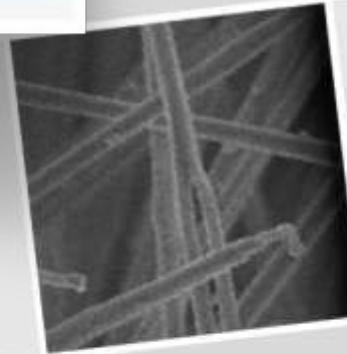
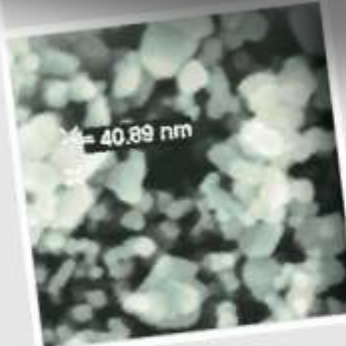


Building College-University  
Partnerships for Nanotechnology  
Workforce Development

# Fundamentals of Metrology and Characterization for Nanotechnology

September 27, 2013

**Recording begins**



Brought to you by:

Brought to You By

The NACK Network, established at the Pennsylvania State College of Engineering, and funded in part by a grant from the National Science Foundation.



Hosted by MATEC NetWorks [www.matecnetworks.org](http://www.matecnetworks.org)

# Welcome to NACK's Webinar

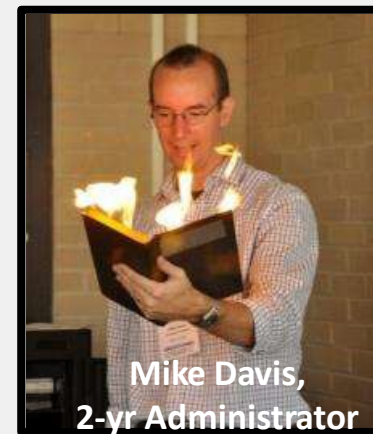


## Today's Presenter:

Dr. Diane Hickey-Davis

Ph.D., Industry

NanoScience Instruments



Moderator: Mike Lesiecki



# In today's webinar:

- How do we see what we can't see?
- Five **common** nanotech instruments
- For each, I'll cover:
  - **What** it does
  - **How** it works
  - Where it's used in **Industry**
  - What **subjects** you can teach with it
  - What **skills** your students can learn from it



# The Scale of Things – Nanometers and More

## Things Natural



Dust mite  
200  $\mu\text{m}$

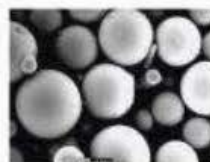


Human hair  
~ 60-120  $\mu\text{m}$  wide

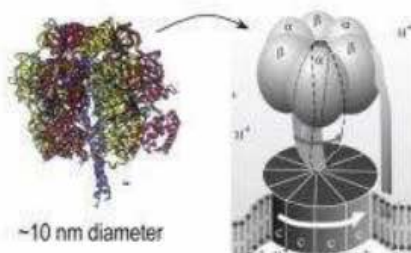
Red blood cells  
(~7-8  $\mu\text{m}$ )



Ant  
~ 5 mm



Fly ash  
~ 10-20  $\mu\text{m}$



~10 nm diameter

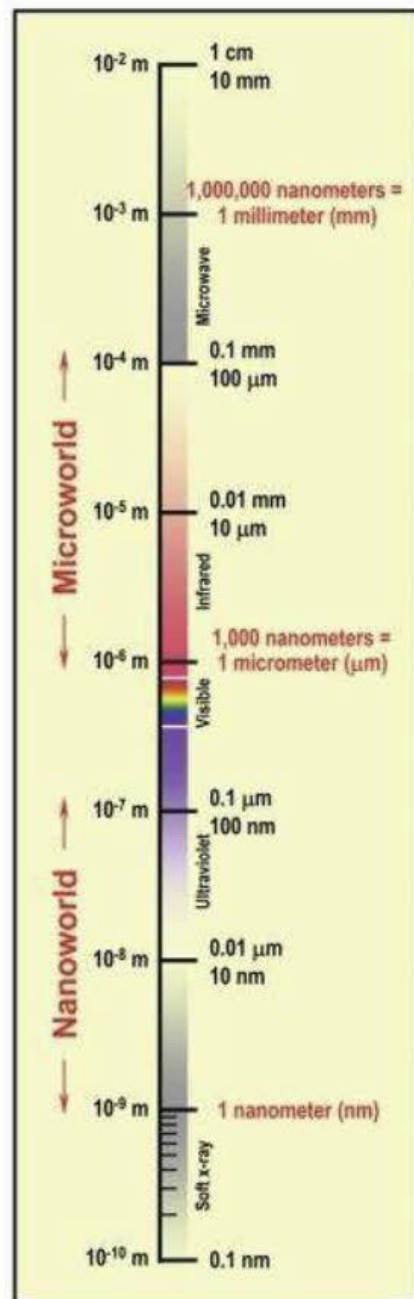
ATP synthase



DNA  
~2-1/2 nm diameter



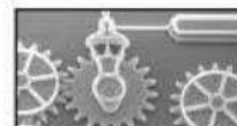
Atoms of silicon  
spacing 0.078 nm



## Things Manmade



Head of a pin  
1-2 mm

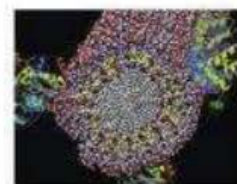


MicroElectroMechanical (MEMS) devices  
10-100  $\mu\text{m}$  wide

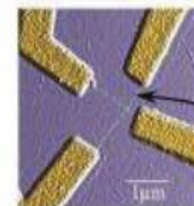
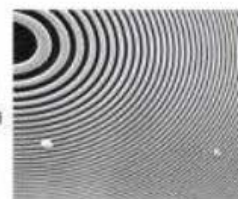


Pollen grain  
Red blood cells

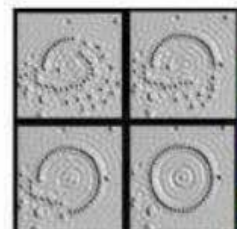
Zone plate x-ray "lens"  
Outer ring spacing ~35 nm



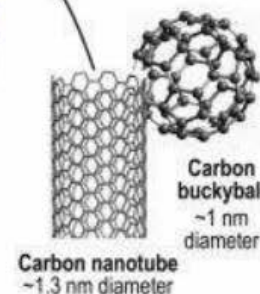
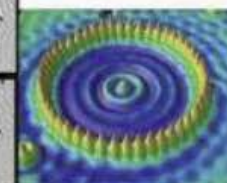
Self-assembled,  
Nature-inspired structure  
Many 10s of nm



Nanotube electrode



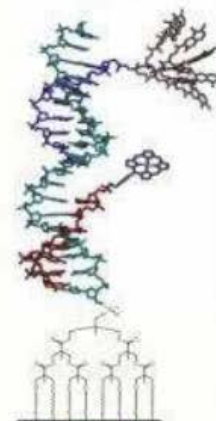
Quantum corral of 48 iron atoms on copper surface  
positioned one at a time with an STM tip  
Corral diameter 14 nm



Carbon nanotube  
~1.3 nm diameter

Carbon buckyball  
~1 nm diameter

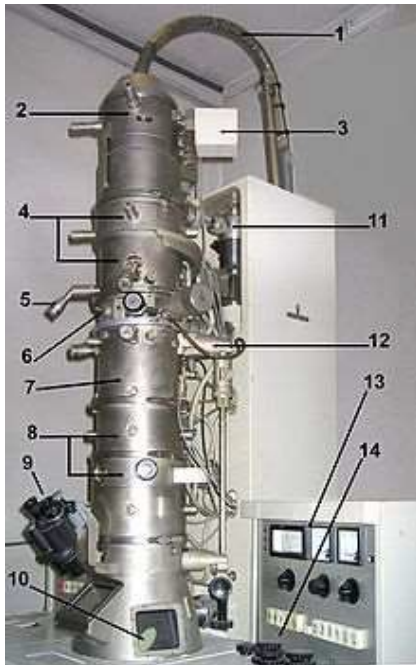
## The Challenge



Fabricate and combine nanoscale building blocks to make useful devices, e.g., a photosynthetic reaction center with integral semiconductor storage.

# Common fears about instruments

- What is it **used** for?
  - [industry]
- Would it be useful for my students to **learn**?
  - [educational benefit]
- What do I do with it once I've got it?
  - [content]
- Will it collect **dust**?
  - [45 min class period; too complicated?; needs expensive replacement parts?]

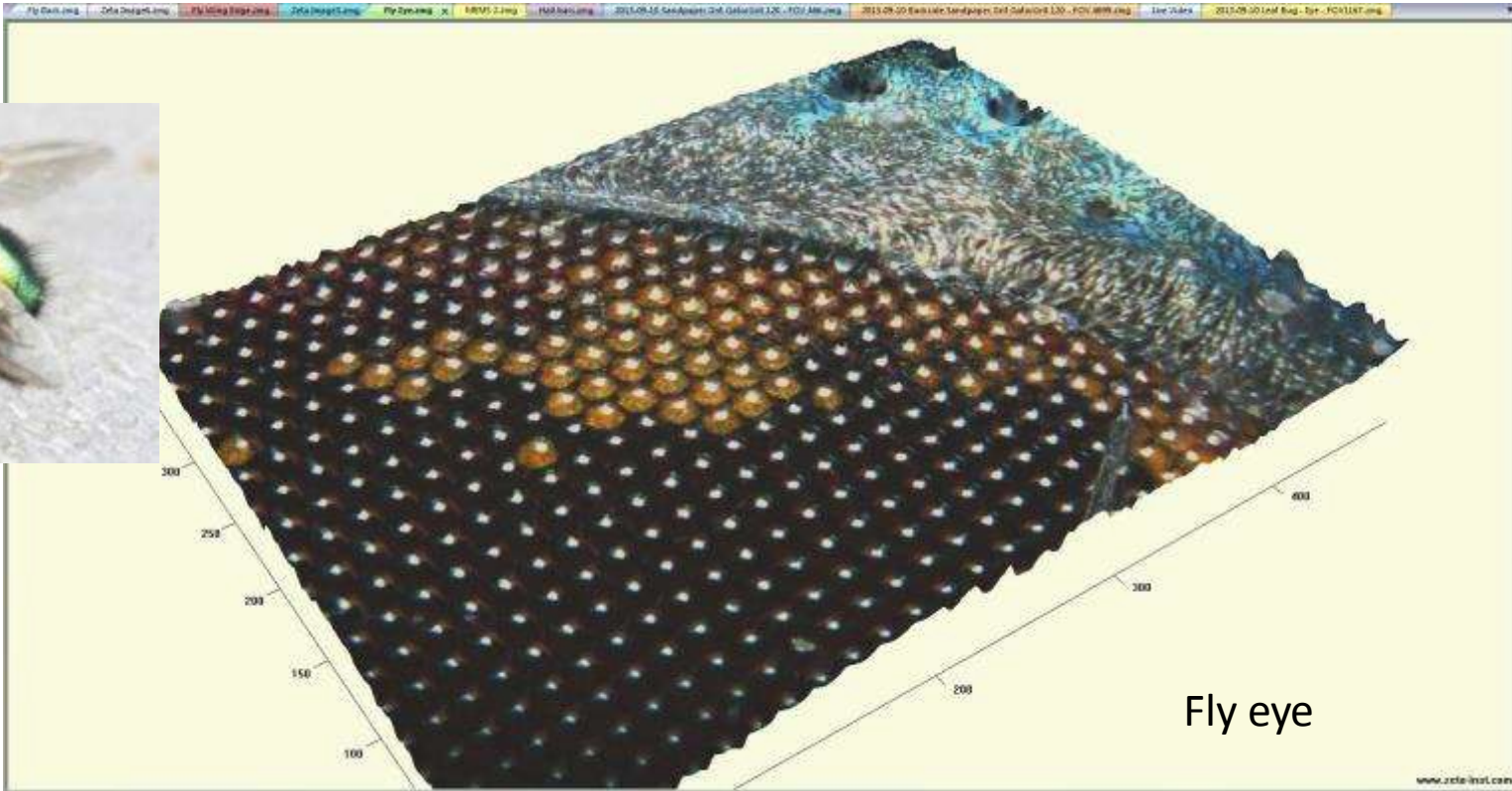


# Today: Alleviate Fears and Provide Info

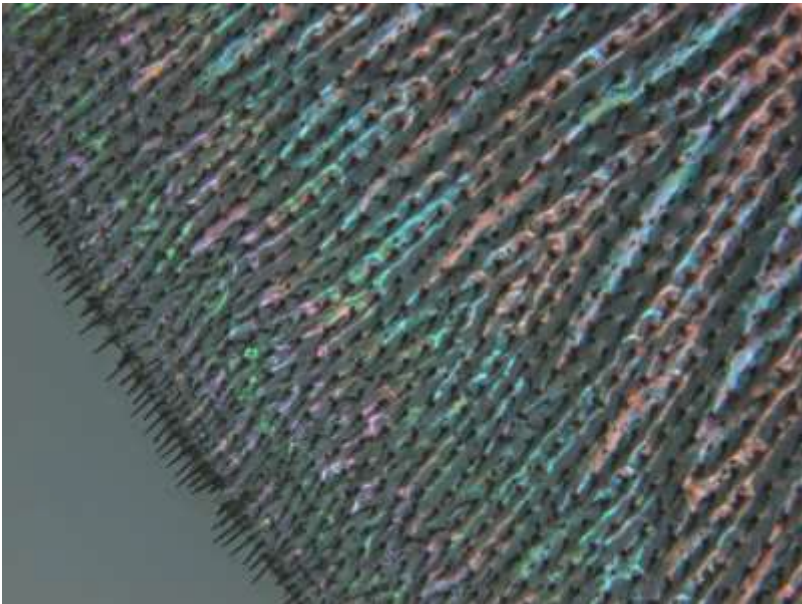
With each instrument, I'll try to communicate:

