 °C	Si Si (100)	Thermal Thermal	[192] [191]
GaS _x	Hexakis(dimethylamido)digallium	H ₂ S	125 °C–225 °C	Si, Fused silica	Thermal	[193]
ZnSe	Zn Zn ZnCl ₂	Se Se (R ₃ Si) ₂ Se	430 °C 430 °C 400 °C	GaAs and lime glass Quartz, glass, and GaAs SiO ₂ and glass	Thermal Thermal Thermal	[195] [196] [194]
CdSe	Cd	Se	150 °C–350 °C	Si	Thermal	[197]
PbSe	Lead(II)bis(2,2,6,6-tetramethyl-3,5-heptanedionato) (Pb(C ₁₁ H ₁₉ O ₂) ₂)	Bis-(triethyl silyl) selane ((Et ₃ Si) ₂ Se)	150 °C–250 °C	Si (100)	Thermal	[280]
Cu ₂ Se, CuSe	CuCl, Cu(II) pivalate	(R ₃ Si) ₂ Se	165 °C and 400 °C	SiO ₂ and glass	Thermal	[194]
In ₂ Se ₃	InCl ₃	(R ₃ Si) ₂ Se	295 °C	SiO ₂ and glass	Thermal	[194]

Table 1. (Continued.)

Material	Reactant A	Reactant B	Growth temperature	Substrate	Type of ALD	References
ZnTe	Zn ZnCl ₂	Te	250 °C	GaAs (100)	Thermal	[281]
		(Et ₃ Si) ₂ Te	400 °C	SiO ₂ and glass	Thermal	[194]
Bi ₂ Te ₃	BiCl ₃ BiCl ₃	(Et ₃ Si) ₂ Te	165 °C	SiO ₂ and glass	Thermal	[194]
		(Et ₃ Si) ₂ Te	160 °C–300 °C	SiO ₂ /Si and soda lime glass	Thermal	[282]
GaAs	GaCl ₃	(Et ₃ Si) ₃ As	125 °C–250 °C	SiO ₂ /Si and soda lime glass substrate	Thermal	[198]
Si	Si ₂ H ₆	SiCl ₄	355 °C–385 °C	SiO ₂	Thermal	[202]
Ge	GeCl ₄	1,4-bis(trimethylsilyl)-1,4-dihydropyrazine	150 °C–300 °C	Pt	Thermal	[203]
	GeCl ₂	Atomic hydrogen	300 °C, 420 °C	Si (100)	Thermal Epitaxy	[199]
	GeCl ₄	Atomic hydrogen	300 °C	Si (100)	Thermal	[200]

significantly higher growth temperatures and resulted in relatively low GPC values [70]. The ALD window for a typical ZnO ALD process using DEZn and H₂O as precursors can nonetheless be estimated to be around 110 °C–170 °C. However, there are variations in ALD growth window even when using the same precursors in different reports [21–24, 26–70]. Although a rather ‘forgiving’ technique, it is quite possible that reactor design also affects the resulting ALD temperature window. Generally, as-grown ALD ZnO films showed intrinsic n-type conductivity which was originating from the presence of defects and impurities in the ZnO crystal. Elevated deposition temperature increased the conductivity of the films, however at high growth temperatures where CVD-growth regime takes over, film resistivity starts to increase again [21–24, 26–70]. Instead of H₂O, when O₂ plasma was used as oxygen source, ZnO films became more stoichiometric as O₂ plasma can oxidize Zn more effectively, confirming that plasma-enhanced ALD (PEALD) can be used to control the stoichiometry of ZnO films [12]. Reduction in oxygen vacancies and interstitial Zn decreases the intrinsic n-type carrier concentration of ZnO, yielding in films with higher resistivity. The growth of epitaxial ZnO films by atomic layer epitaxy is reported on *c*-plane sapphire substrate and GaN [71–85]. Despite the considerable lattice mismatch between sapphire and ZnO (~18%), still no buffer layer was necessary to obtain epitaxial ZnO films. The lattice mismatch between ZnO and GaN is lower which makes deposition of epitaxial high quality ZnO on GaN possible as well [86].

ALD of TiO₂ has been reported using several different compounds of Ti as precursors [87–101]. TiCl₄ is the most commonly used precursor which allows deposition of high quality TiO₂ material (refractive index as high as 2.6) at substrate temperatures ranging from 27 °C to 600 °C [89, 99, 102]. However, TiCl₄ is corrosive and chlorine contamination has been observed in the films grown below 100 °C [102]. Titanium alkoxides (titanium isopropoxide and titanium ethoxide) have been utilized as alternative Ti precursors for ALD of TiO₂ [88, 99, 100, 102, 103]. TiO₂ grown using titanium ethoxide showed low GPCs (0.3–0.4 Å) at substrate temperatures below 300 °C. Lower GPC obtained using titanium ethoxide might be due to larger molecular size compared with TiCl₄. However, other reasons such as incomplete ligand exchange reactions and low number of activated absorption sites might also implicate low GPCs with titanium ethoxide [99]. Moreover, alkoxides start to self-decompose around 300 °C, losing the self-limiting deposition characteristic of ALD and entering CVD growth regime [92]. Among the organometallic Ti precursors, cyclopentadienyl group based precursors have the advantage of higher thermal stability. ALD of TiO₂ has been reported using Cp*Ti(OMe)₃ and ozone reactants where ALD growth window was achieved at growth temperatures higher than 300 °C [92]. A comparative study of TiO₂ nucleation on SiO₂ and hydrogen terminated Si showed that higher GPC can be achieved mainly due to the presence of hydroxyl groups on SiO₂ surface which serve as reactive sites [95]. Tetrakis-(dimethylamido) titanium (TDMAT) and oxygen reactants were also used to grow TiO₂ and the deposited film showed p-type conductivity [104]. It

was hypothesized that p-type conductivity of TiO₂ is a result of native excess of oxygen interstitials in the grown film.

There have been a fair amount of efforts to grow Ga₂O₃ via ALD, a wide bandgap oxide material attracting recent interest for potential power device applications. In the very first report, Ga₂O₃ was grown using gallium(III) acetylacetonate (Ga(acac)₃) and H₂O/O₃ reactants and growth was achieved at deposition temperatures higher than 370 °C [105]. The amine based (Ga₂(NMe₂)₆) precursors and isopropoxide precursors have also been utilized to grow Ga₂O₃ where ALD growth window was relatively narrow [106, 107]. Ga₂O₃ has been grown using Ga(CH₃)₃ in combination with H₂O, O₂ plasma, and ozone [108, 109]. A wide temperature window (100 °C–400 °C) was observed for Ga₂O₃ grown using Ga(CH₃)₃ and oxygen plasma with a GPC of ~0.53 Å.

In₂O₃ growth has been performed initially using InCl₃ and H₂O [110]. InCl₃ chemistry required relatively high growth temperatures in the range of 300 °C–500 °C and yielded a GPC of 0.25–0.40 Å. It has also been reported that InCl₃ can etch the deposited In₂O₃ which is a limitation especially in case of coating nanoporous/3D surfaces where long precursor exposures are required [110, 111]. Nanocrystalline cubic phase In₂O₃ was grown using cyclopentadienyl indium and ozone reactants where growth temperatures were reported in the range of 200 °C–450 °C with GPCs of 1.3–2.0 Å [111]. It was reported that In₂O₃ films grown using ozone as oxygen precursor have film resistivity values higher than 10⁻² Ω cm due to the removal of native donors such as oxygen vacancies (Vo²⁺) and In interstitials (In_i³⁺) [111, 112]. In another study, In(CH₃)₃ and H₂O reactants were used to perform ALD of In₂O₃ in the temperature range between 200 °C and 250 °C, resulting in film resistivity values as low as 2.8 × 10⁻³ Ω cm along with significantly Hall mobility values reaching 84 cm² V⁻¹ s⁻¹ [113]. Yet in another interesting study, cyclopentadienyl indium and combinations of both molecular oxygen and water as co-reactants were used to grow In₂O₃ [114]. Relatively low GPC values were obtained in the temperature range of 100 °C–250 °C when oxygen or water were used individually as oxygen source. Measurements revealed that H₂O performs the function of releasing ligands from the surface while oxygen acts as an oxidizing agent and therefore a synergy between water and oxygen resulted in increased GPC (1.0–1.6 Å) values [114]. Et₂InN(SiMe₃)₂ precursor in combination with H₂O was used to perform ALD of In₂O₃ at substrate temperatures of 225 °C–250 °C where very low film resistivity (2.3 × 10⁻⁴–5.16 × 10⁻⁵ Ω cm) values were obtained which was mainly attributed to the oxygen deficient InO_x phases [115]. More recently, In₂O₃ films were grown using [1,1,1-trimethyl-N-(trimethylsilyl)silanaminato]-indium (InCA-1) and hydrogen peroxide precursors; as grown films showed high conductivity (~10⁻⁴ Ω cm) however, it was shown that N₂O plasma treatment can be applied on films to control the conductivity of materials leading to oxidation of InO_x, where oxygen vacancies act as shallow electron donors at a relatively low temperature of 150 °C [116].

Another significant oxide semiconductor is SnO₂, particularly utilized in chemical/gas sensors. Thermal ALD of

SnO₂ thin films was performed using tin halide and H₂O/O₂ precursors combination which resulted in polycrystalline SnO₂ films with (100) and (120) orientations [117–124]. Epitaxial SnO₂ films were reported using PEALD in which dibutyl tin diacetate and O₂ plasma combination was utilized [125, 126].

2.2. Nitrides

GaN growth via thermal ALD was initially reported using organometallic precursors—either Ga(CH₃)₃ or Ga(C₂H₅)₃—and NH₃ as Group-III and Group-V reactants respectively, at relatively high growth temperatures ranging from 450 °C up to 900 °C [127, 128]. Thermal ALD of GaN films was also demonstrated using GaCl₃ and NH₃ within a temperature range of 500 °C–750 °C. Better thermal stability of GaCl₃ yielded a relatively wide ALD temperature window, while in addition to oxygen, Cl impurities were detected as well [129]. In order to decrease the substrate temperature substantially, PEALD of GaN was first demonstrated using Ga(CH₃)₃ and NH₃ plasma, resulting in a self-limiting growth character within 185 °C–385 °C, which produced amorphous films with high oxygen content [130]. In an effort to decrease the oxygen contamination, hollow-cathode plasma-assisted ALD (HCPEALD) of GaN was demonstrated using Ga(CH₃)₃ and N₂/H₂ or NH₃ plasma reactants at substrate temperatures as low as 200 °C. Deposited films on Si (100) were polycrystalline with hexagonal (002) preferred orientation, however, the same preferred orientation was not observed on *c*-plane sapphire substrates [131]. Use of HCPEALD was found to be quite effective in decreasing the oxygen impurity within GaN films by more than two orders of magnitude [131–136]. Motamedi *et al* reported PEALD of GaN with Ga(C₂H₅)₃ and forming gas mixture (95% N₂/5% H₂) plasma reactants at 275 °C. Transmission electron microscopy (TEM) images revealed that the GaN films were epitaxial at the sapphire substrate interface vicinity (5 nm), transforming to polycrystalline structure for higher thickness values [137]. Interestingly, electrical conductivity measurements showed that deposited GaN films were p-type with a resistivity of 0.033 Ω cm, $1.68 \times 10^{18} \text{ cm}^{-3}$ carrier concentration, and a quite striking hole mobility of $110 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$ [137]. Si(111) might be a better substrate for HCPEALD of GaN using Ga(C₂H₅)₃ and N₂/H₂ plasma reactants as better crystalline quality and higher film density were obtained from GaN grown on Si(111) as compared to films grown on Si(100) and sapphire, which is attributed to the lower lattice mismatch and hexagonal nature of Si(111) substrate surface [138]. Another study showed that Ga(CH₃)₃ might be a better gallium source in comparison with Ga(C₂H₅)₃ for HCPEALD of GaN at low substrate temperatures, yielding GaN films with higher crystalline quality and larger grain sizes [132]. Recently, electron-enhanced ALD of GaN is reported at 100 °C and room temperature using Ga(CH₃)₃, hydrogen (H) radicals, and NH₃ as reactants [139]. The deposited films at 100 °C were polycrystalline with hexagonal wurtzite crystal structure. No oxygen contamination was reported while very significant 10

–35 at% carbon contamination was found inside GaN, leading to nanocrystalline and amorphous films. The growth mechanism for such a low temperature GaN growth is believed to result from the electron stimulated desorption (ESD) of hydrogen which produces dangling bonds facilitating Ga–N bond formation [139].

Within the III-nitride semiconductor family, ALD of AlN has been most extensively studied [140–145]. PEALD of AlN has been studied using AlCl₃ and NH₃/H₂ plasma reactants which produced AlN wurtzite (100) microcrystallites in an amorphous matrix [146]. Thermal [147] and UV assisted ALD [148] of AlN has been reported in a temperature range of 320 °C–470 °C and 240 °C–370 °C, respectively. However, in these studies self-limiting behavior was not observed as surface reactions between Al(CH₃)₃ and NH₃ takes places with sufficient rates only at those temperatures where Al(CH₃)₃ decomposition takes place. Generally, PEALD of AlN has been investigated extensively due to superior reactivity of plasma reactants which decreases the growth temperature significantly. In thermal ALD of AlN, NH₃ has been the most common nitrogen source whereas in plasma-assisted ALD of AlN, either NH₃ or N₂/H₂ plasma have been employed as nitrogen source [144]. ALD of AlN with self-limiting growth behavior has been demonstrated using a combination of Al(CH₃)₃ and N₂/H₂ plasma reactants by several research groups [141–144, 149–151]. Grown films were polycrystalline with wurtzite structure. Optical analysis was carried out and an optical band edge of 5.8–6.04 eV has been reported [141–145, 149–152]. Refractive index values of the films were reported in the range of 1.94–2.05 [141–145, 149–152]. A comparison of thermal and plasma-enhanced ALD of AlN has been reported, where it has been shown that incubation period only occurred for thermal ALD, while growth initiated without any nucleation delay with PEALD, which is attributed to higher nuclei density due to plasma [144]. PEALD grown AlN films also showed higher refractive index below 30 nm which saturated as film thickness was increased above 30 nm [144]. A comparison of as-deposited and annealed AlN films was presented which revealed that hydrogen impurities in the as grown films desorb after annealing at ≥ 400 °C and films started to oxidize after 600 °C [145]. In another report, it was argued that AlN films grown at low temperatures on sapphire have higher crystallinity when compared to the same films grown on silicon substrates [153]. Current transport mechanism was evaluated by fabricating and measuring metal-insulator-semiconductor (MIS) capacitor devices whereby ohmic conduction, trap assisted tunneling, and Frenkel–Poole (FP) emission were determined to be the main electrical transport mechanisms [150]. Trap levels in AlN films were attributed to nitrogen vacancies and DX centers formed with involvement of Si atoms in the film [150]. A comparison of different plasma chemistries for AlN ALD growth showed that N₂-only plasma is not suitable for AlN growth while films grown with NH₃ and N₂/H₂ were nitrogen rich and heavily hydrogenated [140]. Additionally, higher carbon content was found in films grown with N₂/H₂ plasma which might be originating from

an undesirable reaction occurring during the plasma step between nitrogen species and surface CH groups [140].

The most common precursors for ALD of InN are $\text{In}(\text{CH}_3)_3$ and N_2 plasma [154–157]. Atomic layer epitaxy of InN has been reported using $\text{In}(\text{CH}_3)_3$ and N_2 plasma as reactants and novel cubic or a hexagonal phase of InN were synthesized [158]. Two different atomic layer epitaxy growth temperature windows were found between 175 °C–185 °C and 220 °C–260 °C; cubic phase was grown in the low temperature window whereas common hexagonal phase of InN was found in the films grown within the higher temperature window [158]. In another investigation, it was reported that addition of H_2 plasma with N_2 plasma as nitrogen precursor produced InN films with poor crystalline quality, high level of C and O impurities, and significant void structures [154]. Films grown with N_2/H_2 plasma contained a combination of turbostratic and hexagonal phases, while films grown with N_2 -plasma only were single-phase hexagonal InN [154]. A detailed PEALD optimization study of InN has been reported using $\text{In}(\text{CH}_3)_3$ and N_2 plasma as reactants and it was found that longer N_2 plasma exposure time helped in removing carbonaceous ligands more effectively, which at the same time reduced the GPC of InN [157]. In contrast to band gap of single crystal InN (0.7 eV), an effective optical band gap of ~ 1.9 eV was extracted for ALD-grown InN [157].

Finally, boron nitride (BN) has been deposited using $\text{BCl}_3/\text{BBr}_3$ and NH_3 reactants by thermal ALD within 230 °C–750 °C [159, 160]. Films grown were either amorphous or turbostratic, in which BBr_3/NH_3 and BCl_3/NH_3 reactants were utilized. In another report, BN was deposited via laser-assisted ALD using BBr_3/NH_3 reactants which produced turbostratic BN with a relatively high deposition rate [161]. BN was also grown using $(\text{C}_2\text{H}_5)_3\text{B}$ and N_2/H_2 plasma reactants at 350 °C and 450 °C, yielding hexagonal polycrystalline film with relatively low impurities [162]. Thermal decomposition of $(\text{C}_2\text{H}_5)_3\text{B}$ was found to start around 350 °C and therefore the deposition regime of BN at higher temperatures was not self-limiting. BN has been grown by atomic layer epitaxy on Co (0001) template at 327 °C using BCl_3/NH_3 reactants, resulting in epitaxial p-type BN with an average domain size of at least 1900 Å [163]. More recently, atomic layer epitaxy of stoichiometric BN was accomplished at 600 K using BCl_3/NH_3 precursors with very low impurity levels [164].

2.3. Sulfides

One of the very first ALD process (known as atomic layer epitaxy at that time) was demonstrated in early 1975s by Suntola *et al* for the deposition of ZnS for thin-film electro-luminescent displays [165]. Despite ZnS being one of the first ALD coated semiconducting films, the literature on ALD of sulfides is narrower when compared to the vast literature on oxide ALD. Initial atomic layer epitaxy of ZnS process used elemental Zn and S as reactants at 500 °C [165]. Soon, molecular precursors such as ZnI_2 or ZnCl_2 replaced the elemental precursors which provide better ligand exchange mechanisms leading to ideal self-limiting surface reactions [166]. However, halide precursors

required higher source temperatures for vaporization and therefore higher deposition temperatures which cause halide impurity contamination, posing challenges for critical device applications [166]. In order to overcome these issues, organometallic precursors have been used to develop ZnS ALD processes. In almost all the cases, H_2S was used as S source whereas $(\text{C}_2\text{H}_5)_2\text{Zn}$ was used as Zn precursor [167–173]. Use of metal-organic precursors enabled a wide growth temperature range and considerably higher GPCs owing to their higher reactivity and thermal stability [167–173]. When $(\text{C}_2\text{H}_5)_2\text{Zn}$ and H_2S reactants were employed, GPC of ZnS decreased monotonically with increasing growth temperature which was attributed to the decrease in surface functional group coverage with temperature [69, 171]. ZnS grown with metal organic/ H_2S precursor at low temperatures was generally cubic while a change in phase appeared at higher growth temperatures (>300 °C) leading to formation of hexagonal ZnS [171, 172]. *In situ* monitoring of ZnS film growth was investigated during the initial cycles of ALD using scanning tunneling microscopy (STM) technique [168]. It was found that grain morphology is temperature-dependent and grain size increases with deposition temperature from 100 °C to 160 °C [168]. H_2S has been commonly used as S source because of its volatility and reactivity with metalorganic precursors [174]. Nevertheless, H_2S presents several challenges which includes its flammable, corrosive, and toxic character [174]. Therefore, ALD reactors need to be designed carefully for H_2S compatibility. In some reports, H_2S was generated *in situ* for ZnS ALD to eliminate the need to store high pressure H_2S gas [171, 175]. The H_2S precursor was generated by heating thioacetamide to 150 °C in an inert atmosphere, producing acetonitrile and H_2S [171].

There are only a few reports on ALD of CdS. Atomic layer epitaxy of CdS is reported where Cd and S precursors were used at substrate temperature of 340 °C [176]. Atomic layer epitaxy of CdS is also performed using $\text{Cd}(\text{CH}_3)_2$ and H_2S on a ZnSe (100) substrate at room temperature [177]. Annealing of CdS is performed at 250 °C which produced zincblende CdS, a crystal structure similar to the substrate [177]. Bakke *et al* reported ALD of CdS using $\text{Cd}(\text{CH}_3)_2$ and *in situ* generated H_2S on Si (100) or glass substrate in a temperature range of 100 °C–400 °C [178]. Films grown at low temperatures were a mixture of wurtzite and zincblende crystal structures while films grown at higher temperature (400 °C) were dominantly wurtzite [178].

Cu_xS exists in five solid phases at room temperature: chalcocite ($x = 2$), djurleite ($x = 1.96$), digenite ($x = 1.8$), anilite ($x = 1.75$), and covellite ($x = 1$). ALD of Cu_xS has been reported by using three different Cu precursors: $\text{Cu}(\text{thd})_2$ (thd = 2,2,6,6-tetramethyl-3,5-heptanedione), bis(N,N-di-sec-butylacetamidinato)dicopper(I) $\text{Cu}_2(\text{DBA})_2$, and copper(II) acetylacetonate [179–184]. In all these studies, H_2S was used as the sulfur precursor. Depending on different process parameters and precursors employed, different stoichiometry and compositions of Cu_xS were obtained. With the use of $\text{Cu}_2(\text{DBA})_2$ precursor as Cu source material, mainly chalcocite phase (Cu_2S) of Cu_xS was obtained [181, 182]. On the other hand, with the use of $\text{Cu}(\text{thd})_2$ precursor, either covellite (CuS) phase or digenite ($\text{Cu}_{1.8}\text{S}$) phase of Cu_xS was synthesized [183, 184]. When $\text{Cu}(\text{acac})_2$ precursor

was employed, researchers obtained p-type Cu_xS film with either multiphase compounds (made of digenite $\text{Cu}_{1.8}\text{S}$, chalcocite Cu_2S , djurleite $\text{Cu}_{31}\text{S}_{16}$, and covellite CuS) or single-phase digenite $\text{Cu}_{1.8}\text{S}$ film depending on number of growth cycles [179].

