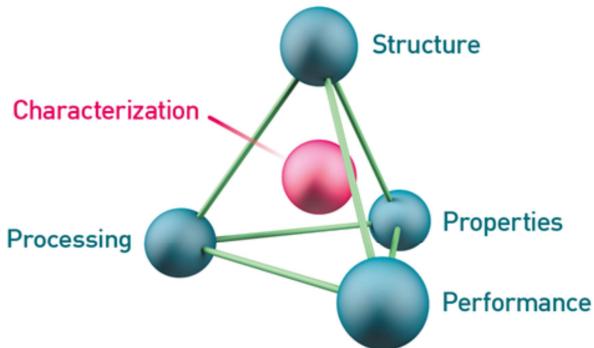


# M5052 CHARACTERIZATION OF MATERIALS AND NANOMATERIALS

*Graduate Program in Nanotechnology*



**PROF. YADIRA I. VEGA CANTÚ**

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## COURSE OBJECTIVES

- Present the principles and applications of some of the basic techniques for characterization of materials and nanomaterials
- Present the principles and applications of some techniques needed specifically for nanomaterials characterization
  
- Learning Objectives: At the end the student should be able to select a characterization technique for a material or nanostructure based on the type of material and properties to measure
  
- Final Project Objective: Linking the class topics to the current research projects of the students



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# SYLLABUS

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1. Introduction to Instrumental Analysis and Interactions of Light and Matter
2. Vibrational Spectroscopy: Infrared Spectroscopy, Raman Spectroscopy, SERS
3. Molecular Spectroscopy:
  - 3.1 UV-vis
  - 3.2 Fluorescence
4. X-Ray Methods:
  - 4.1 Diffraction XRD
  - 4.2 XRF, EDS, XPS
5. Introduction to Optical Microscopy and Nanoscopy
6. Electron Microscopy
  - 6.1 SEM
  - 6.2 TEM
  - 6.3 Other EM (etc.)
7. Scanning Probe Microscopies
  - 7.1 STM
  - 7.2 AFM
  - 7.3 other SPM
8. Thermal Analysis: TGA, DTA, DSC, TMA, DMA



## BACKGROUND KNOWLEDGE REQUIRED OF ENROLLED STUDENTS

---

- Bonding in solids
  - Ionic
  - Covalent
  - Metallic
- Structure of crystalline solids
  - Crystalline structures, amorphous
- General properties of materials
  - Metals
  - Ceramics
- Polymers
- Basic concepts in nanomaterials
  - Nanoparticles
  - Metallic, oxides
  - Quantum dots
  - Carbon nanomaterials
  - Carbon nanotubes, graphene, fullerenes



# RECOMMENDED TEXTBOOKS

---

- J.P. Sibilia [Ed.] “A Guide to Materials Characterization and Chemical Analysis” 2nd. ed., Wiley-VCH (1996)
  - Contains basic descriptions and examples of applications to materials for a large variety of chemical and materials characterization techniques
- Encyclopedias of Science or of Chemical Technology
  - These usually have entries or chapters explaining different analytical techniques
- Douglas A. Skoog, F. James Holler, Stanley R. Crouch  
“Principles of Instrumental Analysis” 7th. ed. Cengage Learning, 2017
  - A classic textbook of instrumental analysis
  - Mexican edition in Spanish may be cheaper: D.A. Skoog, F.J. Holler, S.R. Crouch “Principios de Análisis Instrumental”, 7a ed., México : Cengage Learning, 2018



# SUPPLEMENTARY TEXTBOOKS

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- **W. D. Callister and D. G. Rethwisch**
  - Fundamentals of Materials Science: An Integrated Approach
- **W. Smith, J. Hashemi**, “Foundations of Materials Science and Engineering,”  
McGraw-Hill
- **J. Shackelford**, “Introduction to Materials Science for Engineers”, Pearson, Prentice Hall.
- **G. Hornyak**, et al. “Introduction to Nanoscience and Nanotechnology”, CRC Press.
- Additional/Supplementary reference books:
  - Materials Science
  - Chemistry of Materials
  - Solid State Chemistry



# DR. YADIRA I. VEGA

- Tecnológico de Monterrey
  - School of Engineering and Sciences (IEC)
  - Advanced Manufacturing Focus Group
  - Department of Sciences, area Chemistry and Nanotechnology
  - Innovation Center in Design and Technology
  
- Office: A1-404-C
- Office hours (unofficial): Monday 14:30 - 17:00, Wednesday 11:30 - 13:00 h
- **Please notify in advance**

Contact:

yadira.vega@itesm.mx



Yadira Vega-Cantu



@yivega



Yadira Vega-Cantu

- Preferably, contact me during office hours (8:00 - 18:00)



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## DR. VEGA'S ACADEMIC BACKGROUND



TECNOLÓGICO  
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- B.S. in Chemistry
- M.S. in Chemistry



RICE

- Ph.D. in Chemistry
- Post-doctoral training in Chemistry



- 2005 - 2012 Research Professor
- Advanced Materials Division



UNIVERSIDADE  
FEDERAL  
DE PERNAMBUCO

- 2012 - 2015 Visiting Professor
- Graduate Program in Materials Science



TECNOLÓGICO  
DE MONTERREY®

- 2016 - Currently Research Professor
- School of Engineering and Sciences
- Innovation Center in Design and Technology
- Advanced Manufacturing Focus Group
- Department of Chemistry and Nanotechnology



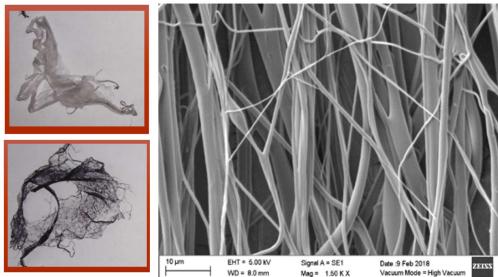
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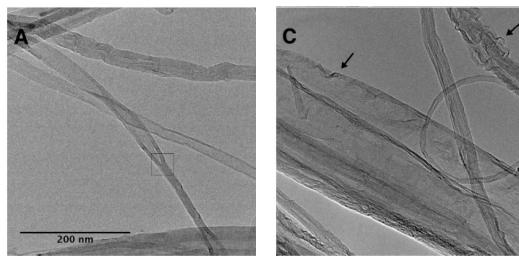
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### Nanocarbon Hybrid Systems



### Nanoribbons



### Composite Materials

C.G. Espinoza-González et al. Polystyrene composites with very high carbon nanotube loadings by *in situ* grafting polymerization

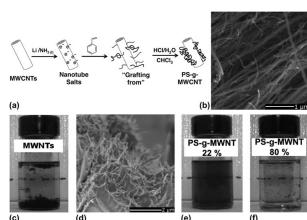


FIG. 1. Reaction scheme and effect of MWNT wt% on dispersibility in chloroform. (a) Reaction scheme. (b) SEM image of the purified MWCNT used as starting material, these do not disperse in chloroform, even with sonication, and flocculate (c). After functionalization the nanotubes are coated with styrene and the composite dispersion is stable over after 3 weeks for 1wt loadings (d), though for the highest loadings they flocculate (e).

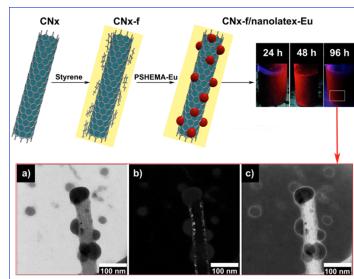


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### Photoluminescent Nanomaterials



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## COURSE ASSESSMENT: DISCUSSION OF EXAMPLES

- After each topic each student should look for one scientific paper showing an example of application of the analysis technique for materials or nanomaterials
- Journal should be indexed in Journal Citations Reports or Scopus, preferably with a Q1 or Q2 ranking
  - Articles from predatory journals are not acceptable
- All papers should be uploaded to a shared folder
- Each student should prepare one slide of their own article and upload it to the shared folder
  - Both Files should follow the format Name-Surname-Paper-Name
  - Also upload supporting information if necessary
- Uploaded examples will be discussed in class
  - All the students should read at least all the slides of their peers for in-class discussion



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# COURSE ASSESSMENT



- Final grade will have a numeric value of 1 - 100
- Exams and activities will be graded with a numeric value of 0 - 100

Final Grade

Activities  
50%

Final Project  
50%

Midterm grades reported will be an average of the grades of homework and activities



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## HOMEWORK & ADDITIONAL ACTIVITIES

Homework (problem sets) every week in Blackboard, will be handed over the following week through Blackboard.