- What it **does**.
- How it **works**, on a high level.
- **Where** it's used.
- What **subjects** can be taught with it.
- What skills your students can **learn** from it. (*this is subjective*)

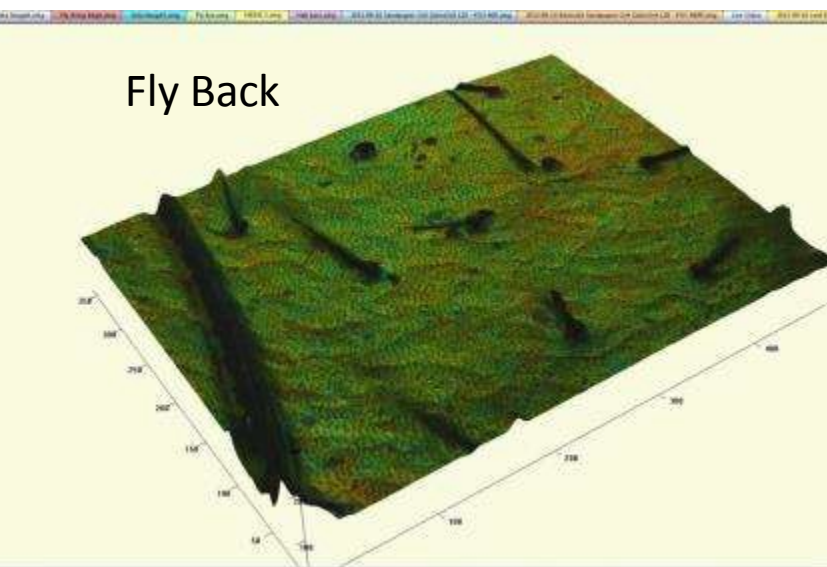




Fly Wing - Edge



Fly Back

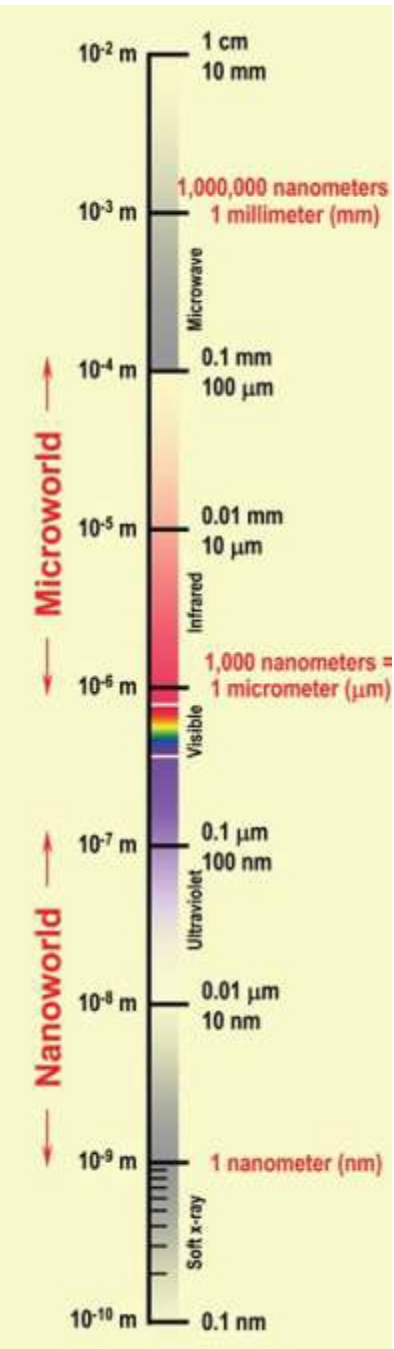




# Optical Microscope

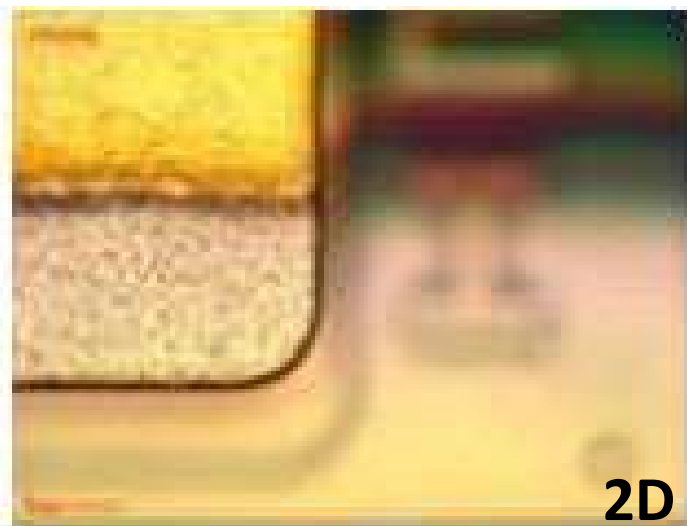
Optical microscopy is ubiquitous in almost any scientific, medical or manufacturing quality environment.

- **What does it do?**
  - It adds a third dimension (3D) to viewing samples, and adds quantitative measurement data.
- **How does it work?**
  - Taking 'slices' of optical images, it reconstructs the focal planes into a 3D image.
- **Where is it used?**
  - Industries: Semiconductor, Manufacturing, Medical devices.
  - Areas: Quality Control, Quality Assurance, Engineering design, Failure Analysis, etc.
- **What subjects can be taught with it?**
  - Integrates easily with Biology, Earth sciences, and the description of the optics for physics.
- **What skills can be taught?**

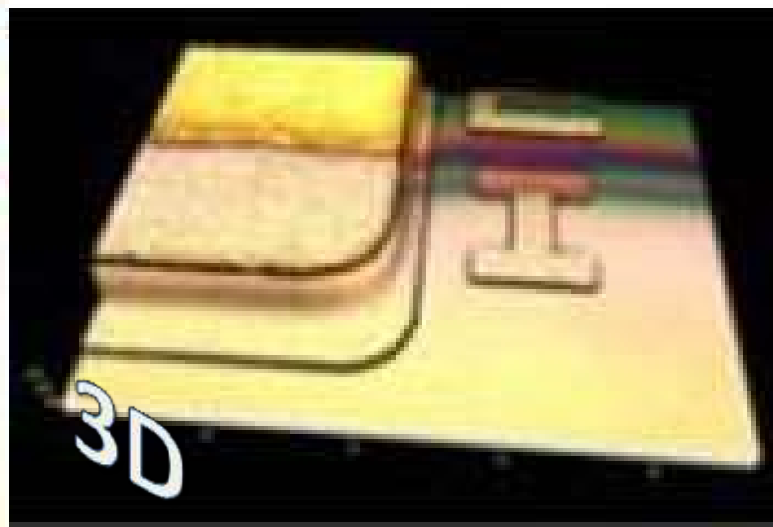
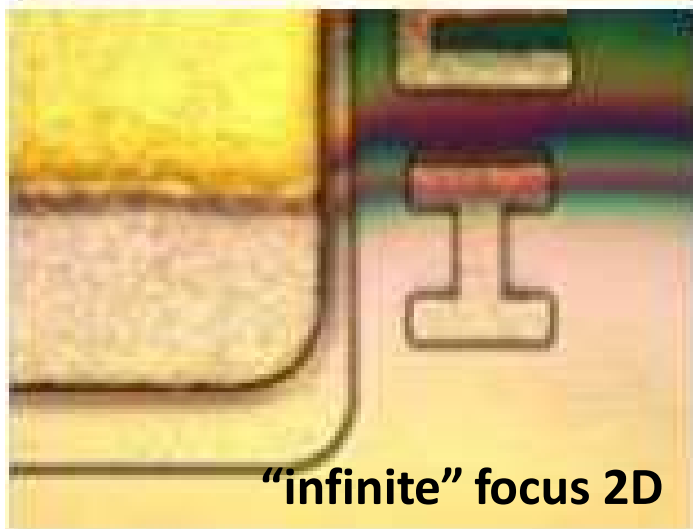


Optical metrology is widely used in:

- **high-tech manufacturing,**
- **quality control, and**
- **failure analysis.**



## A 3<sup>rd</sup> Dimension for Microscopy

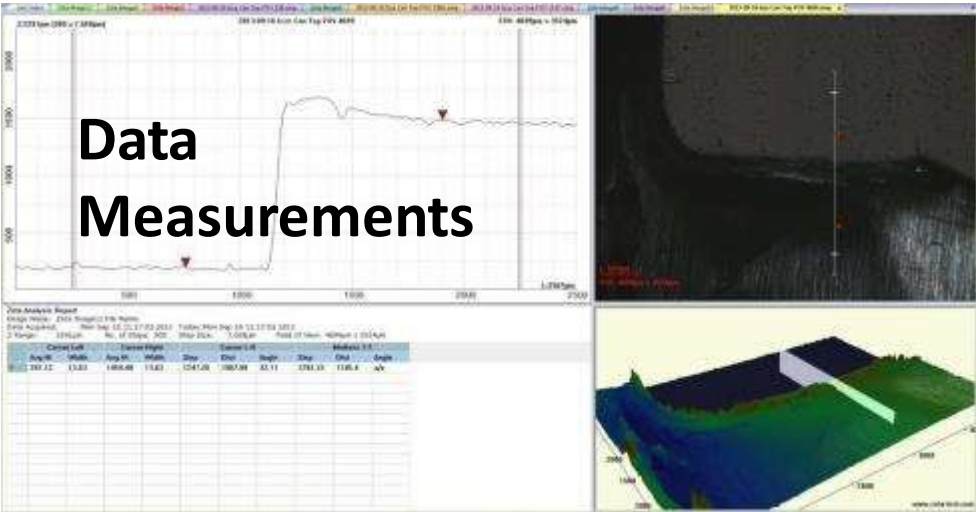
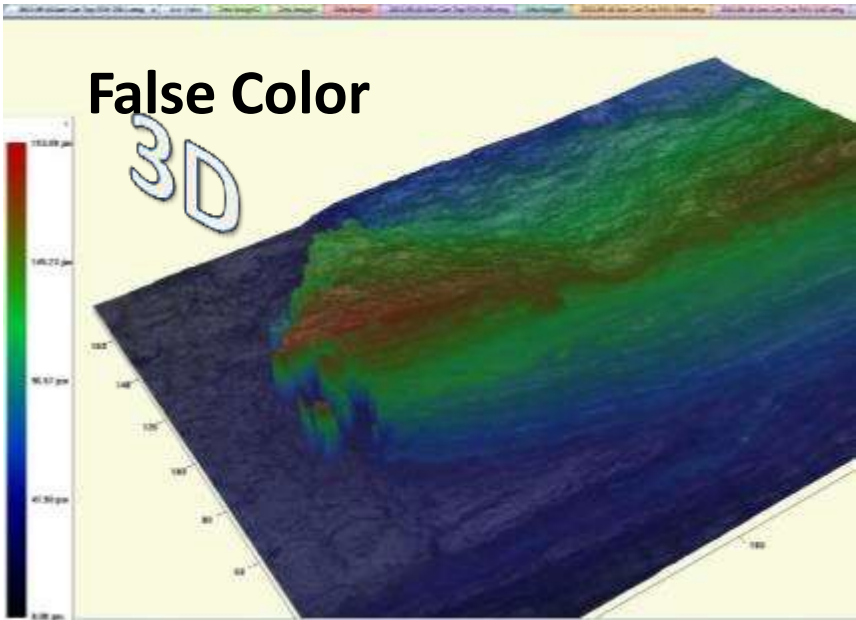


