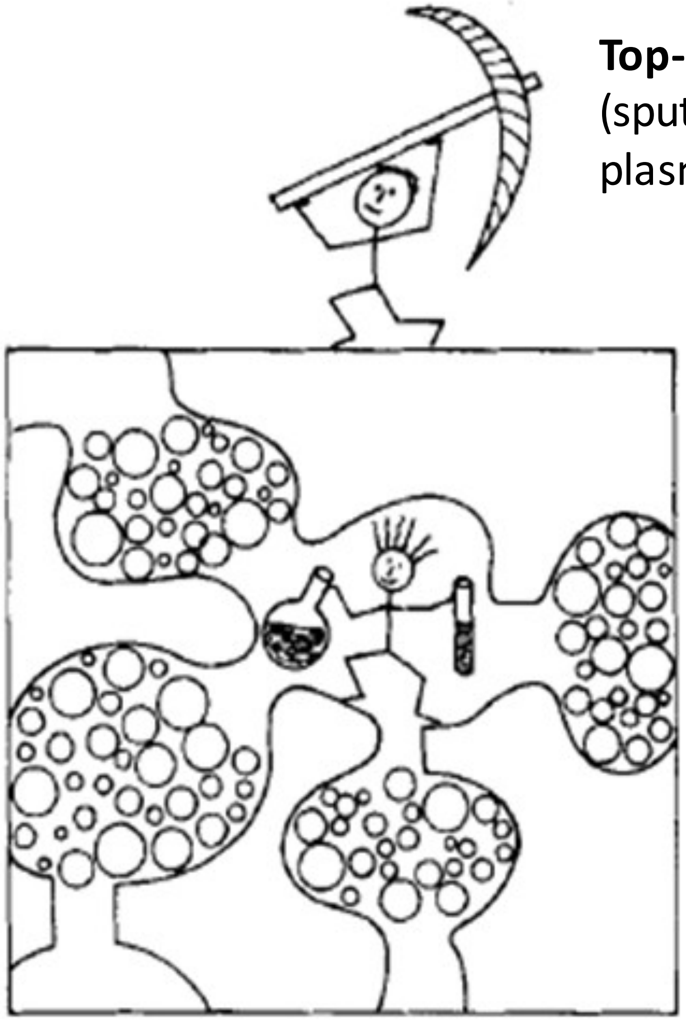
# ChE 430
# Colloidal synthesis of nanoparticles and their energy applications

**MODULE 3: Nucleation and Growth**

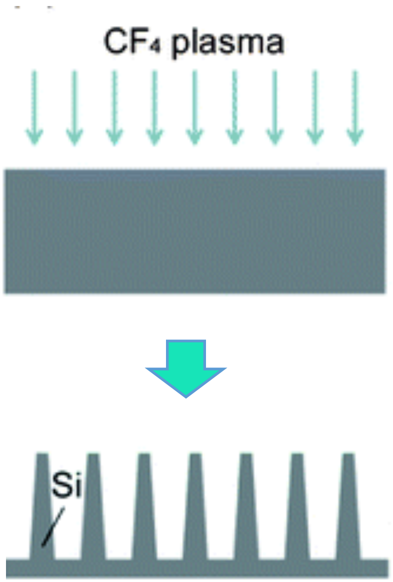
3.1. Introduction
3.2. The Classical Nucleation and Growth Theory
3.3. The Non-Classical Nucleation and Growth Theory
3.4. Chemical reaction mechanisms
3.5. In-situ studies

## 3.1. Introduction

# Top-down and bottom-up



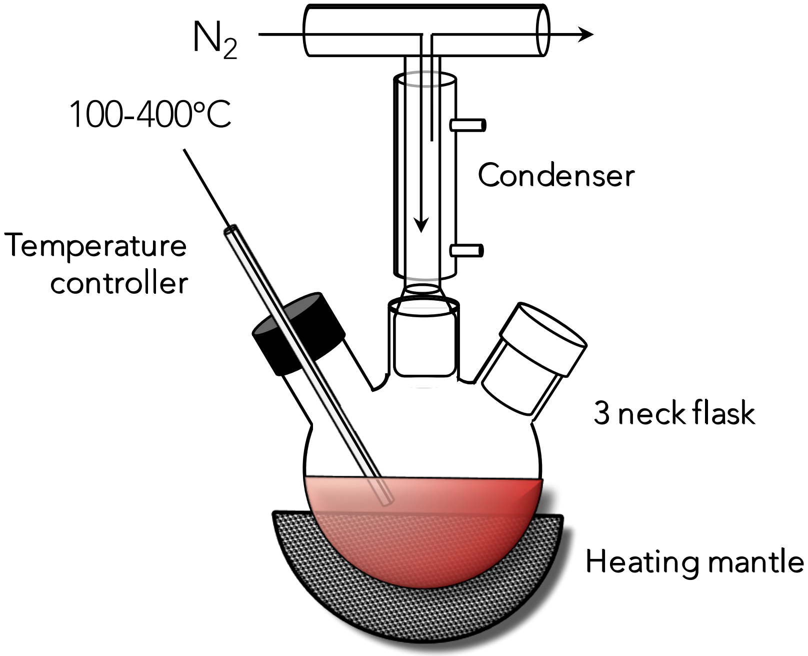
**Top-down approaches**
(sputtering, lithography, plasma etching)



**Bottom-up approaches**
(chemical vapor deposition, laser pyrolysis, chemical synthesis)

# 3.1. Introduction

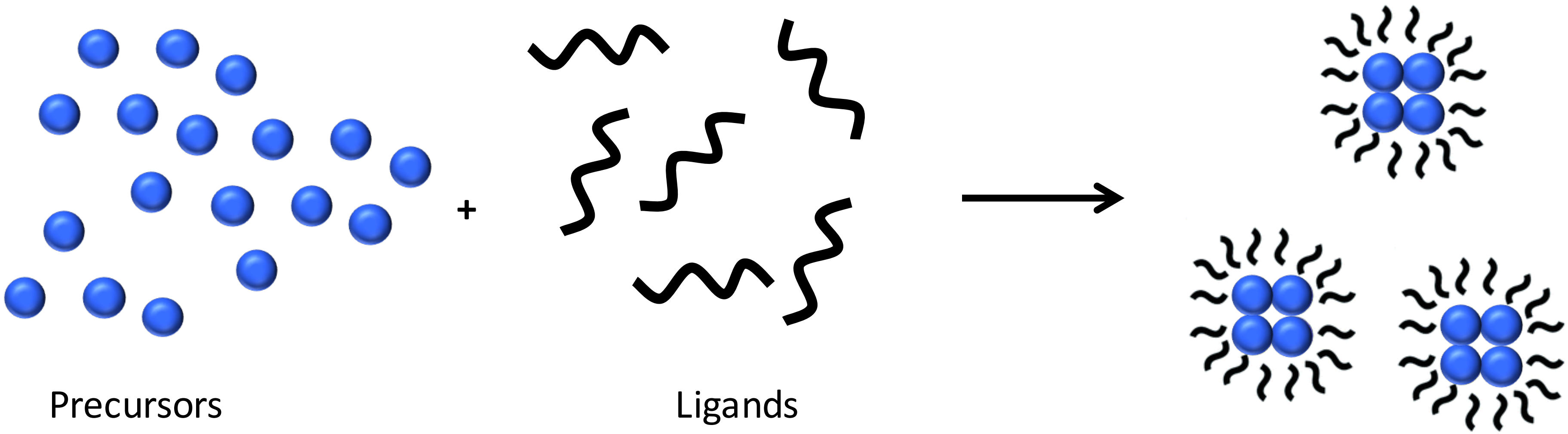
## Colloidal synthesis



**Reaction parameters:**

- Ligands/Precursors reactivity
- Reaction temperature
- Reaction time
- Concentration

# Building nanocrystals from atoms

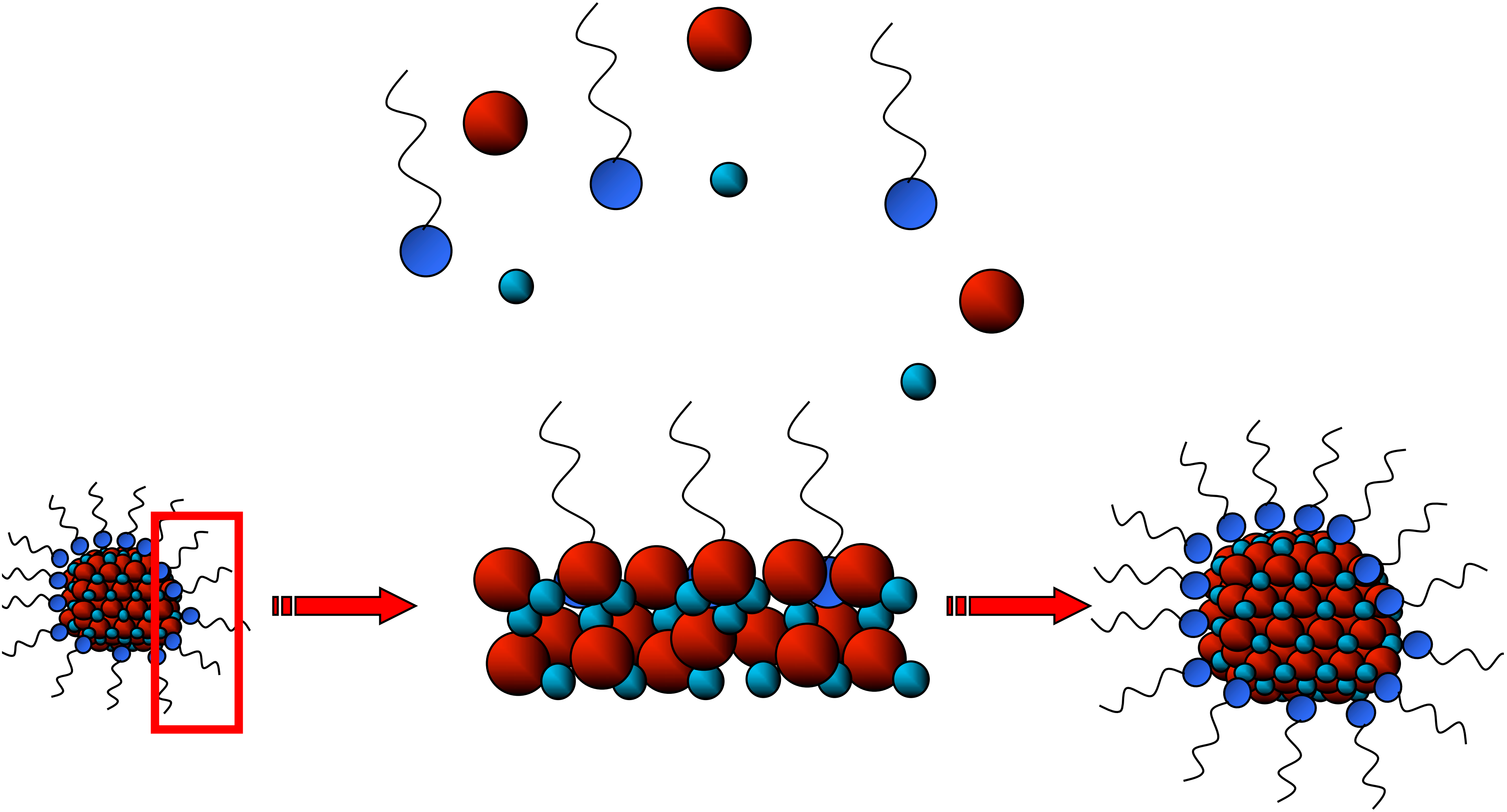


Precursors
- salts
- organometallic complexes
- elemental compounds

Ligands
- carboxylic acids
- amines
- thiols
- phosphonic acids
- phosphines

**3.1. Introduction**

# Ligands are in dynamic equilibrium between NC surface and the growth solution



## 3.1. Introduction

*“..the current state of nanocrystal synthesis is comparable to that of organic synthesis a century ago”*

*(Buhro et al. Chem. Mater. 2014)*

*“While organic reactions that underpin molecular total synthesis can be applied rationally in a by-design manner, the rules and guidelines for constructing high-order hybrid nanoparticles are poorly understood and far from mature”*

*(Schaak et al. J. Am. Chem. Soc. 2015)*

## 3.1. Introduction

**Nucleation:** The process whereby nuclei (seeds) act as templates for crystal growth.

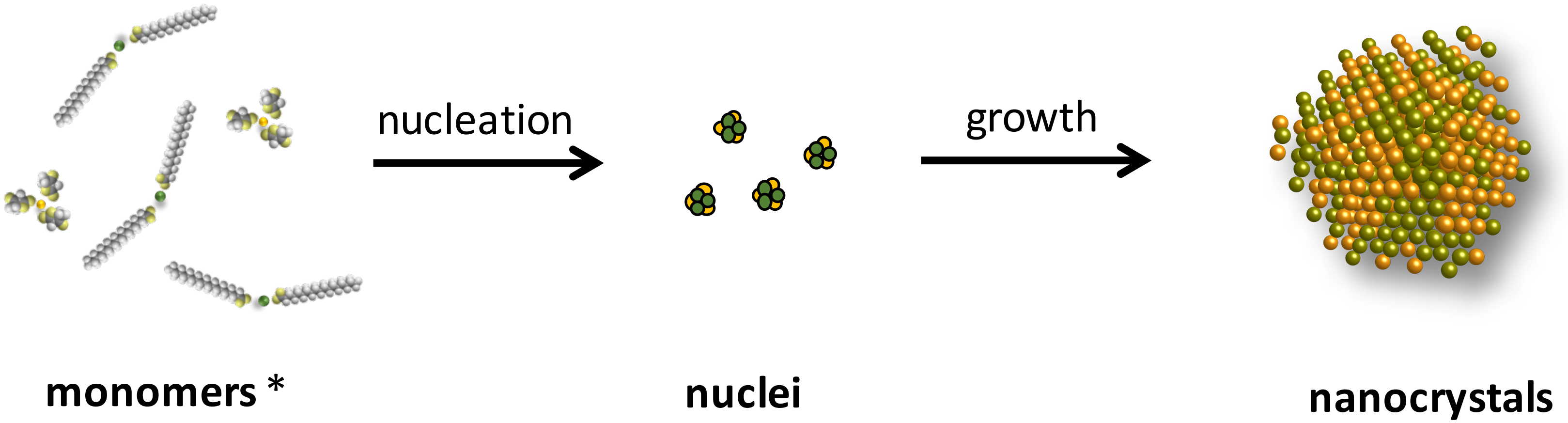
**Homogeneous Nucleation:** it occurs when nuclei form uniformly throughout the parent phase

**Heterogeneous Nucleation:** it occurs when nuclei form on a pre-existing surface (container surface, impurities, nanocrystal seeds)

## 2.2. The Classical Nucleation and Growth Theory

## 2.3. The Non-classical Nucleation and Growth Theory

# 3.2. The Classical Nucleation Theory



nucleation

growth

**monomers** *

**nuclei**

**nanocrystals**

The growth proceeds through addition of monomers to the nuclei

*Monomers might be different from the precursors injected in the flask

# 3.2. The Classical Nucleation Theory

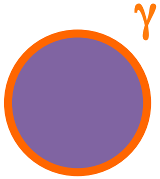
**Free energy of nuclei formation**

$$\Delta G_N = \boxed{4\pi r^2 \gamma} + \boxed{\frac{4}{3}\pi r^3 \Delta G_V}$$

$$\Delta G_V = \frac{-k_B T ln(S)}{V_m}$$

$k_B$ is the Boltzmann's constant
$T$ is the temperature
$S$ is the supersaturation of the solution
$V_m$ is the molar volume

**Surface energy:** energetic penalty to pay in order to establish an interface between the new solid phase and the pre-existing solution phase.



Surface energy between the solid nucleus and the solution phase

**Bulk energy:** energetic gain from the formation of a new spherical nucleus

# 3.2. The Classical Nucleation Theory

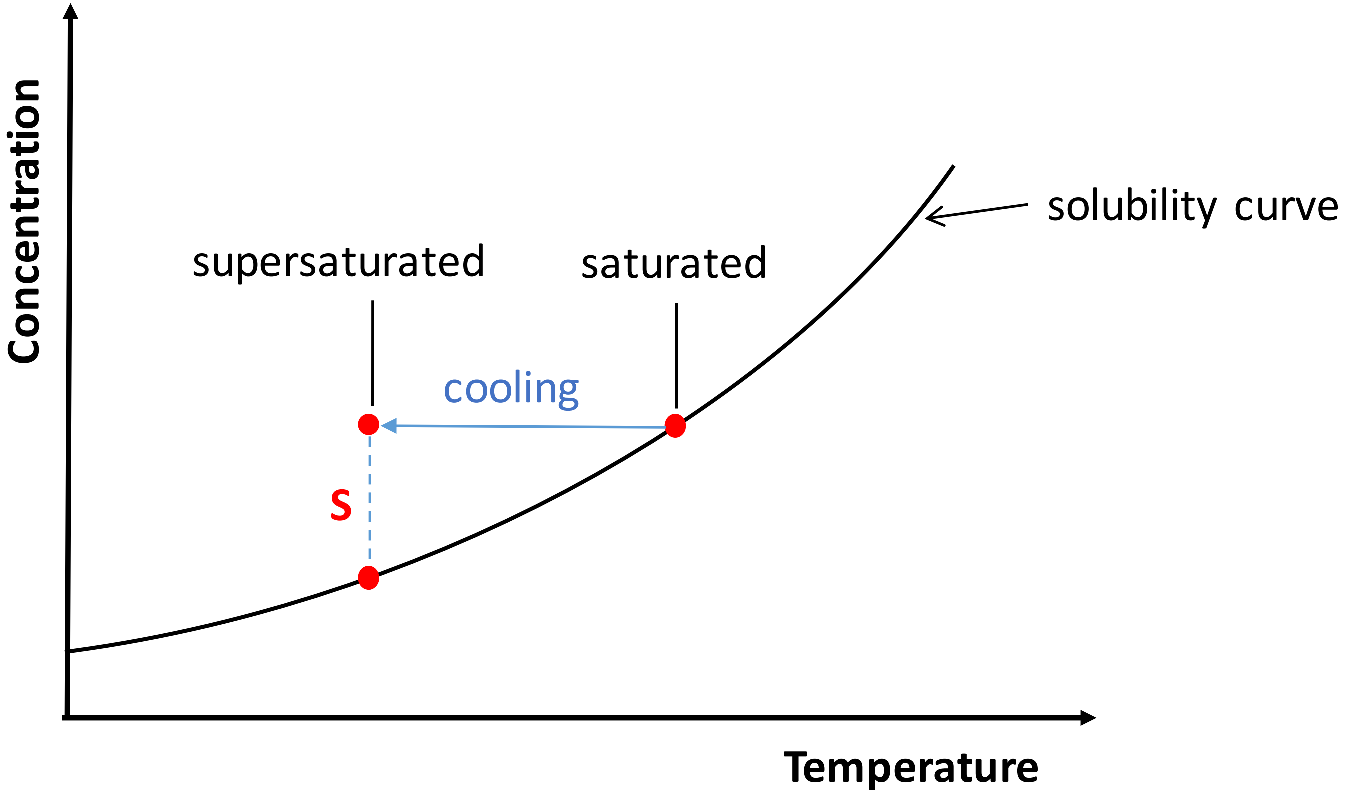
QUESTION: are you familiar with the definition of unsaturated, saturated and supersaturated solution?

What does supersaturation mean?

How do we get out of supersaturation?