- **IMPORTANT:** Submitted homework through Blackboard **should be scanned**. No photographs will be accepted.
- Should be submitted on the specified date and time, otherwise, a number of points will be deducted, depending on the delivery date.
- Problem sets will be solved and corrected in class, on the due date, and **will not be have a numerical grade**.
- Additional to problem sets, students will have other activities, individual and collaborative, which will be graded as part of homework or as participation, depending on the activity.
- % of midterm grades corresponding to “homework” will depend on the number of complete delivered problem sets and activities.



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# PARTICIPATION

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- We will have an active discussion during class, therefore, student participation is extremely important.
- Students have to participate in all designated activities on and off class.
- Other ways to participate: pay attention to lectures, participate on course's discussion boards.



## FINAL PROJECT

---

- Proposal of Characterization of **materials or nanomaterials, can be related to your research project.**
- Must be **novel and original**
- Due on the last day of class
  - Points will be deducted for projects delivered after the due date
- Oral presentation at the end of the semester
  
- **Two progress draft** will be due after the first and second mid term period



# CLASS ATTENDANCE

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- Tec official attendance rules will be observed:
  - **This year, the figure of “EF” (exceso de faltas) disappears; which means there is not an absence limit in order to be granted a final grade. HOWEVER:**
  - Attendance *still* will be registered at the beginning of the class session. If a student arrives after roll-call is her or his obligation to inform the instructor at the end of the session, and to make sure the absence is removed.
  - Tardiness will be counted in 15 minutes sets. If a student is late more than one hour, a complete absence will be registered.
  - Absences and tardiness will be counted starting today
  - **If you are late to any exam, no extra time will be given.**
  - **If you do not show up in a day where a quiz or other graded activity was done, those will not be replaced and a grade of “0.00” will be granted.**
  - If you fall asleep, are talking to others, not paying attention, then instructors could consider to register an absence.

→ *Class attendance will be registered even without conducting a roll call*



## COURSE GUIDELINES AND POLICIES

---

- All exams and activities are individual:
  - Copying or transcribing in an exam will invalidate the answer or even the complete exam without opportunity for a second-chance exam.
  - Nullified exams will be graded with a “DA”
  - Any kind of academic dishonesty will not be tolerated: to offer or to receive aid, plagiarism, cheating, etc.
- Students should not chat during lectures and presentations.
- It's very important to participate in and out of lectures.
- At the end of the semester, there are will be no extra activities of make up projects, nor second-chance exams.



# USE OF CELL PHONES, TABLETS, LAPTOPS, ETC.

---

- Comprehension and discussion during lectures is very important, so the students should avoid any distractions.
- Mobile devices will be used to look for information during lectures and activities.
- Devices should stay in “mute” or “do not disturb” mode during lectures.
- Phone calls should be taken only in case of emergency, please leave the classroom to do so. Preferably let the professor know in advance.
- **Recording (filming) lectures or people can be done only upon previous authorization.**



## Recommendations for success...

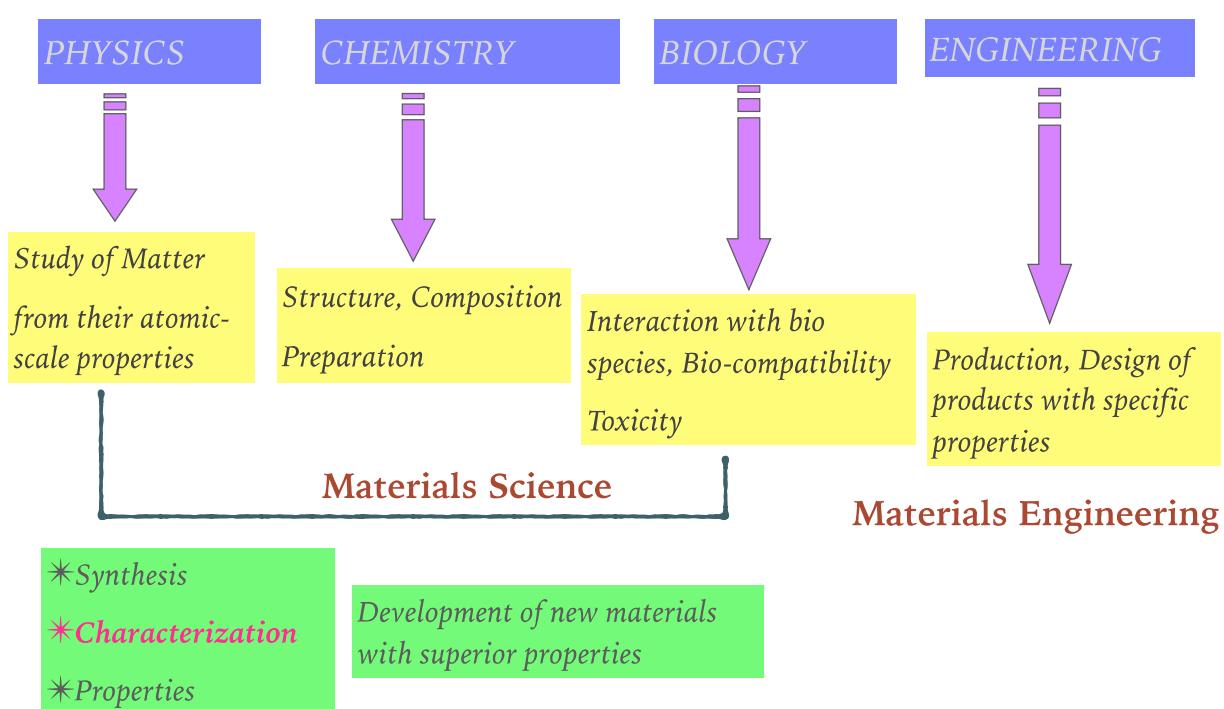
- ***Self-study is critical***
- ***Find a study buddy, or even better, a study group***
- ***Do not skip class***
- ***Do not wait until you fail an exam to seriously start studying or to ask for help***
- ***When doing homework, make sure you understand thoroughly every exercise. Do not just “deliver homework”***
- ***Do not hesitate to ask ANY question***
- ***Be well prepared for the exams***
- ***Be responsible for your own situation, actions and your own success***