# Failure Analysis – Metal Parts

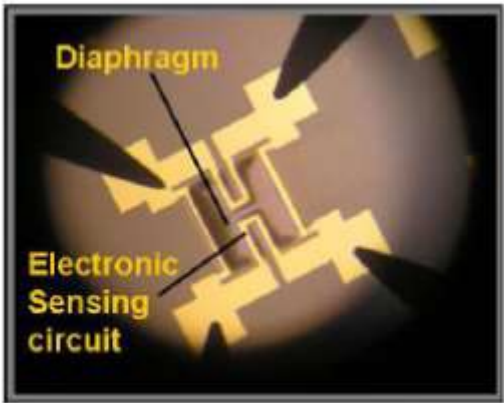




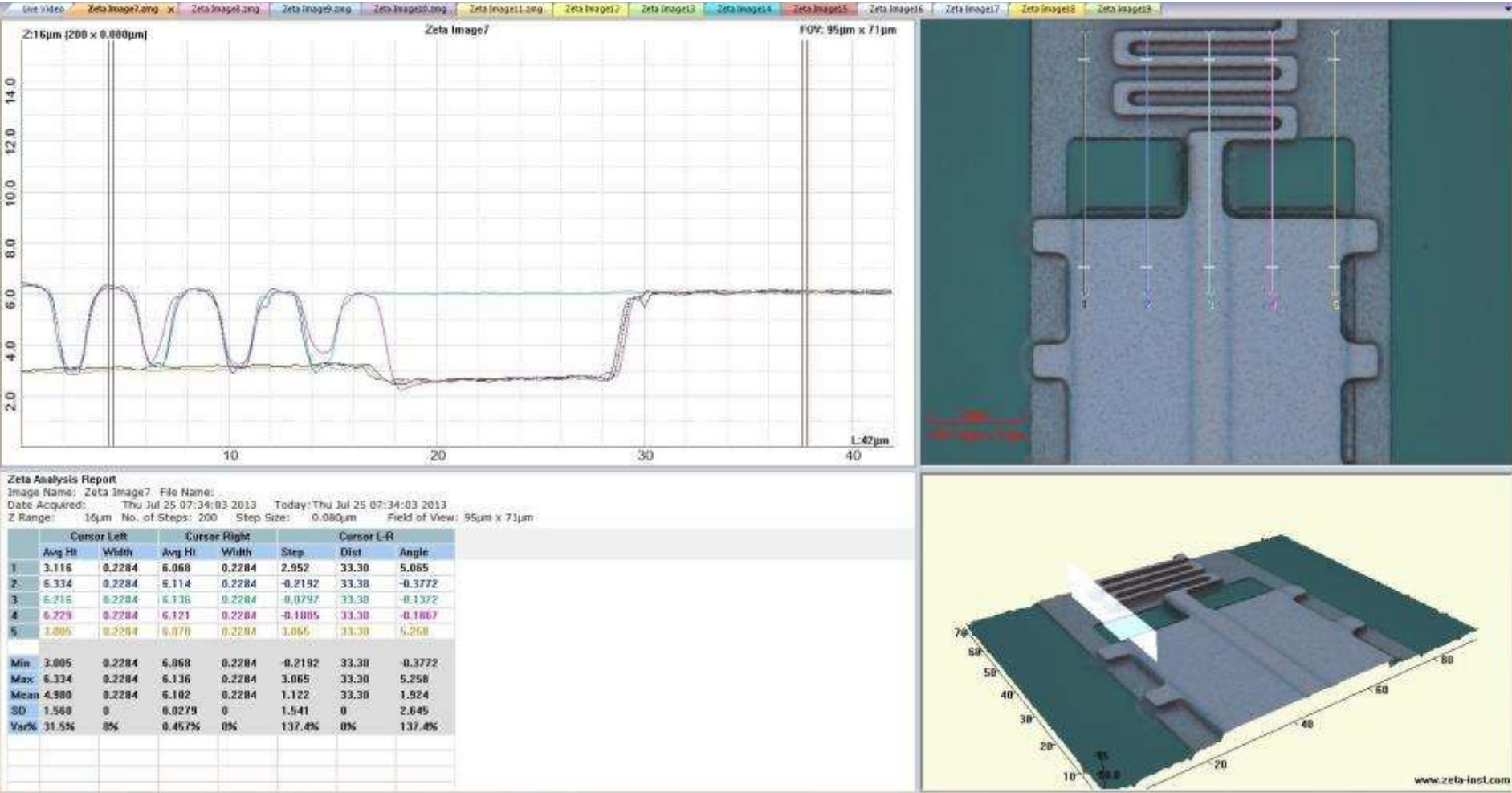
# Failure Analysis – Metal Parts



# Failure Analysis – Semiconductor Parts

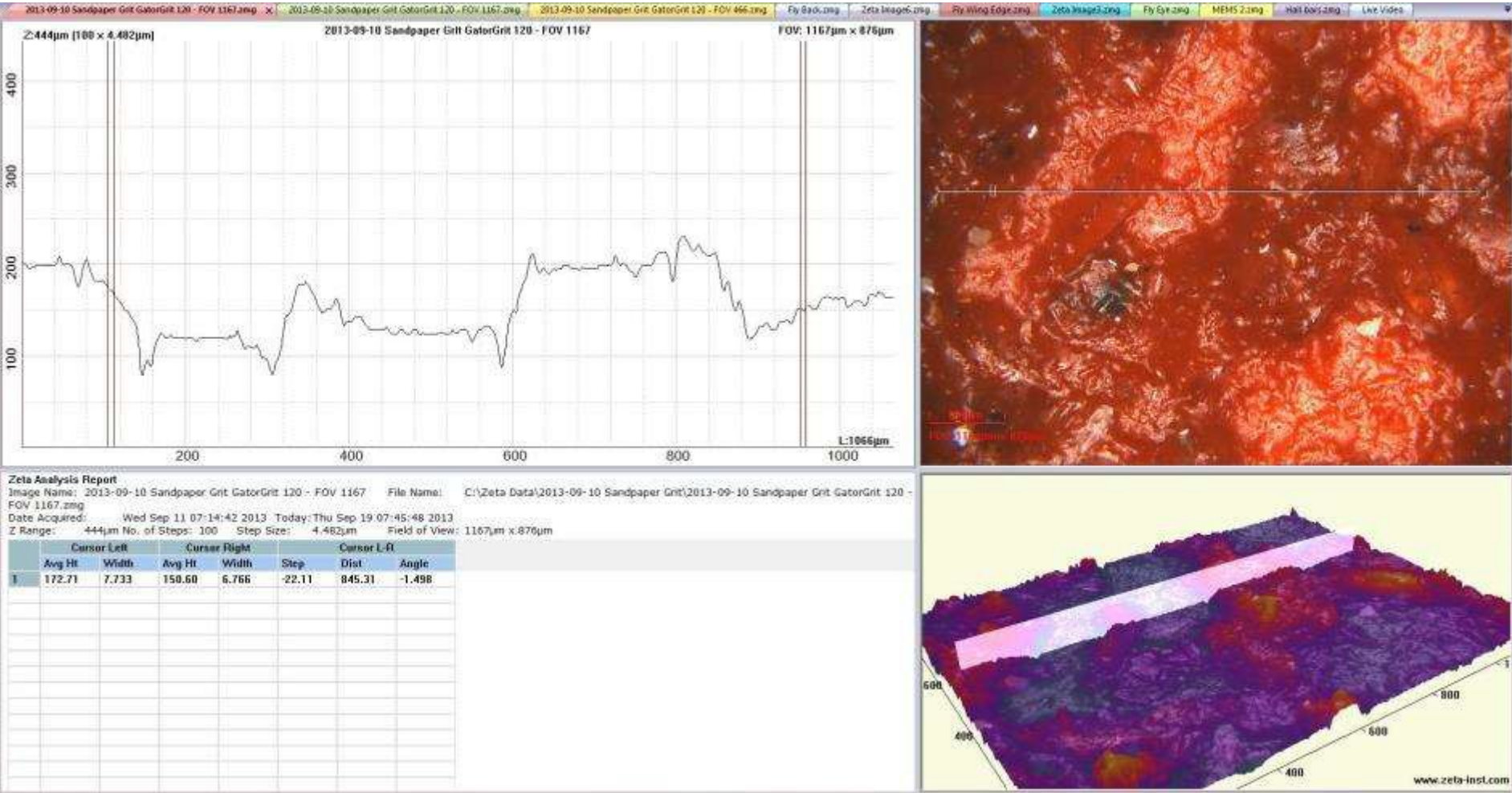


# MEMS Fabrication – Argonne National Lab





# Companies like 3M use 3D Optical Profilers for Quality Control and/or R&D of Sandpaper





# From the Optical Microscope ...



Ant  
~ 5 mm





**Lisa Del Muro** @lisa\_delmuro

28 Aug

got a WOW as soon as class moved the sample from the optical to SEM. one student asked why image in black/white :)

[pic.twitter.com/j1Q1EAXET8](https://pic.twitter.com/j1Q1EAXET8)

Retweeted by shreya

Hide photo

Reply

Retweeted

Favorite

More



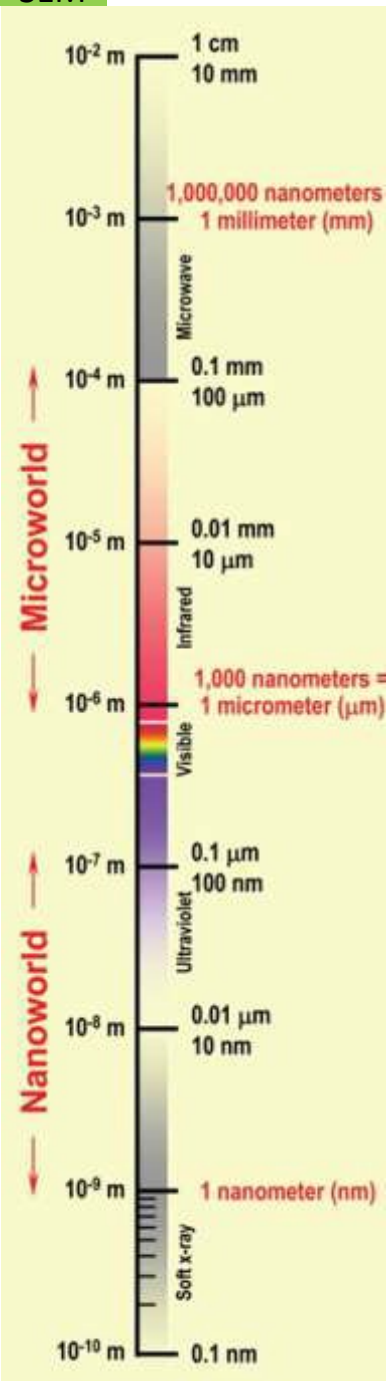
1

RETWEET



12:22 PM · 28 Aug 13 · Details

Flag media



# Scanning Electron Microscope

- What does it do?
  - Let's let Abby, from the TV Show NCIS, explain...
- How it works, on a high level.
- Where it's used.
- What subjects can be taught with it.
- What skills your students can learn from it. (*this is subjective*)