ALD of PbS films has been studied by only one research group [185–187]. Bis(2,2,6,6-tetramethyl-3,5-heptanedionato)lead(II) ($\text{Pb}(\text{tmhd})_2$) and H_2S were the precursors employed at a growth temperature of 160 °C [185–187]. The grown PbS film was polycrystalline with grain sizes ranging from 30 to 150 nm. Effect of size quantization on band gap of ALD grown PbS were demonstrated by fabricating PbS quantum wells with sub-10 nm thickness [185]. Bandgap of the films was varied from 0.4 to 2.75 eV by varying only the number of ALD cycles [185].

ALD of another sulfide semiconductor, SnS was initially reported by Kim *et al* using $(\text{Sn}(\text{acac})_2)$ and H_2S reactants [188]. GPC of SnS was relatively low (0.24 Å); the reason might be that Sn–O bonds (532 kJ mol⁻¹) are stronger than Sn–S bonds (464 kJ mol⁻¹) which results in an unfavorable thermodynamic enthalpy change [188]. SnS growth on Al_2O_3 substrate had nucleation issues and displayed very small GPCs which might be caused by $\text{Al}(\text{acac})^*$ site blocking surface species [188]. Films contained 15–20 at% oxygen after air exposure and such oxidized SnS films exhibited a band gap of ~1.87 eV, higher than the reported SnS bulk band gap value (~1.3 eV) [188]. Higher GPCs (0.86–0.90 Å) were obtained with the use of Bis(*N,N'*-diisopropylacetamidinato)tin(II) [$\text{Sn}(\text{MeC}(\text{N}-i\text{Pr})_2)_2$], $\text{Sn}(\text{amd})_2$ tin precursor at lower growth temperatures [189]. Impurities in the film deposited with $\text{Sn}(\text{amd})_2$ precursors were negligible and films were p-type with a band gap value of 1.30–1.42 eV which is closer to bulk band gap value of SnS films [189]. ALD of SnS has also been reported with tetrakis(dimethylamino)tin (TDMASn , $[(\text{CH}_3)_2\text{N}]_4\text{Sn}$) precursor where it has been shown that crystal structure of SnS_x can be tuned by changing the growth temperature [190]. Below 120 °C, SnS_x films were amorphous while SnS_x films were SnS_2 hexagonal at 140 °C and 150 °C, and orthorhombic above 160 °C [190].

ALD of In_2S_3 has been reported by McCarthy *et al* using In(III) *N,N'*-diisopropylacetamidinate ($\text{In}(\text{amd})_3$) and H_2S reactants in a temperature range of 140 °C–240 °C [191]. Deposited films were n-type with free electron concentrations up to 10¹⁸ cm⁻³ and carrier mobilities in the order of ~1 cm² V⁻¹ s⁻¹ [192]. There is a single report for ALD of GaS_x in which films were synthesized using hexakis(dimethylamido)digallium and H_2S reactants at a growth temperature range of 125 °C–225 °C [193]. Growth was self-limiting in the temperature range of 125 °C–225 °C while the films were amorphous as promising lithium ion battery anode material. For further reading about ALD of sulfides, reader may consult to the review of Dasgupta *et al* which specifically focuses on ALD of metal sulfide materials [174].

2.4. Selenides and tellurides

ALD of selenides and tellurides have been only limited to selenides and tellurides of Zn and Cd, as those are rare cases

where constituent elements can be used as precursors [194–197]. Selenides and tellurides are less explored because their hydride compounds are toxic and would require extensive safety precautions. Alkyl compounds of selenium and tellurium have been more commonly employed as CVD reactants but in ALD they are unable to provide efficient ligand exchange reactions with the common metal precursors [194]. In a breakthrough discovery, ALD of various selenides and tellurides has been reported using alkylsilyl compounds of Se and Te [194]. Compared to common alkyls and alkylamides of Se and Te, $(\text{R}_3\text{Si})_2\text{Se}$ and $(\text{R}_3\text{Si})_2\text{Te}$ offered straight forward elimination of ligands of the metal precursors. In $(\text{R}_3\text{Si})_2\text{Se}$ and $(\text{R}_3\text{Si})_2\text{Te}$, there is an unfavorable hard-soft Lewis acid-base pair, as in these compounds a hard Lewis acid is bonded to the heavy group 16 elements which are soft Lewis bases. When there is an exchange reaction of these compounds with metal chlorides, silicon becomes bonded to harder base thus allowing an easy ligand exchange reaction [194].

2.5. Arsenides

To date, there is only one recent report on ALD of arsenide compounds where synthesis of GaAs was demonstrated. Dechlorosilylation reaction between GaCl_3 and $(\text{Et}_3\text{Si})_3\text{As}$ precursors was found to be successful in depositing GaAs films via ALD [198]. The films were uniform, amorphous, and stoichiometric, while film crystallization was achieved by post-deposition annealing process [198].

2.6. Elemental semiconductors

ALD of elemental semiconductors is less explored due to the challenge of synthesizing crystalline group-IV semiconductor films with low impurities [199, 200]. ALD of Si was initially explored using $\text{Si}(\text{C}_6\text{H}_{10})_2$ precursor [201]. Upon reacting with substrate, hydrocarbon rings of $\text{Si}(\text{C}_6\text{H}_{10})_2$ precursor transform into dimethylbutadiene (C_6H_{10}), which is very volatile and readily desorb. This chemistry provided Si with high level of carbon impurities. ALD of Si was demonstrated using Si_2H_6 and SiCl_4 precursors in temperature range of 355 °C–385 °C [202]. Films were smooth with arithmetic average roughness of 0.26 nm, however, no elemental characterization data was reported from the bulk of film.

Ge ALD was performed using alternating exposures of GeCl_4 and atomic hydrogen. After the first self-limiting exposure of GeCl_4 , atomic hydrogen extracted surface terminating chlorine in the second exposure which resulted in Ge film growth [200]. Recently, Ge ALD was demonstrated using GeCl_4 and 1,4-bis(trimethylsilyl)-1,4-dihydropyrazine as reducing agent. Smooth continuous films were obtained only on platinum substrates. X-ray photoelectron spectroscopy (XPS) and x-ray diffraction (XRD) data revealed the formation of PtGe_2 alloy initially, followed by smooth Ge film growth [203].

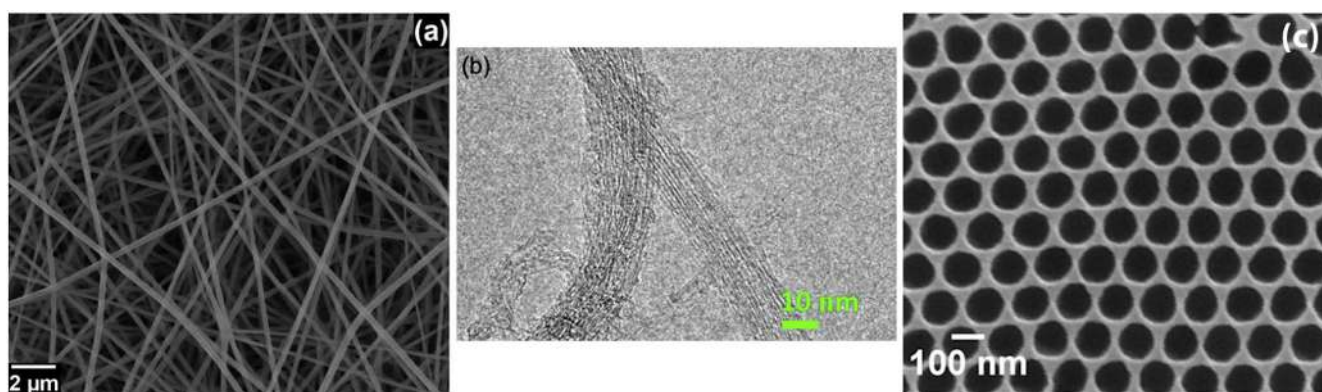


Figure 1. Various nanostructured templates used for template assisted ALD. (a) SEM image of nylon 6,6 polymeric nanofibers. (b) TEM image of single wall carbon nanotubes. (c) SEM image of porous AAO template. Reprinted with permission from [284, 335]. Copyright (2014, 2015) AIP publishing, Elsevier.

3. Nanostructured semiconductor growth via ALD

Mainly two approaches exist for nanostructured materials growth via ALD: (i) Bottom-up growth via catalyst-assisted or catalyst-free method (ii) Template-assisted growth. In template-assisted strategy, material growth is carried out on nanostructured templates (figure 1) such as carbon nanotubes, polymers, or anodic aluminum oxide (AAO) [283–285]. Subsequent to growth, template material might be removed via high-temperature treatment (calcination) or physical/chemical etching to obtain various kinds of freestanding functional nanostructures.

Bottom up growth method can be further classified into mainly two types: catalyst-assisted and catalyst-free methods. Catalyst-assisted growth typically occurs via vapor liquid solid growth where nanostructures are fabricated along a supersaturated catalytic liquid metal such as Ni or Au [283]. Catalyst free methods generally utilize selective area growth of preferential growing crystallographic planes over non-preferential planes by altering the processing conditions in order to favor growth of material in one dimension [286, 287]. In certain chemistries of ALD, growth of islands is observed rather than formation of continuous films during the initial stages of deposition [288–290]. Quantum dots and nanoparticles are synthesized by taking advantage of the so-called incubation period of ALD. This way of synthesizing 0D nanostructures can also be classified as catalyst-free bottom up growth approach. Below we summarize the reported major achievements in the field of 0D, 1D, 2D, and core-shell structured semiconductor materials.

3.1. 0D structures

Table 2 provides a summary of ALD-grown 0D semiconductor nanostructures. ALD of ZnO has been carried out on SiO₂ film having nanoscale voids between them which allow subsequent deposition within the voids of the films [291, 292]. ZnO nanodots were fabricated in this way within SiO₂ film and resulted in a significant blue shift in the photoluminescence properties of ZnO, mainly due to the quantum confinement effect. ZnO filling between voids of a SiO₂ film

has affected the mechanical, photoluminescence, and photosensitivity properties of the film [291, 292]. Nanoparticulate TiO₂ growth has been reported on CNTs during the incubation period of TiO₂ growth [293]. TiO₂ was grown in uniform thin film form on CNTs at 100 °C, but the growth at 300 °C resulted in nanoparticles during the initial cycles of the ALD process [293]. It was argued that island ALD growth mode at 300 °C occurred because of recombination between the byproducts and TiCl₄ molecules [293]. Fabrication of TiO₂ nanoparticle chains has been reported on CNTs by calcination of conformally TiO₂ coated multi-walled carbon nanotubes (MWCNTs) [294]. During annealing process, thin tubular layer of TiO₂ has been transformed into nanoparticle chains with an enhanced ultra-high surface area [294].

Quantum dots of semiconducting PbS (figure 2) and CdS have been synthesized by utilizing the incubation period observed during the initial stages of ALD where growth of islands is observed rather than formation of continuous films [288–290]. Note that this type of growth mode has only been observed in certain ALD chemistries [288, 289]. This very strategy of synthesizing quantum dots using ALD provides several unique advantages. Size of quantum dots can be varied by simply changing the number of growth cycles. Excellent infiltration of quantum dots can be obtained on high-surface area nanostructured templates by utilizing the high conformality of ALD processes. Moreover, as opposed to conventional quantum dot fabrication techniques, ALD eliminates the use of solutions and solution-based byproducts [288–290]. Islands with sub-10 nm diameters were observed during the initial ALD cycles (10–40 growth cycles) of PbS on Si nanowire array (figure 2) and PL measurements showed a blue shift when number of ALD cycles are decreased confirming successful quantum confinement in PbS quantum dots (figure 3) [290].

3.2. 1D structures

ALD grown 1D nanostructures are summarized in table 3. Majority of reports on ZnO nanostructures utilized nanoscale templates with the intention of disposing the template after deposition in order to obtain various kinds of ZnO

Table 2. Overview of the growth conditions employed to synthesize 0D nanostructures using ALD. The materials deposited, the morphology, precursors used, the temperature during ALD, and preparation method are mentioned.

Material	1st precursor	2nd precursor	Growth temperature	Morphology	Preparation method	References
ZnO	Zn(C ₂ H ₅) ₂	H ₂ O	180 °C	Quantum dots	ALD of ZnO on silica nanoparticles	[292]
	Zn(C ₂ H ₅) ₂	H ₂ O ₂	200 °C, 300 °C	Nanodots	ALD of ZnO on Si and SiO ₂ films	[208]
	Zn(C ₂ H ₅) ₂	H ₂ O	200 °C	Nanoparticles	ALD of ZnO on nylon nanofibers	[295]
TiO ₂	Tetrakis(dimethylamido) titanium (TDMAT)	O ₃	100 °C	Nanoparticle chains	TiO ₂ growth on carbon nanotubes followed by annealing	[294]
	TiCl ₄	H ₂ O	200 °C, 300 °C	Nanoparticles	Growth during initial growth cycles of TiO ₂ on carbon nanotubes	[293]
CdS	Dimethyl cadmium	H ₂ S	150 °C	Quantum dots	Utilizing the incubation period of CdS growth by ALD on TiO ₂	[288]
PbS	Bis(2,2,6,6-tetra-methyl-3,5-heptanedionato)lead(II) (Pb(tmhd) ₂)	H ₂ S	160 °C	Quantum dots	Utilizing the nucleation stage of ALD PbS growth	[290]
	Bis(2,2,6,6-tetra-methyl-3,5-heptanedionato)lead(II) (Pb(tmhd) ₂)	H ₂ S	160 °C	Quantum dots	Utilizing the nucleation stage of ALD PbS growth	[289]
SnSe	Et ₄ Sn	H ₂ O	200 °C–450 °C	Sphere shaped particles	Growth temperature dependent morphology of SnSe structures	[296]

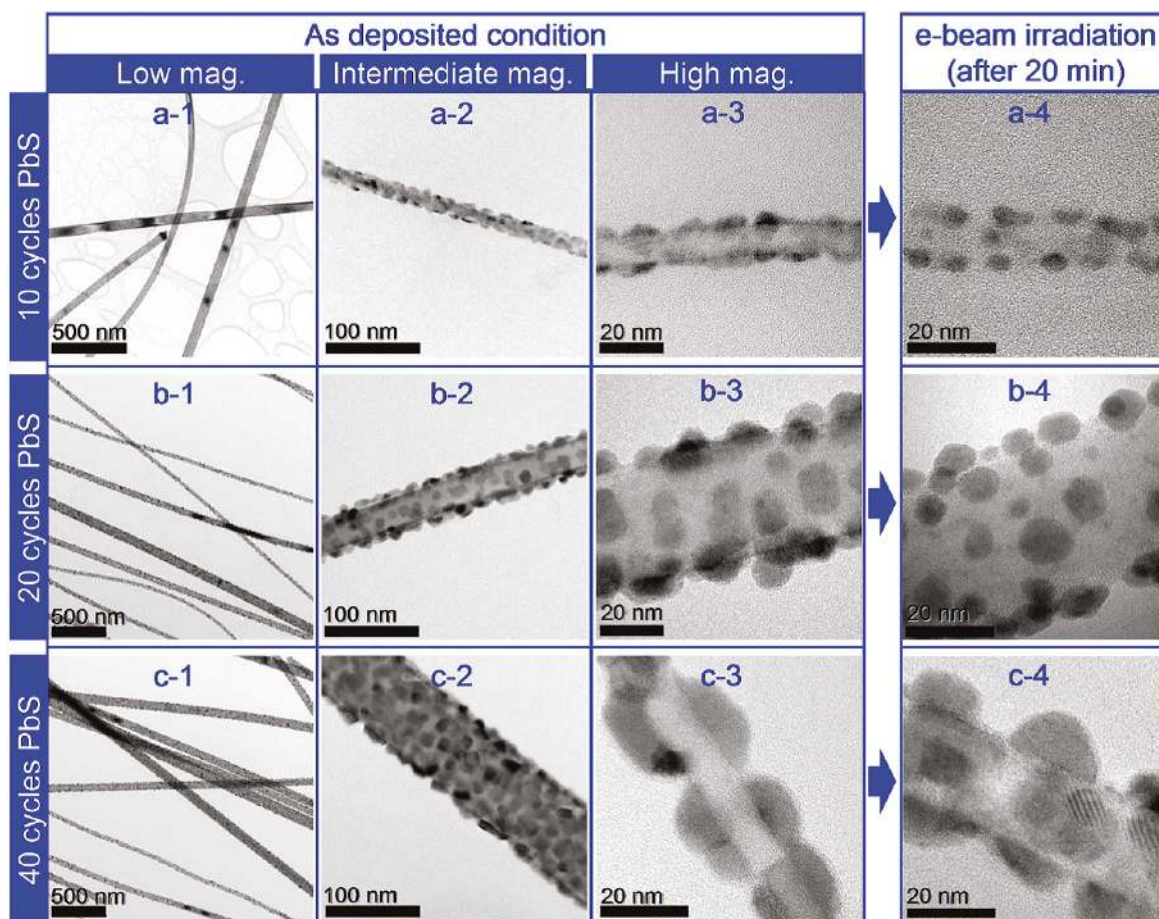


Figure 2. TEM images of Si nanowires coated by ALD PbS shown at different magnifications after (a) 10 cycles, (b) 20 cycles, and (c) 40 cycles. Also shown is the effect of 20 min e-beam irradiation on the dot-coated Si nanowires. (As a result of the e-beam irradiation process, the crystallite morphology changes to a dome shape to lower the surface to volume ratio providing better contrast in TEM imaging.) Reprinted with permission from [290]. Copyright (2011) American Chemical Society.

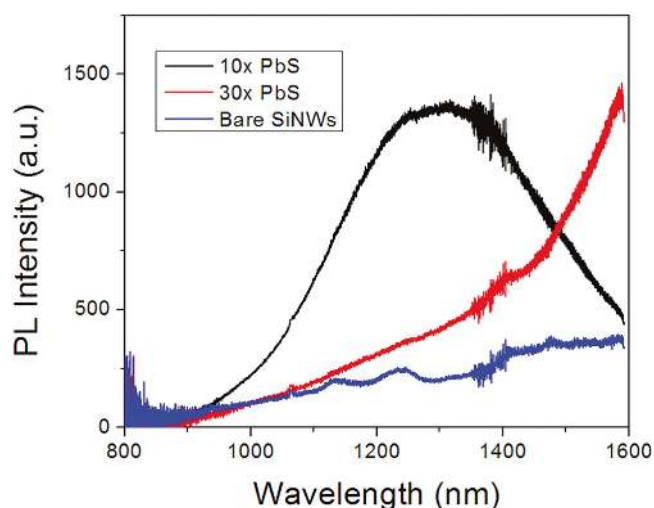


Figure 3. Photoluminescence spectra for bare Si nanowires (blue), 10 cycles of ALD PbS (black), and 30 cycles of ALD PbS (red). Reprinted with permission from [290]. Copyright (2011) American Chemical Society.

nanostructures [297–301]. Mainly three types of templates have been used: anodized alumina (AAO) templates, polymeric templates, and bio-nano-templates made out of biological materials [298, 301–304]. These templates have also been used to study the conformality of ZnO as well as to make multi-segmented ZnO nanotubes [305, 306]. ZnO growth inside the pores of AAO was limited by precursor diffusion into the high aspect ratio wells and therefore long exposure times were necessary to obtain conformal coatings (120 s exposure time for aspect ratio of ~ 5000) [305, 306]. Moreover, with the utilized long exposure times, also extensive purging times were required to ensure the complete removal of the excess reactants and byproducts which will ensure the elimination of any unwanted gas-phase CVD reaction.

ZnO 1D structures such as nanorods or nanowires have been fabricated by utilizing lithography techniques or by performing growth directly on nanostructured templates [307–312]. In one study, combination of phase-shift lithography and plasma was utilized to synthesize a photoresist nanodot pattern on Si which was used to etch Si afterwards

Table 3. Overview of the growth conditions employed to synthesize 1D nanostructures using ALD. The materials deposited, the morphology, precursors used, the temperature during ALD, and preparation method are mentioned.