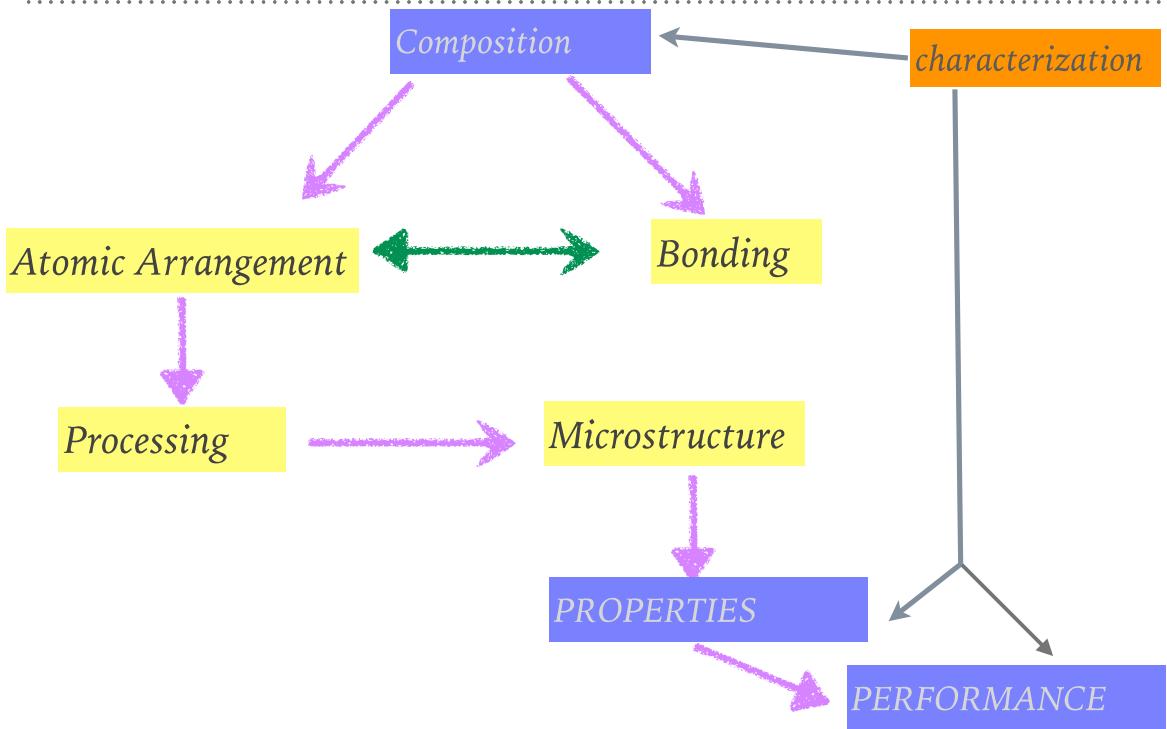
# INTRODUCTION: STUDY OF MATERIALS



## STUDY OF MATERIALS



# PROPERTIES OF MATERIALS



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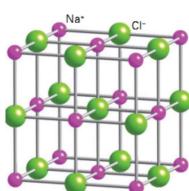
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## CLASSIFICATION BY ATOMIC ORGANIZATION

### Crystalline solids



rock salt crystals

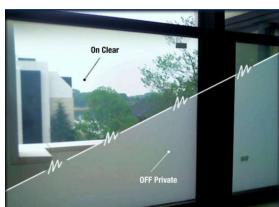
### Semi crystalline / amorphous solids



glass

Polymers

### Liquid crystals



liquid crystals on windows



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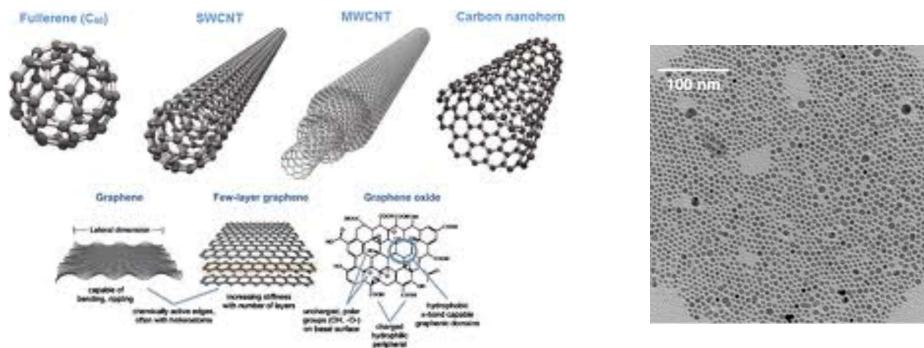
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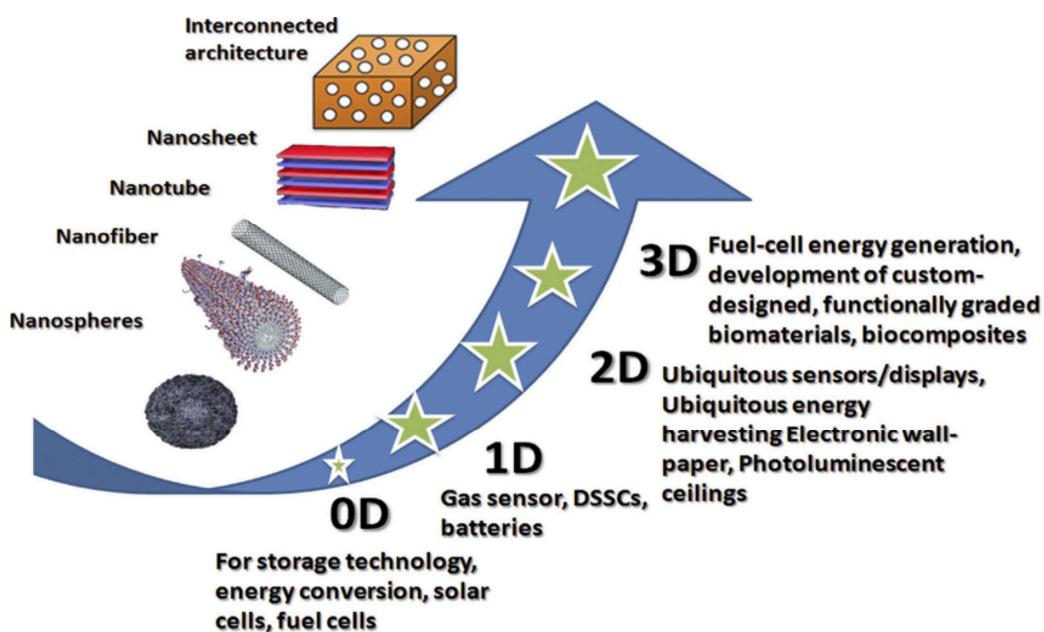
# EXAMPLE: ADVANCED MATERIALS

## Nanomaterials

- Materials produced by simple materials at the atomic level
- Produced by nanotechnology: Ability to arrange atoms in different ways (bottom up)
- Dimensions in the order of nanometers ( $10^{-9}$ ), less than 100 nm



## CLASSIFICATION BY DIMENSIONALITY

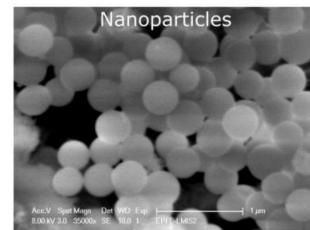
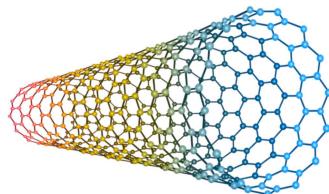
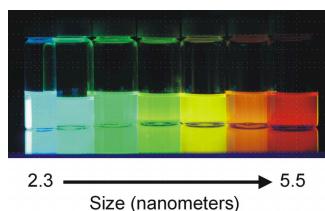


[https://www.researchgate.net/publication/303423807\\_Nitrogen-doped\\_Titanium\\_Dioxide\\_an\\_overview\\_of\\_material\\_design\\_and\\_dimensionality\\_effect\\_over\\_modern\\_applications/figures?lo=1](https://www.researchgate.net/publication/303423807_Nitrogen-doped_Titanium_Dioxide_an_overview_of_material_design_and_dimensionality_effect_over_modern_applications/figures?lo=1)



# NANOMATERIALS & NANOTECHNOLOGY

## NEED UNDERSTANDING OF MATER FROM THE ATOMIC AND MOLECULAR POINT OF VIEW



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## STUDYING OF MATERIALS AND NANOMATERIALS