# NCIS' "Abby" – explaining the SEM

<http://www.youtube.com/watch?v=W4HnH6Ar6pw>

# Five Microscopes

to fit the scale of things



**3D Optical**  
3D Optical  
Profiler



**Scanning**  
**Electron (SEM)**



**Atomic Force**  
**(AFM)**



**Transmission**  
**Electron (TEM)**

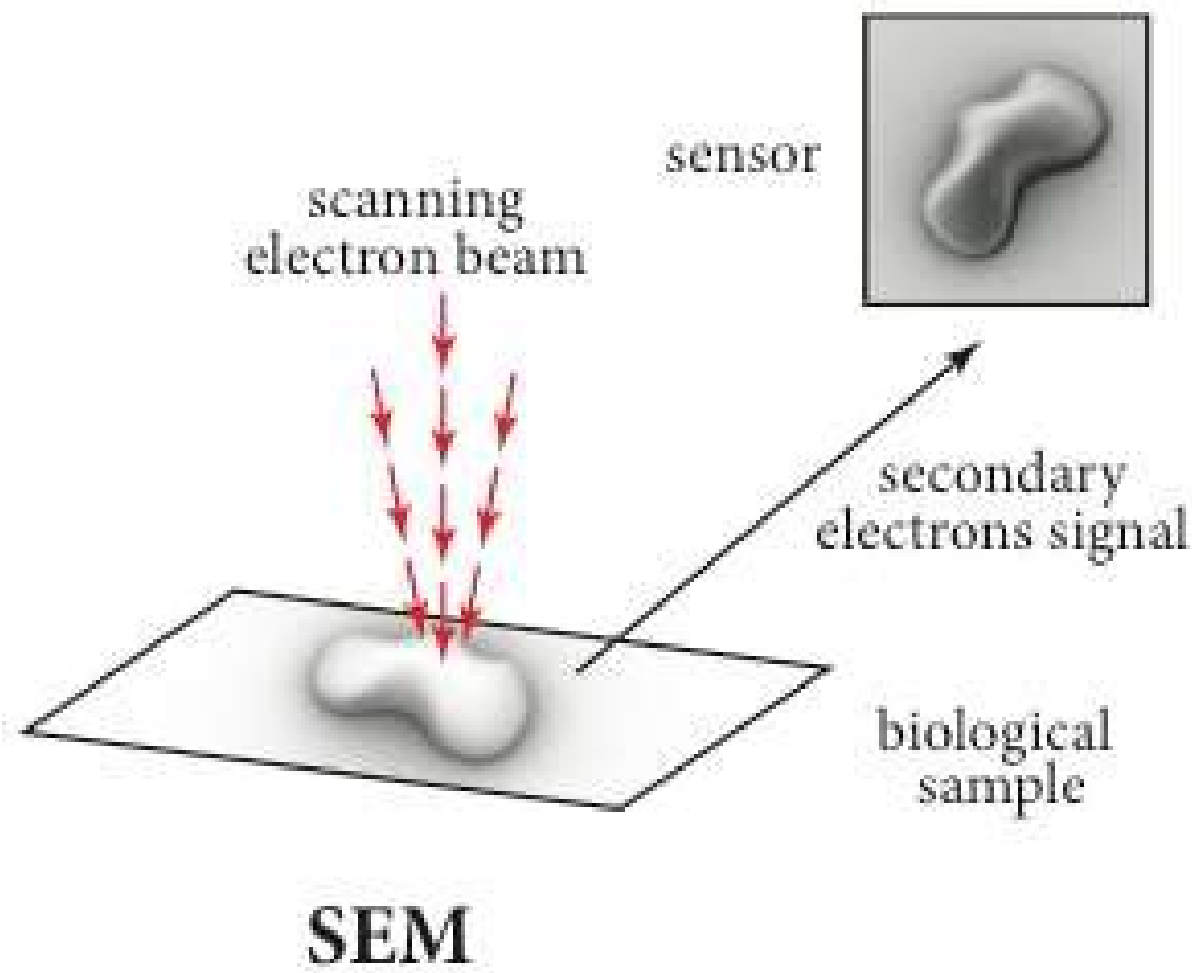


**Scanning**  
**Tunneling (STM)**

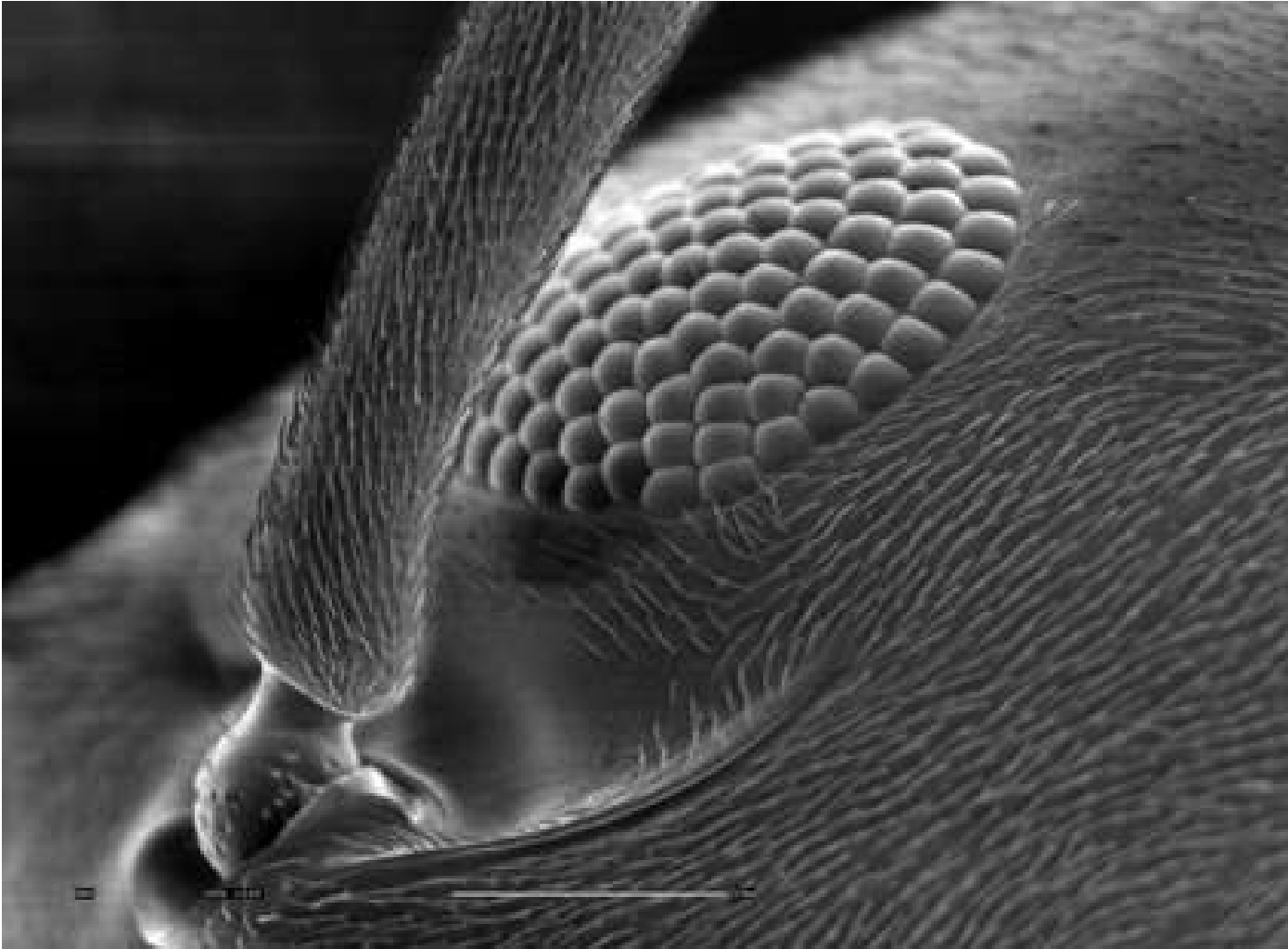
# Scanning Electron Microscope

- What it does.
- How it works, on a high level.
- Where is it used?
- What subjects can be taught with it.
- What skills your students can learn from it. (*this is subjective*)

# How it works



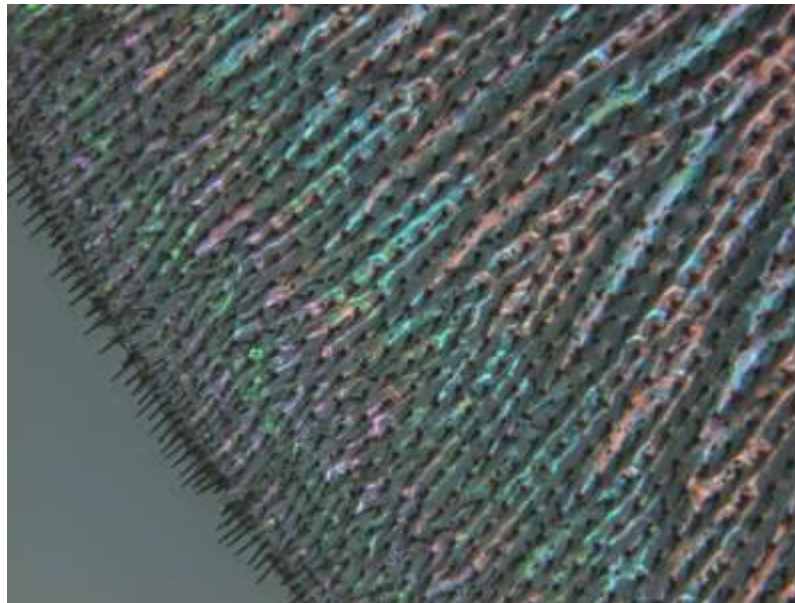
# Why is it black and white?







Color on the  
Optical  
Microscope  
...  
Hmmm...

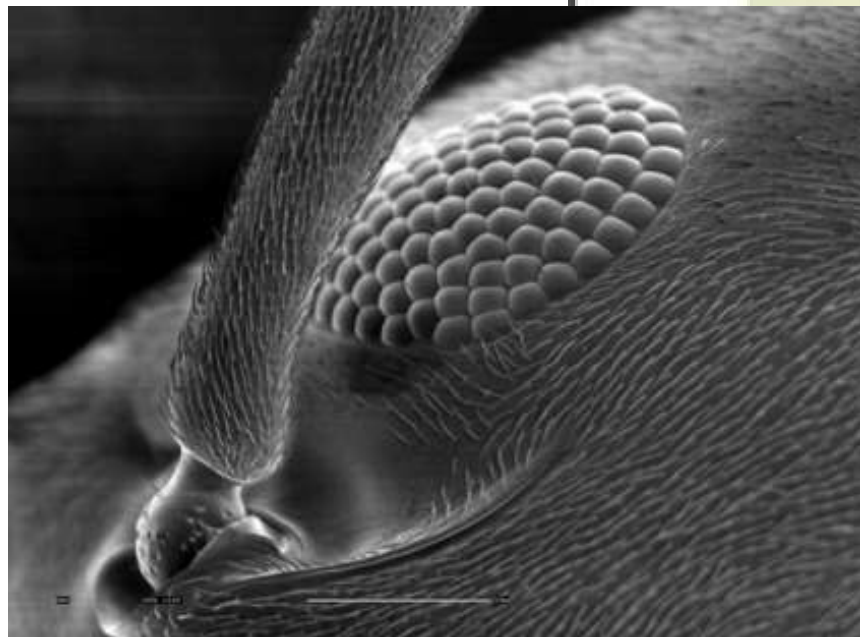


Fly Wing - Edge

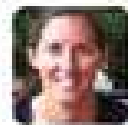


Fly Back

No color on the  
electron microscope?



How does color work  
again?



