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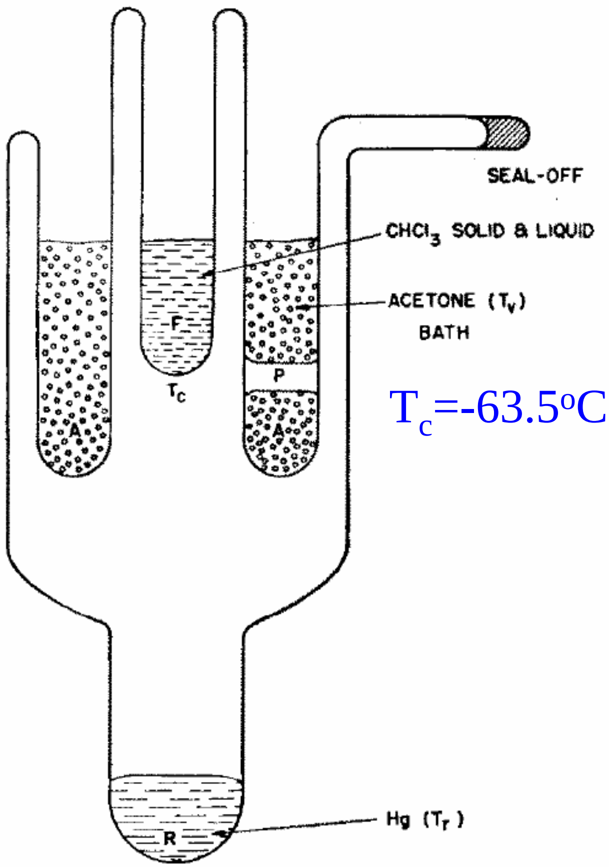
  - (a)
  - (b)

Synthesis of Nano Materials- 1D from gas phase

❑ Evaporation and Condensation

ex) Hg



T_c=-63.5°C

$$\alpha = \frac{p}{p_o}$$: supersaturation ratio

- T_v= 25°C platelet, 0.3 mm length, ~0.03 μ thickness (α=1600)
  25~-50°C platelet+whisker
  -50°C whisker, 1~2 mm length, ~0.01 μ radius (α=100)
- Zn: 350°C (α~3)
  Cd: 250°C (α~20)
  Ag: 850°C (α~10)
  CdS: 800°C (α~2)
- axial screw dislocation proposed but not confirmed
  microtwin and stacking fault also proposed
- higher growth rate than the condensation rate
  (dislocation-diffusion theory)

G.W. Sears, Acta Metal. 3 (1955) 361.

Synthesis of Nano Materials- 1D from gas phase

❑ Evaporation and Condensation

- thermal evaporation- ZnO- ZnO powder at 1400^{o}C

nanobelt

- no screw dislocation except stacking fault
- clean, atomistically sharp, free of sheath of amorphous
- absence of amorphous globules on the tip (not VSL mechanism)
- not related to crystal structure (SnO_2- rutile In_2O_3- C-rare earth CdO-NaCl cubic Ga_2O_3- monoclinic PbO_2-rutile)

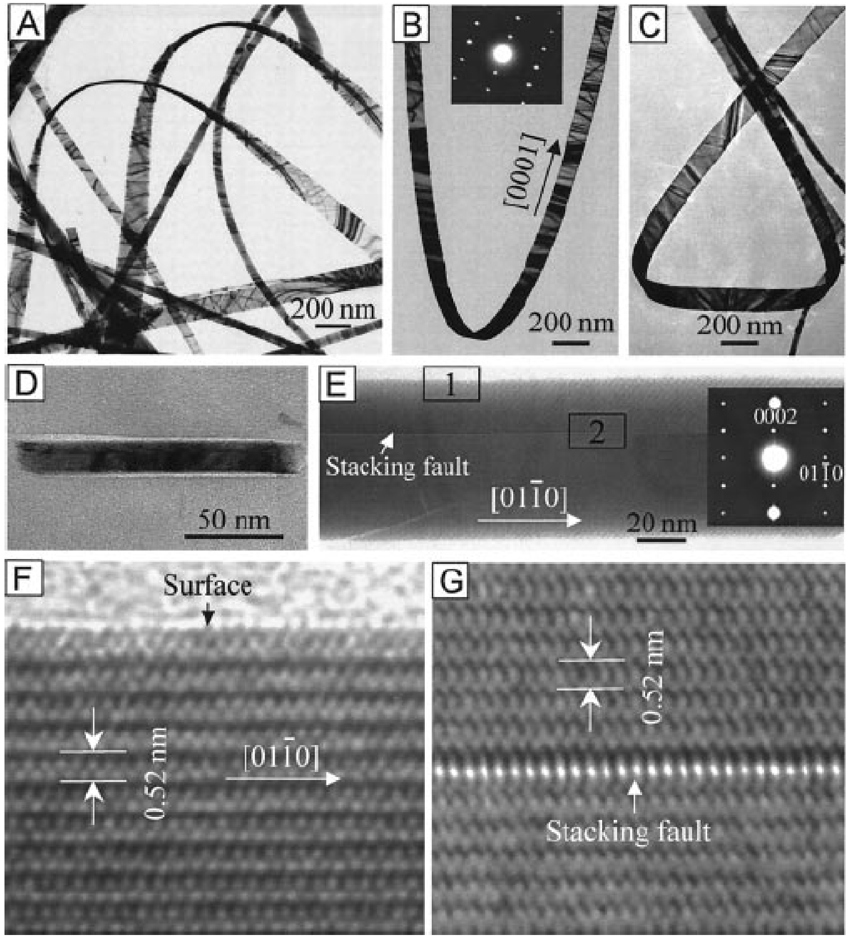


Fig. 2. TEM and HRTEM images of ZnO nanobelts showing their geometrical shape. (A to C) TEM images of several straight and twisted ZnO nanobelts, displaying the shape characteristics of the belts. (D) Cross-sectional TEM image of a ZnO nanobelt, showing a rectanglelike cross section with width-to-thickness ratio of ~9. The cross-sectional TEM specimen was prepared by slicing nanobelts embedded in epoxy with an ultramicrotone. (E) TEM image of a nanobelt growing along [01$\bar{1}$0], showing only one stacking fault present in the nanobelt; this type of nanobelt is the only one that has a defect. The inset shows the electron diffraction pattern. (F) HRTEM image from box 1 in (E), showing a clean and structurally perfect surface (the amorphous contrast seen above the surface is from the amorphous carbon film used to support the nanobelt for TEM imaging). (G) HRTEM image from box 2 in (E), showing the stacking fault.

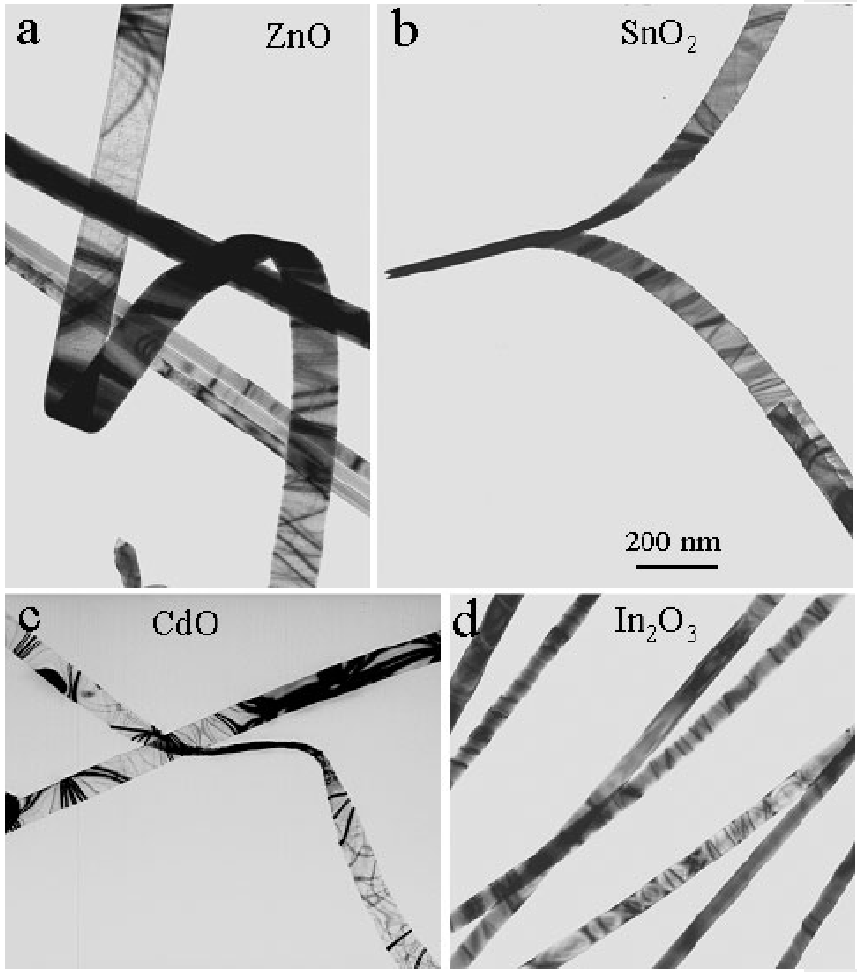
Z.W. Pan, Science, 291 (2001) 1947.

Synthesis of Nano Materials- 1D from gas phase

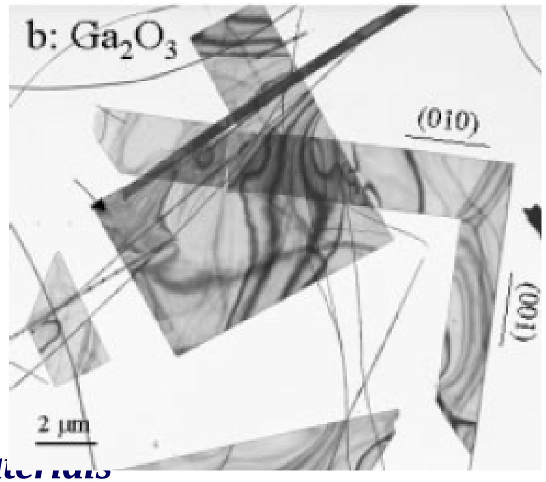
❑ Evaporation and Condensation

Nanobelts	Crystal structure	Growth direction/plane	Top surfaces	Side surfaces
ZnO	Wurtzite	[0001]	$\pm(2\bar{1}\bar{1}0)$	$\pm(0\bar{1}10)$
ZnO	Wurtzite	$[01\bar{1}0]$	$\pm(2\bar{1}\bar{1}0)$	±(0001)
Ga_2O_3	Monoclinic	(010)	±(100)	$\pm(10\bar{1})$
Ga_2O_3	Monoclinic	(001)	±(100)	±(010)
SnO_2	Rutile	[101]	$\pm(10\bar{1})$	±(010)
In_2O_3	C-Rare earth	[001]	±(100)	±(010)
CdO	NaCl	[001]	±(100)	±(010)
PbO_2	Rutile	[010]	(201)	$\pm(10\bar{1})$

nanobelt



nanosheet



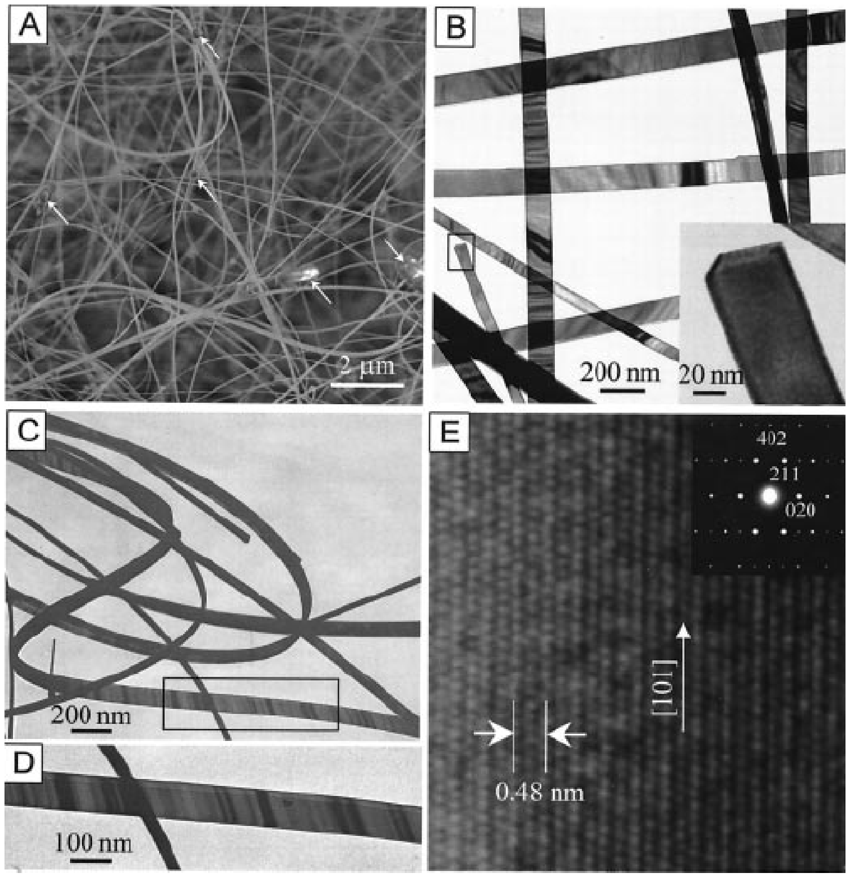
Z.L. Wang, Adv. Mater. 15 (2003) 432.

Synthesis of Nano Materials- 1D from gas phase

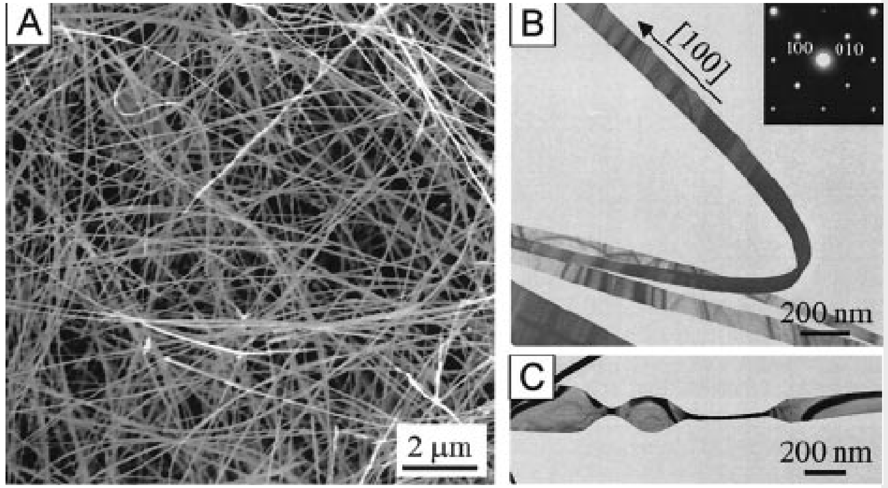
❑ Evaporation and Condensation

- thermal evaporation- SnO_2, In_2O_3, CdO

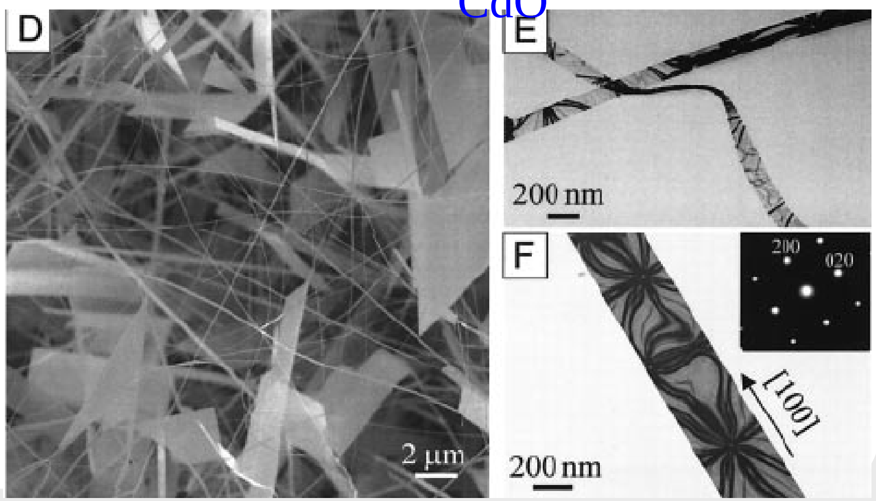
SnO_2



In_2O_3



CdO

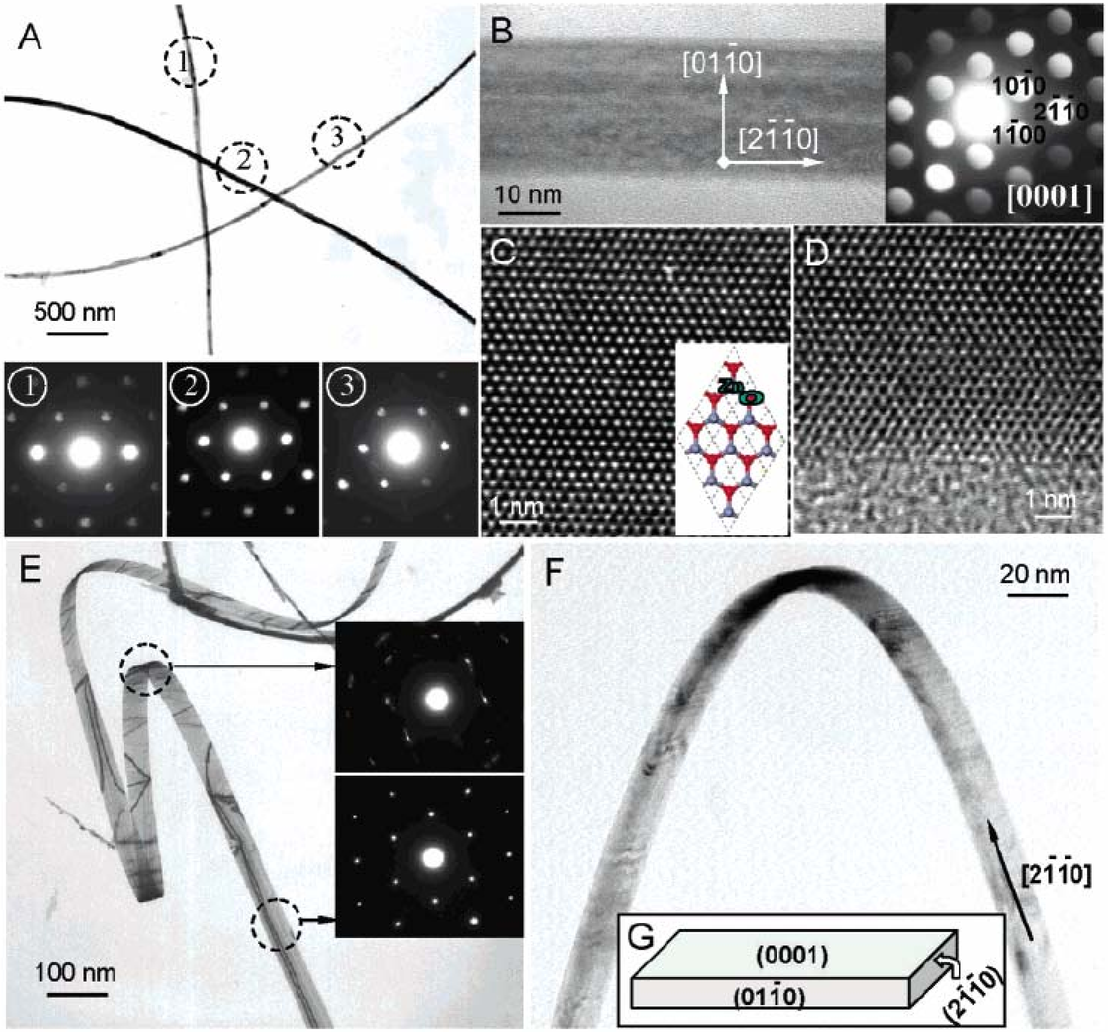


Z.W. Pan, Science, 291 (2001) 1947.

Synthesis of Nano Materials- 1D from gas phase

❑ Evaporation and Condensation

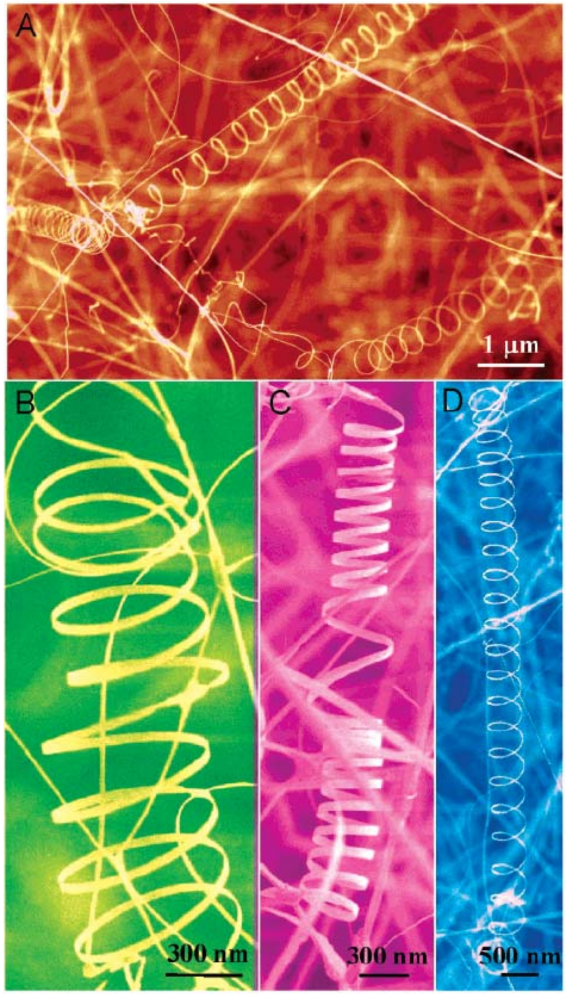
- (0001) polar surface-dominated ZnO nanobelts
  - zinc and oxygen-terminated ±(0001) surface
  - spontaneous polarization
  - minimization of the total energy contributed by spontaneous polarization and elasticity
    → helical growth

