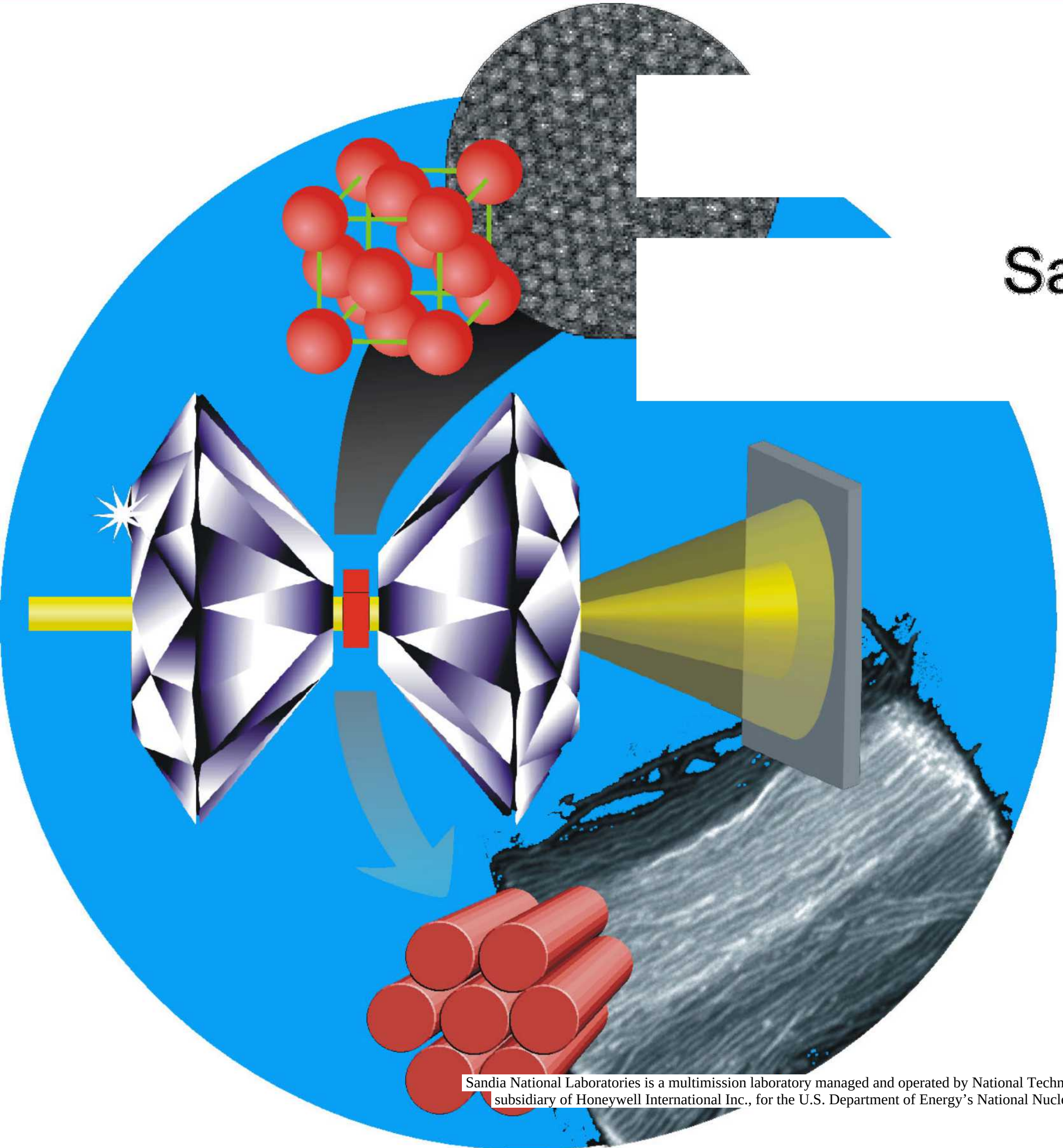
Pressure-Induced Phase Transformation, Optical Coupling, and Coalescence of Nanoparticle Superlattices

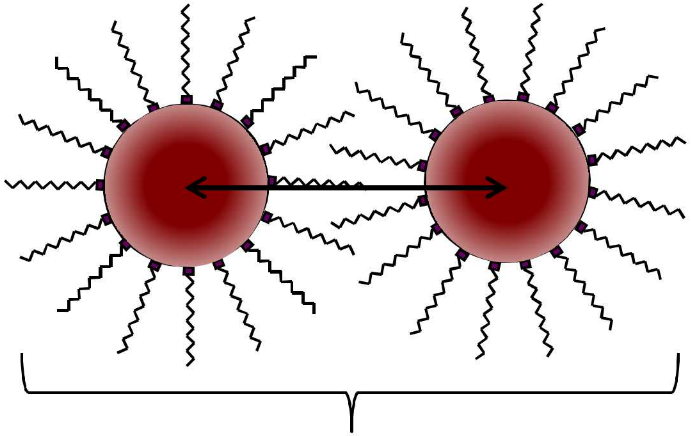
SAND2018-10240C



Hongyou Fan

Sandia National Laboratories

University of New Mexico



Nanoparticle interactions and coupling

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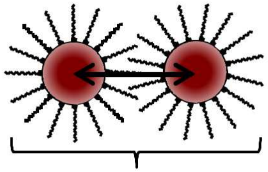
Outline

1. Introduction - Nanoparticle Self-Assembly



- Nanoparticle interactions
- Collective properties of nanoparticle arrays
- Current Progress

2. Pressure-Induced Assembly and Formation of 1-3D Nanostructures

- High pressure induced assembly
- 1-3D metallic nanostructures (Au and Ag)
- Semiconductor nanowires (CdSe, etc.)
- Pressure-tuned nanoparticle coupling and collective property

3. Summary

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Applications of Nanoparticles and Arrays

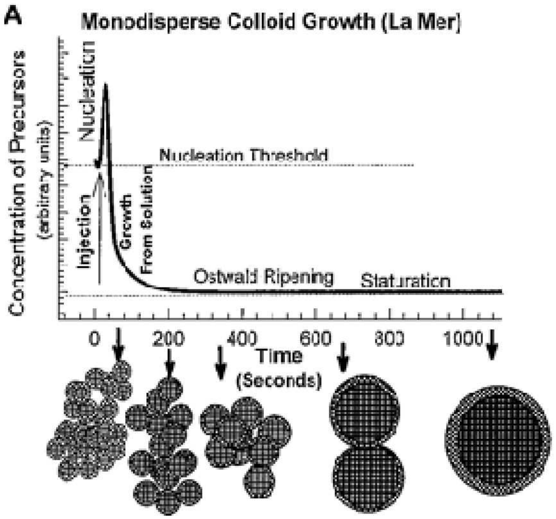
- **Size/shape-dependent and collective optical, electrical, and magnetic properties.**
  e.g., surface plasmon resonance (SPR), tuning refractive index for optical coatings, tuning dielectric constant, QD/solar cells, electron transport/conductance, magnetic memory, etc.
- **Chemical and biological sensing, imaging, & therapeutics.**
  e.g., Surface enhanced Raman scattering (SERS) based chem-/bio-sensor systems, MRI contrast agents, cell killing, etc.
- **Catalyst arrays for orientated growth of nanomaterials and arrays.**
  e.g., carbon nanotube arrays/films, nanowire arrays, etc.

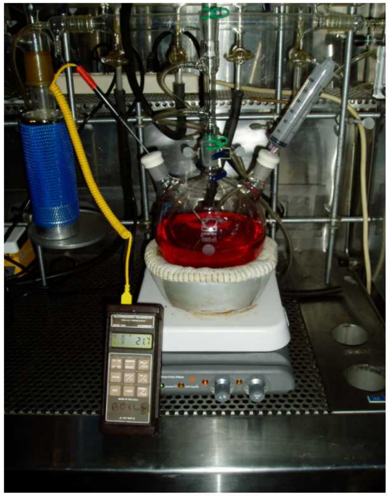
(CdSe) quantum dot size increases

Sanderson, K., *Nature* vol.459, 760-761, 2009.

Synthesis of Nanoparticles at Ambient Pressure

(1) “Hot Soap” solution method





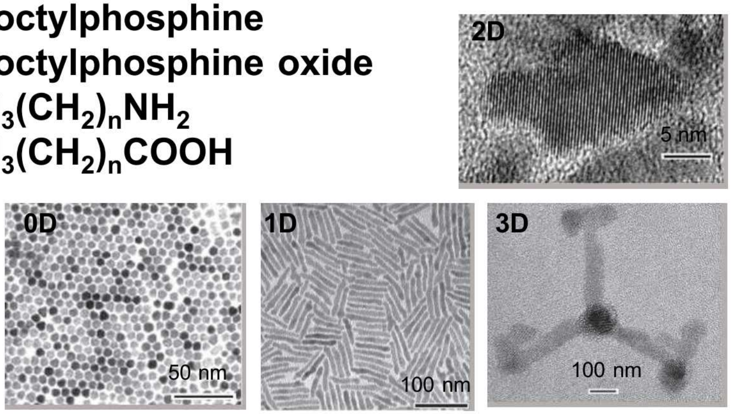
Murray, C.B. *et al.*, *Annu. Rev. Mater. Sci. 30,* 545-610, **2000**.

Semiconductor and magnetic nanoparticles:

e.g., CdSe, FePt, etc.

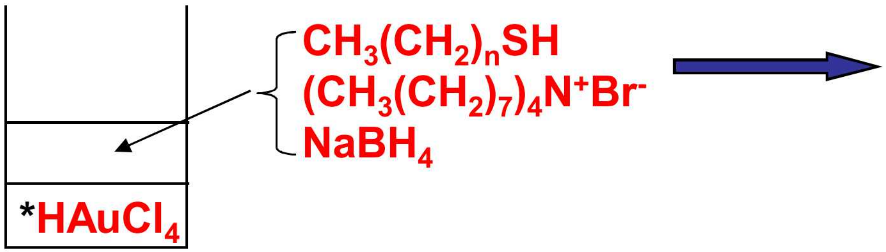
Organic ligands:

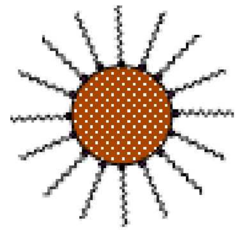
Trioctylphosphine
Trioctylphosphine oxide
$CH_3(CH_2)_nNH_2$
$CH_3(CH_2)_nCOOH$



Yin, Y., *et al.*, *Nature*, v.437, 664-670, 2005.

(2) Phase transition synthesis of metal nanocrystals





Gold & silver nanoparticles

* Brust, M., *et al.*, *J. Chem. Soc.-Chem. Comm.* 7, 801-802, 1994.

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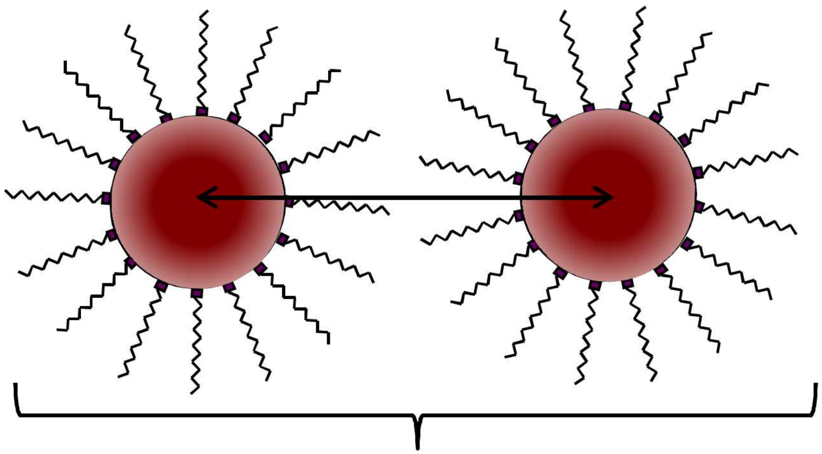
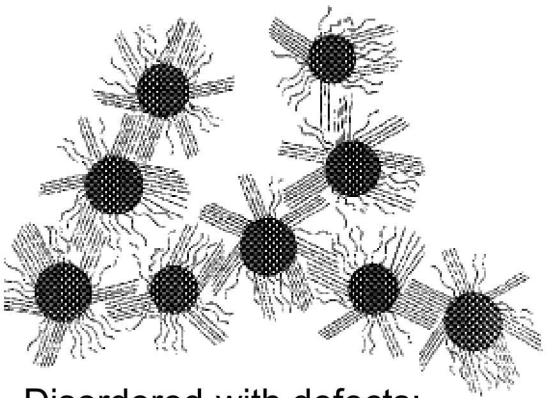
Nanoparticle Assembly at Ambient Pressure: Balanced Nanoparticle Interactions

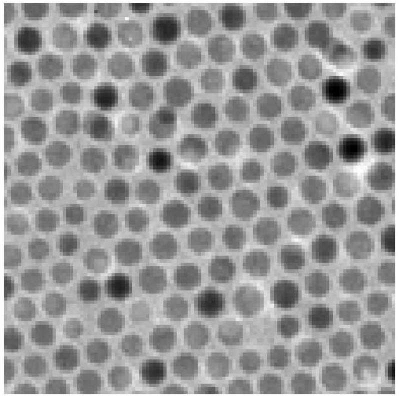


Balanced Nanoparticle interactions:

- Attraction
- Van der Waals
- Charge interactions
- Dipole-dipole
- …



Disordered with defects: vacancy, grain boundary, etc.

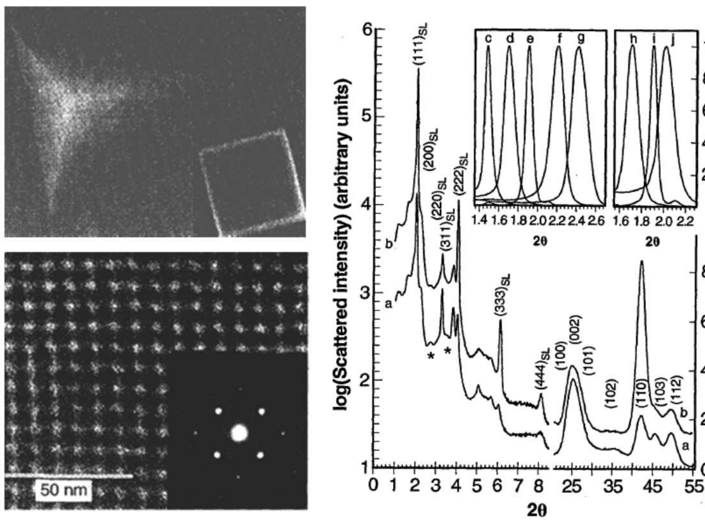


Ordered array

Lennox, R.B. *et al*. *Chem. Eur. J.* vol. 2, 359-363, 1996,

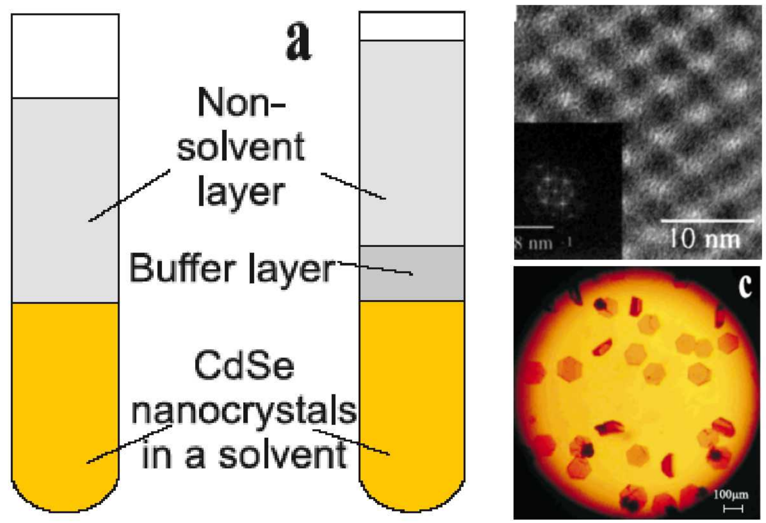
Nanoparticle Self-Assembly at Ambient Pressure: 1. Balanced Nanoparticle Interactions

1. Solvent anealing



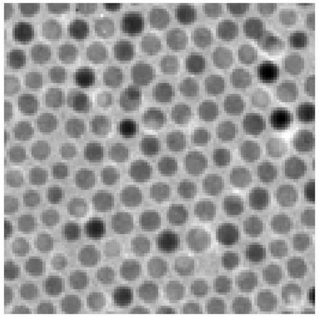
Murray, et al., *Science*, 1995.

2. Heterogeneous nucleation



Talapin, et al., *Adv. Mater*. 2001.

3. Solvent Evaporation



Ordered thin film

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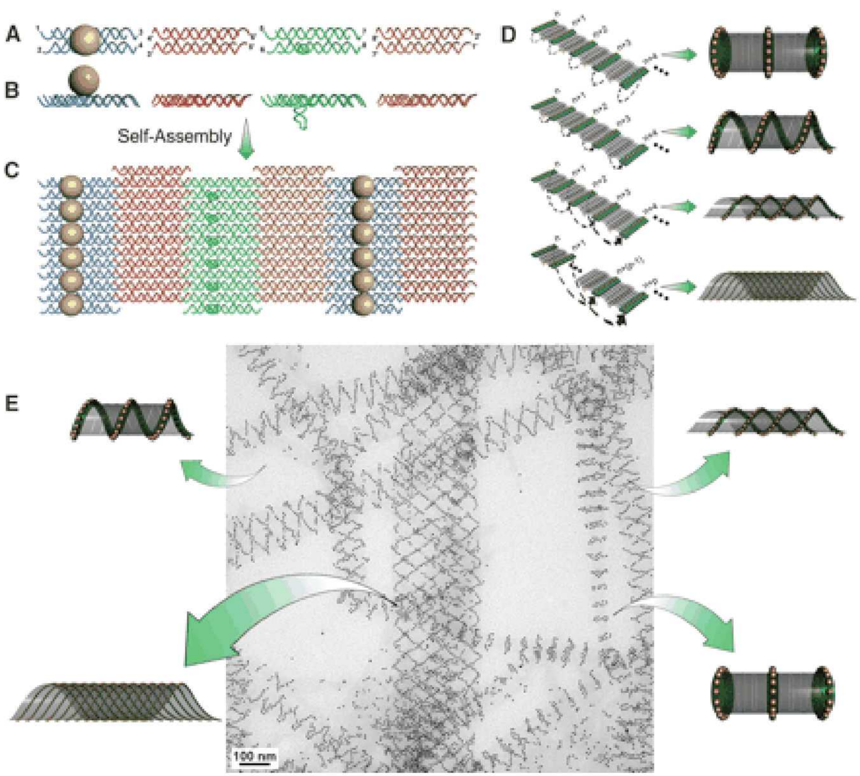
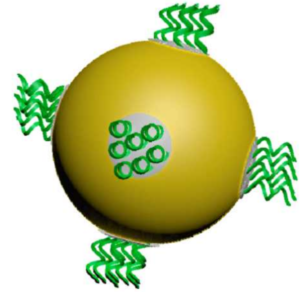
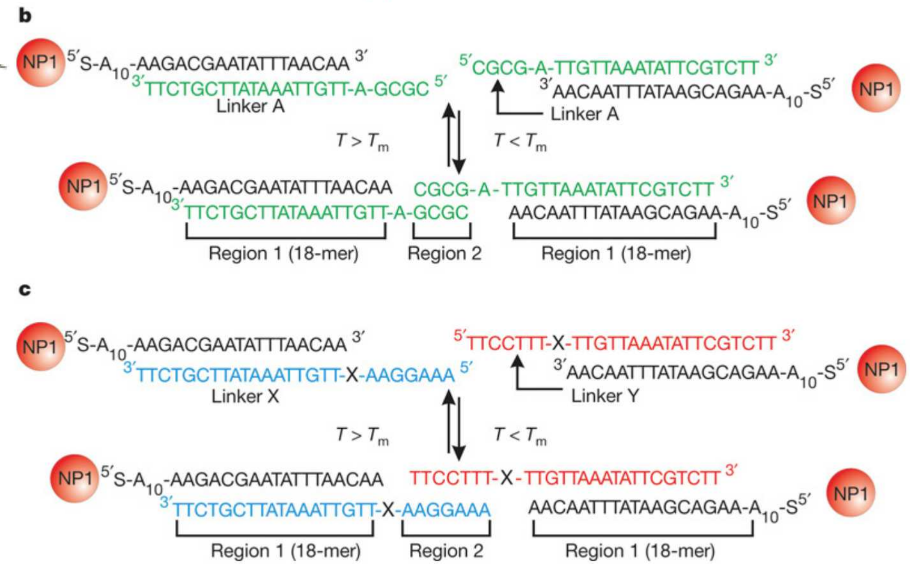
Nanoparticle Self-Assembly at Ambient Pressure: 2. DNA-Programmable Nanoparticles