Lisa Del Muro @lisa\_delmuro

28 Aug

got a WOW as soon as class moved the sample from the optical to SEM. one student asked why image in black/white :)

[pic.twitter.com/j1Q1EAXET8](https://pic.twitter.com/j1Q1EAXET8)

Retweeted by shreya

Hide photo

Reply

13 Retweeted

Favorite

More



1

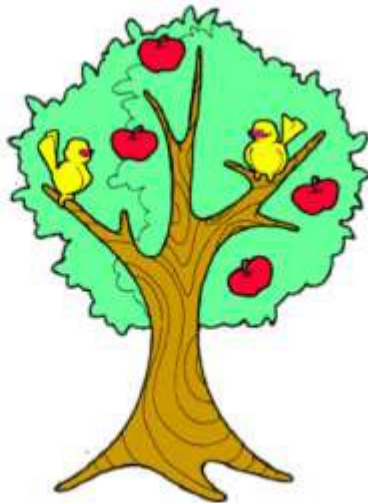
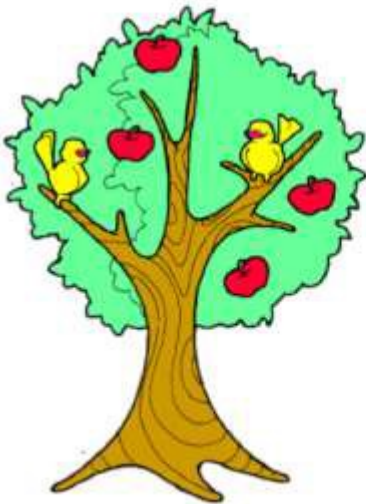
RETWEET



12:22 PM · 28-Aug-13 · Details

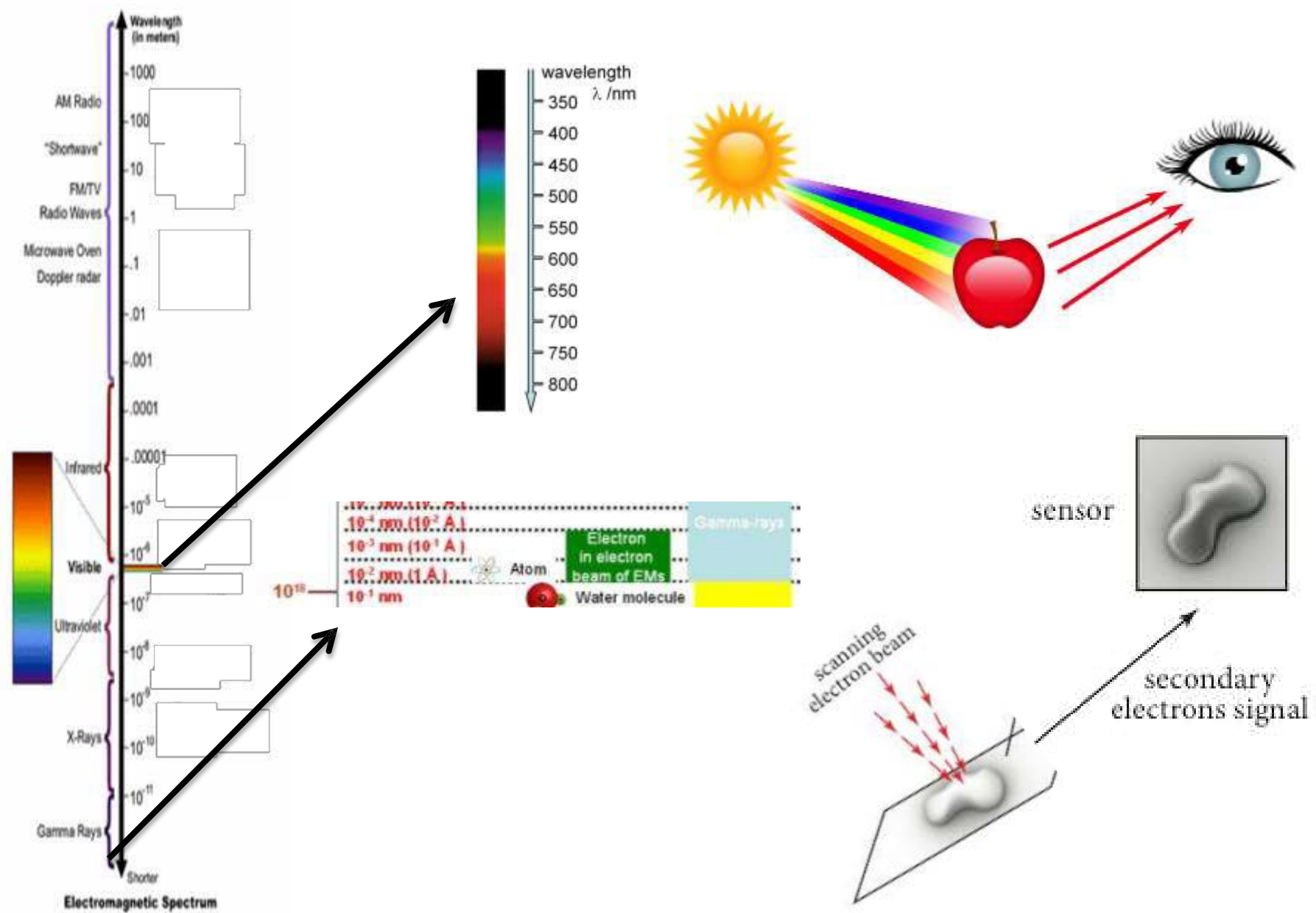
Flag media

# Why Does an Apple Look Red?



Title your paper, and record each of your observations.

SEM – How it works, described visually





*Well,  
technically...*

In EMs, the kinetic energy (eV) gained by an electron as it is accelerated in the electron gun is equal to the electron's drop in potential energy ( $V_0$ ). Therefore, we have,

$eV_0 = m_0 v^2 / 2$  ----- [4787a]

where,

- $v$  -- The electron velocity,
- $m_0$  -- The electron rest mass.

Based on Newtonian theory, the relation between the wavelength ( $\lambda$ ) of a particle (e.g. electron here), moving at a velocity,  $v$ , is given by the de Broglie wave equation:

$\lambda = h / m_0 v$  ----- [4787b.a]  
 $= h / (2 m_0 e V_0)^{1/2}$  ----- [4787b.b]

where,

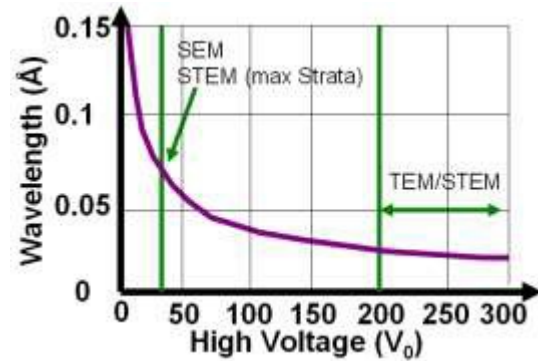
- $h$  -- The [Planck's constant](#).

Because the accelerated electrons have a speed of approximately light speed ( $c$ ), they should be treated using relativistic quantum mechanics, which are important in electron microscopy since the electrons are typically accelerated to potentials of 30 to 400 keV (about half the speed of light). By introducing the correction for relativistic effects, the wavelength is given by,

$$\lambda = h / \left[ 2 m_0 e V_0 \left( 1 + e V_0 / 2 m_0 c^2 \right) \right]^{1/2}$$

The wavelength of the electrons is dramatically decreased with increase of the acceleration voltage; therefore, the spatial resolution of the microscopes is significantly improved. Note that, in electron microscopy it is normally sufficient only to replace the mass and wavelength of the electrons with the corresponding relativistic values [1]. Therefore, the relation between  $\lambda$  and  $V_0$  can be given by,

$$\lambda = \frac{1.23}{\sqrt{V_0 + 10^{-6} V_0^2}} \text{ nm}$$

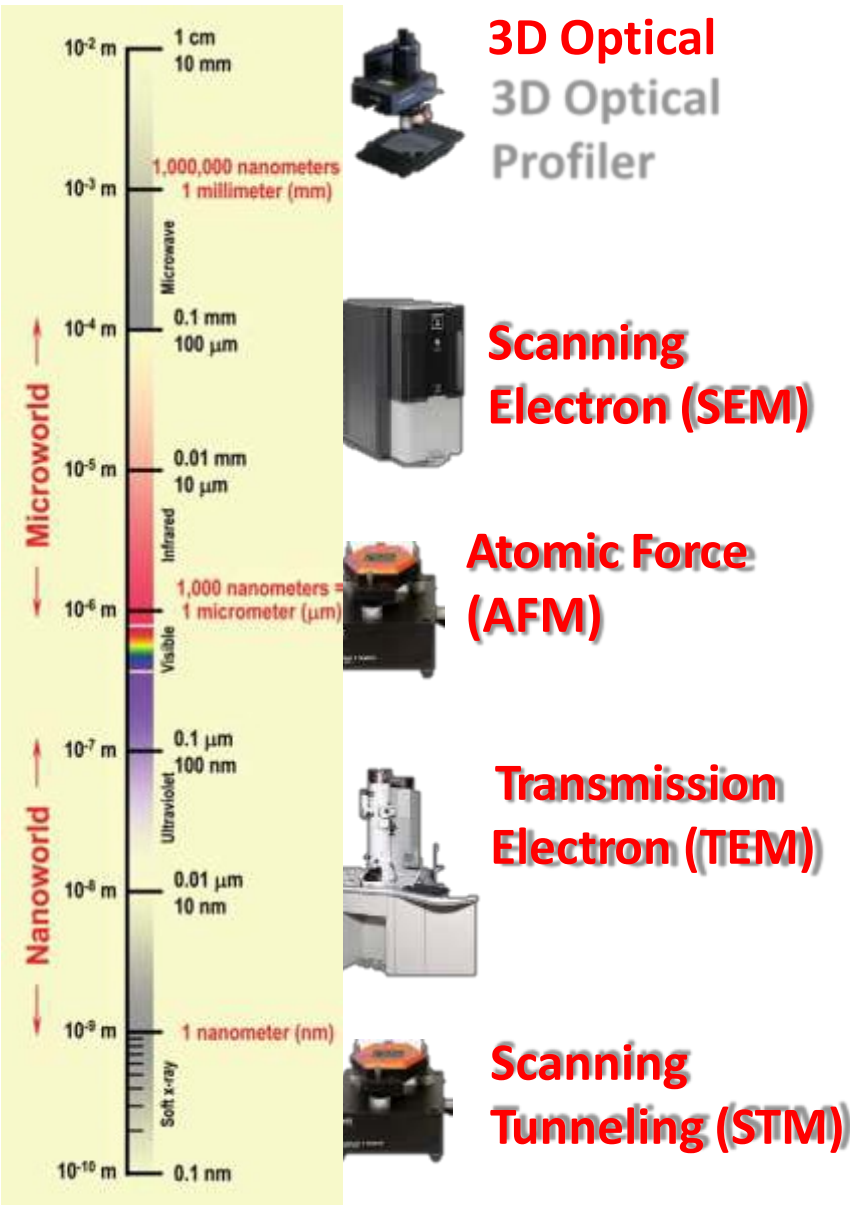


# Five Microscopes

to fit the scale of things

## Scanning Electron Microscope

- What it does.
- How it works, on a high level.
- **Where is it used?**
  - Scanning Electron Microscopy is used in virtually every high technology and scientific area.
  - Additionally, there is a strong focus on including SEM in forensic analysis, as depicted here...
- What subjects can be taught with it.
- What skills your students can learn from it. *(this is subjective)*





# CSI NY

<http://www.youtube.com/watch?v=dWe65XWsqHY>

# SEM in Education

**Then....**



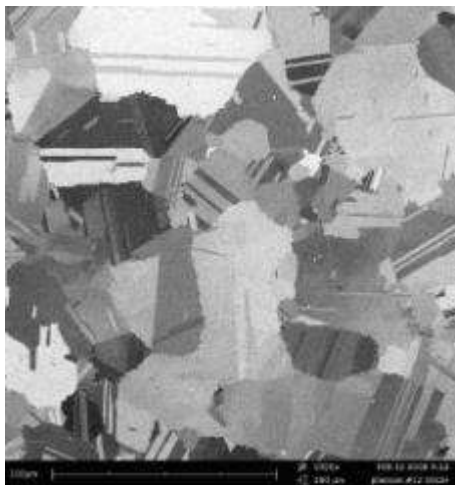
**.... And Now...**

## Phenom SEM and Students

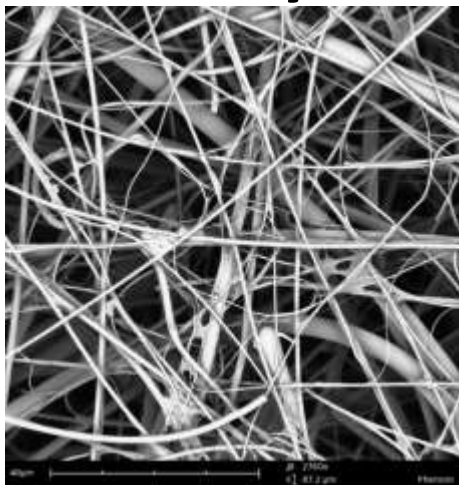


<http://www.youtube.com/watch?v=OyrVL-Dy5RY>

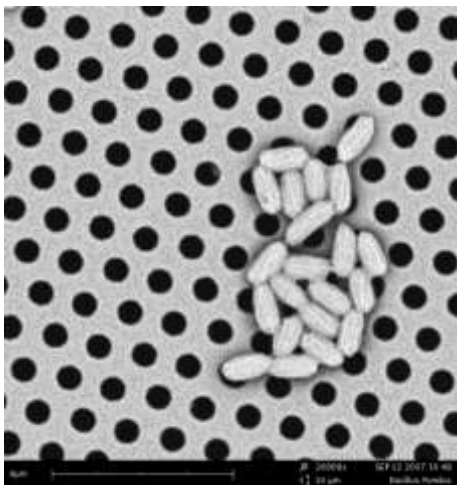
# Where SEM is used in industry:



**Aerospace:** nickel-base supper alloy



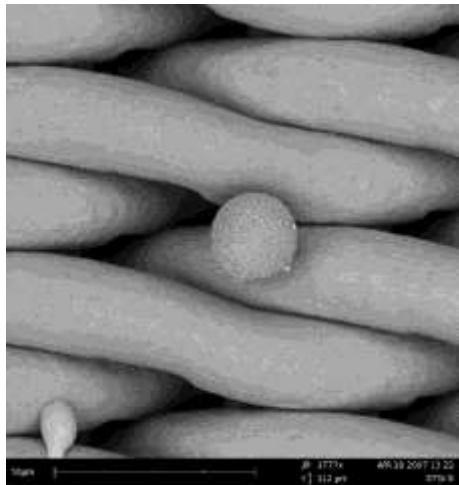
**Consumer Goods:** hepa filter



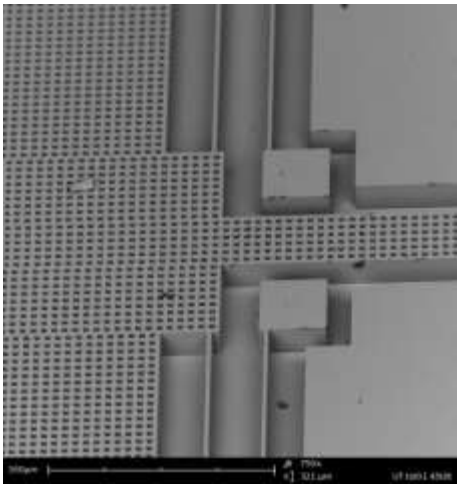
**Medical:** bacteria



**Pharmaceutical:** powder compound



**Industrial:** metal filter



**Semiconductor:** micro structure (MEMS)

Also, colored SEM images can be used as art:

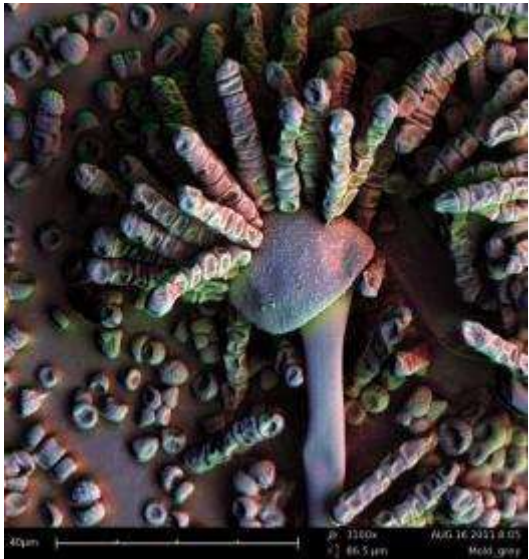
Fly mouth



Penny



Green Mold

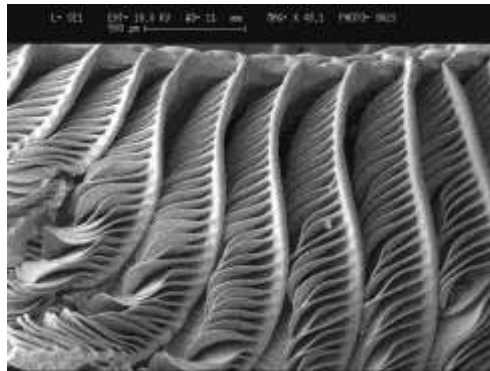
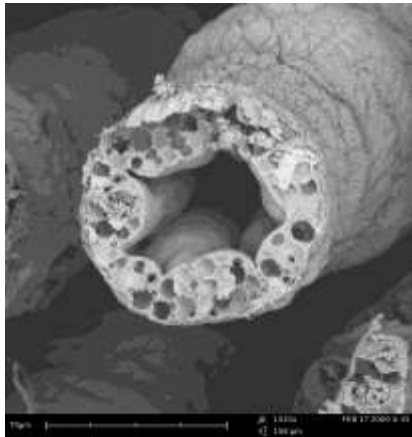




# Used in teaching, for example: Occidental College



*"We love the (SEM)." –Dr. Gary Martin,  
Biology Professor @ Occidental College*



## **Courses taught with the SEM:**

- Introductory Biology
- Upper division Invertebrate biology

## **Other activities:**

- Student research
- Book chapters
- Publications



# Another example: Western New England University



*“This instrument enriches our program and helps students get interested and excited about science.” –Dr. Robert Gettens, BioEngineering Professor @ WNE College*



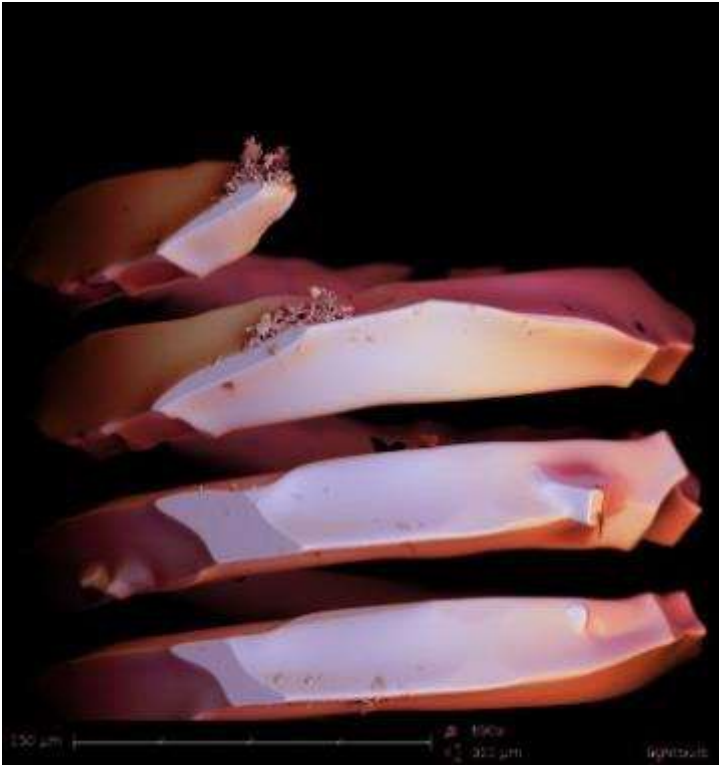
## **Departments using the SEM:**

- Biomedical Engineering
- Chemistry

## **Other activities:**

- Student research
- Industrial Collaborations
- Summer program for high school students

# And just cool stuff that you break: Colorized SEM images of a broken light bulb filament

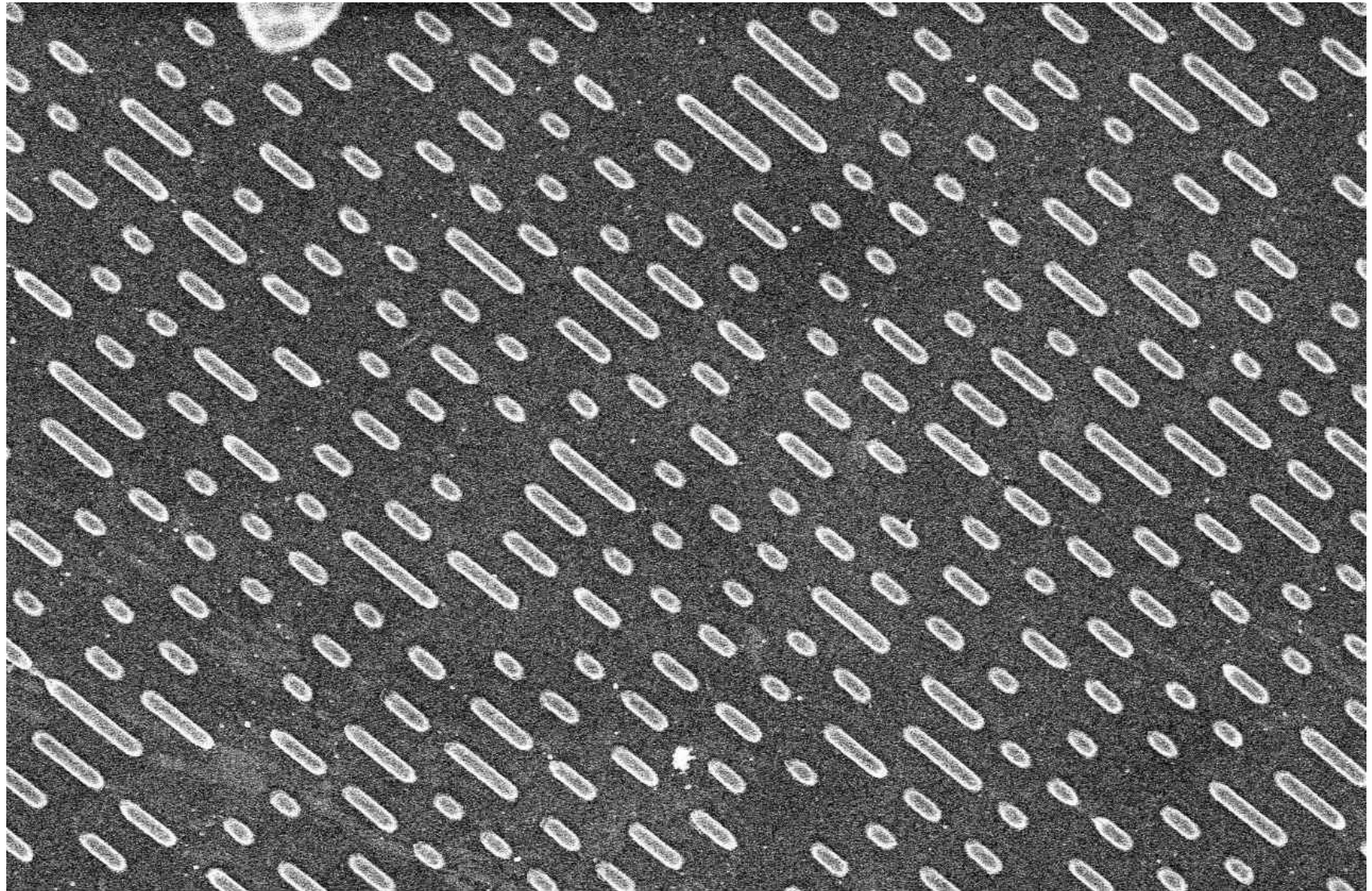


Questions?



Please type all questions or  
comments into the Chat Box

What do you think this is an SEM image of?