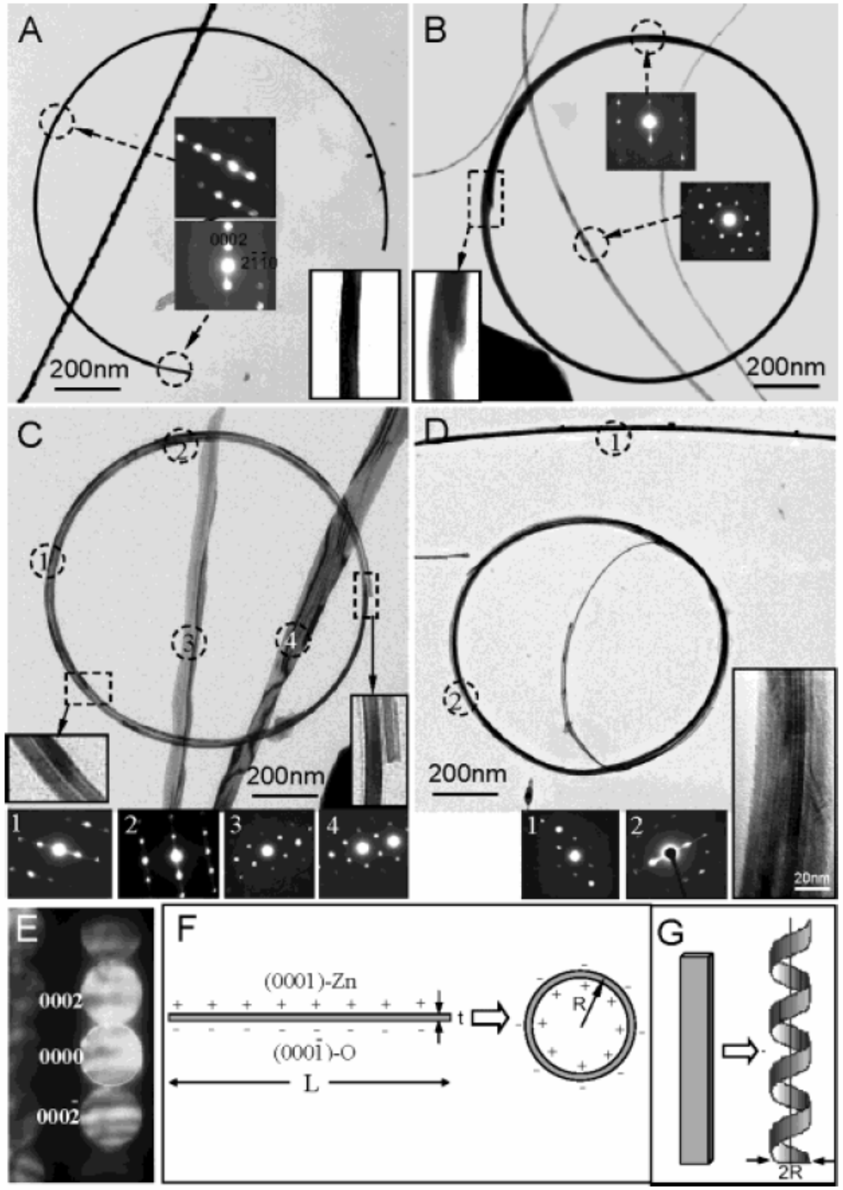


X. Yang, Nano Lett. 3 (2003) 1625.

Synthesis of Nano Materials- 1D from gas phase

❑ Evaporation and Condensation



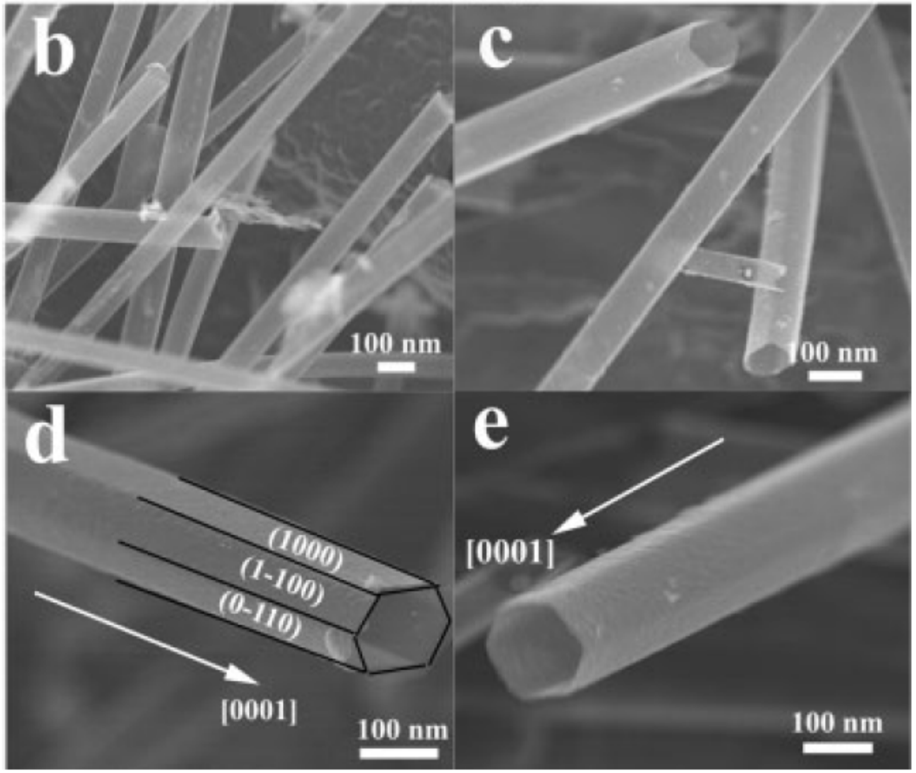


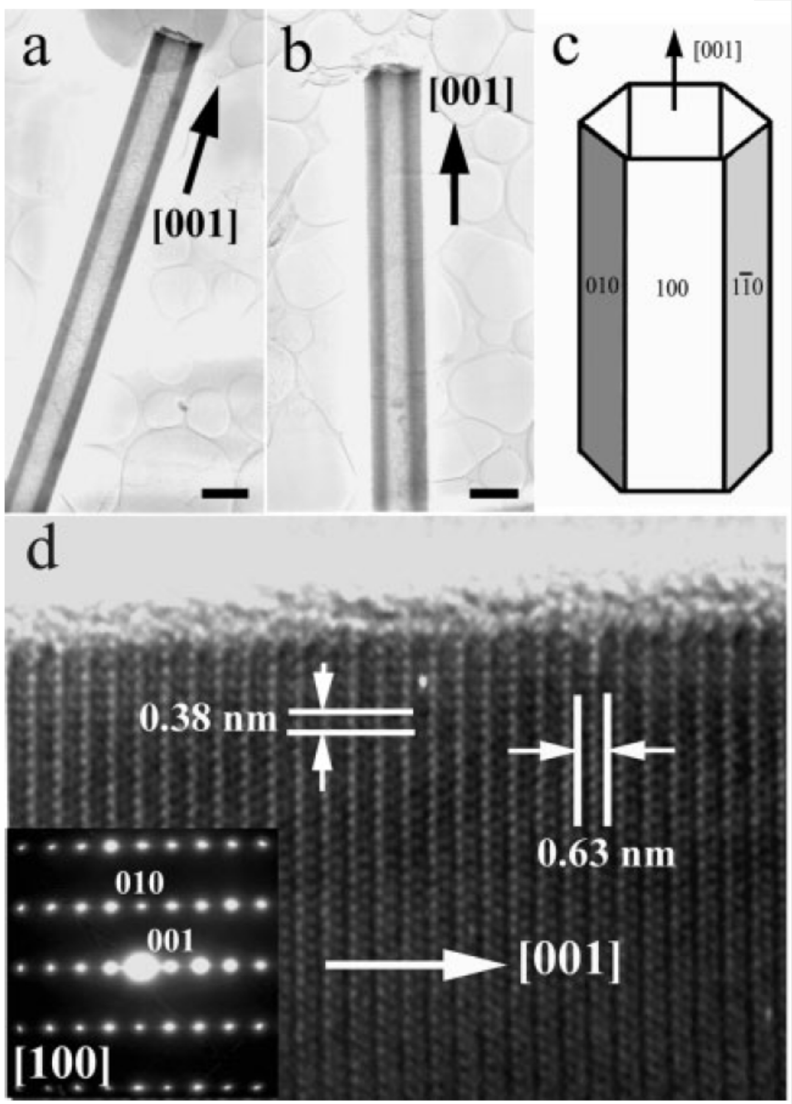
X. Yang, Nano Lett. 3 (2003) 1625.

Synthesis of Nano Materials- 1D from gas phase

❑ Evaporation and Condensation

- ZnS (induction furnace, 1500~1700^{o}C sapphire substrate)





L.-W. Yin, Adv. Mater. 17 (2005) 1972.

Synthesis of Nano Materials- 1D from gas phase

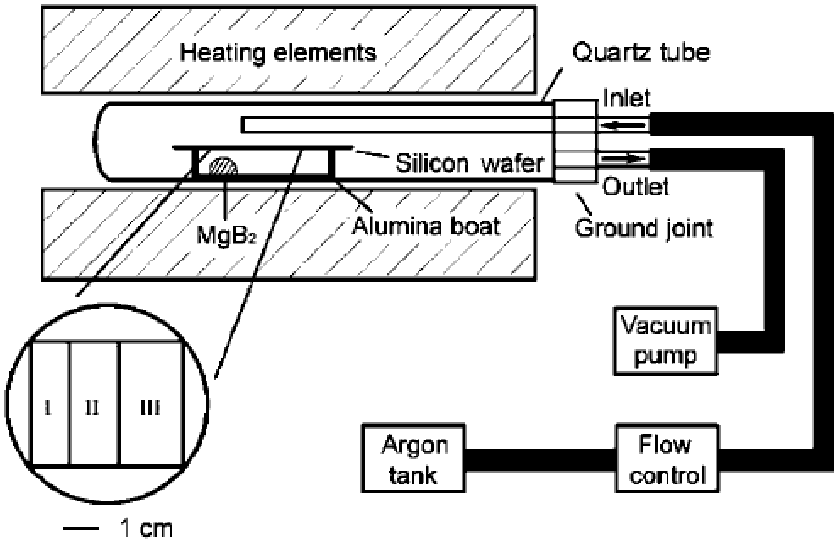
Evaporation and Condensation

ex) MgO (thermal evaporation of MgB_2 at 800~900°C)

Oxidation of Mg or direct reaction

$$2\,MgB_{2(s)} \longrightarrow Mg_{(g)} + MgB_{4(s)}$$

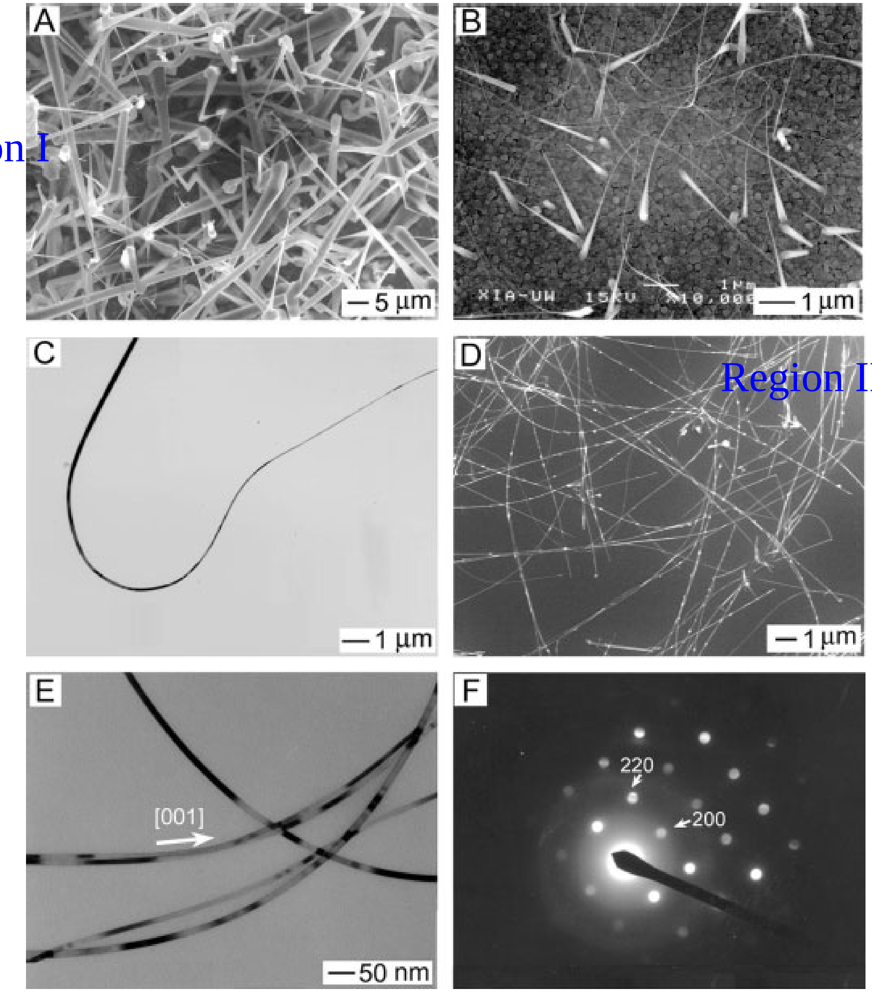
$$4\,MgB_2 + O_2 \longrightarrow 2\,MgO + 2\,MgB_4$$



Region I

Region II

Region III



Y. Yin, Adv. Func. Mater. 12 (2002) 293.

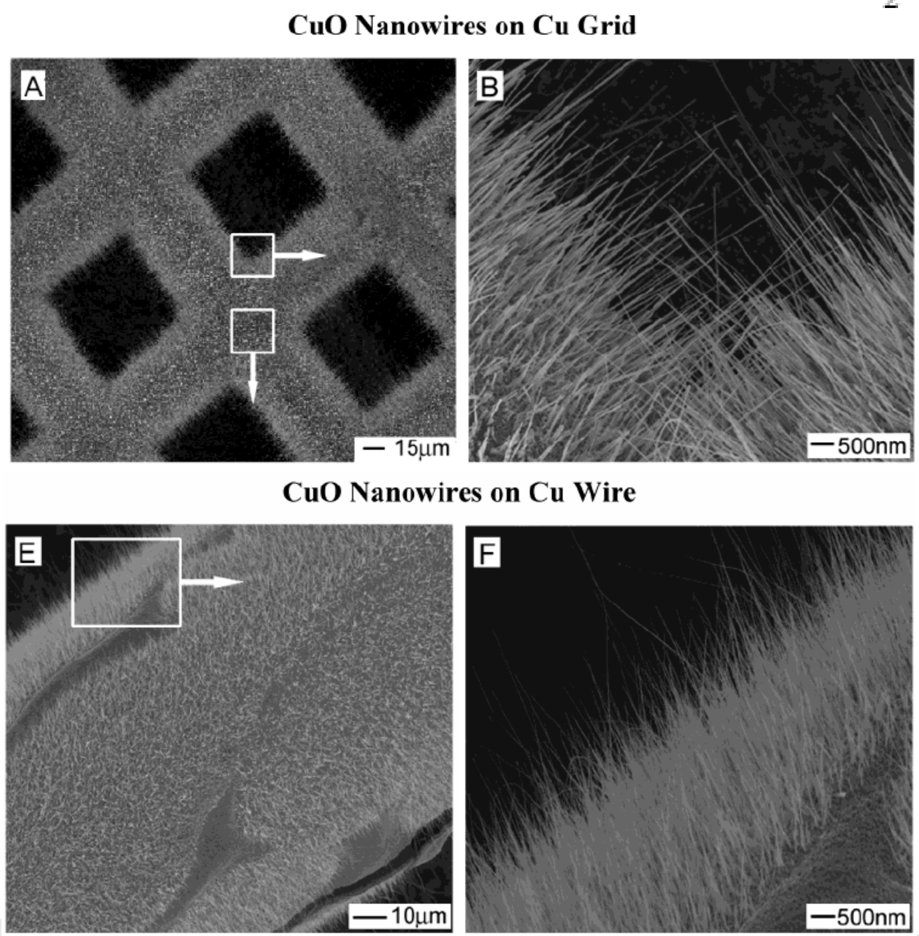
Synthesis of Nano Materials- 1D from gas phase

❑ Evaporation and Condensation

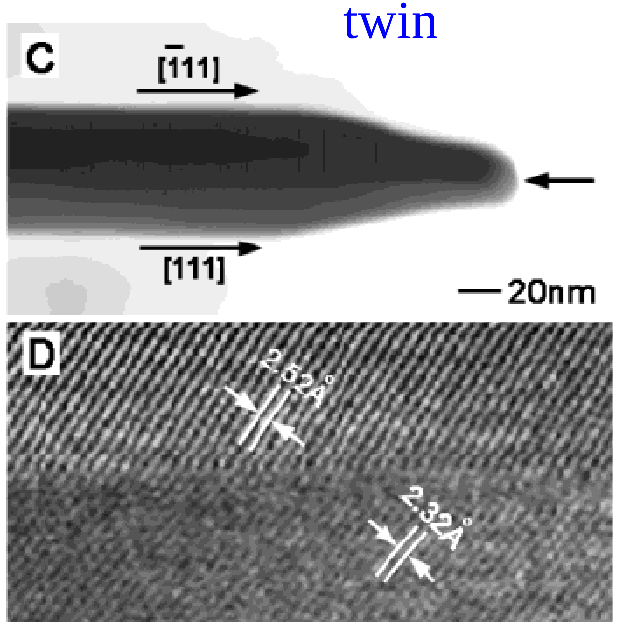
ex) CuO (thermal oxidation of s

$$4Cu + O_2 \rightarrow 2Cu_2O$$

$$2Cu_2O + O_2 \rightarrow 4CuO$$



bicrystal
twin



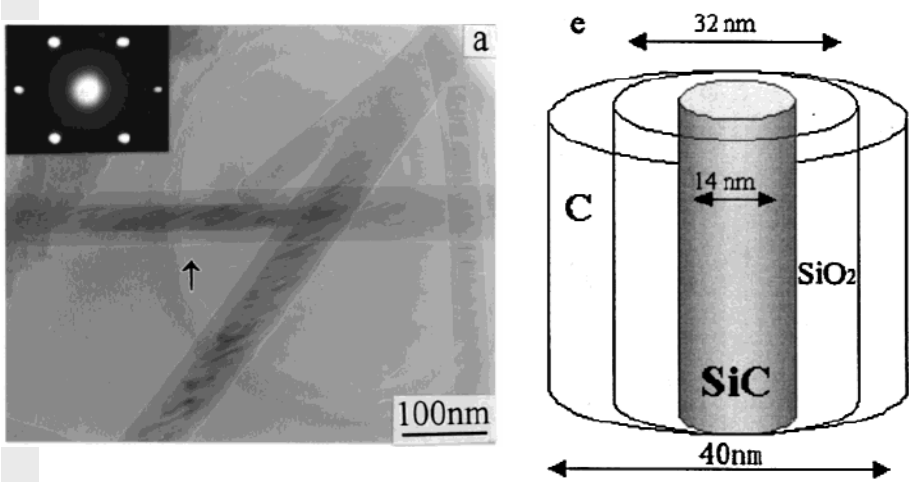
X. Jiang, Nano Lett. 2, 1333 (2002).

Synthesis of Nano Materials- 1D from gas phase

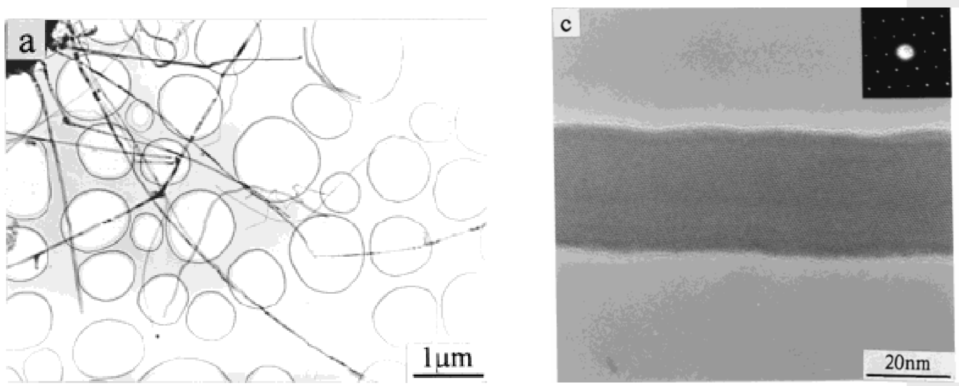
❑ Evaporation and Condensation

product	raw materials	synthesis temp (°C)	carrier gas	diameter (nm)	axis
Si_3N_4 and SiO_2	Si or (Si/SiO_2)	1200−1400	N_2	for Si_3N_4, most are in the range of 15−35 nm. for SiO_2, <300 nm; most <50 nm.	⊥{0001}
SiO_2	Si or (Si/SiO_2)	1100−1300	Ar	most <50 nm	
SiC	SiO_2, nanoscale C particles	1550	Ar	most <50 nm	⊥{200}
Ga_2O_3	Ga	650	N_2 (Ar)	most <50 nm	//(001)
ZnO	Zn	650	N_2 (Ar)	most <50 nm	//[0001]
In	In	1350−1400	Ar	30−40 nm	//(001)

SiC



Si_3N_4



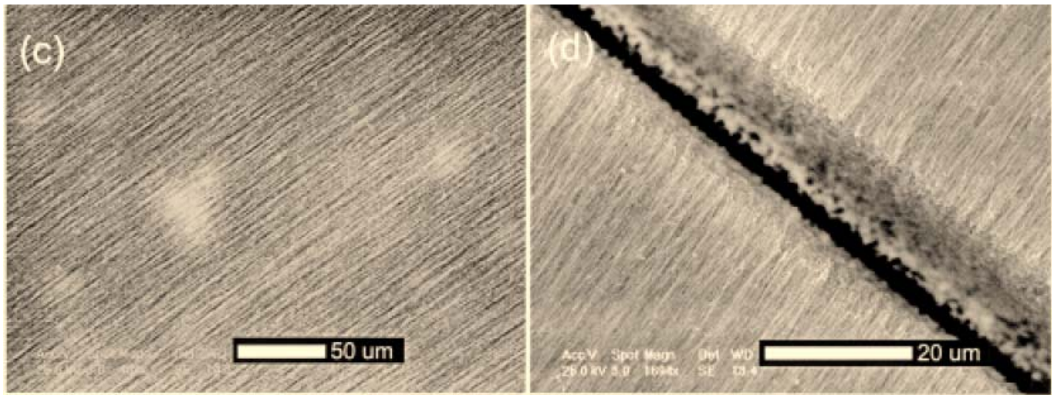
Y. Zhang, Chem. Mater. 14 (2002) 3564.

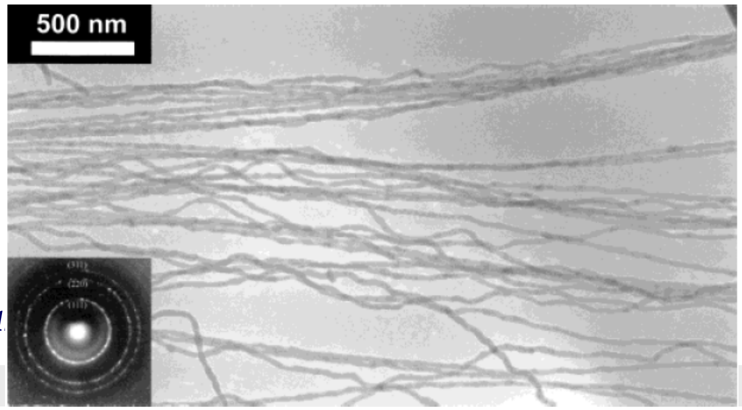
Synthesis of Nano Materials- 1D from gas phase

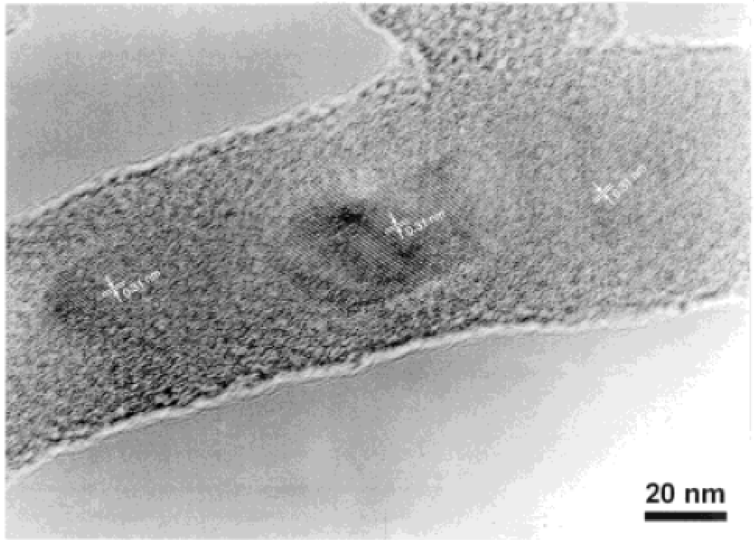
❑ Evaporation and Condensation (reduction)

ex) Si nanowires on Si substrate

- SiO powders, Ar with 5% H_2
- 1300 → 930^{o}C
- core (Si, 20 nm)-shell (SiO_2, 5 nm) structure)





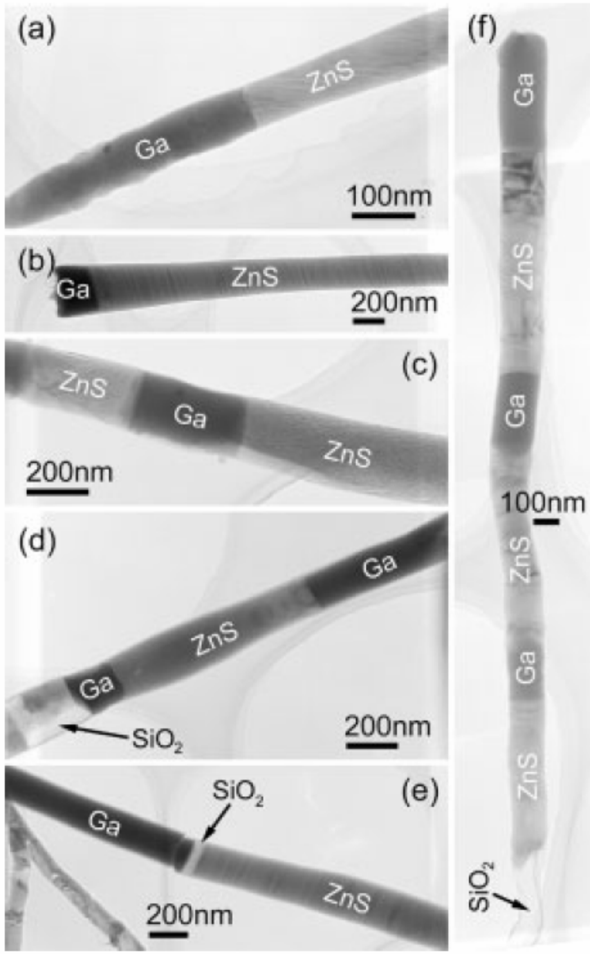


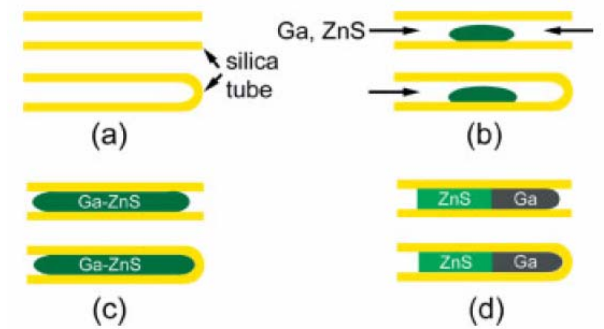
W. Shi, Adv. Mater. 12 (2000) 1343.