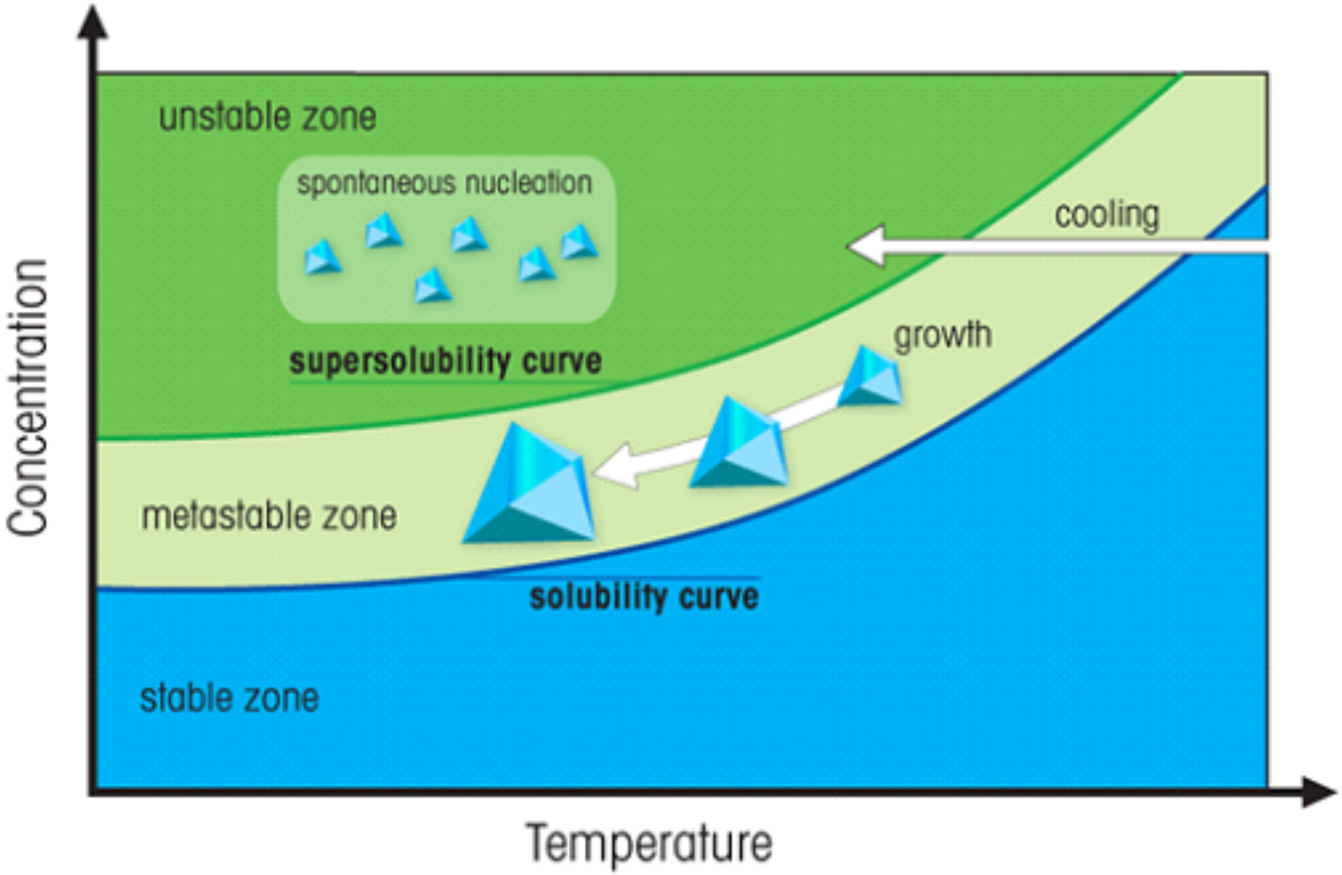
## 3.2. The Classical Nucleation Theory

**Supersaturation (S):** The difference between the actual concentration and the solubility concentration at a given temperature



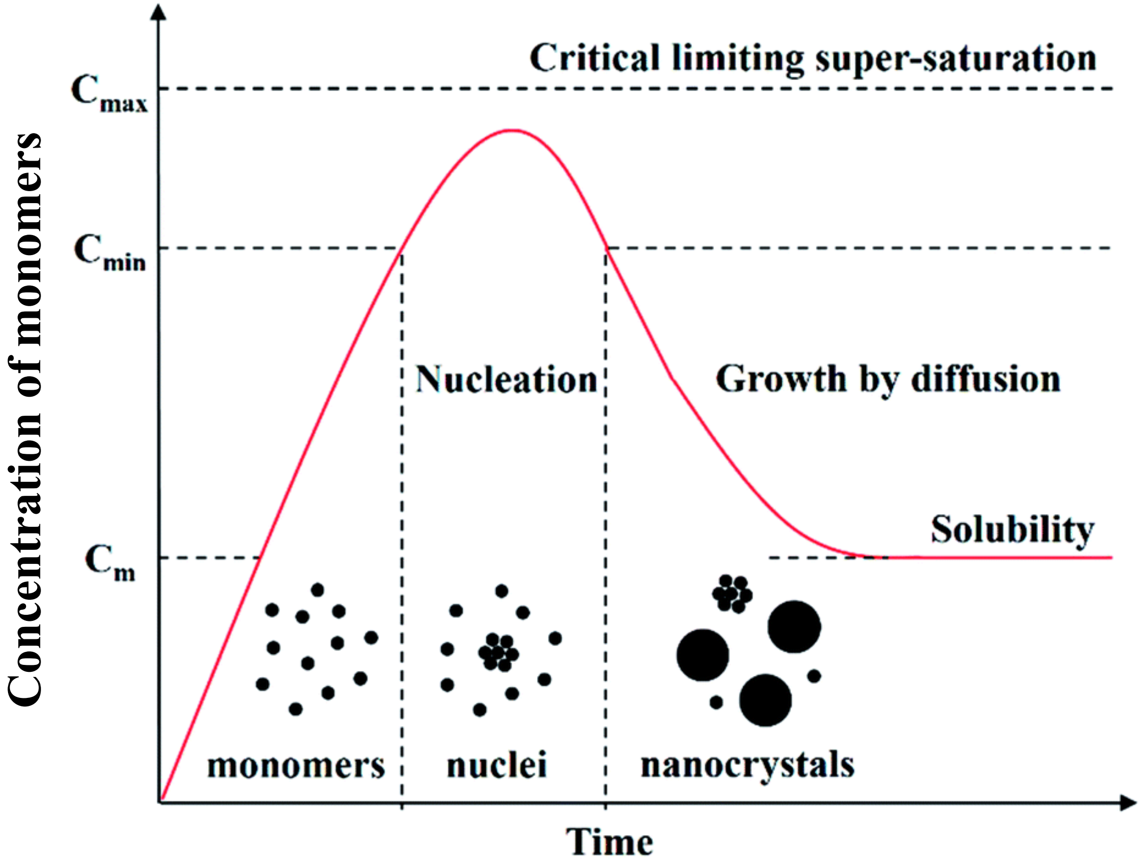
## 3.2. The Classical Nucleation Theory

**Supersaturation:** The difference between the actual concentration and the solubility concentration at a given temperature



# 3.2. The Classical Nucleation Theory

## La Mer diagram

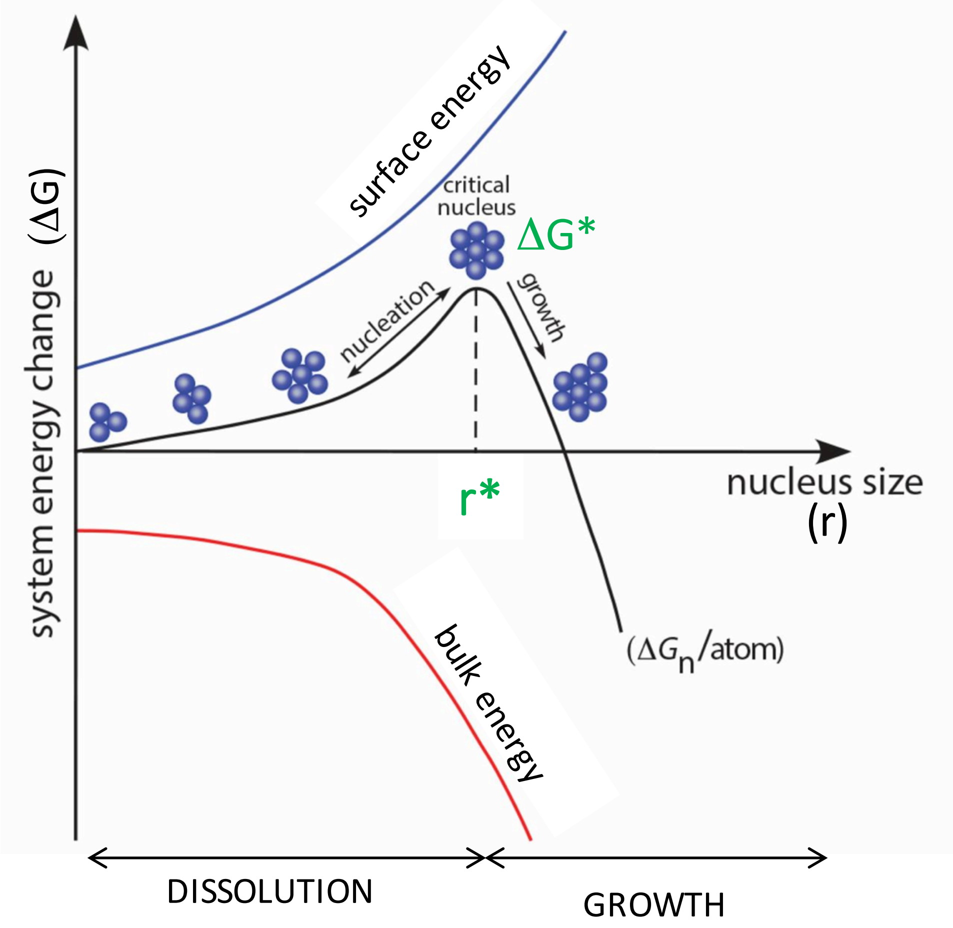


The goal is the achieve supersaturation which will lead to one brief outburst of nucleation. Above the critical limiting super-saturation, only spontaneous nucleation and no growth will occur.

# 3.2. The Classical Nucleation Theory

**Free energy of nuclei formation**

$$\Delta G_N = \boxed{4\pi r^2\gamma} - \boxed{\frac{4}{3}\pi r^3 \frac{k_B T ln(S)}{V_m}}$$



$$\Delta G^* = \frac{4}{3}\pi\gamma(r^*)^3$$ from $\frac{d\Delta G}{dr} = 0$

$$r^* = \frac{2\gamma V_m}{RT \ln S}$$ from $\Delta G = \Delta G^*$

r < r* nuclei form but redissolve (Gibbs-Thomson effect)

r > r* nuclei form and grow

(The **critical-nucleus size** is in the range of tens to hundreds of atoms, 1-3 nm)

## 3.2. The Classical Nucleation Theory

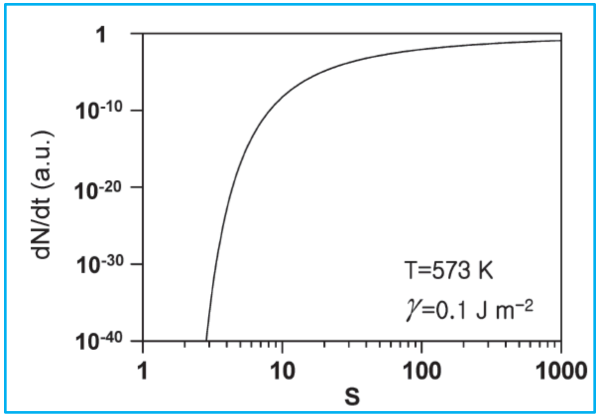
The **rate of nucleation of N particles** during time *t* can be described using an Arrhenius type equation:

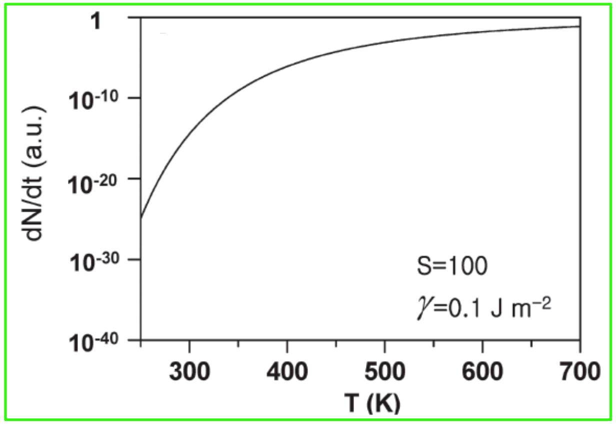
$$\frac{dN}{dt} = A\exp\left[\frac{-\Delta G_N}{k_B T}\right]$$

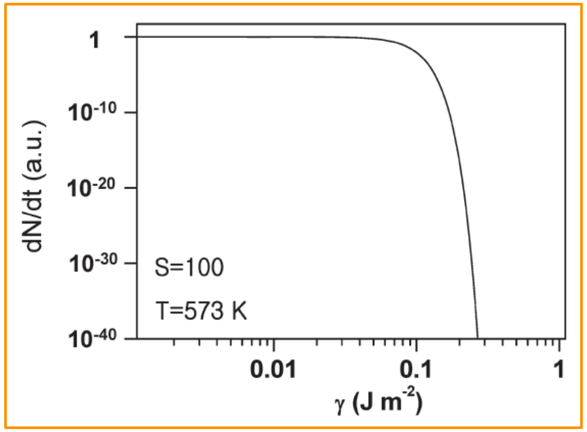
$$= A\exp\left[-\frac{16\pi \boxed{\gamma}^3 V_m^2}{3k_B^3 \boxed{T}^3 N_A^2 (\ln \boxed{S})^2}\right]$$

Experimentally controllable parameters influencing the nucleation rate:

1. saturation
2. temperature
3. surface free energy







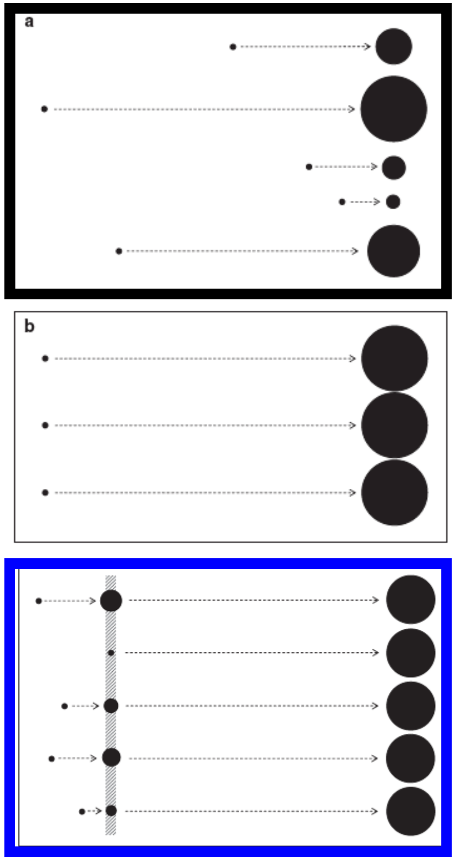
As the level of supersaturation is increased from *S* = 2 to 4, the nucleation rate is increased by ≈ $10^{70}$ times

Hyeon et al. *Small* **2011**

# 3.2. The Classical Nucleation Theory

Size distribution is largely dependent on the time over which nuclei are formed:

- A narrow time window for nucleation leads to **monodisperse** nanocrystals (assuming we are in a focusing regime)
- A wider time window for nucleation leads to **polydisperse** nanocrystals

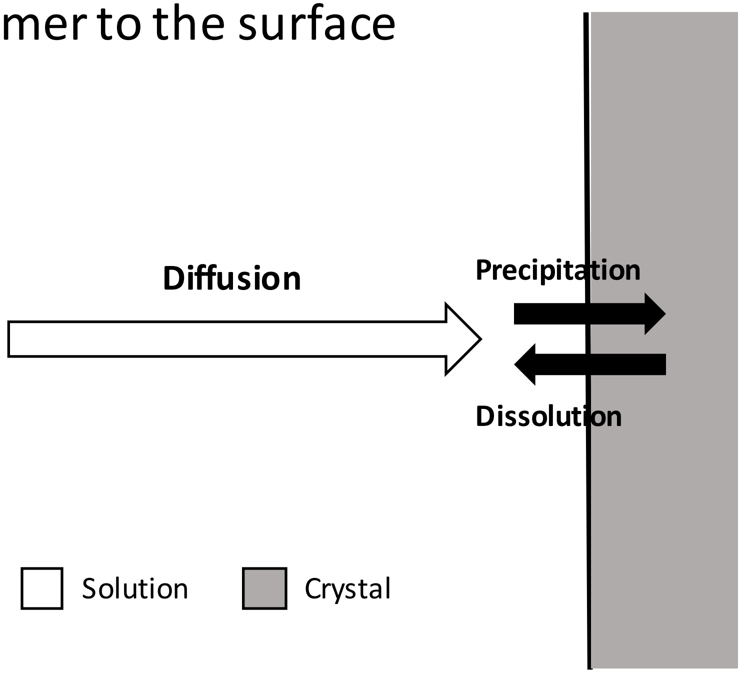


ideal conditions

Hyeon et al. *Small* **2011**

# 3.2. The Classical Nucleation Theory

1. Diffusion of monomer to the surface
2. Surface reaction



**Diffusion**

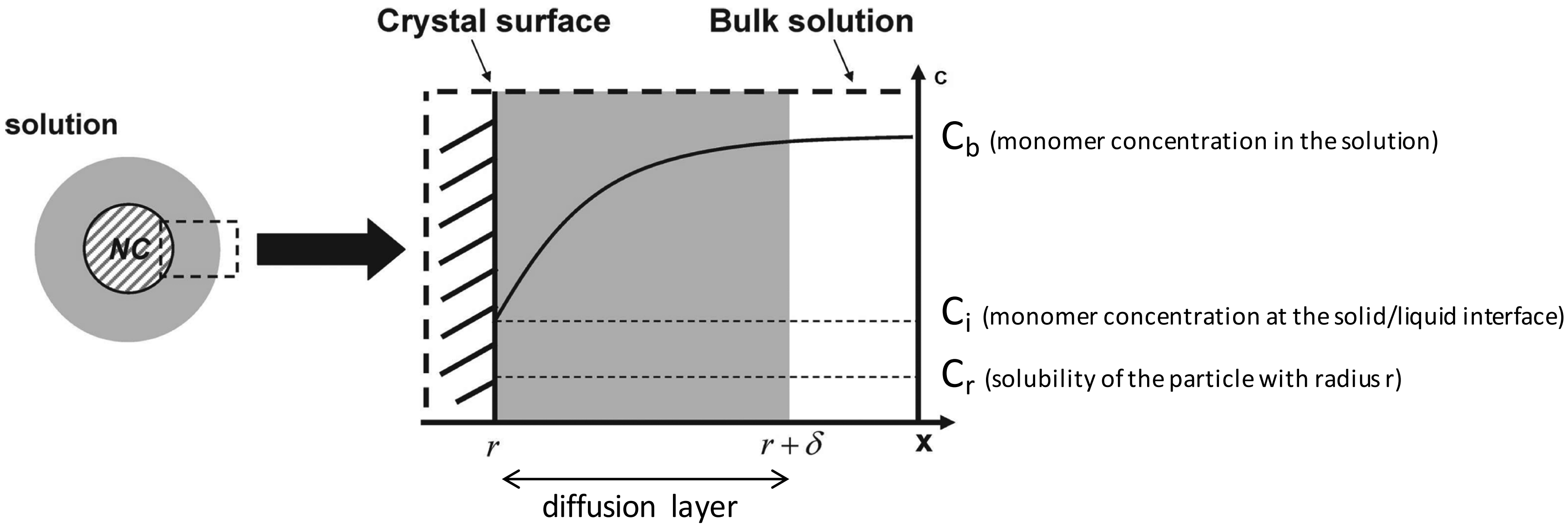
**Precipitation**

**Dissolution**

□ Solution ▨ Crystal

We can model the growth using the Fick’s Law (ChE-204)

# 3.2. The Classical Nucleation Theory



$C_b$ (monomer concentration in the solution)

$C_i$ (monomer concentration at the solid/liquid interface)

$C_r$ (solubility of the particle with radius r)

ASSUMPTION: steady-state (*J* = *const*)

$$J = 4\pi x^2 D \frac{dC}{dx}$$

*J* is the total flux of monomers
*x* is the radius of the spherical plane
*D* is the diffusion coefficient
*C* is the concentration at distance x