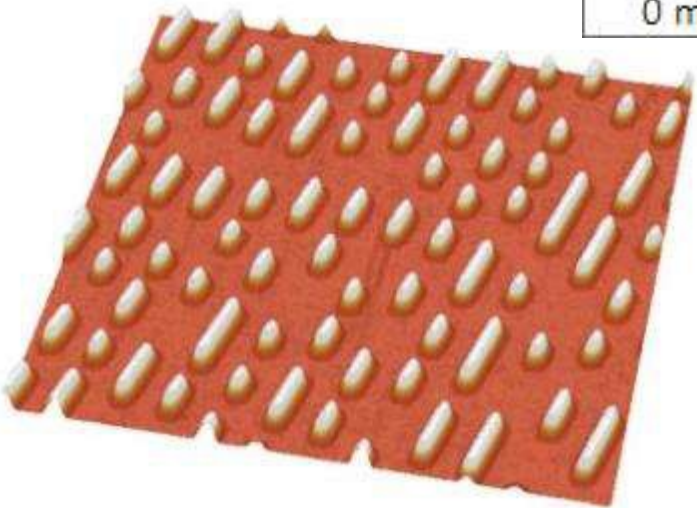
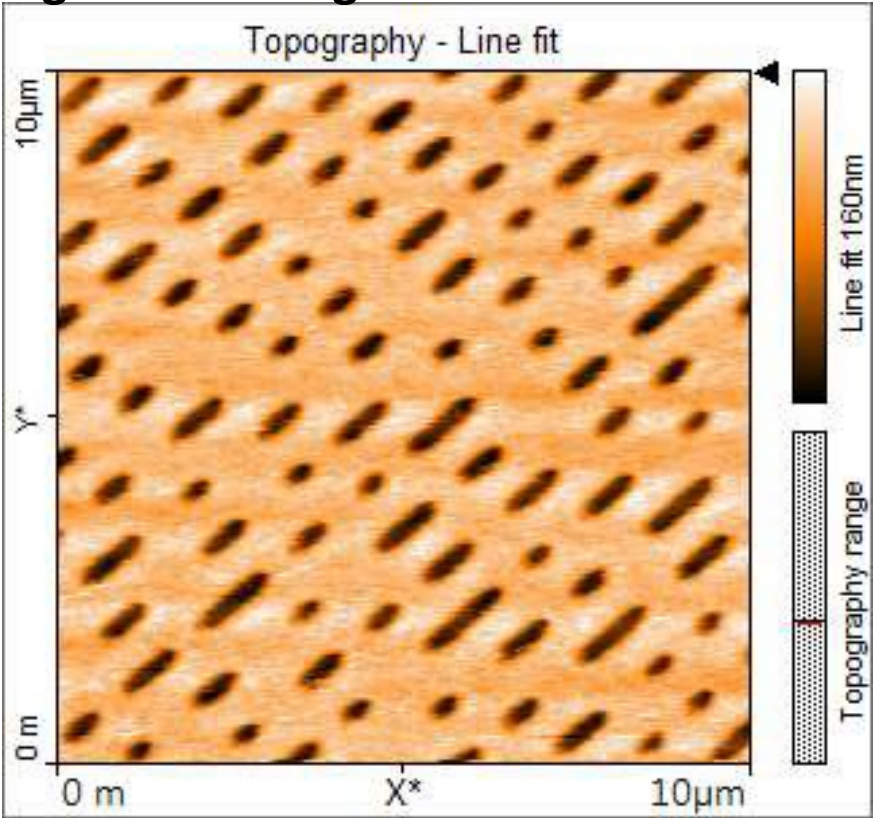


Yale 5.0kV 5.0mm x3.00k SE(M)

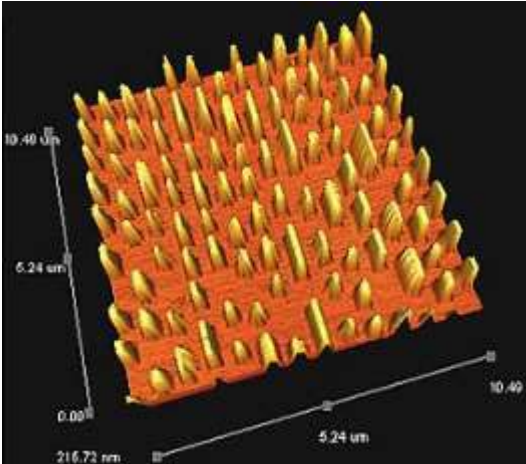
10.0um



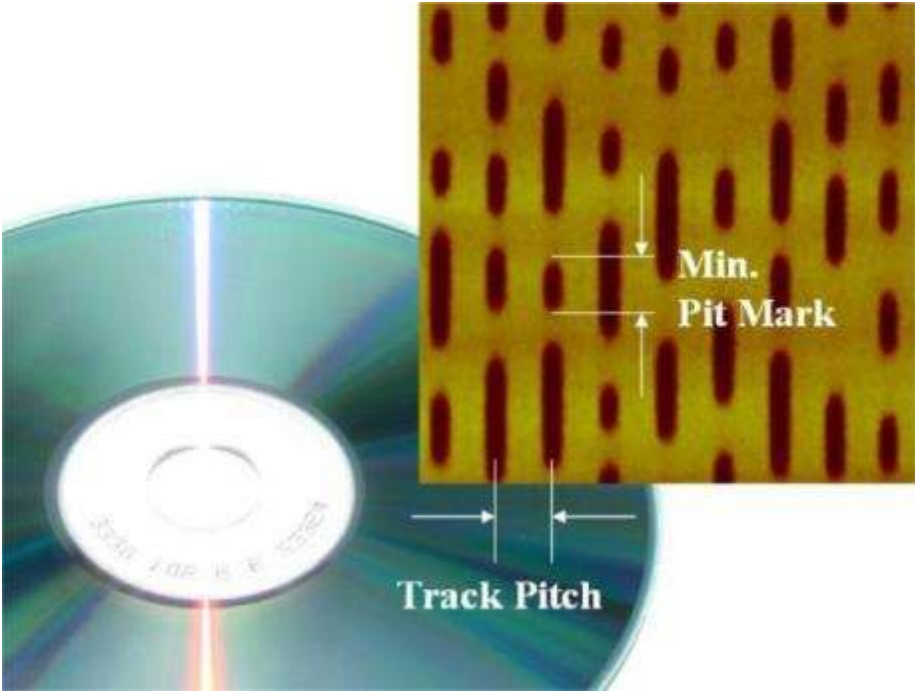
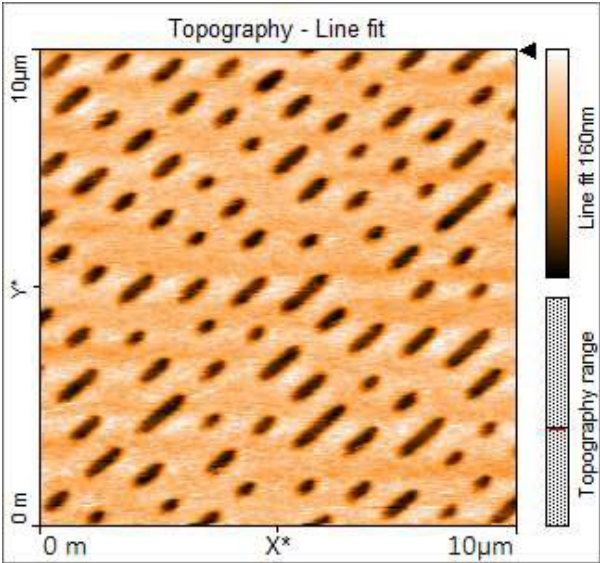
Maybe it's easier to figure out using an Atomic Force Microscope?



*Still no idea?*

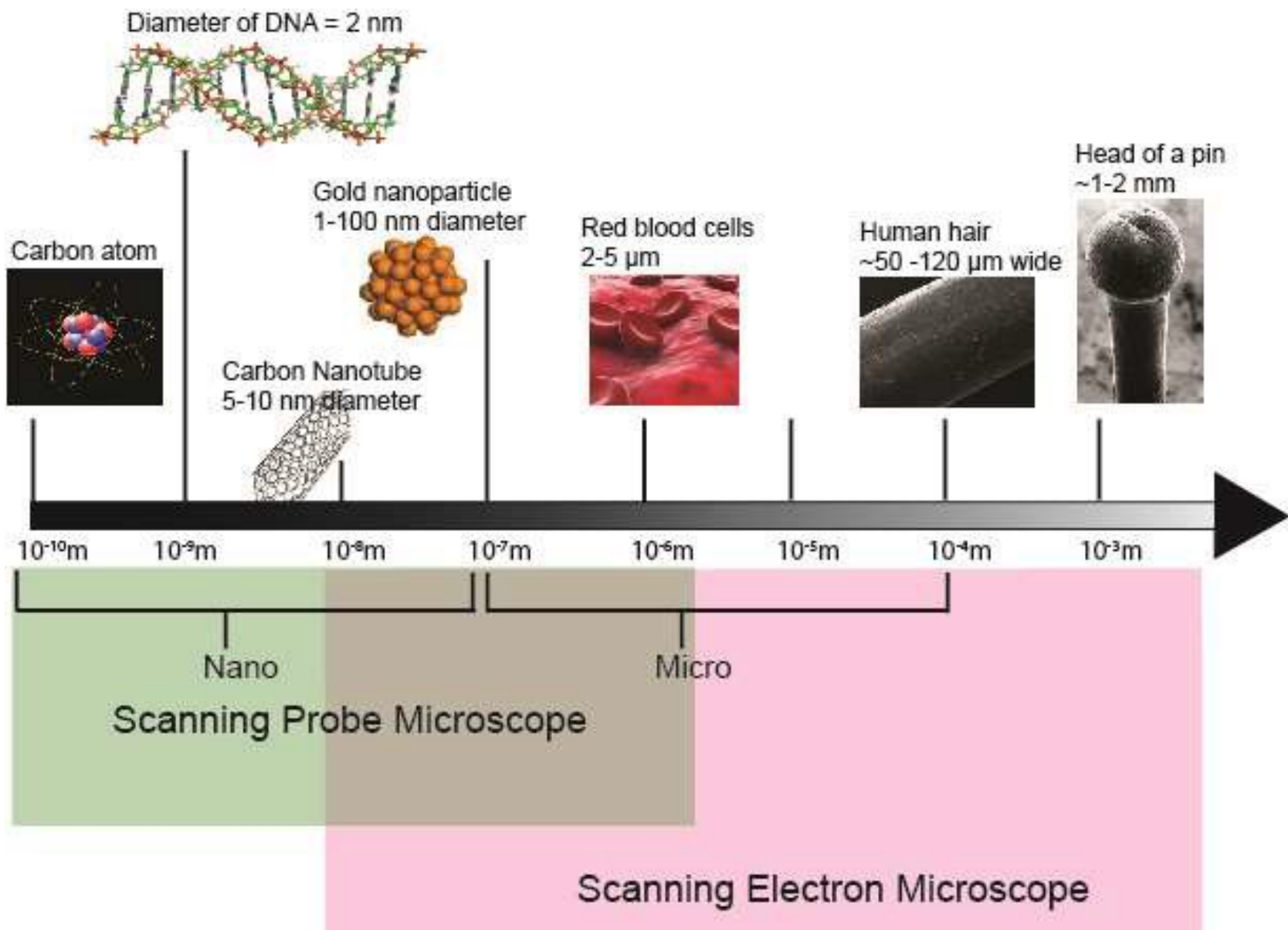


# A DVD!

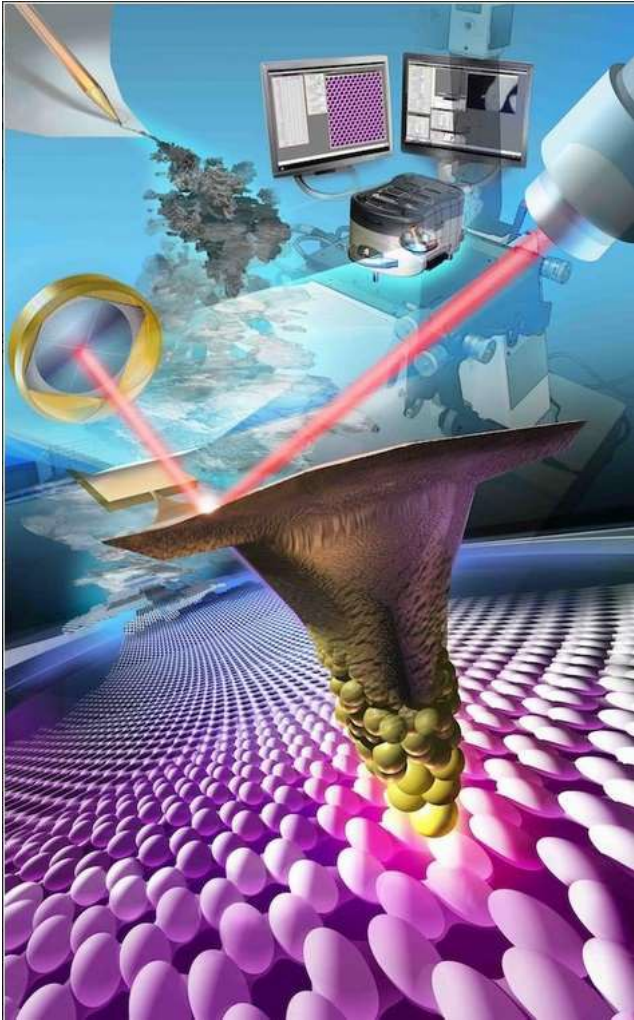
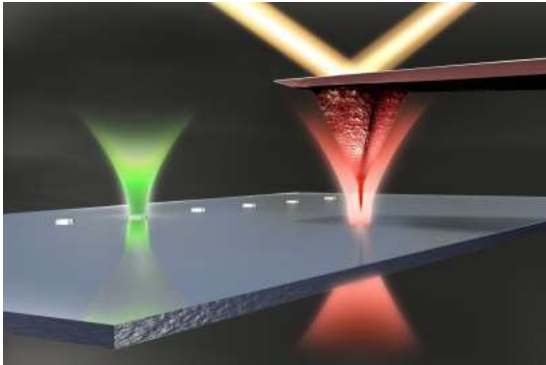




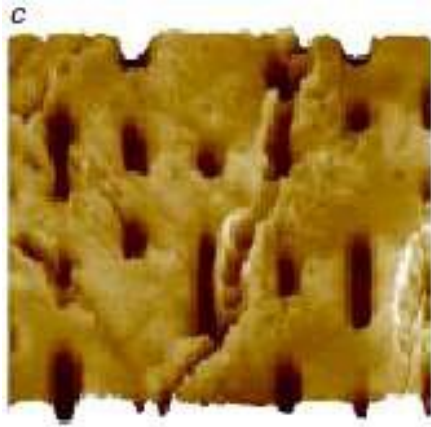
# Is AFM essential to “nano”?