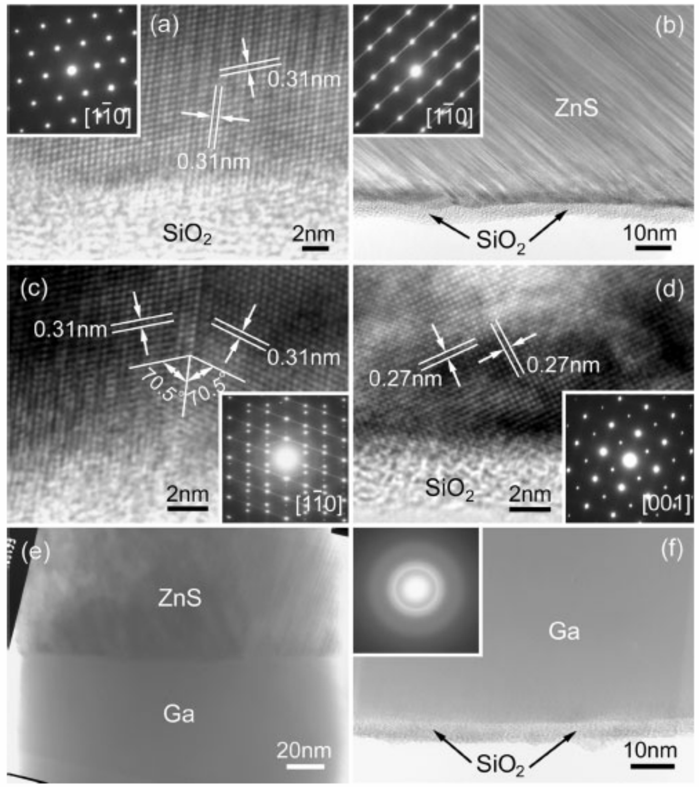
Synthesis of Nano Materials- 1D from gas phase

❑ Evaporation and Condensation (heterojunction)

ex) silica-shielded Ga-ZnS







J. Hu, Adv. Mater. 17 (2005) 1964.

Synthesis of Nano Materials- 1D from gas phase

❑ Vapor-Liquid-Solid (VLS) Growth

- VLS (vapor-liquid-solid) growth mechanism is a typical example of creation of new technology from old technology
- Important milestones in the history of VLS mechanism
  - Observation of 1 dimensional growth in Si wafer
  - Wagner suggests growth mechanism
  - Ceramic whiskers are fabricated by this mechanism
  - Carbon nano tubes are fabricated by this mechanism
  - Semiconductor nanowires are fabricated by this mechanism
  - Nowadays, VLS mechanism is one of the most important and famous technology in nanoscale science

Synthesis of Nano Materials- 1D from gas phase

❑ Vapor-Liquid-Solid (VLS) Growth

- Observation of 1 dimensional growth in Si wafer

- Si whiskers are dislocation free
- Impurities are essential
- Whisker growth is a two step process: fast leader and slow layer growth
- Extension growth occurs by addition of materials at the tips
- Growth direction is <111>
- Liquid like globule are observed

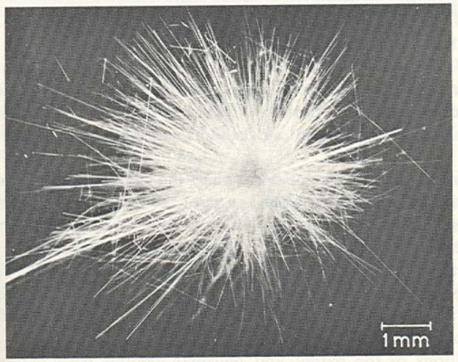
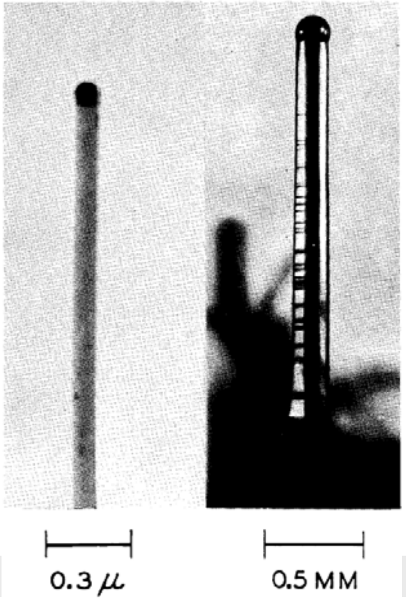


Figure 3.5 Silicon filaments extending radially from a nucleation site.



R.S. Wagner, Appl. Phys. Lett. 4 (1964) 89.
R.S. wagner, J. Appl. Phys. 35 (1964) 2993.

Synthesis of Nano Materials- 1D from gas phase

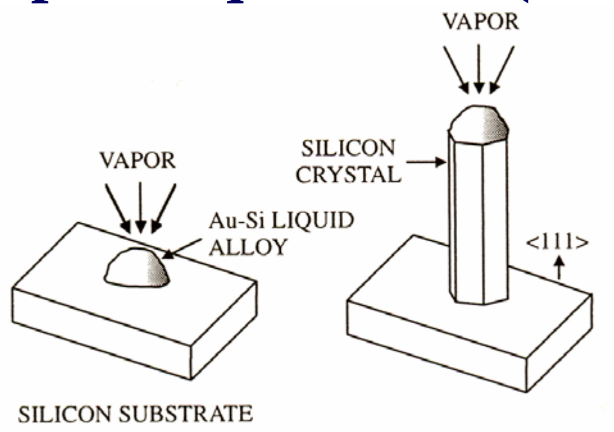
❑ Vapor-Liquid-Solid (VLS) Growth

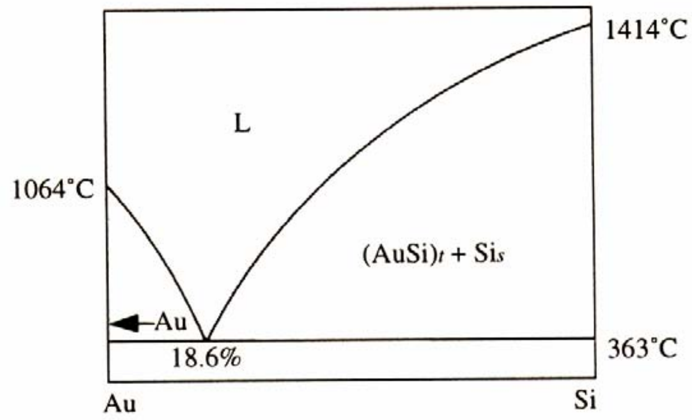
- Wagner suggests growth mechanism

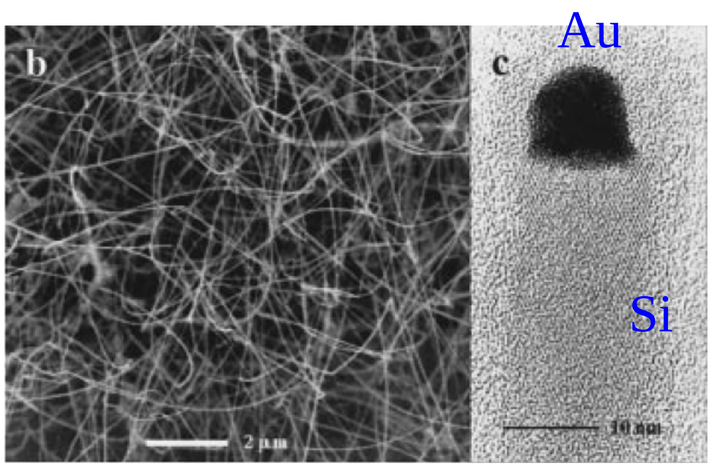
- Catalyst or impurity must form a liquid solution with crystalline materials to be grown at the deposition temperature.
- $K = C_s/C_l < 1$ (distribution coefficient)
- Vapor pressure of catalyst or impurity over the liquid droplet must be very small
- Must be inert chemically
- Interfacial energies play an important role (wetting angle)
- One of the constituents can serve as the catalyst for a compound nanowire growth
- Solid-liquid interface must be well defined crystallographically (single crystal substrate)

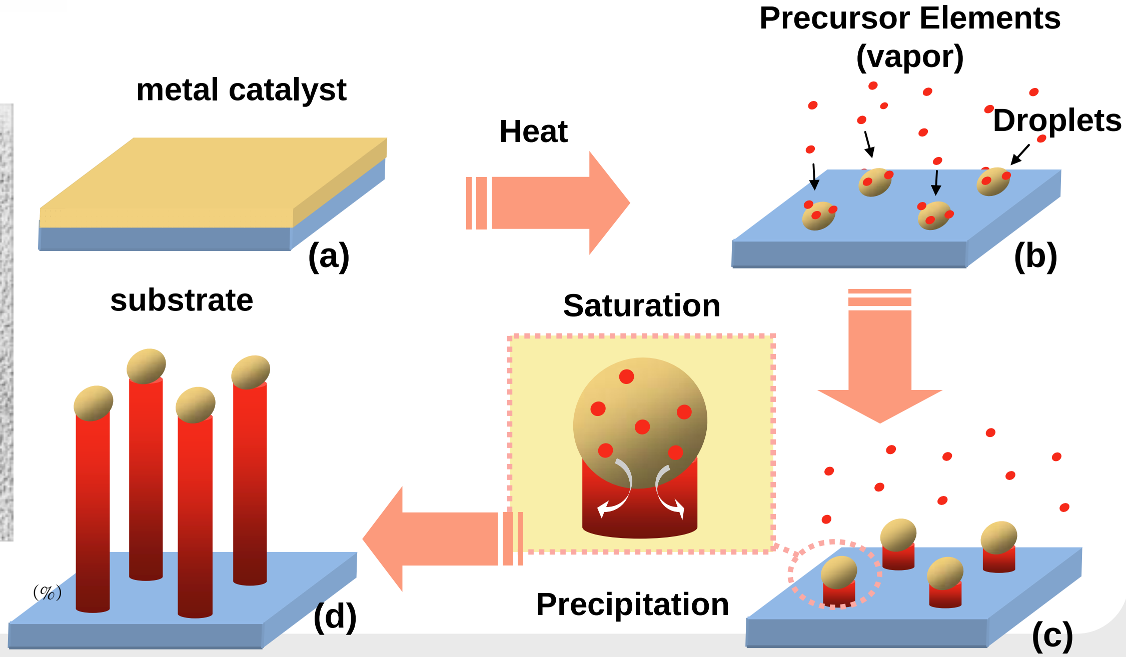
Synthesis of Nano Materials- 1D from gas phase

❑ Vapor-Liquid-Solid (VLS) Growth









J. Hu, Acc. Chem. Res. 32, 435 (1999).

Synthesis of Nano Materials- 1D from gas phase

❑ Vapor-Liquid-Solid (VLS) Growth

- direct observation of VLS

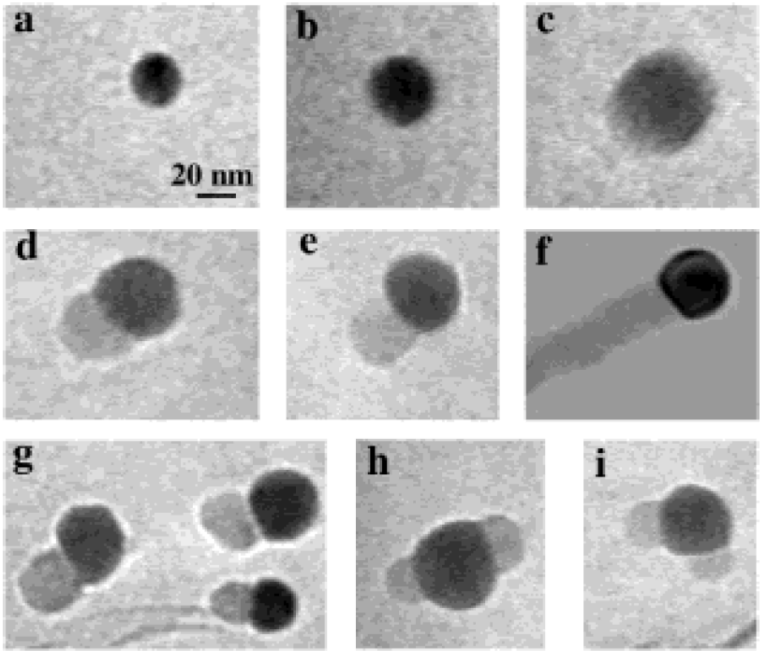


Figure 1. In situ TEM images recorded during the process of nanowire growth. (a) Au nanoclusters in solid state at 500 °C; (b) alloying initiates at 800 °C, at this stage Au exists in mostly solid state; (c) liquid Au/Ge alloy; (d) the nucleation of Ge nanocrystal on the alloy surface; (e) Ge nanocrystal elongates with further Ge condensation and eventually a wire forms (f). (g) Several other examples of Ge nanowire nucleation, (h,i) TEM images showing two nucleation events on single alloy droplet.

Ge nanowire
via Au

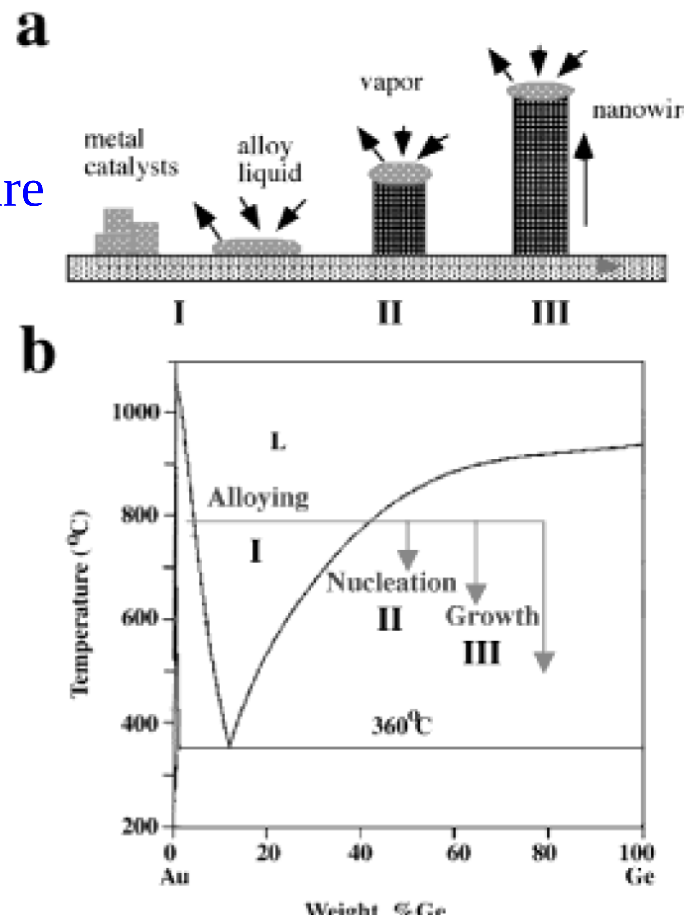


Figure 2. (a) Schematic illustration of vapor−liquid−solid nanowire growth mechanism including three stages (I) alloying, (II) nucleation, and (III) axial growth. The three stages are projected onto the conventional Au−Ge binary phase diagram (b) to show the compositional and phase evolution during the nanowire growth process.

Y. Wu, J. Am. Chem. Soc. 13 (2001) 3165.

Synthesis of Nano Materials- 1D from gas phase

❑ Vapor-Liquid-Solid (VLS) Growth- growth rate

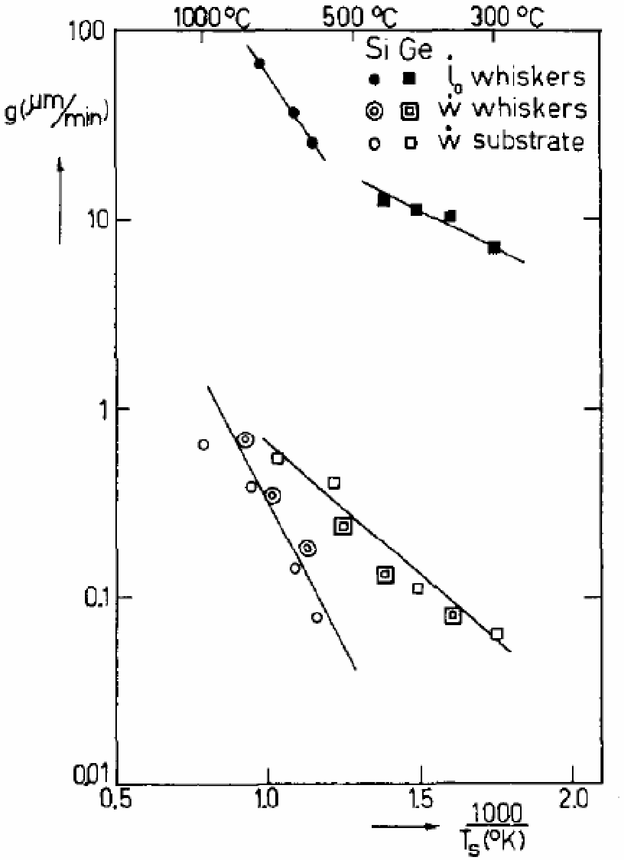
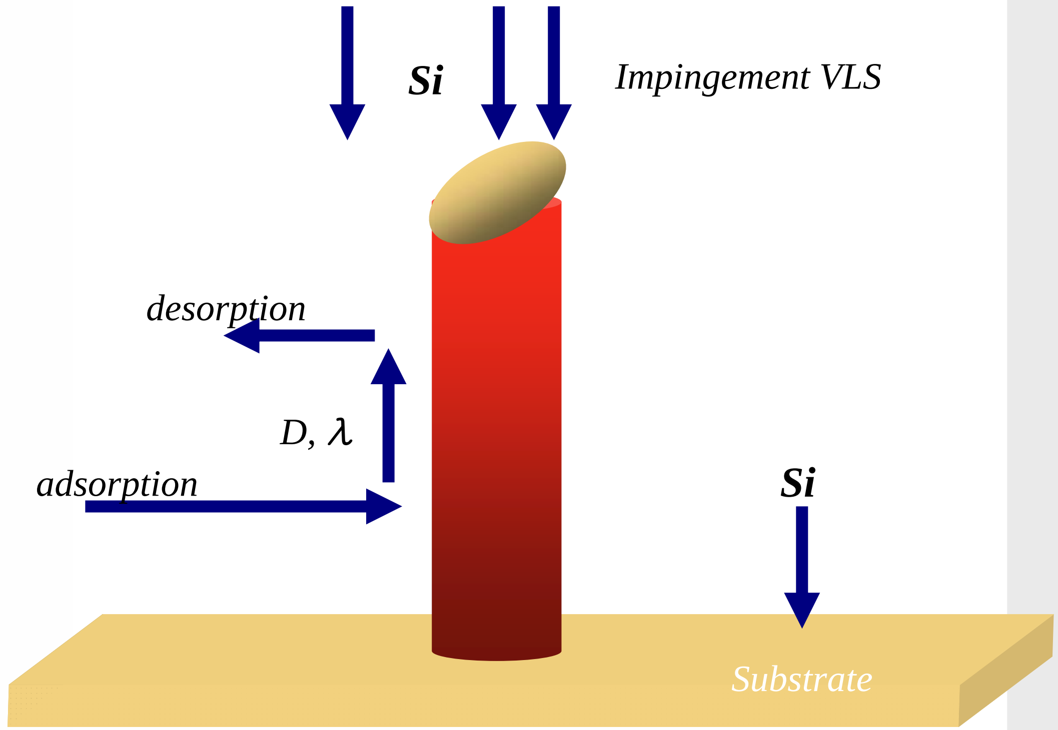


Fig. 9. Arrhenius plots of the growth rates, g, of silicon and germanium; for conditions with H_2, and SiH_4 or GeH_4 at a pressure of 10 Torr.



Axial growth rate: V=I(VLS) +4λ/d thL/λ

G.A. Bootsma, J. Cryst. Growth 10 (1971) 223.

Synthesis of Nano Materials- 1D from gas phase

❑ Vapor-Liquid-Solid (VLS) Growth- size of nanowire

- size of nanowire ← size of liquid droplet
- thin catalyst layer
- colloids

size of liquid droplet← film thickness

Ex) Si-Au

10 nm Au → 150 nm in diameter

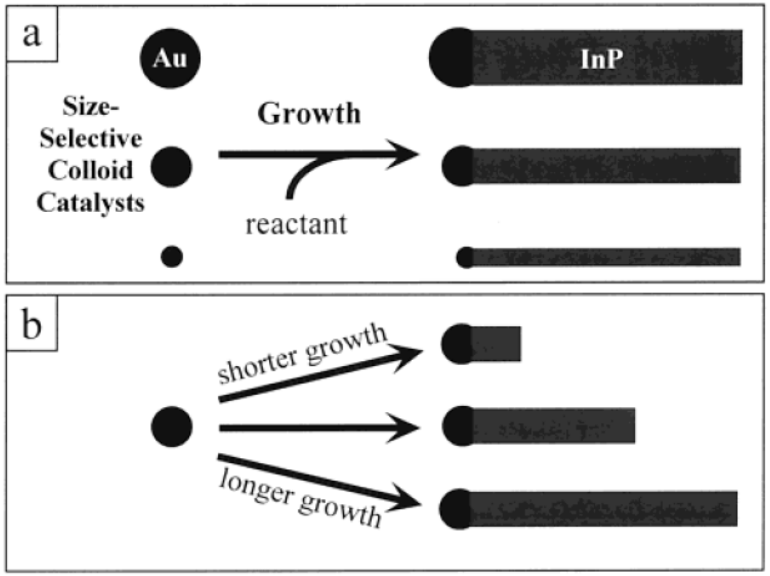
5 nm → 80 nm Ge nanowire

limitation

$$r_{\min} = \frac{2\gamma_{LV} V_m}{RT \ln \alpha} \quad \alpha: \text{supersaturation}$$

high $\alpha \rightarrow$ lateral growth

radial size instability



8.4 nm Au → 11.4 nm GaP

18.5 nm → 20 nm

28.2 nm → 30.2 nm

M.S. Gudiksen, J. Phys. Chem. B105 (2001) 4062.