# 3.2. The Classical Nucleation Theory

By integrating C(x) between *r* and *r* + $\delta$, we obtain:

$$J = \frac{4\pi D r(r+\delta)}{\delta}(C_b - C_i)$$

The consumption rate of monomers at the surface is:
(ChE-204, exercize 4.6)

$$J = 4\pi r^2 k_d (C_i - C_r)$$

*K is the rate constant of a simple first order deposition reaction*

We can write the growth rate as

$$\frac{dr}{dt} = \frac{JV_m}{4\pi r^2}$$

$$\frac{dV}{dt} = \frac{d}{dt}\left(\frac{4}{3}\pi r^3\right) = \frac{4}{3}\pi\frac{d}{dt}(r^3) = \frac{4}{3}\pi(3r^2)\frac{dr}{dt} = 4\pi r^2\frac{dr}{dt}$$

$$\frac{dV}{dt} = JV_m$$

$V_m$ is molar volume

# 3.2. The Classical Nucleation Theory

$$\frac{\mathrm{d}r}{\mathrm{d}t} = \frac{\frac{D}{r}\left(1+\frac{r}{\delta}\right)V_{\mathrm{m}}(c_{\mathrm{b}}-c_r)}{1+\frac{D}{k_{\mathrm{d}}r\left(1+\frac{r}{\delta}\right)}}$$

From Gibbs-Thompson equation:

$$c_r = c_\infty \exp\left(\frac{2\sigma V_{\mathrm{m}}}{rRT}\right) \approx c_\infty\left(1+\frac{2\sigma V_{\mathrm{m}}}{rRT}\right)$$

$$c_b = c_\infty \exp\left(\frac{2\sigma V_{\mathrm{m}}}{r^*RT}\right) \approx c_\infty\left(1+\frac{2\sigma V_{\mathrm{m}}}{r^*RT}\right)$$

$C_\infty$ is the solubility of the bulk material
$\sigma$ is the interfacial energy
$V_m$ is the molar volume
$R$ is the universal gas constant

Since the diffusion layer δ is usually of the orders of microns, we can assume r **<<** δ and derive:

$$\boxed{\frac{\mathrm{d}r}{\mathrm{d}t} = \frac{2\sigma V_{\mathrm{m}}^2 c_\infty}{RT(1/D+1/k_{\mathrm{d}}r)}\,\frac{(1/r^*-1/r)}{r}}$$

## 3.2. The Classical Nucleation Theory

### Lifshitz-Slyozov-Wagner (LSW) Theory (1950)

If the growth is DIFFUSION-LIMITED, $D \ll K_D$

$$\boxed{\frac{\mathrm{d}r}{\mathrm{d}t} = \frac{2\sigma D V_\mathrm{m}^2 c_\infty}{RT} \frac{(r/r^* - 1)}{r^2} = K_\mathrm{D}(r/r^* - 1)/r^2}$$

ASSUMPTIONS:

- Spherical particles in a supersaturated solution
- Processes such as nucleation and aggregation that introduce new particles are negligible

# 3.2. The Classical Nucleation Theory

If the growth is DIFFUSION-LIMITED,

size distribution

$$\frac{dr}{dt} = \frac{K_D}{r}\left(\frac{1}{r^*} - \frac{1}{r}\right) \longrightarrow \frac{d(\Delta r)}{dt} = \frac{K_D \Delta r}{\bar{r}^2}\left(\frac{2}{\bar{r}} - \frac{1}{r^*}\right)$$

$$\frac{\bar{r}}{r^*} \geq 2 \qquad \frac{d(\Delta r)}{dt} \leq 0$$

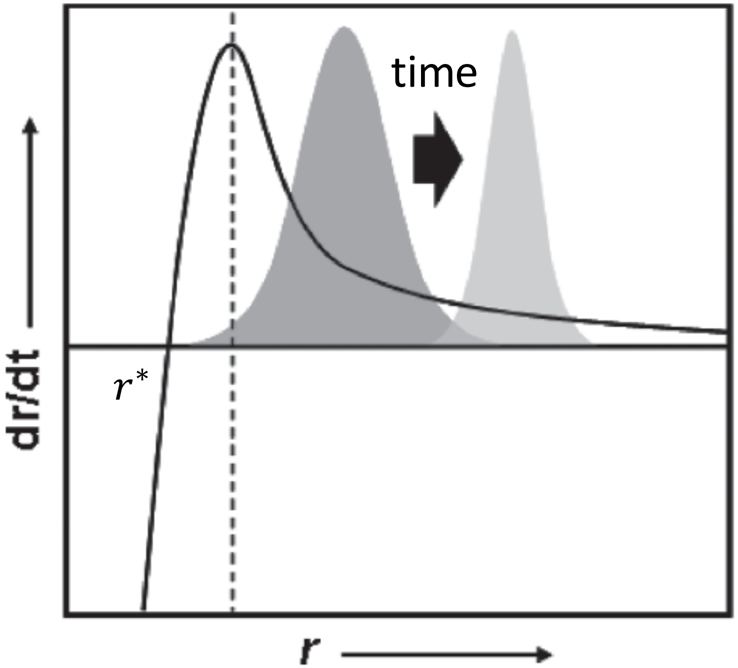
The size distribution will be self-sharpening over time

$$\frac{\bar{r}}{r^*} < 2 \qquad \frac{d(\Delta r)}{dt} > 0$$

The size distribution will become broader over time

Uncommon because in the earlier growth stages S is high, thus r* is small

**FOCUSING REGIME**



# 3.2. The Classical Nucleation Theory

If the growth is REACTION-LIMITED, $D \gg K_D$

$$\frac{dr}{dt} = K_D\left(\frac{1}{r^*} - \frac{1}{r}\right) \longrightarrow \frac{d(\Delta r)}{dt} = \frac{K_D\,\Delta r}{\bar{r}^2}$$

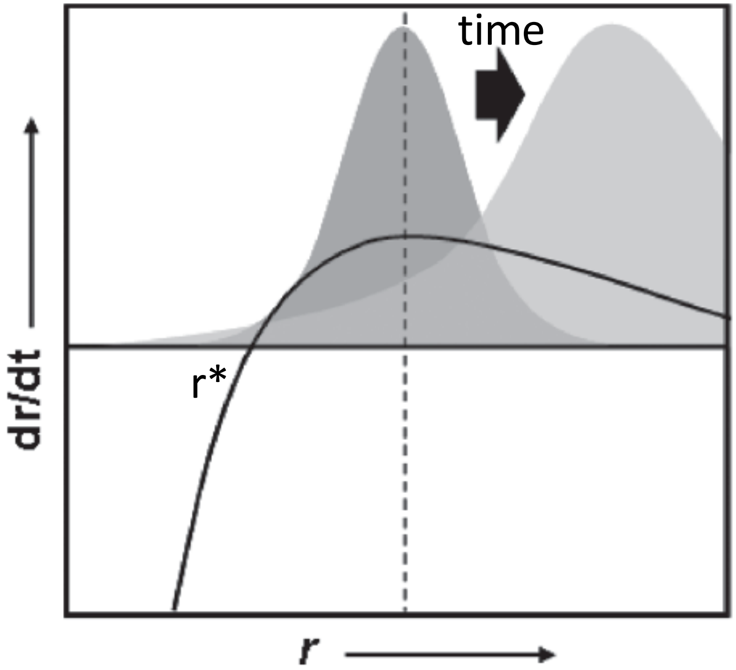
**DEFOCUSING REGIME**

$r = r^*$ $\quad \dfrac{dr}{dt} = 0$

$r < r^*$ $\quad \dfrac{dr}{dt} < 0$ The particle will dissolve

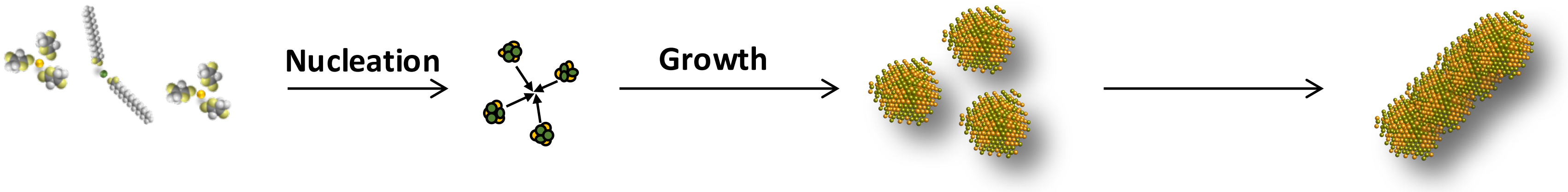
$r > r^*$ $\quad \dfrac{dr}{dt} > 0$ The particle will grow

For any $\bar{r}$ $\quad \dfrac{d(\Delta r)}{dt} > 0$



Bigger particles will grow faster than smaller particles (Ostwald Ripening)

# 3.3. The Non-Classical Nucleation Theory (or Aggregative Nanocrystal Growth )



Nucleation

Growth

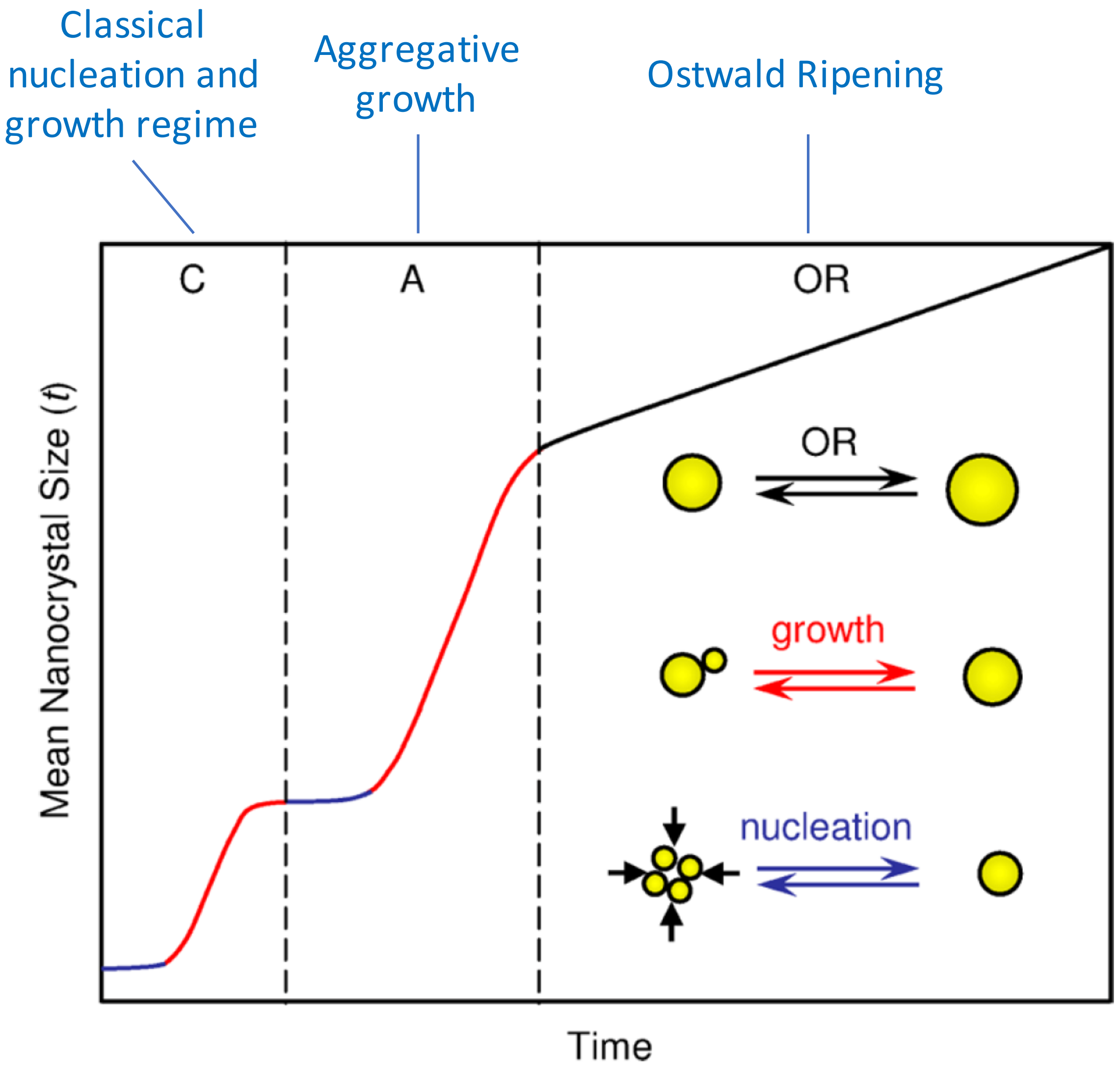
monomers

nuclei/clusters

nanocrystals

The growth proceeds by coalescence of nuclei

# 3.3. The Non-Classical Nucleation Theory


Classical nucleation and growth regime

Aggregative growth

Ostwald Ripening

Buhro et al. Chem. Mater. 2014

## 3.3. The Non-Classical Nucleation Theory

DRIVING FORCES FOR COALESCENCE:

- Van der Waals forces
- Hydrophobic attractions
- Charge-charge interactions
- Dipolar interactions

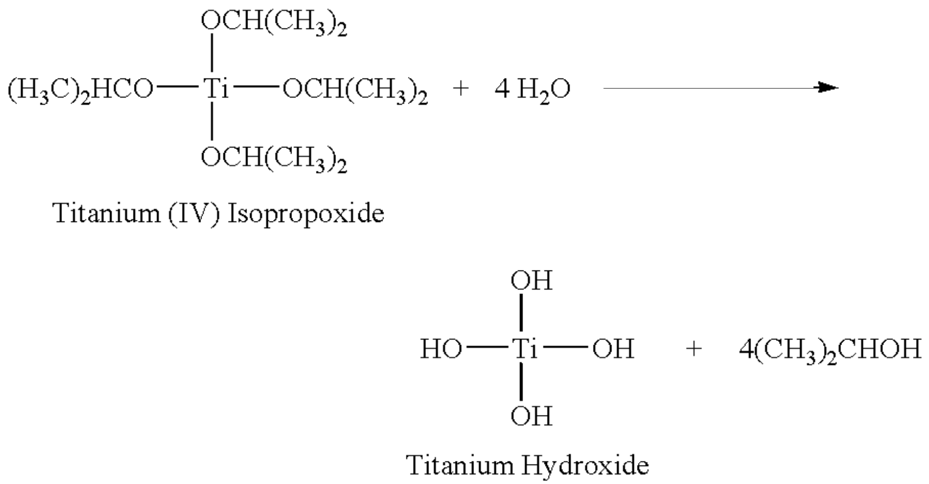
DRIVING FORCE FOR ALIGNMENT:

The thermodynamic driving force for the reduction of the defect-free energy becomes significant at the nanoscale, where it induces the rotational motion of the primary particles and the lattice reconstruction.

# 3.4. Chemical reaction mechanisms

## 3.4.1 Metal Oxides (MO)

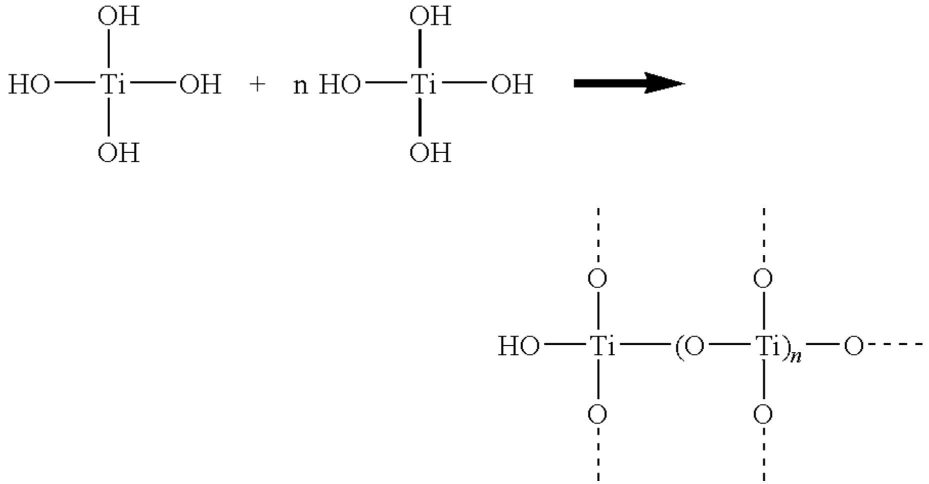
### Sol-gel synthesis of MO thin films: Hydrolysis-Condensation Reaction



$$(H_3C)_2HCO-\underset{OCH(CH_3)_2}{\overset{OCH(CH_3)_2}{Ti}}-OCH(CH_3)_2 + 4\,H_2O \longrightarrow HO-\underset{OH}{\overset{OH}{Ti}}-OH + 4(CH_3)_2CHOH$$

Titanium (IV) Isopropoxide

Titanium Hydroxide

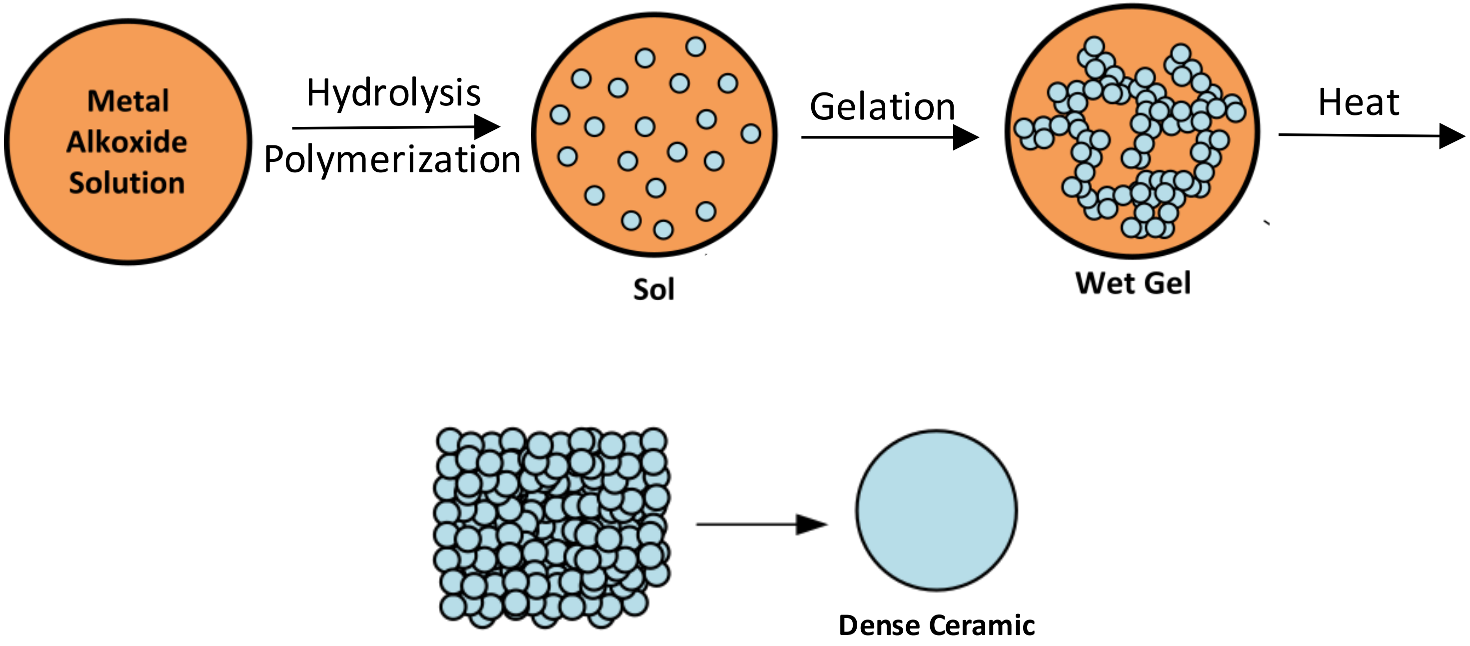


$$HO-\underset{OH}{\overset{OH}{Ti}}-OH + n\ HO-\underset{OH}{\overset{OH}{Ti}}-OH \longrightarrow HO-\underset{O}{\overset{O}{Ti}}-(O-\underset{O}{\overset{O}{Ti}})_n-O\text{- - - -}$$