Ligand-receptor interactions

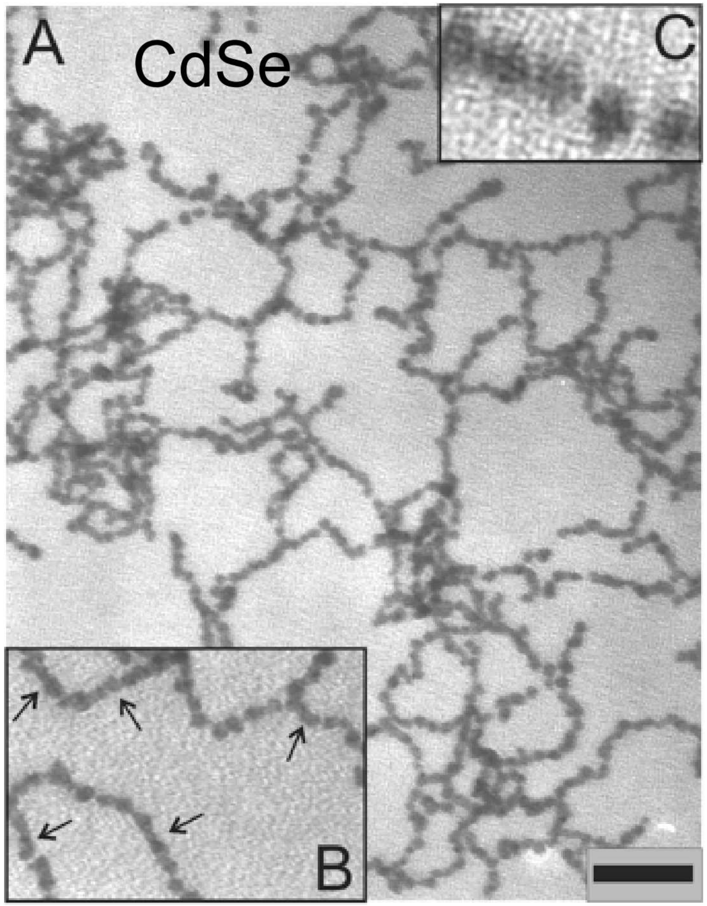






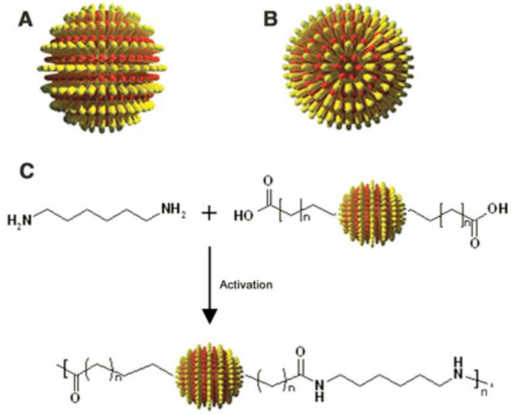
Nanoparticle Self-Assembly at Ambient Pressure: 3. Dipole-Dipole Interactions and Chemical Reactions

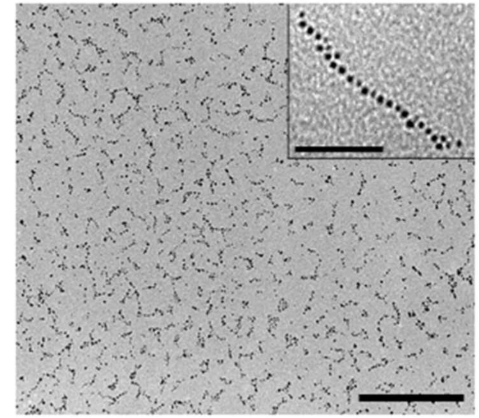
Dipole-Dipole Interaction



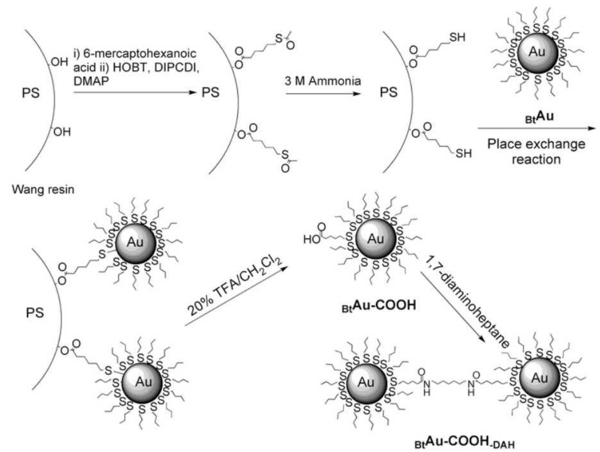
Z. Y. Tang, *et al., Science* 297, 237, 2002.

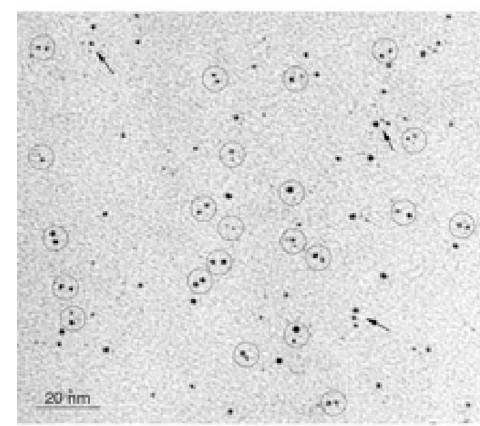
Chemical Reactions





G. A. DeVries, *et al., Science* 315, 358, 2007.





J. G. Worden, *et al., Chem. Comm.*, 518, 2004.

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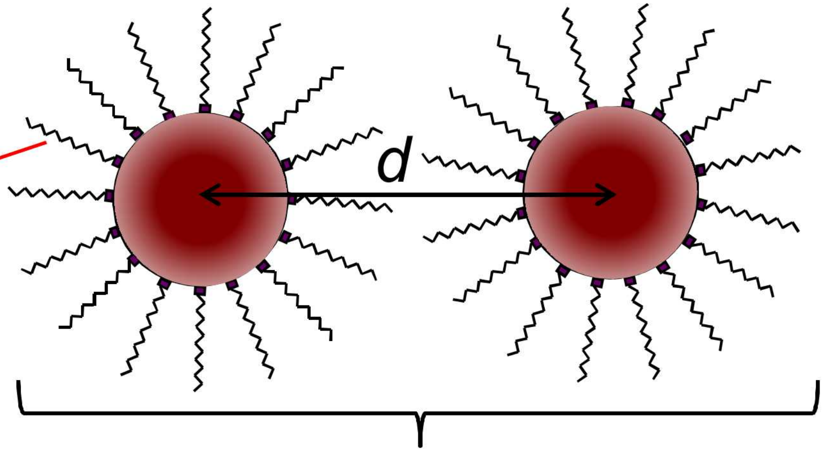
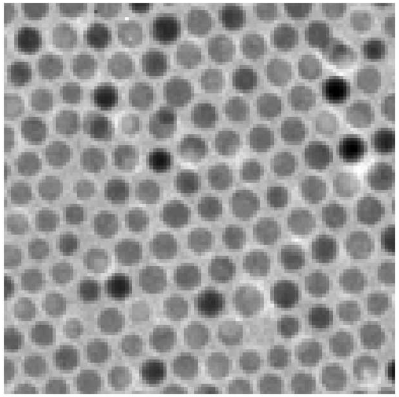
Nanoparticle Interactions and Coupling

Nanoparticle coupling depends on interparticle separation distance

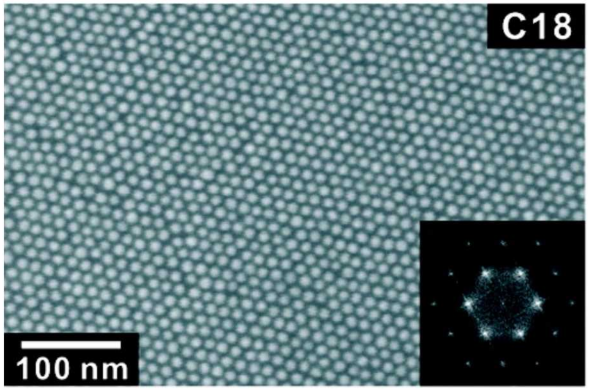
Organic ligands

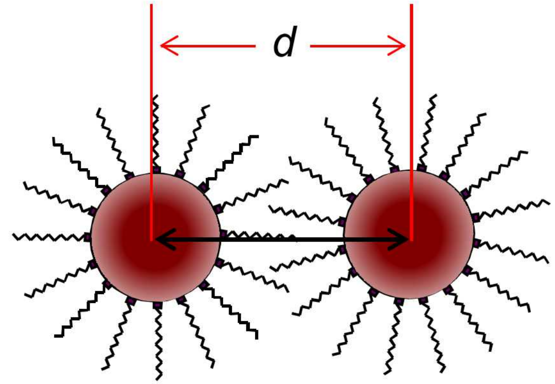


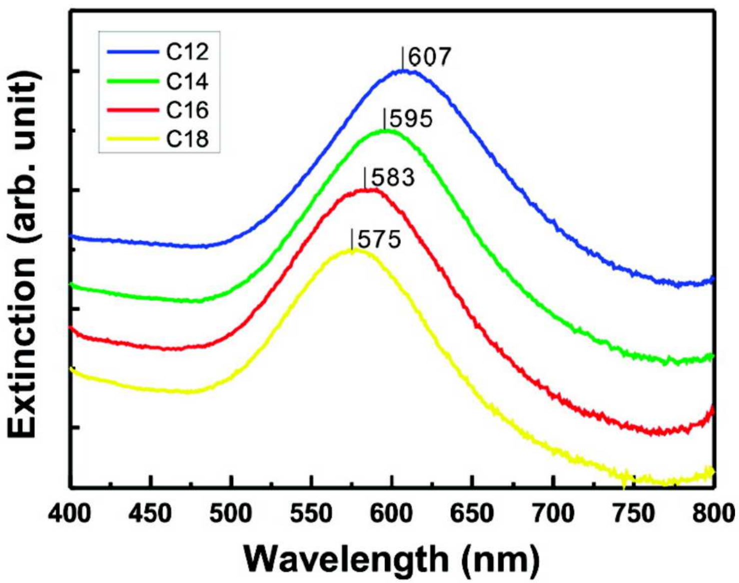


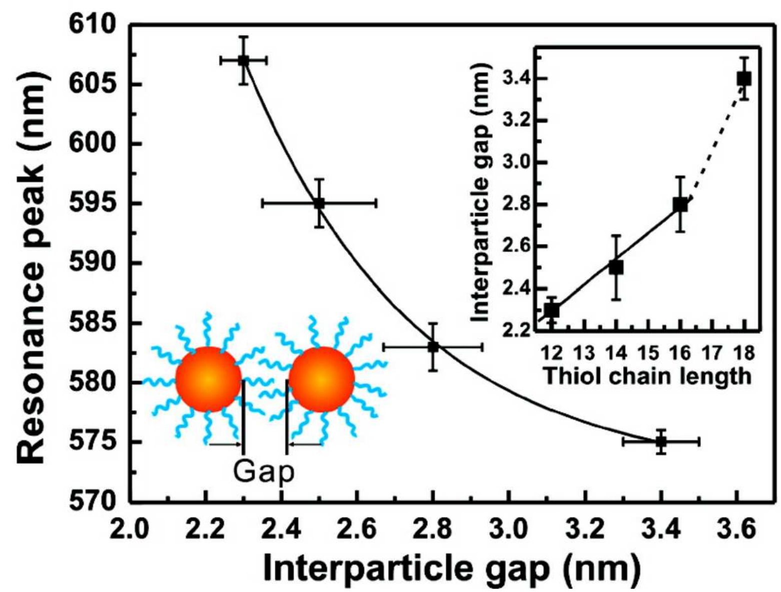
Balanced nanoparticle interactions:
•Attraction
•Van der Waals
•Charge interactions
•Dipole-dipole
•…

Tuning Plasmonic Response from Alkanethiolate-Stabilized Gold Nanoparticle Superlattices









Chi-Fan Chen, *et al.*, *J. Am. Chem. Soc.,* 130, 824-826, 2008.

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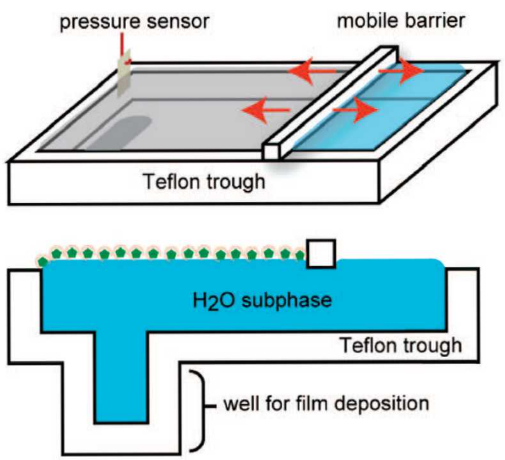
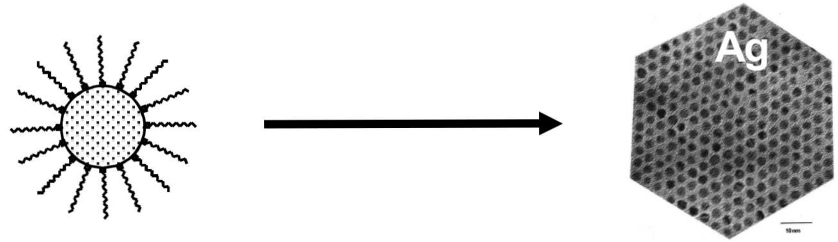
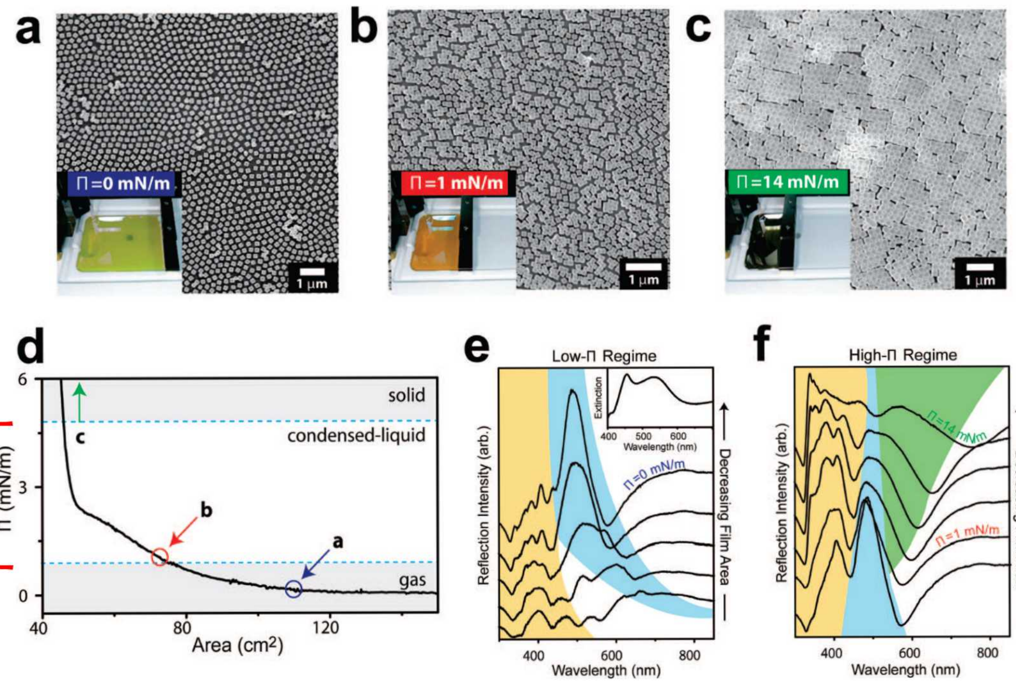
Langmuir-Blodget to Tune Nanoparticle Separaion

LB process





C. P. Collier, *et al*, *Science*, 227, 1978-1981, 1997.



- uniaxial pressure
- 2-dimensional
- limited pressure range

Tao, A. R. *et al*., *Acc. Chem. Res.*, vol. 41, No. 12, 1662-1673, 2008.

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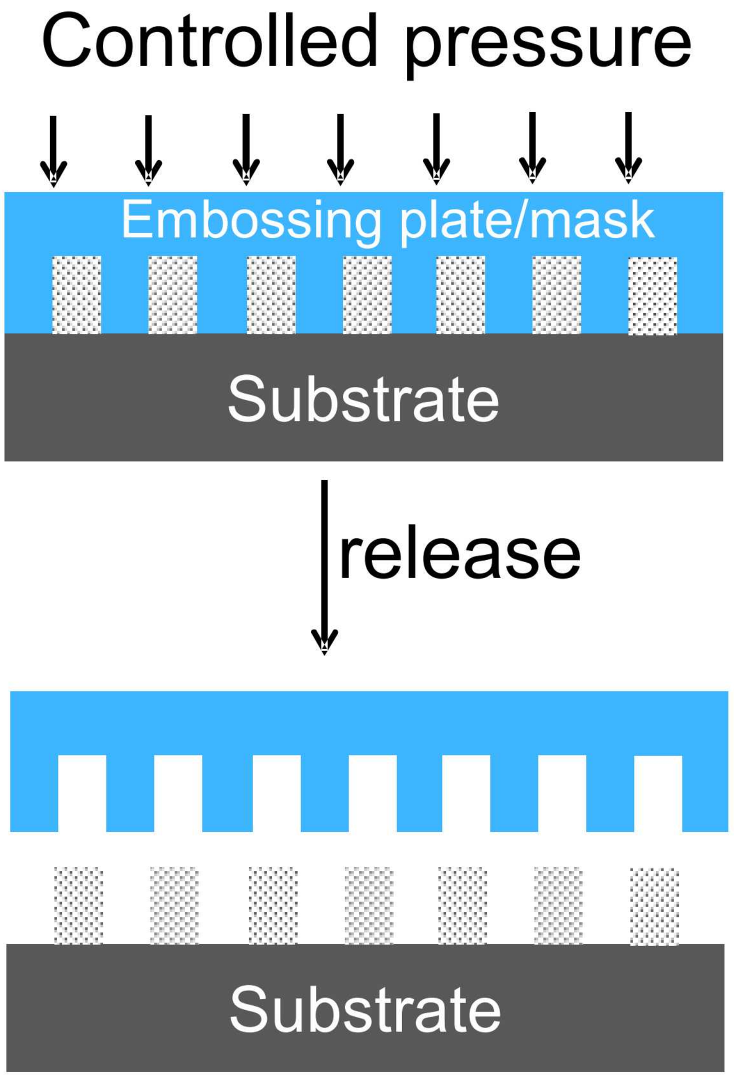
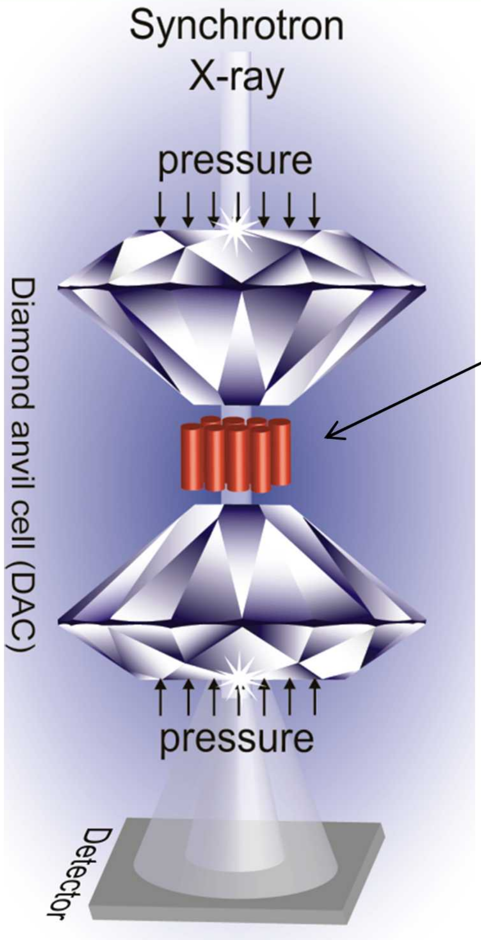
Directed Assembly- Nanoparticle Assembly under Pressure:
Mimic Manufacturing Processes - Embossing or Imprinting



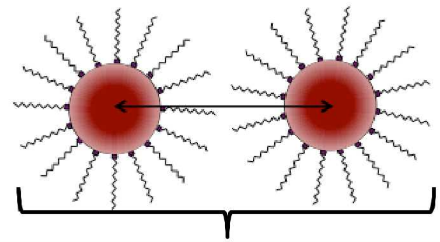
Features:
- Rapid
- Cost effective
- High throughput
- High fidelity

Pressure-Induced Assembly and Fabrication

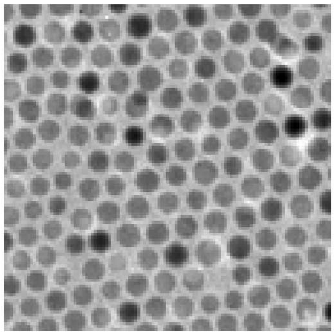
An external pressure overcomes balanced interparticle interactions, enables engineering of nanoparticle assembly, allowing fine-tuning of lattice structure and interparticle separation distance, and to fabricate new nanoparticle architectures.