J. Am. Chem. Soc. 122 (2000) 8801.

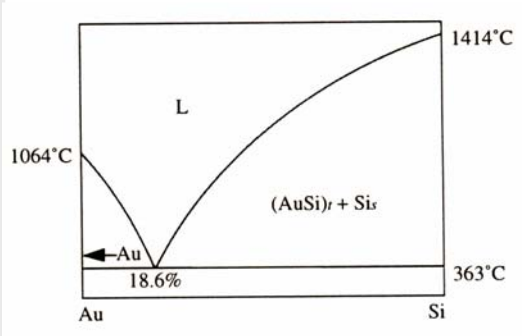
Y. Wu, Chem Mater, 12 (2000) 605.

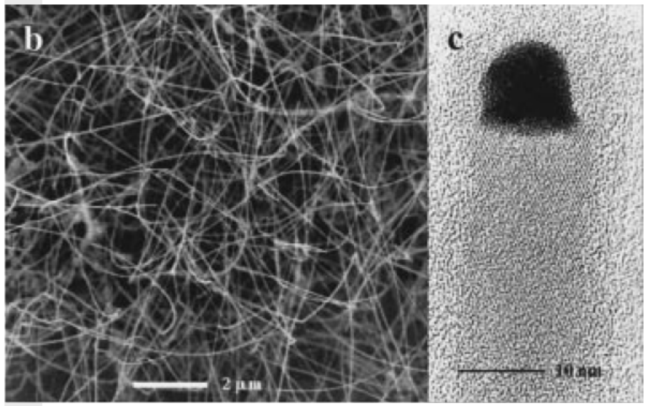
Synthesis of Nano Materials- 1D from gas phase

❑ Vapor-Liquid-Solid (VLS) Growth- catalyst

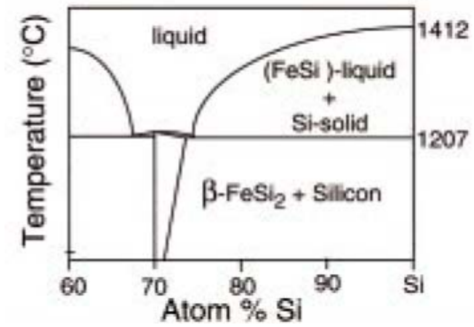
- ex) Si

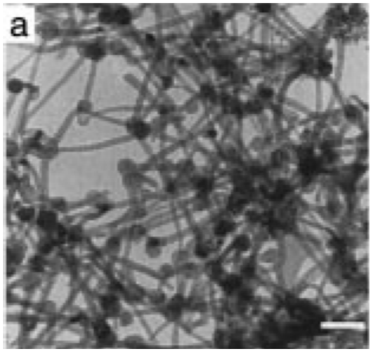
i) Au

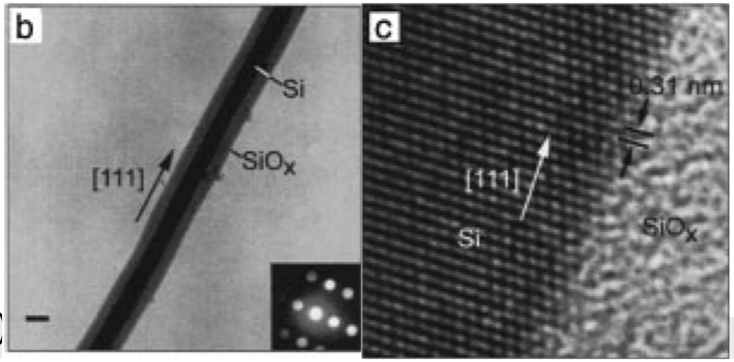




ii) Fe







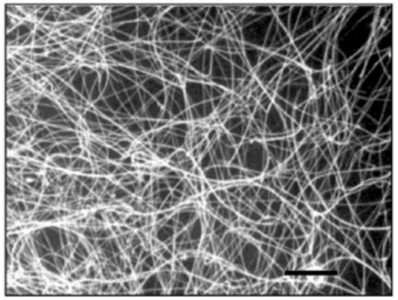
J. Hu, Acc. Chem. Res. 32, 435 (1999)

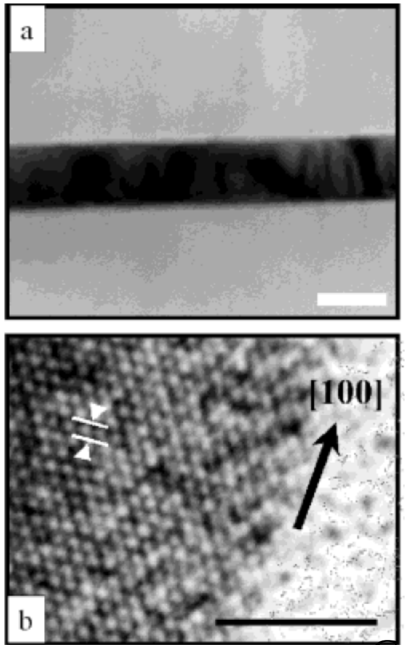
Synthesis of Nano Materials- 1D from gas phase

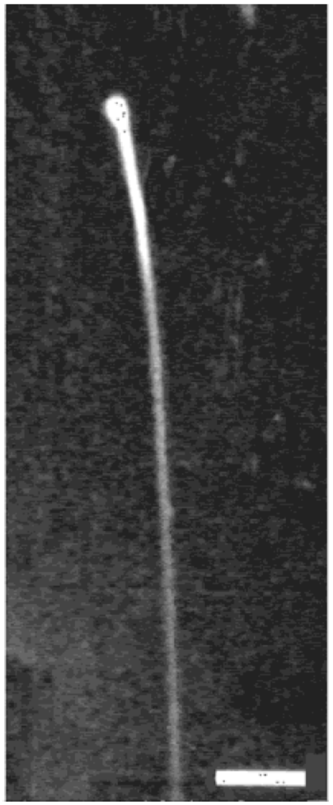
❑ Vapor-Liquid-Solid (VLS) Growth- catalyst

- ex) GaN

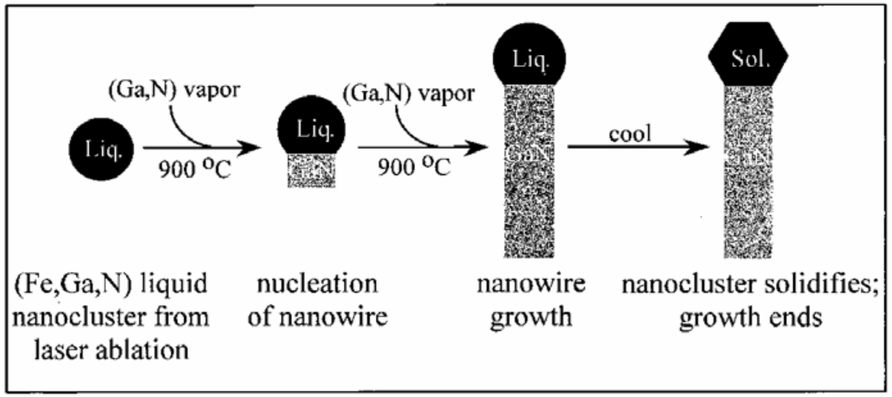
i) In

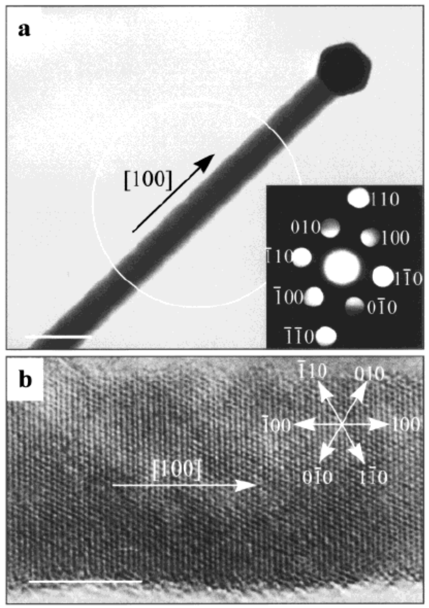






ii) laser assisted (Fe)





C.C. Chen, Adv. Mater. 12 (2000)738. X. Duan, J. Am. Chem Soc. 122 (2000) 188.

Synthesis of Nano Materials- 1D from gas phase

❑ Vapor-Liquid-Solid (VLS) Growth- catalyst

- ex) GaN

iii) NiO, FeO

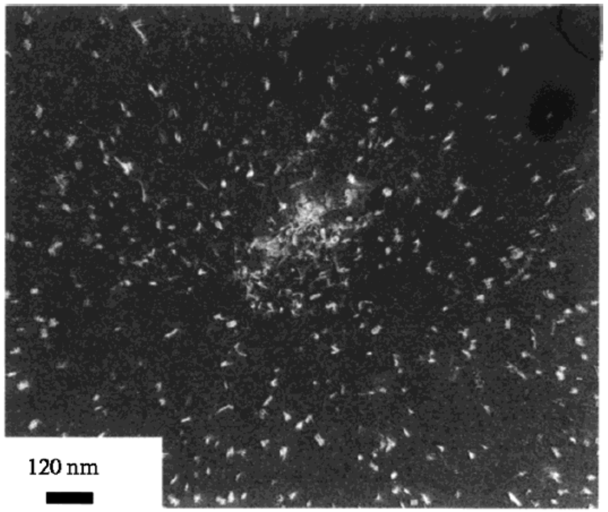


Fig. 1. SEM image of NiO particles distributed on the $LaAlO_3$ substrate.

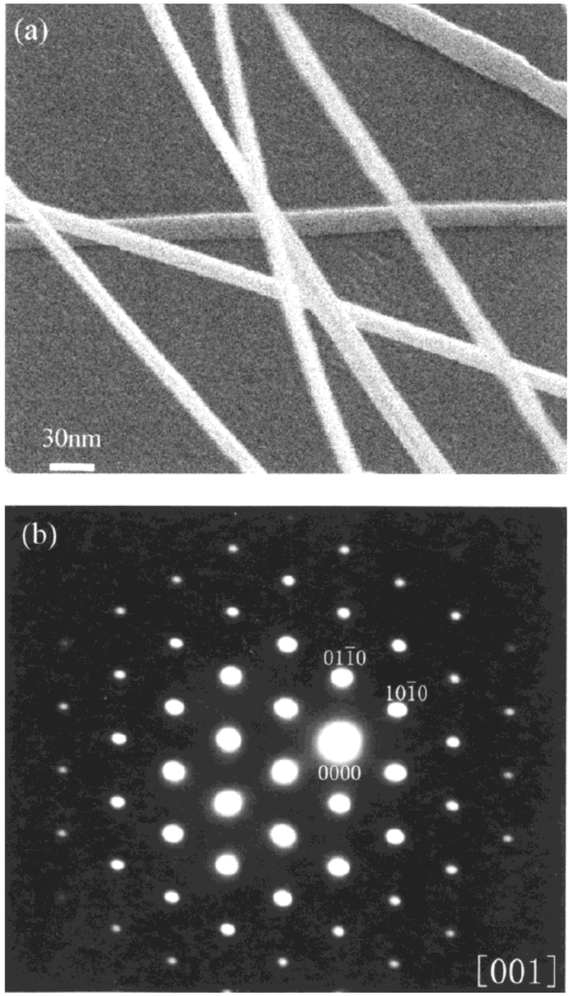


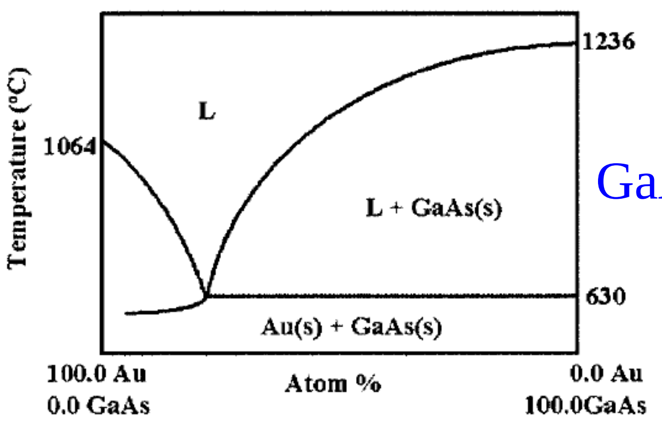
Fig. 3. a) SEM image of the straight and smooth GaN nanowires. b) Typical SAED pattern of a grown GaN nanowire taken along [001].

X. Chen, Adv. Mater. 1432 (2000)738.

Synthesis of Nano Materials- 1D from gas phase

❑ Vapor-Liquid-Solid (VLS) Growth

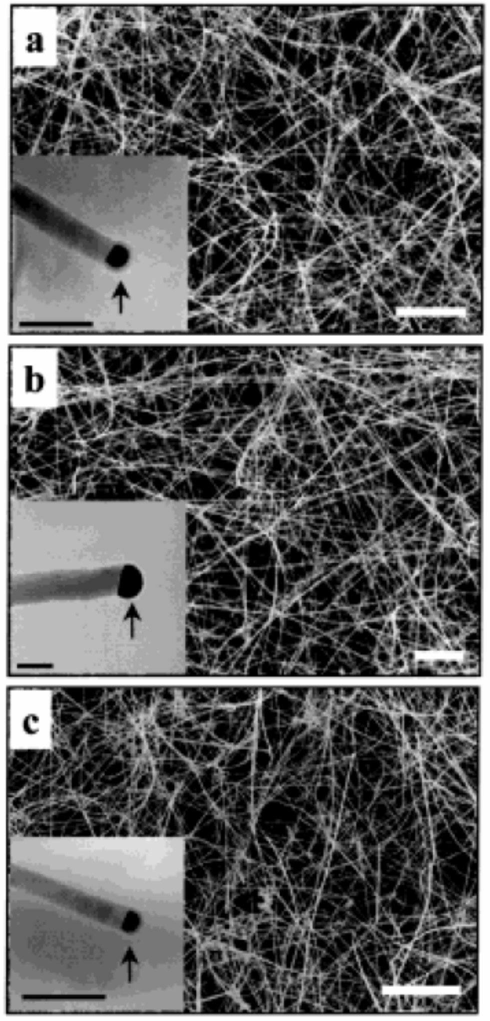
- ex) Compound Semiconductors

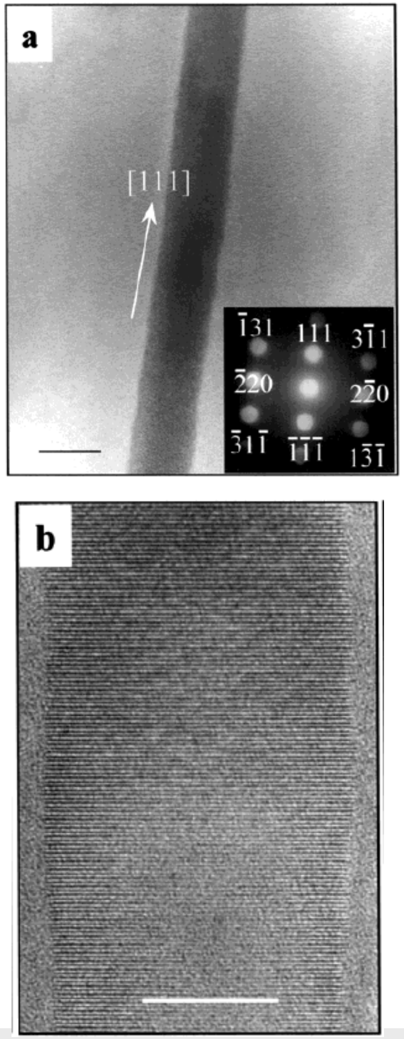


GaAs

GaP

$GaAs_{0.6}P_{0.4}$





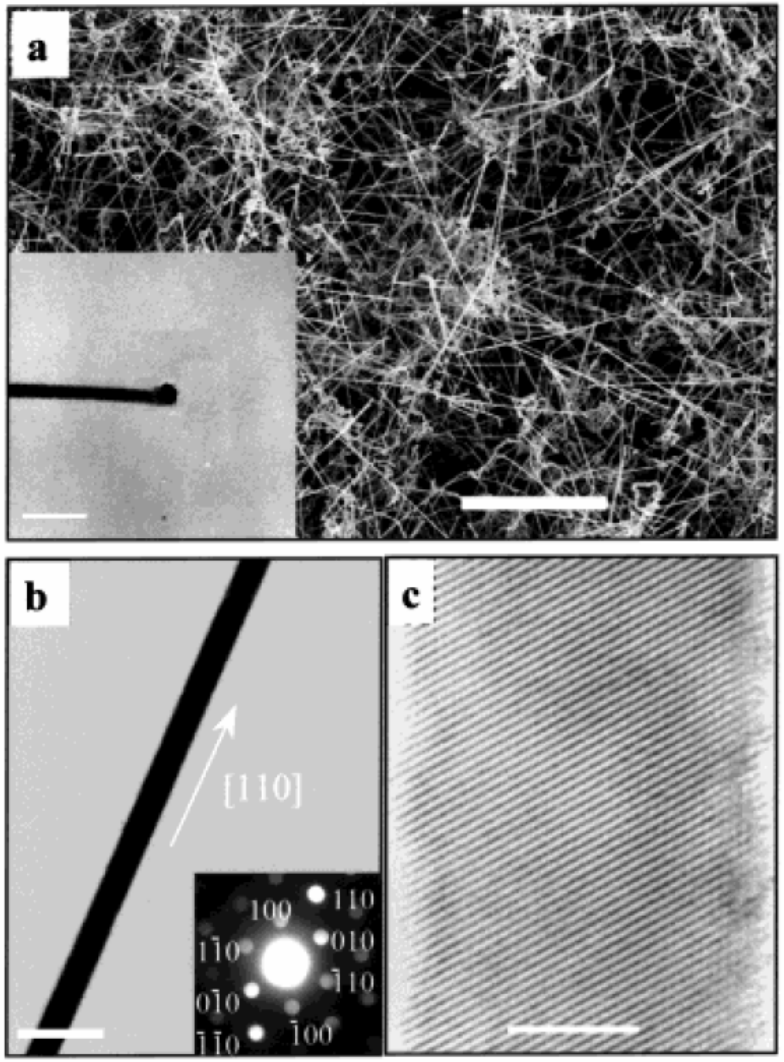
GaAs

X. Duan, Adv. Mater. 12 (2000)298.

Synthesis of Nano Materials- 1D from gas phase

❑ Vapor-Liquid-Solid (VLS) Growth

- ex) Compound Semiconductors



CdSe

Material	Growth Temperature [°C]	Minimum Diameter [nm]	Average Diameter [nm]	Structure	Growth Direction	Ratio of Components
GaAs	800–1030	3	19	ZB	<111>	1.00 : 0.97
GaP	870–900	3–5	26	ZB	<111>	1.00 : 0.98
$GaAs_{0.6}P_{0.4}$	800–900	4	18	ZB	<111>	1.00 : 0.58 : 0.41
InP	790–830	3–5	25	ZB	<111>	1.00 : 0.98
InAs	700–800	3–5	11	ZB	<111>	1.00 : 1.19
$InAs_{0.5}P_{0.5}$	780–900	3–5	20	ZB	<111>	1.00 : 0.51 : 0.51
ZnS	990–1050	4–6	30	ZB	<111>	1.00 : 1.08
ZnSe	900–950	3–5	19	ZB	<111>	1.00 : 1.01
CdS	790–870	3–5	20	W	<100>, <002>	1.00 : 1.04
CdSe	680–1000	3–5	16	W	<110>	1.00 : 0.99
$Si_{1-x}Ge_x$	820–1150	3–5	18	D	<111>	$Si_{1-x}Ge_x$

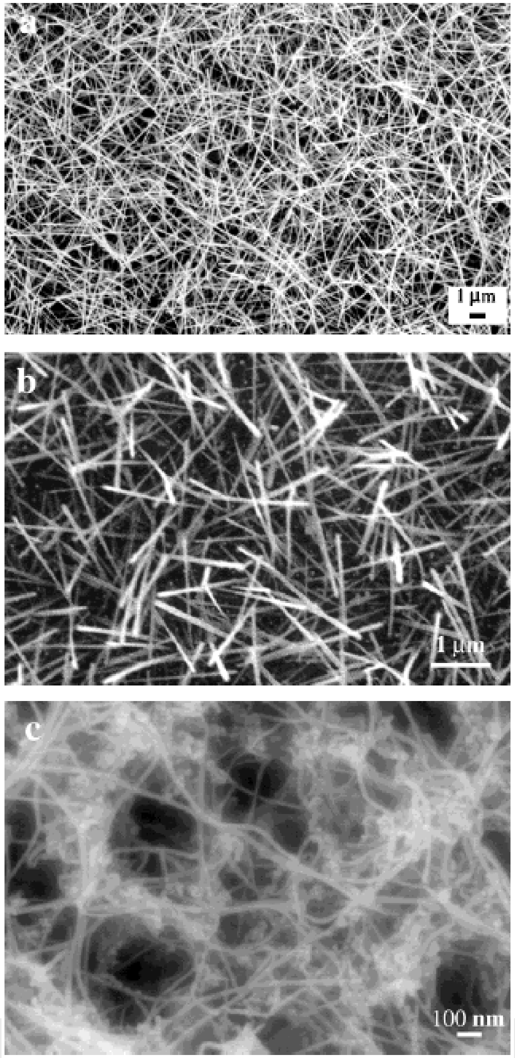
GaAs- Au, Ag, Cu catalyst
Rest-Au catalyst

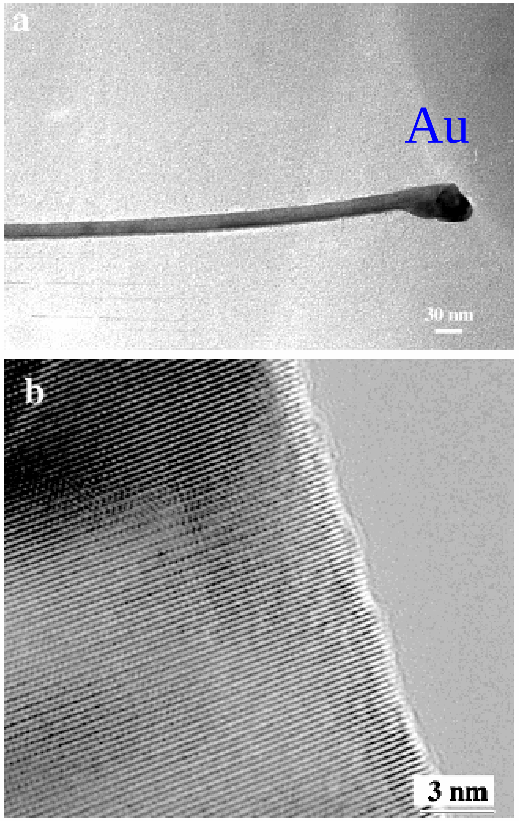
X. Duan, Adv. Mater. 12 (2000)298.