# 3.4. Chemical reaction mechanisms

## 3.4.1 Metal Oxides (MO)

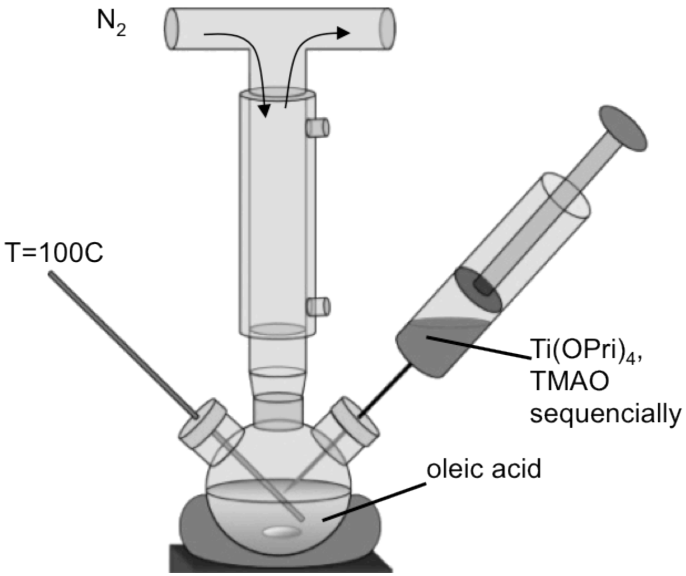
### Sol-gel synthesis of MO thin films: Hydrolysis-Condensation Reaction

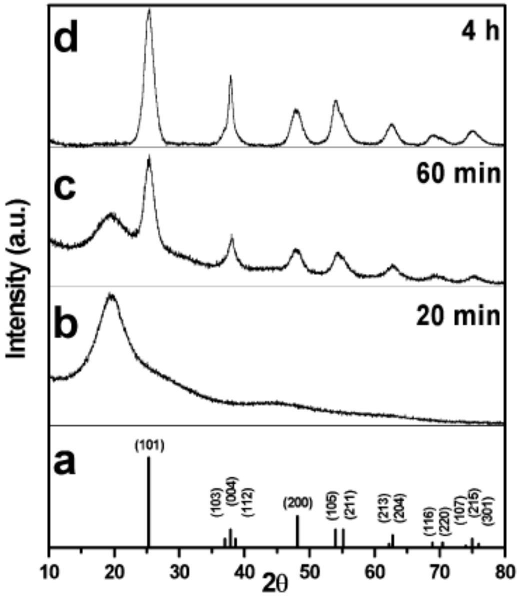


# 3.4. Chemical reaction mechanisms

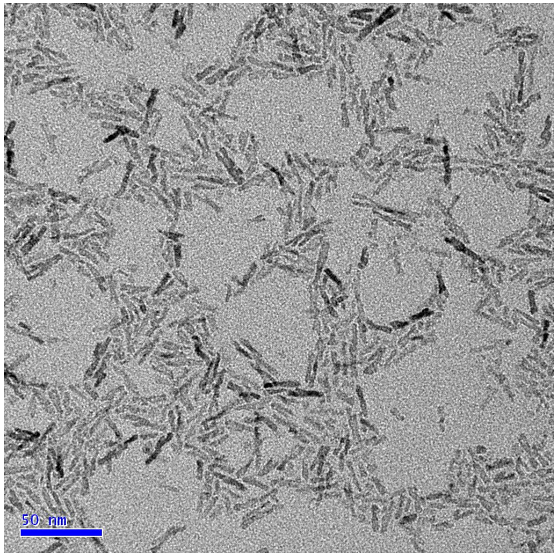
## 3.4.1 Metal Oxides (MO)

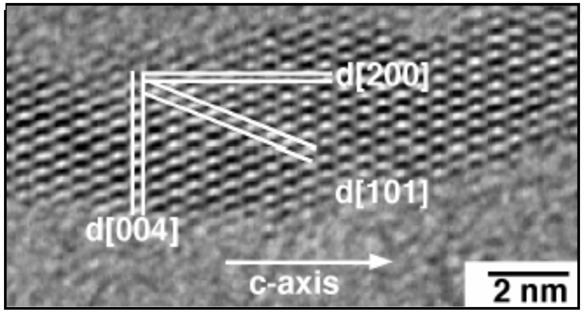
## Synthesis of anatase $TiO_2$ nanorods





To note the sharper (004) peak





REAGENTS
Titanium tetraisopropoxide: Ti precursor
Trimethylamino-*N*-oxide (TMAO: base to catalyze hydrolysis)
Oleic acid: ligand
Ethanol: non-solvent for clean-up
Hexane: solvent

Cozzoli et al. *JACS* (**2003**)

# 3.4. Chemical reaction mechanisms

## 3.4.1 Metal Oxides (MO)

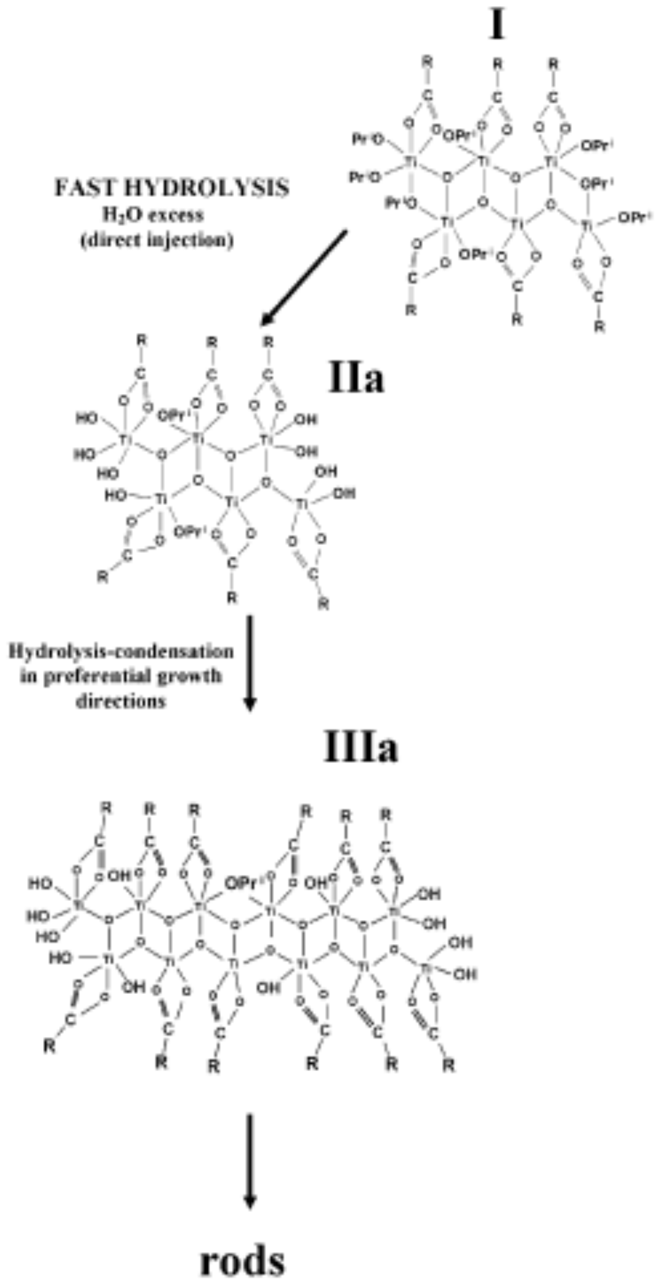
$$Ti(OPr^{i})_4 + H_2O \xrightarrow[\text{TMAO}]{\text{Oleic Acid}} TiO_2 \text{ NANORODS}$$

Hydrolysis/condensation
with Oleic Acid as ligand

$$Ti(OR)_4 + xH_2O \rightarrow Ti(OH)_x(OR)_{4-x} + xROH \qquad (1)$$

$$(OR)_{4-x}(OH)_{x-1}Ti{-}OH + XO{-}Ti(OH)_x(OR)_{3-x} \rightarrow$$
$$(OR)_{4-x}(OH)_{x-1}\mathbf{Ti{-}O{-}Ti}(OR)_{3-x}(OH)_x + XOH \quad (2)$$

where $R = -CH(CH_3)_2$; $X = H, R$.



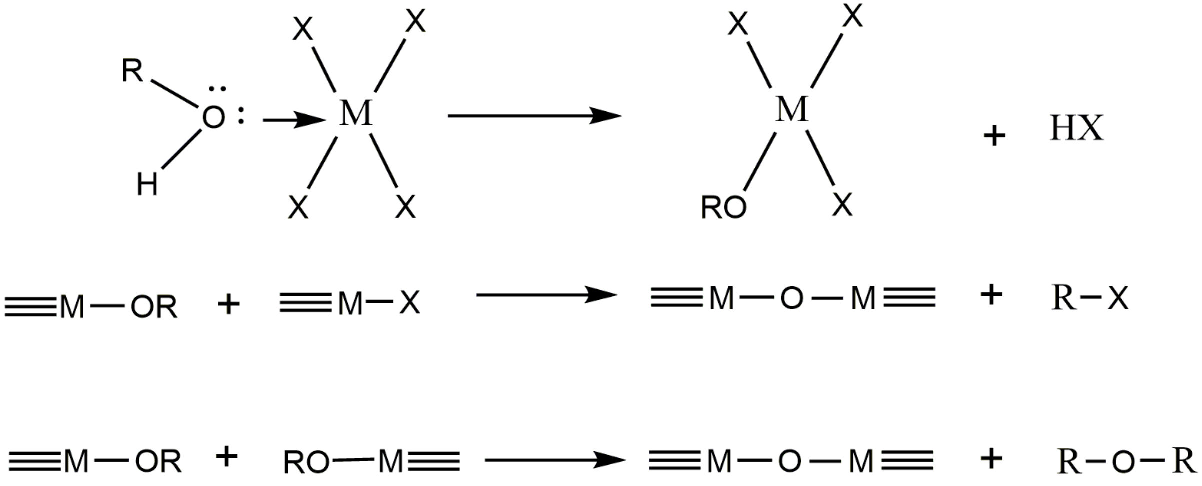
## 3.4. Chemical reaction mechanisms

### 3.4.1 Metal Oxides (MO)

Some typical reactions for the synthesis of metal oxide nanocrystals

- **Alcoholysis**:

- Metal halides and alcohols



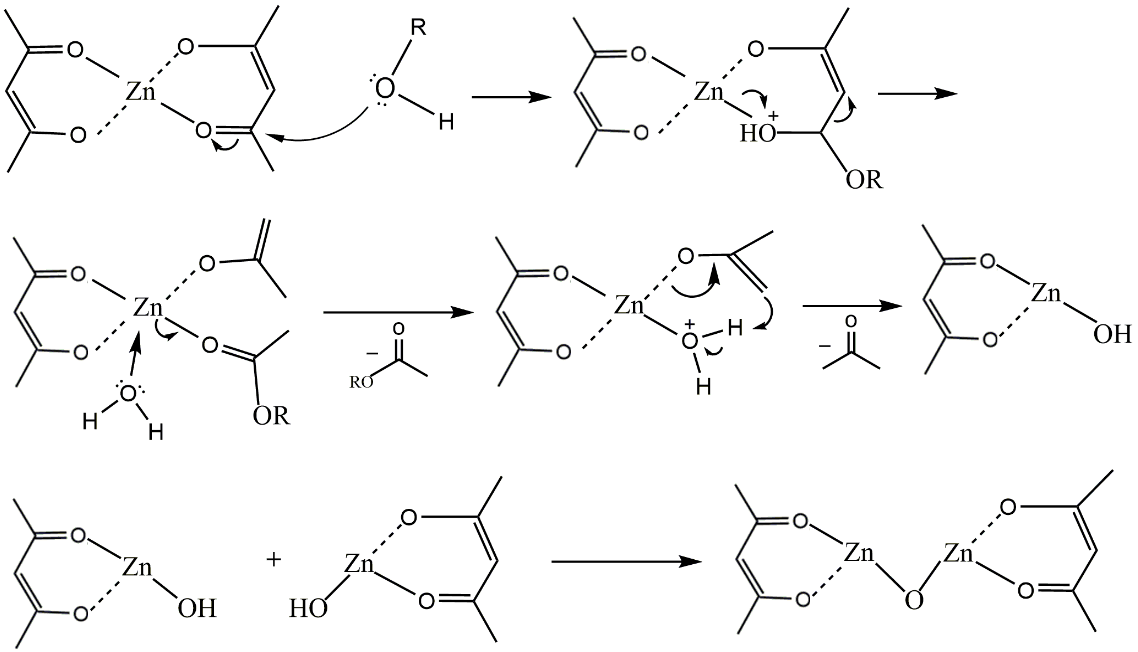
# 3.4. Chemical reaction mechanisms

## 3.4.1 Metal Oxides (MO)

### Some typical reactions for the synthesis of metal oxide nanocrystals

- **Alcoholysis**:

- Metal acetylacetonates and alcohols



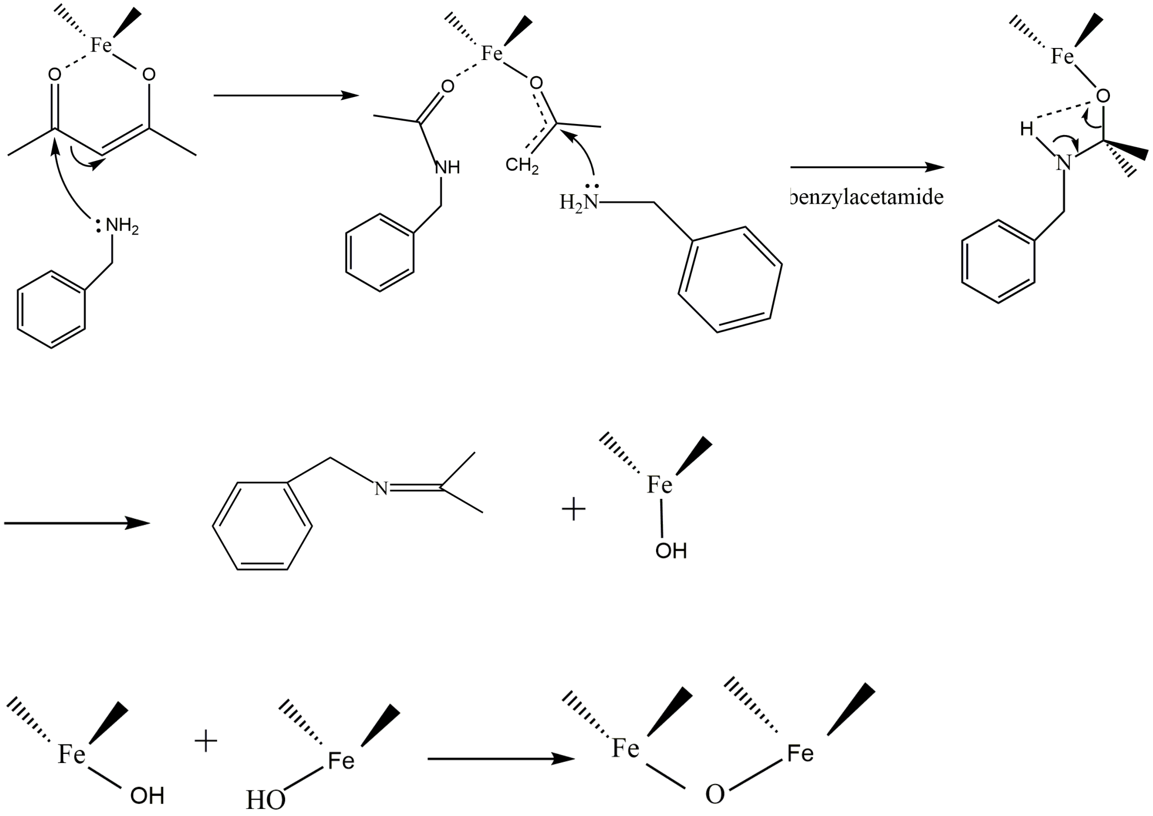
# 3.4. Chemical reaction mechanisms

## 3.4.1 Metal Oxides (MO)

### Some typical reactions for the synthesis of metal oxide nanocrystals

- **Aminolysis**:

- Metal acetylacetonates and amines

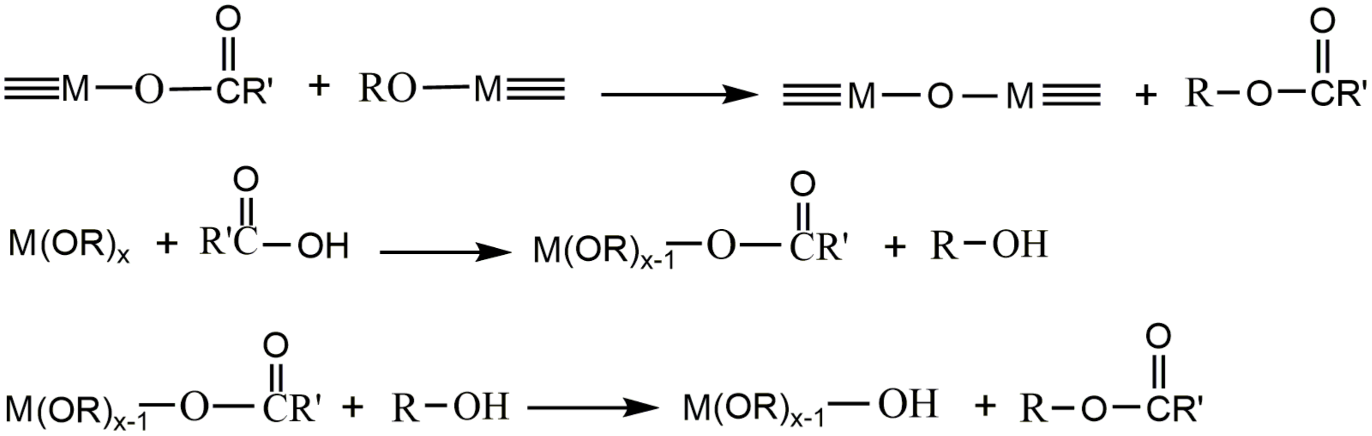


# 3.4. Chemical reaction mechanisms

## 3.4.1 Metal Oxides (MO)

### Some typical reactions for the synthesis of metal oxide nanocrystals

- **Ester Elimination**:



$$\equiv M-O-\overset{O}{\overset{\parallel}{C}}R' + RO-M\equiv \longrightarrow \equiv M-O-M\equiv + R-O-\overset{O}{\overset{\parallel}{C}}R'$$

$$M(OR)_x + R'\overset{O}{\overset{\parallel}{C}}-OH \longrightarrow M(OR)_{x-1}-O-\overset{O}{\overset{\parallel}{C}}R' + R-OH$$

$$M(OR)_{x-1}-O-\overset{O}{\overset{\parallel}{C}}R' + R-OH \longrightarrow M(OR)_{x-1}-OH + R-O-\overset{O}{\overset{\parallel}{C}}R'$$

## 3.4. Chemical reaction mechanisms

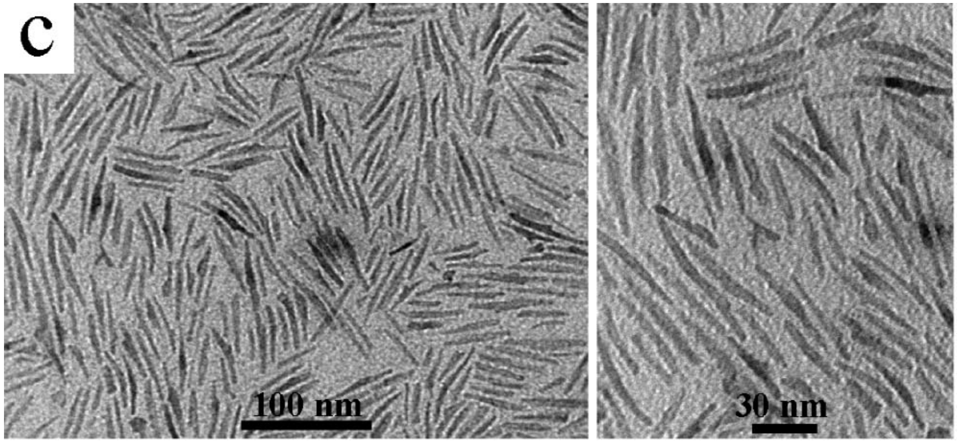
### 3.4.1 Metal Oxides (MO)