Diamond Anvil Cell (DAC)



Balanced Nanoparticle interactions:
- Attraction
- Van der Waals
- Charge interactions
- Dipole-dipole
- …



- Provide controlled pressure fields:
  - Hydrostatic & uniaxial
  - Controlled pressure range
- Allow in-situ structural and property characterizations
  - Absorption, emission, etc.
  - Crystal structure, phase transition, etc.
  - Structural evolution with pressure

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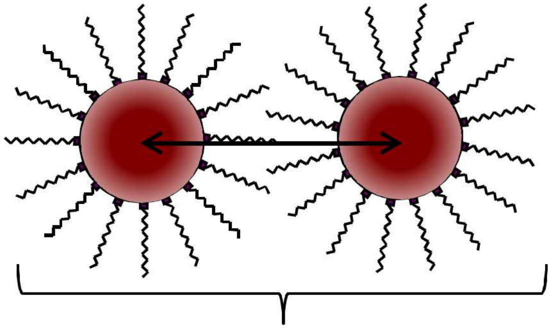
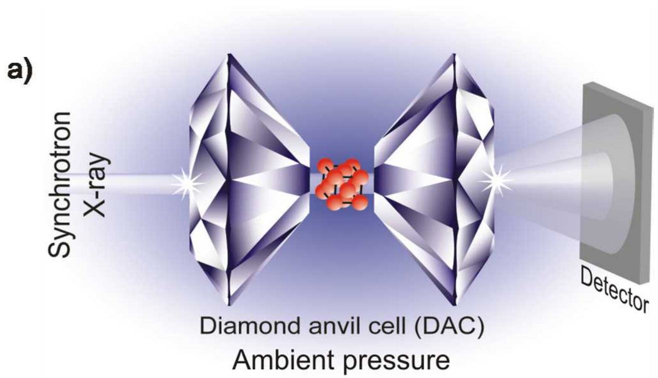
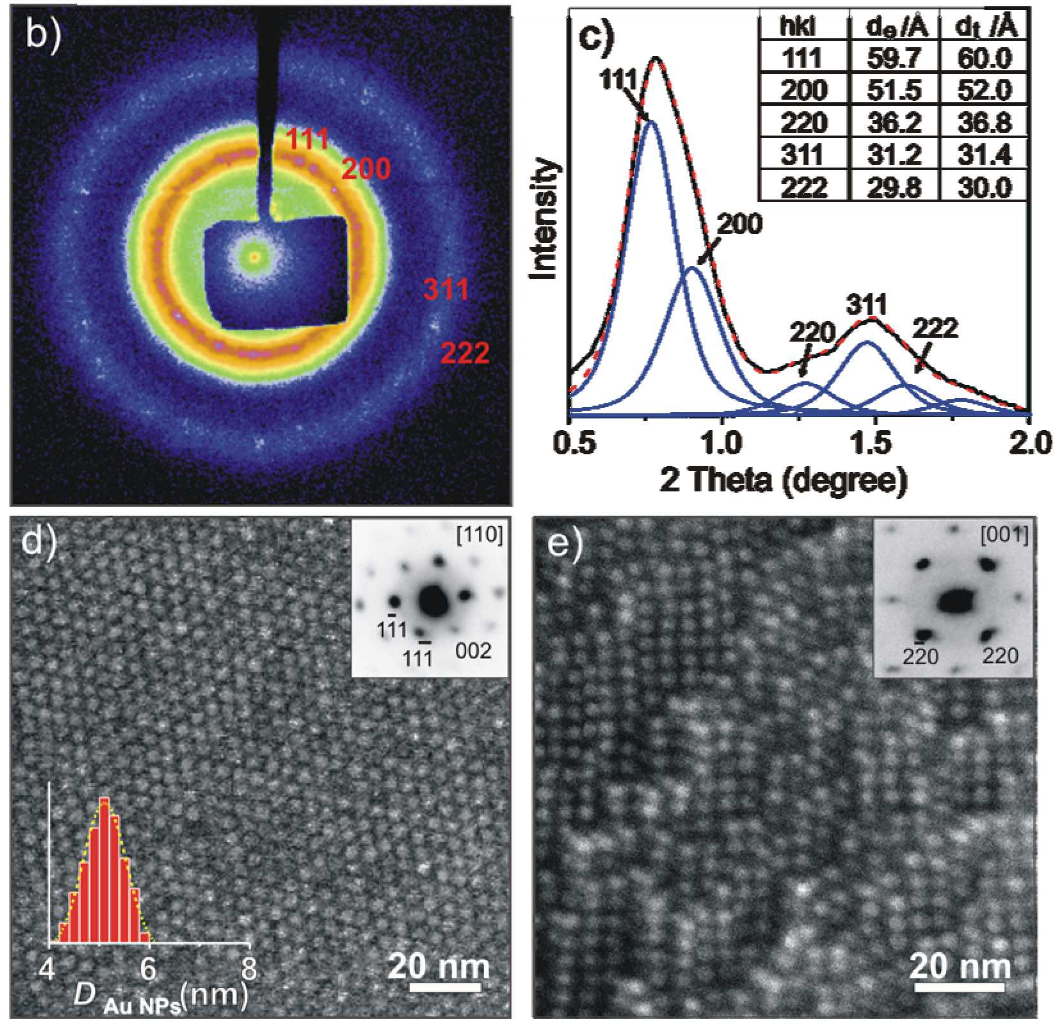
Before Compression – Starting Materials: Ordered Spherical Gold Nanoparticle Arrays

5 nm gold nanoparticles and *fcc* lattice (a = 10.4 nm)



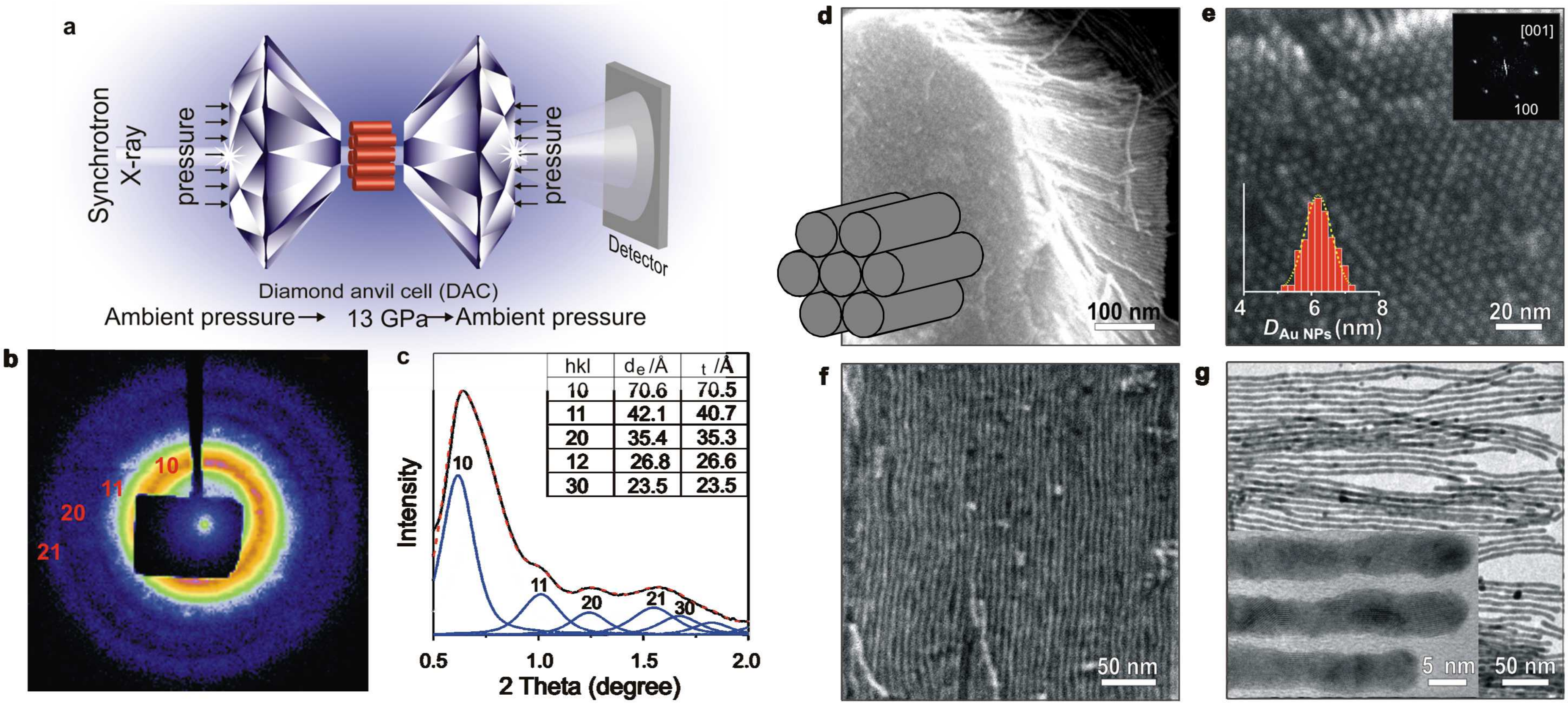
Balanced particle interactions





Wu H., Fan H., *et al. Angew. Chem. Int. Ed.,* 49, 8431-8434, 2010.

After Compression – Formation of 1D Nanowires



hkl	d_e /Å	t /Å
10	70.6	70.5
11	42.1	40.7
20	35.4	35.3
12	26.8	26.6
30	23.5	23.5

Wu H., Fan H., *et al. Angew. Chem. Int. Ed.,* 49, 8431-8434, 2010.

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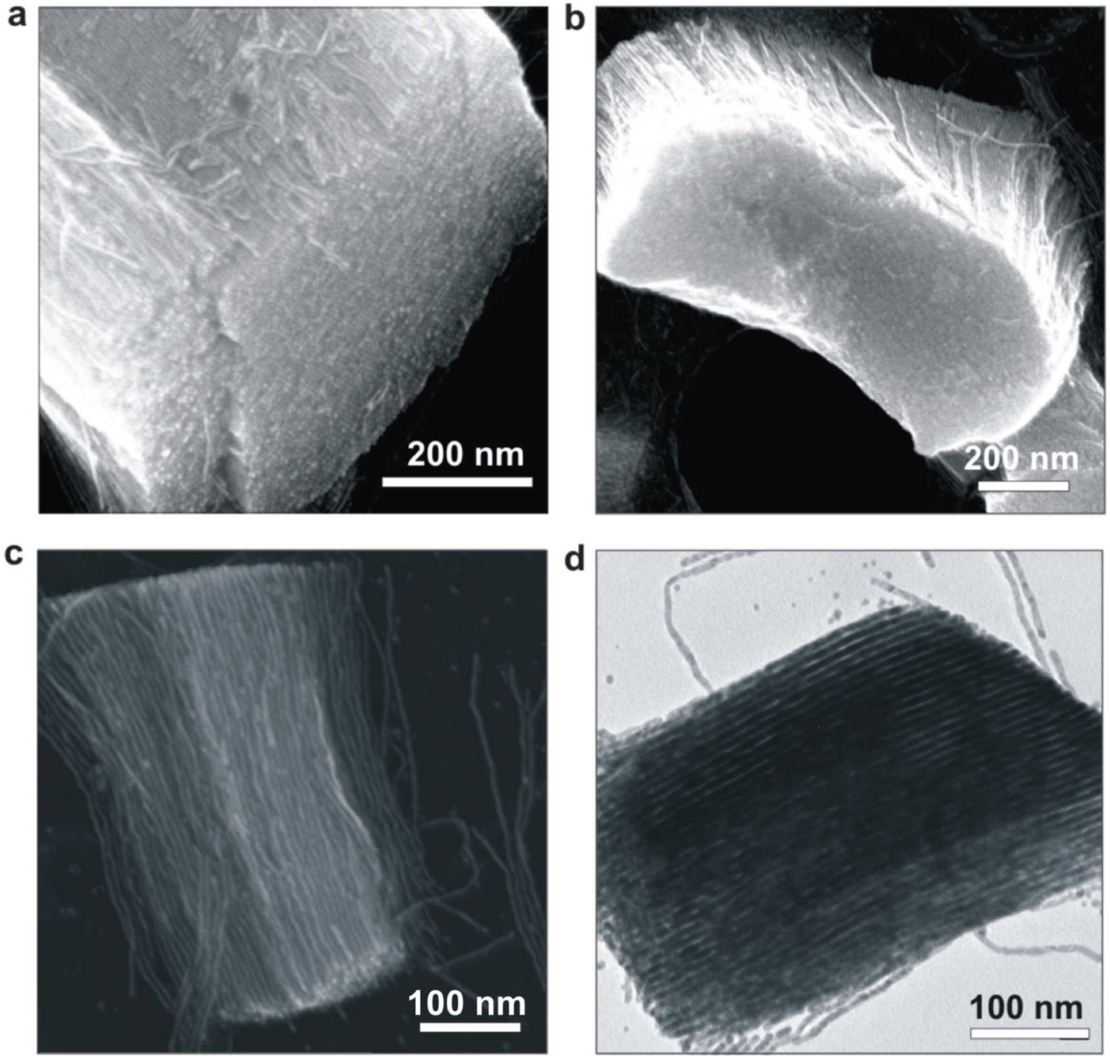
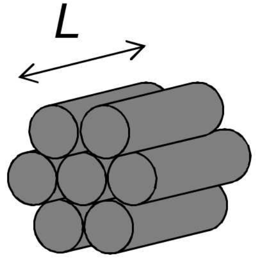
Bundles of 1D Nanowire Arrays



Uniform length



Wu H., Fan H., *et al. Angew. Chem. Int. Ed.,* 49, 8431-8434, 2010.

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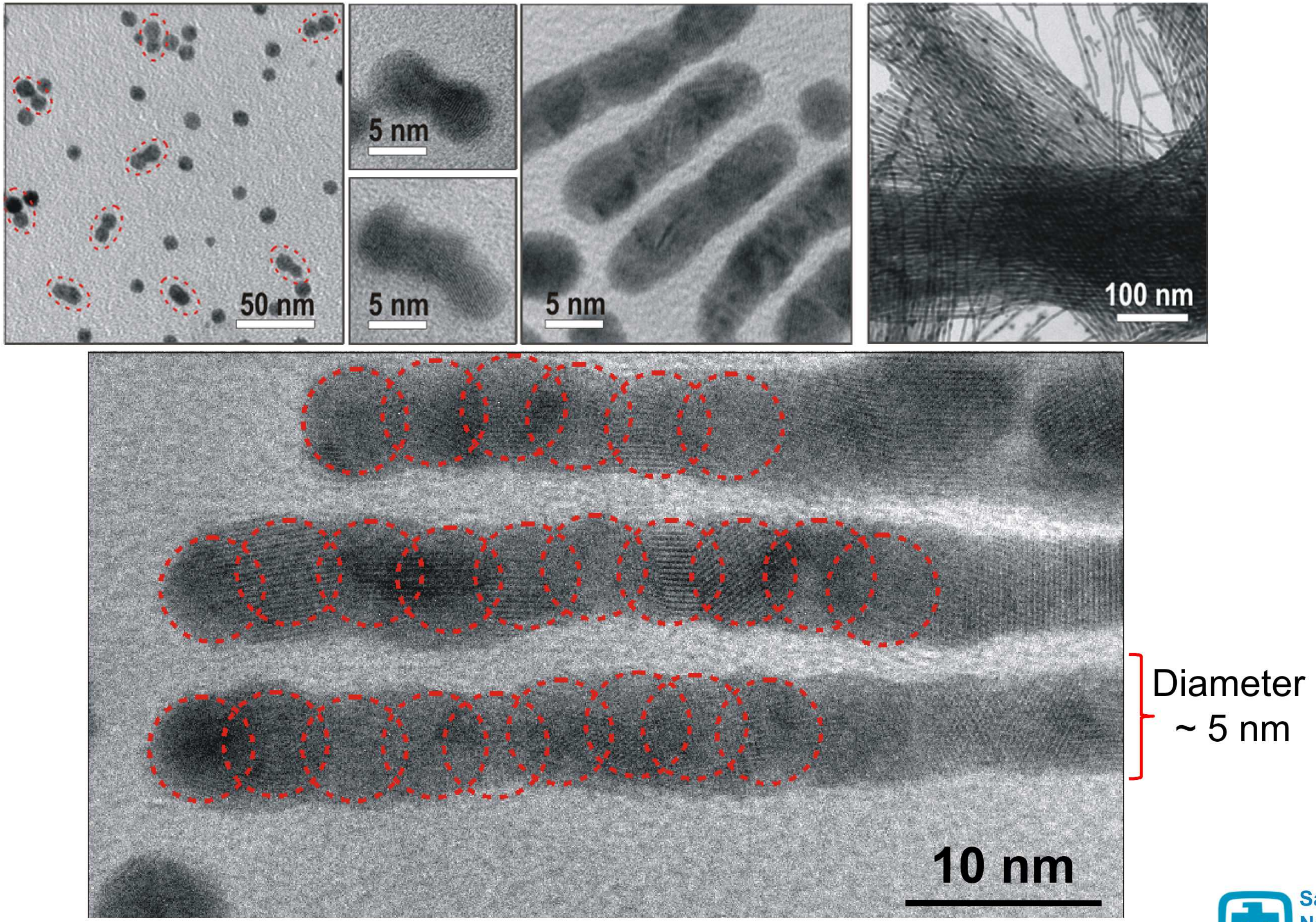
Pressure Tuned 1D Nanostructures



Wu H., *et al. Angew. Chem. Int. Ed.,* 49, 8431-8434, 2010.

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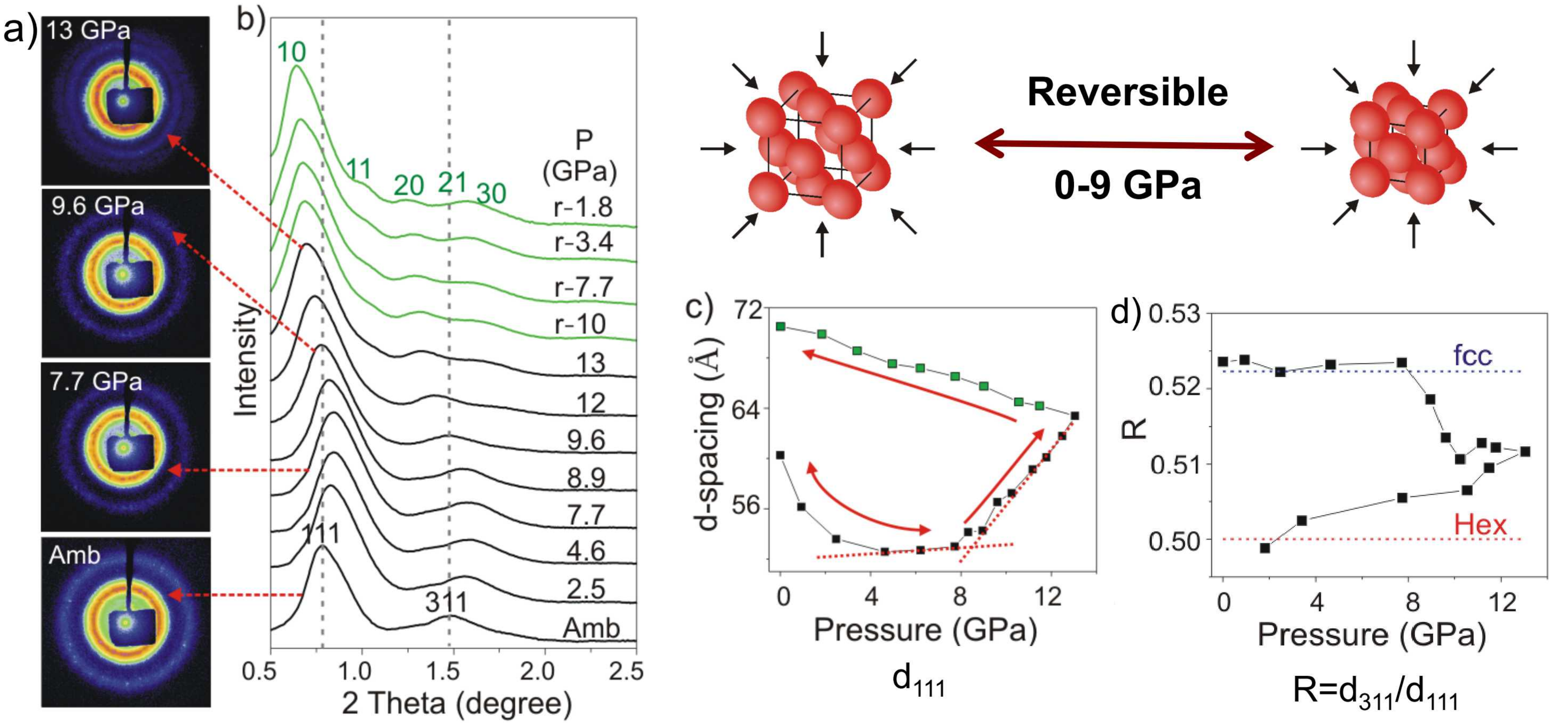
In-situ Synchrotron X-ray Studies of Nanoparticle Assembly under Pressure

An external pressure overcomes specific interparticle interactions, enables engineering of nanoparticle assembly, allowing fine-tuning of lattice structure and interparticle separation distance, and to fabricate new nanoparticle architectures.