Material	1st precursor	2nd precursor	Growth temperature	Morphology	Preparation method	References
ZnO	Zn(C ₂ H ₅) ₂	H ₂ O	250 °C	Nanotube and nanorod arrays	Deposition of ZnO on AAO/Si templated followed by AAO etching	[308]
	Zn(C ₂ H ₅) ₂	H ₂ O	150 °C	Nanotubes	ALD of ZnO on aerogels fibrils followed by calcination	[316]
	Zn(C ₂ H ₅) ₂	H ₂ O	150 °C	Nanotubes	ALD of ZnO on Polyacrylonitrile fibers followed by annealing or O ₂ plasma treatment	[299]
	Zn(C ₂ H ₅) ₂	H ₂ O	120 °C	Nanotubes	ALD of ZnO on SAMs-AAO	[306]
	Zn(C ₂ H ₅) ₂	H ₂ O	200 °C	Nanotubes	ALD of ZnO on Polyvinylacetate and AAO followed by annealing or etching	[337]
	Zn(C ₂ H ₅) ₂	H ₂ O	150 °C	Rod like morphology	ALD ZnO on MgO films	[338]
	Zn(C ₂ H ₅) ₂	H ₂ O	177 °C	Nanorods	Hydrothermal ZnO growth on ALD ZnO seed layer	[321]
	Zn(C ₂ H ₅) ₂	H ₂ O	100 °C	Nanorods	Hydrothermal ZnO growth on ALD ZnO seed layer	[300]
	Zn(C ₂ H ₅) ₂	H ₂ O	70 °C	Nanorods	Using polymer/Si porous structures to direct ALD of ZnO	[297]
	Zn(C ₂ H ₅) ₂	H ₂ O	100 °C	Nanorods	Wet chemical procedure on ALD grown ZnO	[320]
	Zn(C ₂ H ₅) ₂	H ₂ O	200 °C	Nanorods	ZnO deposition on high aspect ratio Si microwire arrays	[324]
	Zn(C ₂ H ₅) ₂	H ₂ O	200 °C	Nanorods	ALD of ZnO on AAO	[310]
	Zn(C ₂ H ₅) ₂	H ₂ O	200 °C	Nanorods	ALD of ZnO on AAO followed by chemical etching of AAO	[312]
	Zn(C ₂ H ₅) ₂	H ₂ O	70 °C–300 °C	Nanofibers	ALD of ZnO on inner shell membrane of hen's egg	[303]
	Zn(C ₂ H ₅) ₂	H ₂ O	150 °C	Nanofibers	ALD of ZnO on Polyvinylacetate nanofibers followed by annealing	[298]
	Zn(C ₂ H ₅) ₂	H ₂ O	200 °C	Nanofibers	ALD of ZnO on nylon nanofibers	[302]
	Zn(C ₂ H ₅) ₂	H ₂ O	150 °C	Nanofibers	ALD of ZnO on polyvinylpyrrolidone (PVP) fibers followed by annealing	[301]
	Zn(C ₂ H ₅) ₂	H ₂ O	200 °C	Nanofibers	ALD of ZnO on nylon nanofibers	[295]
	Zn(C ₂ H ₅) ₂	H ₂ O	50 °C–130 °C	Nanofibers	ALD of ZnO on polyacrylonitrile nanofibers	[339]
	Zn(C ₂ H ₅) ₂	H ₂ O	180 °C, 300 °C	Nanopillars	Preferential ALD of ZnO on ZnO seed layer	[304]
Zn(C ₂ H ₅) ₂	H ₂ O	200 °C	Nanowires	Hydrothermal growth of ZnO on ALD grown ZnO seed layer	[323]	
Zn(C ₂ H ₅) ₂	H ₂ O	115 °C	Nanowires	Substitution of Si nanowires by ZnO NWs with a dry etching technique and atomic layer deposition	[307]	

Table 3. (Continued.)

Material	1st precursor	2nd precursor	Growth temperature	Morphology	Preparation method	References
	Zn(C ₂ H ₅) ₂	H ₂ O	200 °C	Nanoneedles	Hydrothermal growth of ZnO on ALD ZnO/ PAN nanofibers	[325]
	Zn(C ₂ H ₅) ₂	H ₂ O	200 °C	Nanofibers	ALD of ZnO on nylon nanofibers during incubation period	[295]
TiO ₂	Titanium(IV) isopropoxide	H ₂ O	70 °C	Nanotubes	TiO ₂ growth into AAO membranes	[326]
	TiCl ₄	H ₂ O	100 °C	Nanotubes	TiO ₂ growth into AAO membranes	[328]
	Titanium tetraisopropoxide	H ₂ O	150 °C, 250 °C	Nanotubes	TiO ₂ growth into AAO membranes and carbon nanotubes	[327]
	TiCl ₄	H ₂ O	250 °C	Nanotubes	TiO ₂ growth on carbon nanotubes	[329]
	TiCl ₄	H ₂ O	250 °C–300 °C	Nanotubes	TiO ₂ growth into AAO membranes	[340]
	TiCl ₄	H ₂ O	150 °C	Nanotubes	TiO ₂ growth into AAO membranes	[330]
	TiCl ₄	H ₂ O	600 °C	Nanorods	TiO ₂ growth into AAO membranes	[341]
Ga ₂ O ₃	Gallium (III) alkyl amidinate [mono- acetamidinatodiethylgallium(III), compound 1]	H ₂ O, O ₂	450 °C	Nanowires	Self-seeding growth	[342]
SnO ₂	SnCl ₄	H ₂ O	150 °C–400 °C	Nanotubes	SnO ₂ growth into AAO membranes fol- lowed by polishing and etching	[331]
	Tin(IV) alkoxides	Carboxylic acid	75 °C–250 °C	Nanotubes	SnO ₂ growth on carbon nanotubes and boron nitride nanotubes	[332]
	SnCl ₄	H ₂ O	200 °C–400 °C	Nanotubes	SnO ₂ growth on carbon nanotubes	[343]
	Tin tert-butoxide	Acetic acid	200 °C	Nanotubes	SnO ₂ growth on carbon nanotubes	[344]
GaN	Ga(CH ₃) ₃	N ₂ /H ₂ plasma	200 °C	Flexible nanofibers	GaN growth on electrospun nanofibers	[285]
AlN	Al ₂ (CH ₃) ₆	NH ₃ plasma	200 °C	Nanofibers	GaN growth on electrospun nanofibers fol- lowed by calcination	[333]
ZnS	Zn(C ₂ H ₅) ₂	H ₂ S	75 °C–180 °C	Nanotubes	ZnS growth on AAO template	[334]
Cu ₂ S	Bis(N,N'-disec-butylacetamidinato)dicopper(I) (CuAMD)	H ₂ S	135 °C	Nanotubes	ALD of Cu ₂ S on single walled carbon nanotubes	[335]
GaS _x	Ga ₂ (NMe ₂) ₆	H ₂ S	150 °C	Nanotubes	ALD of GaS _x on single walled carbon nanotubes	[336]

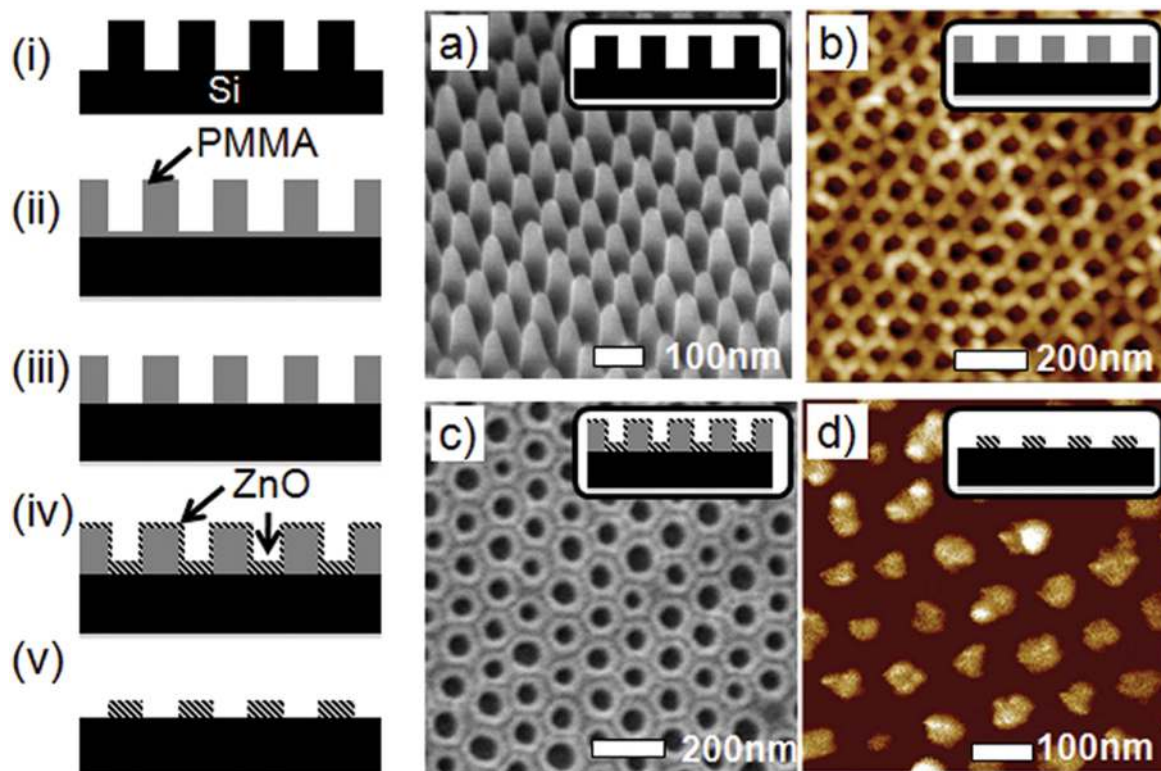


Figure 4. (i)–(v) Illustration of steps to fabricate ZnO nanoarrays using selective area ALD within templates defined by copolymer-derived NIL molds. (i) High-resolution silicon nanopillar molds (as shown in panel (a) as field effect scanning electron microscope (FESEM) image measured at 45° tilt). (ii) Nanoporous polymethylmethacrylate (PMMA) films obtained upon NIL (as shown in panel (b) by tapping mode atomic force microscope (AFM) image) and subsequently exposed to controlled O₂ plasma reactive ion etching (RIE) to obtain (iii) porous templates with through-holes. (iv) ALD of ZnO on the nanoporous templates (as shown by FESEM image in panel (c)), followed by removal of templates to obtain (v) ZnO nanoarrays (as shown by tapping mode AFM image in panel (d)). Reprinted with permission from [297]. Copyright (2012) American Chemical Society.

[307]. Another coating of photoresist and subsequent etching of Si nanowires resulted in a mask for ALD of ZnO nanowires. In another report, polymer templates synthesized through a combination of block copolymer lithography (BCL) and nanoimprint lithography (NIL) were used as a nanotemplate for subsequent ALD to obtain highly oriented and high-quality ZnO nanopatterns (illustration of fabrication steps are summarized in figure 4) [297]. The fabricated uniform array of ZnO nanostructures possessed sub-100 nm feature and spatial resolutions, exhibiting narrow distributions in size and separation, and superior mechanical stability. AAO template has been commonly used for ZnO nanorod fabrication; AAO features long-range ordered vertically aligned nanopores with varying aspect ratios whose dimensions can be easily controlled by adjusting the process conditions [308, 310, 312, 313]. It is a fairly simple technique to obtain ZnO nanorods by growing on these templates followed by the removal process of AAO, usually accomplished by chemical wet etching. The pulse/exposure time of the precursors needed to conformally coat inside the AAO pores is typically long as precursor molecules need time to diffuse-in and react with the available adsorption sites within the high aspect ratio template walls.

Low temperature ALD of ZnO has enabled the use of bio-templates in ZnO nanostructuring. Cicada wings have been used as templates to fabricate ZnO nanopillars by taking advantage of the fact that nanopillars on the wing surface block ZnO growth [314, 315]. Other studies utilized fibrils of hen's egg shell and nanocellulose fibers as bio-nanotemplates for ZnO coating [303, 316]. Figure 5 shows the fabricated ZnO nanotubes through bio templating; in this strategy, first nanocellulose hydrogel is dried to aerogel, followed by coating with ZnO ALD to form composite organic/inorganic nanofibers, and finally calcinated to inorganic hollow nanotubes [316]. Peptide amphiphile nanofibrous templates were used as a soft template for ALD of ZnO and TiO₂ and resulting functional nanonetworks (figure 6) found applications in photocatalysis for organic dye degradation and as anodic material in dye sensitized solar cells [317, 318]. Overall, the use of bio-templates in ZnO nano-structuring is relatively less explored and this field might hold potential for future device applications.

There also are studies in which fabrication of ZnO nanorods or nanodots has been reported without using any templates. These studies take advantage of the incubation period of ZnO growth to form ZnO islands which can then be used as seeds for the growth of nanopillars [208, 304, 319]. In some reports, ALD-grown ZnO thin films have been utilized

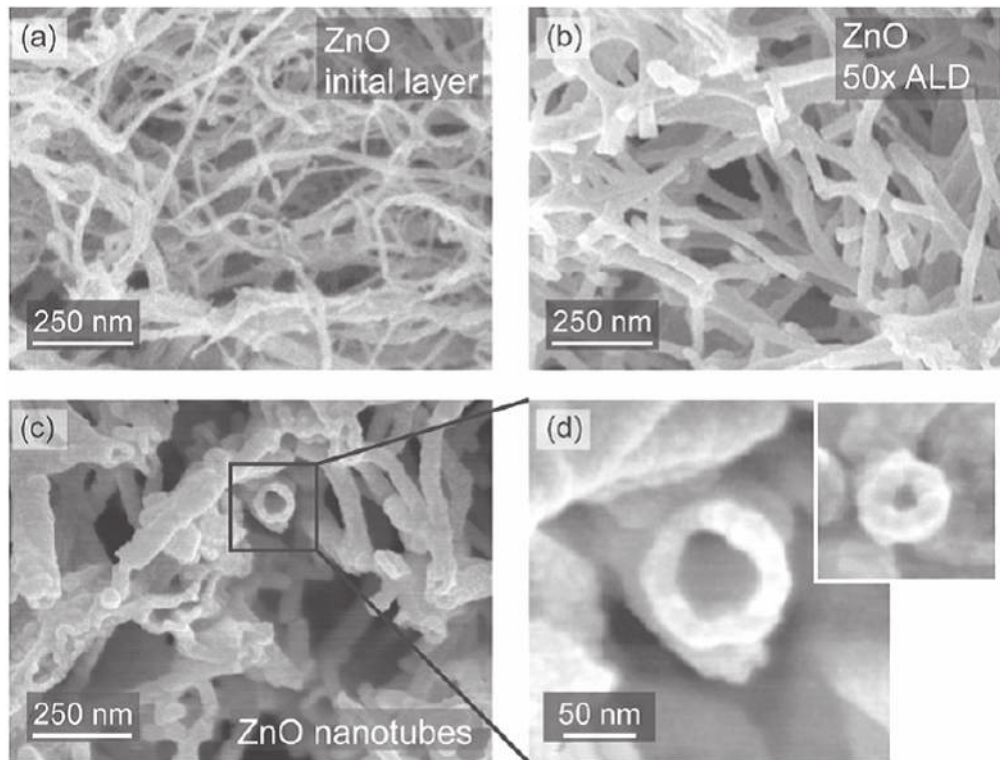


Figure 5. (a) A thin uniform ZnO layer formed on nanocellulose fibrils after initial exposure to the zinc precursor. (b) ZnO layer thickness is increased upon the ALD process (here 50 cycles). (c) Calcinated, hollow ZnO nanotubes are visibly rough. (d) Close-ups on ZnO nanotubes show that they are hollow. Reprinted with permission from [316]. Copyright (2011) American Chemical Society.

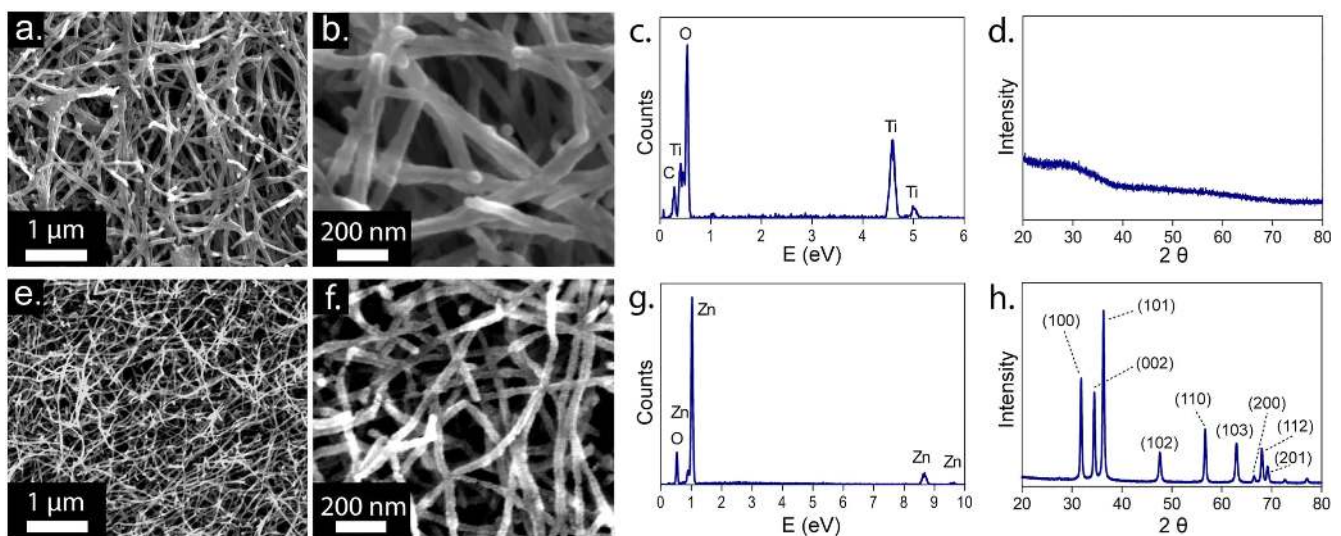


Figure 6. Characterization of as-synthesized TiO₂ and ZnO nanonetworks deposited with 350 and 100 ALD cycles, respectively. (a) and (b) Scanning electron microscope (SEM) images, (c) energy dispersive x-ray spectroscopy (EDX) spectrum, and (d) XRD pattern of TiO₂ nanonetworks. (e) and (f) SEM images, (g) EDX spectrum, and (h) XRD pattern of ZnO nanonetworks. Reprinted with permission from [318]. Copyright (2013) Nature publishing group.

as template for the subsequent growth of ZnO nanorods using hydrothermal process [320–324]. In these reports, ALD-grown ZnO provides a high-quality textured film to either act as a seed layer for nanorod growth or to protect the substrate from reagents of hydrothermal process. In another approach, ALD ZnO coated polyacrylonitrile (PAN) nanofibers were

used as a template for over growth of ZnO nanoneedles via hydrothermal growth (figure 7) [325].

Nanostructured TiO₂ fabrication has been reported mostly in the form of nanotubes and the most common precursors utilized are TiCl₄ and H₂O [326–330]. Majority of reports utilized AAO as a template for TiO₂ growth which

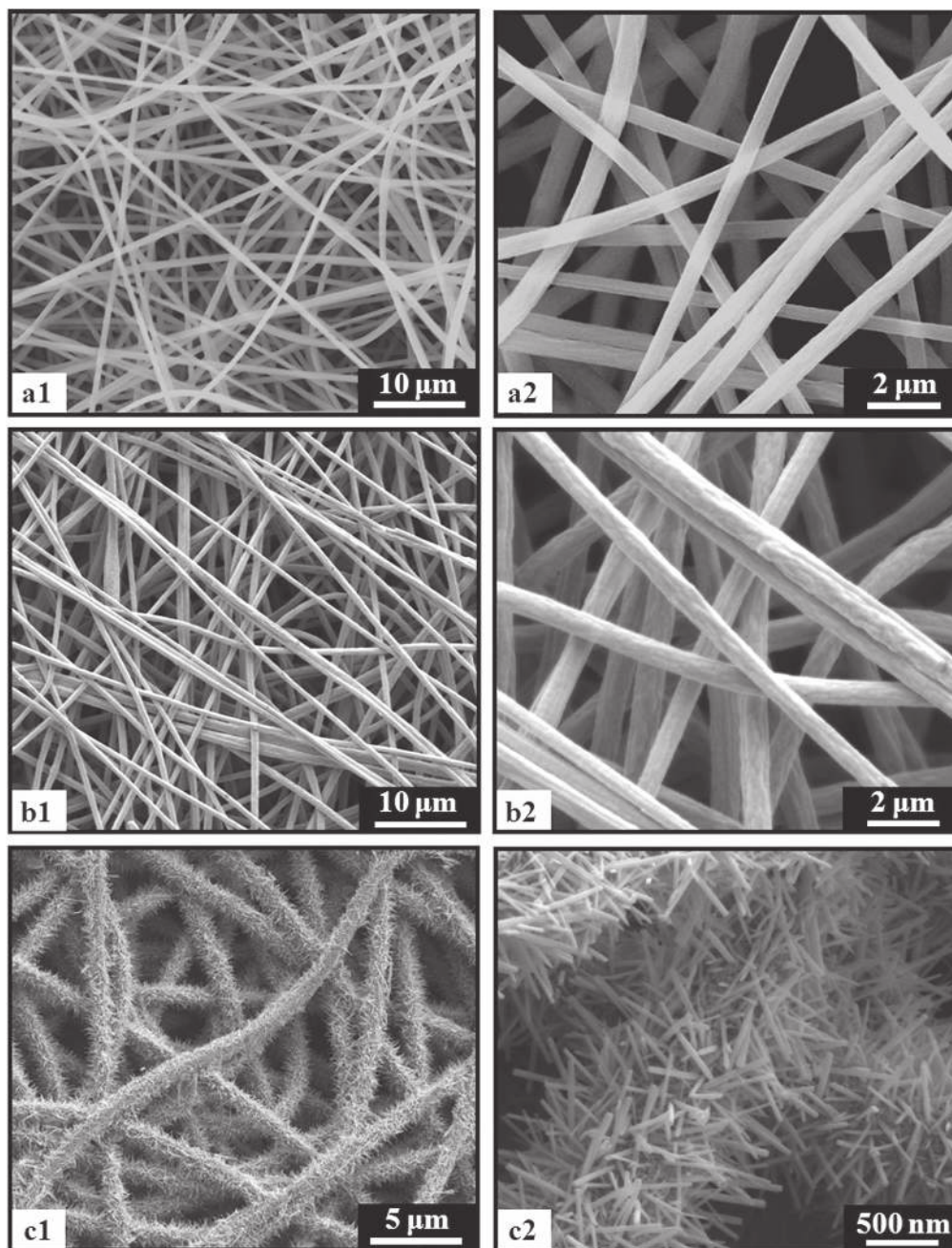


Figure 7. Representative SEM images of (a1 and 2) pristine PAN, (b1 and 2) PAN/ZnO seed, and (c1 and 2) PAN/ZnO needle nanofibers at different magnifications. Reprinted with permission from [325]. Copyright (2014) Elsevier.

was subsequently etched using NaOH solution to obtain TiO₂ nanotubes [326–328]. Crystal morphology of TiO₂ nanotubes has been tuned from amorphous to anatase phase when growth temperature was increased from 150 °C to 250 °C [327]. Figure 8 shows TiO₂ nanotubes fabricated via titania deposition into AAO membranes followed by AAO removal through etching [328].