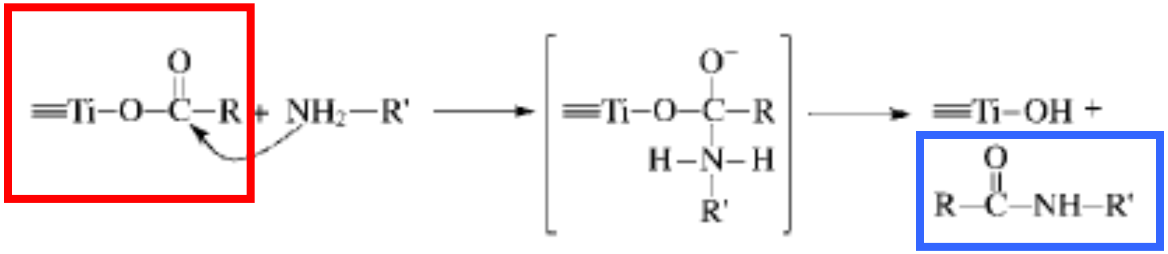
### Synthesis of brookite $TiO_2$ nanorods

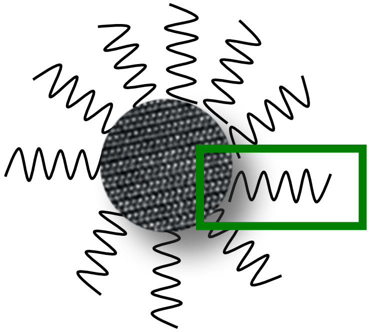
**REAGENTS**

$TiCl_4$
oleyalmine
oleic acid

$\xrightarrow[\text{1h}]{\text{290C}}$







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aminolysis of Ti-oleate complex at 280C

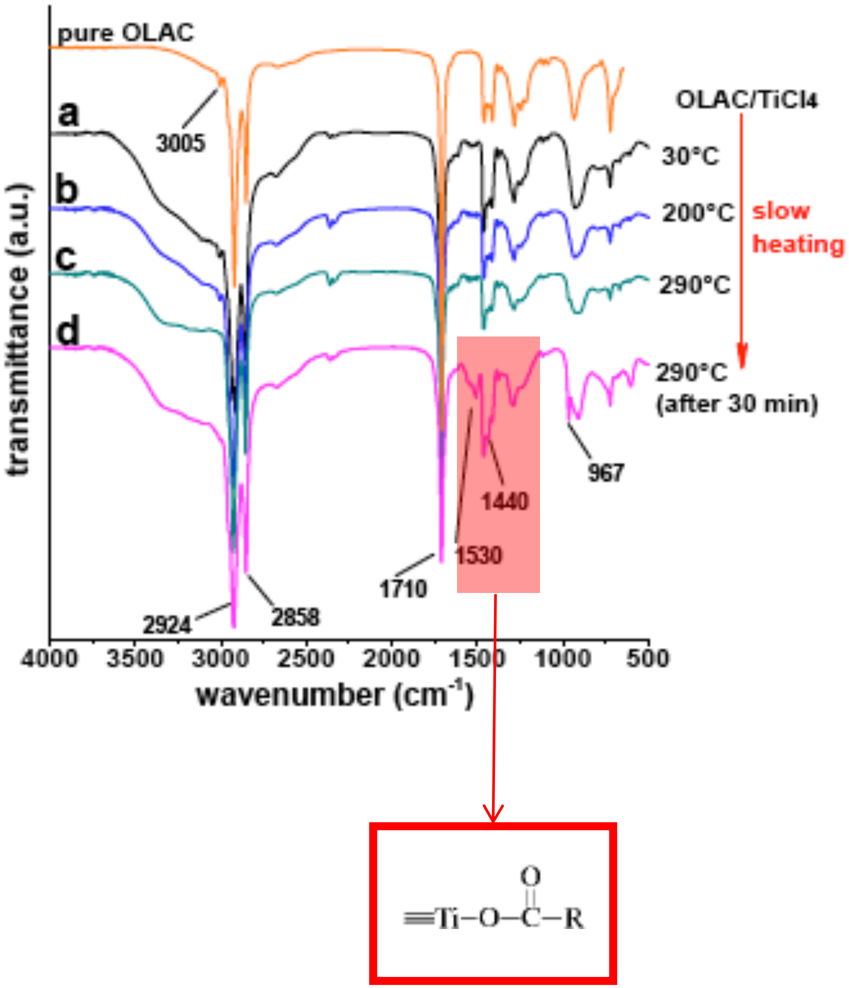
Buonsanti et al. *JACS* (**2008**)

# 3.4. Chemical reaction mechanisms

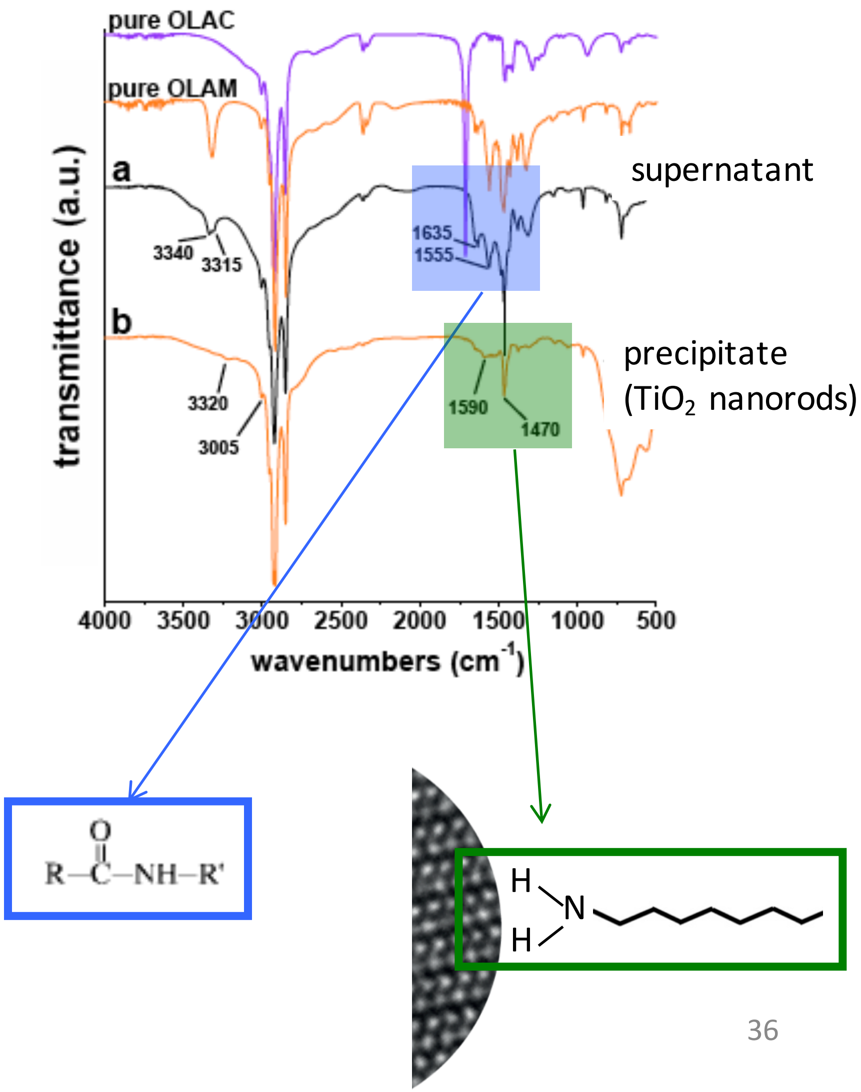
## 3.4.1 Metal Oxides (MO)

Synthesis of brookite $TiO_2$ nanorods

**FTIR on $RCOOH+TiCl_4$ mixture**



**FTIR on $RCOOH+RNH_2+TiCl_4$ mixture**



Buonsanti et al. *JACS* (**2008**)

# 3.4. Chemical reaction mechanisms

## 3.4.1 Metal Oxides (MO)

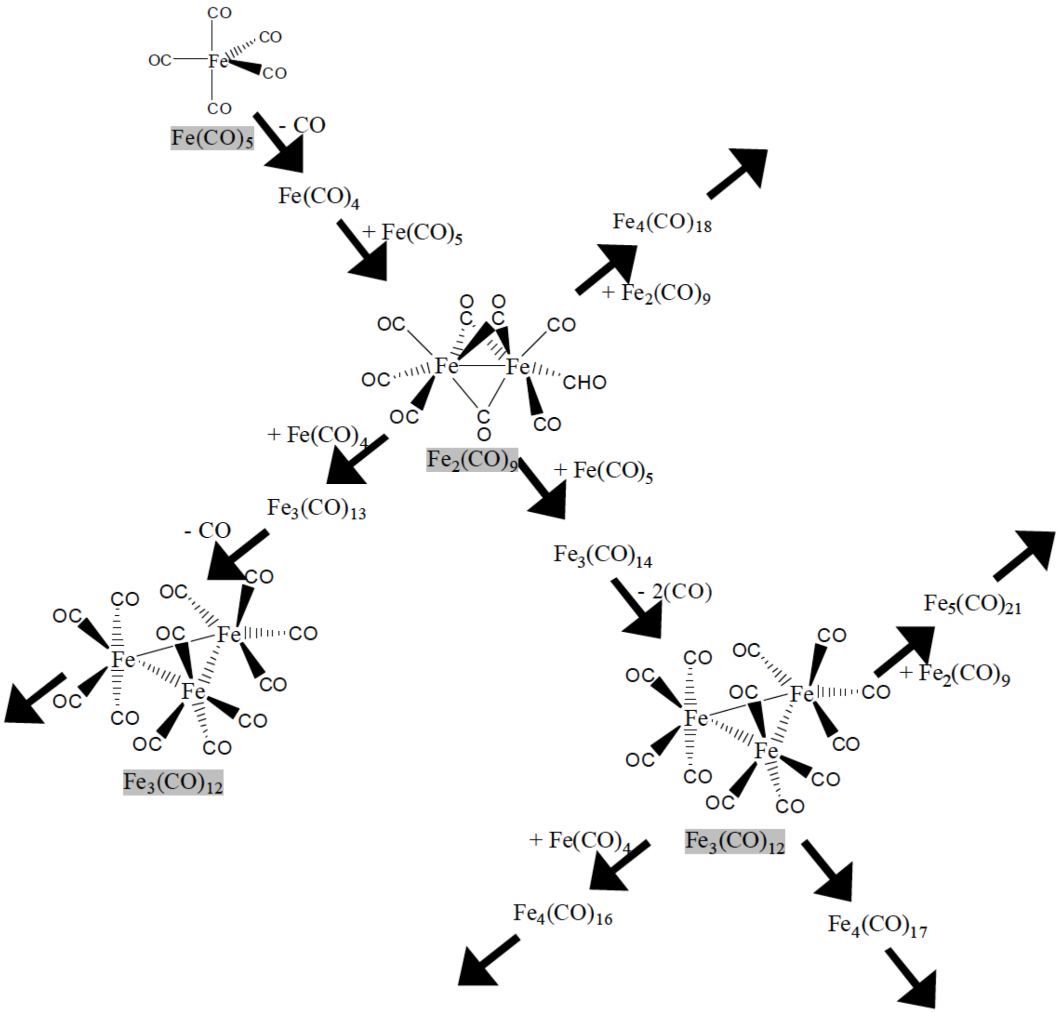
### Some typical reactions for the synthesis of metal oxide nanocrystals

- **Thermal Decomposition**:

  Organo-metallic precursor:

  $Fe(CO)_5 \longrightarrow Fe + 5CO$

  Typical solvent: octadecene
  Typical ligand: oleic acid



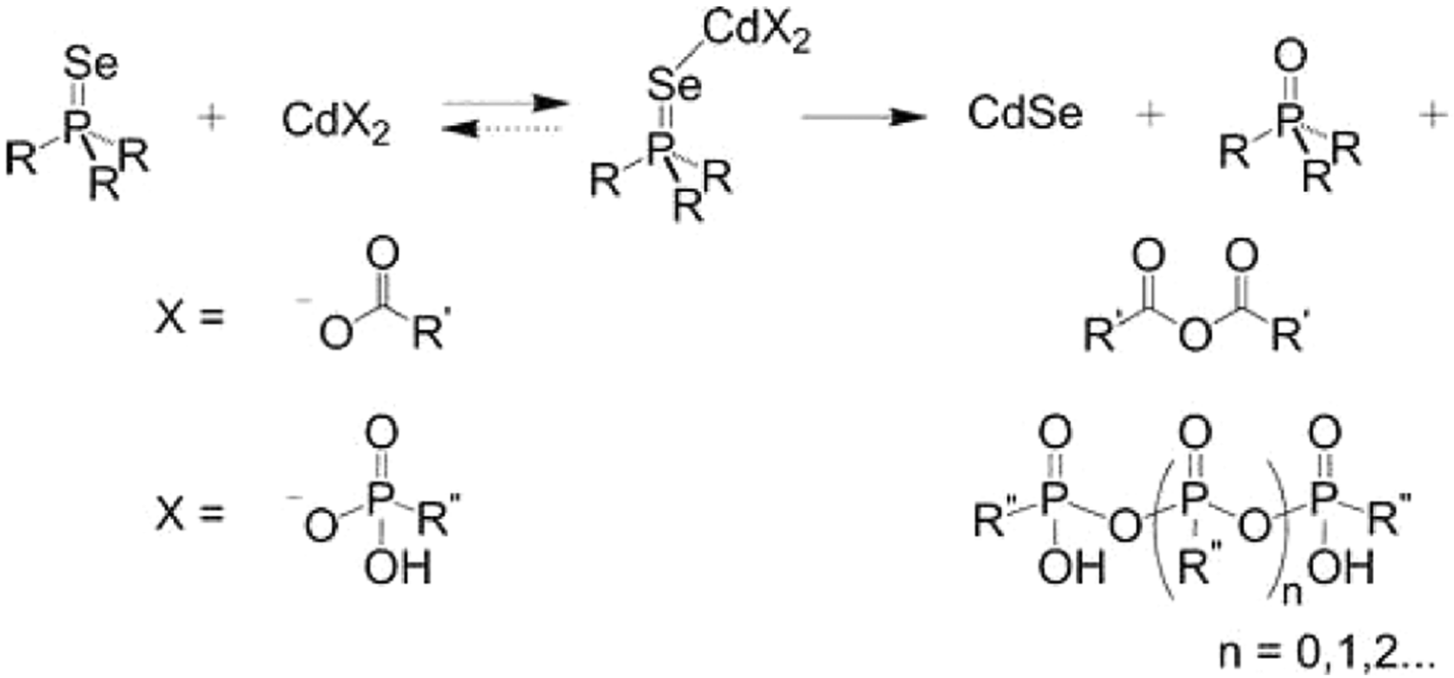
# 3.4. Chemical reaction mechanisms

## 3.4.2 Semiconductors (II-VI, III-V, IV-VI)

It usually occurs by thermal decomposition of highly reactive precursors

Typical synthesis for CdSe quantum dots:

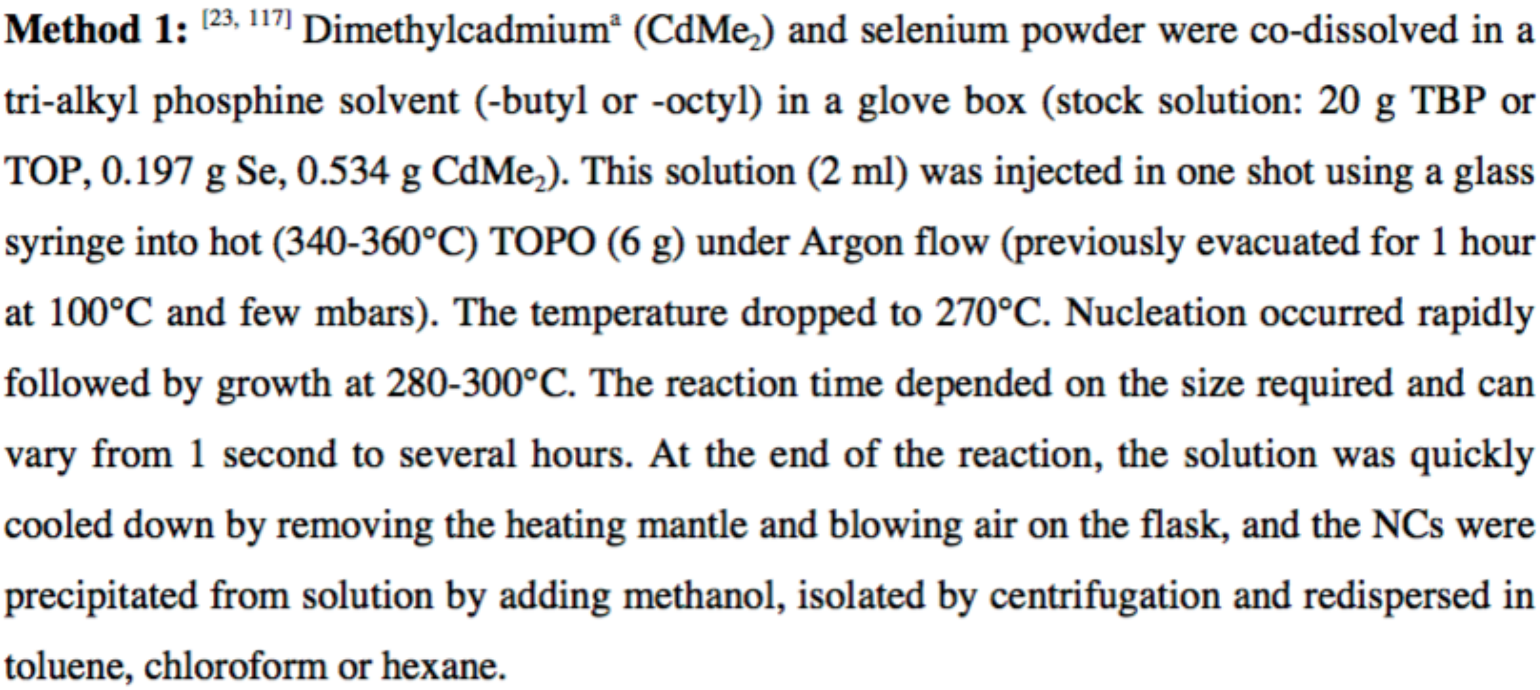
(i.e. TOP-Se)



# 3.4. Chemical reaction mechanisms

## 3.4.2 Semiconductors (II-VI, III-V, IV-VI)

### Other examples of synthesis for CdSe QDs



**Method 1:** [23, 117] Dimethylcadmium[a] ($CdMe_2$) and selenium powder were co-dissolved in a tri-alkyl phosphine solvent (-butyl or -octyl) in a glove box (stock solution: 20 g TBP or TOP, 0.197 g Se, 0.534 g $CdMe_2$). This solution (2 ml) was injected in one shot using a glass syringe into hot (340-360°C) TOPO (6 g) under Argon flow (previously evacuated for 1 hour at 100°C and few mbars). The temperature dropped to 270°C. Nucleation occurred rapidly followed by growth at 280-300°C. The reaction time depended on the size required and can vary from 1 second to several hours. At the end of the reaction, the solution was quickly cooled down by removing the heating mantle and blowing air on the flask, and the NCs were precipitated from solution by adding methanol, isolated by centrifugation and redispersed in toluene, chloroform or hexane.