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# NANOTECHNOLOGY



## WHAT IS NANO?

“Nano” derived from the Greek

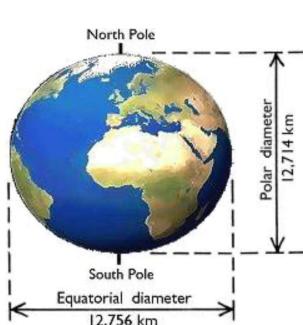
“Nannos” → very short man



# NANO... IT'S A MATTER OF SCALE

**Nano =  $10^{-9}$**

1 nanometer = 0.000 000 001



$10^{-9}$  m



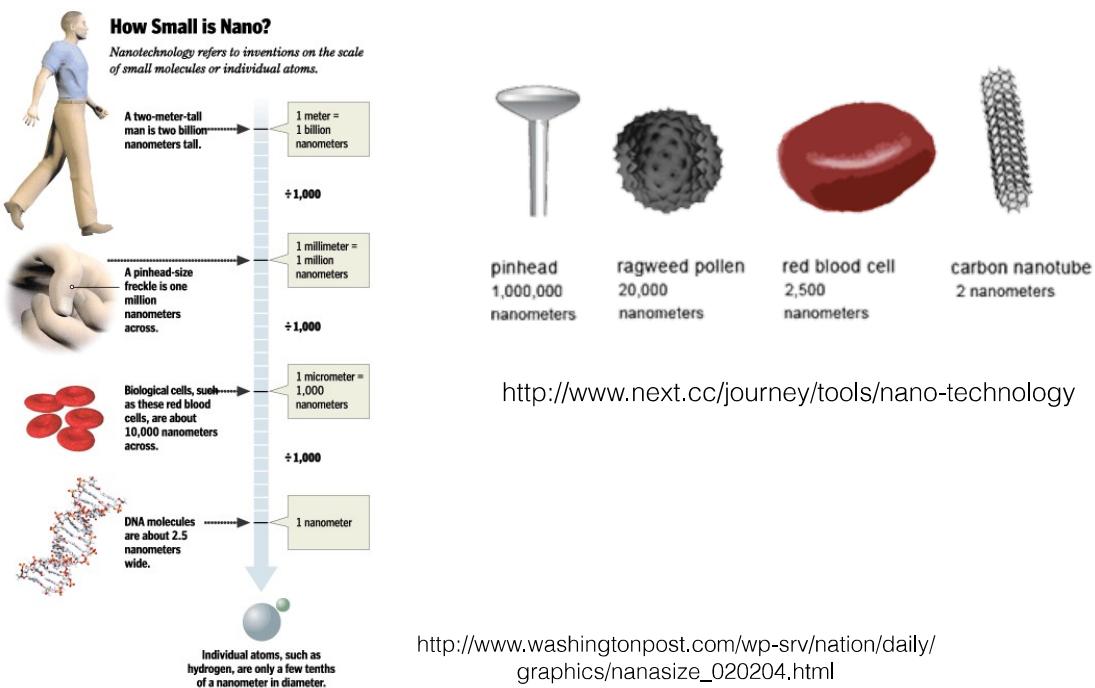
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## HOW SMALL IS NANO?



SOURCE: National Science and Technology Center

BY SETH HAMBLIN AND LOUIS SPIRITO—THE WASHINGTON POST



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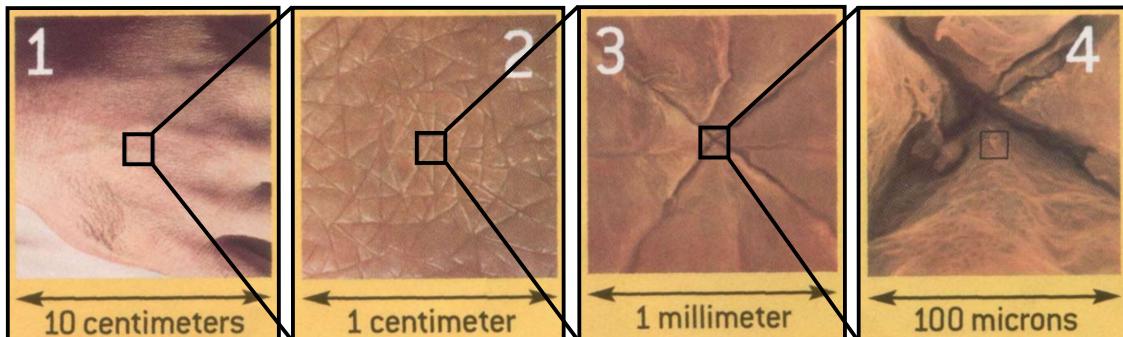
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# HOW SMALL IS NANO?

---

Consider a human hand:



**Skin**

M. Hersam, *Introduction to Nanometer Scale, Science and Technology*, hersam-group.northwestern.edu/



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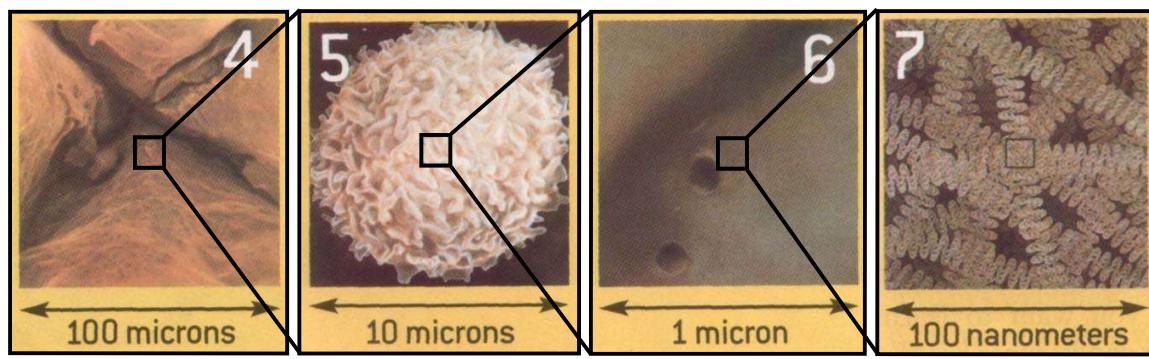
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# HOW SMALL IS NANO?

---

Consider a human hand:



**Skin**

**White blood  
cell**

**DNA**

M. Hersam, *Introduction to Nanometer Scale, Science and Technology*, hersam-group.northwestern.edu/



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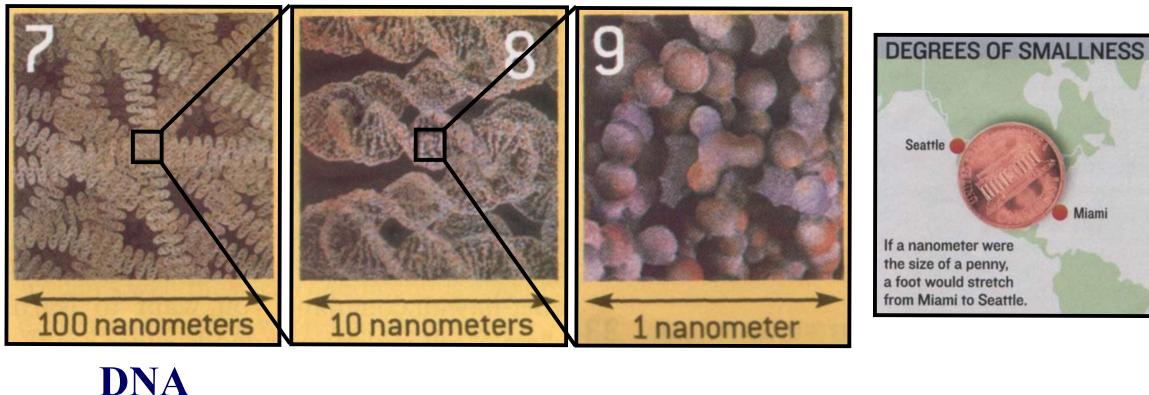
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# HOW SMALL IS NANO?

Consider a human hand:



M. Hersam, *Introduction to Nanometer Scale, Science and Technology*, hersam-group.northwestern.edu/



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## NANOSCIENCE & NANOTECHNOLOGY

**THE SCIENCE AND TECHNOLOGY OF THE SMALL**

"Nanotechnology is the understanding and control of matter at dimensions of roughly 1 to 100 nm, where unique phenomena enable novel applications."