Wu H. & Fan H. *et al*., *J. Am. Chem. Soc.*, 132, 12826-12828, 2010
& *Angew. Chem. Int. Ed.*, 49, 8431-8434, 2010.

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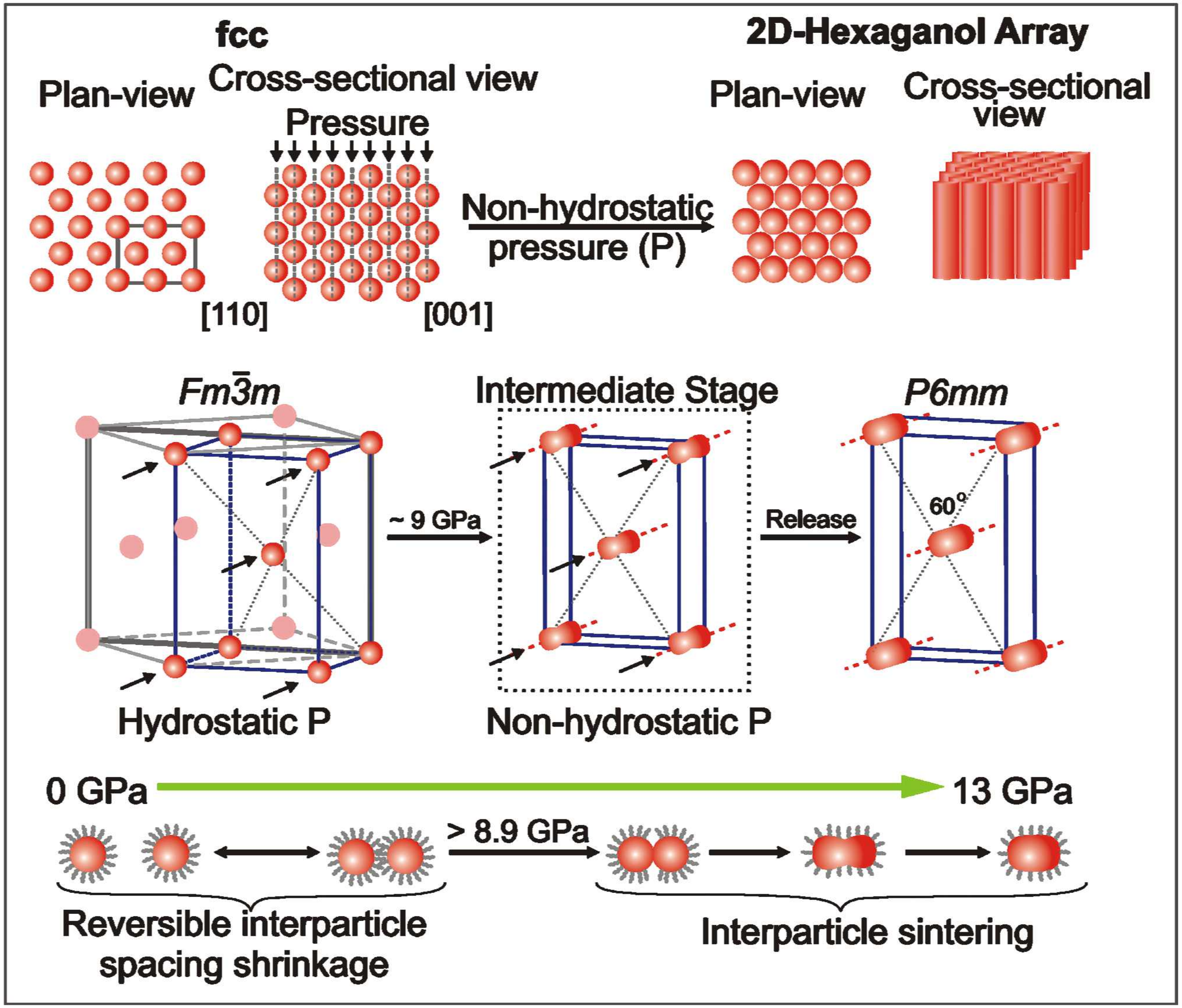
Pressure-Induced Nanoparticle Assembly Processes



Wu & Fan *et al.*, *J. Am. Chem. Soc.*, 132, 12826-12828, 2010 & *Angew. Chem. Int. Ed.,* 49, 8431-8434, 2010.

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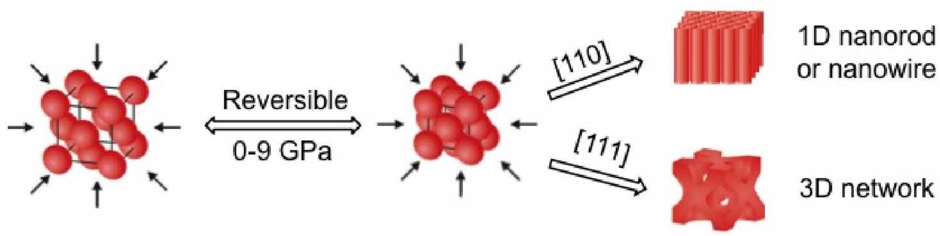
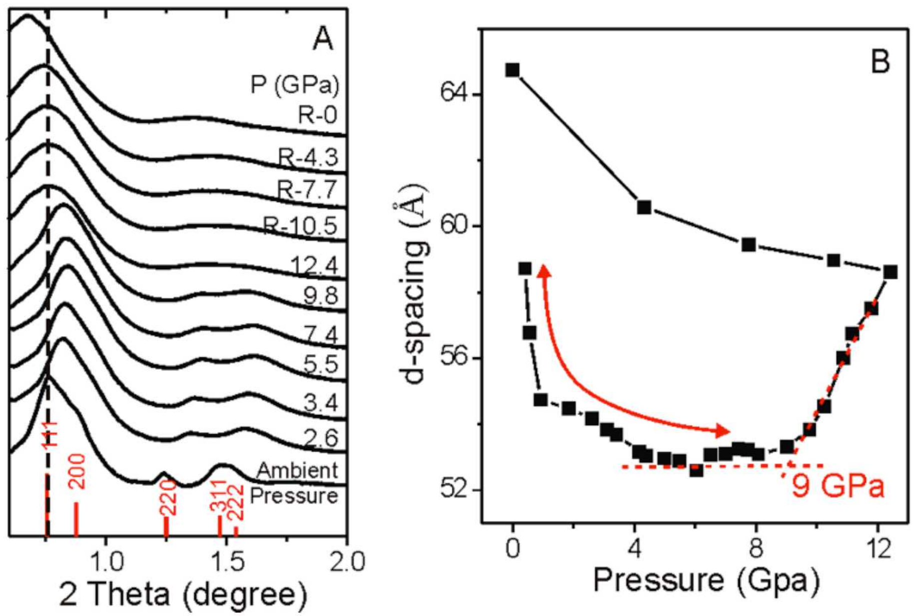
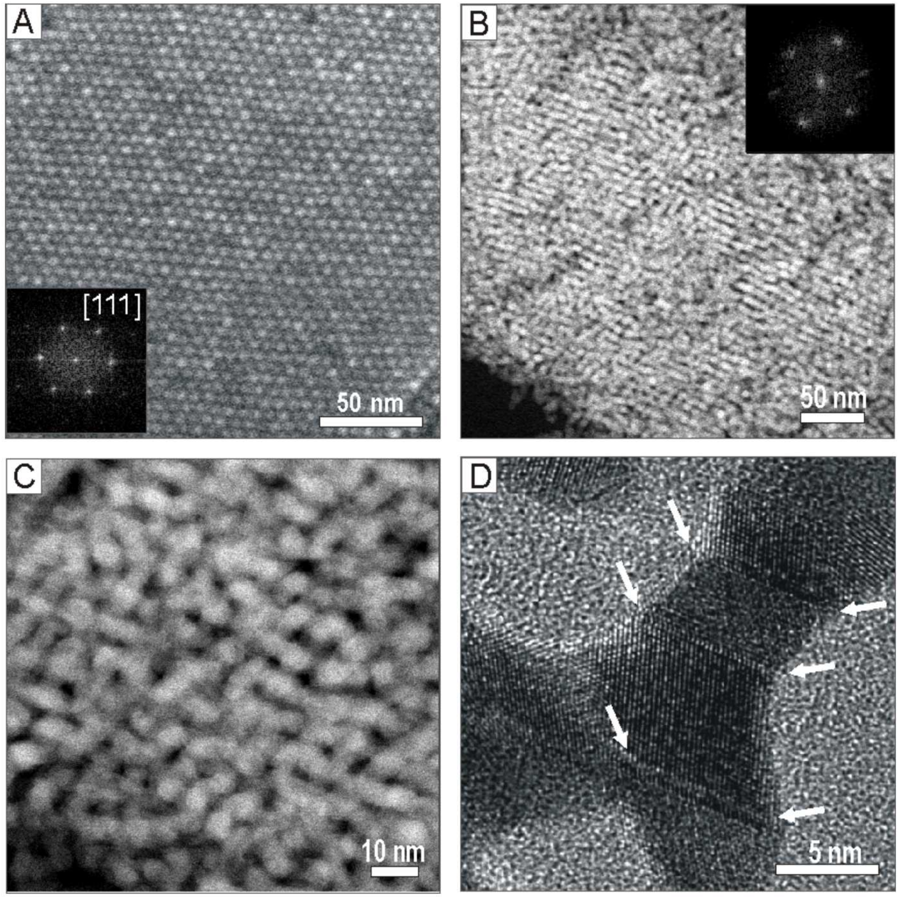
Pressure-Induced Formation of 3D Nanostructures

Interconnected 3D gold networks are formed depending on initial nanoparticle packing







Wu H., Fan, H. *et al.*, *J. Am. Chem. Soc.*, 132, 12826, 2010 & *J. Am. Chem. Soc. 136*, pp 7634, 2014.

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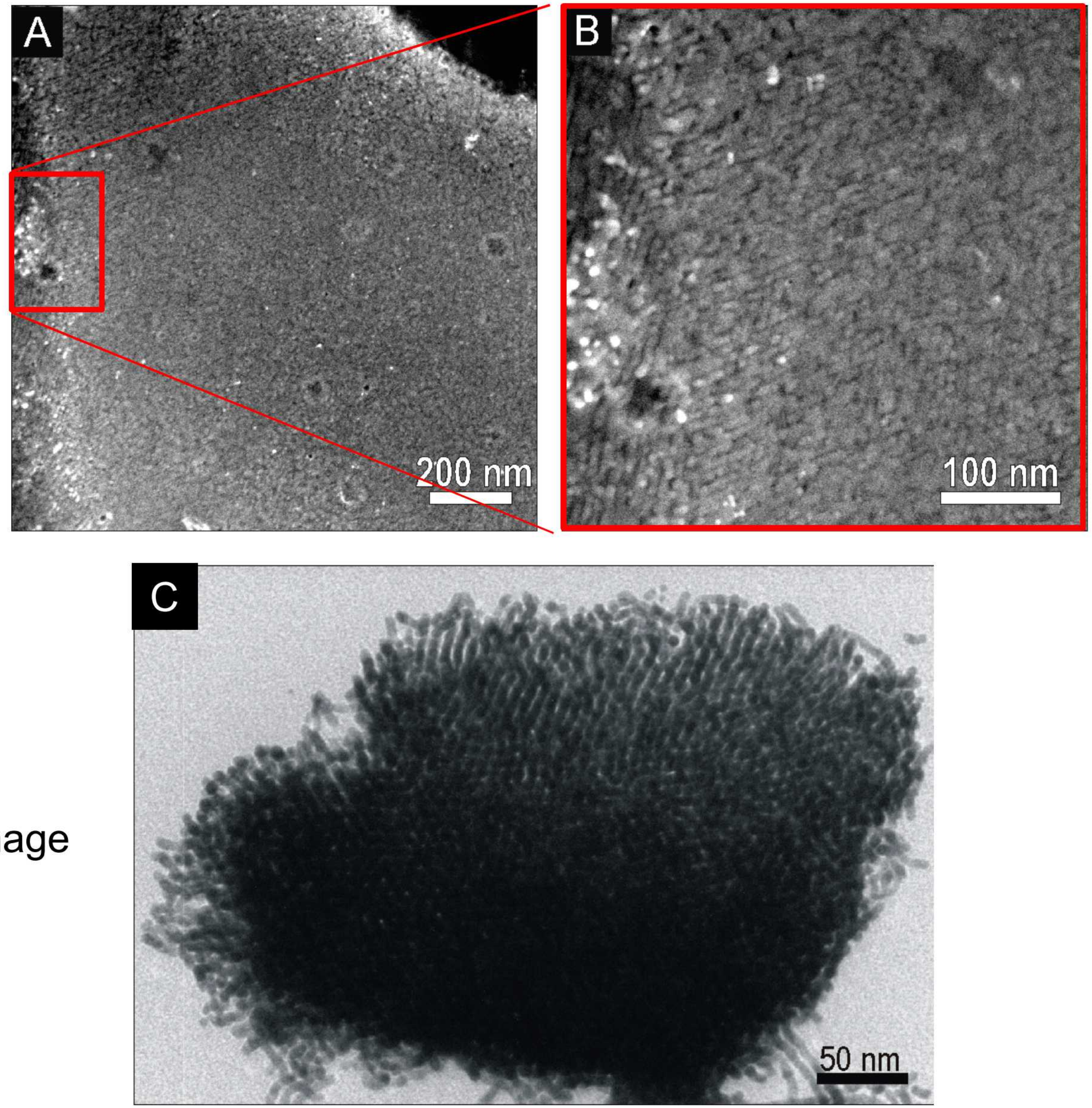
Electron Microscopy of 3D Networks

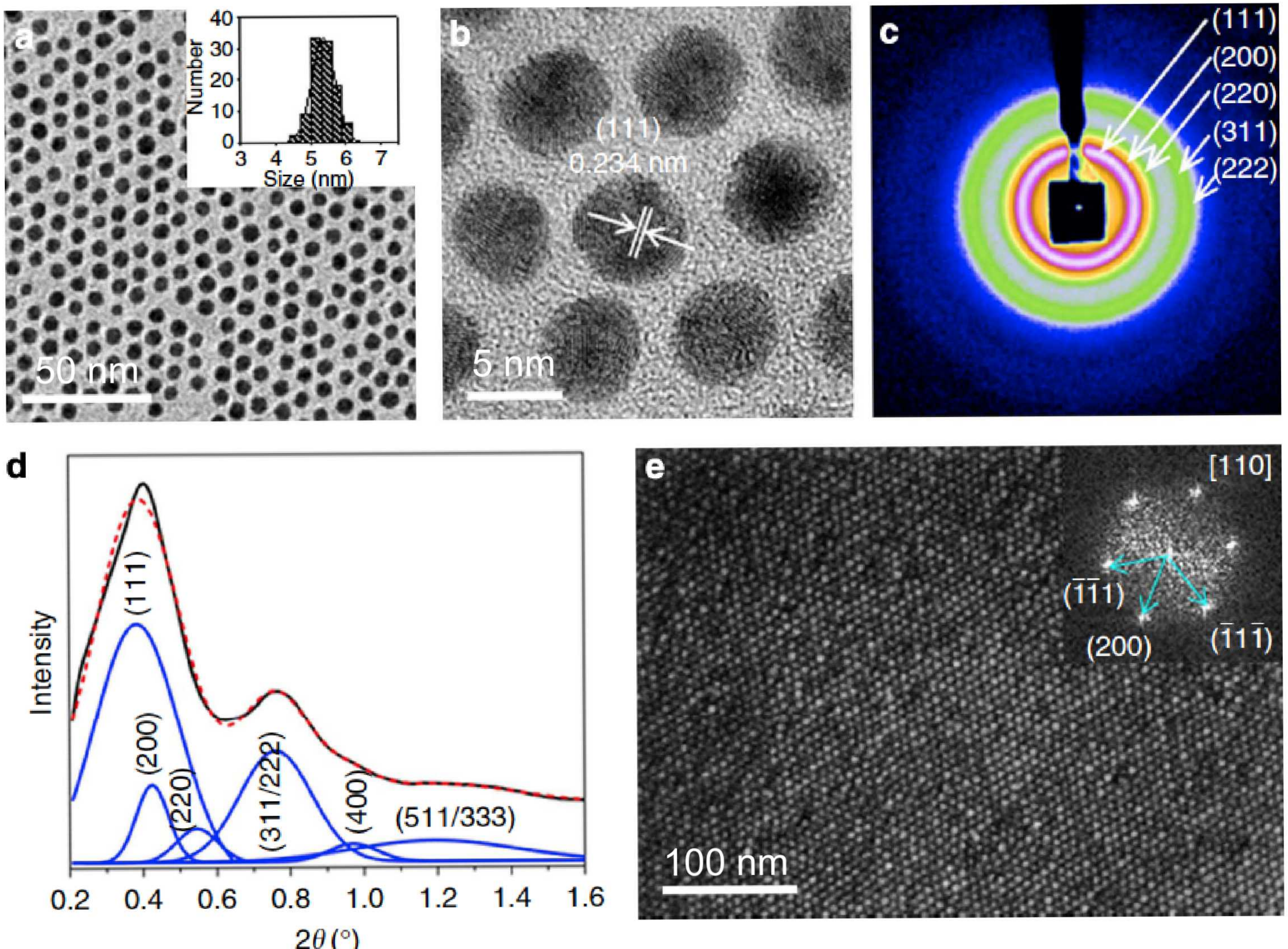
SEM images

TEM image



Wu, H. et al. *J. Am. Chem. Soc.* 2014, 136, 7634−7636.

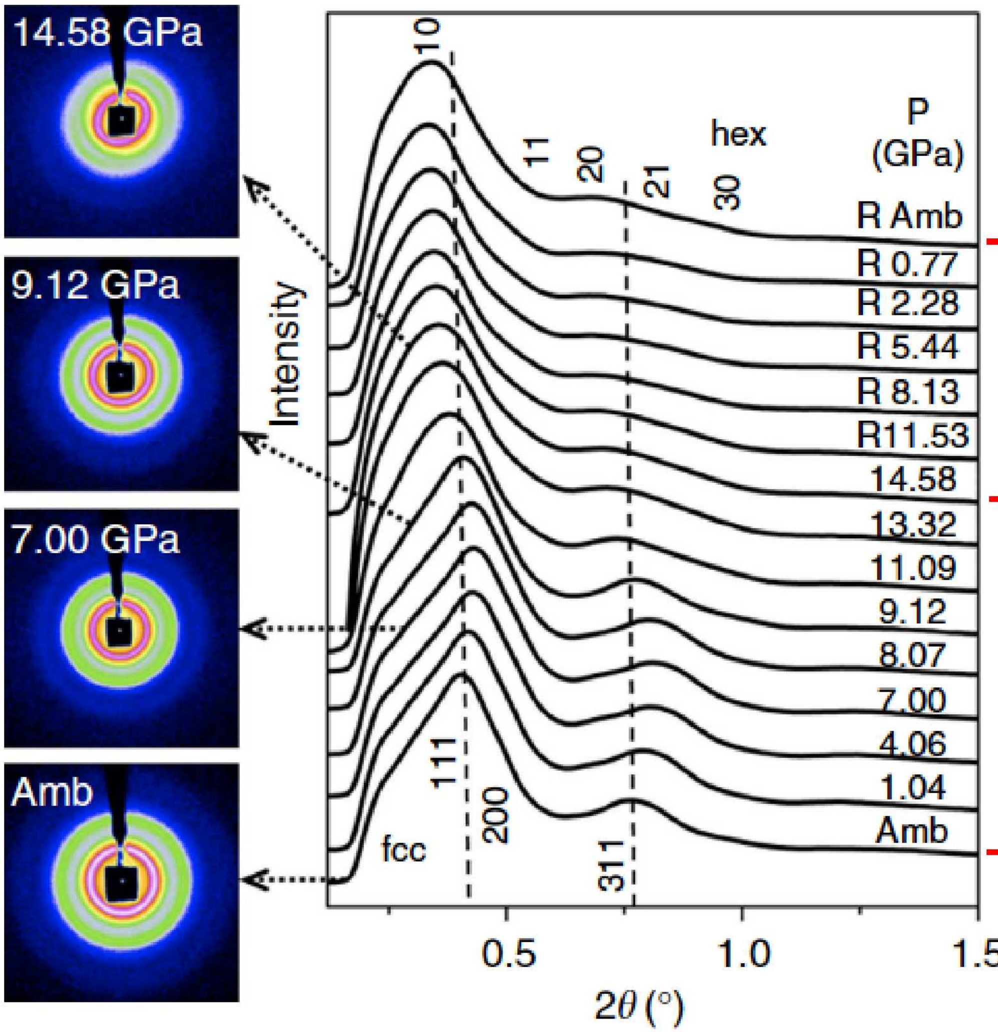
Before Compression – Starting Materials: Ag Nanoparticles



Wang, *et al*. *J. Am. Chem. Soc*. 133, 14484–14487 (2011).

Li, B.; Fan H., *et al*., *Nat.Commun.* 5:4179 doi: 10.1038/ncomms5179 (2014).