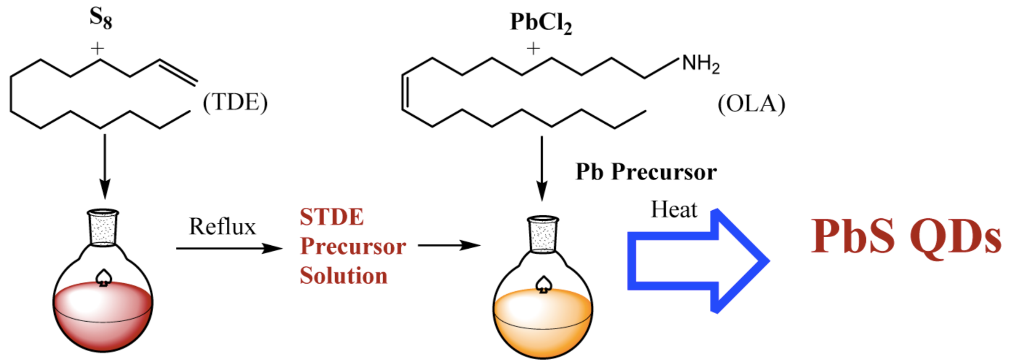
TBP = tributylphosphine
TOP = trioctylphosphine
TOPO = trioctlyphosphine oxide

Murray, Norris, Bawendi JACS (1993)

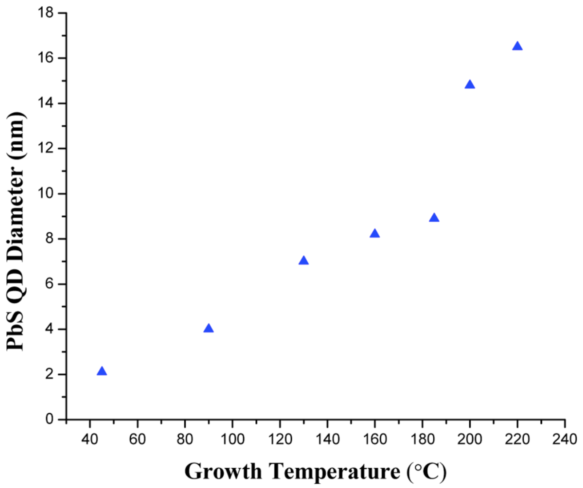
# 3.4. Chemical reaction mechanisms

## 3.4.2 Semiconductors (II-VI, III-V, IV-VI)

## Synthesis of PbS QDs



Noninjection Synthesis



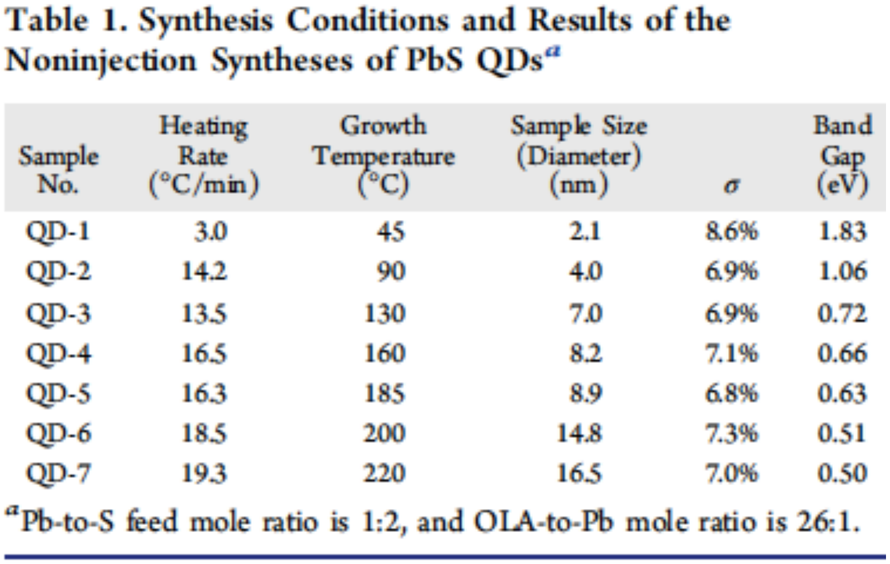


**Table 1. Synthesis Conditions and Results of the Noninjection Syntheses of PbS QDs**$^a$

| Sample No. | Heating Rate (°C/min) | Growth Temperature (°C) | Sample Size (Diameter) (nm) | σ | Band Gap (eV) |
|---|---|---|---|---|---|
| QD-1 | 3.0 | 45 | 2.1 | 8.6% | 1.83 |
| QD-2 | 14.2 | 90 | 4.0 | 6.9% | 1.06 |
| QD-3 | 13.5 | 130 | 7.0 | 6.9% | 0.72 |
| QD-4 | 16.5 | 160 | 8.2 | 7.1% | 0.66 |
| QD-5 | 16.3 | 185 | 8.9 | 6.8% | 0.63 |
| QD-6 | 18.5 | 200 | 14.8 | 7.3% | 0.51 |
| QD-7 | 19.3 | 220 | 16.5 | 7.0% | 0.50 |

$^a$Pb-to-S feed mole ratio is 1:2, and OLA-to-Pb mole ratio is 26:1.

Pan et al. *J. Chem. Edu.* (**2015**)

# 3.4. Chemical reaction mechanisms

## 3.4.3 Metals

### Representative chemical reduction schematics for metallic nanoparticles

**Pathway 1: reduction followed by bonding of atoms**

$$M^{x+} + xe \rightarrow M^0$$

$$M^0 + M_n^0 \rightarrow M_{n+1}^0$$

**Pathway 2: bonding of atoms as ions followed by reduction**

$$M^{x+} + e \rightarrow M^{(x-1)+}$$

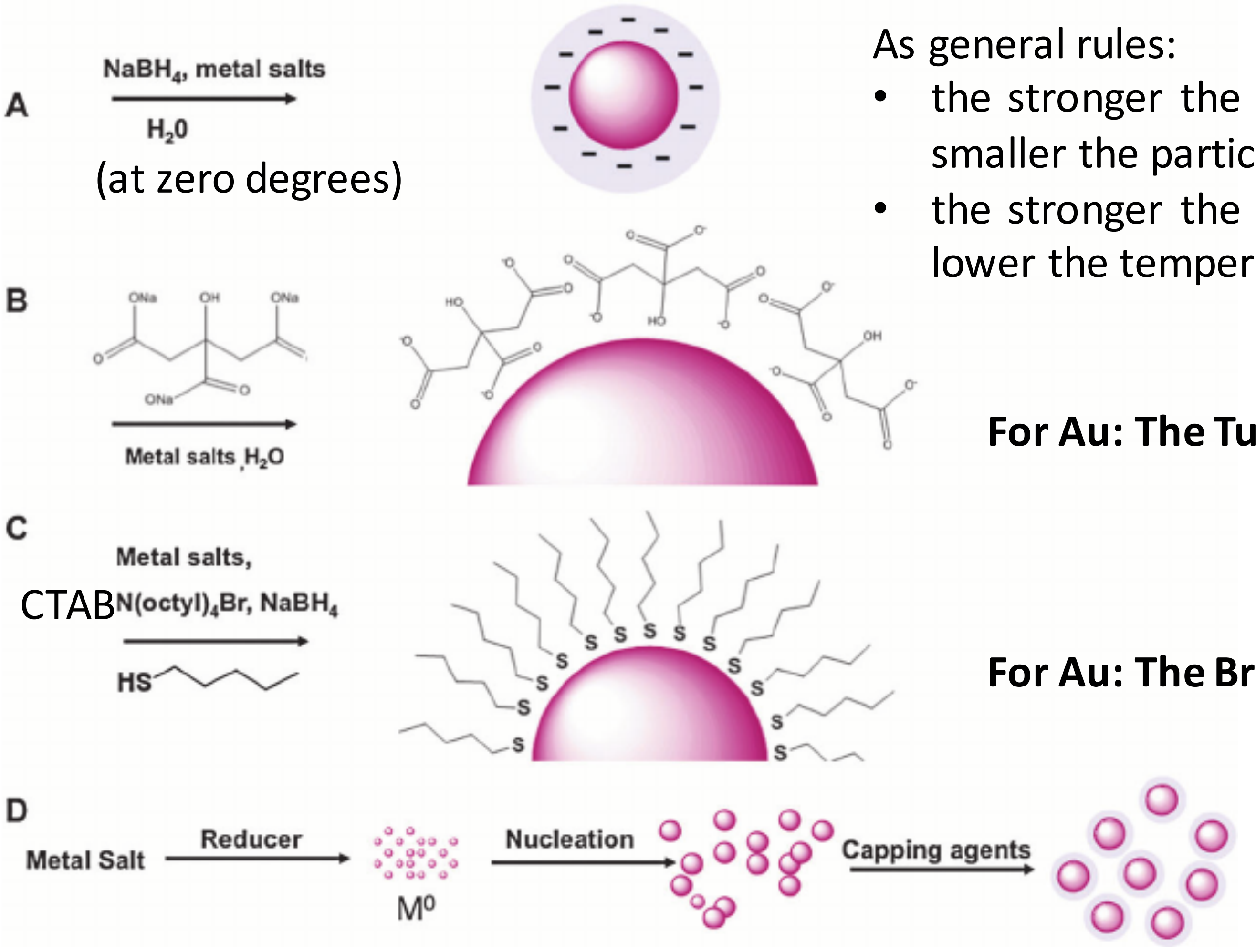
$$M^{(x-1)+} + M^{(x-1)+} \rightarrow M_2{}^{(2x-2)+}$$

$$M_2{}^{(2x-2)+} + xe \rightarrow M_x$$

## 3.4. Chemical reaction mechanisms

### 3.4.3 Metals

**Representative chemical reduction schematics for metallic nanoparticles**



(at zero degrees)

CTAB

As general rules:

- the stronger the reducing agent is, the smaller the particles are
- the stronger the reducing agent is, the lower the temperature for nucleation is

**For Au: The Turkevich Method**

**For Au: The Brust Method**

# 3.4. Chemical reaction mechanisms

## 3.4.3 Metals

## Synthesis of gold nanoparticles

- **the Turkevich Method** (1951, reduction of tetrachloroaurate by trisodium citrate)

$$2AuCl_4^- + Ctr^{3-} + 2H_2O \xrightarrow{100\ ^\circ C} 2Au$$
$$+ 3CH_2O + 3CO_2 \uparrow + 8Cl^- + 3H^+$$

- **Reduction of gold chloride by oleylamine**

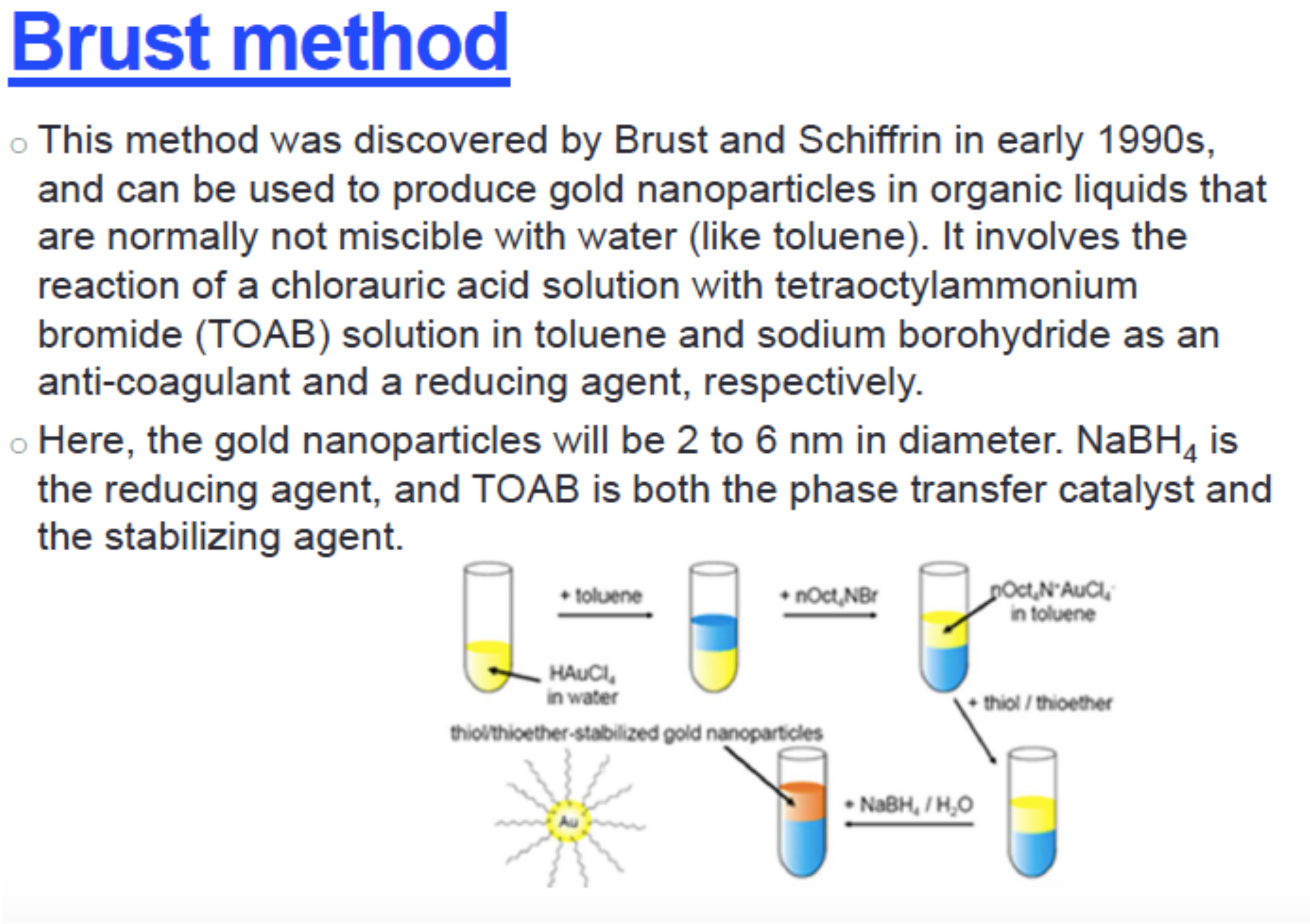
some reduced N-species

$$\underset{\text{in toluene}}{AuCl_3} + \text{oleylamine} \xrightarrow{80^\circ C} Au + \begin{matrix} NH{=}CH{-}R \\ N{\equiv}C{-}R \end{matrix}$$

# 3.4. Chemical reaction mechanisms

## 3.4.3 Metals

Synthesis of gold nanoparticles



## Brust method

- This method was discovered by Brust and Schiffrin in early 1990s, and can be used to produce gold nanoparticles in organic liquids that are normally not miscible with water (like toluene). It involves the reaction of a chlorauric acid solution with tetraoctylammonium bromide (TOAB) solution in toluene and sodium borohydride as an anti-coagulant and a reducing agent, respectively.
- Here, the gold nanoparticles will be 2 to 6 nm in diameter. $NaBH_4$ is the reducing agent, and TOAB is both the phase transfer catalyst and the stabilizing agent.

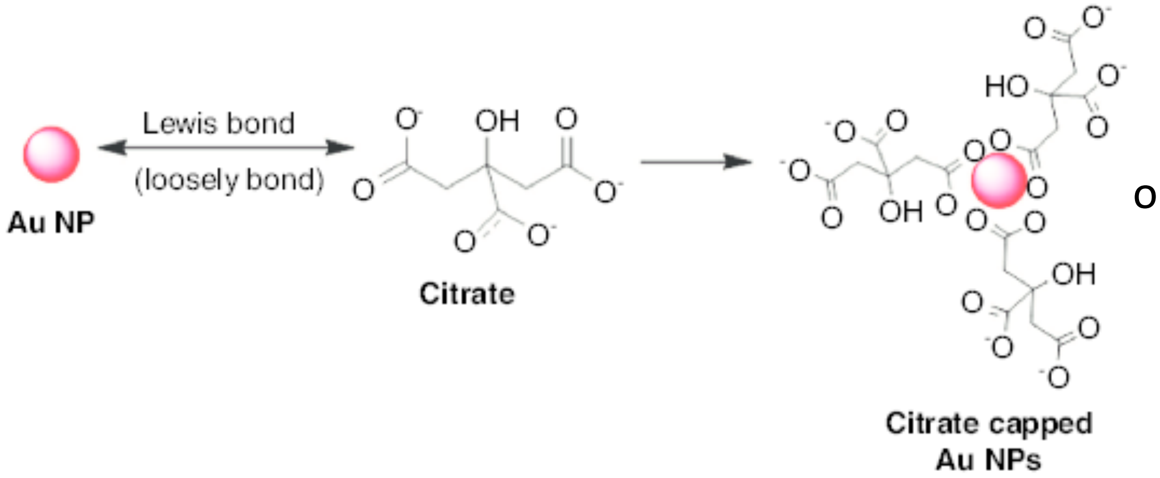
# 3.4. Chemical reaction mechanisms

## 3.4.3 Metals

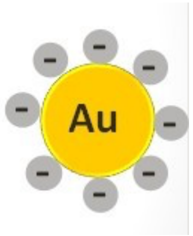
## Synthesis of gold nanoparticles

Thiols have a stronger interaction with the surface of gold, so the colloidal stability is better

$$HAuCl_4 + 3\,Na_3C_6H_5O_7 \longrightarrow 2Au + 3\,Na_3C_5H_6O_5 + 3NaCl + 5Cl^- + 5H^+ + 3CO_2$$