"Nanoscience is the study of materials that exhibit remarkable properties, functionality and phenomena due to the influence of small dimensions"

"Nanotechnologies are characterized by characterization, production and application of structures and systems by controlling shape and size at nanometre scale"

"Nanoscience is the study of very small things and across all other science fields, such as chemistry, biology, physics, materials science, and engineering"



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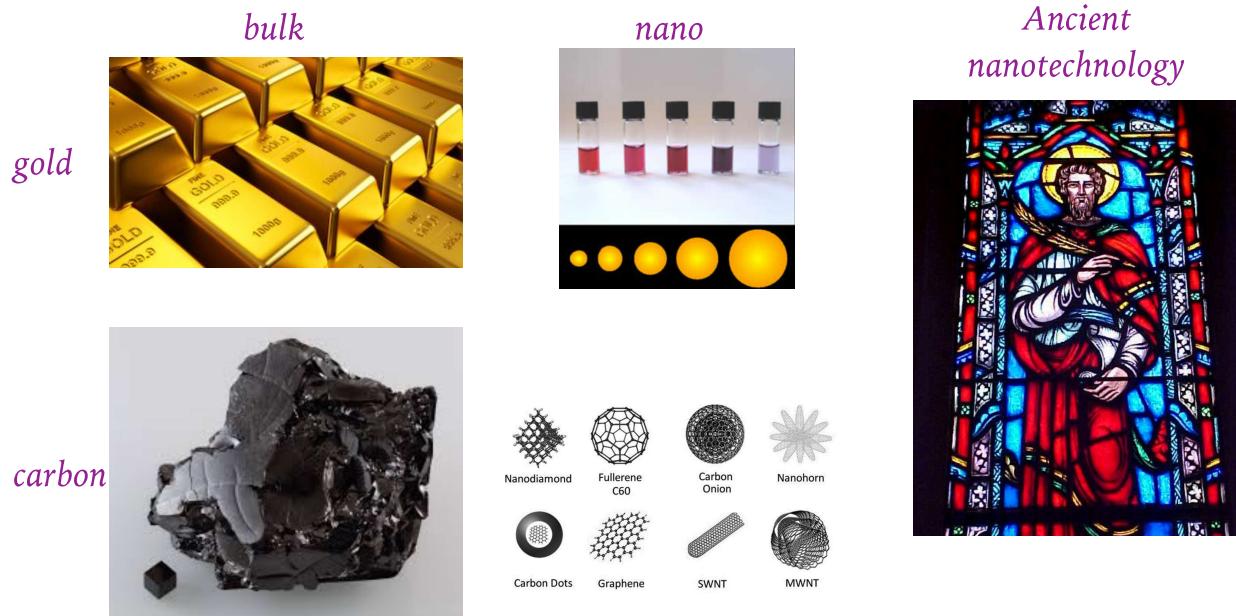
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# NANOTECHNOLOGY

## Materials Properties change at the nanoscale



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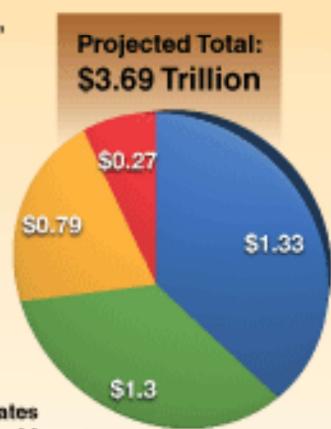
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## THE SCIENCE OF ATOM-SIZE OBJECTS WILL RESHAPE THE ECONOMY

### Large Global Nanotech Market Seen

Europe and Asia are expected to continue generating more revenue from nanotechnology-enabled products than the United States in 2018, experts say. Those regions are projected to account for more than two-thirds of global revenue that year, while the United States is expected to represent about a fifth.

**Projected Global Revenue from Nanotechnology-Enabled Products, by Region, in \$ Trillions, 2018**



Source: Lux Research Inc., December 2015

● Europe  
● Asia  
● United States  
● Rest of world

<http://library.cqpress.com/cqresearcher/document.php?id=cqresrre2016061000>



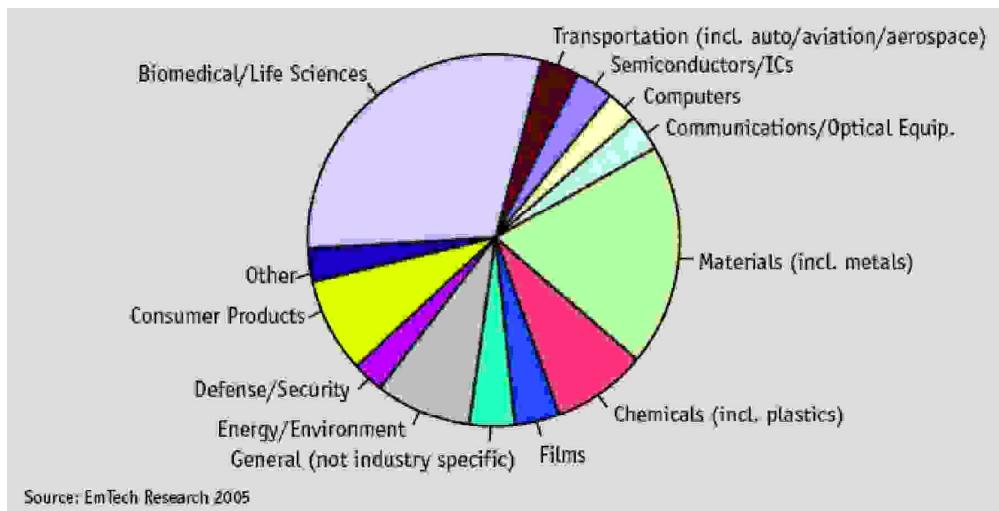
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# APPLICATIONS OF NANOTECHNOLOGY BY INDUSTRY



## A FEW BASIC DEFINITIONS

**Nanoscale:**

in the order of 1 – 100 nm

**Nanostructure**

structure of size between  
molecular and microscopic  
scales

**Nanomaterial:**

material having at least one  
dimension at the nanoscale

**Nanoscience:**

study of phenomena and  
processes at the nanoscale

**Nanotechnology:**

the use of nanoscience for  
practical applications



# NANOSCIENCE AND NANOTECHNOLOGY

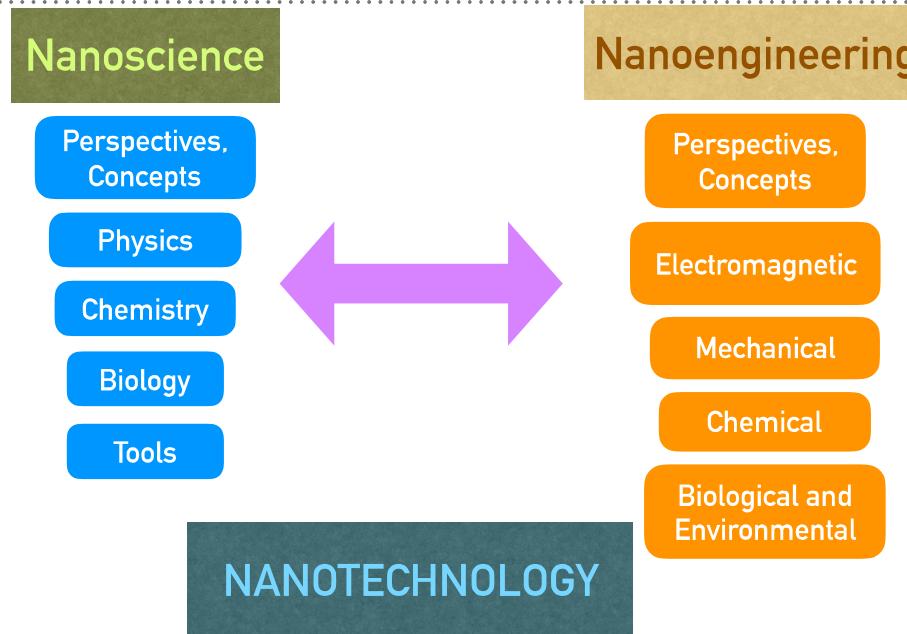
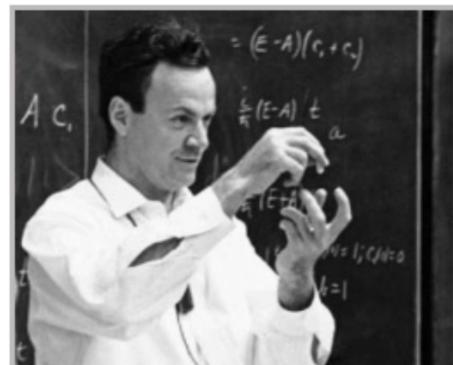