SnCl₄ and H₂O precursors were employed to grow SnO₂ nanotubes using AAO as a template followed by etching of AAO [331]. Interestingly, three temperature-dependent growth modes were observed (figure 9): (i) layer-by-layer growth at temperature $T < 200$ °C; (ii) layer-by-particle growth at temperature 200 °C $\leq T < 400$ °C; (iii) evolutionary particles

at temperatures $T \geq 400$ °C [331]. It was believed that layer-by-layer growth occurred at low temperatures because of ligand exchange reactions and negligible change in reactive sites. Layer-by-particle growth occurred because of competing effects between ligand exchange and chlorination, i.e., lower growth temperatures prompt ligand exchange while higher temperatures can cause chlorination. Lastly, the evolutionary particle growth occurred at high growth temperatures mainly because of chlorination effects which caused certain nucleation sites on the substrate [331]. The layers were amorphous whereas particles were identified as crystalline. Hence in this way, morphology and phase of SnO₂ nanotubes were controlled via ALD growth temperature [331]. In another report it

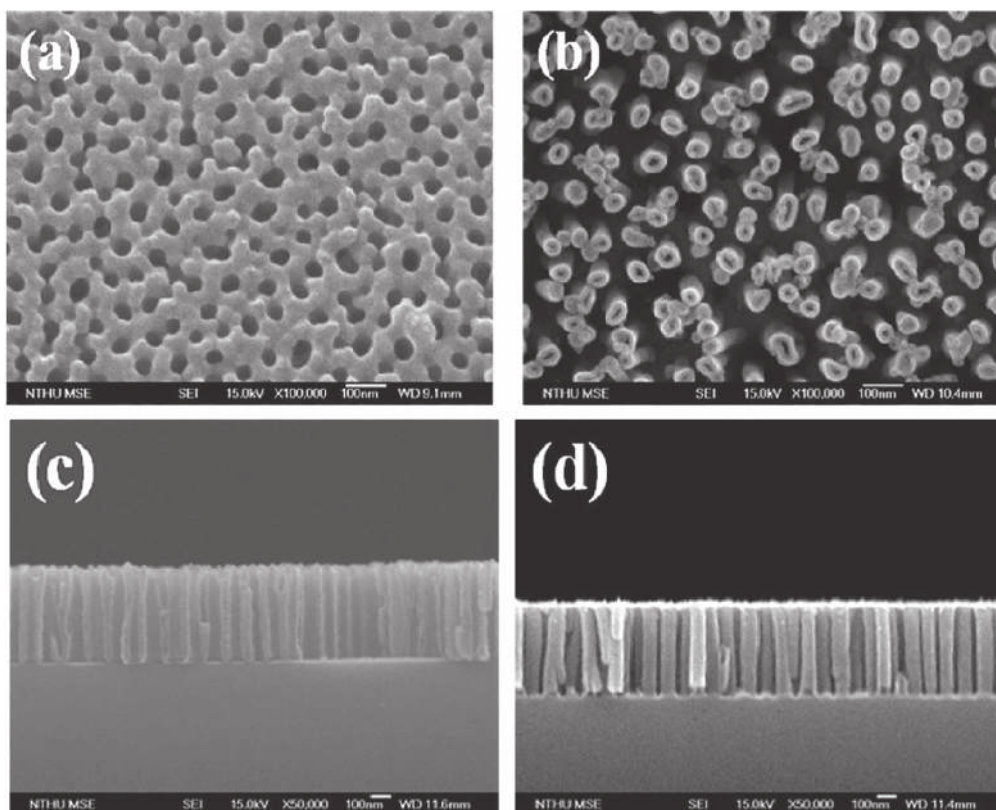


Figure 8. SEM images of (a), (c) TiO_2 in the AAO template, and (b), (d) pure TiO_2 nanotubes after removal of the AAO template. Reprinted with permission from [328]. Copyright (2011) American Chemical Society.

was demonstrated that the use of a tin(IV) alkoxide and carboxylic acid precursors are highly suitable for the fabrication of SnO_2 nanotubular structures at temperatures as low as 75°C using templates such as carbon nanotubes (CNTs) and boron nitride nanotubes [332].

Flexible polymer–GaN organic–inorganic core–shell nanofibers (figure 10) were synthesized by performing remote PEALD growth of GaN on electrospun nylon 6,6 nanofibers at 200°C [285]. The fabrication process resulted in ~ 28 nm thick conformal polycrystalline wurtzite GaN shell layer on polymeric-core nanofibers with an average fiber diameter of ~ 70 nm. The low process temperatures enabled the preservation of the flexibility of the resulting core–shell polymer/GaN nanofiber templates [285]. Polycrystalline hexagonal AlN hollow nanofibers (figure 11) were fabricated via PEALD using similar sacrificial electrospun polymeric nanofiber templates [333]. Deposition of AlN was carried out again at 200°C on electrospun nylon 6,6 nanofibers followed by a high-temperature *in situ* calcination at 500°C for 2 h to remove the sacrificial polymeric nanofiber template [333]. Recently, vertically aligned GaN, AlN, and InN hollow nano-cylindrical arrays (HNCs) on Si substrates were fabricated using anodized aluminum oxide (AAO) membrane based template-assisted plasma-assisted atomic layer deposition (PEALD). Fabrication scheme consisted of the following steps: (i) Electrochemical anodization of aluminum foil to obtain free-standing AAO membrane, followed by transfer and sticking of AAO membrane to Si substrate, (ii) Si patterning with Ar and CHF_3 based reactive ion etching (RIE) using AAO membrane as hard mask

material to achieve nanoporous network on Si substrate, (iii) Conformal growth of GaN, AlN, and InN on nanoporous Si via low-temperature PEALD, (iv) Ar based RIE of PEALD coated III-nitride material from top surface of Si and SF_6 based isotropic RIE of surrounding Si to attain highly ordered vertical GaN, AlN, and InN hollow nano-cylinder arrays. Materials characterization revealed that ordered vertical arrays of III-nitride hollow nanocylinders were successfully integrated inside Si(100); a representative image of GaN HNCs fabricated using this strategy is given in figure 12.

High aspect ratio (≥ 300) ZnS nanotubes were fabricated by performing growth into highly ordered pores of AAO templates at deposition temperatures as low as 75°C [334]. The tubes exhibited smooth wall surface and their dimensions can be precisely tailored by varying the electrochemical and ALD processing parameters [334]. Cu_2S was grown on networks of single walled carbon nanotubes (SWCNTs) to obtain SWCNT-n- Cu_2S composite nanotube structure [335]. The resulting structures demonstrated an intimate contact between SWCNTs and Cu_2S , yet preserving the porosity for efficient transport of charges [335]. Similar strategy has been utilized to obtain Ga_xS -SWCNTs composite nanotubular structure [336]. Figure 13 shows TEM images of ALD-coated Ga_xS on SWCNTs yielding highly conformal Ga_xS -SWCNT core–shell 1D templates with the outer shell thickness depending on the number of Ga_xS growth cycles. The EELS maps in figures 13(g)–(i) correspond to the boxed region in Figure 13(f), and reveal that the coating is uniform and comprised of Ga and S.

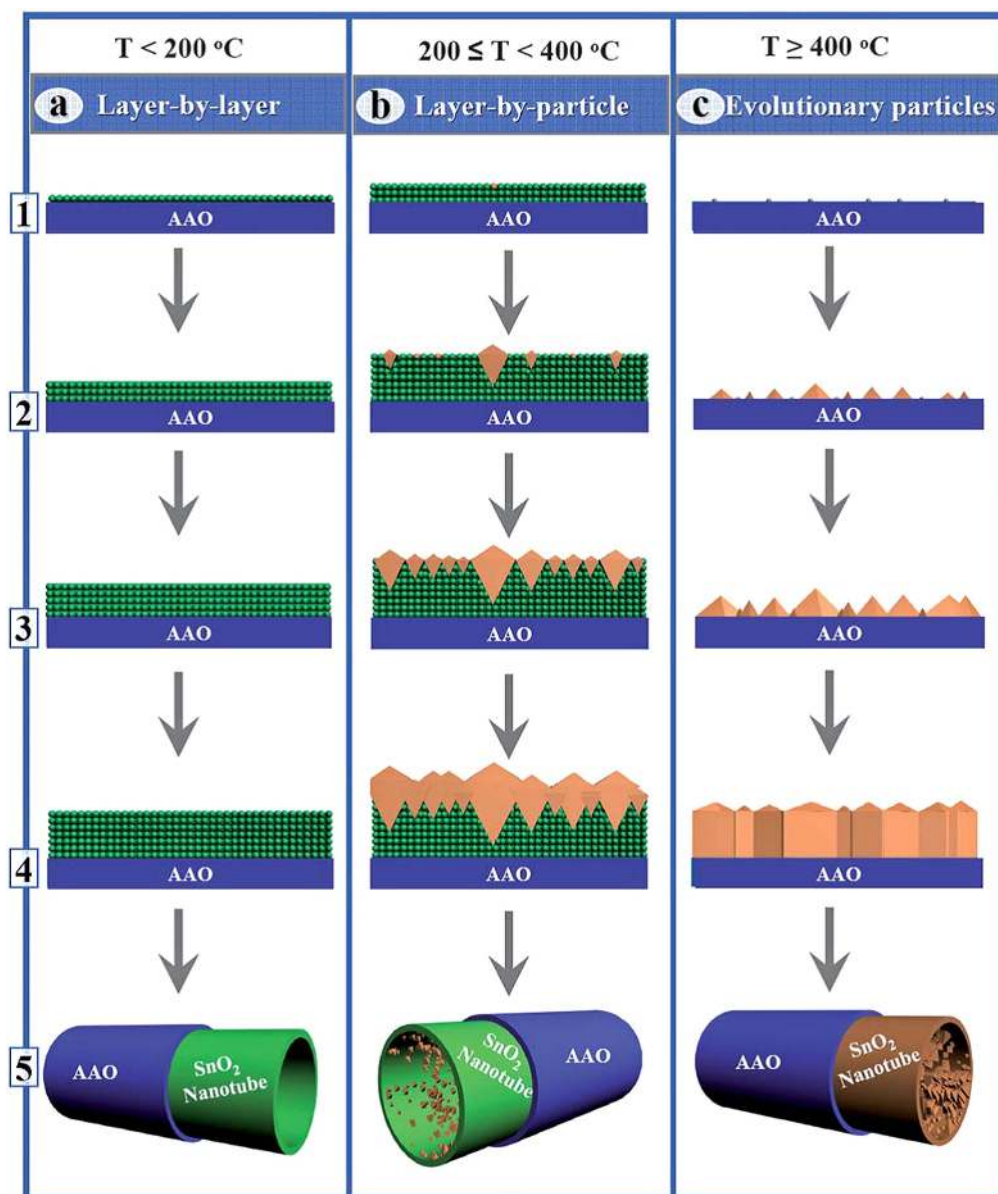


Figure 9. Three growth models for ALD-SnO₂ nanotube arrays. At a temperature lower than 200 °C, the ALD-SnO₂ was built up in AAO pores in a mode of (a) layer-by-layer growth, as schematically shown in (a1-4) with uniform films of increasing thickness; in the range 200 °C–400 °C, the ALD-SnO₂ experienced a mode of (b) layer-by-particle growth: (b1) amorphous layers prevailed at the initial stage but random nucleation happened with increased films, (b2) crystals grew laterally and radially while new nuclei appeared with increased films, (b3) crystals saturated on the surface and amorphous layers stopped growing, (b4) crystals stopped their lateral growth and competed with each other for their radial growth; at a temperature no less than 400 °C, the ALD-SnO₂ transferred to a mode of (c) evolutionary particle: (c1) nuclei formed immediately on the substrate surface at the starting of the ALD process, (c2) crystals grew quickly with increased ALD-cycles, (c3) crystals saturated on the substrate surface, (c4) crystals grew predominantly in their radial direction. In (a5), (b5), and (c5), the produced nanotubes were schematically illustrated. Reprinted with permission from [331]. Copyright (2011) The Royal Society of Chemistry.

3.3. 2D structures

Although ALD is known to be self-limiting, initial nucleation generally occurs through the formation of multi-layer islands which makes it difficult to achieve the layer controllability needed to deposit 2D semiconductors such as transition metal dichalcogenides (TMDs) [345–348]. It is therefore essential to maximize the self-limiting behavior of ALD process in order to achieve the layer controllability needed for 2D structures which requires careful selection of precursors and optimization of process conditions. Initial reports of WS₂ ALD growth

employed WF₆ and H₂S reactants where zinc catalyzed the adsorption and reaction of WF₆ [349, 350]. In later reports, single crystal WS₂ nanosheets on SiO₂ substrates were synthesized by the sulfurization of a WO₃ film prepared by ALD. Utilization of ALD for preparation of WO₃ meant that WS₂ layers retained inherent characteristics of ALD processes such as thickness controllability, reproducibility, high conformality, and uniformity [351, 352]. MoS₂ thin films were deposited by ALD using Mo(CO)₆ and H₂S plasma as precursors which resulted in hexagonal nanocrystalline films [346]. In another report, MoCl₅ and H₂S were employed to grow MoS₂ where

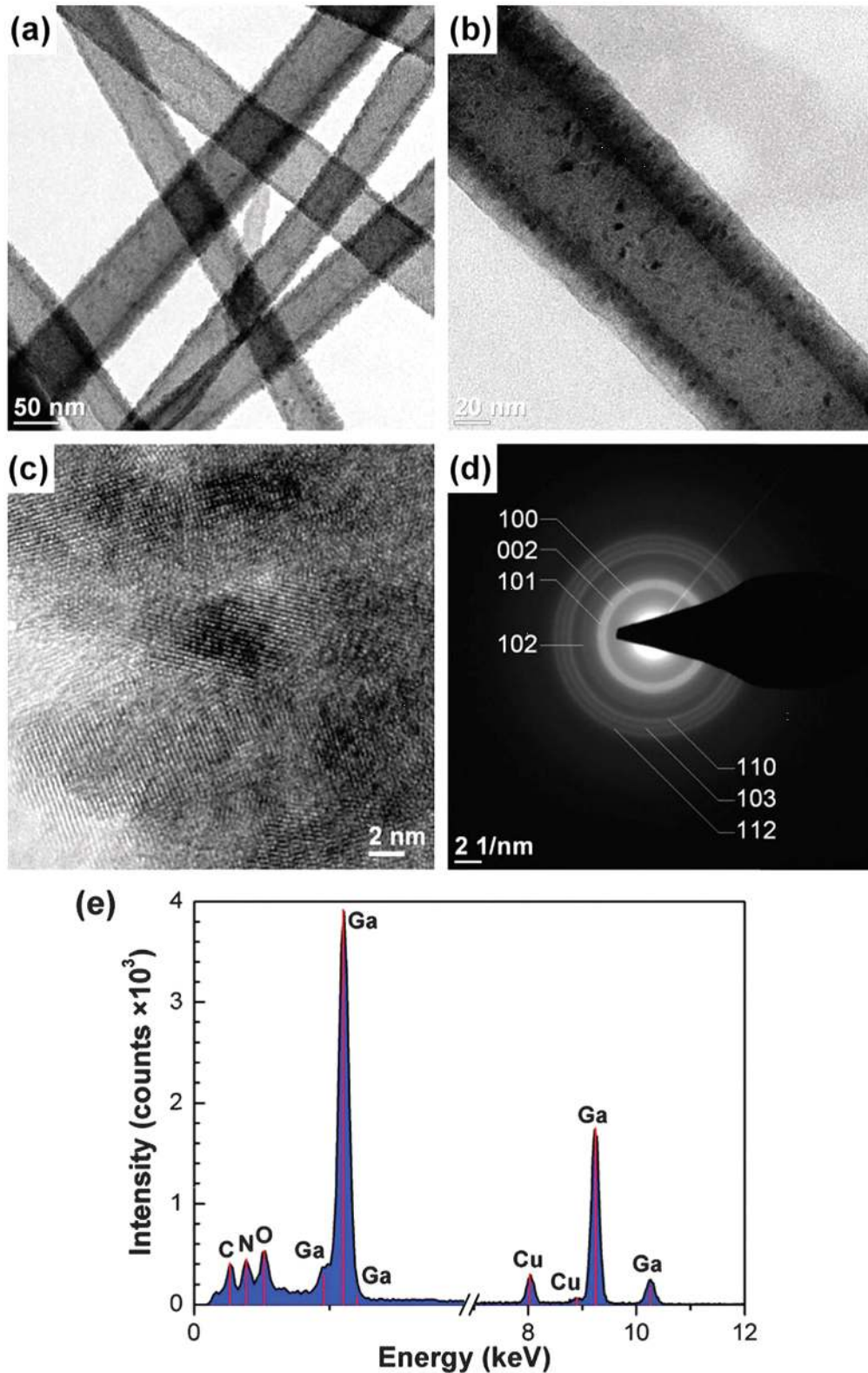


Figure 10. Representative (a) and (b) TEM, (c) high-resolution TEM images, (d) selected area electron diffraction (SAED) pattern, and (e) EDX spectrum of nylon-GaN core-shell nanofiber(s). Reproduced with permission from from [285]. Copy right (2015) The Royal Society of Chemistry.

number of layers formed were controlled by varying the growth temperature [348]. It is believed that lack of dangling bonds on MoS₂ surface caused the growth to self-terminate after few layers of MoS₂ have been grown at a certain temperature [348].

Recently, low temperature (400 °C) growth of graphene has also been demonstrated using PEALD [353]. Hexagonal carbon rings and carbon atoms were observed (figure 14) using aberration-corrected transmission electron microscopy which indicated highly crystalline structure of graphene.

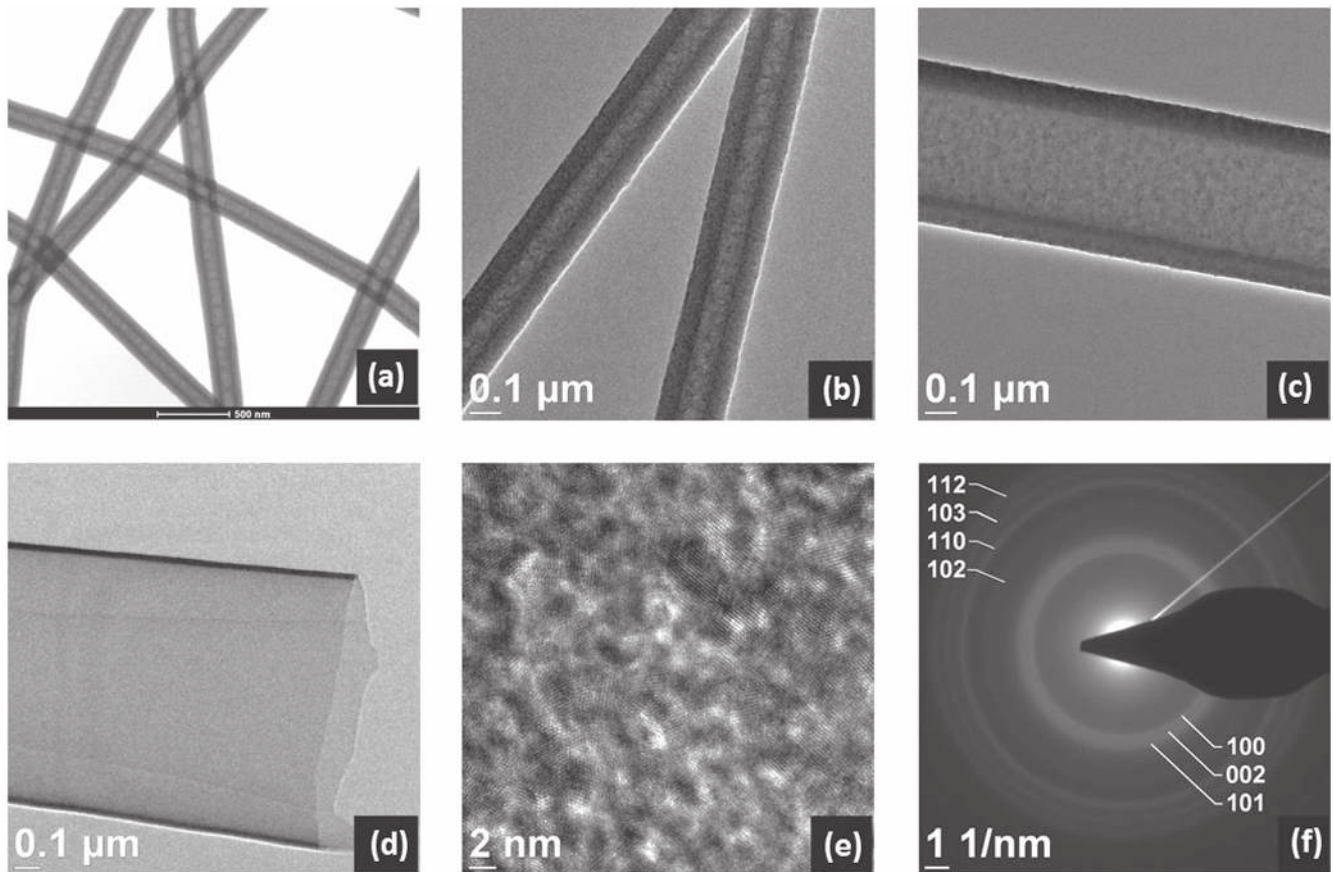


Figure 11. AlN hollow nanofibers synthesized by the deposition of 800 cycles AlN, followed by *in situ* calcination. (a) Bright field TEM image of hollow nanofibers synthesized using nylon 6,6 template having an average fiber diameter of ~ 70 nm. (b)–(d) TEM images of hollow nanofibers synthesized using templates with average fiber diameters of ~ 70 , ~ 330 , and ~ 740 nm, respectively. (e) High-resolution TEM image, and (f) SAED pattern of AlN hollow nanofibers synthesized using a template having ~ 330 nm average fiber diameter. Reprinted with permission from [333]. Copyright (2013) John Wiley and Sons.