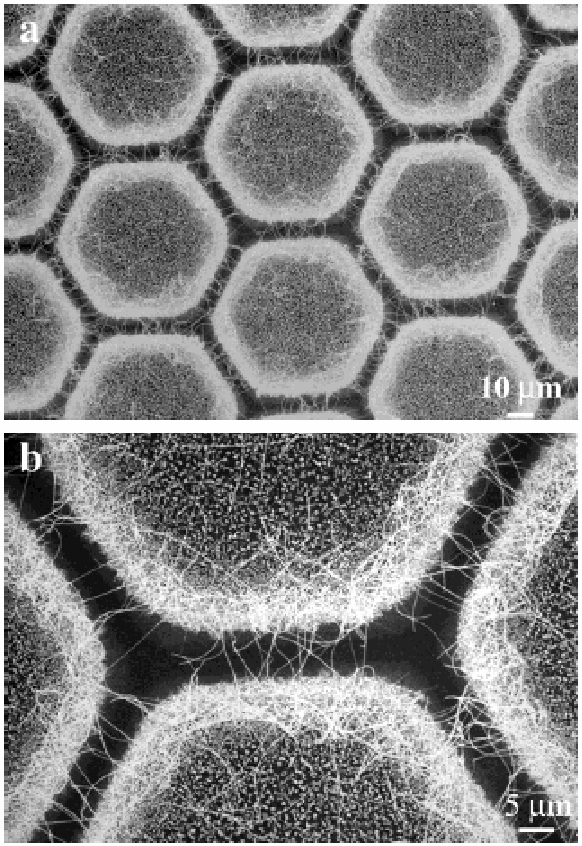
Synthesis of Nano Materials- 1D from gas phase

❑ Vapor-Liquid-Solid (VLS) Growth

- ex) ZnO





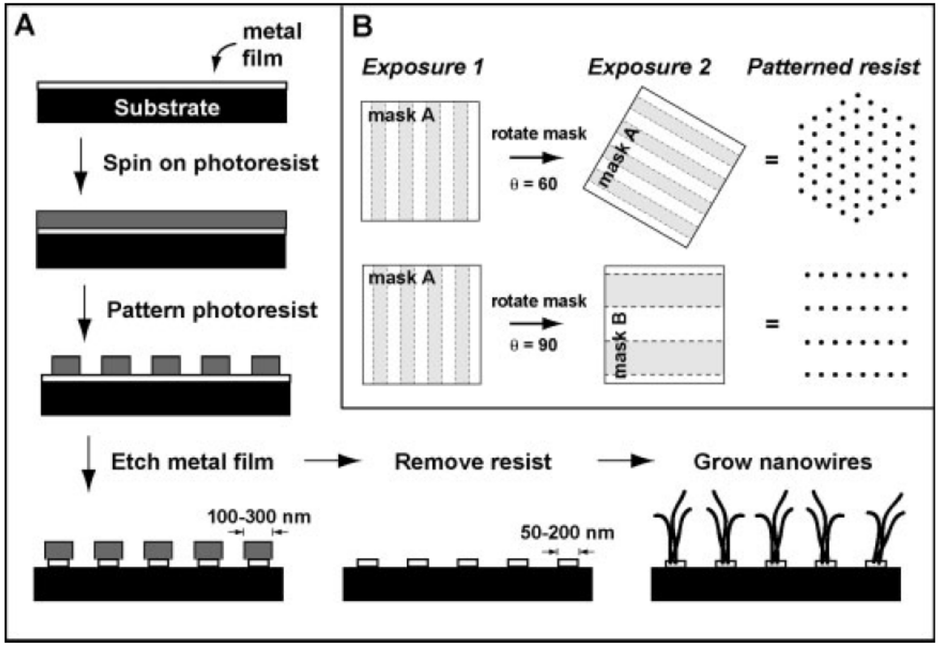


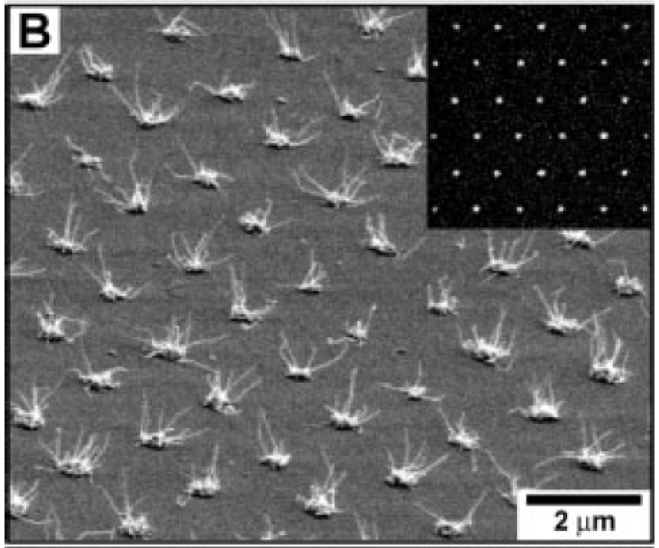
M.H. Huang, Adv. Mater. 13 (2001)113.

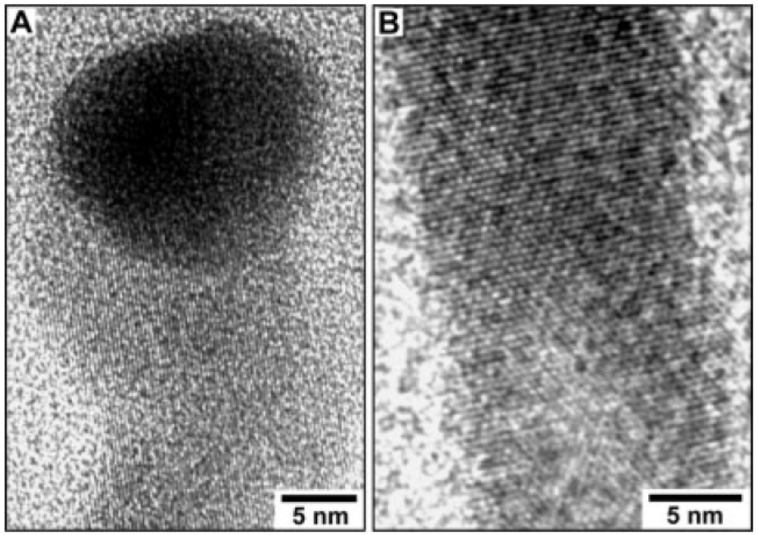
Synthesis of Nano Materials- 1D from gas phase

❑ Vapor-Liquid-Solid (VLS) Growth

- ex) ZnO







E.C. Greyson, Adv. Mater. 16 (2004)1348.

Synthesis of Nano Materials- 1D from gas phase

❑ Vapor-Liquid-Solid (VLS) Growth- vertical growth

- ex) ZnO

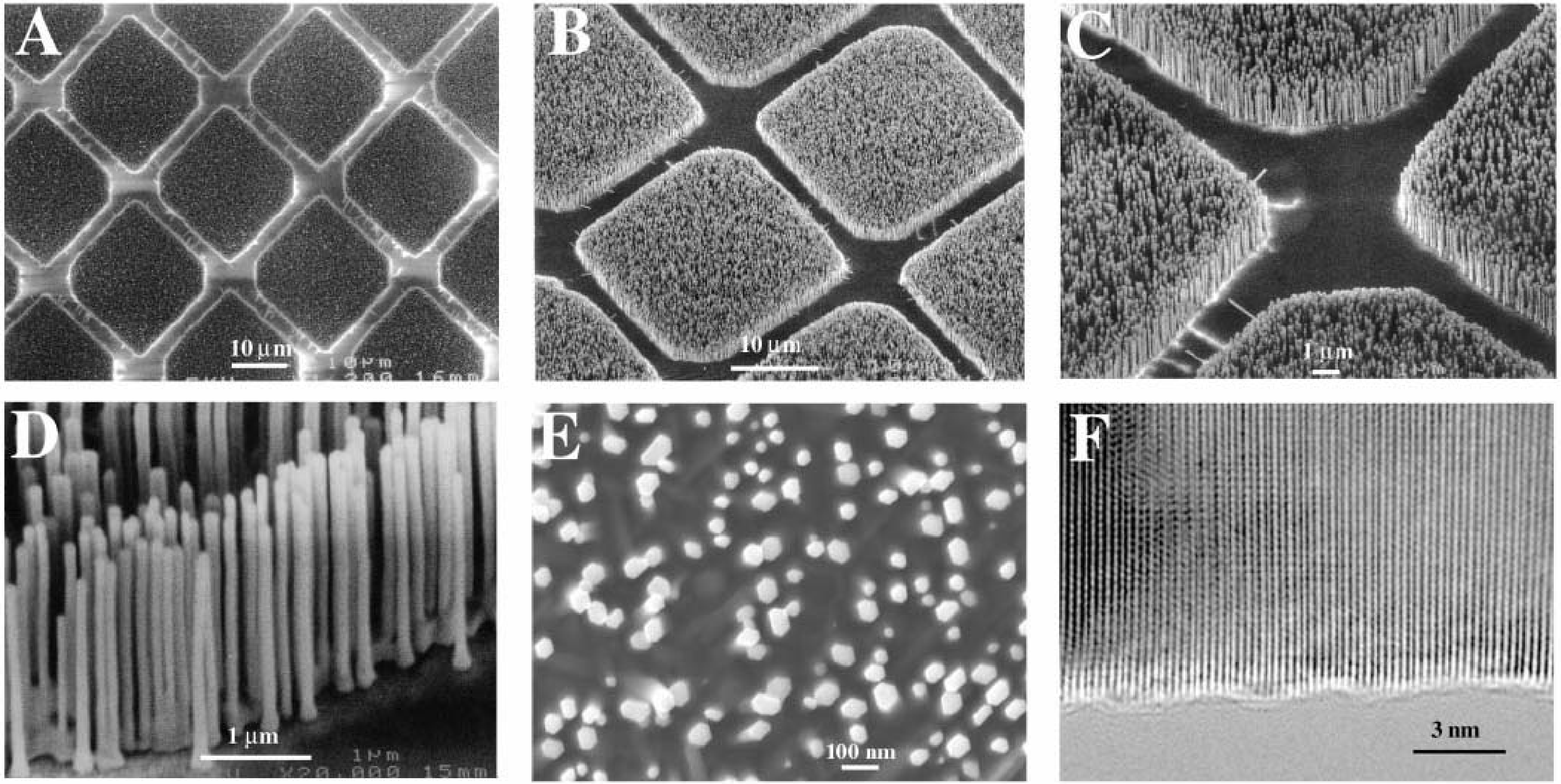


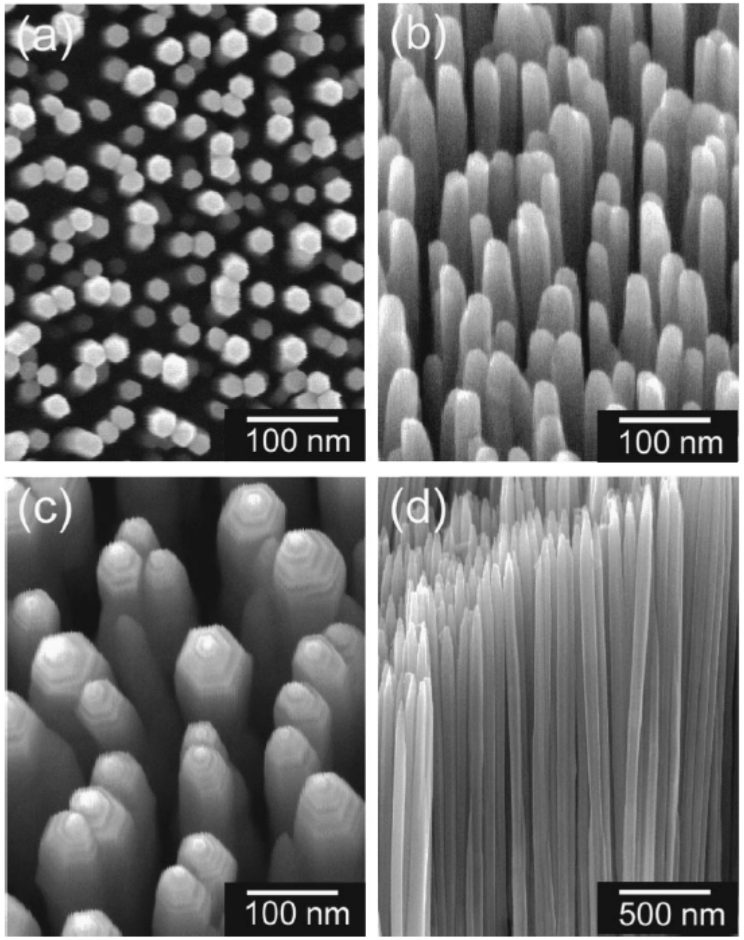
Fig. 1. (A through E) SEM images of ZnO nanowire arrays grown on sapphire substrates. A top view of the well-faceted hexagonal nanowire tips is shown in (E). (F) High-resolution TEM image of an individual ZnO nanowire showing its <0001> growth direction. For the nanowire growth, clean (110) sapphire substrates were coated with a 10 to 35 Å thick layer of Au, with or without using TEM grids as shadow masks (micro contact printing of thiols on Au followed by selective etching has also been used to create the Au pattern). An equal amount of ZnO powder and graphite powder were ground and transferred to an alumina boat. The Au-coated sapphire substrates were typically placed 0.5 to 2.5 cm from the center of the boat. The starting materials and the substrates were then heated up to 880° to 905°C in an Ar flow. Zn vapor is generated by carbothermal reduction of ZnO and transported to the substrates where ZnO nanowires grow. The growth generally took place within 2 to 10 min (*15*).

M. Huang, Science, 292 (2001) 1897.

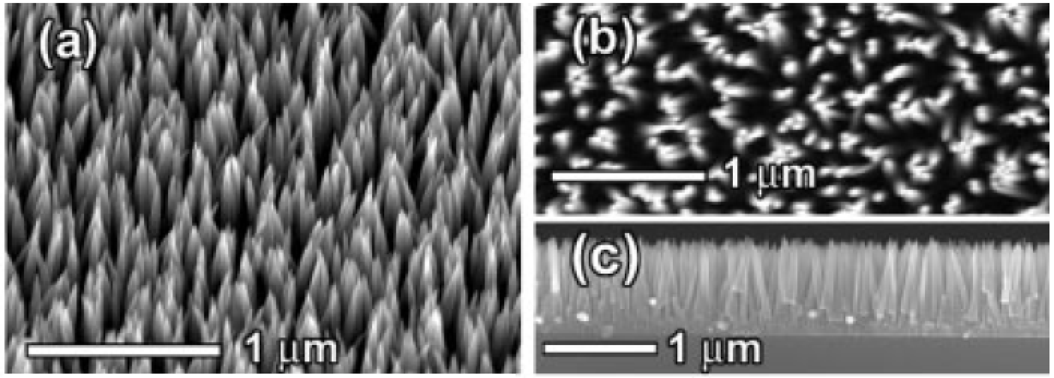
Synthesis of Nano Materials- 1D from gas phase

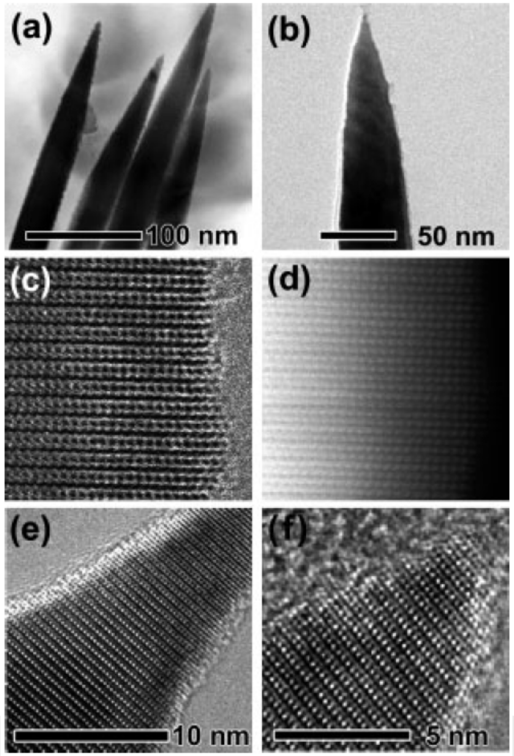
❑ Vapor-Liquid-Solid (VLS) Growth- vertical growth

- ex) ZnO (noncatalyst)



W.I. Park, Appl. Phys. Lett. 80 4232 (2002).





W.I. Park, Adv. Mater. 14 (2002) 1841.

Synthesis of Nano Materials- 1D from gas phase

❑ Vapor-Liquid-Solid (VLS) Growth- selective growth

- ex) InAs (electron beam lithography)

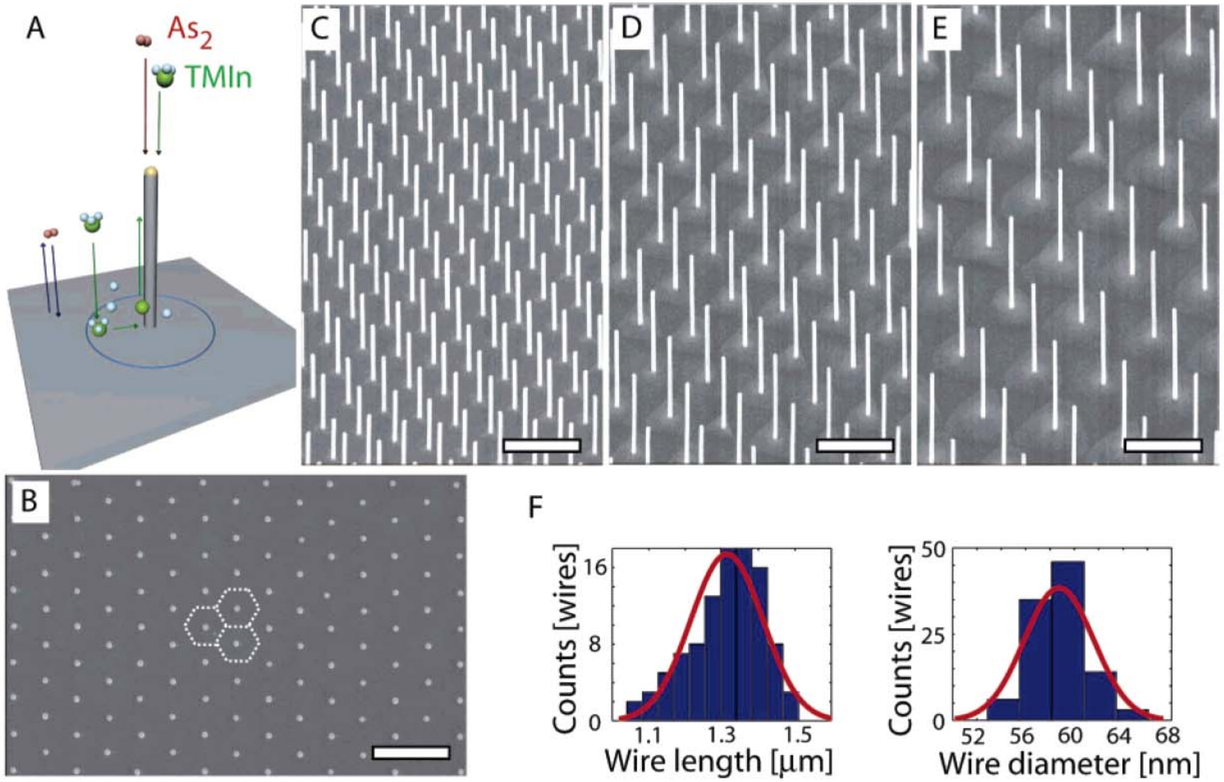


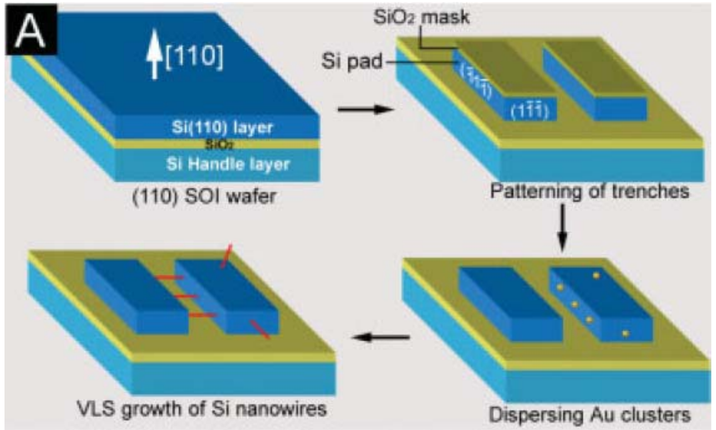
Figure 1. InAs nanowire growth from arrays of Au disks. (A) Schematics of precursor transport to the growth point. In can contribute to growth via direct impingement or via diffusion from the maximum surface collection area (marked by a blue circle). As, on the other hand, can only contribute via direct impingement. (B) Top down view of positioned Au particles, defined by EBL. The dotted areas indicate the available surface collection area. (C−E) Tilted views of nanowire arrays with the same diameter but with an interwire distance of 0.5 μm, 0.75 μm, and 1 μm. As the interwire distance increases, the wire growth rate increases. Small triangular pyramids are visible at the base of the wires, which consume some material but are of subordinate interest to the wire growth, since no relation between pyramid sizes and wire lengths could be determined. (F) Histograms of wire diameter and wire length. Red curves are fits to the data. All scale bars are 1 μm.

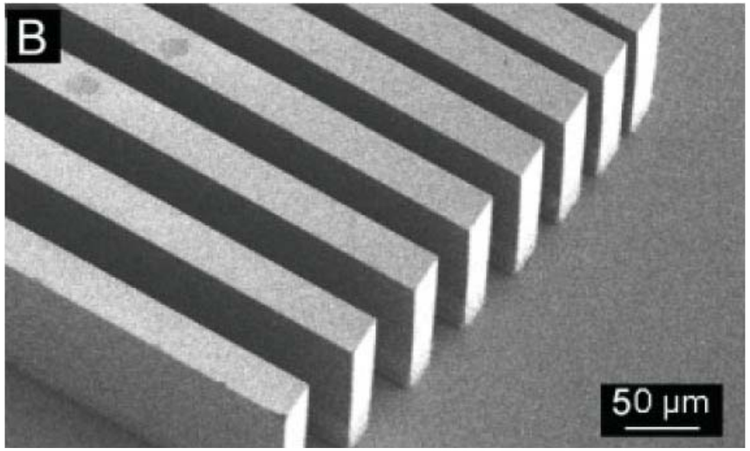
L. E. Jensen, Nano Lett. 4 (2004)1961.