Figure modified from: G. L. Hornyak, H. F. Tibbals, J. Dutta, J. J. Moore; "Introduction to Nanoscience & Nanotechnology" . CRC Press. 2009



**"THERE'S  
PLENTY OF  
ROOM AT THE  
BOTTOM"**

*Cal Tech 1959*

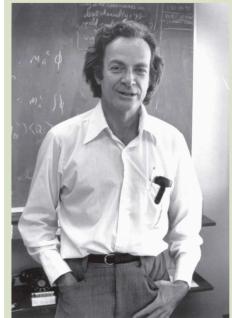


Physicist Richard Feynman, the father of nanotechnology.



*I would like to describe a field, in which little has been done, but in which an enormous amount can be done in principle. This field is not quite the same as the others in that it will not tell us much of fundamental physics (in the sense of, 'What are the strange particles?') but it is more like solid-state physics in the sense that it might tell us much of great interest about the strange phenomena that occur in complex situations. Furthermore, a point that is most important is that it would have an enormous number of technical applications. What I want to talk about is the problem of manipulating and controlling things on a small scale.*

RICHARD FEYNMAN, CALTECH, 1959 [FIG. 1.0],  
*"There's Plenty of Room at the Bottom"*



Nobel Prize laureate Richard Feynman at the blackboard.

G. L. Hornyak, H. F. Tibbals, J. Dutta, J. J. Moore; "Introduction to Nanoscience & Nanotechnology". CRC Press. 2009



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## BRIEF HISTORY OF NANOSCIENCE AND NANOTECHNOLOGY

1959

Richard Feynman  
 "There's Plenty of  
 Room at the Bottom"

1979

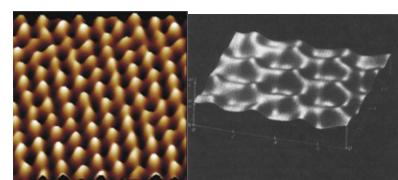
Norio Taniguchi uses the term  
 "Nano-Technology" to describe  
 materials manipulation in the  
 nanoscale (in the context of  
 miniaturization of  
 microprocessors)

1960 -  
 1980

Mesoscopic systems:  
 Materials between the  
 micro and nanoscale

1981

Development of the  
 Scanning Tunneling  
 Microscope (STM)  
 Individual atoms could be  
 seen  
 First tool capable of  
 manipulation at the atomic  
 scale  
 Modern nanotechnology  
 began



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## BRIEF HISTORY OF NANOSCIENCE AND NANOTECHNOLOGY (2)

1983  
-1985

Discovery of  
Semiconducting Quantum  
Dots  
Coloidal CdS particles  
semiconductors

1985

Discovery of  
Fullerenes

H. Kroto, R. Curl, R.  
Smalley (Rice University)  
Carbon nanotechnology  
began

1986

Development of the  
Atomic Force  
Microscope (AFM)

1986

K. Erick Drexler publishes  
“Engines of Creation: The Coming Era of  
Nanotechnology”

Proposes “molecular nanotechnology”  
Concept of “molecular assembler”  
This idea is considered to be not feasible  
by nano science researchers



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## BRIEF HISTORY OF NANOSCIENCE AND NANOTECHNOLOGY (3)

1991

Discovery of Multiwalled  
Carbon Nanotubes by  
Iijima

1993

Discovery of Single-  
Walled Carbon  
Nanotubes

By Iijima and by  
Bethune et al.

1999

USA launches the National  
Nanotechnology Initiative

The recognition of Nanotechnology as a  
distinct field of study launches several  
research projects and initiatives around  
the world.

Nanoscience and Nanotechnology are  
now recognized as interdisciplinary  
fields of study which allow applications  
not formerly possible and enable new  
business opportunities



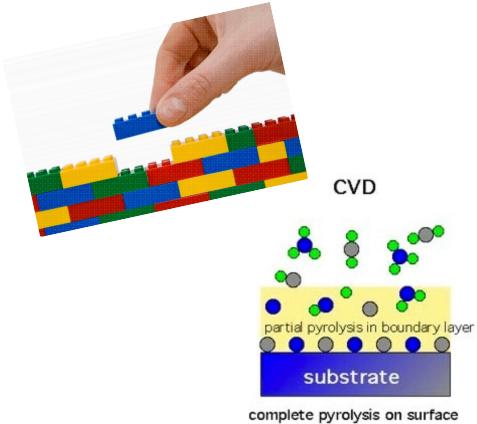
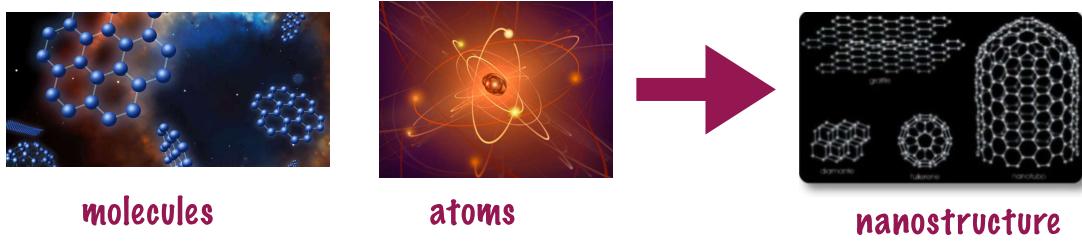
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# PRODUCING NANOSTRUCTURES: BOTTOM-UP



- Nanostructures from molecules and atoms
- CVD
- Wet chemistry
- Self-assembly



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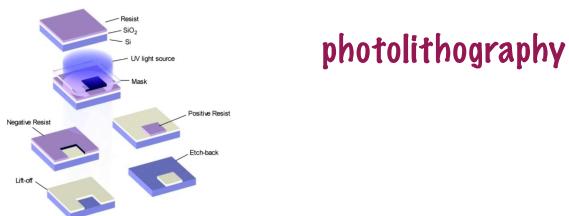
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# PRODUCING NANOSTRUCTURES: TOP DOWN

*“Every block of stone has a statue inside it and it is the task of the sculptor to discover it.” - Michelangelo*



*La fille du Marin* by Philippe Faraut



photolithography

Graphene by  
the Scotch  
tape method



- Nanostructures from removing the excess material. From bulk or microscale to nano.



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## TOP DOWN: MEMS/NEMS



- Micro - & Nano-Electro-Mechanical Systems

- Produced with technology developed for semiconductor microcircuits and computer chips
- Litography masks, physical and chemical etching, etc.