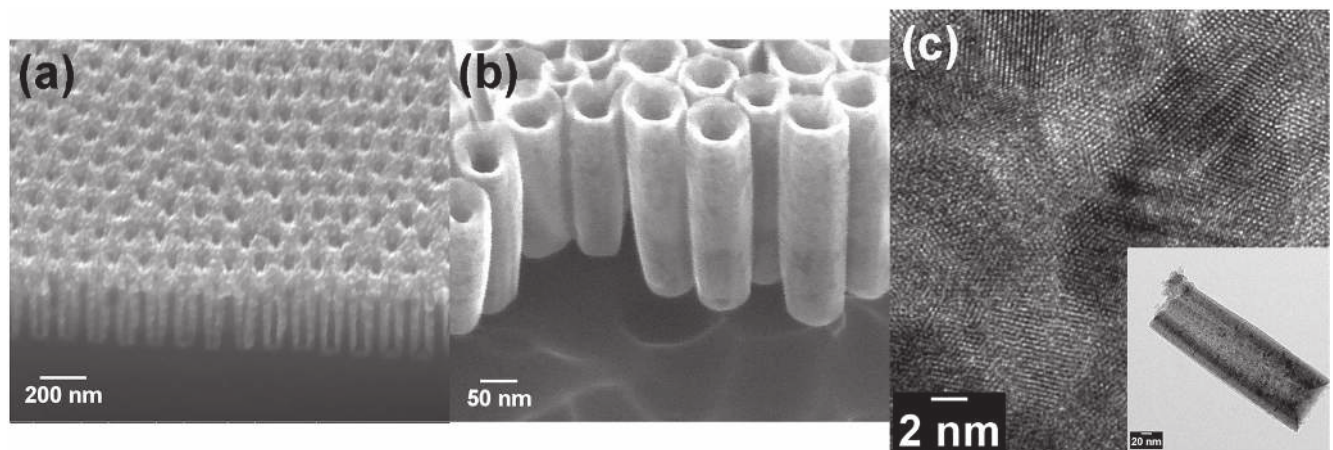


Figure 12. (a) SEM image of conformally coated GaN on porous Si structure using PEALD. (b) SEM image of GaN hollow nano-cylinders obtained after RIE etching of top GaN layer and surrounding Si substrate material. (c) High-resolution TEM image of GaN nano-cylinder, (Inset) TEM image of a single GaN hollow nano-cylinder.

Benzene is chosen as the carbon source and authors demonstrated that benzene molecules can be dehydrogenated and connected to each other to form the graphene structure [353]. An overview of semiconductor 2D nanostructures synthesized using ALD is presented in table 4.

3.4. Core–shell structures

Core–shell structures are formed when a 0D or 1D nanostructure, such as a nanoparticle or nanorod is coated with a thin layer of another material. Such core–shell nanostructures

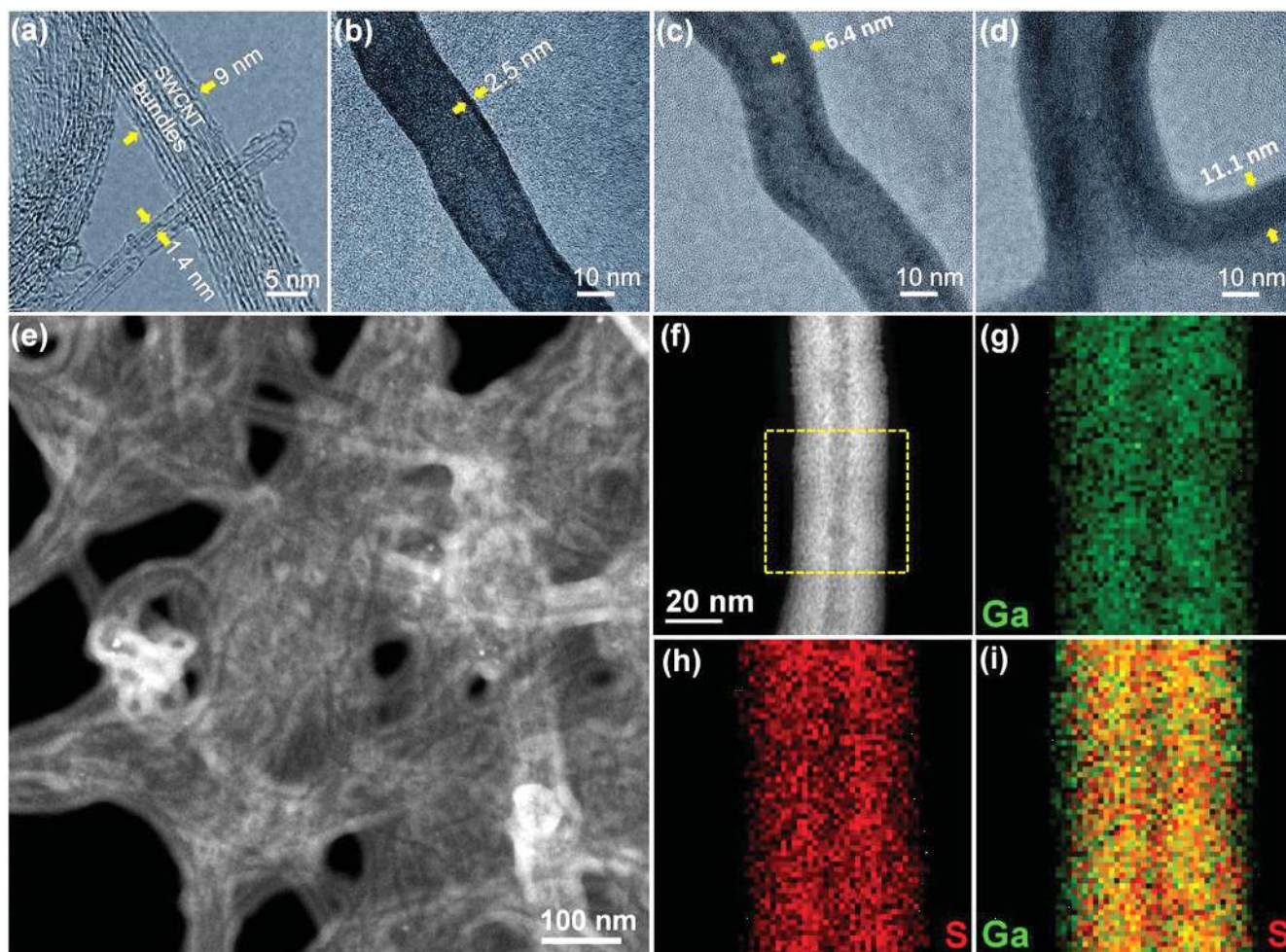


Figure 13. TEM (a)–(d) and STEM (e)–(i) images of: (a) SWCNTs and their bundles; SWCNTs coated by (b) 50-cycle, (c) 100-cycle, and (d) 150-cycle ALD GaS_x; (e), (f) ADF-STEM images; and STEM-EELS mapping of (g) gallium, (h) sulfur, and (i) both gallium and sulfur. Reprinted with permission from [336]. Copyright (2014) John Wiley and Sons.

might show enhanced material properties when compared to the core-only nanostructure, which provide significant potential for numerous applications

ALD of ZnO has been implemented to coat particles of Si [358], SiO₂ [359–362], TiO₂ [363], and Al₂O₃ [364] for applications in photocatalysis. A viscous flow or fluidized bed reactor configuration is suitable for coating nanoparticles using ALD. More commonly, ALD of ZnO has been performed on 1D structures such as nanorods, nanotubes, nanofibers, and nanowires made of Si or SiO₂ [365–372], SnO₂ [373–376], TiO₂ (figure 15) [377], and Al₂O₃ [378, 379]. Alternatively, ALD of TiO₂ has been performed on ZnO nanofibers (figure 15) to produce TiO₂–ZnO core–shell nanofibers [377]. Generally, the fabrication of these structures are more application centered and concentrated on photoluminescence and chemo-resistive properties of the fabricated nanostructures [358, 361, 368, 369, 378, 379]. Typically, 1D core part of these structures is prepared using alternative techniques such as hydrothermal or vapor liquid solid (VLS) growth due to the relative difficulty of fabricating 1D structures directly with ALD [362, 363, 365, 367, 370, 373, 376, 380–383]. Taking advantage of high conformality and uniformity of ALD

coatings, shell portions of these structures are commonly grown using ALD [368, 375, 382, 384–386]. AlN/BN core shell hollow nanofibers (HNFs) (figure 16) have been synthesized by successive ALD of AlN and low temperature sequential CVD of BN on electrospun nylon polymer nanofibrous templates [284]. Material characterization showed that crystalline BN/AlN nanofibers were fabricated with relatively low impurity content. The nanoneedle-like 3D morphology of BN ALD-coating is particularly noteworthy in this study [284]. Overview of core–shell nanostructures prepared in part or totally by ALD is provided in table 5.

4. Applications of ALD-grown nanostructured semiconductors

Material/crystalline properties of low-temperature ALD-grown thin-film semiconductors are considerably weaker than high-temperature epitaxial counterpart films grown via MOCVD, CVD, MBE, etc. Therefore, it's quite hard for ALD-grown semiconductor films to compete with epitaxial films in terms of device performance. Despite this drawback,

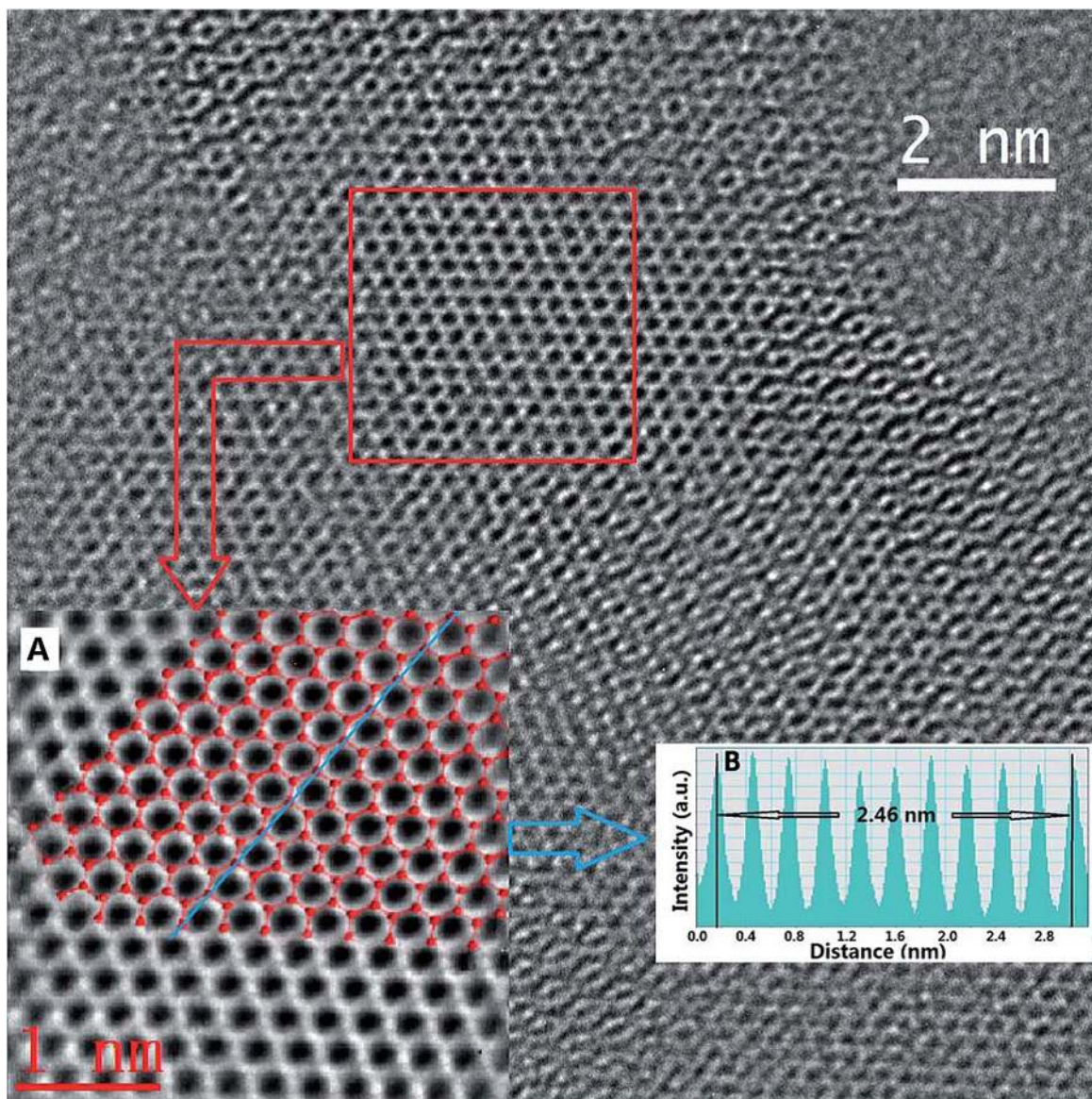


Figure 14. High-resolution images of the graphene deposited by 10 ALD cycles. The atomic-resolution image was obtained using an FEI Titan G2 60–300 at an acceleration voltage of 300 kV (the scale bar is 2 nm). Inset (A): filtered image of the area within the red rectangular frame. Inset (B): pixel intensity profile of the graphene along the blue line in inset (A), which was used to calculate the distance between the second-nearest neighboring carbon atoms in the hexagonal carbon rings using the Digital Micrograph software. Reprinted with permission from [353]. Copy right (2014) The Royal Society of Chemistry.

ALD-grown thin-film semiconductors might find use particularly in applications where low-temperature deposition is critical. Flexible electronics is such a field which can be utilized in wearable and implantable devices including chemical and optical sensors, active matrix displays with integrated thin-film transistors (TFT), etc. ZnO and GaN have been the main materials of choice for the development of ALD-grown active device layers for TFT, photodetector, and sensors [397–401].

On the other hand, as overviewed in section 3, ALD is an attractive method for the fabrication of complex and high-surface area 0D, 1D, 2D, and core–shell nanostructures which can be hardly accomplished via conventional high-temperature epitaxial growth techniques. For this reason, in this part, we would like to overview the device applications where only nanostructured semiconductors grown/fabricated by ALD are

utilized. Among various device applications demonstrated in literature based on nanostructured semiconductors, energy storage, catalysis, photocatalysis, solar cells, opto-electronics, and chemical sensing constitute the vast majority of reports.

4.1. Energy storage

Many of the deficiencies of Li-ion batteries such as limited power density, storage capacity, and slow charging/discharging can be alleviated to certain extent by nanostructuring the electrode which can implicate shorter Li^+ and electron diffusion paths. By having a large contact area with the electrolyte (low thickness) and/or the current collector may allow the use of materials with low conductivity which will result in high power capability. Besides, certain nano-scale

Table 4. Overview of the growth conditions employed to synthesize 2D nanostructures using ALD. The materials deposited, the morphology, precursors used, the temperature during ALD, and preparation method are mentioned.

Material	1st precursor	2nd precursor	Growth temperature	Morphology	Preparation method	References
MoS ₂	Molybdenum hexacarbonyl (Mo(CO) ₆)	H ₂ S	120 °C–200 °C	Nanosheets	ALD of MoS ₂ on Si	[354]
	Mo(CO) ₆	H ₂ S	175 °C–225 °C	Nanosheets	ALD of MoS ₂ on SiO ₂	[346]
	MoCl ₅	H ₂ S	475 °C	Nanosheets	ALD of MoS ₂ on SiO ₂ /Si and quartz	[355]
	MoCl ₅	H ₂ S	350 °C–450 °C	Nanosheets	ALD of MoS ₂ on SiO ₂ /Si	[347]
	MoCl ₅	H ₂ S	500 °C–900 °C	Nanosheets	ALD of MoS ₂ on SiO ₂ /Si	[348]
	Mo(CO) ₆	Dimethyl disulfide	100 °C	Nanosheets	ALD of MoS ₂ on Carbon fiber papers and Si	[356]
	Mo(NMe ₂) ₄	(HS(CH ₂) ₂ SH)	50 °C followed by annealing	Nanosheets	ALD of MoS ₂ on SiO ₂ /Si and silica nanoparticles	[345]
WS ₂	WF ₆	H ₂ S	300 °C–350 °C	Nanosheets	ALD of WS ₂ on Si, SiO ₂ , stainless steel, and polycrystalline Si	[350]
	WF ₆	H ₂ S	250 °C–300 °C	Nanosheets	ALD of WS ₂ on Rolling element bearing	[349]
	WH ₂ (iPrCp) ₂	O ₂	300 °C followed by sulfurization of WO ₃	Nanosheets	ALD of WS ₂ on SiO ₂ /Si substrates	[351]
	WH ₂ (iPrCp) ₂	O ₂	300 °C followed by sulfurization of WO ₃	Nanosheets	ALD of WS ₂ on SiO ₂ /Si substrates	[352]
	Bis(tert-butylimido)-bis(dimethylamido) tungsten (((tBuN) ₂ (Me ₂ N) ₂ W))	O ₂	150 °C followed by CS ₂ vulcanization	Nanosheets	ALD of WS ₂ on Quartz	[357]
Graphene	Benzene	H ₂	400 °C	Layer of graphene	ALD of graphene on Cu foil	[353]

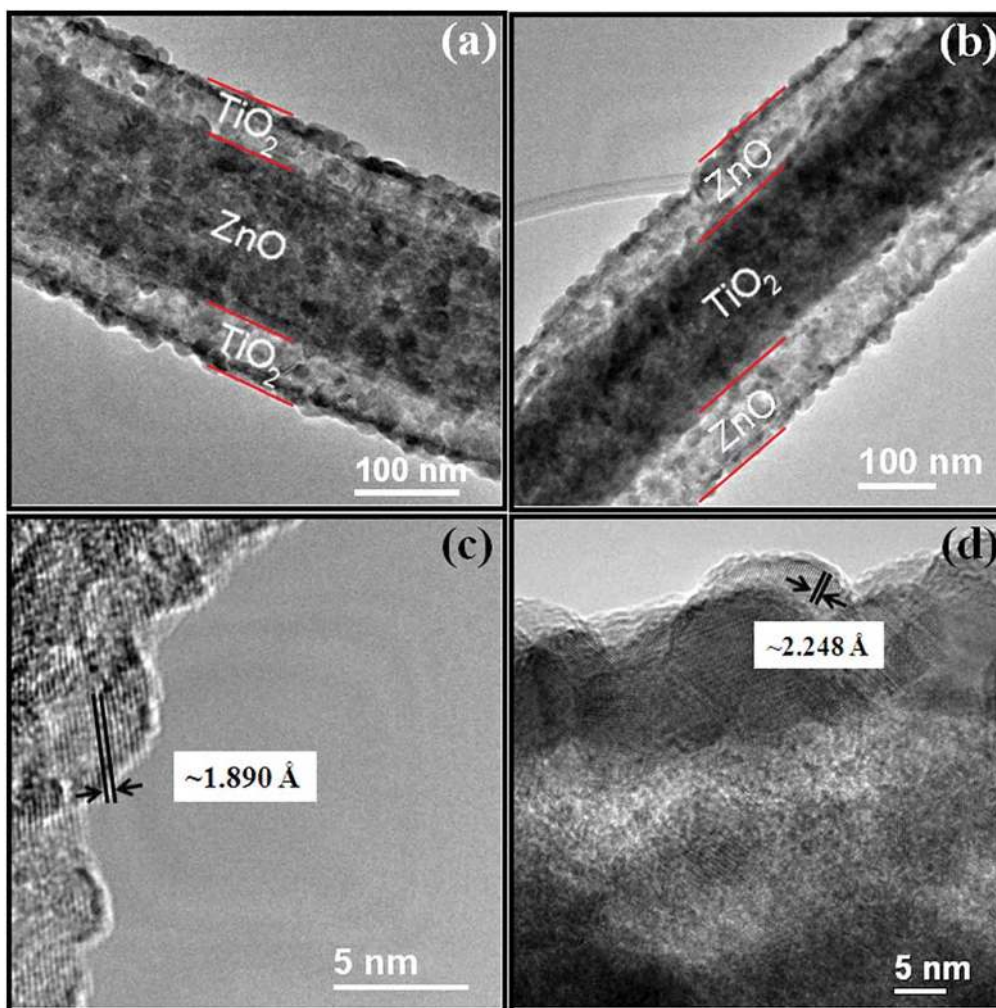


Figure 15. Representative TEM images of the core-shell heterojunction nanofibers (a) ZnO-TiO₂, (b) TiO₂-ZnO; (c) and (d) HRTEM images of the 'shell' regions of (a) and (b), respectively. Reprinted with permission from [377]. Copy right (2014) The Royal Society of Chemistry.

quantum-size effects can increase the electrode storage capacity as well. Moreover, nanostructured electrodes can withstand stresses due to volume expansion and contraction which enables enhanced reversible cycling of the electrode material [404–408].

As described in previous sections, ALD with its conformal growth mechanism, can be effectively used to produce nanostructures via template-assisted strategy. Such nanostructures can then be used as electrode materials to enhance the energy storage capacity of Li-ion and other alternative (Li-O₂, Li-polymer, Li-air, etc) batteries. Conformal and ultra-thin ALD-grown coatings can also be used as electrode protection/passivation layers to reduce electrolyte decomposition on the surface of the electrode and dissolution of electrode material into the electrolyte [415–418]. Some examples from the literature are presented in this section.