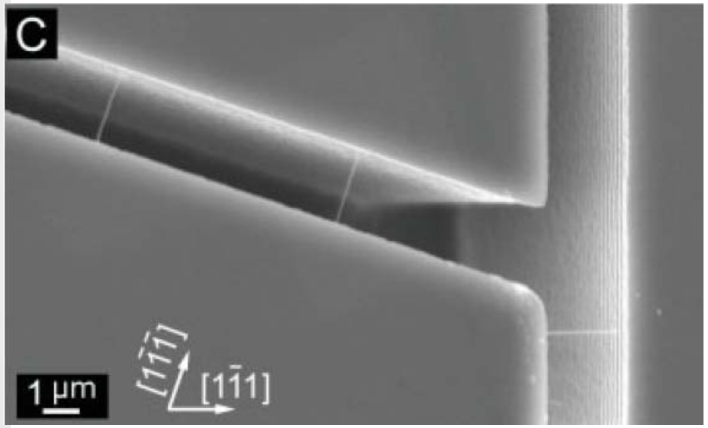
Synthesis of Nano Materials- 1D from gas phase

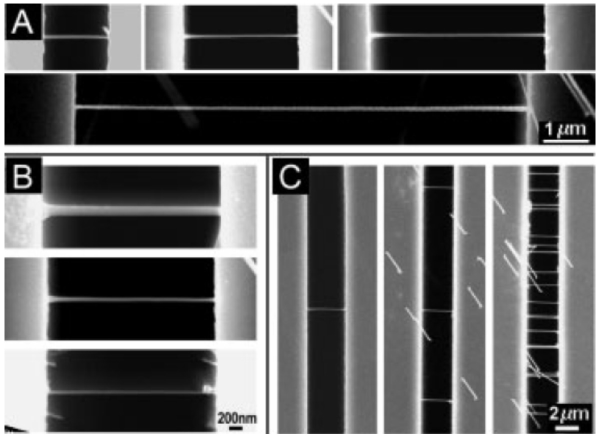
❑ Vapor-Liquid-Solid (VLS) Growth- lateral growth

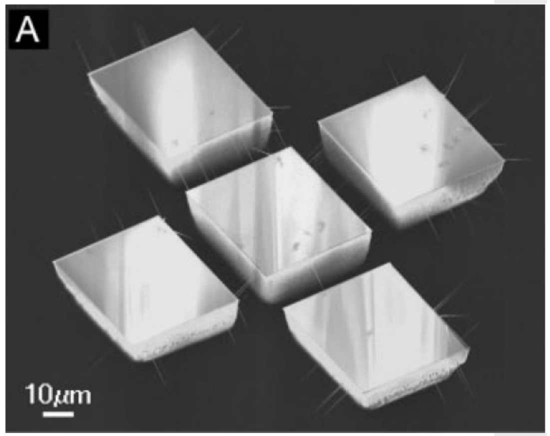
- ex) Si









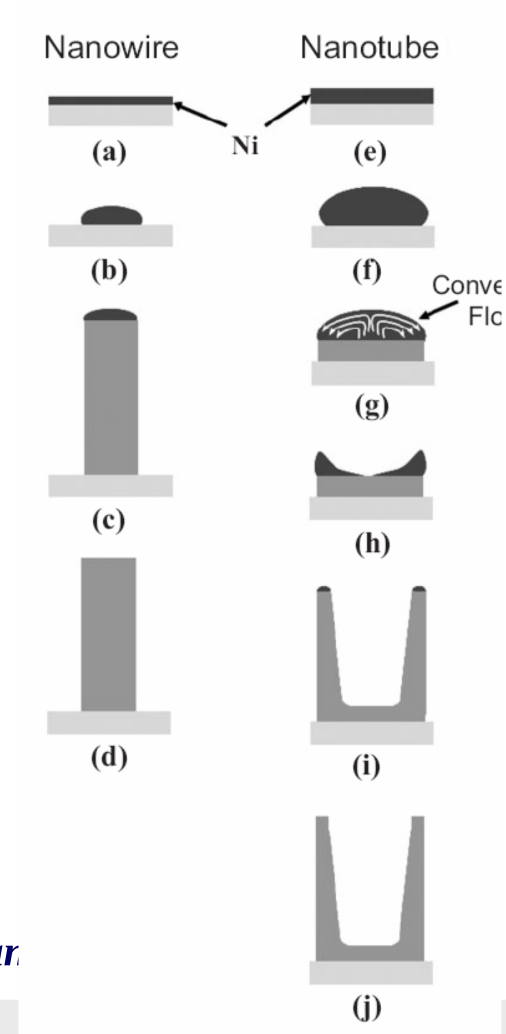


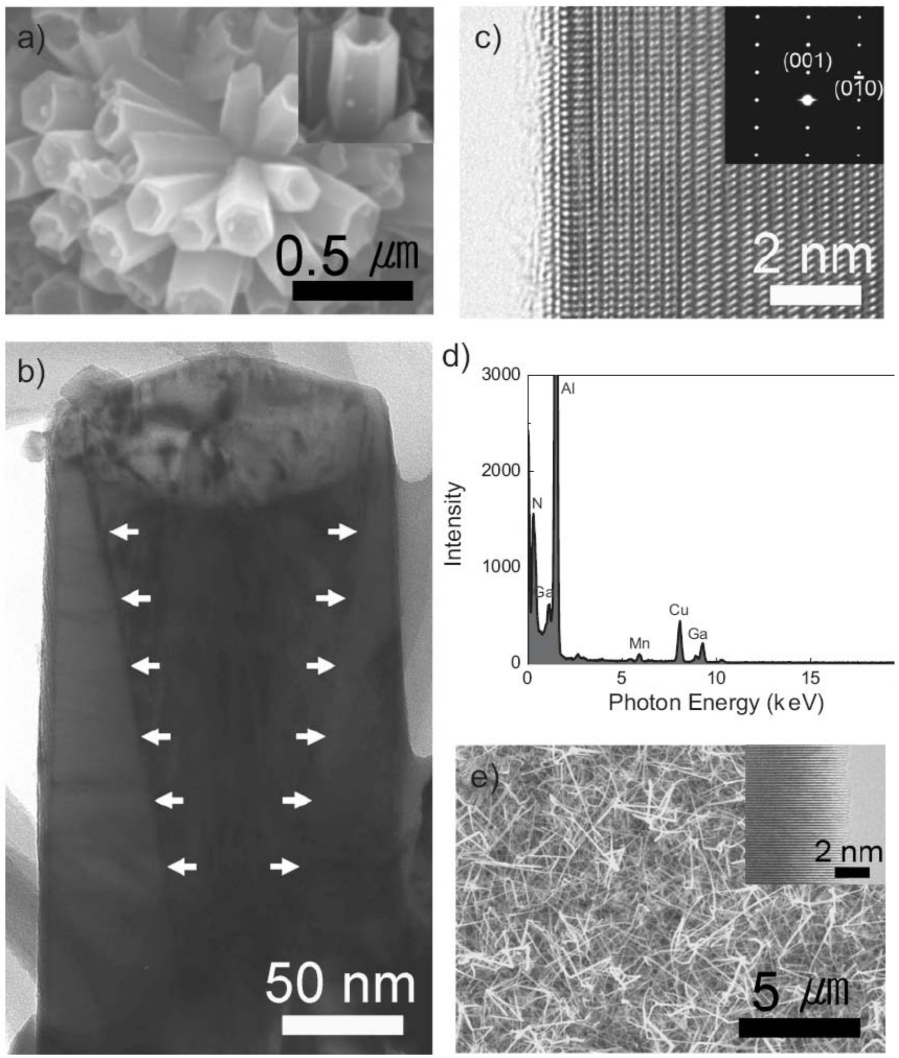
R. He, Adv. Mater. 17 (2005) 2098.

Synthesis of Nano Materials- 1D from gas phase

❑ Vapor-Liquid-Solid (VLS) Growth- nanotube

- ex) AlGaN:Mn



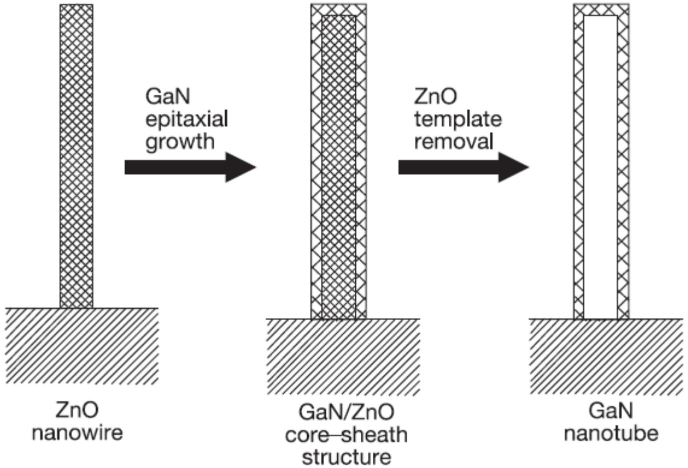


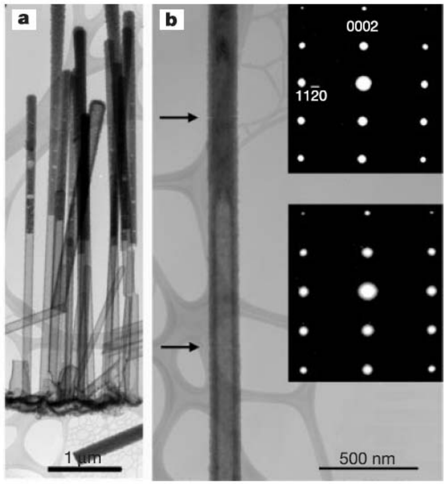
H. K. Seong, Adv. Mater 18 (2006)3019.

Synthesis of Nano Materials- 1D from gas phase

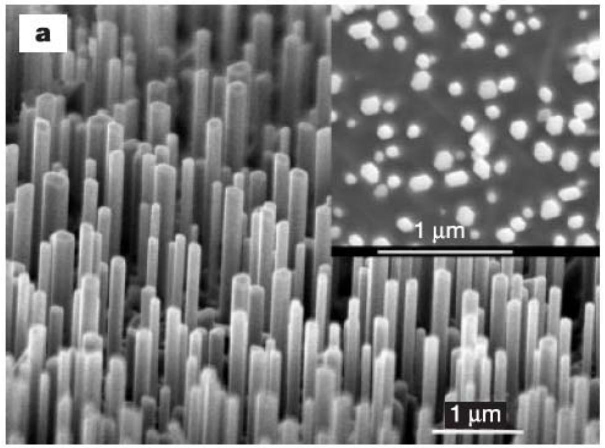
❑ Vapor-Liquid-Solid (VLS) Growth- nanotube

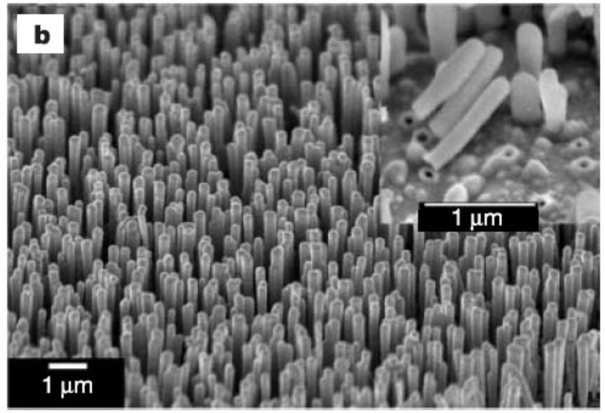
- ex) GaN



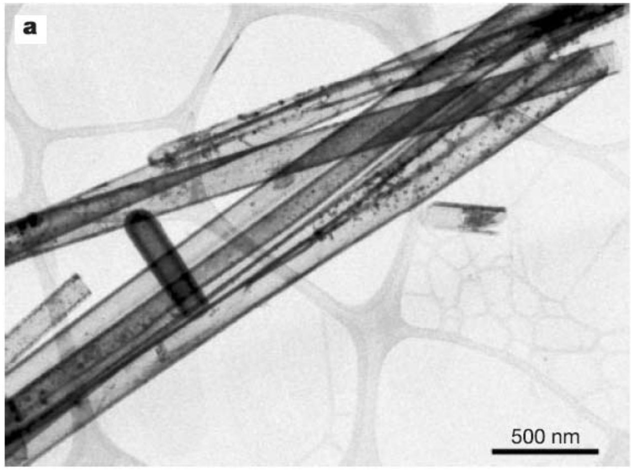


ZnO





GaN

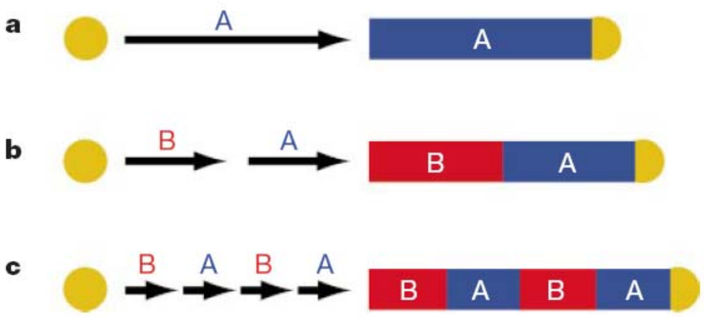


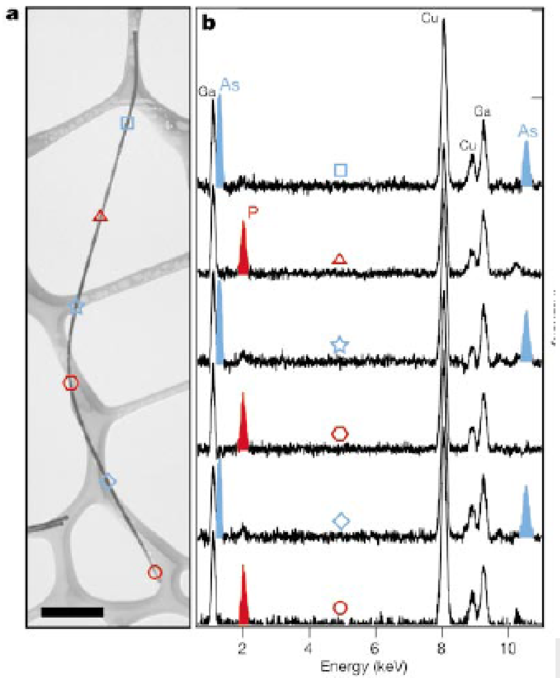
J. Goldberger, Nature 422 (2003)599.

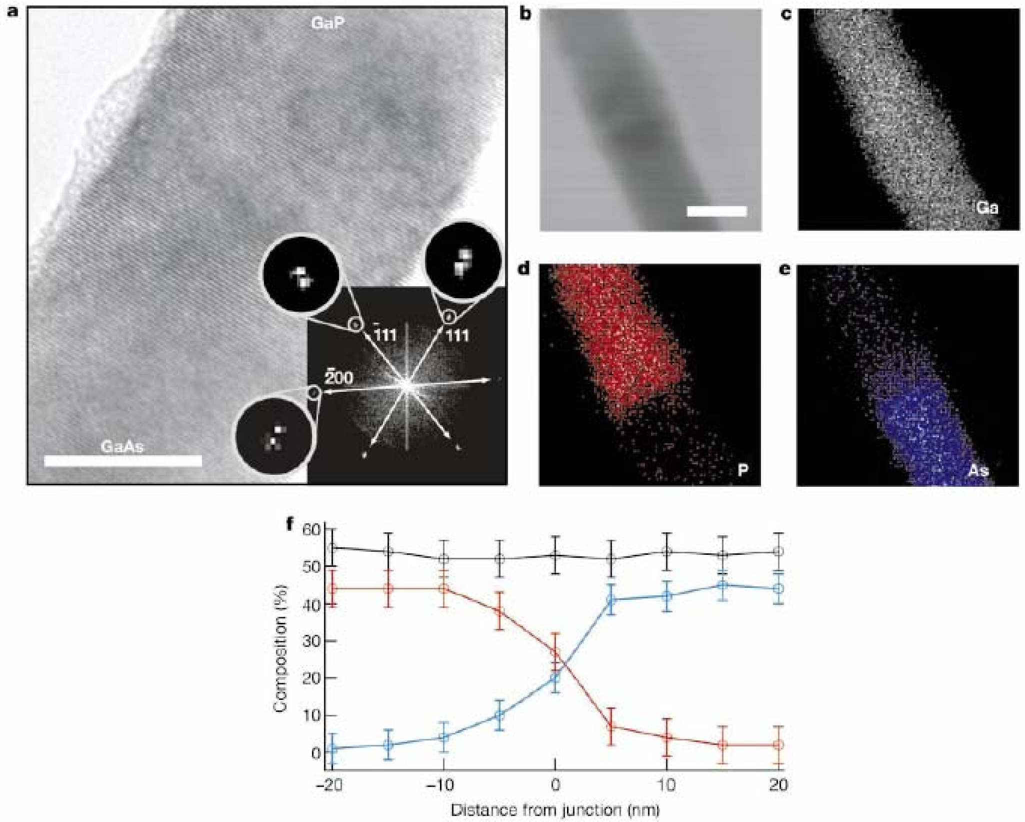
Synthesis of Nano Materials- 1D from gas phase

❑ Vapor-Liquid-Solid (VLS) Growth- superlattice

- ex) GaAs/GaP





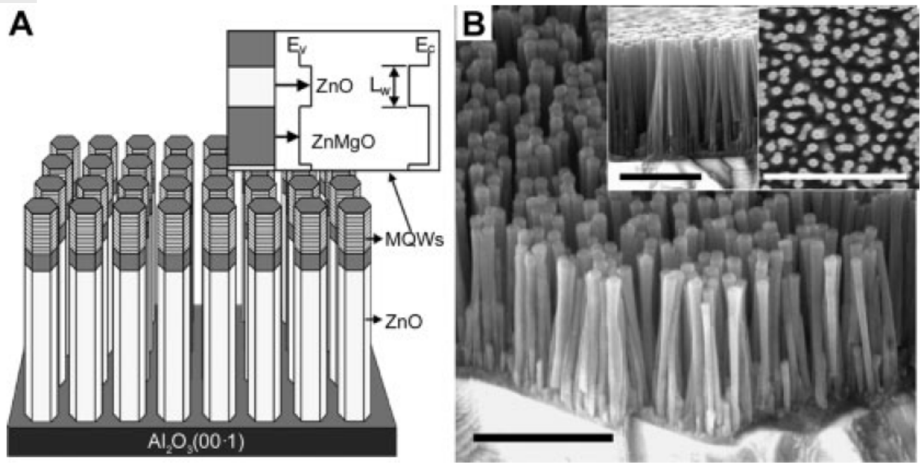


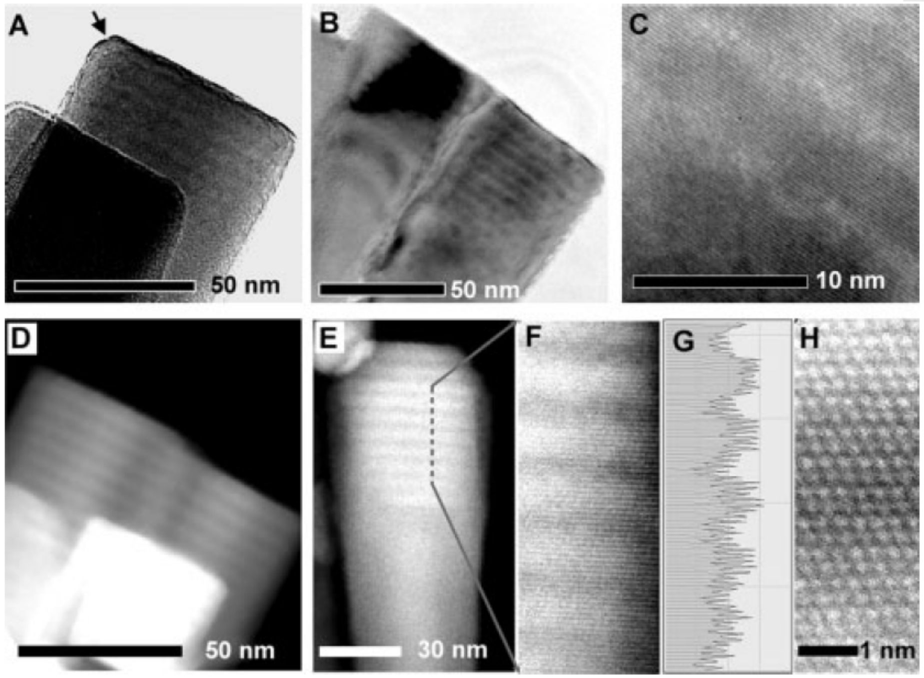
M.S. Gudiksen , Nature 415 (2002) 617.

Synthesis of Nano Materials- 1D from gas phase

❑ Vapor-Liquid-Solid (VLS) Growth- heterostructure

- ex) ZnO/ZnMgO (noncatalyst)



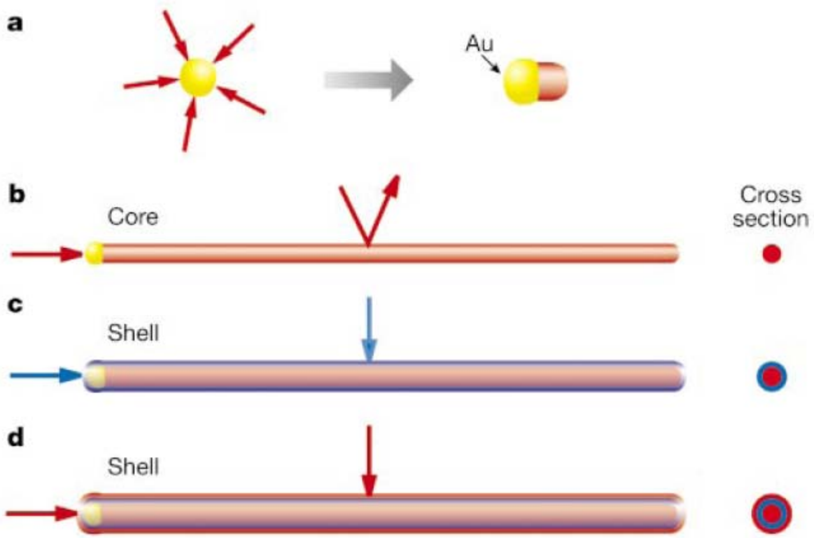


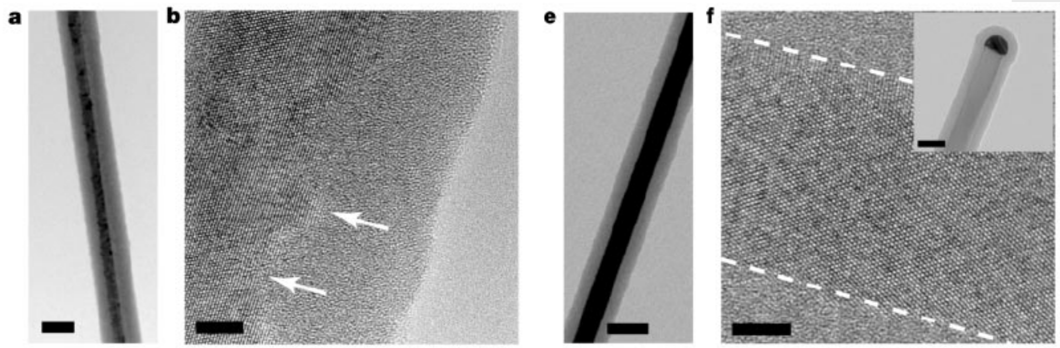
W.I. Park, Adv. Mater. 15 (2003) 526.