Structural Evolution of Ag Nanoparticle Arrays during Compression and Release



R-Ambient
Hexagonal
***a* = 94.63 Å**

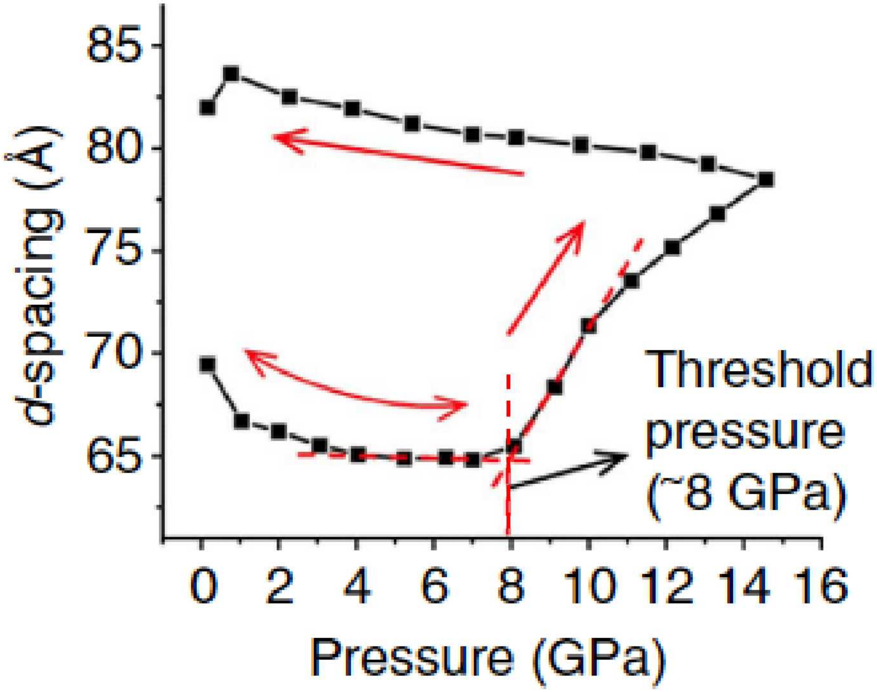
14.58 GPa
Hexagonal
a= 90.44 Å

Ambient
Cubic, *fcc*
a= 119.83 Å

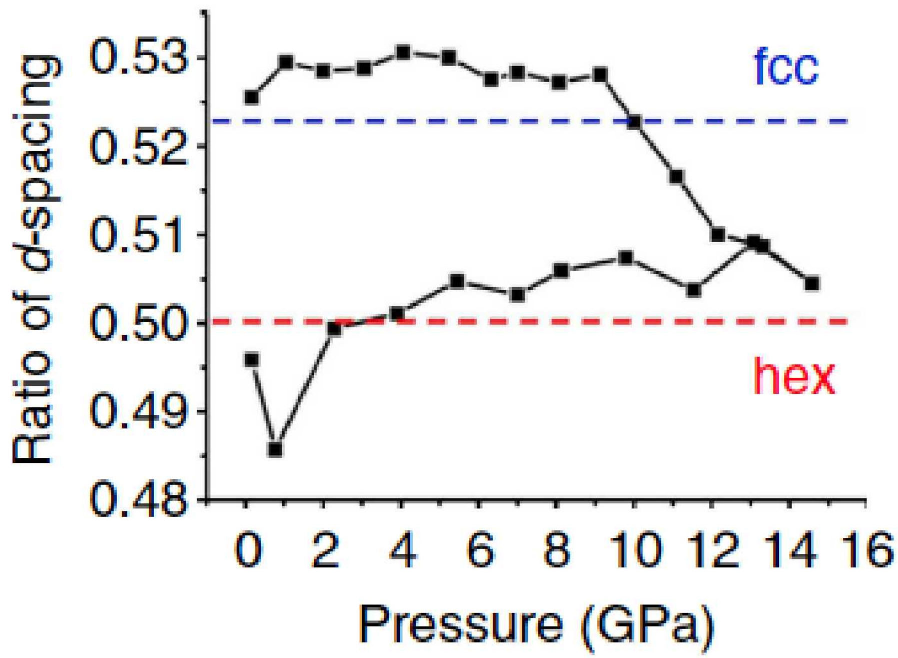
Wang, et al. *J. Am. Chem. Soc.* 2011, 133, 14484–14487.

Li, B.; Fan H., *et al.*, *Nat.Commun.* 5:4179 doi: 10.1038/ncomms5179 (2014).

Structural Evolution of Ag Nanoparticle Assemblies during Compression and Release



d_{111}



d_{311}/d_{111}

Li, W. et al., *Nano Lett.* 2014, 14, 4951−4958.
Li, B.; Fan H., *et al.*, *Nat.Commun.* 5:4179 doi: 10.1038/ncomms5179 (2014).

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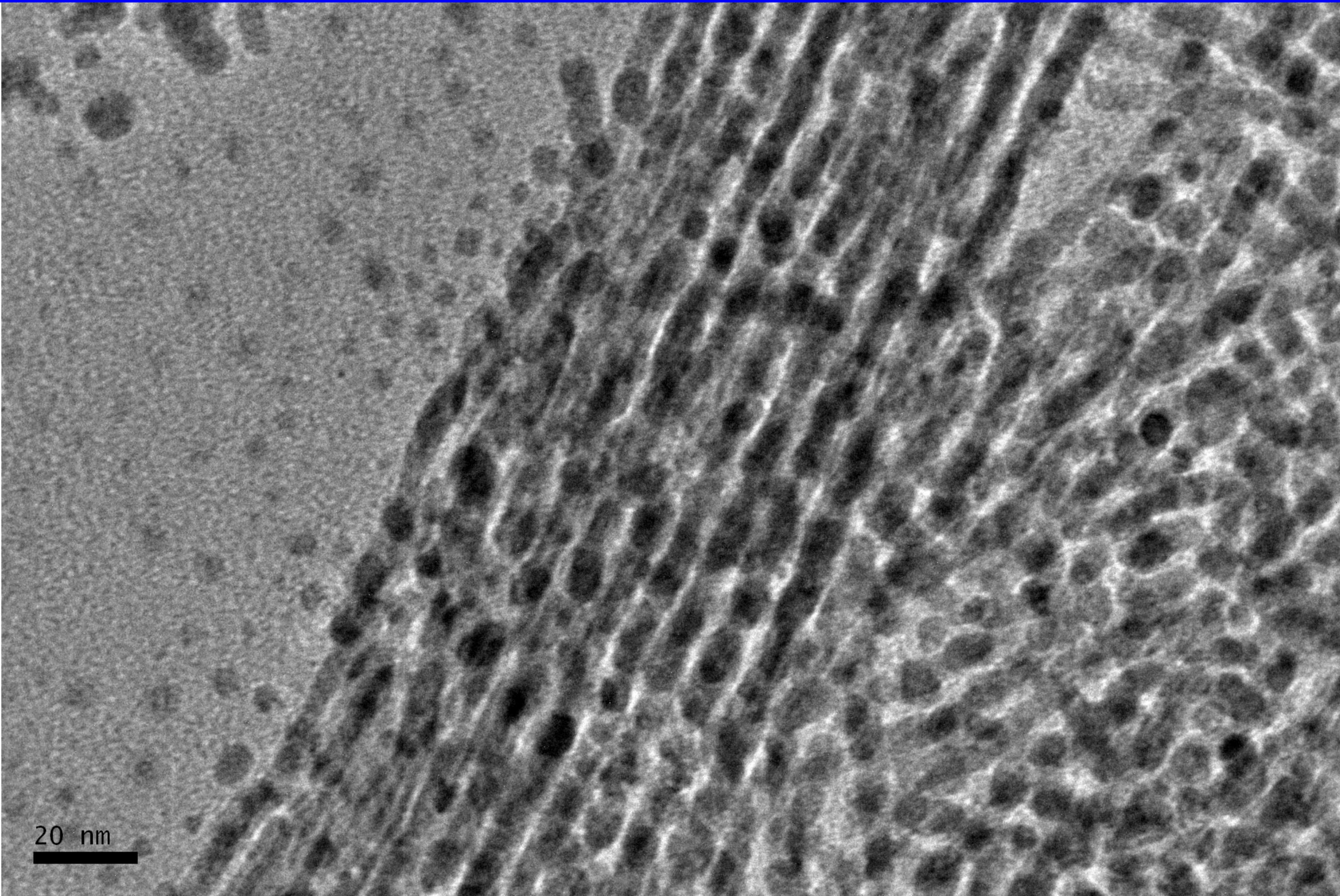
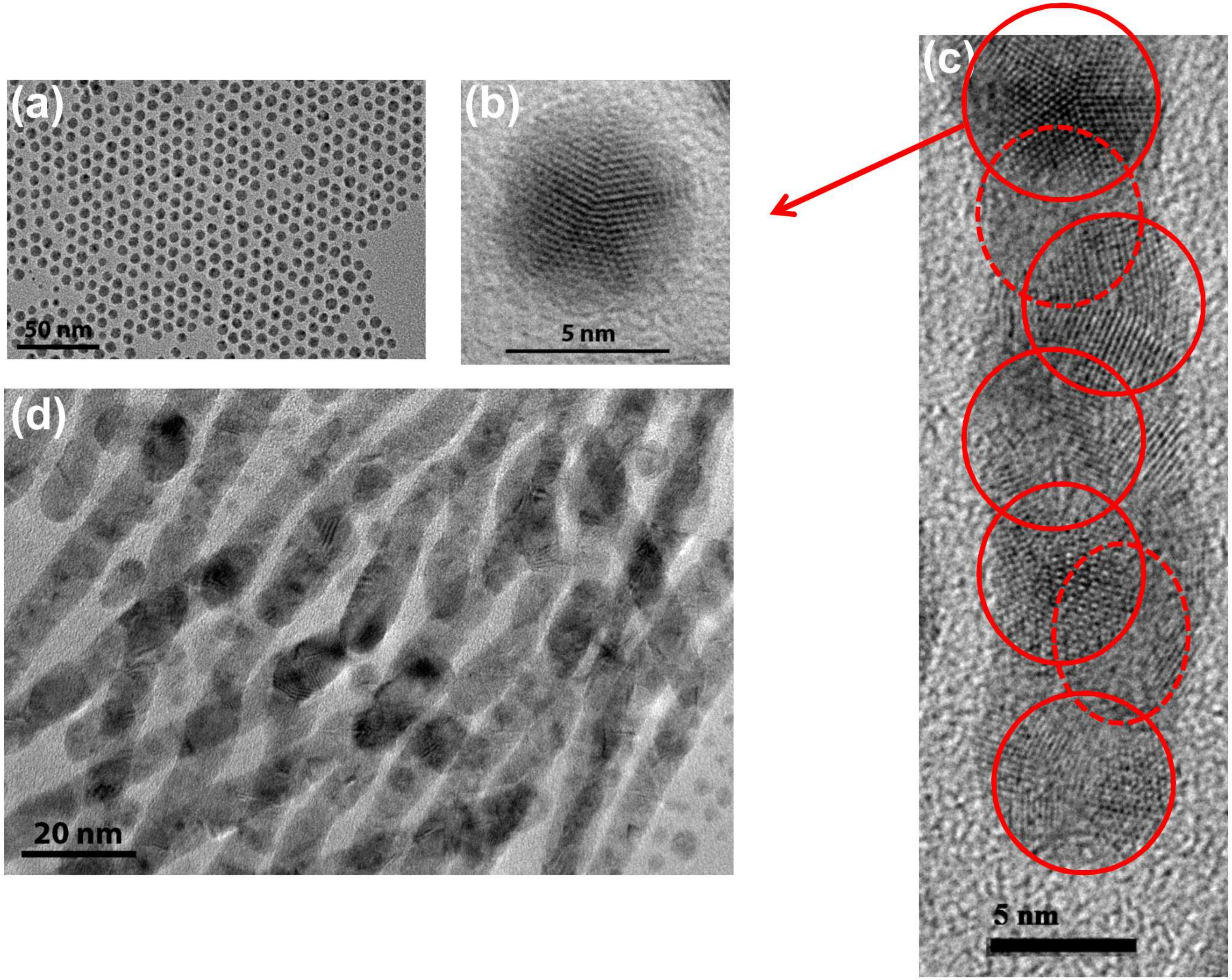
1D Ag Nanowires



Oriented Consolidation and Formation of Ag Nanowires



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Li, B.; Fan H., *et al*., *Nat.Commun.* 5:4179 doi: 10.1038/ncomms5179 (2014).

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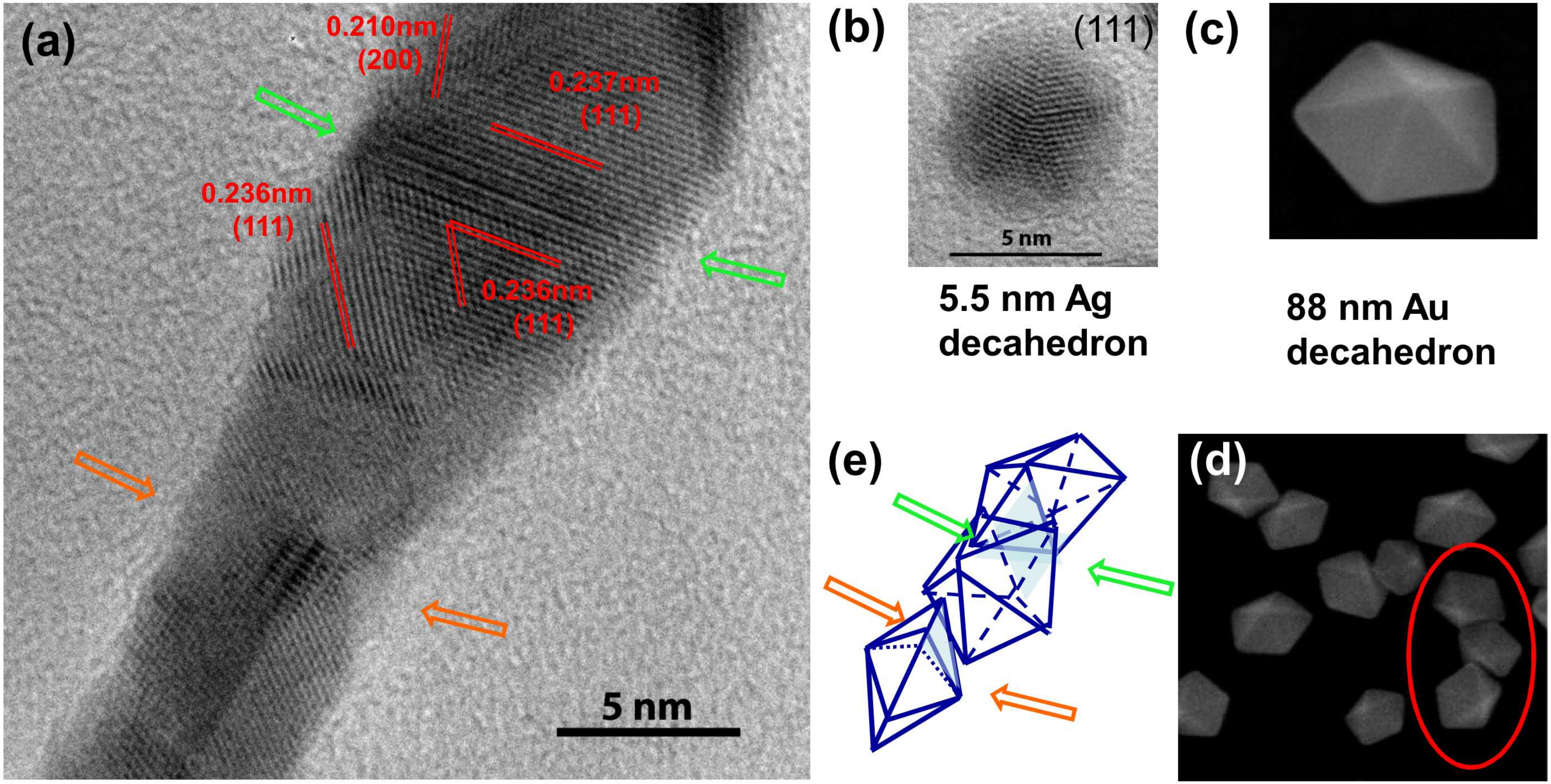
Oriented Consolidation and Formation of Ag Nanowires

Ag nanoparticles connect together through (111) planes to (111) planes since (111) is the major plane on surface of decahedron-shaped Ag nanoparticles.



Song, H. *et al.*, *J. Phys. Chem. C,* 112, 2469-2475, 2008.

Wang, et al. *J. Am. Chem. Soc.* 2011, 133, 14484–14487.

Li, B.; Fan H., *et al.*, *Nat.Commun.* 5:4179 doi: 10.1038/ncomms5179 (2014).

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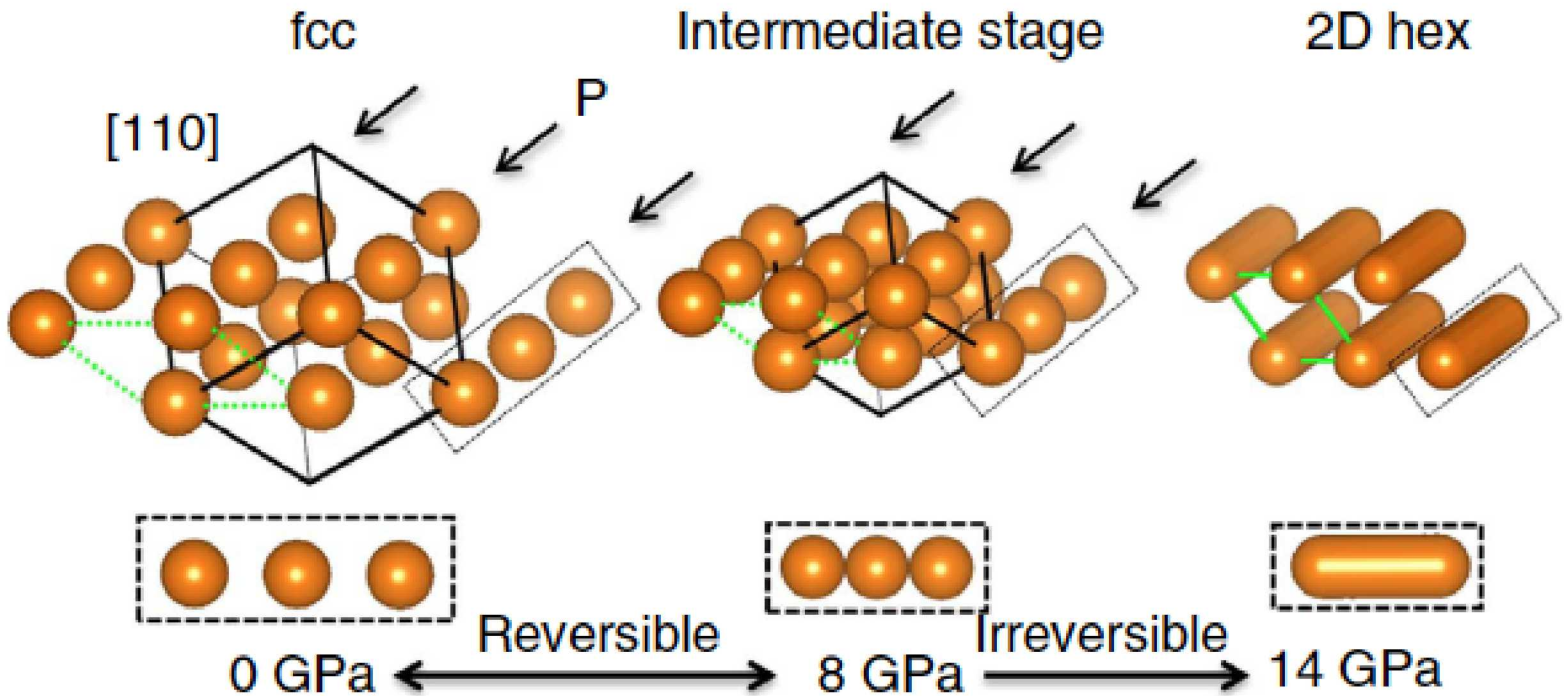
Stress-Induced Assembly and Consolidation of Nanoparticle Superlattices



Li, W. et al., *Nano Lett.* 2014, 14, 4951−4958

Li, B.; Fan H., *et al.*, *Nat.Commun.* 5:4179 doi: 10.1038/ncomms5179 (2014).