Battery application of ALD nanocoatings was first demonstrated in energy storage devices using particle based electrodes where TiN was coated on Li₄Ti₅O₁₂ aimed for improving inter-particle electrical conductivity [402]. The

film in this study was quite thin (~6 nm) and therefore, did not cause any performance problems with Li⁺ ion diffusion process [402]. ALD has also assisted in making composite nanoparticle electrodes for Li-ion batteries. In one such report, SnO₂/graphene nanosheets composite electrode was fabricated by performing SnO₂ ALD on graphene nanosheet powder [403]. ALD-grown TiO₂ has also been employed in Li-ion batteries [404, 405]. TiO₂ has been used as an anode material and features the advantage of relatively high redox potential [392, 406–414]. TiO₂ was coated on peptide nanofibers followed by calcination at high temperature which removed peptide material as well as crystallized the TiO₂ to anatase phase [405]. The resulting TiO₂ hollow nanonetwork demonstrated higher storage capacity and charge rate performance (figure 17) when compared with TiO₂ nanopowder electrodes, which was attributed to prominent nanosize effects in TiO₂ nanonetwork [405]. In another study, TiO₂ coated on Au nanoparticles was utilized as Li ion battery anode material (figure 18) [413]. Power performance of the electrode improved with the decrease in TiO₂ thickness by reducing the

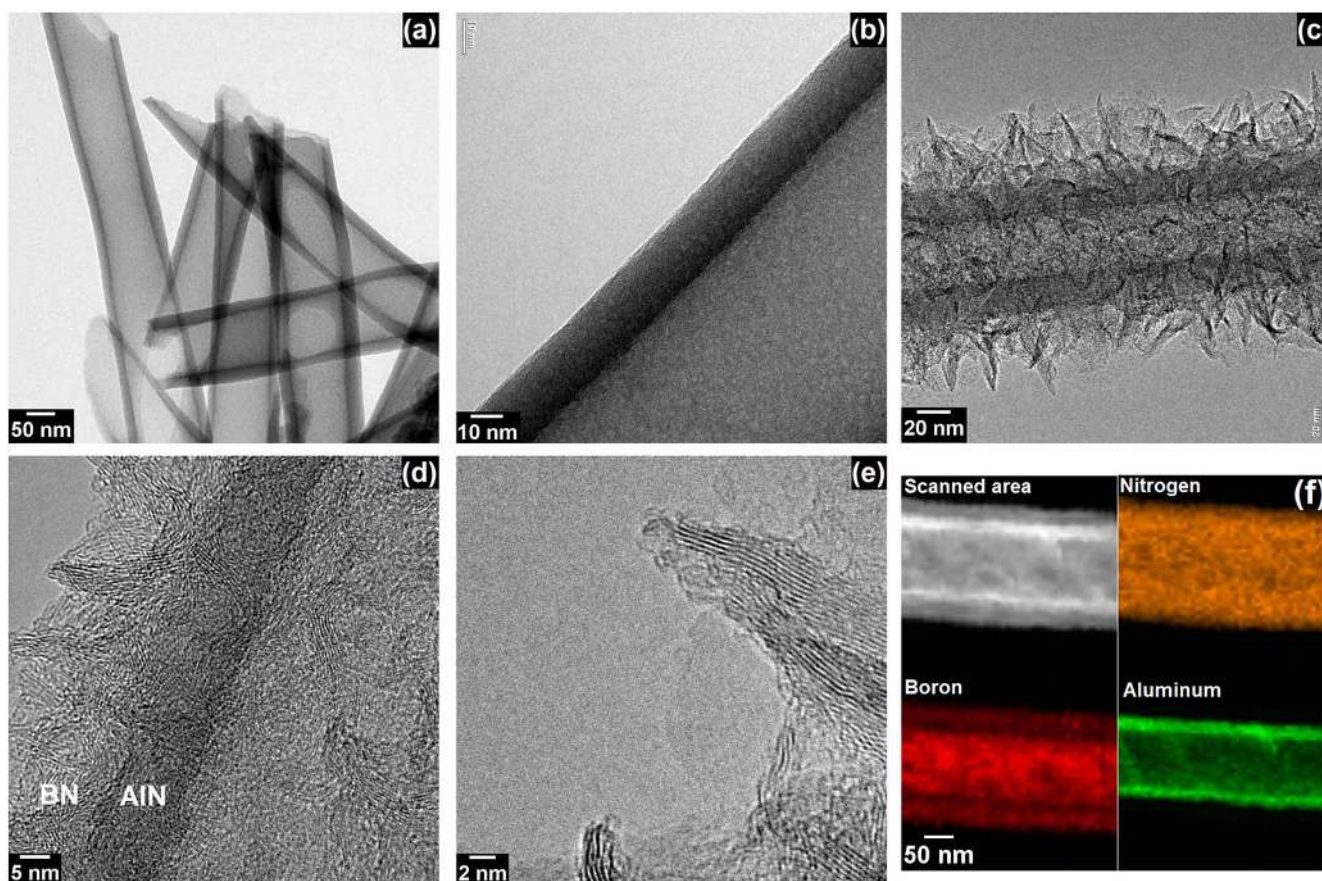


Figure 16. (a) and (b) Representative bright field TEM images of AlN HNFs. (c) and (d) Representative bright field TEM images of AlN/BN bishell HNF having an average inner fiber diameter of ~ 100 nm with an average wall thickness of ~ 20 nm and ~ 35 nm of AlN and BN, respectively. (e) Representative HR-TEM image of AlN/BN bishell HNF. (f) EDX elemental map of aluminum, boron, and nitrogen from an individual AlN/BN HNF. Reprinted with permission from [284]. Copyright (2014) AIP publishing.

Li^+ diffusion pathway, enhancing the Li^+ solid solubility, and minimizing the voltage drop across the electrode/electrolyte interface (figure 19) [413].

For Li-S batteries, ALD has been used to develop a 3D structured thin TiO_2 shell which acts as a cathode material and provides high electronic and ionic conductivity [415]. An inverse opal structure has been formed which aids against polysulfide dissolution due to O_2 vacancies which facilitate S-Ti-O interactions. These oxygen vacancies were created from reduction of TiO_2 with hydrogen [415]. Polysulfide dissolution or shuttle effect is a common problem with Li-S batteries where polysulfides react with Li metal at electrode to form lower order polysulfides. This phenomena leads to the formation of a high-impedance solid-electrolyte interface (SEI) layer that consumes the electrolyte and active material and causes anode decay eventually leading to battery cell failure.

TiN and TaN grown using remote plasma ALD have been applied in 3D all-solid-state micro-batteries as Li diffusion barrier layers [416, 417]. The Li diffusion barrier properties of ALD TiN was significantly better than sputtered TiN barriers. ALD grown TiN in Si pores was shown to behave as excellent barrier layer and current collector [418]. In that study, low pressure chemical vapor deposition (LPCVD) grown Si was used as an anode material and was

coated with ALD TiN to serve as Li barrier and current collector [418]. ALD grown V_2O_5 was demonstrated to be a good alternative cathode material [419]. Especially, the V_2O_5 annealed at 400°C offered excellent electrochemical stability and cyclability for Li^+ ions [419]. ALD grown Co_3O_4 was shown to be a decent anode material as well where good cycling performance and high storage capacity were obtained in electrochemical tests [420].

4.2. Photocatalysis

Hydrogen produced from water splitting is a potentially clean fuel with its high energy density and environmental friendliness. Production of hydrogen from water splitting needs an external energy source and solar energy is an attractive choice, forming a complete renewable energy cycle. Photoelectrochemical splitting of water on semiconductor electrode materials (n-type TiO_2) is an efficient and inexpensive way of producing clean hydrogen fuel [421–430]. The major challenges in this research field range from reactor design to the discovery of novel high-surface area nanostructured materials that can achieve decent activity levels [421–430]. ALD has played a significant role in fabricating a large variety of nanostructured semiconductor materials for applications in photocatalysis.

Table 5. Overview of the growth conditions employed to synthesize core-shell nanostructures using ALD. The materials deposited, the morphology, precursors used, the temperature during ALD, and preparation method are mentioned.

Core-shell Materials	Precursors for 1st material	Precursors for 2nd material	Growth temperature	Morphology	Preparation method	References
ZnSe–CdSe	Elemental Zn and Se	Elemental Cd and Se	400 °C–420 °C	Nanowires	Growth initiation using gold nanoparticles and the vapor–liquid–solid mechanism	[387]
ZnO–ZrO ₂	Zn(CH ₂ CH ₃) ₂ and H ₂ O	—	177 °C	Nanoparticles	ZnO growth on ZrO ₂ nanoparticles	[388]
ZnO–BaTiO ₃	Zn(CH ₂ CH ₃) ₂ and H ₂ O	—	177 °C	Nanoparticles	ZnO growth on BaTiO ₃ nanoparticles	[388]
SiO _x –ZnO	(C ₂ H ₅) ₂ Zn and H ₂ O	SiO _x growth through CVD	170 °C	Nanowires	ZnO shell layer growth on SiO _x nanowires	[365]
ZnO–SiO ₂	(C ₂ H ₅) ₂ Zn and H ₂ O	Commercially purchased SiO ₂ particles	177 °C	Nanoparticles	ZnO growth on SiO ₂ nanoparticles	[362]
ZnO–TiO ₂	(C ₂ H ₅) ₂ Zn and H ₂ O	Commercially purchased TiO ₂ particles	177 °C	Nanoparticles	ZnO growth on TiO ₂ nanoparticles	[362]
ZnO–TiO ₂	(C ₂ H ₅) ₂ Zn and H ₂ O ₂	Commercially purchased TiO ₂ nanopowder	50 °C, 100 °C, and 177 °C	Nanoparticles	ZnO growth on TiO ₂ particles	[363]
ZnO–polystyrene (PS)	Zn(CH ₂ CH ₃) ₂ and H ₂ O	—	80 °C and 90 °C	Nanocrystals	ZnO growth on monodisperse PS colloidal nanospheres	[380]
ZnO–SnO ₂	(C ₂ H ₅) ₂ Zn and H ₂ O	SnO ₂ nanofibers prepared by electrospinning	150 °C	Nanofibers	ZnO growth on SnO ₂ nanofibers	[373]
TiO ₂ –ZnO	(C ₂ H ₅) ₂ Zn and H ₂ O	TiO ₂ nanofibers prepared by electrospinning	150 °C	Nanofibers	ZnO growth on TiO ₂ nanofibers	[381]
Bi ₂ O ₃ –ZnO	(C ₂ H ₅) ₂ Zn and H ₂ O	Thermal evaporation of Bi ₂ O ₃ nanowires	—	Nanowires	ZnO growth on Bi ₂ O ₃ nanowires	[383]
ZnO–Si	(C ₂ H ₅) ₂ Zn and H ₂ O	Si nanowires growth using CVD	25 °C	Nanowires	ZnO growth on Si nanowires	[367]
CoFe ₂ O ₄ –TiO ₂	Titanium	Directed assembly of colloidal CoFe ₂ O ₄ in a Langmuir–Blodgett monolayer	200 °C	Nanoparticles/film composites	TiO ₂ growth on CoFe ₂ O ₄ nanoparticles/film composites	[384]
CoFe ₂ O ₄ –ZnO	Isopropoxide and acetic acid (C ₂ H ₅) ₂ Zn and H ₂ O	Directed assembly of colloidal CoFe ₂ O ₄ in a Langmuir–Blodgett monolayer	70 °C	Nanoparticles/film composites	ZnO growth on CoFe ₂ O ₄ nanoparticles/film composites	[384]
ZnTe–ZnO	(C ₂ H ₅) ₂ Zn and H ₂ O	ZnTe nanowires growth using gold catalyzed vapor–liquid–solid mechanism	60 °C, 100 °C, 150 °C and 200 °C	Nanowires	ZnO growth on ZnTe nanowires	[382]
ZnO–Si	(C ₂ H ₅) ₂ Zn and H ₂ O	Si nanopillars fabricated by self-masking dry etching in hydrogen-containing plasma	200 °C	Nanopillars	ZnO growth on Si nanopillars	[370]

Table 5. (Continued.)

Core-shell Materials	Precursors for 1st material	Precursors for 2nd material	Growth temperature	Morphology	Preparation method	References
ZnMnTe/ZnO	(C ₂ H ₅) ₂ Zn and H ₂ O	ZnMnTe nanowires fabricated through MBE technique using gold-catalyzed vapor-liquid-solid mechanism.	200 °C, 250 °C, and 300 °C	Nanowires	ZnO growth on ZnMnTe nanowires	[389]
ZnO-SnO ₂	SnCl ₄ and H ₂ O	ZnO nanorods prepared by thermal evaporation	350 °C	Nanorods	SnO ₂ growth on ZnO nanorods	[376]
ZnO-Al ₂ O ₃	Al ₂ (CH ₃) ₆ and H ₂ O	ZnO nanorods synthesized by aqueous chemical route	180 °C	Nanorods	Al ₂ O ₃ growth on ZnO nanorods	[385]
ZnO-ZnO	(C ₂ H ₅) ₂ Zn and H ₂ O	ZnO nanorods synthesized by aqueous chemical route	180 °C	Nanorods	ALD ZnO growth on ZnO nanorods	[385]
ZnO-Si	(C ₂ H ₅) ₂ Zn and H ₂ O	Si nanopillars obtained by dry etching using silver nanodots as mask	200 °C	Nanopillars	ZnO growth on Si nanopillars	[371]
ZnO-SiO ₂	(C ₂ H ₅) ₂ Zn and H ₂ O	Silica nanosprings grown using vapor liquid solid mechanism	175 °C	Nanosprings	ZnO growth on silica nanosprings	[368]
ZnO-SnO ₂	(C ₂ H ₅) ₂ Zn and H ₂ O	SnO ₂ nanowires growth via vapor liquid solid mechanism	150 °C	Nanorods	ZnO growth on SnO ₂ nanorods	[375]
ZnO-SnO ₂	(C ₂ H ₅) ₂ Zn and H ₂ O	SnO ₂ nanowires growth via vapor liquid solid mechanism		Nanowires	ZnO layer growth on SnO ₂	[386]
⊗ ZnO-Si	(C ₂ H ₅) ₂ Zn and H ₂ O	Si nanotips fabrication using high-density electron cyclotron resonance (ECR) plasma etching	90 °C	Nanotips	Nanowires ZnO layer growth on Si	[372]
SnO ₂ -TiO ₂	TiCl ₄ and H ₂ O	Rod-like SnO ₂ Particles prepared through a basic synthetic route.	300 °C	Nanoparticles	Nanotips TiO ₂ grown on SnO ₂ particles	[390]
ZnO-Si	(C ₂ H ₅) ₂ Zn and H ₂ O	Si nanowires fabrication through electroless metal deposition	50 °C	Nanowires	ZnO growth on Si nanowires	[369]
ZnO-ZnSnO ₄	(C ₂ H ₅) ₂ Zn and H ₂ O	Synthesis of Zn ₂ SnO ₄ nanorods through thermal evaporation of a mixture of ZnO, SnO ₂ , and graphite powders	150 °C	Nanorods	ZnO growth on ZnSnO ₄ nanorods	[374]
ZnO-Al doped ZnO	(C ₂ H ₅) ₂ Zn, Al ₂ (CH ₃) ₆ , and H ₂ O	Fabrication of ZnO nanowires using thermal evaporation	150 °C	Nanowires	Al-doped ZnO growth on ZnO nanowires	[378]
ZnO-Cu	(C ₂ H ₅) ₂ Zn and H ₂ O	Cu e-beam evaporation	120 °C	Nanoclusters	ZnO growth on Cu nanoclusters	[391]
ZnO-TiO ₂	(C ₂ H ₅) ₂ Zn and H ₂ O	Tetrakis(dimethylamido)titanium and H ₂ O	200 °C	Nanofibers	ALD of TiO ₂ and ZnO on electrospun polymer fibers	[377]

Table 5. (Continued.)

Core-shell Materials	Precursors for 1st material	Precursors for 2nd material	Growth temperature	Morphology	Preparation method	References
ZnS-CuS	Bis (2,2,6,6-tetramethyl-3,5-heptanedionato)zinc and H ₂ S	Bis(2,2,6,6-tetramethyl-3,5 heptanedionato)copper and H ₂ S	200 °C	Multilayer films	ZnS and CuS deposited on top of each other	[180]
TiO ₂ -SnO ₂	Tetrakis(dimethylamino)tin (IV) and H ₂ O	Titanium(IV) isopropoxide and H ₂ O	175 °C and 160 °C for SnO ₂ and TiO ₂ growth, respectively.	Nanotube arrays	TiO ₂ -SnO ₂ nanotubes fabricated using porous alumina membrane template	[392]
ZnO-HfO ₂	Hafnium tetrakis(ethylmethylamino) and H ₂ O	ZnO nanowires prepared through hydrothermal method	200 °C	Nanowires	HfO ₂ growth on ZnO nanowires	[393]
ZnO-Al ₂ O ₃	Al ₂ (CH ₃) ₆ and H ₂ O	ZnO nanorods prepared through hydrothermal method	100 °C	Nanorods	Al ₂ O ₃ growth on ZnO nanorods	[394]
ZnO-Fe ₂ O ₃	(C ₂ H ₅) ₂ Zn and H ₂ O	Thermal oxidation of Fe foils to obtain Fe ₂ O ₃ nanorods	150 °C	Nanorods	ZnO growth on Fe ₂ O ₃ nanorods	[395]
ZnO-TiO ₂	TiCl ₄ and H ₂ O	(C ₂ H ₅) ₂ Zn and H ₂ O	120 °C, 160 °C, 200 °C, or 240 °C	Multilayers	ZnO and TiO ₂ deposited on top of each other	[396]
ZnO-TiO ₂ , TiO ₂ -ZnO	Tetrakis(dimethylamino)tin (IV) and H ₂ O	(C ₂ H ₅) ₂ Zn and H ₂ O	200 °C	Nanofibers	ZnO ALD on TiO ₂ nanofibers or vice versa	[377]
BN-AlN	TMA and N ₂ /H ₂	(C ₂ H ₅) ₃ B and N ₂ /H ₂	AlN at 100 °C and BN at 450 °C	Nanofibers	AlN growth on polymeric nanofibers followed by BN growth	[284]

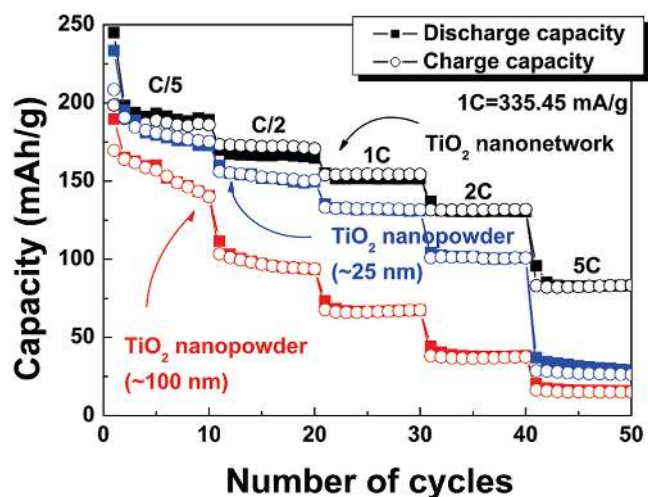


Figure 17. Rate capability of the TiO₂ nanonetwork, the 25 nm- and 100 nm-TiO₂ nanopowders from C/5 to 5 C for 10 cycles. Reprinted with permission from [405]. Copyright (2009) American Chemical Society.

ALD-grown nanostructured TiO₂ and ZnO semiconductors are active materials for applications in photocatalysis for organic dye degradation, photoelectrochemical water splitting, and metal-insulator-semiconductor (MIS) photocatalysts for hydrogen generation [421–430]. A good example features TiO₂/TiSi₂ core/shell nanostructures which were fabricated by combining CVD and ALD [431]. High surface area material network was particularly useful in improving photon absorption which in combination with high electrical conductance, provides superior charge transport properties. Photoelectrochemical water splitting was shown with this nanonetwork with a peak efficiency of 16.7% [431]. In another study, CdS/TiO₂ nanotube-array coaxial heterogeneous structures were fabricated and characterized for photocatalytic reactions such as water splitting and degradation of organic pollutants [432]. CdS was deposited conformally on TiO₂ nanotubes using electrochemical ALD; an analog of ALD based on surface limited reactions using under-potential deposition method [432]. The coaxial CdS/TiO₂ heterostructure significantly enhanced active contact area with electrolyte which reduced the distance that electrons and holes must travel resulting in enhanced photon absorption and photocurrent generation [432]. ALD of hematite was reported on TiSi₂ nanonets [433]. The nanonets performed a dual role of structural support and charge collector, allowing for an enhanced photon to charge conversion and resulting in enhanced photocurrents [433].

Paracchino *et al* reported a highly active photocathode for solar H₂ production which consisted of electrodeposited cuprous oxide protected against photocathodic decomposition in water by a thin ALD coating of Al-doped ZnO and TiO₂ [434]. Finally, electrodeposited Pt nanoparticles activated the nanostructures for hydrogen evolution. The electrodes showed photocurrents up to -7.6 mA cm^{-2} at a potential of 0 V versus the reversible hydrogen electrode at mild pH [434].

Pan *et al* fabricated TiO₂ porous structures by depositing TiO₂ on a reticular sponge template followed by a subsequent heat treatment [429]. Annealing of free standing TiO₂ porous structure resulted in phase transition and production of oxygen vacancies [429]. The porous TiO₂ structure demonstrated excellent photocatalytic ability owing to co-action of high surface area, oxygen vacancies, and the optimal crystal structure [429]. Singh *et al* reported design and fabrication of high-surface-area photocatalysts by depositing TiO₂ on fibrous nanosilica (KCC-1) using ALD [427]. Size quantization effects were observed from TiO₂ nanoparticles which were obtained after heat treatment of these ALD-coated samples of KCC-1 [427]. Enhanced photocatalytic activity was observed which was attributed to unique textural properties and morphology of KCC-1 and TiO₂ nanoparticle formation and their size quantization. Zhang *et al* fabricated novel model of MIS photocatalysts for hydrogen generation in the ultraviolet to near-infrared region [421]. The MIS photocatalysts consisted of metal co-catalyst (Pt), electron tunneling layer (ALD grown TiO₂), and photoactive non-stoichiometric core (Ti³⁺ doped TiO₂ nanocrystal). The MIS photocatalysts exhibited efficient hydrogen generation ($52 \mu\text{mol h}^{-1} \text{ g}^{-1}$), good reusability (16 h), and long-term stability (>7 d) [421]. ALD of ZnO was conducted on electrospun nylon 6,6 nanofibers (figure 20) and the core-shell nylon 6,6-ZnO nanofiber mats were utilized as a filtering/membrane material for treatment of organic pollutants for water purification (figure 21) owing to their efficient photocatalytic properties, structural flexibility, and stability [302]. ALD growth cycles numbers were changed to grow either ZnO nanoparticles or a continuous film on electrospun polymeric nanofibers (figure 22) for the fabrication of flexible photocatalytic nanofibrous membranes [295].