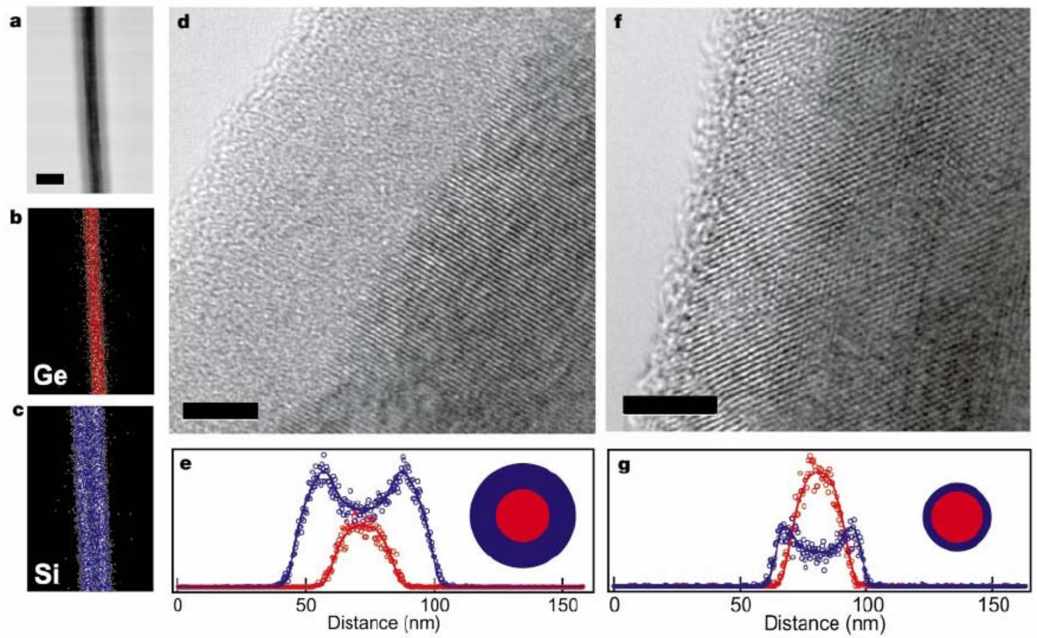
Synthesis of Nano Materials- 1D from gas phase

❑ Vapor-Liquid-Solid (VLS) Growth- core-shell

- Ge/Si





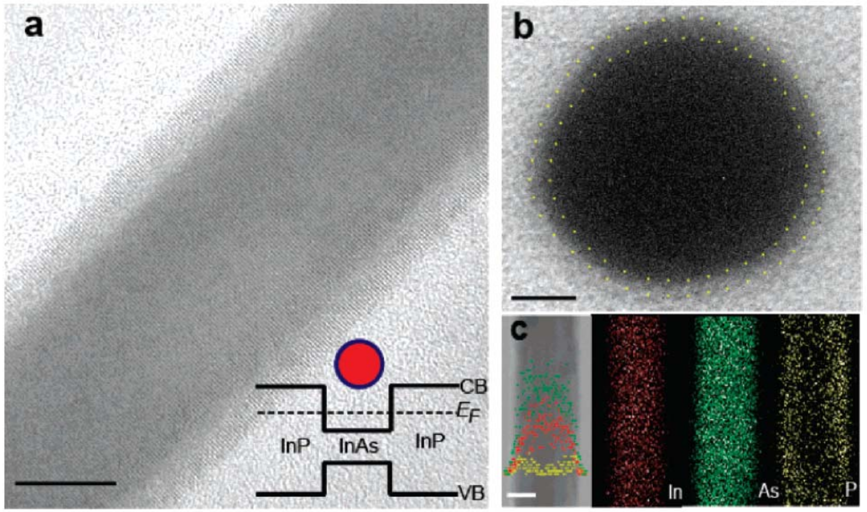


L. J. Lauhon, Nature 420 (2002) 57.

Synthesis of Nano Materials- 1D from gas phase

❑ Vapor-Liquid-Solid (VLS) Growth- core-shell

-InP/InAS

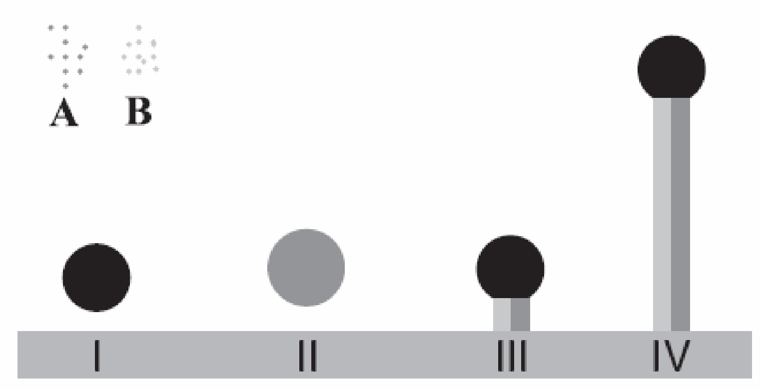


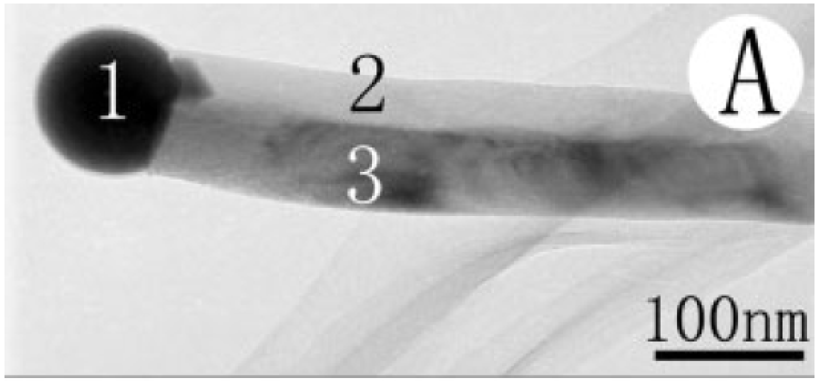
X. Jiang, Nono Lett. 7 (2007) 3214.

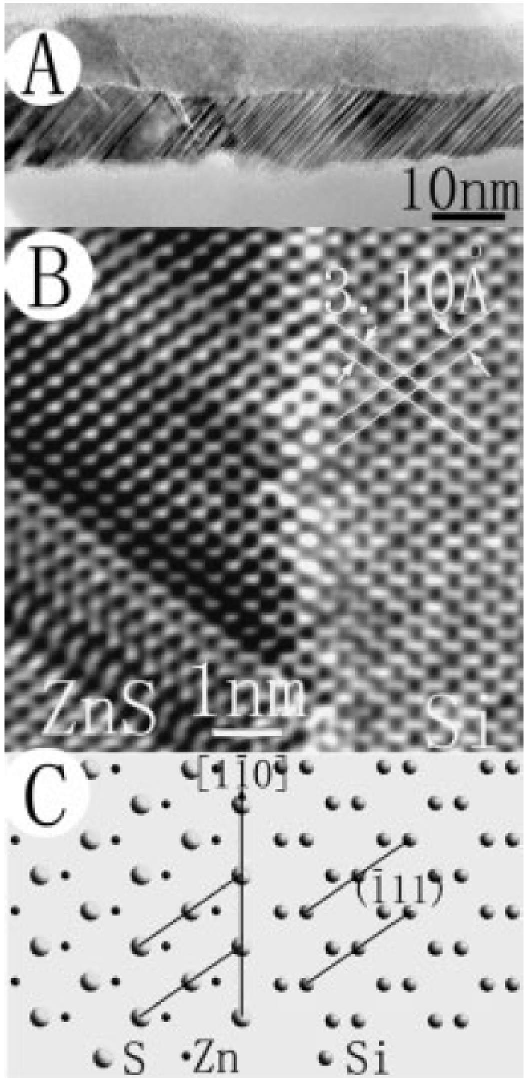
Synthesis of Nano Materials- 1D from gas phase

❑ Vapor-Liquid-Solid (VLS) Growth- composite

-single catalyst confined ZnS/Si



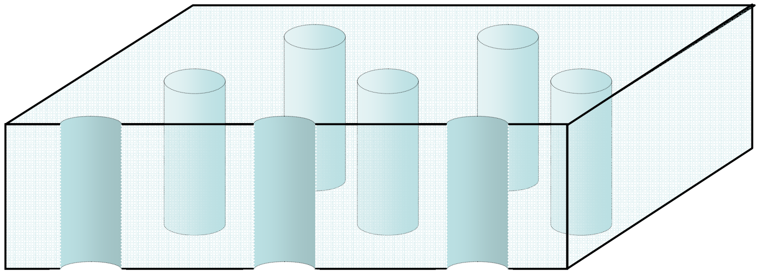




Z. John, Adv. Mater. 17 (2005) 225.

Synthesis of Nano Materials- 1D from gas phase

❑ Template-based



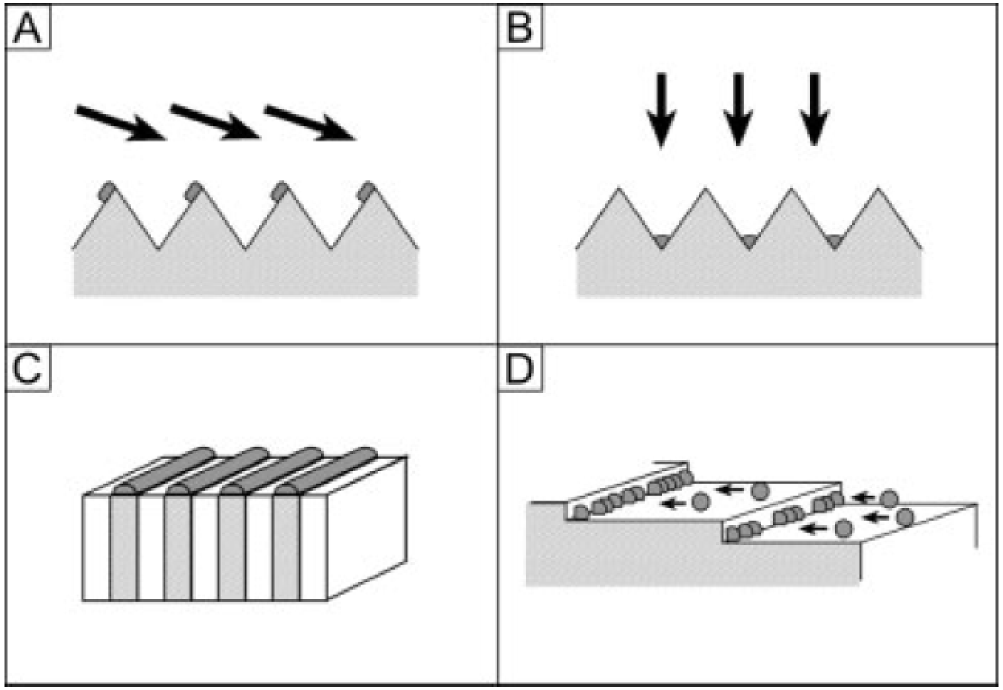
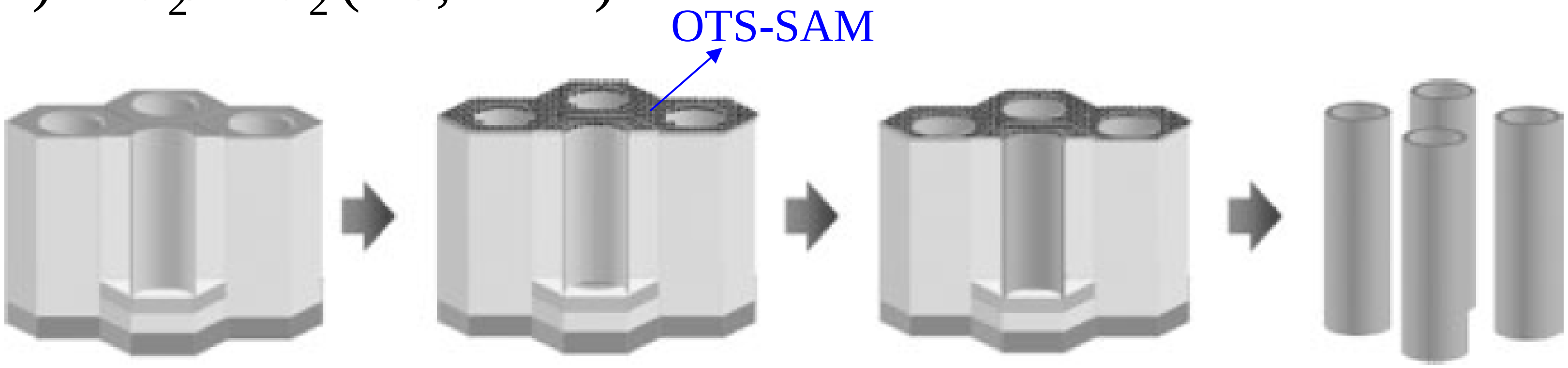


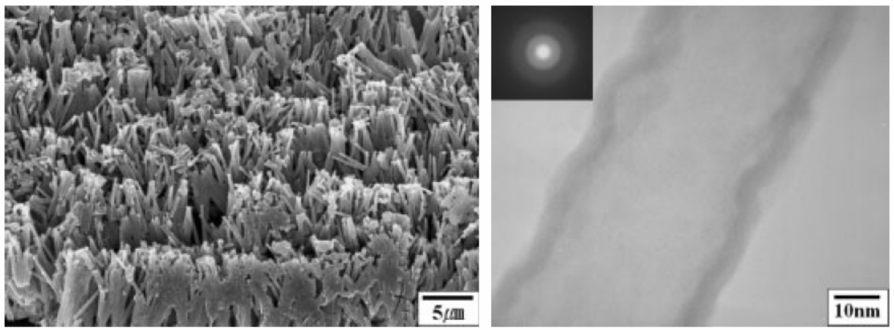
Fig. 6. Schematic illustrations of procedures that generated 1D nanostructures by A) shadow evaporation [58]; B) reconstruction at the bottom of V-grooves [60]; C) cleaved-edge overgrowth on the cross-section of a multilayer film [64]; and D) templating against step edges on the surface of a solid substrate [68].

Synthesis of Nano Materials- 1D from gas phase

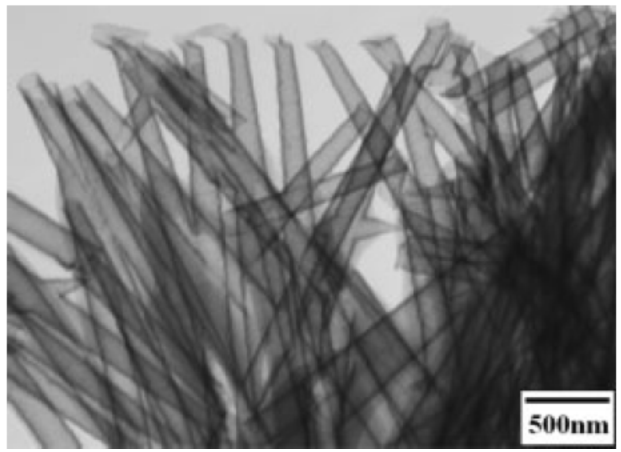
❑ Template-based

ex) TiO_2/ZrO_2 (PC, ALD)





TiO_2



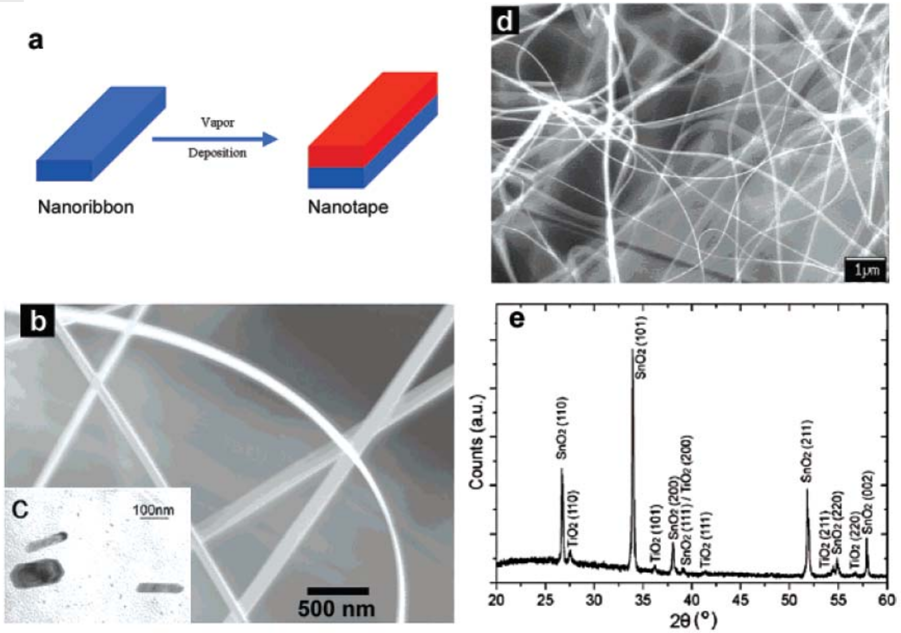
ZrO_2

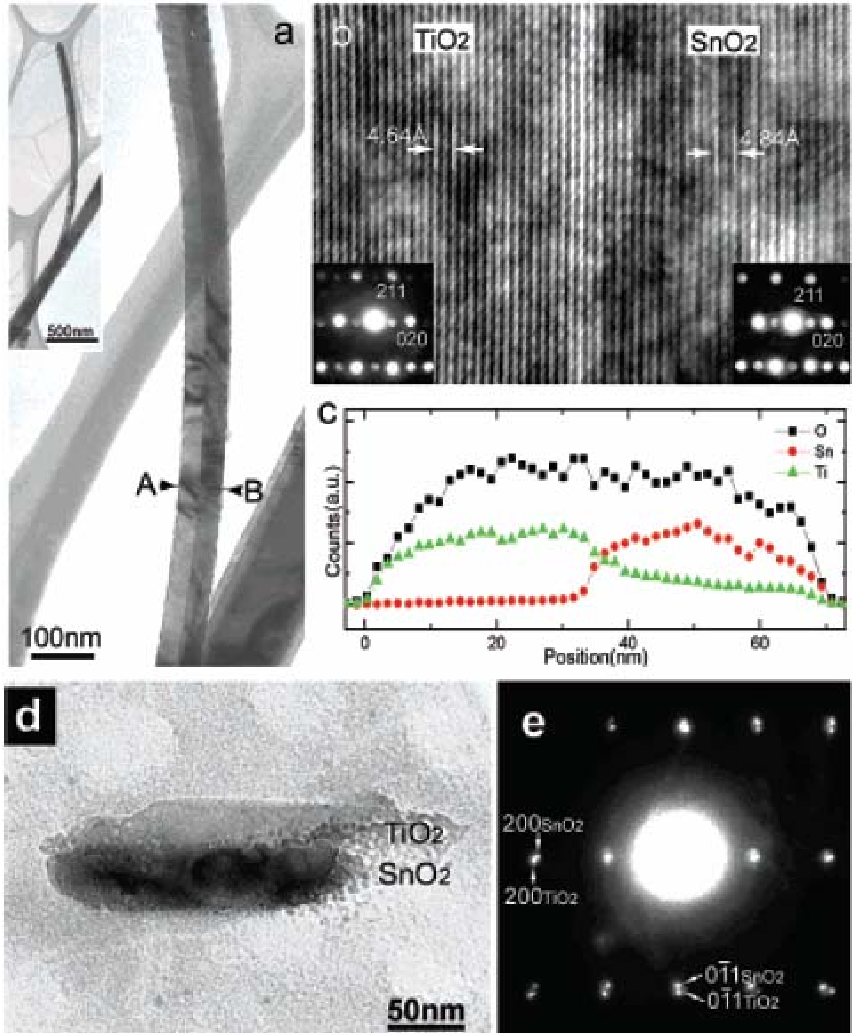
H. Shin, Adv. Mater, 16 (2004) 1197.

Synthesis of Nano Materials- 1D from gas phase

❑ Template-based

ex) $TiO_2@SnO_2$ (PLD)



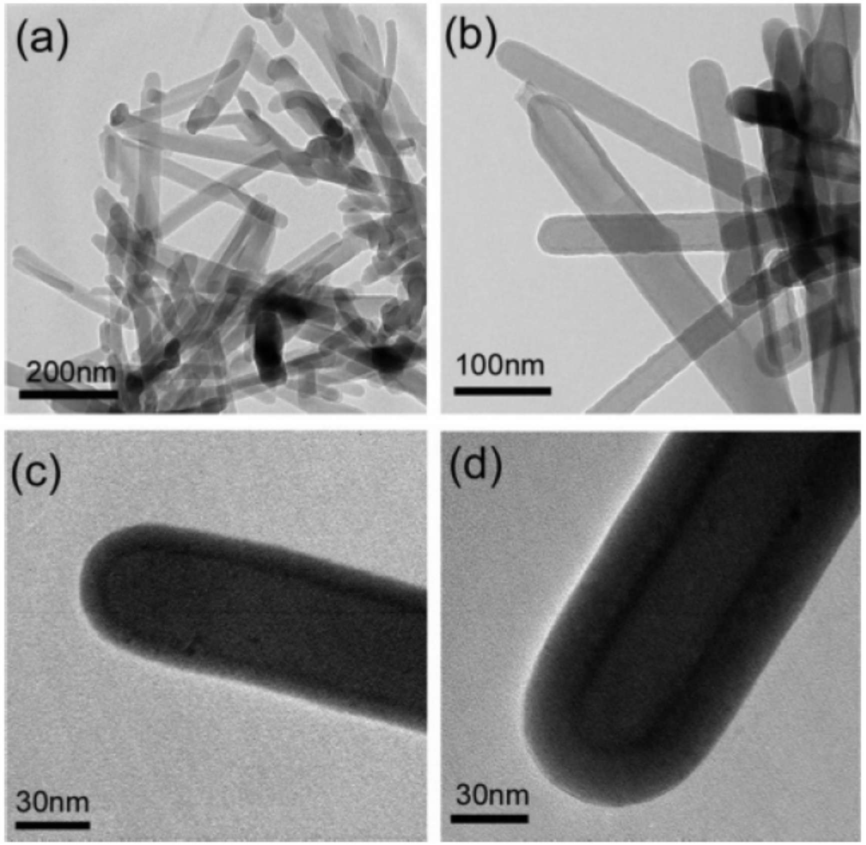


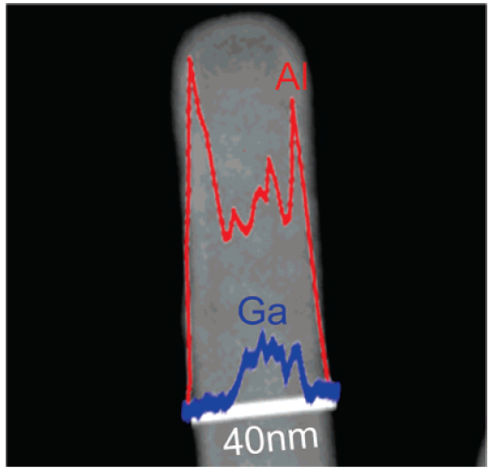
R. He, Nano Lett, 2 (2002) 1109.

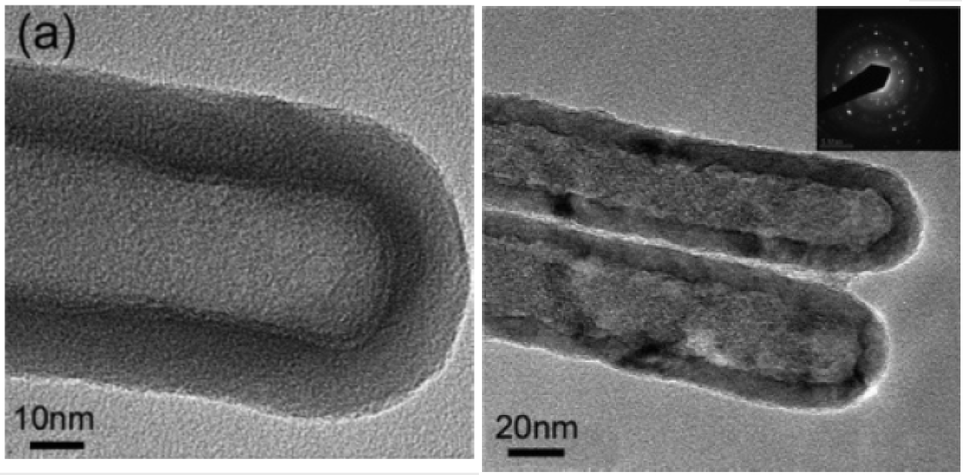
Synthesis of Nano Materials- 1D from gas phase

❑ Template-based

ex) Al_2O_3@GaQ(Tris-(8-hydroxyquinoline) Gallium) (ALD)





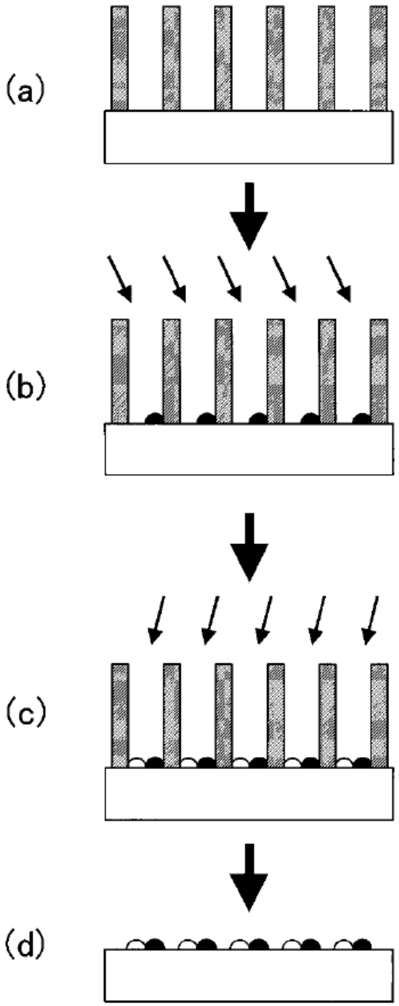


C.-C. Wang, nano Lett. 7 (2007) 1566.