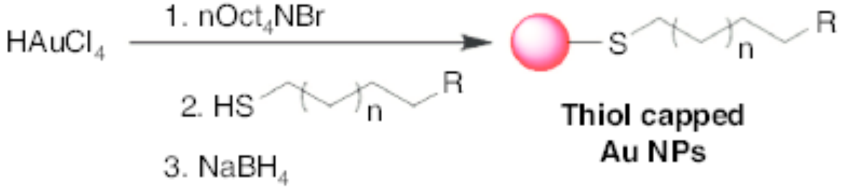


or



**in water**

It is not clear what is the main mode of surface passivation

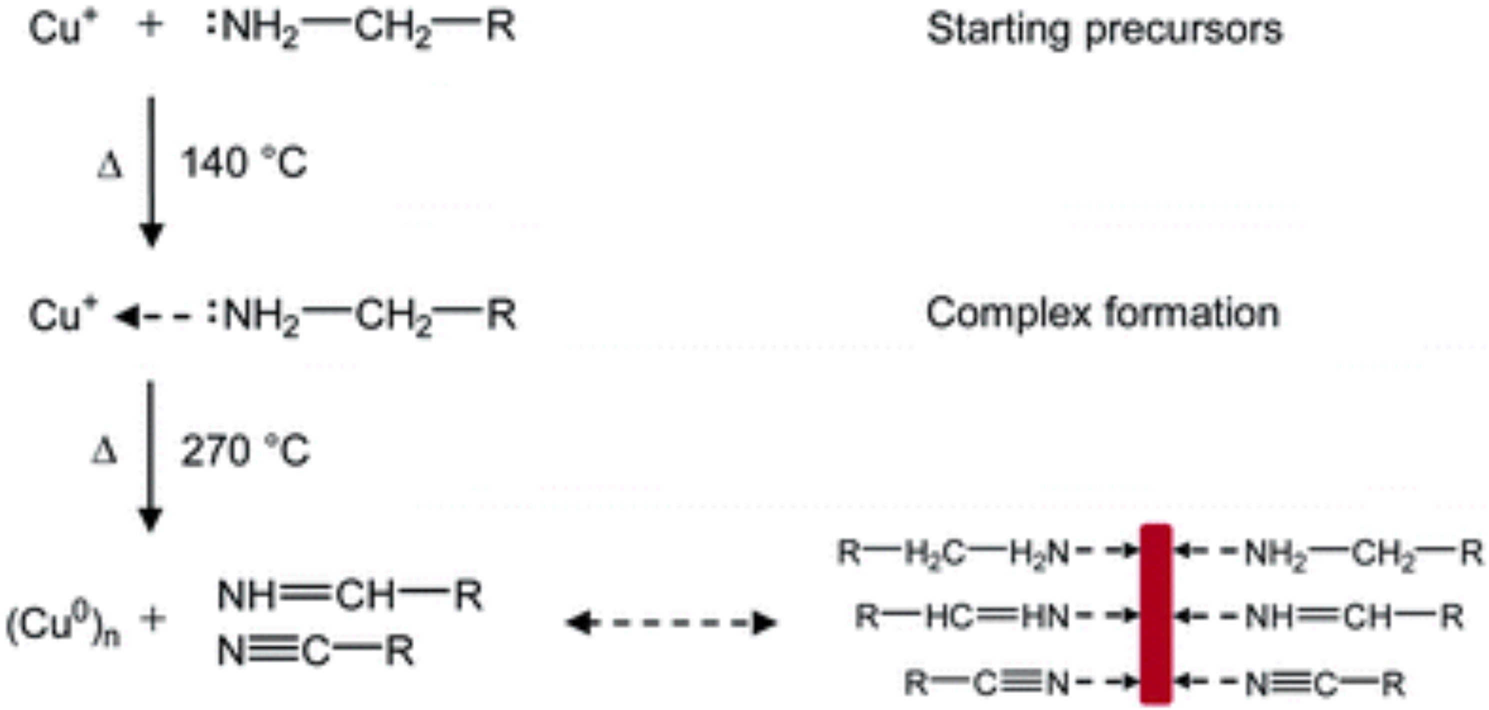


**in toluene**

# 3.4. Chemical reaction mechanisms

## 3.4.3 Metals

## Synthesis of copper nanoparticles

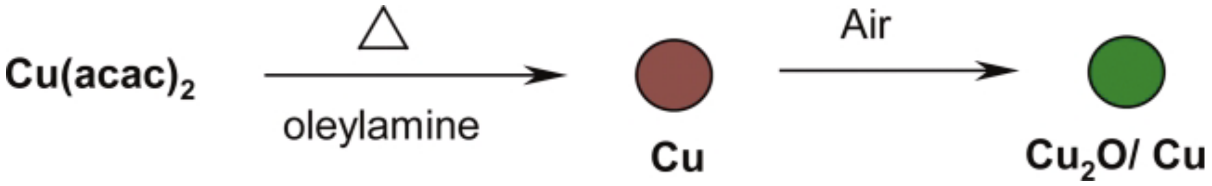


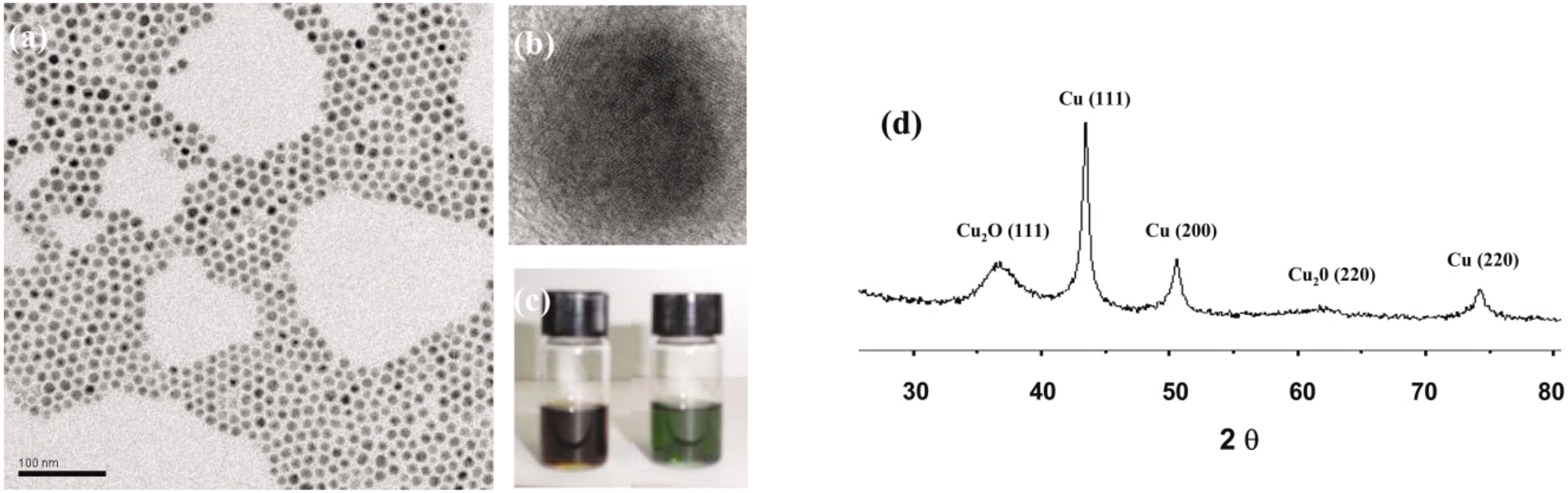
Sreethawong et al. J. Mater. Chem. A 2014, 2, 3417

# 3.4. Chemical reaction mechanisms

## 3.4.3 Metals

## Synthesis of copper nanoparticles





Hyeon et al *Chem Comm* (**2004**)

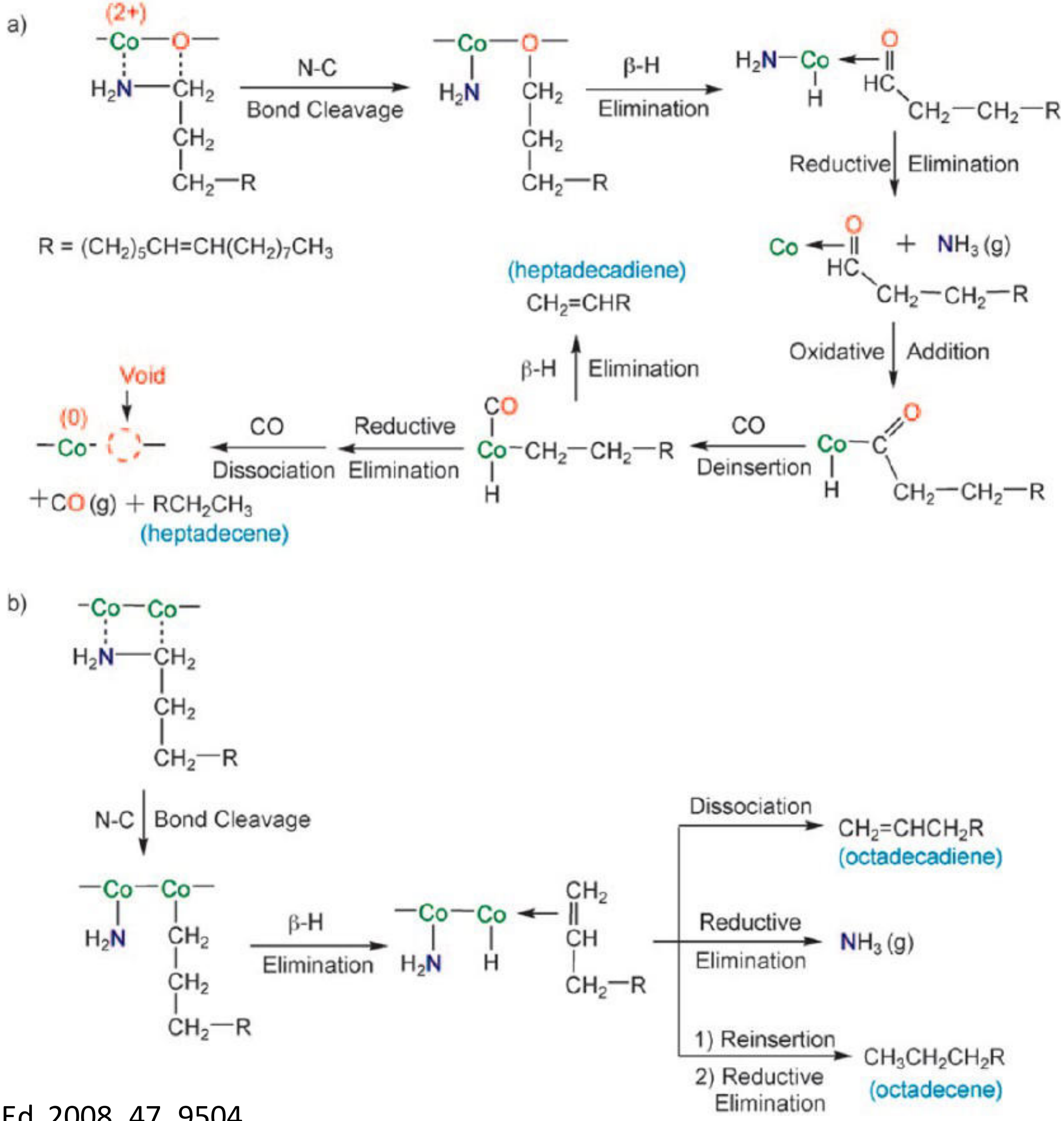
# 3.4. Chemical reaction mechanisms

## 3.4.3 Metals

Some examples

**COBALT**

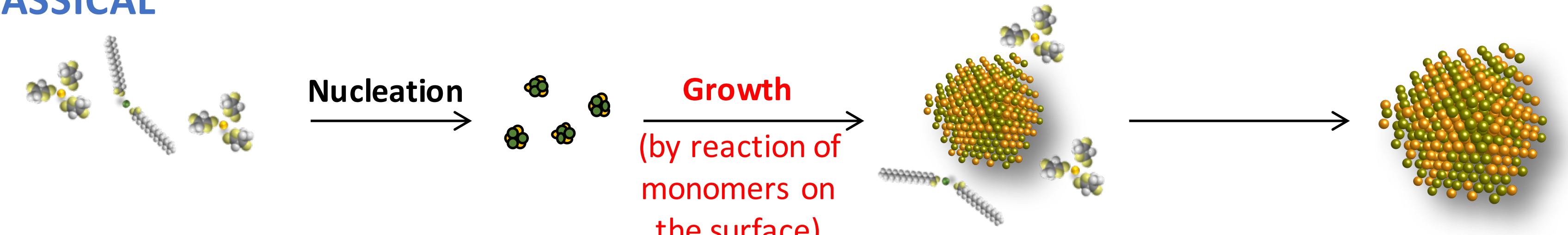
- **CoO reduction by oleylamine**



Park et al. et al. Angew. Chemie. Int. Ed. 2008, 47, 9504

# 3.5. In-situ studies

## CLASSICAL



Nucleation

Growth

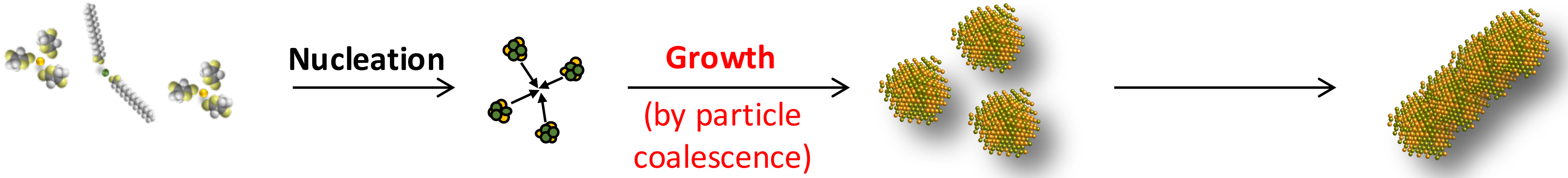
(by reaction of monomers on the surface)

monomers

nuclei

nanocrystals

## NON-CLASSICAL



Nucleation

Growth

(by particle coalescence)

monomers

nuclei/clusters

nanocrystals

### Chemical reactions

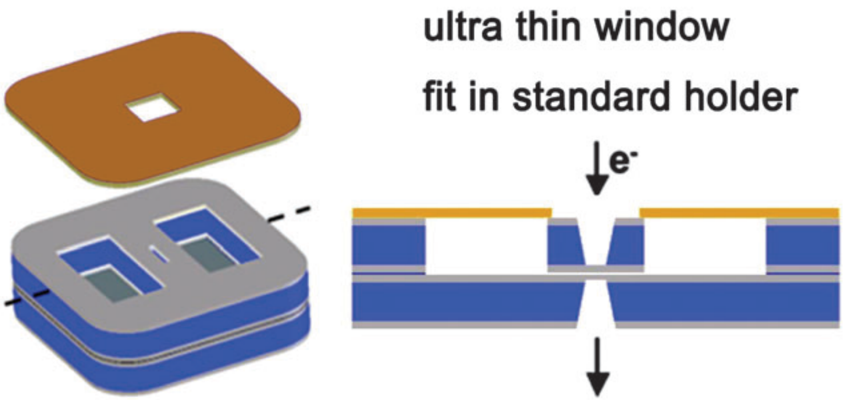
- ✓ FT-IR
- ✓ NMR
- ✓ X-Ray Absorption

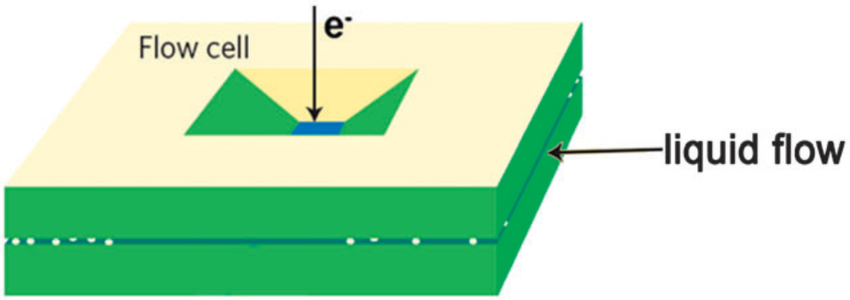
### Nucleation and growth

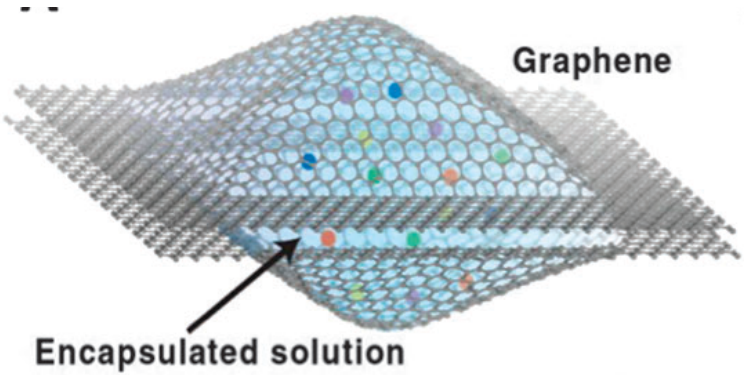
- ✓ UV-Vis
- ✓ **TEM**
- ✓ XRD
- ✓ X-Ray Absorption

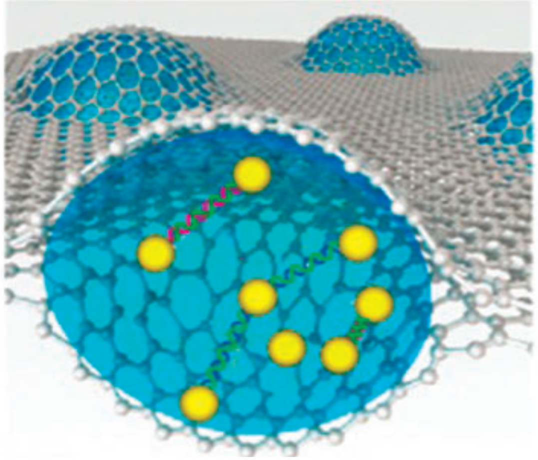
# 3.5. In-situ studies

## 3.5.1. In-situ transmission electron microscopy







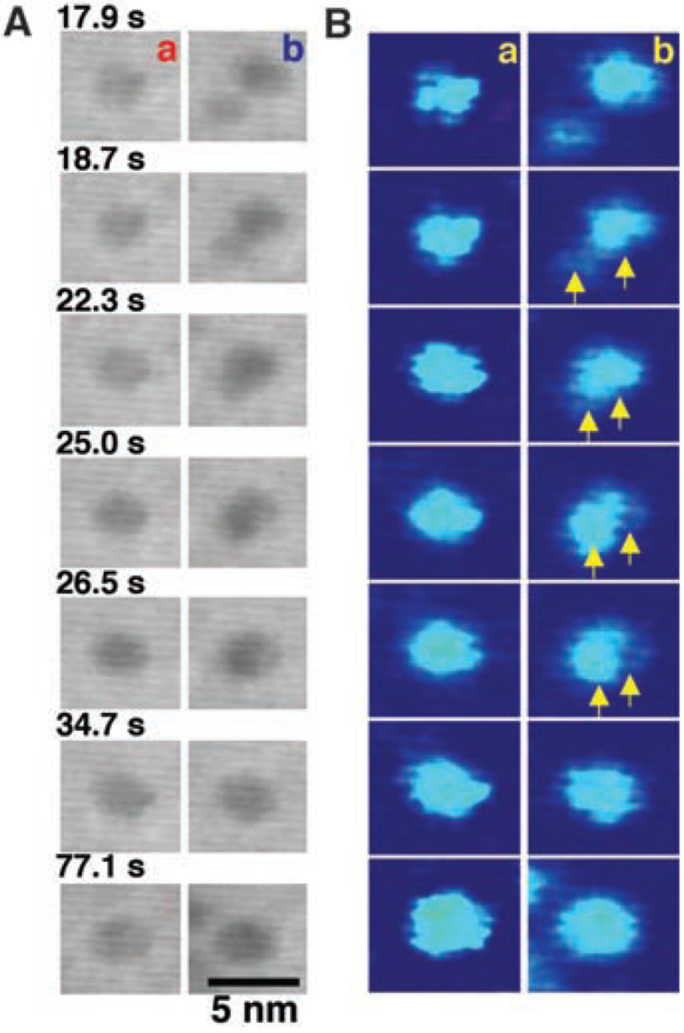


Zheng, Alivisatos et al. Science (2009), Science (2011), Science (2015)

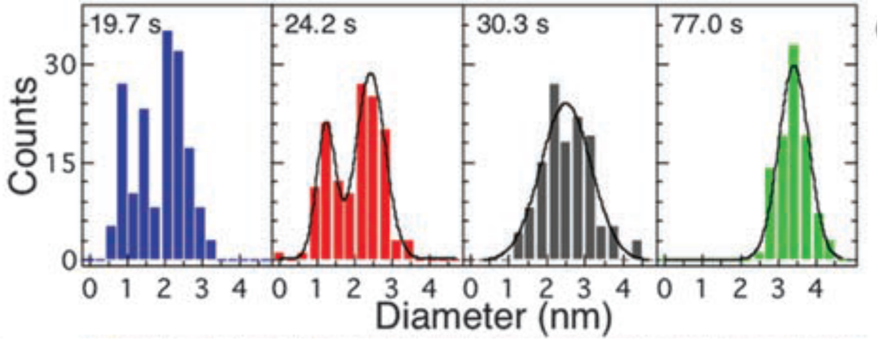
# 3.5. In-situ studies

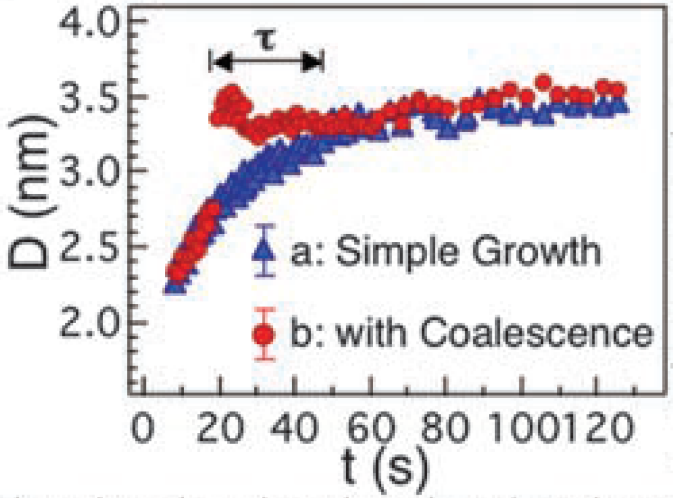
## 3.5.1. In-situ transmission electron microscopy