4.3. Photovoltaics and optoelectronics

Solar cell device technology have been under continuous development over the past five decades. Their wide spread usage is hindered by two main factors; limited conversion efficiency and high cost. ALD as a conformal thin film coating technique has been extensively utilized for surface passivation and humidity barrier of Si-based solar cells to improve cell efficiency [435]. Here, we will focus on the use of ALD to fabricate semiconductor nanostructures for applications in photovoltaics and optoelectronics.

ALD has been used to fabricate high surface area ZnO nanotube photoanodes using AAO templates for application in dye sensitized solar cells (DSSCs) [436]. ALD was used to coat AAO pores which provides a path for charge collection over tens of micrometer thickness. ZnO nanotube cells showed superior photovoltage and fill factor (FF) as compared to similar ZnO based devices [436]. In another study, ALD was used to deposit ITO in a porous template as a high-surface area photoelectrode, which was sequentially coated with amorphous TiO₂ [437]. Due to radial collection of electrons, this new photoelectrode architecture revealed higher current densities as compared to their counterpart control devices which lack efficient current collection [437]. Hamann *et al* fabricated photoanodes

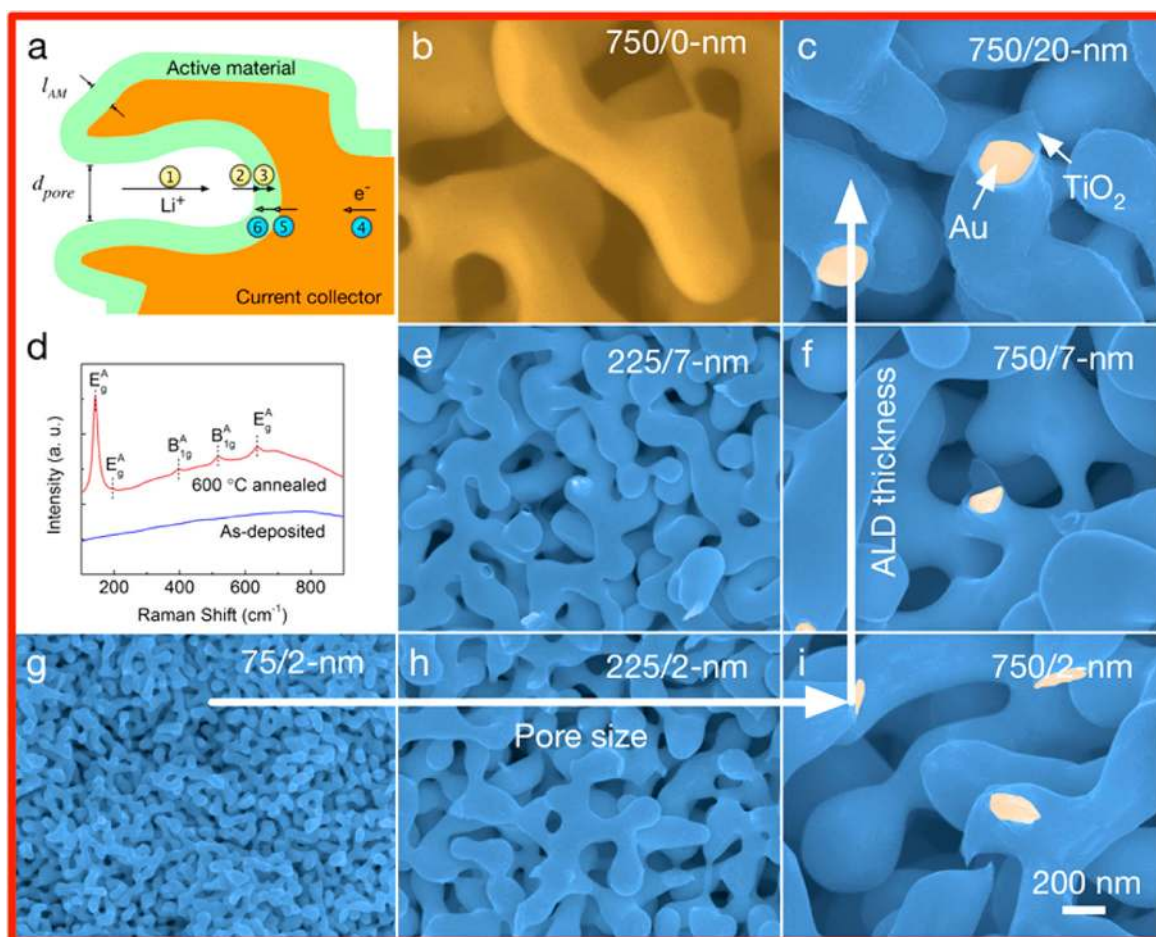


Figure 18. Relevant transport processes and microstructure of 3D np-Au/TiO₂ electrodes for lithium ion batteries. (a) An illustration of relevant transport processes in 3D porous LIB electrodes: (1) Li^+ transport through the electrolyte; (2) charge transfer at the electrolyte/electrode interface; (3) Li^+ transport in the active material; (4) electron transport in the current collector; (5) electron transfer from the current collector to the active material; (6) Li^+ /electron recombination. (b) Scanning electron micrograph (SEM) of np-Au with a pore size of 750 nm. (c), (e)–(i) SEM images of TiO₂ ALD coated np-Au samples after annealing at 600 °C for various pore size (75, 225, and 750 nm) and TiO₂ film thickness (2, 7, and 20 nm). All SEM images were taken at the same magnification. (d) Raman spectra from a TiO₂-coated np-Au sample measured before and after annealing at 600 °C revealing the phase transition from as-deposited amorphous TiO₂ to anatase. Reprinted with permission from [413]. Copyright (2015) American Chemical Society.

using high surface area mesoporous aerogel thin films as templates [438]. ZnO with a controlled variable thickness was conformally deposited on aerogels using ALD [438]. The electrodes used in DSSCs showed enhanced light harvesting and power conversion efficiency compared to other ZnO based DSSCs. Owing to its ease of fabrication, flexibility of design, and excellent initial performance, ALD-coated aerogel-based photoanodes are promising alternatives to nanoparticle based electrodes in DSSCs [438]. In another report, Park *et al* fabricated ZnO nanocrystallites coated with ultrathin TiO₂ ALD layer to increase the power conversion efficiency of DSSCs [438]. TiO₂ layer increased both FF and open circuit voltage as a result of decreased surface charge recombination without compromising on photocurrent density [438]. Foong *et al* employed a liquid-phase atomic layer deposition (LALD) process to coat TiO₂ in porous anodic alumina templates followed by alumina dissolution for

application in hybrid solar cells [439]. The LALD overcomes the vacuum conditions needed in conventional gas phase ALD and provides yet an ALD-like GPC and high conformality [439]. The viability of TiO₂ nanotube arrays grown on indium tin oxide (ITO)–glass electrodes was also examined for use in model hybrid poly(3-hexylthiophene) (P₃HT):TiO₂ solar cell devices [439].

Chandiran *et al* fabricated mesoscopic photoanodes for solid-state perovskite absorber solar cells by employing ALD grown TiO₂ overlayer on the mesoporous nanoparticle TiO₂ films [440]. The nanoparticle-TiO₂-ALD TiO₂ film is infiltrated with perovskite absorber. Authors showed that their structured photoanodes block electron recombination effectively and it overrides the commonly used multi-step passivation prevailing in the DSC community [440]. Observations revealed that even a 2 nm thick TiO₂ can impede the electron back reaction effectively from FTO and TiO₂ surface

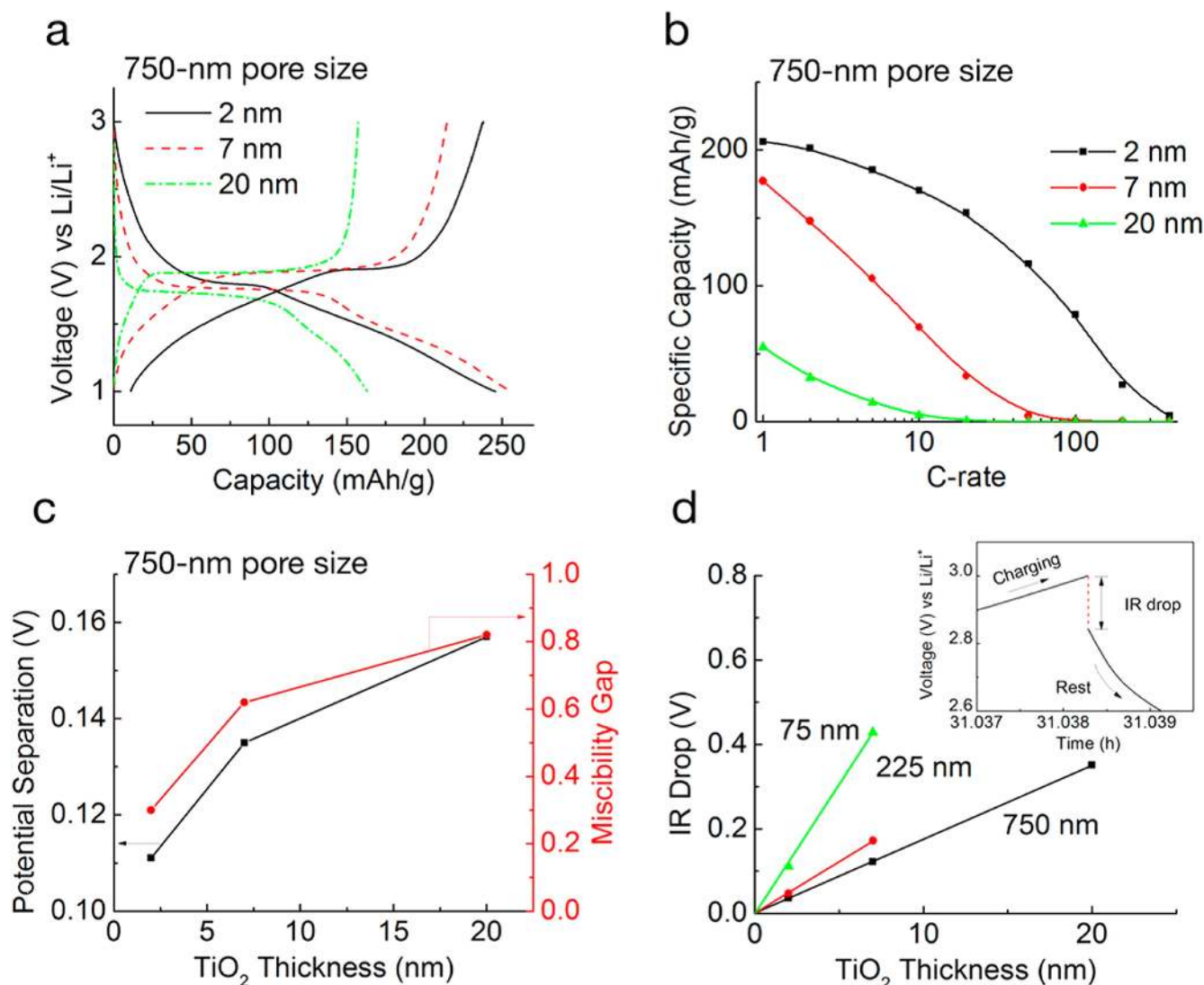


Figure 19. Effect of the TiO₂ layer thickness on (a) charge/discharge voltage profiles (measured at 0.1 C) and (b) the rate performance of nanoparticle-Au/TiO₂ electrodes with 750 nm pores. (c) Potential separation and miscibility gap measured at 0.1 C as a function of the TiO₂ layer thickness. (d) IR drop/TiO₂ layer thickness dependency for various pore sizes measured by the current interrupt method. The inset shows a typical charge-rest-discharge voltage profile collected from a 750/7 nm sample (charge/discharge at 10 C, interrupted at 3 V). Note the linear correlation between IR drop and TiO₂ layer thickness. Reprinted with permission from [413]. Copyright (2015) American Chemical Society.

resulting in a photovoltaic power conversion efficiency of 11.5% [440]. In another similar work, authors examined the passivation effect of ALD TiO₂ on hydrothermally grown one-dimensional (1D) TiO₂ nanorod (NR) arrays (figure 23) for solid-state perovskite-sensitized solar cells [441]. Their findings revealed that 4 nm thick ALD-grown TiO₂ passivated NR sample controls the back flow reactions and shows a power conversion efficiency as high as $\eta = 12.53\%$ for the CH₃NH₃PbI₃ perovskite absorbing layer [441].

Nanostructured ALD ZnO and TiO₂ are the leading materials used for UV photodetection owing to their suitable band gap [339, 442–446]. High-quality ZnO–TiO₂ core shell nanowires revealed enhanced UV sensing properties owing to superior anti-reflection properties and electron–hole

separation mechanism. The preparation comprised of two steps: ZnO nanowires fabrication by hydrothermal synthesis followed by ALD of TiO₂. The photoresponsivity was 495 A/W at 373 nm under -10 V which is ~ 8 times higher than photodetector fabricated from bare ZnO NWs (figure 24) [445]. Three-dimensional polyacrylonitrile/ZnO material were prepared by combination of electrospinning and atomic layer deposition. The UV photoresponse current for this configuration was enhanced by a factor of 250 compared to a flat electrode [339]. TiO₂/Ag nanorods (figure 25) were fabricated by successive glancing angle deposition of Ag on Ag film/Si template and ALD of TiO₂. Nanostructured devices showed a photo response enhancement factor of 1.49×10^2 under 3 V reverse bias (figure 26) [442].

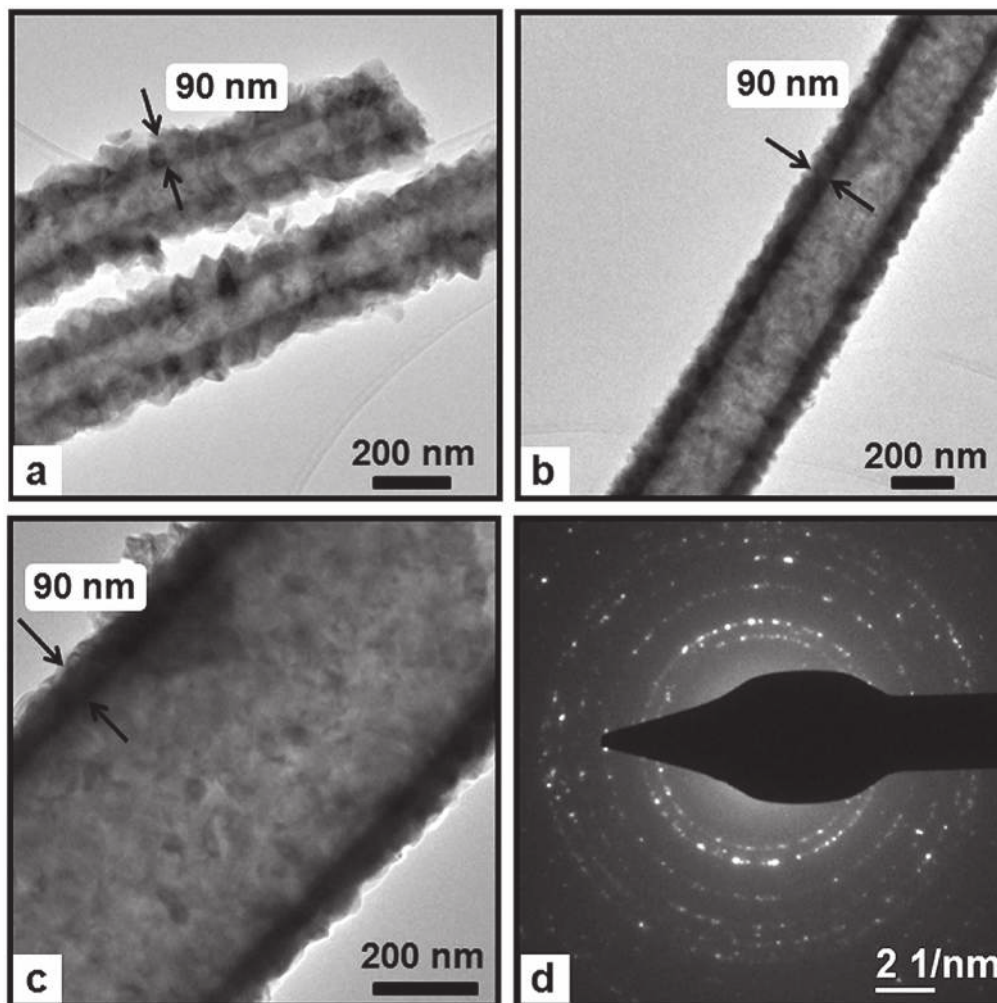


Figure 20. Representative TEM images of (a) 8%-nylon 6,6/FA-ZnO, (b) 5%-nylon 6,6/HFIP-ZnO, (c) 8%-nylon 6,6/HFIP-ZnO core-shell nanofibers; (d) representative SAED pattern of the core-shell nylon 6,6-ZnO nanofibers (8%-nylon 6,6/HFIP-ZnO NF). Reprinted with permission from [302]. Copyright (2012) American Chemical Society.

4.4. Chemical sensing

Gas sensors work on the basis of the modulation of the materials' physical properties (e.g., electrical, optical) in presence of the target species/molecules. Thin-film and nanostructured metal-oxide semiconductors are most widely used as active layers in chemo-resistive gas sensors, where SnO_2 is one of the most intensely studied semiconductor material [312, 395, 447–454]. Owing to the strong correlation between grain size and sensor response, use of ALD-grown nanostructured and heterostructured materials show promise in the enhancement of gas sensing capacity.

Chemical sensors find important applications in everyday life which modify their properties (e.g., electrical, optical) in presence of target species. In general, ALD-grown semiconductor metal oxides have been utilized as active layers in chemo-resistive sensors and the most prominent one is SnO_2 [312, 395, 447–454]. One of the recent notable work in this area was from Kim *et al* who reported superior toluene sensing properties of SnO_2 -ZnO core-shell nanowires functionalized with Pt nanoparticles (figure 27)

[453]. SnO_2 nanowires were fabricated via vapor-liquid-solid growth on patterned electrode and ZnO shell layers were grown subsequently via ALD process. Pt nanoparticles were attached to core-shell nanowires via γ -ray radiolysis [453]. An exceptional sensitivity of 279 was obtained for 100 ppb of toluene, where the response was measured R_a/R_g , where R_a and R_g represent the device resistance values in the absence and presence of an analyte gas, respectively. The dual effect of the expanded electron-depleted region in the ZnO layer and the catalytic effect of the functionalized Pt NPs are the reason behind exceptional toluene sensitivity and selectivity [453].

Park *et al* examined the ethanol gas sensing properties of $\text{In}_2\text{O}_3/\text{ZnO}$ core-shell nanowires [450]. The core-shell nanowires were grown by evaporation of indium powder in an oxidizing atmosphere and subsequent atomic layer deposition of ZnO [450]. The $\text{In}_2\text{O}_3/\text{ZnO}$ core-shell nanowires exhibited more than 6-fold higher response to 1000 ppm ethanol at 300 °C than pristine In_2O_3 nanowires (figure 28) [450].

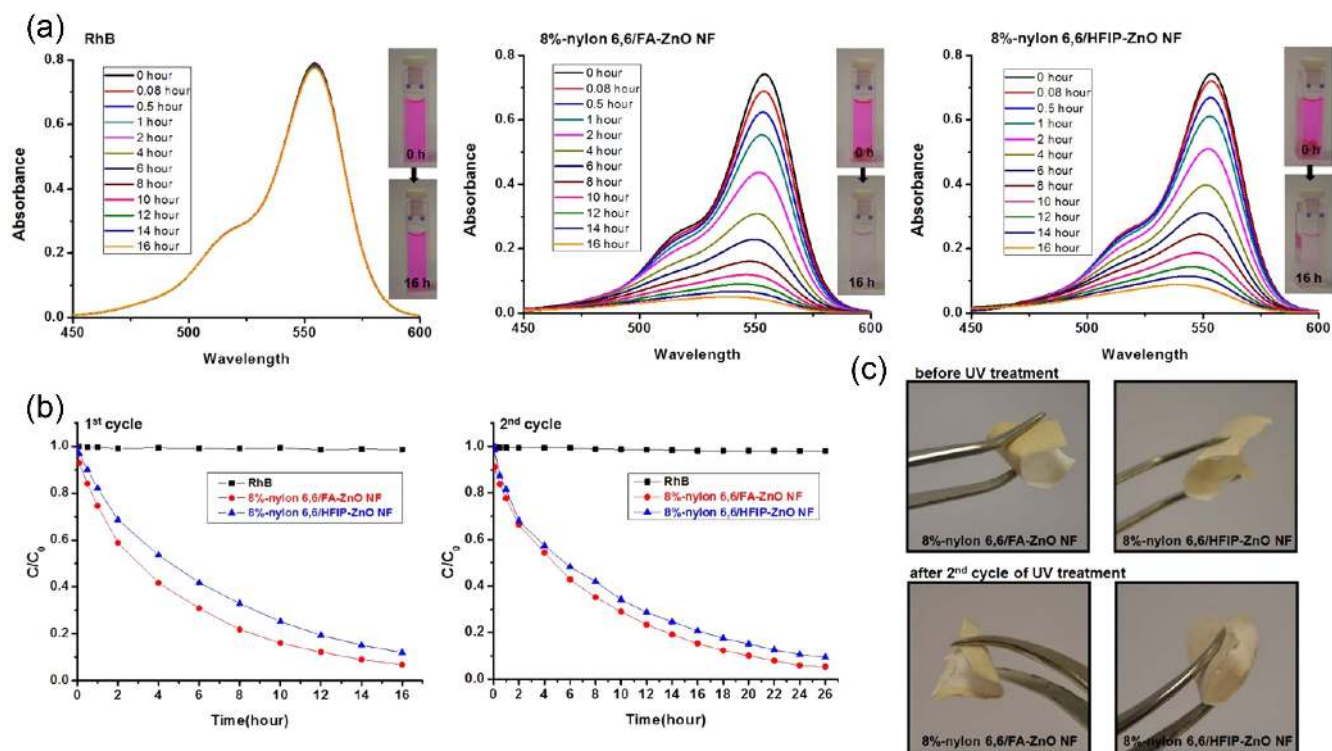


Figure 21. (a) UV-vis spectra of the Rh-B solution with and without core-shell nylon 6,6-ZnO nanofibers as a function of the UV irradiation time for 1st cycle experiment, (b) the rate (C/C_0) of Rh-B degradation of the Rh-B solution with and without core-shell nylon 6,6-ZnO nanofibers by exposing UV light with 365 nm wavelength for 1st and 2nd cycle experiments; (c) representative photographs of the flexible nylon 6,6-ZnO core-shell nanofibers before UV treatment and after 2nd cycle of UV treatment. Reprinted with permission from [302]. Copyright (2012) American Chemical Society.