Synthesis of Nano Materials- 1D from gas phase

❑ Template-based

ex) AAO on Si, evaporation of Au and Ag



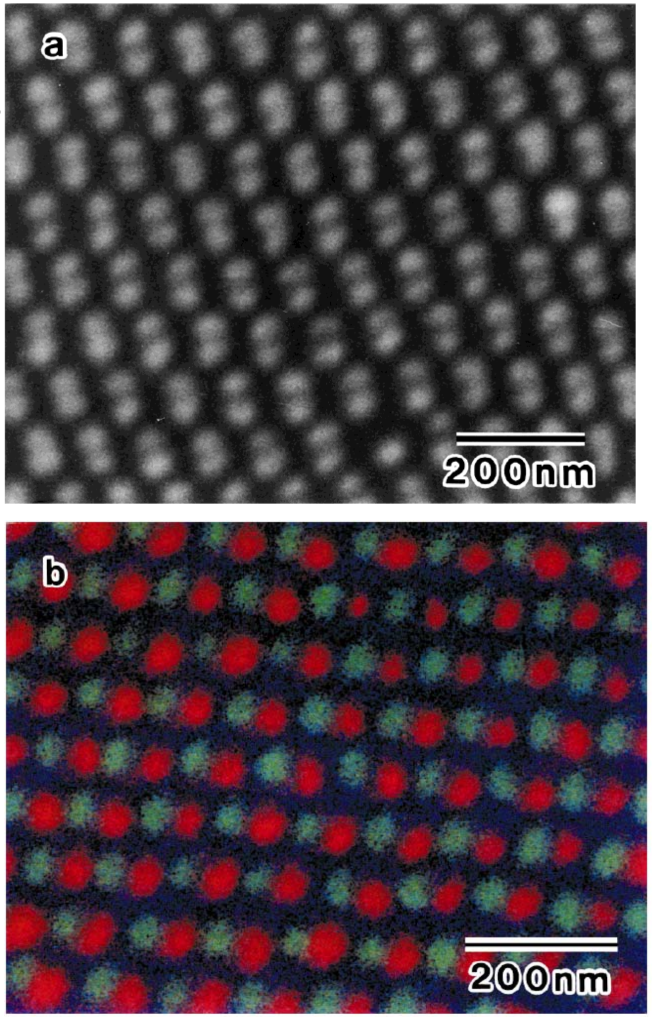


Fig. 3. Images of the array of bimetallic dots of Au and Ag: a) SEM image, b) SAM image. In (b) the signals from Au, Ag, and Si are shown in green, red, and blue, respectively.

H. Masuda, Adv. Mater. 12 (2000) 1031.

Synthesis of Nano Materials- 1D from gas phase

❑ Template-based

ex) oblique evaporation of metal
on V-grooved InP (001)

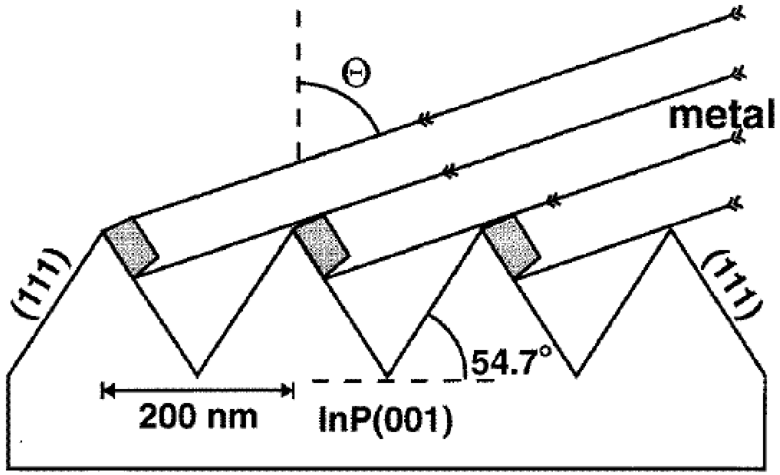
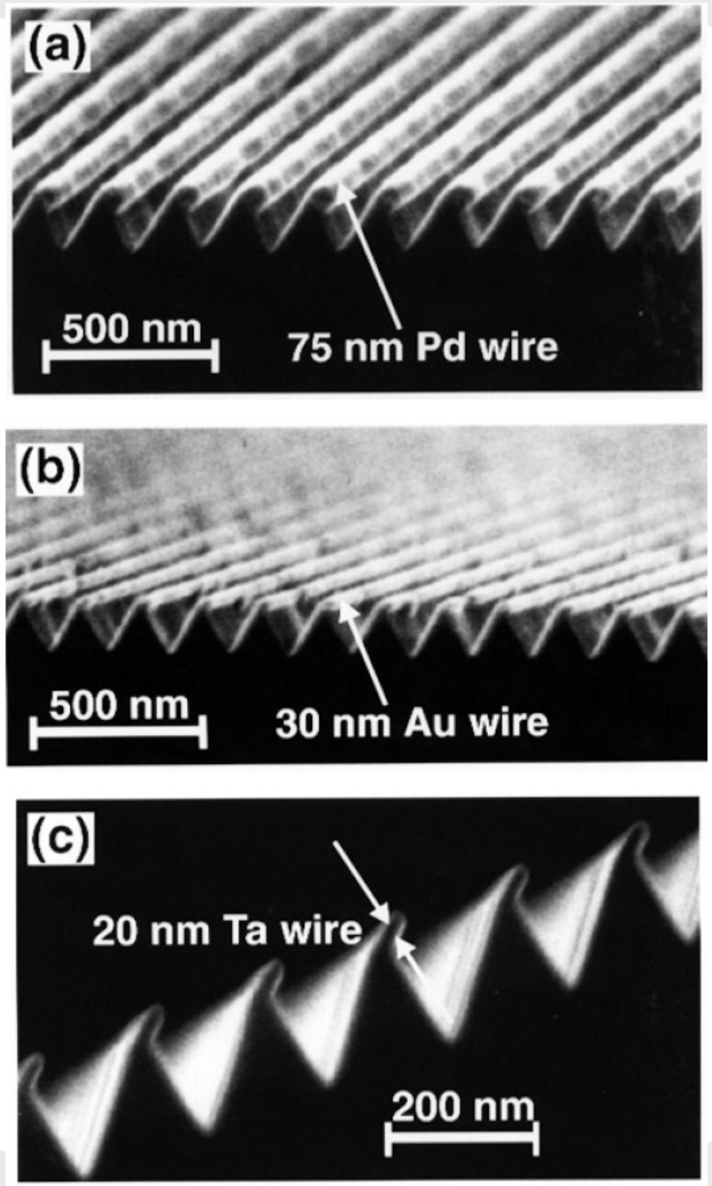


Figure 1. Schematic picture of the oblique evaporation of metal onto V-grooved InP(001) substrates.



J. Jorritsma, Nanotechnology 7 (1996) 263.

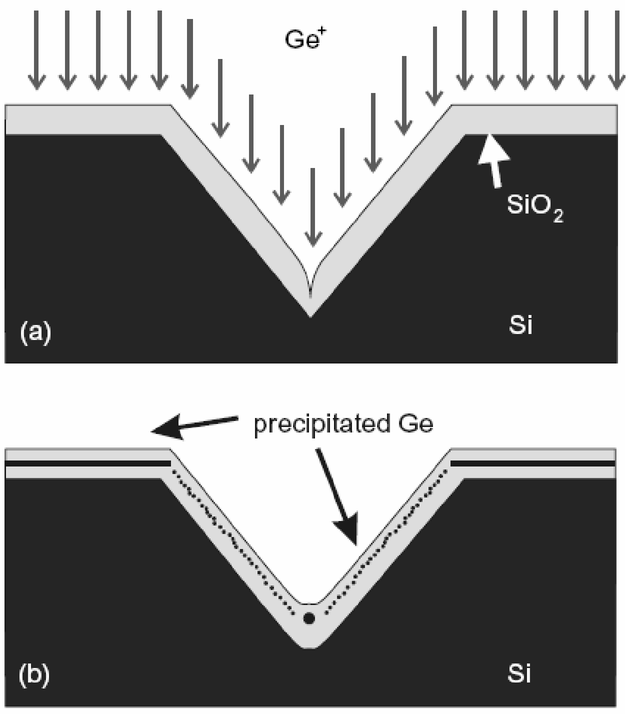
Synthesis of Nano Materials- 1D from gas phase

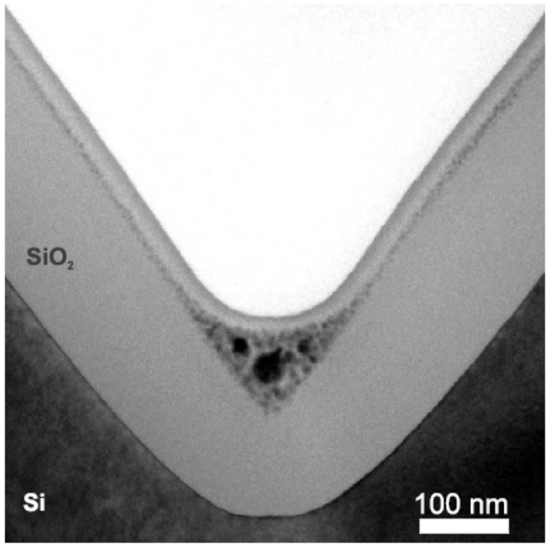
❑ Template-based (V-groove)

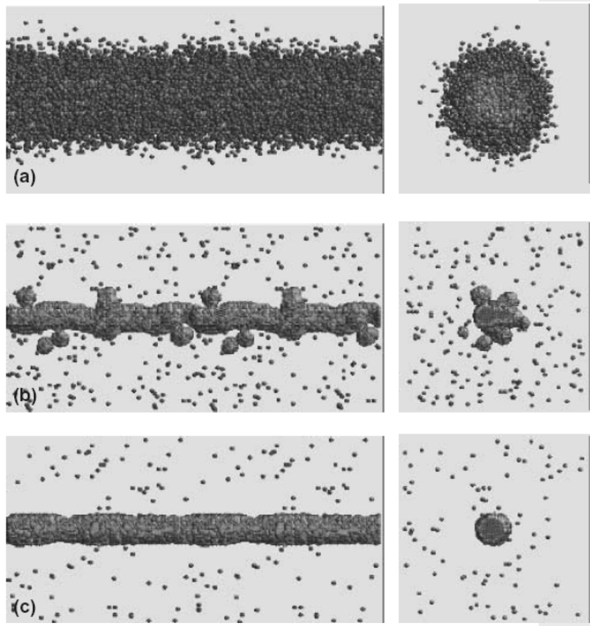
ex) Ge-nanowire

V-groove on (001) Si by anisotropic etching and oxidation

implantation of Ge, annealing in N_2 atmosphere, coalescence of Ge precipitates







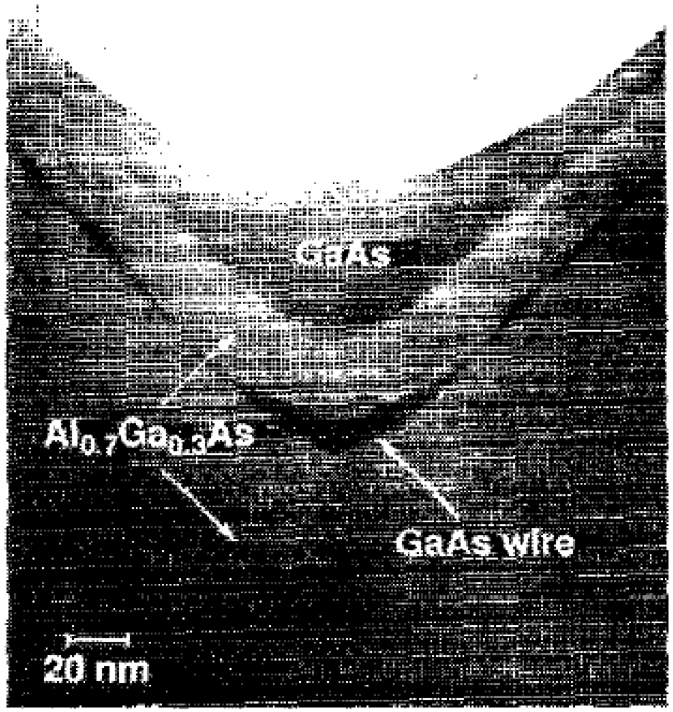
T. Muller, Nuclear Instrum. Mehtods Phys. Res. B175 (2001) 468.

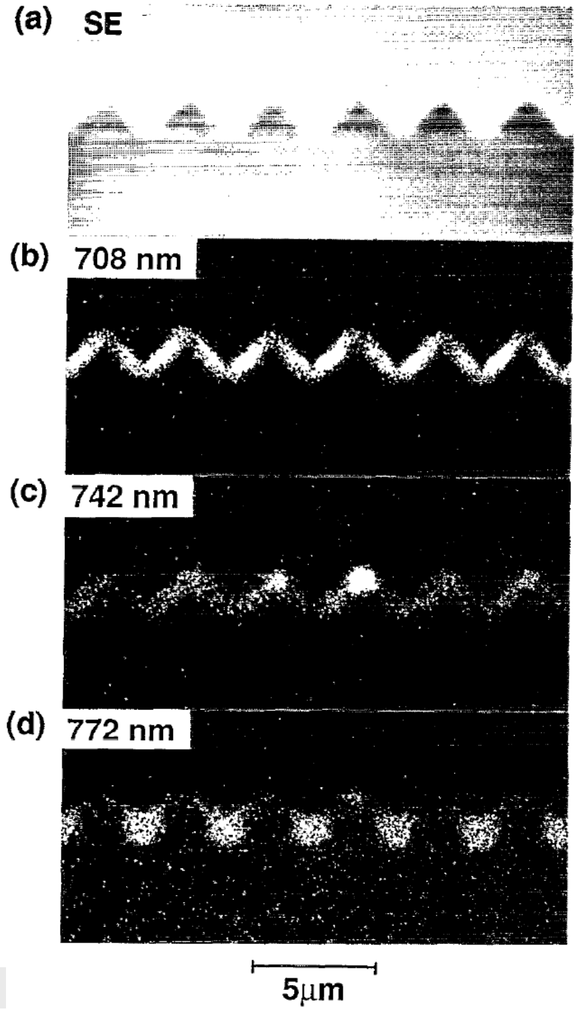
Synthesis of Nano Materials- 1D from gas phase

❑ Template-based (V-groove)

ex) GaAs-quantum wire

organometallic chemical vapor deposition on the V-grooved (100) GaAs





E. Kapon, Appl. Phys. Lett. 60 (1992) 477.

Synthesis of Nano Materials- 1D from gas phase

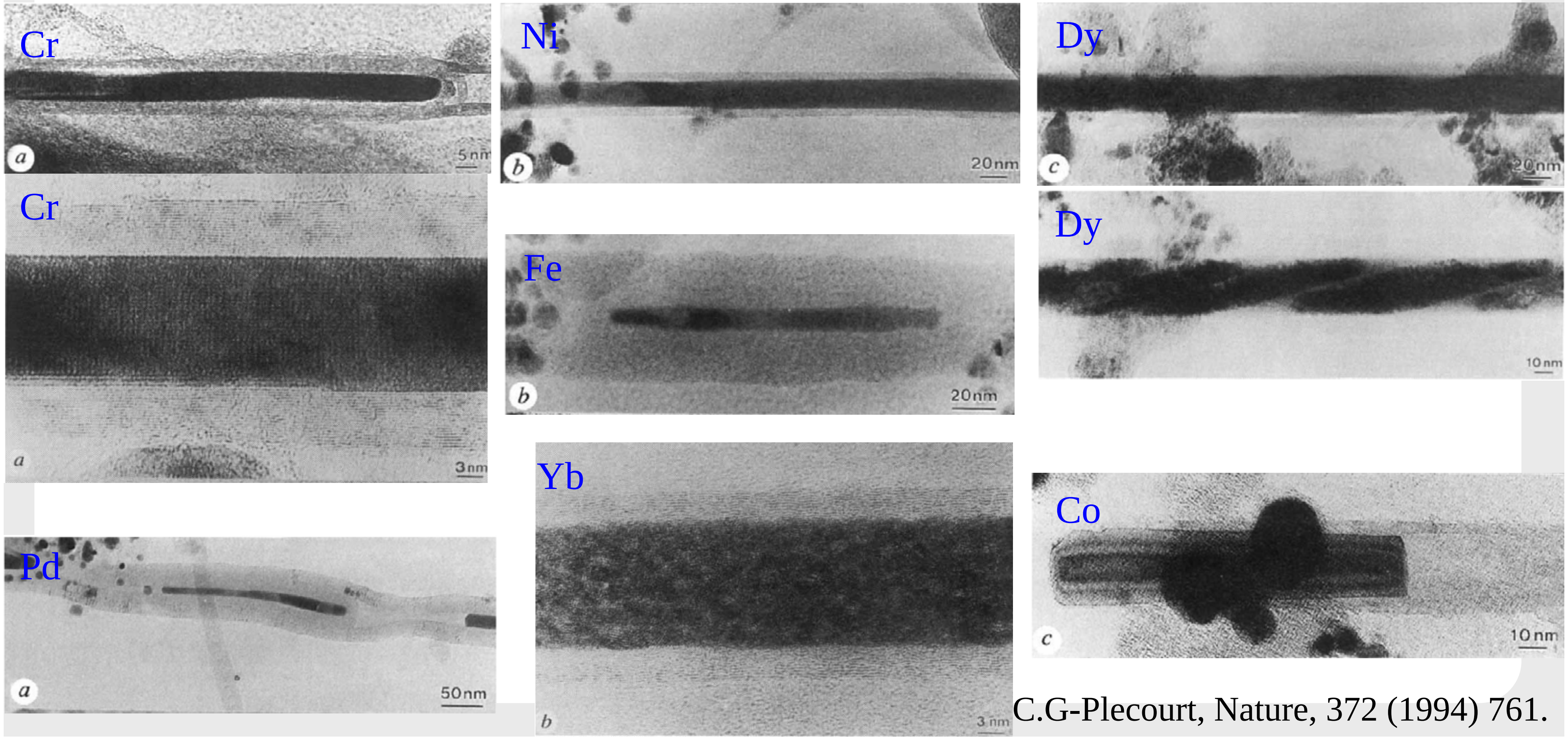
❑ Template-based (carbon nanotube)

ex) metal compounds inside carbon nanotube- arc discharge synthesis

cathode- graphite rod

anode- graphite rod with a hole filled with graphite and metal powder

metal carbide

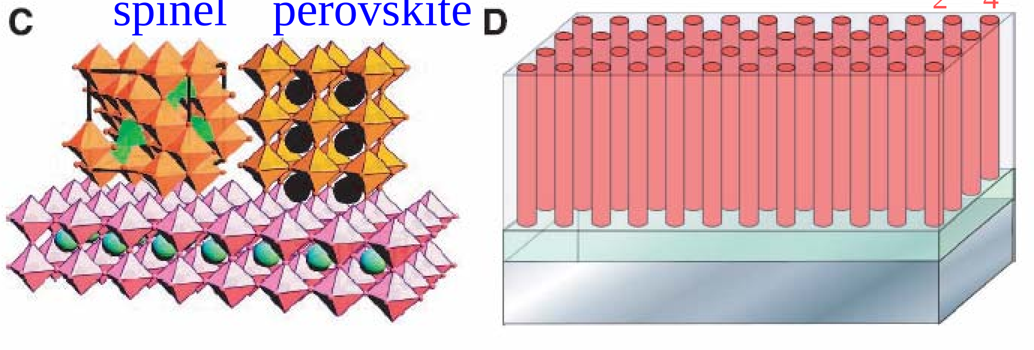


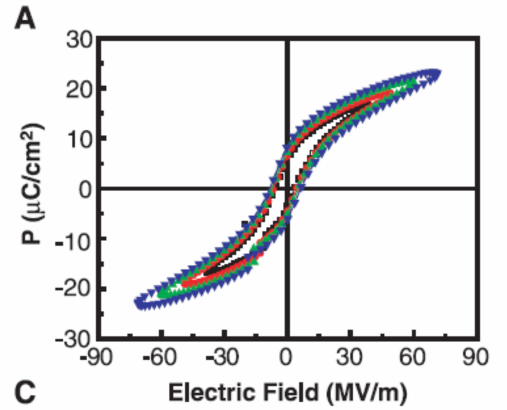
C.G-Plecourt, Nature, 372 (1994) 761.

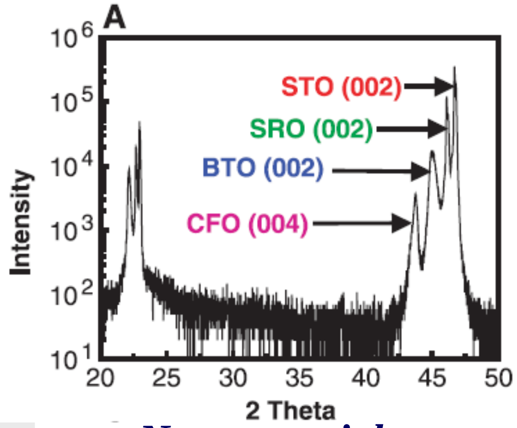
Synthesis of Nano Materials- 1D from gas phase

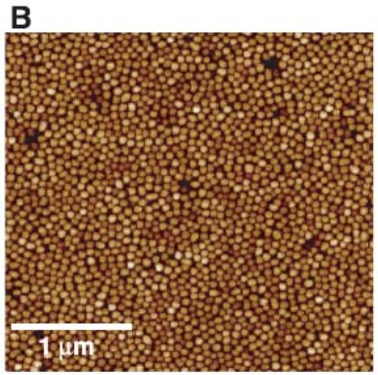
❑ Self-Assembly

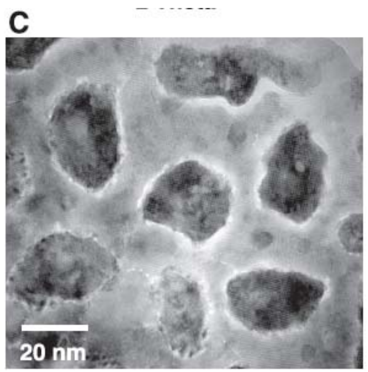
ex) $BaTiO_3$-$CoFe_2O_4$ nanostructure (PLD, Ba-Ti-Co-Fe oxide target)

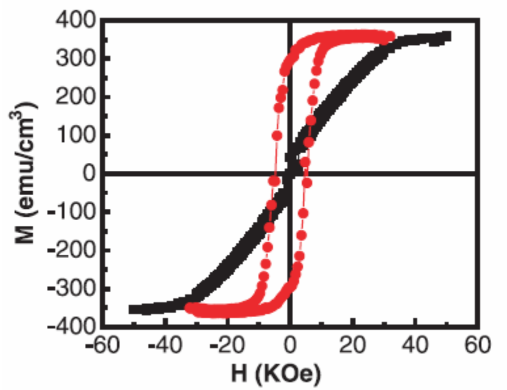












H. Zheng,, Science, 303 (204) 661.