CORROBORATING THE NON-CLASSICAL GROWTH



A. Growth of Pt nanoparticles by monomer addition
B. Growth of Pt nanoparticles by coalescence



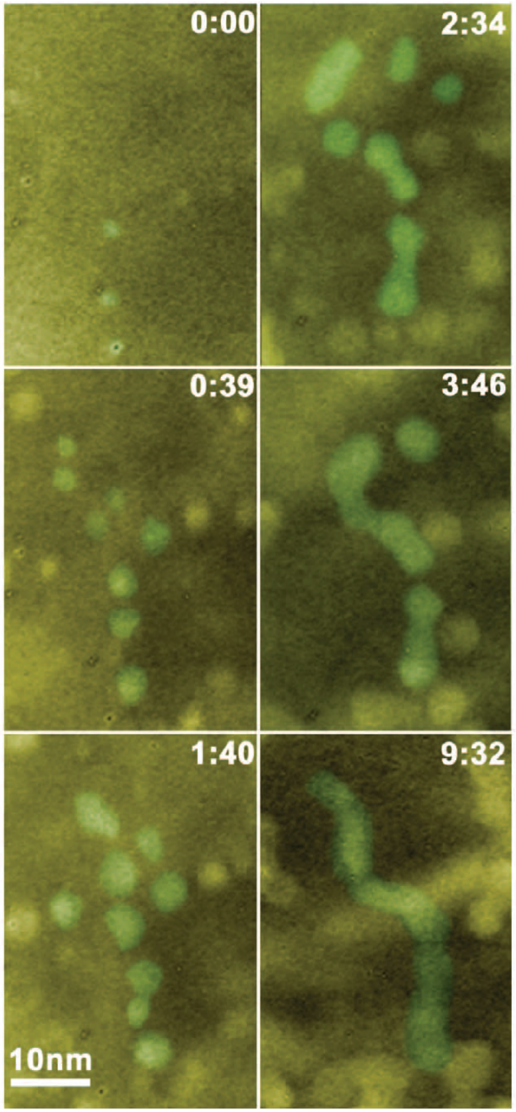


Zheng, Alivisatos et al. Science (2009)

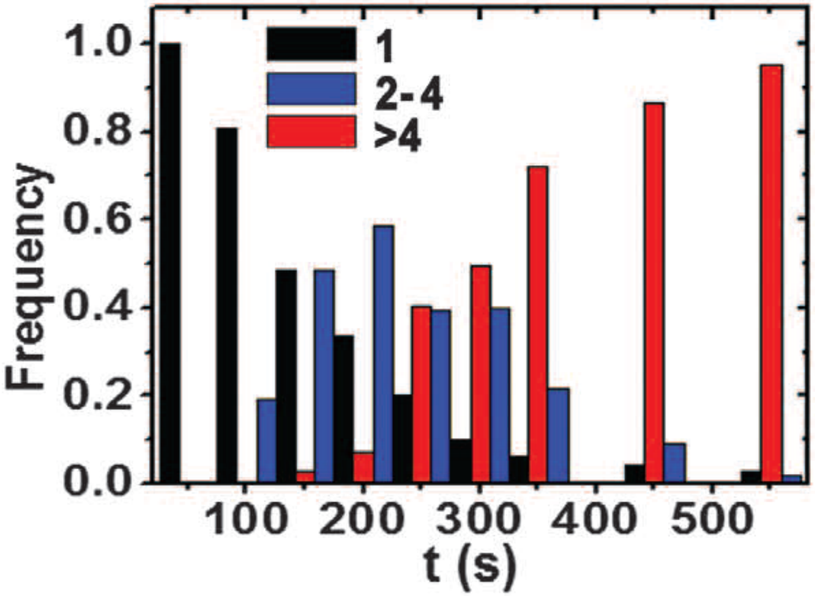
# 3.5. In-situ studies

## 3.5.1. In-situ transmission electron microscopy

CORROBORATING THE NON-CLASSICAL GROWTH



$Pt_3Fe$ nanorods grow by coalescence of primary nanoparticles

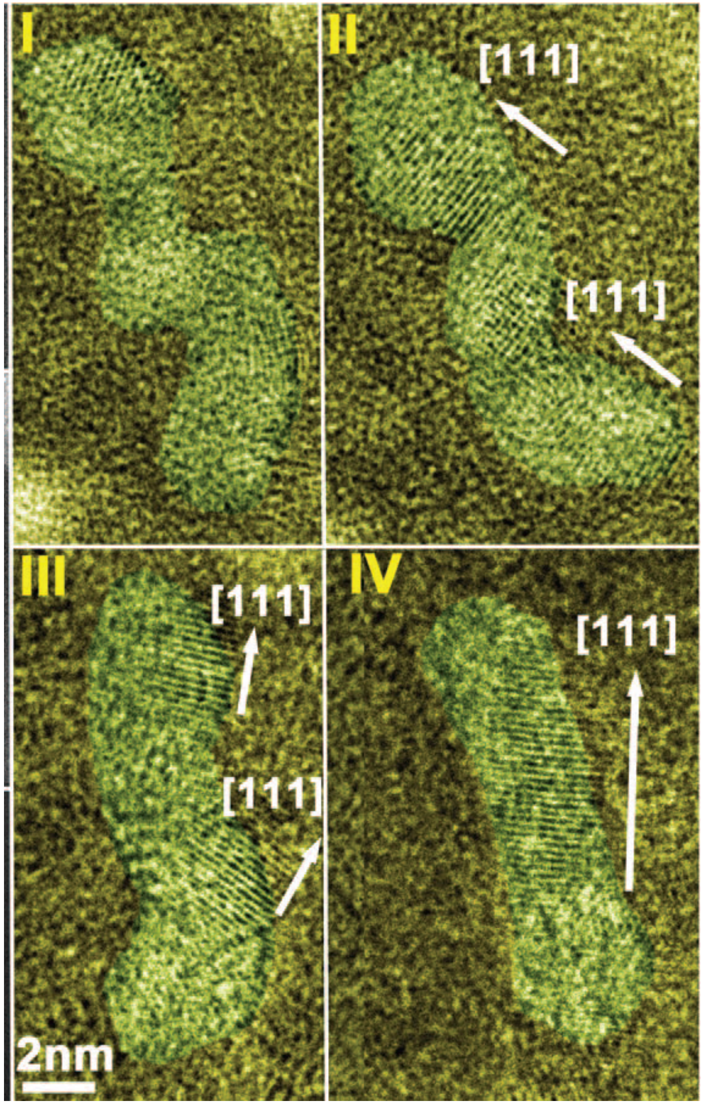


Zheng et al. Science (2011)

# 3.5. In-situ studies

## 3.5.1. In-situ transmission electron microscopy

CORROBORATING THE NON-CLASSICAL GROWTH



$Pt_3Fe$ nanorods turn into single crystals and orient along the [111] direction upon crystal orientation (ORIENTED ATTACHMENT)

Zheng et al. Science (2011)

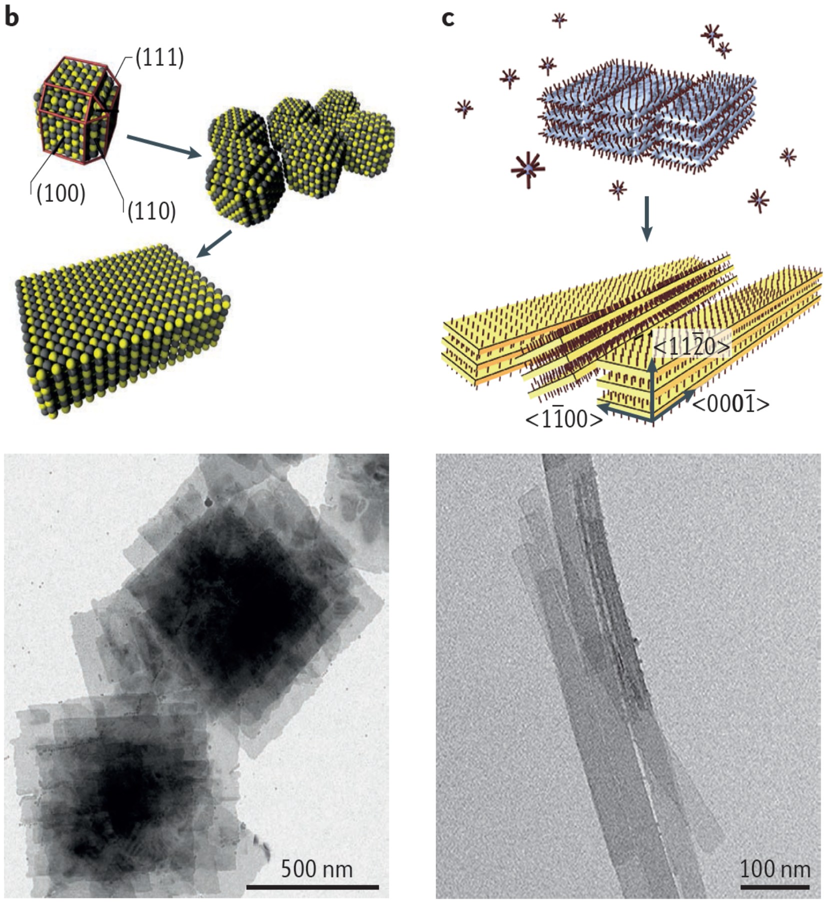
# 3.5. In-situ studies

## 3.5.1. In-situ transmission electron microscopy

CORROBORATING THE NON-CLASSICAL GROWTH

PbS nanosheets formed by (110) attachment of PbS nanoparticles

CdSe nanoribbons formed from $(CdSe)_{13}$ clusters

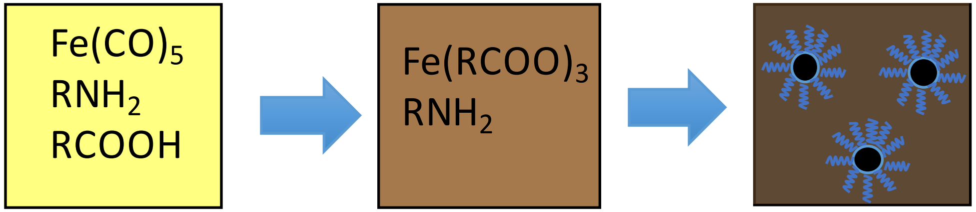


Hyeon et al. Nature Reviews Materials (2016)

# 3.5. In-situ studies

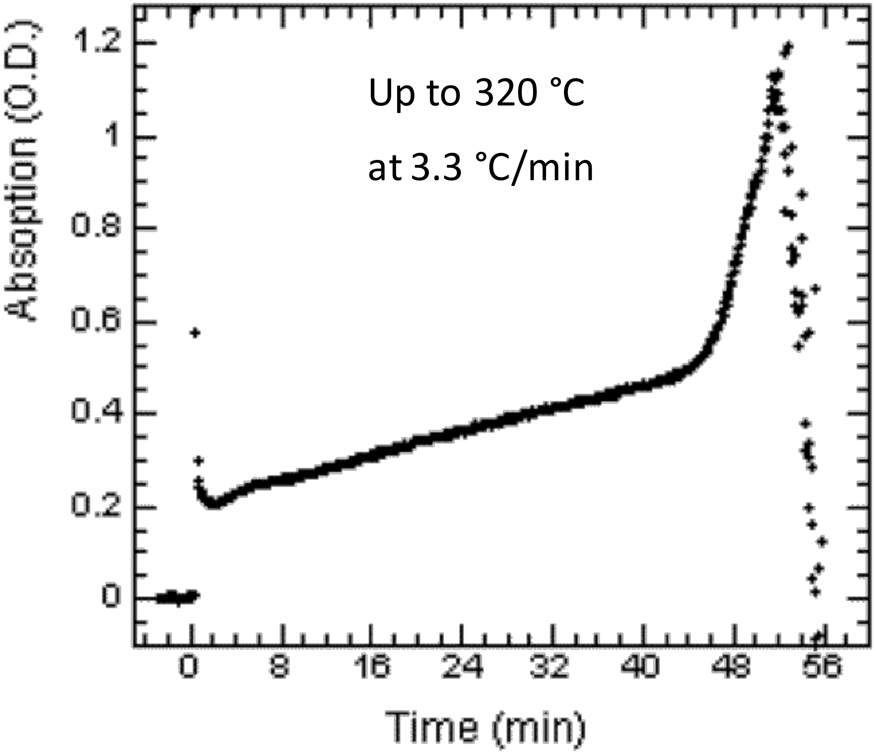
## 3.5.2. In-situ infrared spectroscopy

### Formation and decomposition of iron oleate



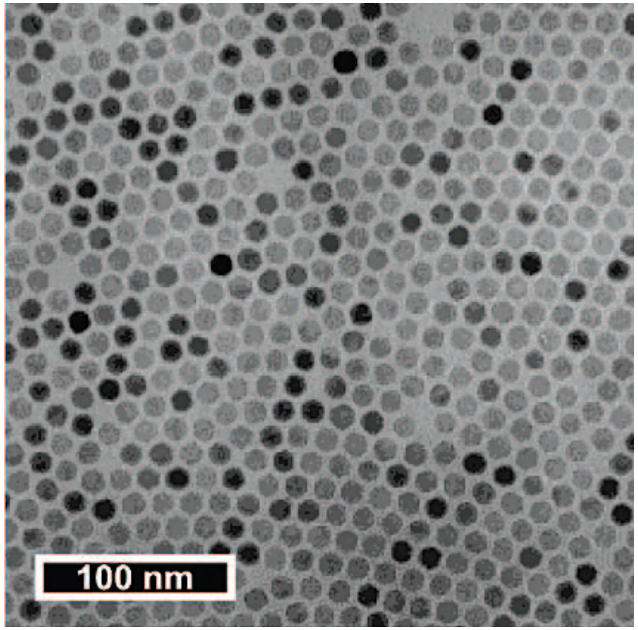
$Fe(CO)_5$
$RNH_2$
$RCOOH$

$Fe(RCOO)_3$
$RNH_2$



Up to 320 °C

at 3.3 °C/min

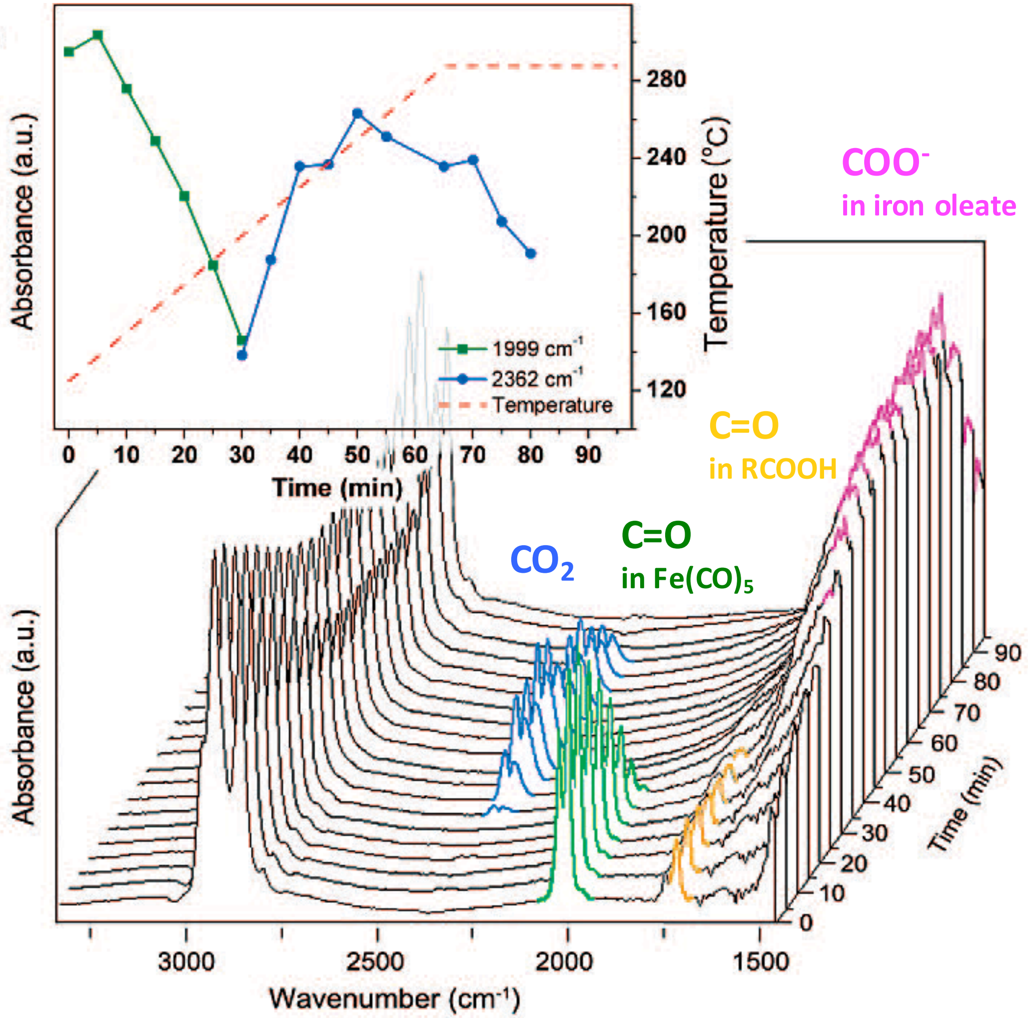


Alivisatos et al. *JACS* **2006**

# 3.5. In-situ studies

## 3.5.2. In-situ infrared spectroscopy

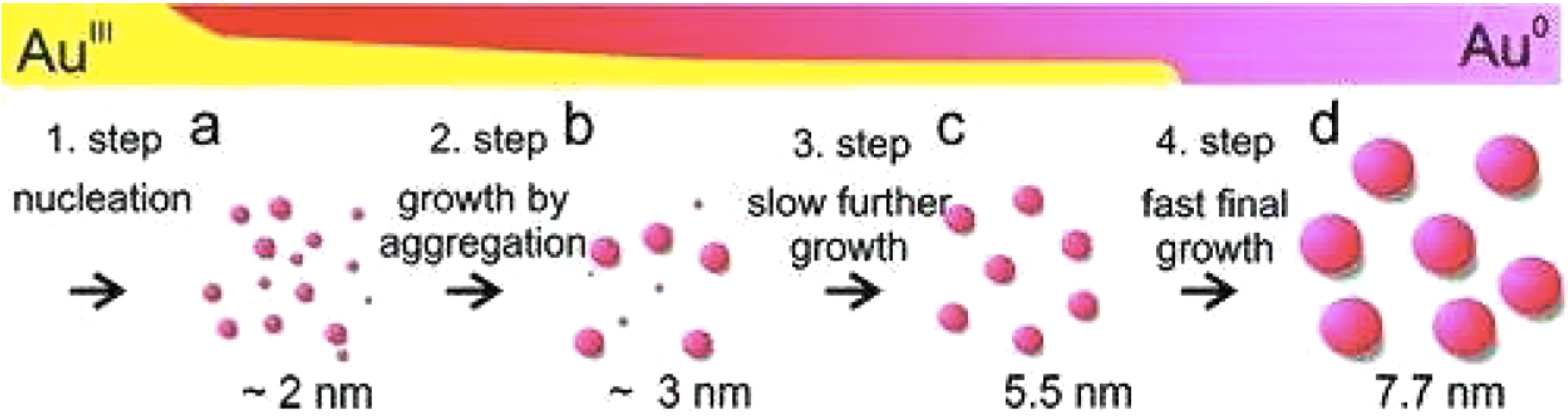
### Formation and decomposition of iron oleate



T. Hyeon JACS (**2007**); *Acc. Chem. Res.* (**2013**)

# 3.5. In-situ studies

## 3.5.3. In-situ X-Ray Absorption Studies



Teacher: Dr. Michal Strach this afternoon ☺

Sorry, not for this course ;)

Polte et al. J. Am. Chem. Soc. (2010)

**Main references for this module:**

Buhro et al. *Chem. Mater.* **2014**, 26, 5

Hyeon et al. *Nature Review* **2016**, 1, 1

Niederberger et al. *Chem.Eur. J.* **2017**, 23, 8542

Mahiddine et al. *Chem Rev.* **2014**, 114, 7610