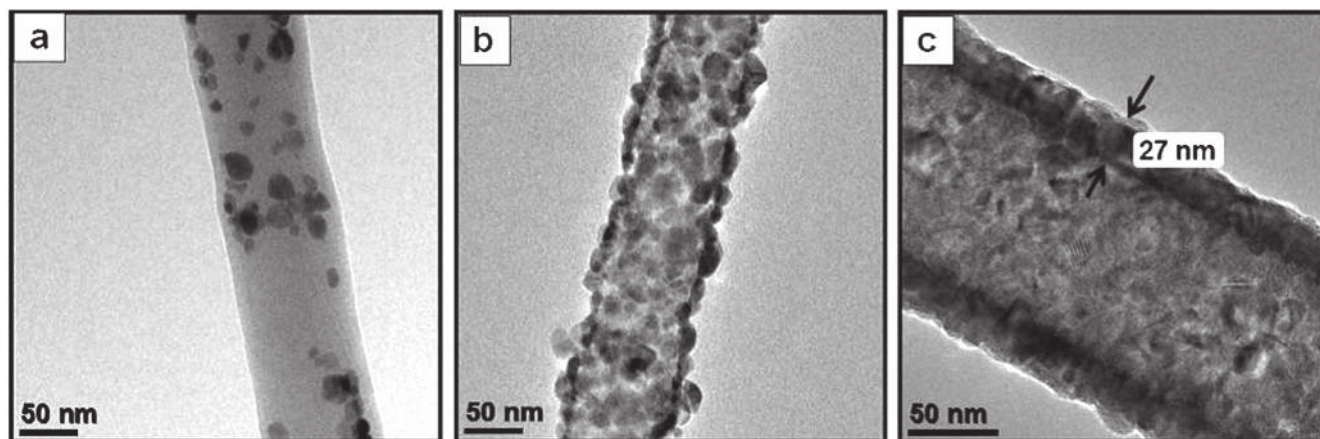


Figure 22. Representative TEM images of nanofibers: (a) nylon-ZnO nanoparticles, (b) nylon-ZnO nanoparticles (highly dense), and (c) nylon-ZnO nanocoating. Reprinted with permission from [295]. Copyright (2013) Royal Society of Chemistry.

Katoch *et al* reported fabrication of TiO_2/ZnO double-layer hollow fibers (DLHFs) as superior sensing materials for reducing gases [447]. The DLHFs were synthesized by sequentially growing TiO_2 and ZnO on sacrificial polymer templates followed by a final thermal treatment [447]. The outer ZnO layer becomes more resistive after donating electrons to inner TiO_2 layer. After the exposure of reducing gases such as CO, the outer resistive ZnO layer partially regains its original resistivity [447].

5. Conclusions and outlook

ALD-grown nanoscale compound semiconductor materials have already demonstrated significant potential for energy conversion and storage, opto-electronics, flexible/wearable electronics, photocatalysis, and sensing applications. ALD shows its superiority particularly in high-surface area nano-templated semiconductors, where lack of precise thickness control and 3D conformality are detrimental. However, the

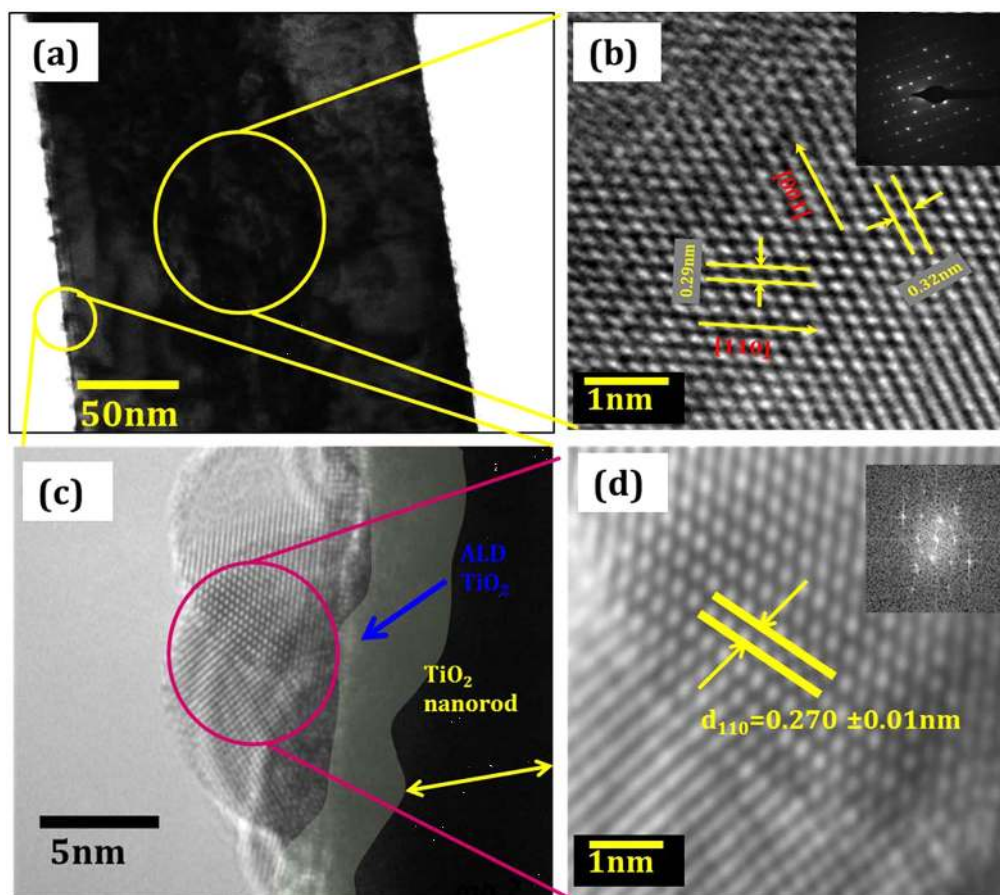


Figure 23. TEM and HRTEM images of TiO_2 nanorods coated with $\text{CH}_3\text{NH}_3\text{PbI}_3$. (a) TEM image of a $\text{CH}_3\text{NH}_3\text{PbI}_3$ -covered TiO_2 nanorod. (b) HRTEM image of a TiO_2 nanorod. The inset shows the SAED pattern. (c) Highly magnified HRTEM image of the $\text{CH}_3\text{NH}_3\text{PbI}_3$ and TiO_2 . The ALD layers and TiO_2 nanorods are indicated by the respective colors. (d) HRTEM image and (inset) live FFT pattern of the selected area of $\text{CH}_3\text{NH}_3\text{PbI}_3$ nanoparticles in (c). Reprinted with permission from [441]. Copyright (2015) American Chemical Society.

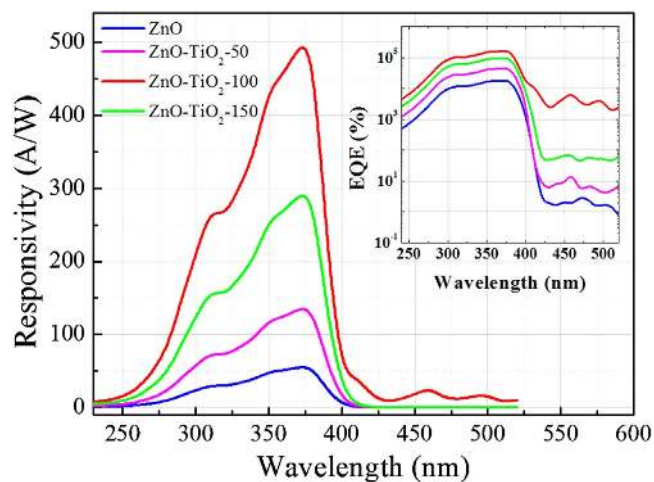


Figure 24. Photoresponsivity spectra of the photodetectors fabricated from the bare ZnO NWs and from the ZnO- TiO_2 core-shell NWs. Reprinted with permission from [445]. Copyright (2014) Elsevier.

full potential of these materials is hampered by the insufficient crystalline quality due to relatively high impurity incorporation within the films as a result of low substrate temperatures and incomplete ligand-exchange surface reactions. Nevertheless, there is an increasing effort in developing novel ALD

recipes for semiconductor materials using alternative precursor chemistries, innovative reactor designs, and finely tuned deposition parameters. As materials and device engineering rapidly approaches the atomic-scale era, there is an increasing need for precisely engineered atomic-scale materials covering an even wider spectrum of alternative set of materials. Device sizes and individual layer dimensions are shrinking towards single-digit nm and even sub-nm scale, where poly-crystalline material might even meet local epitaxial quality by featuring sufficiently large crystal sizes. Moreover, the needed device layer thicknesses are reducing below the critical thickness values which might eliminate the lattice mismatch problem for semiconductor heterostructures. As a result, ALD will most probably be an even stronger contender to deliver new materials solutions at the atomic-scale, including compound and elemental semiconductors for future complex device architectures.

Finally, we would like to share our opinions regarding future perspectives, challenges, and opportunities for ALD-grown nanoscale semiconductors:

- The crystalline quality of low-temperature ALD-grown layers should be further improved via systematic studies featuring various alternative activation mechanism including plasma, radical/electron, UV/photo/e-beam-assisted

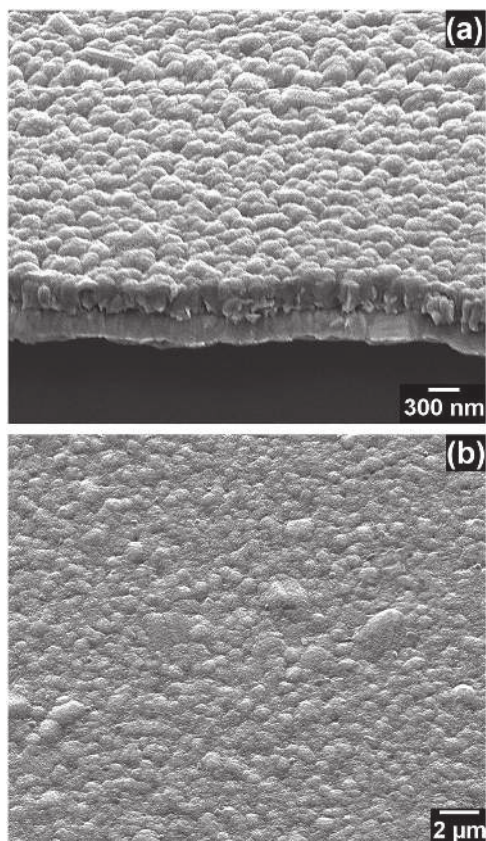


Figure 25. (a) Tilted and (b) top view SEM images of TiO_2 deposited on Ag nanorods/Ag thin film/Si by ALD, respectively. Reprinted with permission from [442]. Copyright (2015) AIP publishing.

approaches, substrate bias, optimized reactor designs, and novel precursor chemistries

- In the post-Moore era, CMOS technology needs more than ever novel materials solutions with atomic-scale precision engineering. ALD is already an integral part of CMOS processing and will play an even more critical role in feature sub-10 nm or single-digit nm technology nodes (7, 5, 3, 2 nm), where low-temperature grown atomic-scale semiconductors might find use within the 3D device architectures.
- Atomic-scale materials processing era will unleash quantum-size effects in low-dimensional semiconductors, thereby introducing challenges as well as opportunities for novel device designs. ALD-grown semiconductors might play a significant role at such reduced dimensions. Moreover, even non-semiconducting materials such as certain semi-metals might exhibit semiconductor-like properties due to quantum confinement effect at few atomic-layer dimensions [455]. The exploitation of materials at the atomic level will enable an unprecedented power of materials and device engineering.
- ALD-grown semiconductors might enable a wide range of emerging device applications for future internet of things (IoT) platform and cyber-physical systems, which necessitates compact, autonomous sensors with integrated energy harvesting and storage systems, and wireless

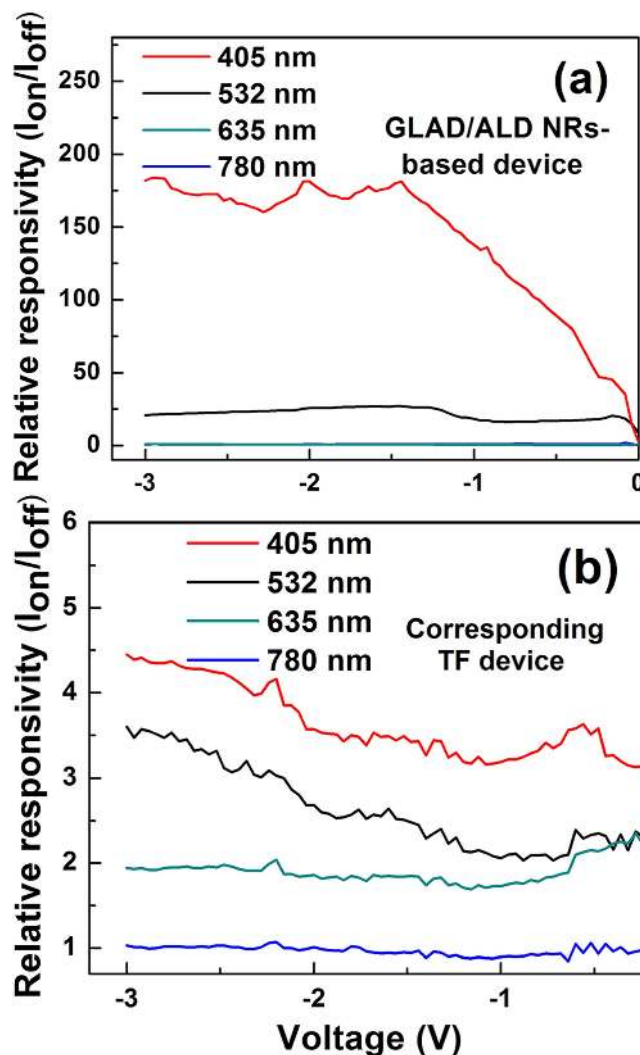


Figure 26. Relative responsivity values measured versus applied reverse bias of the fabricated (a) Al/ TiO_2 /Ag(NRs)/Ag(TF)/Si and (b) Al/ TiO_2 /Ag(TF)/Si photodiodes under illumination of different laser diodes. Reprinted with permission from [442]. Copyright (2015) AIP publishing.

communication transmitter/receiver modules on cost-effective substrates such as flexible and low-temperature compatible polymers

- ALD know-how in semiconductor materials will trigger other atomic layer processing techniques including atomic layer etching and atomic layer doping to form a complete semiconductor processing toolbox with atomic-scale precision and control
- In order to develop atomic-scale device structures without the need for highly complex and expensive advanced lithography techniques, an ultimate advancement in materials processing would be to develop self-aligned processes, which necessitates selective material deposition and etching recipes. Towards this lithography-free device fabrication goal, selective atomic layer processing of dielectrics, metals, and semiconductors should be developed. Such a bottom-up processing tool-box will lead to atomic-level precision manufacturing capability.

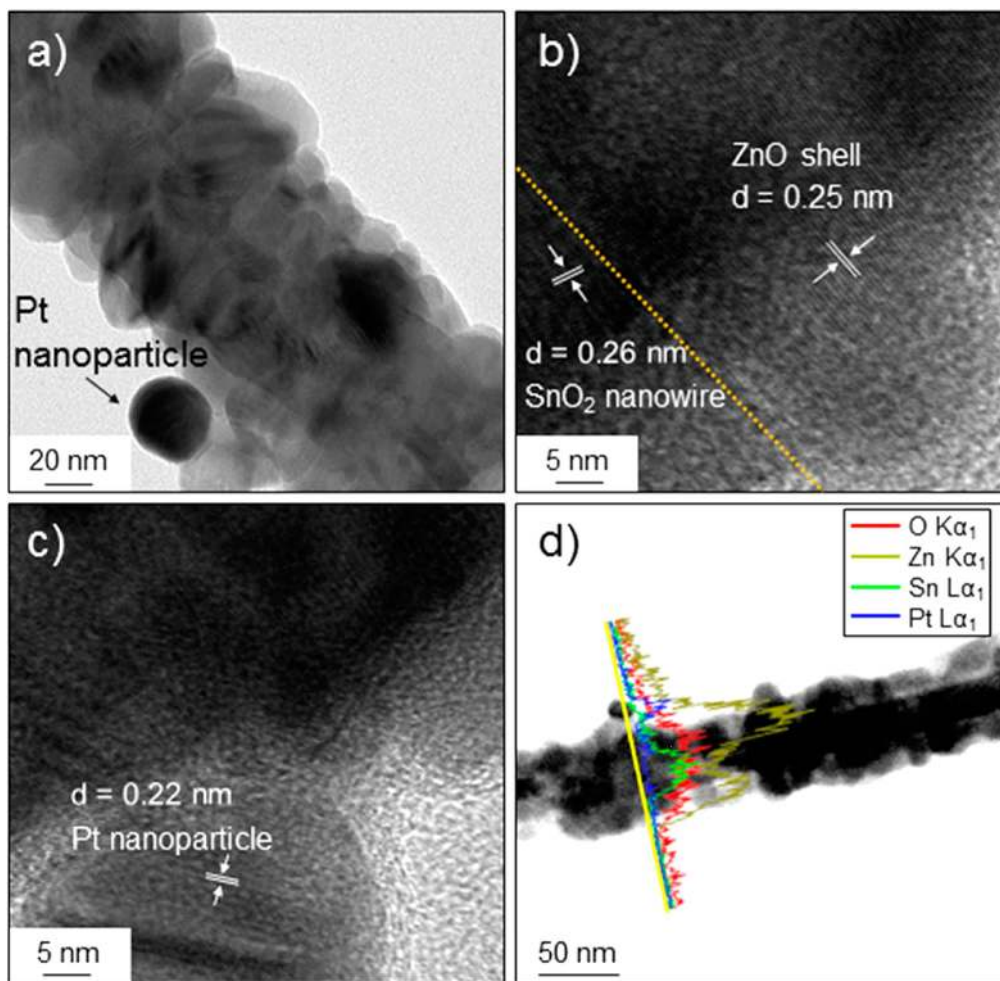


Figure 27. TEM images of the Pt NP-functionalized SnO₂-ZnO core-shell NWs with a ZnO shell thickness of 50 nm. (a) A low-magnification TEM image. (b), (c) High-resolution lattice images. (d) EDS line profiles for O, Sn, Zn, and Pt. Reprinted with permission from [453]. Copyright (2015) American Chemical Society.

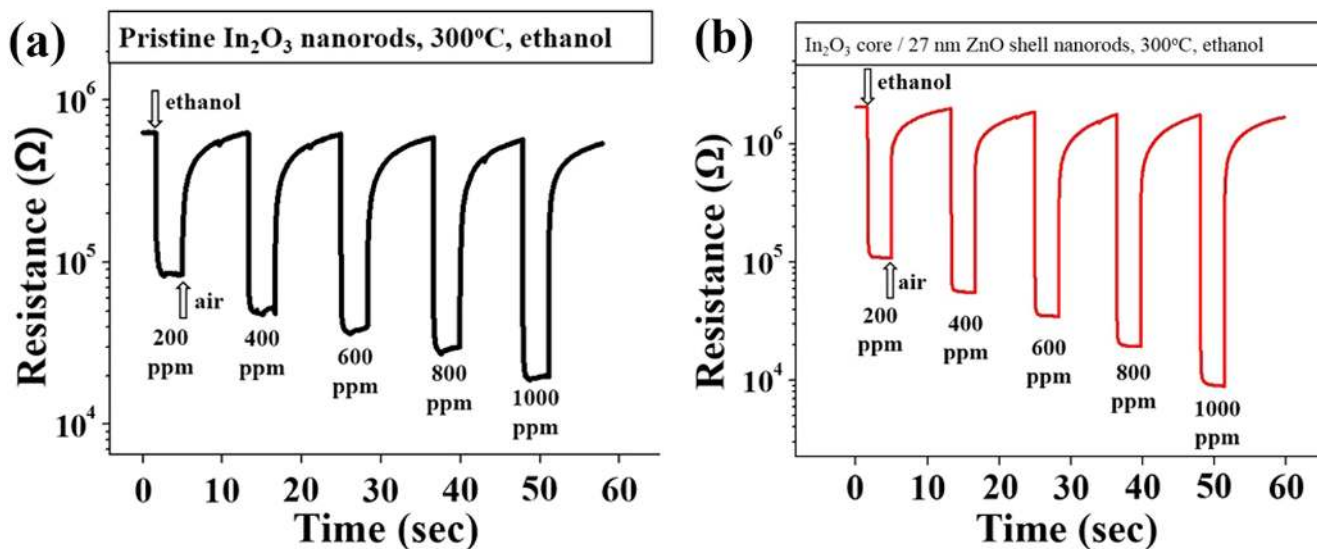


Figure 28. Electrical responses of the gas sensors fabricated from (a) pristine In₂O₃. (b) In₂O₃-core/ZnO-shell nanowires. Reprinted with permission from [450]. Copyright (2014) American Chemical Society.

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