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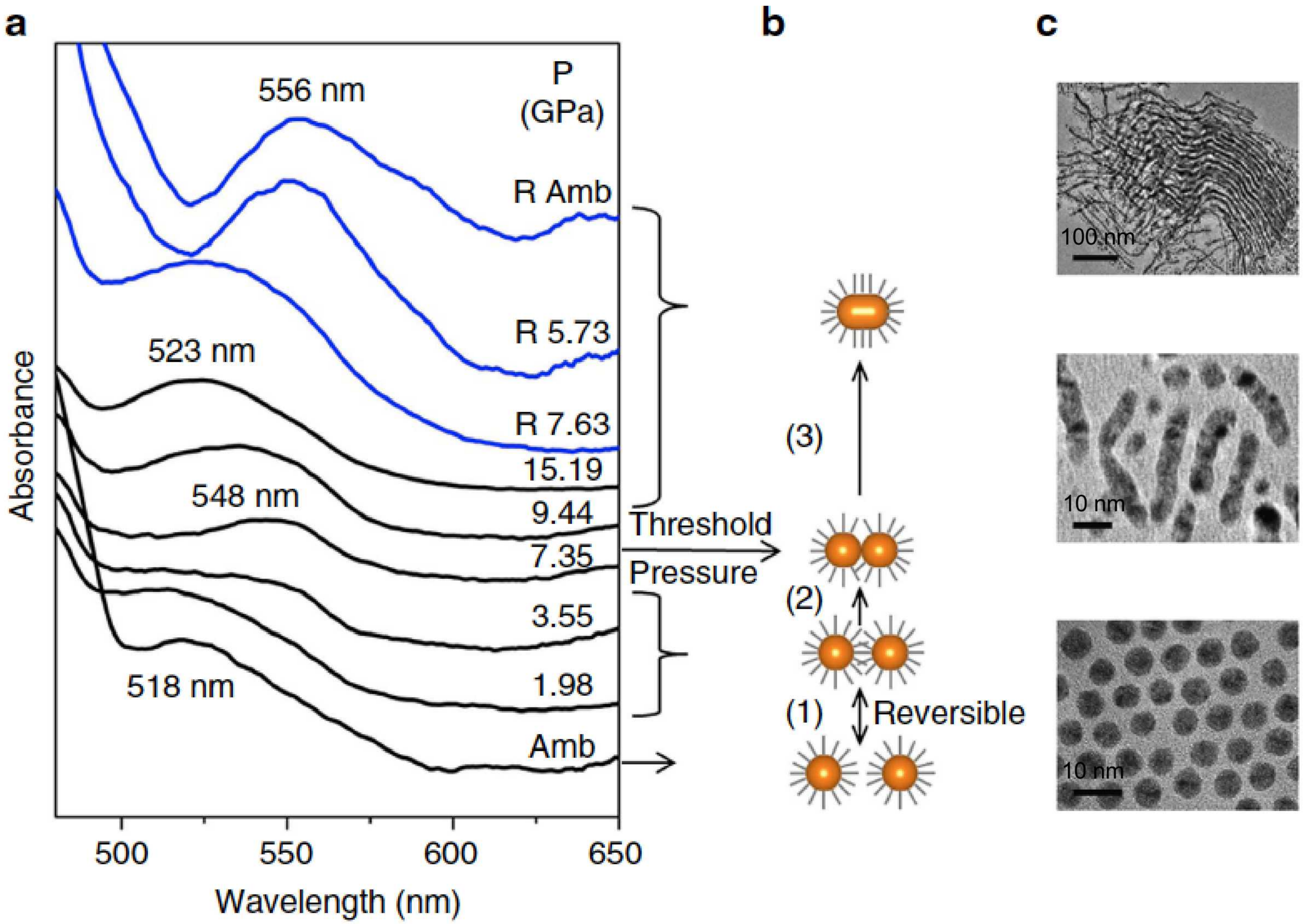
Pressure-Tuned Nanoparticle Interactions and Coupling

Structure - optical property correlation of Ag nanoparticle arrays



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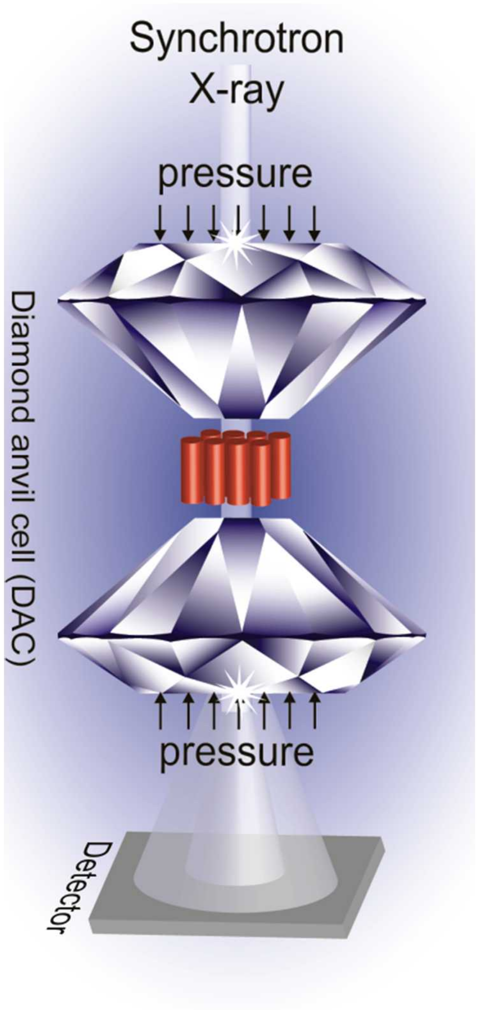
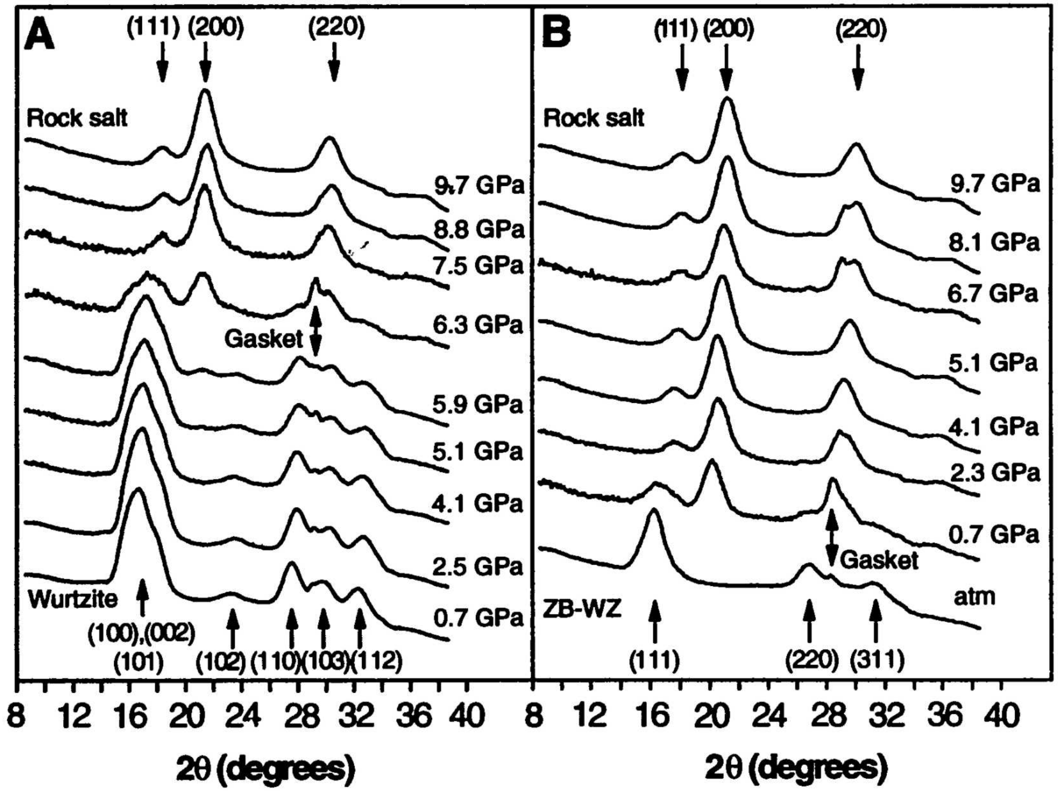
Adv. Mater. 2016, 28, 1989–1993.
Li, B.; Fan H., *et al*., *Nat.Commun.* 5:4179 doi: 10.1038/ncomms5179 (2014).

Semiconductor Nanoparticles (CdSe) under Pressure

Previous studies were focused on structural transformation in atomic lattice of CdSe nanoparticles and associated optical property changes under pressure.



Wurtzite → Rock salt → Zinc Blende



Tolbert S.H., Alivisatos A.P., *Science,* 265: 373-376, 1994
& *J. Chem. Phys.* 102: 4642-56, 1995.

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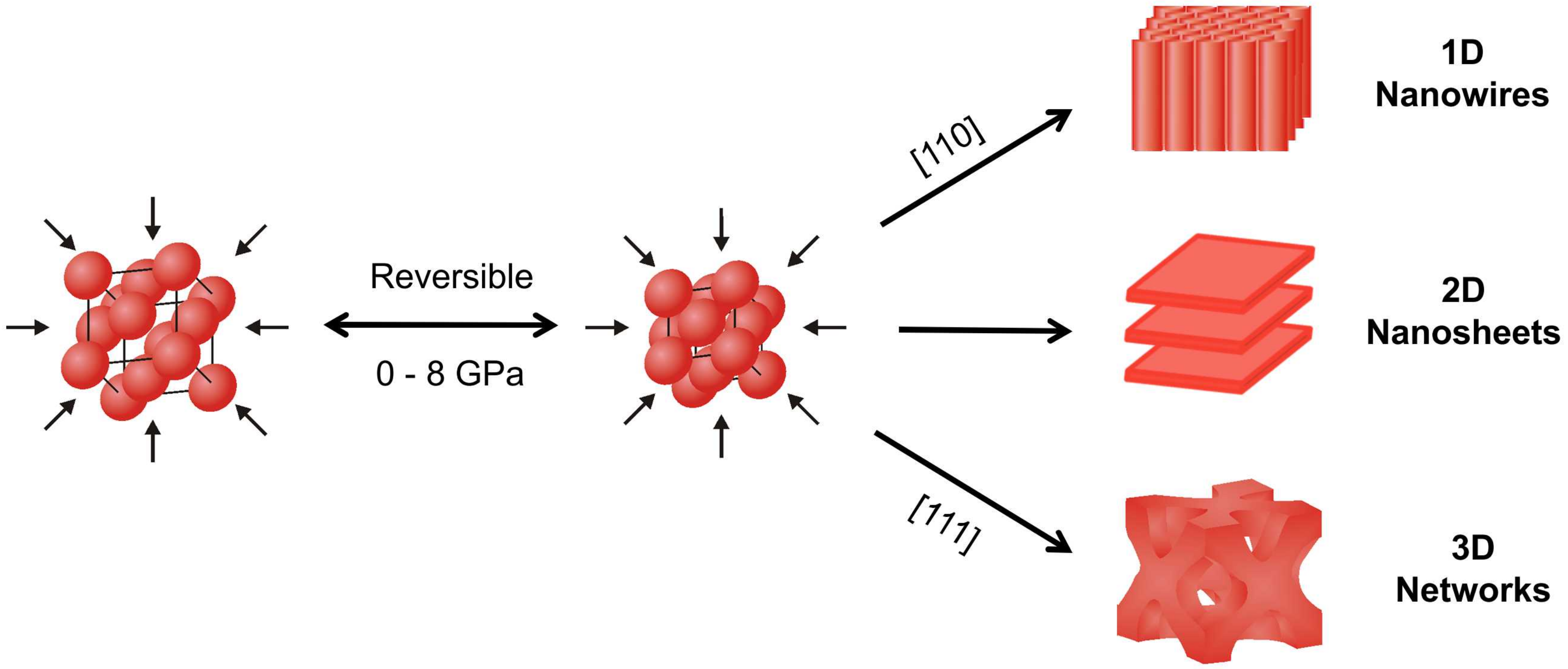
Semiconductor Nanoparticles (CdSe) under Pressure

Our studies were focused on structural transformation in CdSe nanoparticle mesophase and fabrication of new nanostructures under pressure.



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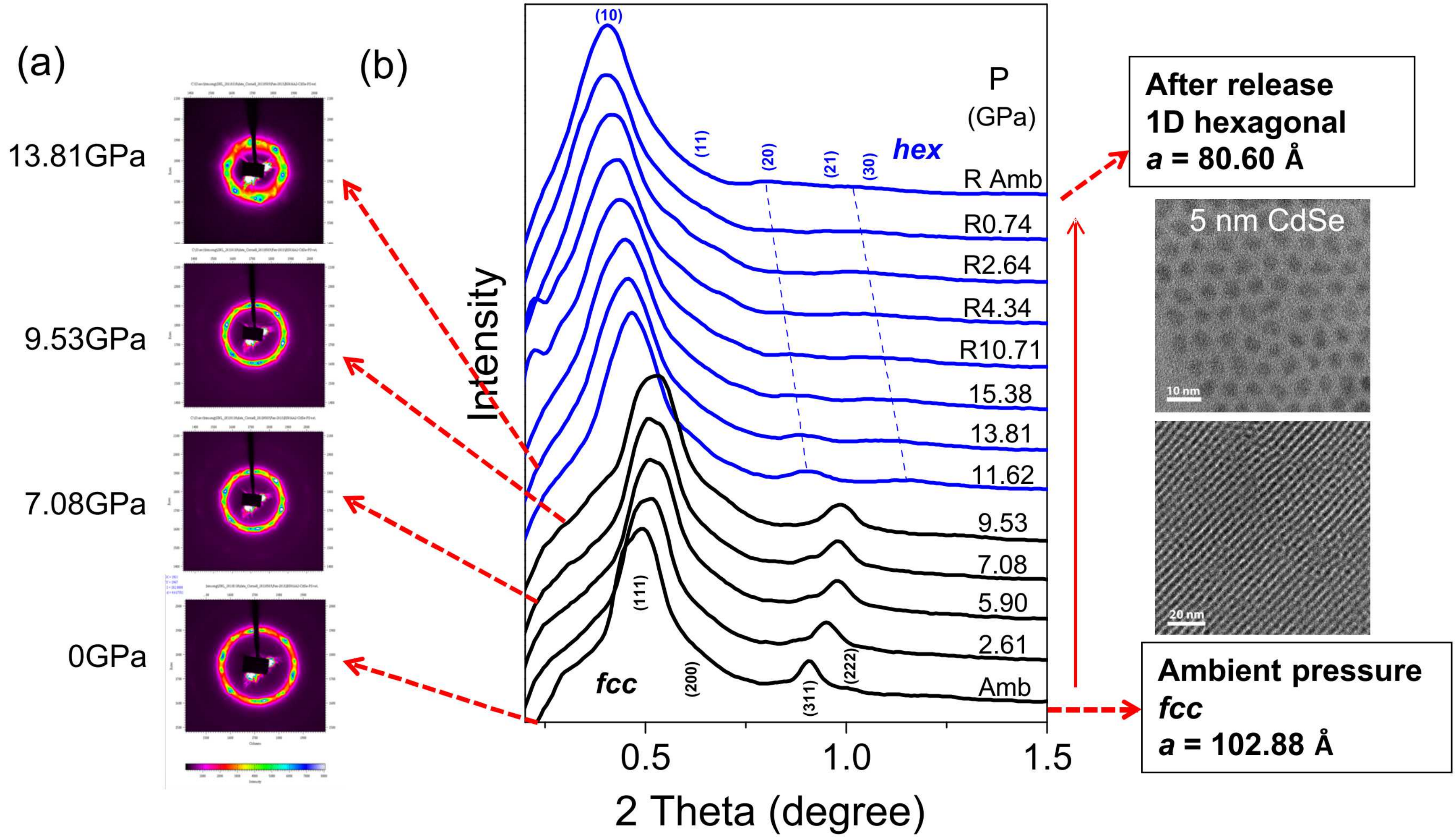
Li, B. et al. *Nat. Commun*. 8, 14778 doi: 10.1038/ncomms14778 (2017).

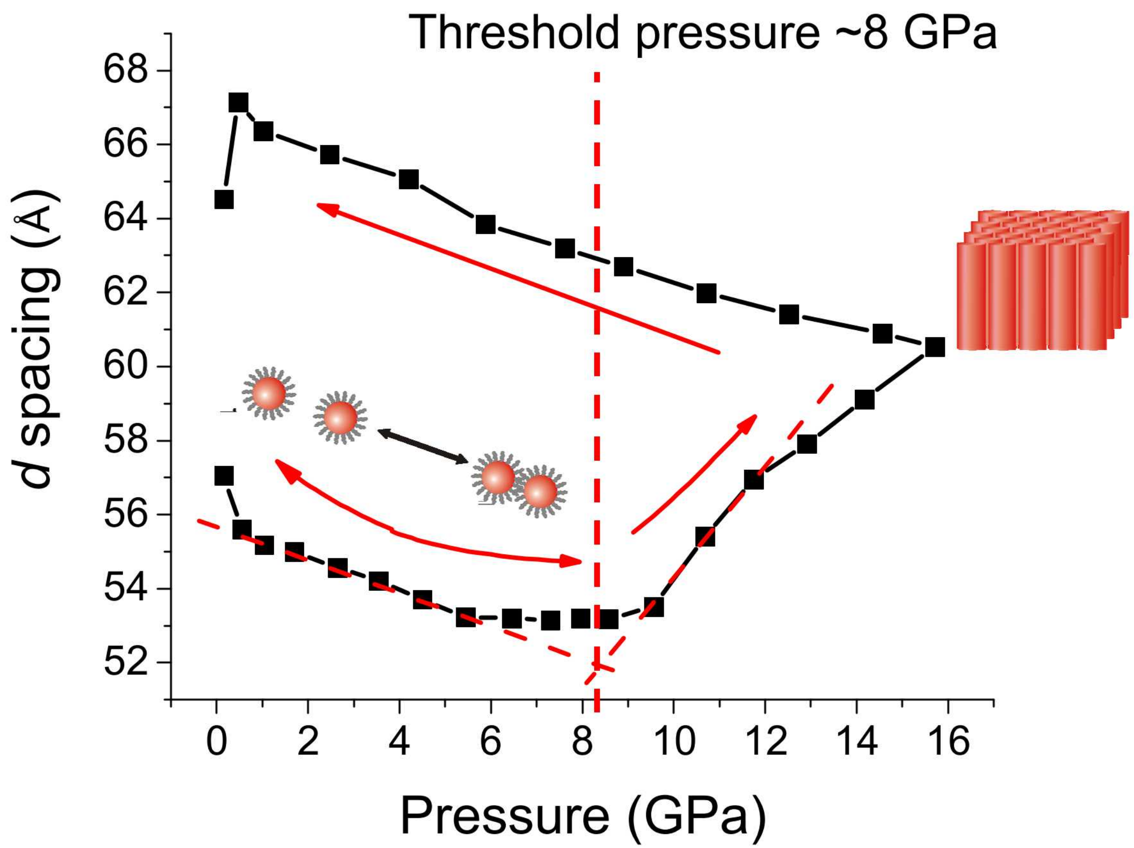
Structural Evolution of Ordered, Self-assembled CdSe Nanoparticle Arrays under Pressure

5 nm CdSe nanoparticles coated with Octadecylphosphonic acid (ODPA), Trioctylphosphonic oxide (TOPO)



B. Li, K. Bian, et al, *Science Advances* 3, e1602916 (2017).

Tunable Interparticle Spacing in CdSe Arrays during Compression and Release



Graph shows the *d*-spacing of the first Bragg reflection in each HP-SAXS spectrum.

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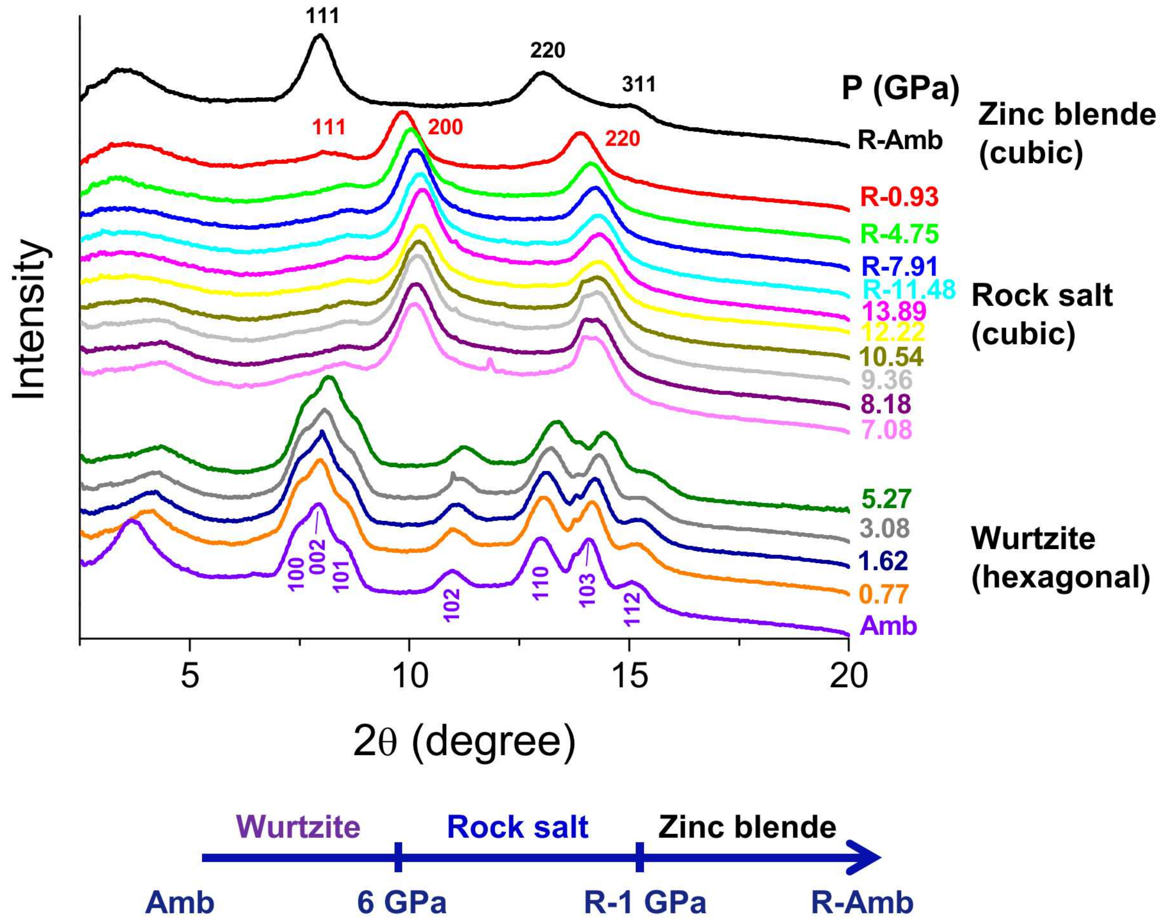
B. Li, K. Bian, et al, *Science Advances* 3, e1602916 (2017).