<http://www.memx.com/>



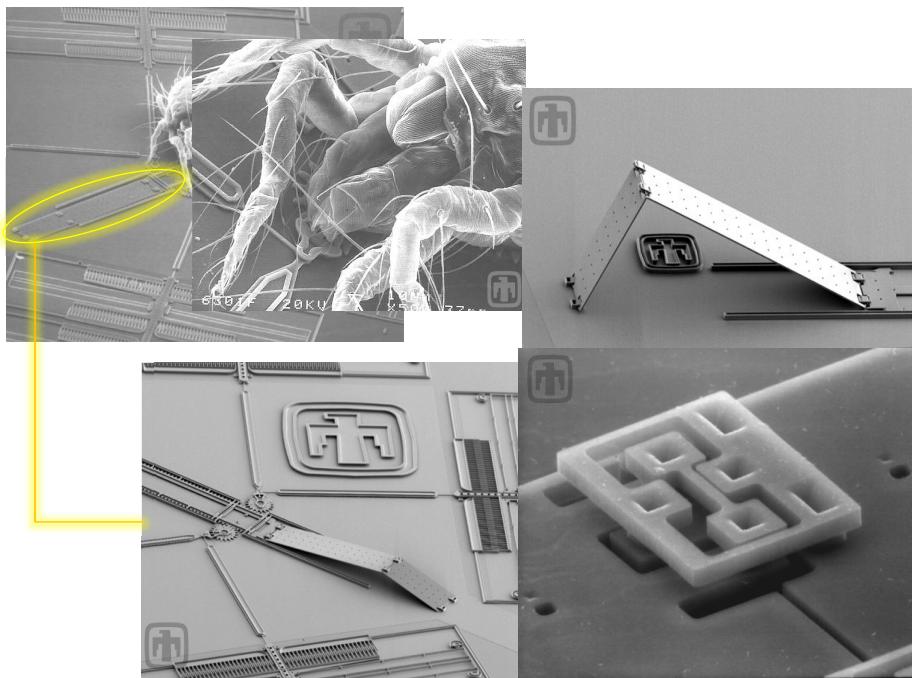
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## MEMS & NEMS



[mems.sandia.gov/gallery/images\\_bugs\\_on\\_mem.html](http://mems.sandia.gov/gallery/images_bugs_on_mem.html)

[mems.sandia.gov/gallery/images\\_mirrors.html](http://mems.sandia.gov/gallery/images_mirrors.html)



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# NANOTECHNOLOGY-BASED FORMULATIONS

- Incorporation of nanostructured materials
  - Most produced by **bottom-up** methods

## Important Aspects

- Uniform **dispersion** & **distribution** in a medium is very important
- Many are used as **intermediate goods**, instead of final consumer products



<http://www.nanowerk.com/spotlight/spotid=23934.php>



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# HOW OLD ARE THE OLDEST KNOW APPLICATIONS OF NANOTECHNOLOGY?

## NANOMATERIALS BEFORE THE NANOTECHNOLOGY ERA



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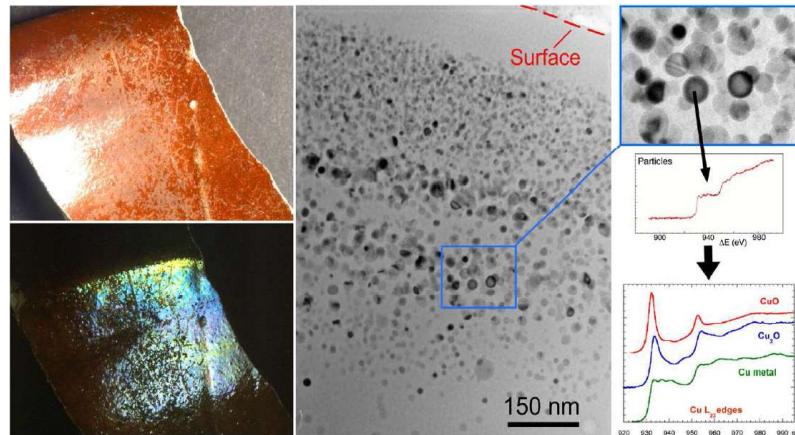
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# NANOPARTICLES ON ANCIENT TIMES

- Bronze age: (1200 -1100 B.C.) glasses found in Italy contain copper nanoparticles that give them a red color

- Celtic red enamels (400–100 B.C.) contain Cu (and cuprous oxide) nanoparticles



Philippe Sciau (2012). "Nanoparticles in Ancient Materials: The Metallic Lustre Decorations of Medieval Ceramics", in *The Delivery of Nanoparticles*, Dr. Abbass A. Hashim (Ed.), ISBN: 978-953-51-0615-9, InTech, <http://www.intechopen.com/books/the-delivery-of-nanoparticles/nanoparticles-in-ancient-materials-the-metallic-lustre-decorations-of-medieval-ceramics>



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# NANOPARTICLES ON ANCIENT TIMES

- Damascus Steel (Middle age) in 2006 carbon nanotubes were discovered in a blade

<http://www.nature.com/news/2006/061113/full/news061113-11.html>



- Ancient Chinese and Japanese ceramics: contained Cu oxides nanoparticles

Metal\_nanoparticles\_in\_contemporary\_potters\_master\_pieces\_Lustre\_and\_red\_pigeon\_blood\_potteries\_as\_models\_to\_understand\_the\_ancient\_pottery\_ancient-pottery.pdf



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# NANOPARTICLES ON ANCIENT TIMES

- Lycurgus Cup, Rome, 4th century: **Au** and **Ag** nanoparticles
  - "The most spectacular glass of the period, fittingly decorated, which we know to have existed"



- Dichroic glass
- Transmission Electron Microscopy analysis in 1980 showed the presence of nanoparticles (50-100 nm) of a Au & Ag (30:70) alloy



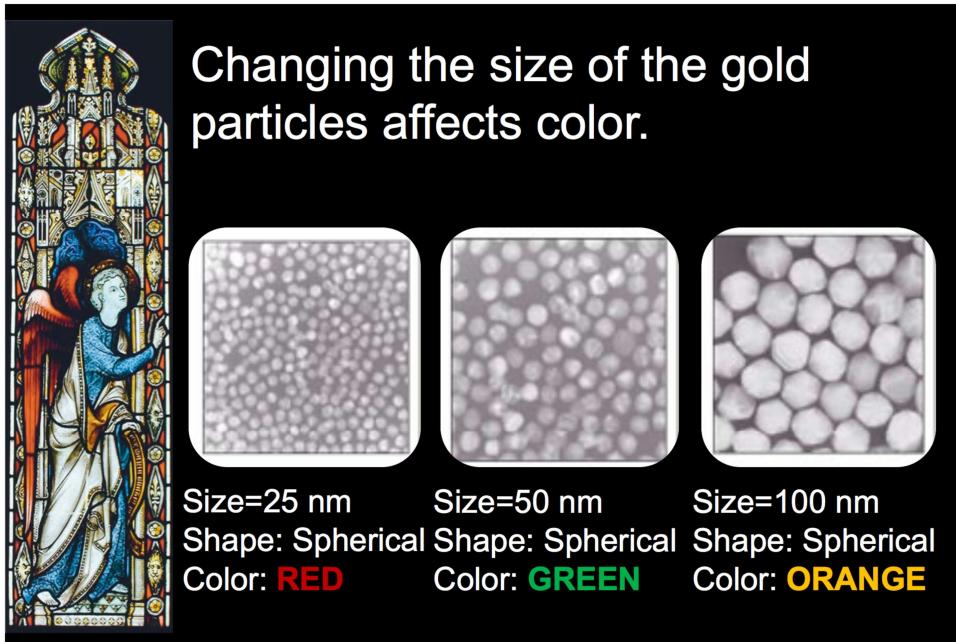
[https://en.wikipedia.org/wiki/Lycurgus\\_Cup](https://en.wikipedia.org/wiki/Lycurgus_Cup)

[www.britishmuseum.org/researchcollection\\_onlinecollection\\_object\\_details.aspx?objectId=61219&partId=1&searchText=lycurgus+cup&page=1](http://www.britishmuseum.org/researchcollection_onlinecollection_object_details.aspx?objectId=61219&partId=1&searchText=lycurgus+cup&page=1)