Atomic Lattice Phase Transition of CdSe Nanoparticles during Compression and Release



B. Li, K. Bian, et al, *Science Advances* 3, e1602916 (2017).

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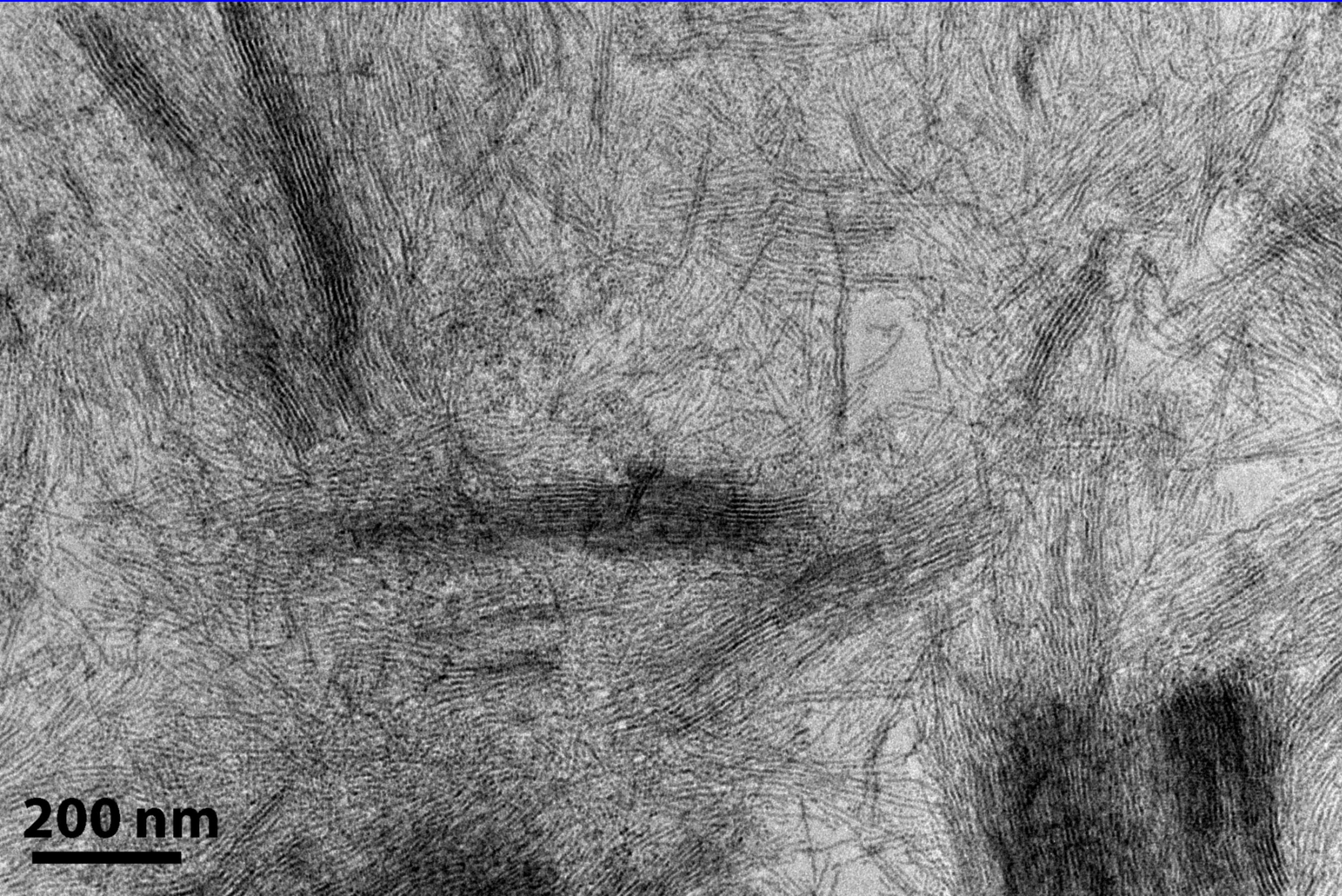
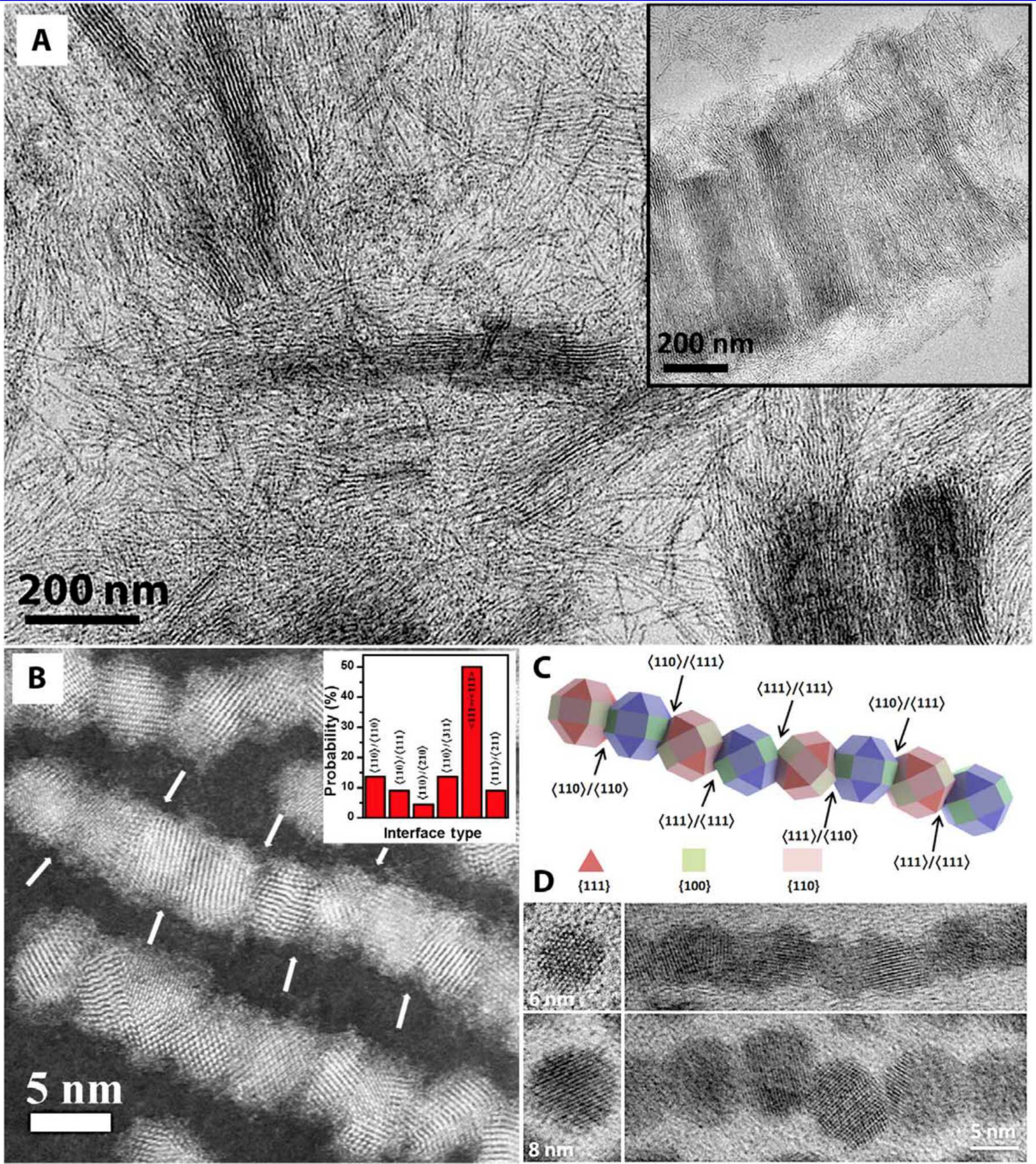
Transmission Electron Microscopy Image of CdSe Nanowires



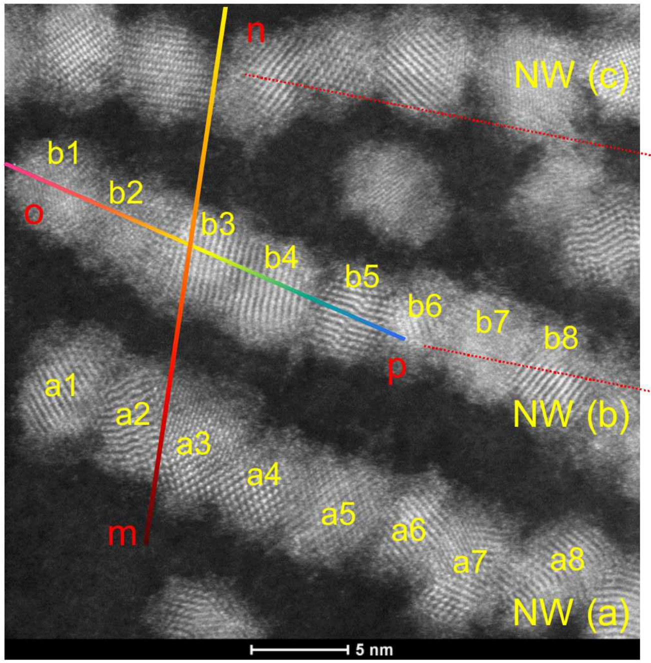
Transmission Electron Microscopy Image of CdSe Nanowires



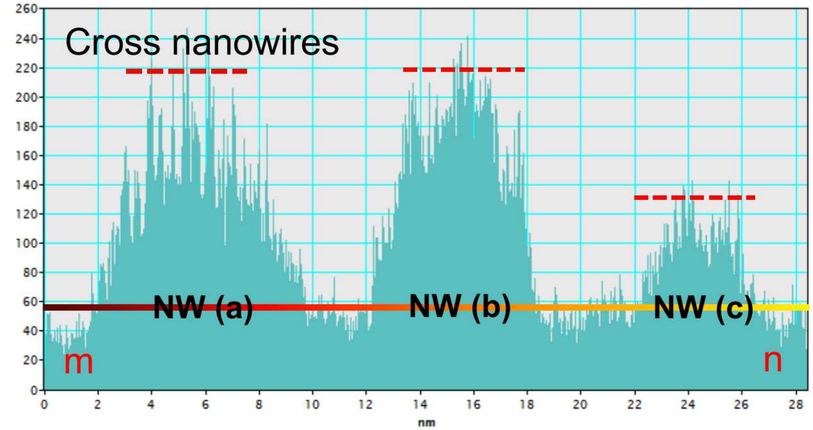
B. Li, K. Bian, et al,
Science Advances
3, e1602916 (2017).

Scanning Transmission Electron Microscopy Image of CdSe Nanowires

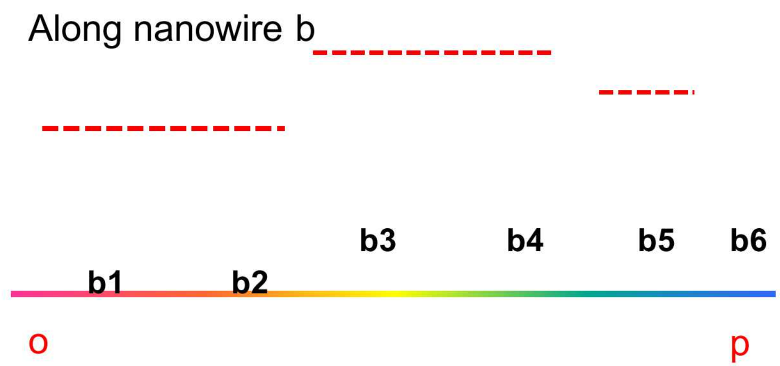
STEM image



TEM by Dr. Ping Lu

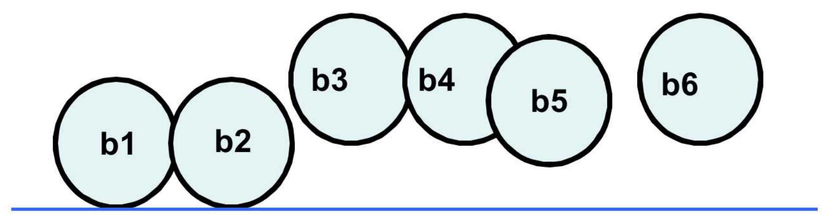


Height profile

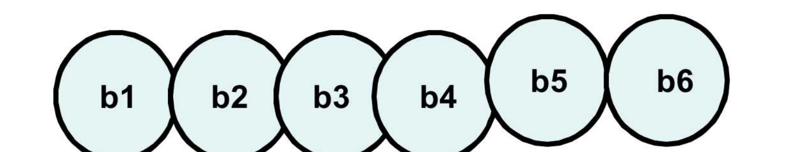


Nanowire (b)

Side view

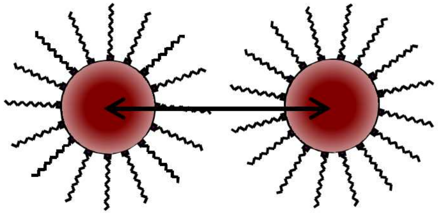


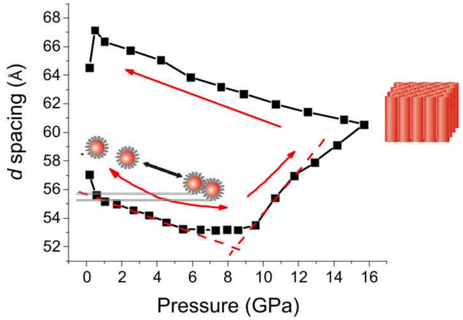
Top view

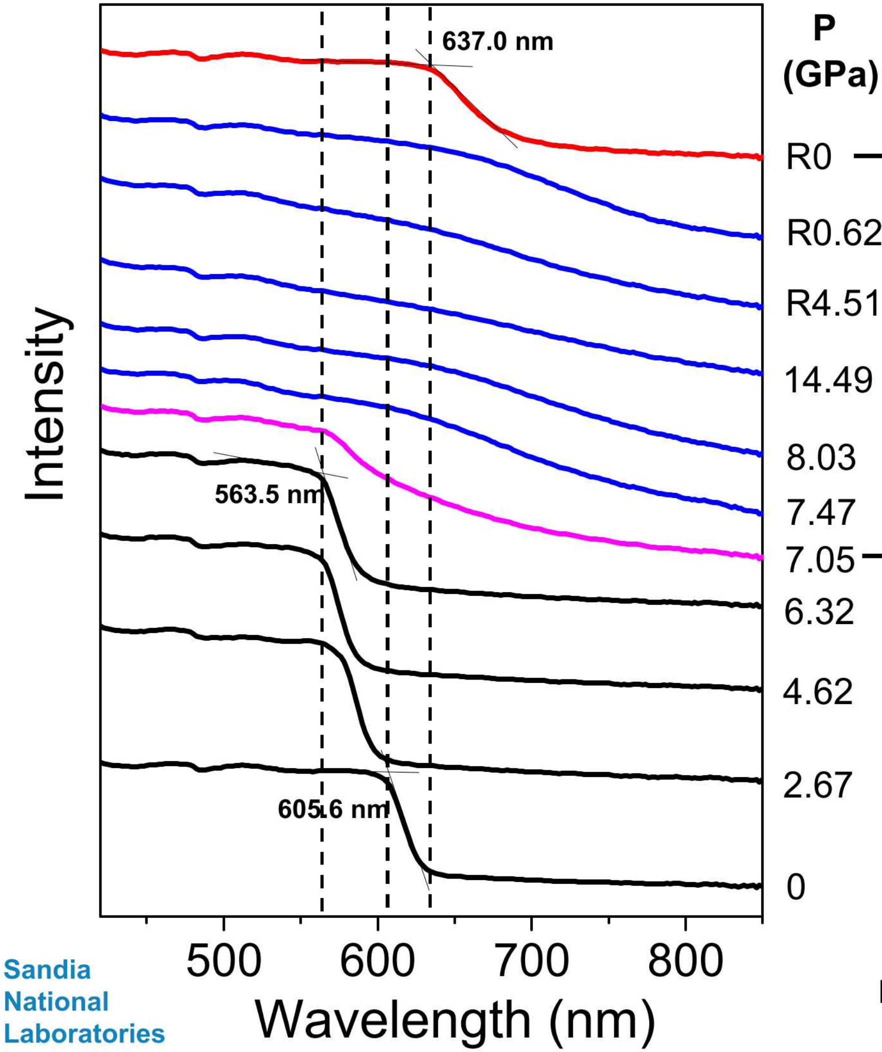


B. Li, K. Bian, et al, *Science Advances* 3, e1602916 (2017).

Stress Induced CdSe Nanoparticles Interactions







R0 → Absorption peak became sharper and stronger. Absorption peak shows 31 nm red shift (from 605.6 nm to 637.0 nm).

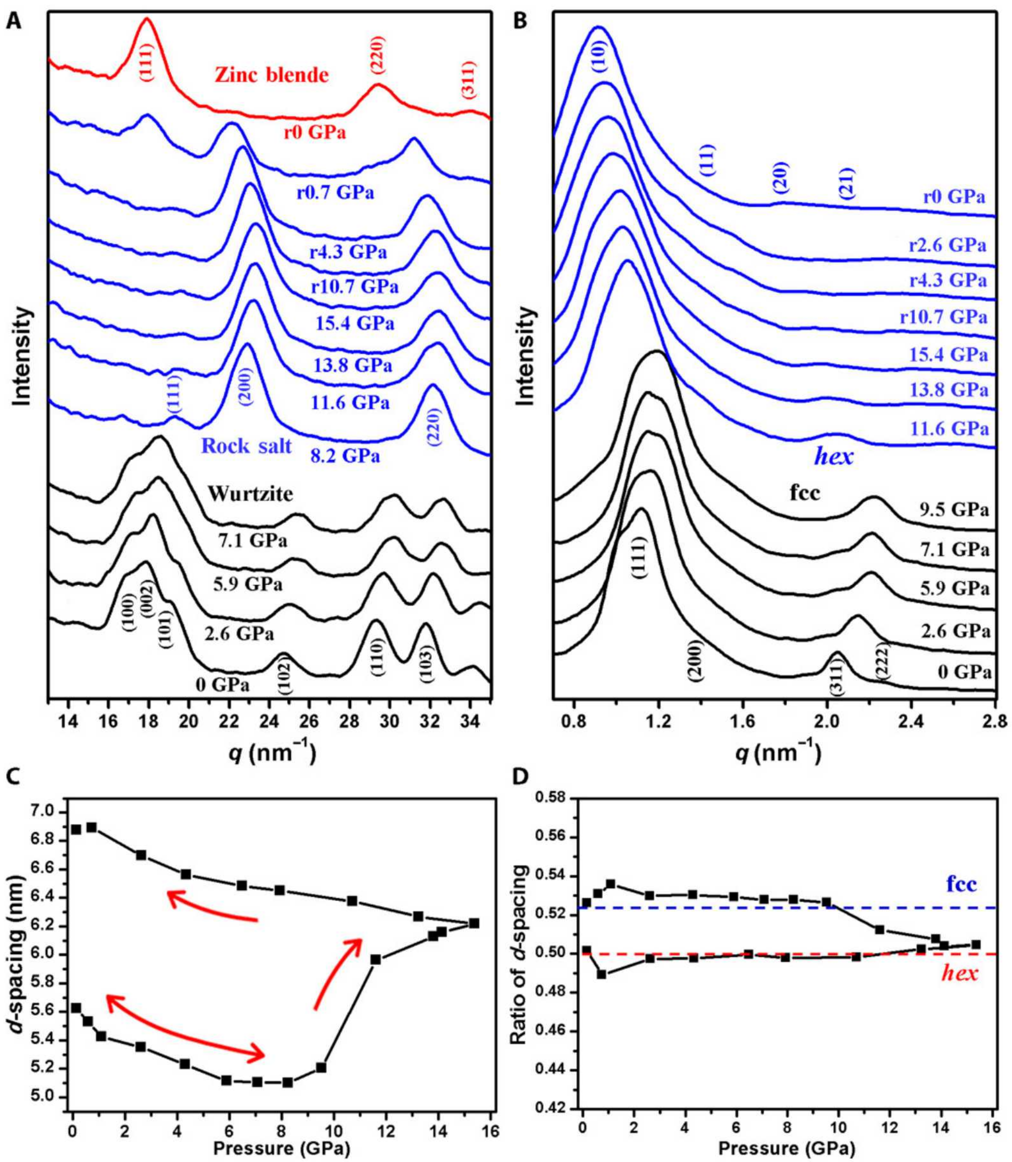
Absorption peak further extends to longer wavelength (up to about 800 nm), indicating nanowires formation.

7.05 → Absorption peak starts to extend to longer wavelength at around 7 GPa, indicating the nanoparticles become consolidated.

Absorption peak blue shifts due to nanoparticle aggregation under pressure. The peak blue-shifts until pressure reaches to 4.62GPa, then remains almost no change until 6.32GPa, indicating the nanoparticles become very close or start to contact at ~ 4.62GPa.

B. Li, K. Bian, et al, *Science Advances* 3, e1602916 (2017).

Pressure Induced Phase Transition on Multiple Scales

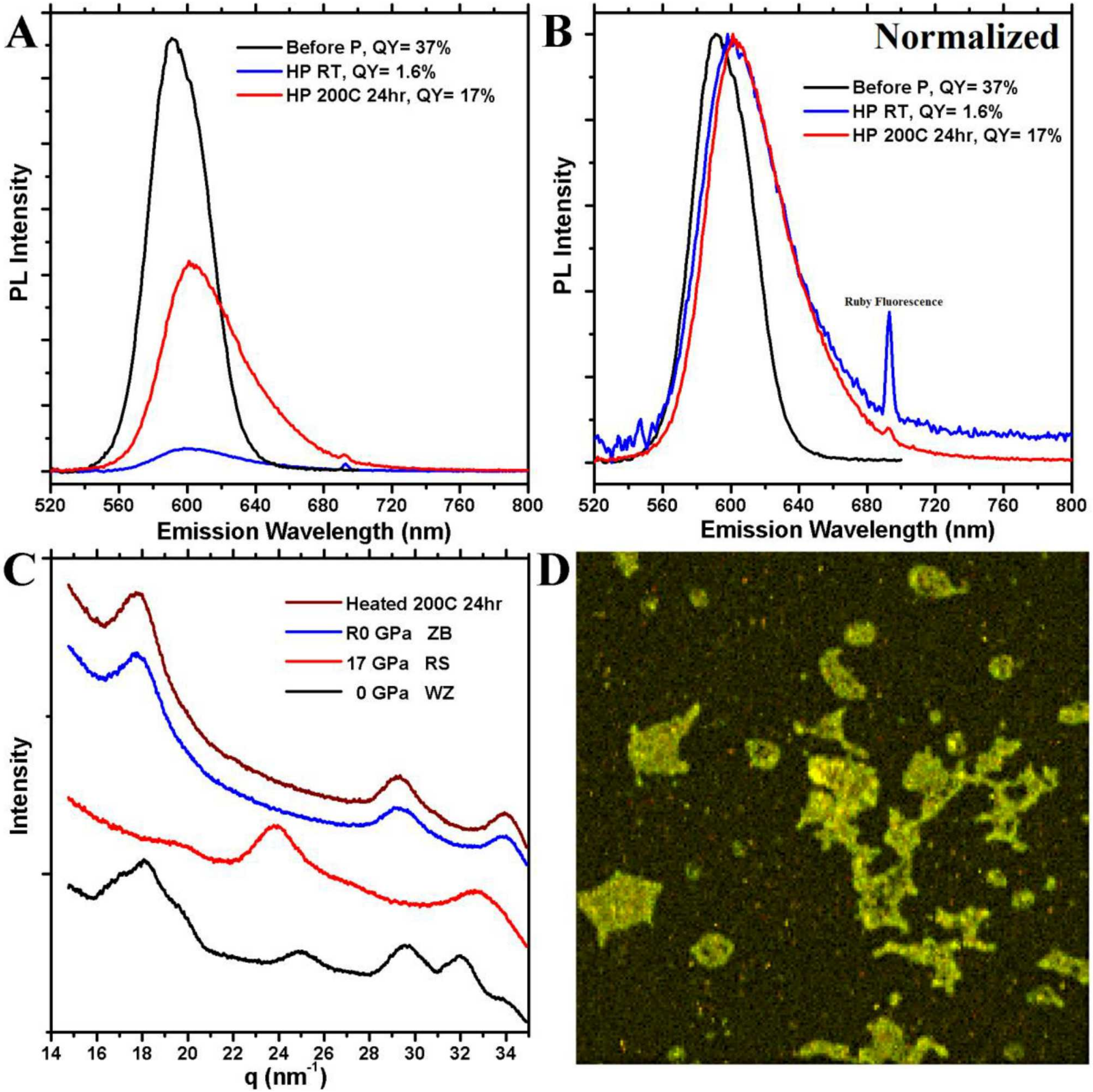


Atomic scale phase transition

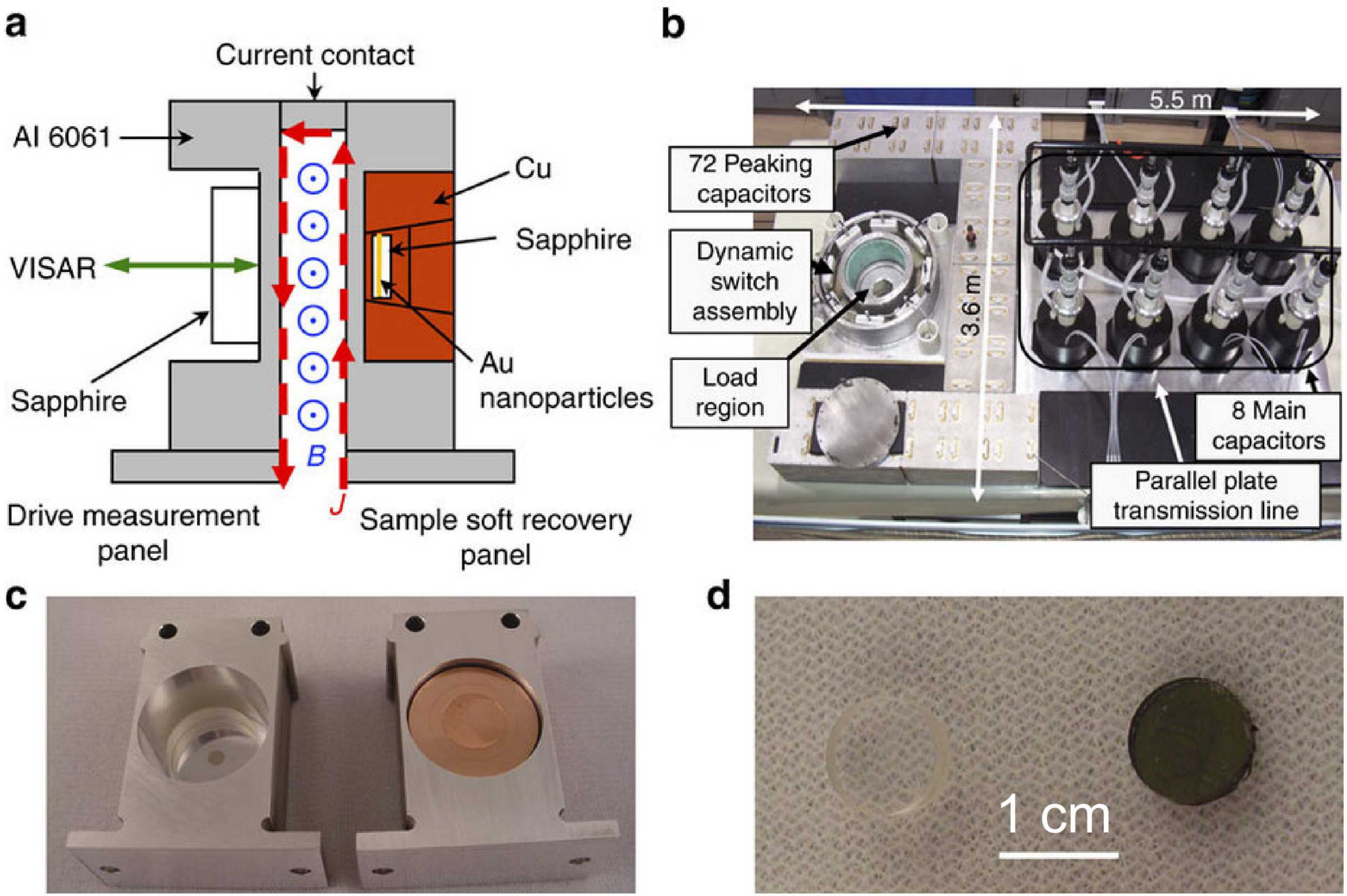
Mesoscale phase transition

B. Li, K. Bian, et al, ***Science Advances*** 3, e1602916 (2017).

Optical Property of CdSe Nanowires under Pressure

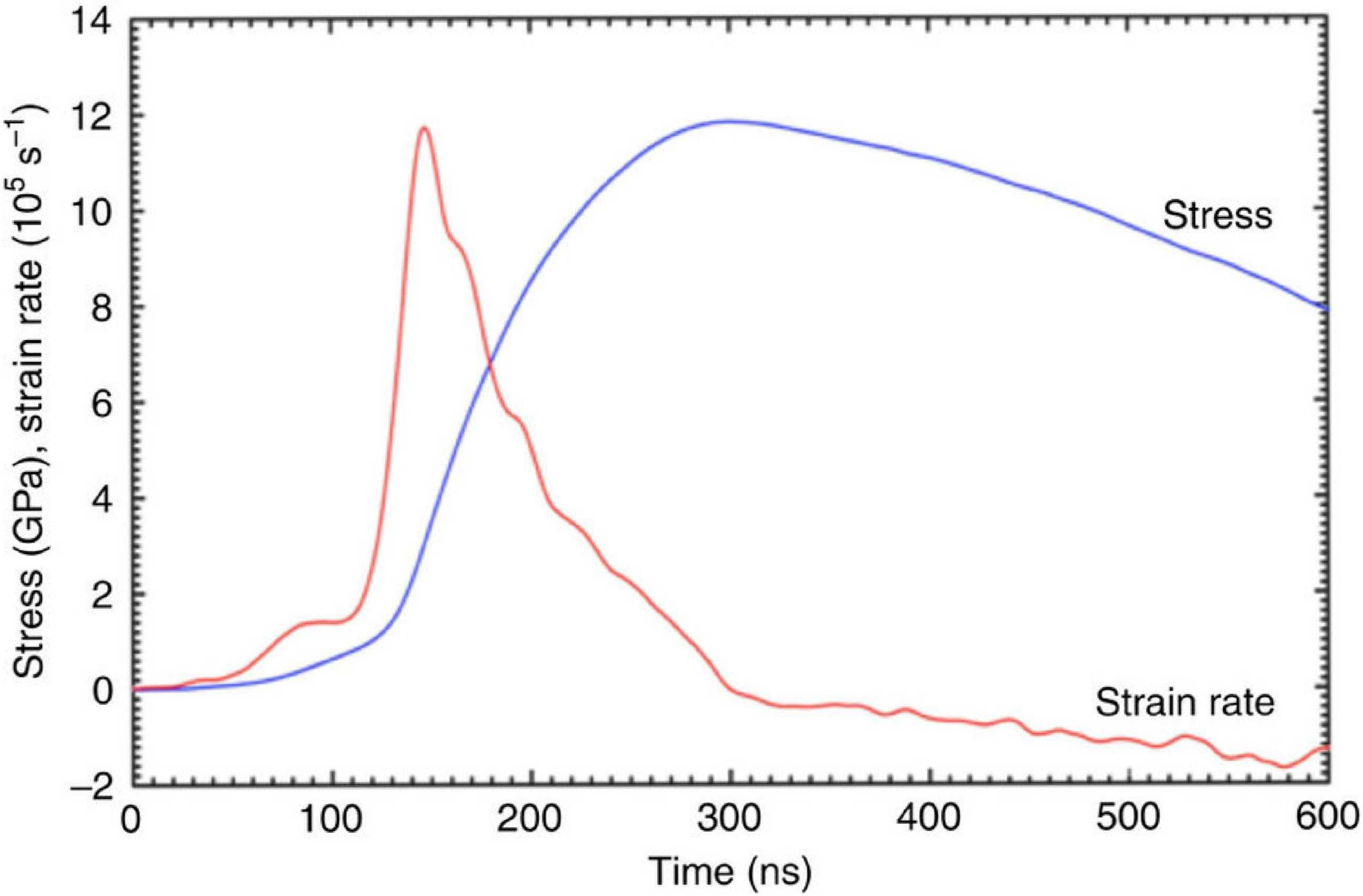


Ultrafast assembly and synthesis of gold nanostructures using nanosecond compression via pulsed power



Li, B. et al. *Nat. Commun*. 8, 14778 doi: 10.1038/ncomms14778 (2017).

Ultrafast assembly and synthesis of gold nanostructures using nanosecond compression via pulsed power



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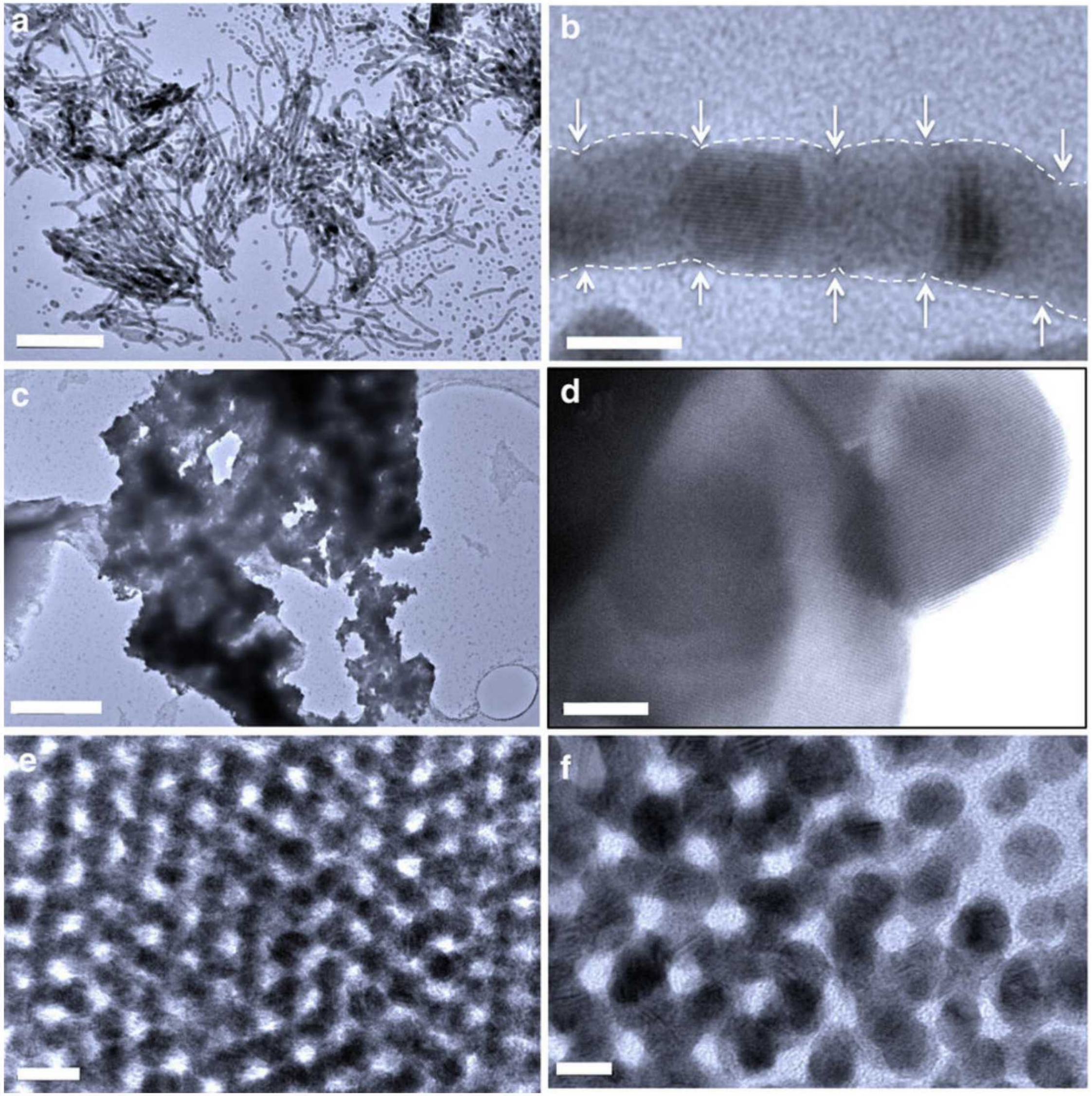

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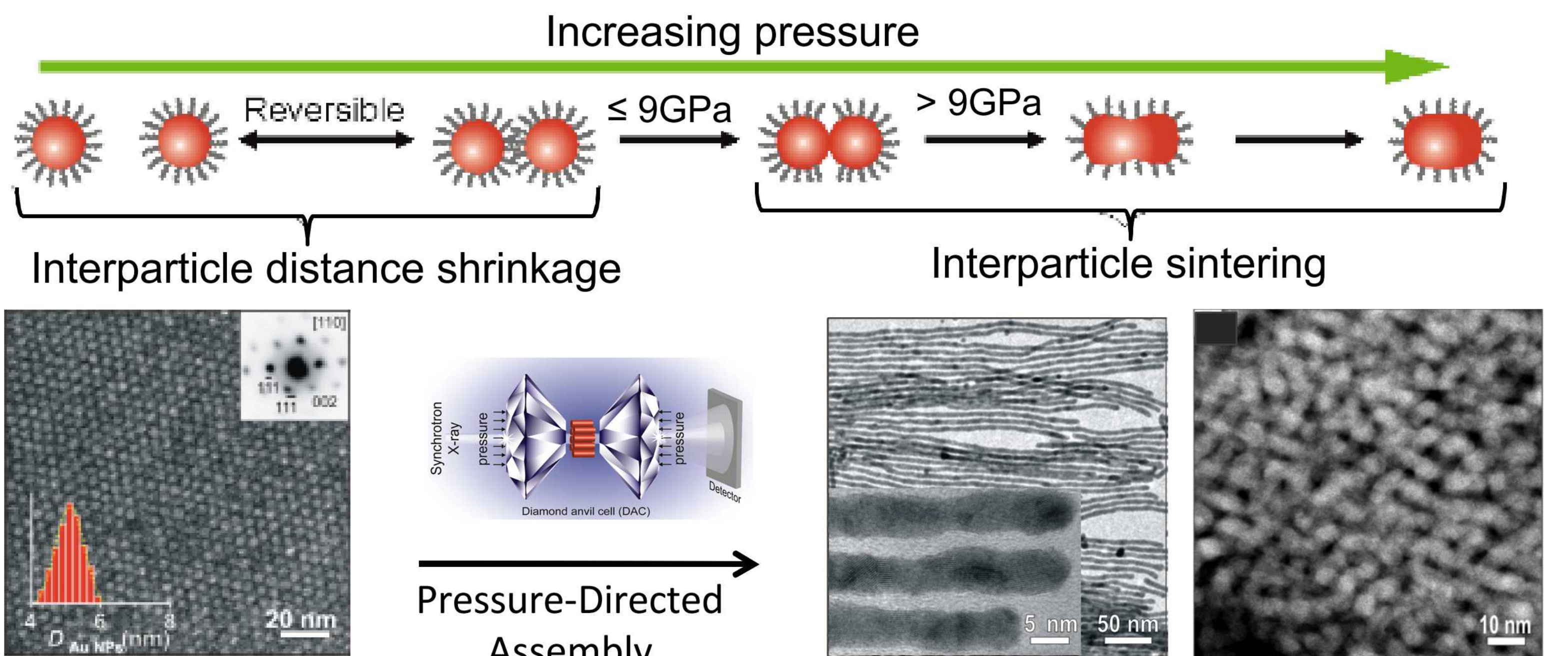
Li, B. et al. *Nat. Commun*. 8, 14778 doi: 10.1038/ncomms14778 (2017).

Summary: Pressure-Induced Nanoparticle Engineering

Pressure-Directed Assembly presents a paradigm shift in engineering nanoparticle arrays:

• Allow precise, systematic, and reversible tuning of interparticle distance for interrogation of new chemical and physical properties.

• Produce new chemically and mechanically stable 1-3D nanostructures, which is not possible for current top-down and bottom up methods.



3D spherical Nanoparticle array (Before compression)

1-3D nanostructures (After compression)

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Acknowledgements

Dr. Zaicheng Sun
UNM postdoc

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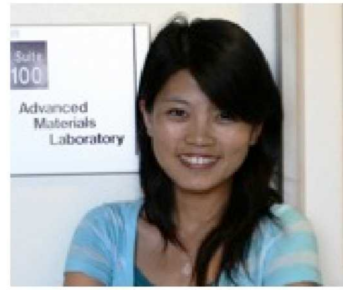
UNM postdoc



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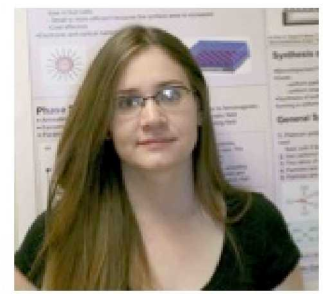

Samantha Schmitt
UNM student

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