# NANOPARTICLES ON ANCIENT TIMES

- Stained Glass: metallic nanoparticles

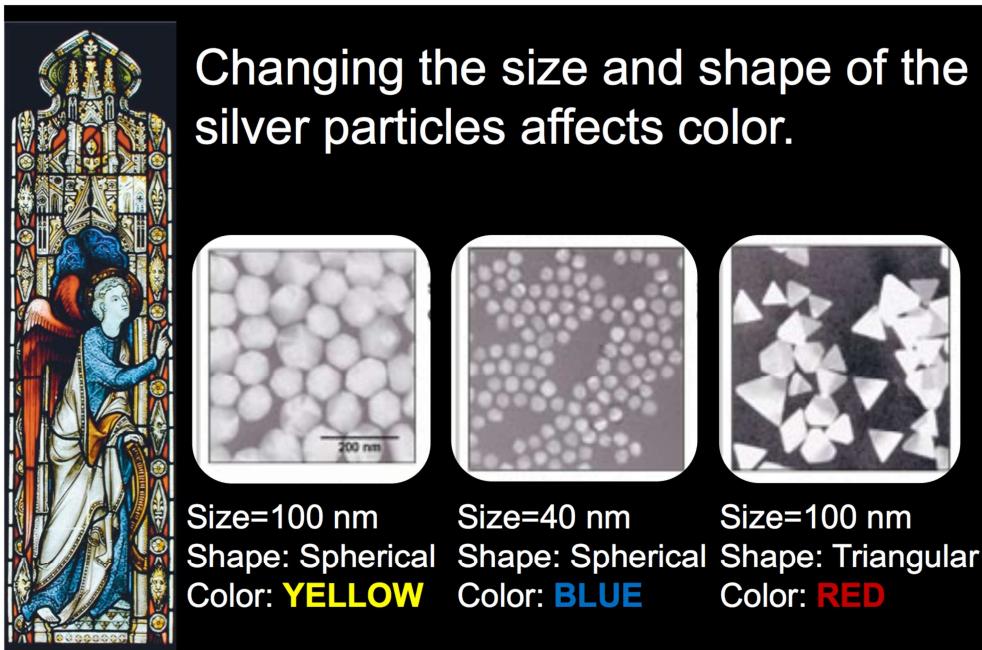


[http://www.nisenet.org/sites/default/files/catalog/uploads/2474/stainedglasscart\\_classpresentation\\_jan10.ppt.pdf](http://www.nisenet.org/sites/default/files/catalog/uploads/2474/stainedglasscart_classpresentation_jan10.ppt.pdf)



# NANOPARTICLES ON ANCIENT TIMES

- Stained Glass: metallic nanoparticles



[http://www.nisenet.org/sites/default/files/catalog/uploads/2474/stainedglasscart\\_classpresentation\\_jan10.ppt.pdf](http://www.nisenet.org/sites/default/files/catalog/uploads/2474/stainedglasscart_classpresentation_jan10.ppt.pdf)



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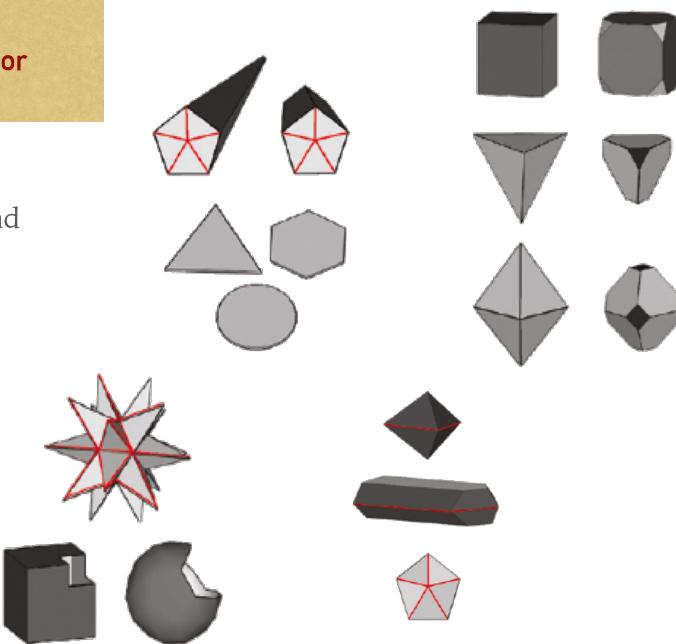
## NANOPARTICLES TODAY

Structures of any material **about 100 nm or less**

### Composition:

metallic, ceramic, polymeric, biological or hybrid

- Many morphologies are possible
- Named according to **composition** and **geometry**:
  - E.g.: Titanium nanorods, gold nanoclusters, zinc oxide nano whiskers, carbon nanotubes, etc.



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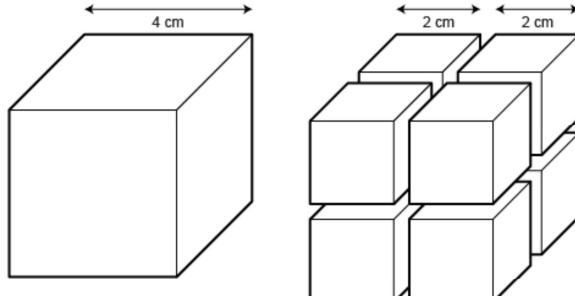
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# NANOPARTICLES TODAY

- Intentional and controlled synthesis is what defines these particles as products from nanoscience and nanotechnology

## High surface to volume ratio



Surface area  
=  $(4 \text{ cm} \times 4 \text{ cm} \times 6 \text{ faces}) = 96 \text{ cm}^2$

Surface area of one cube  
=  $(2 \text{ cm} \times 2 \text{ cm}) \times 6 \text{ faces} = 24 \text{ cm}^2$

Total surface area  
=  $24 \text{ cm}^2 \times 8 \text{ cubes} = 192 \text{ cm}^2$

- In 2 g of Al nanoparticles of 100 nm diameter there are enough particles to give 300,000 to every human being on Earth

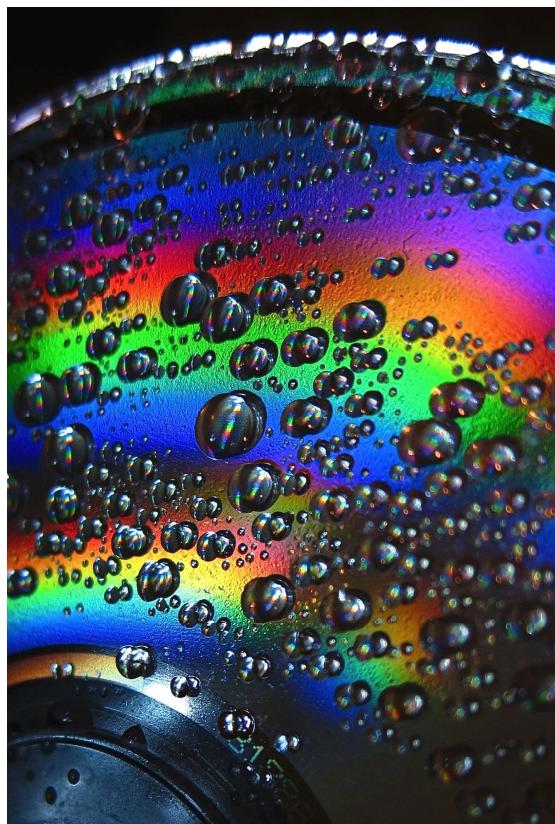
[http://www.bbc.co.uk/schools/gcsebitesize/science/21c/materials\\_choices/nanotechnologyrev1.shtml](http://www.bbc.co.uk/schools/gcsebitesize/science/21c/materials_choices/nanotechnologyrev1.shtml)



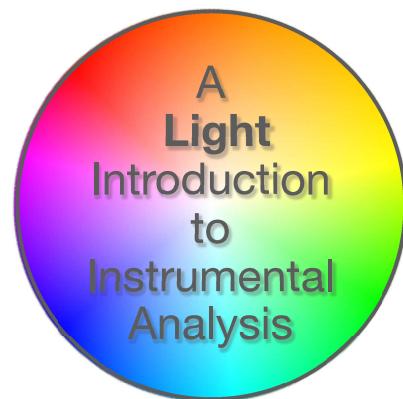
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# THE ELECTROMAGNETIC SPECTRUM



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