# AFM – The Poster Child of Nano ...



Why won't my CD play after my 2-year old has slid it across the floor?



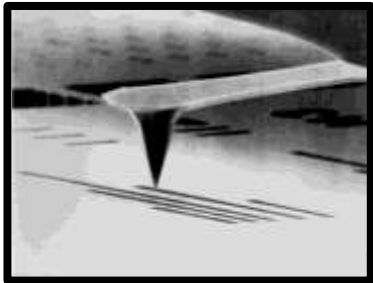
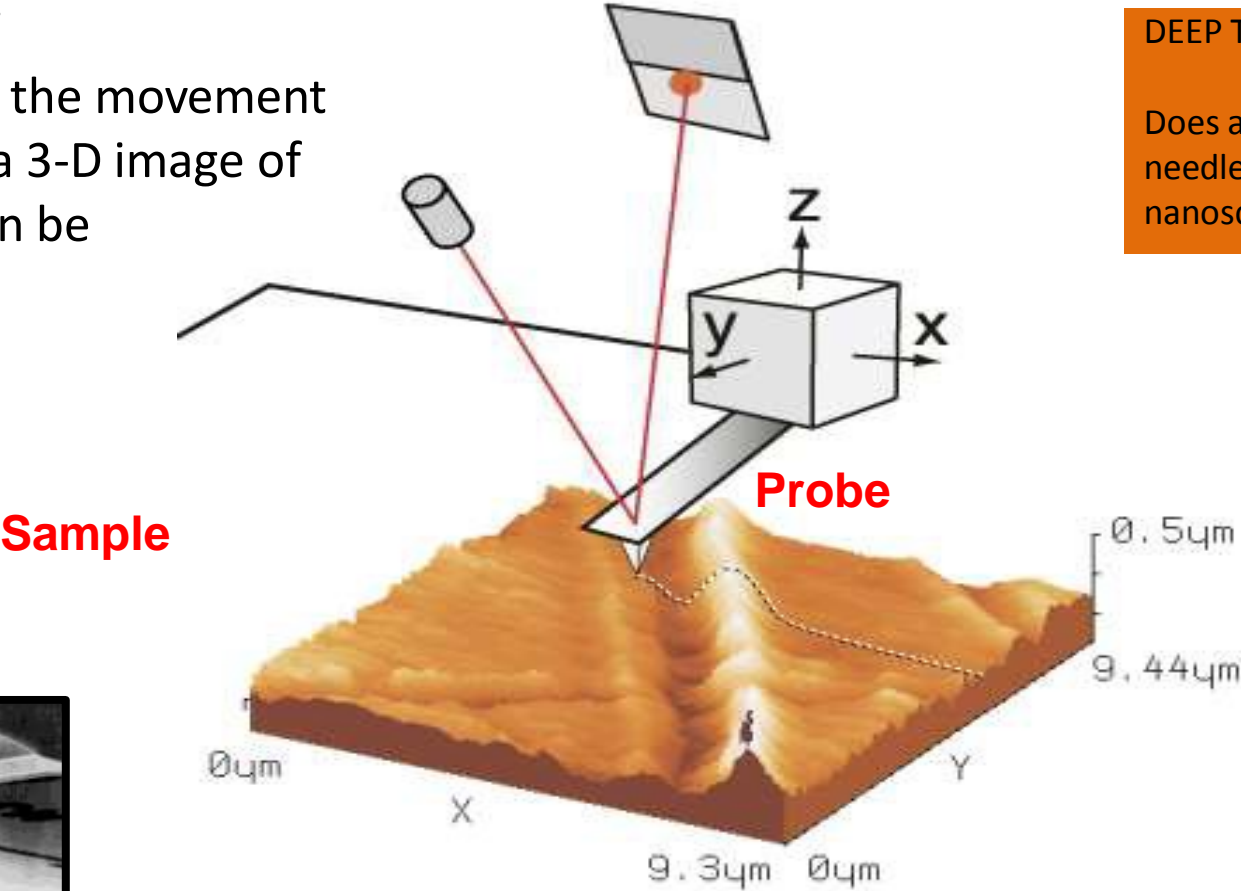
In simple terms, the atomic force microscope works by scanning a sharp probe over the surface of a sample in a raster pattern.

By monitoring the movement of the probe, a 3-D image of the surface can be constructed.

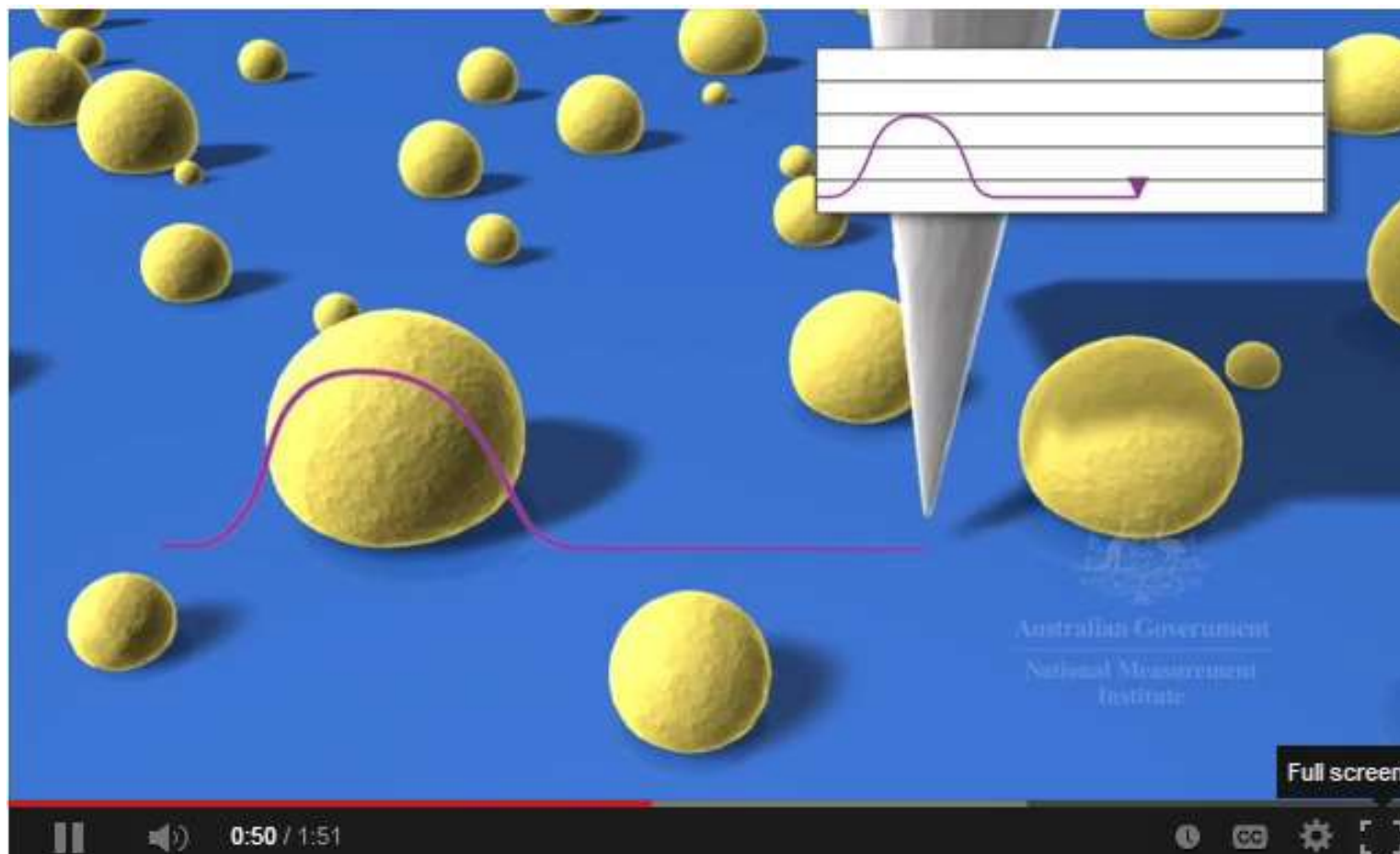


DEEP THOUGHT...

Does a record player  
needle touch at the  
nanoscale?



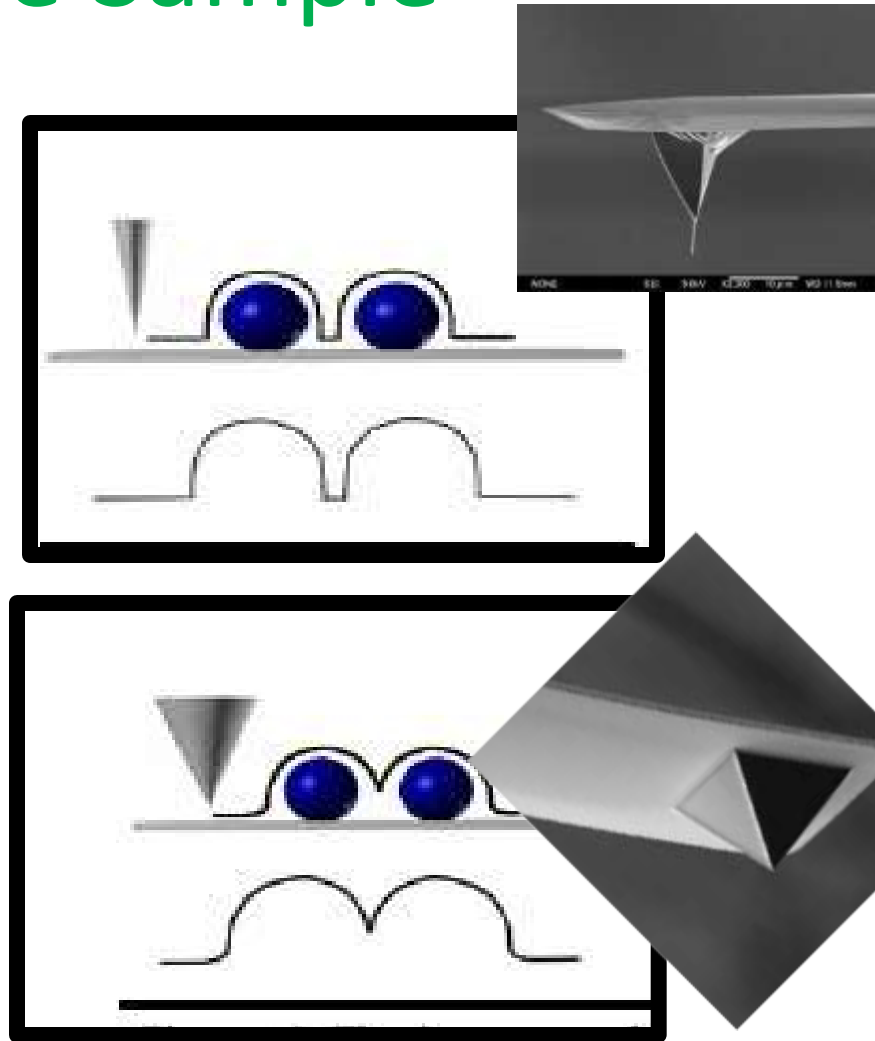




<http://www.youtube.com/watch?v=Ha53tFTsmW8>

# Scanning the Sample

- Tip brought within nanometers of the sample (van der Waals)
- Radius of tip limits the accuracy of analysis/ resolution
- Stiffer cantilevers protect against sample damage because they deflect less in response to a small force





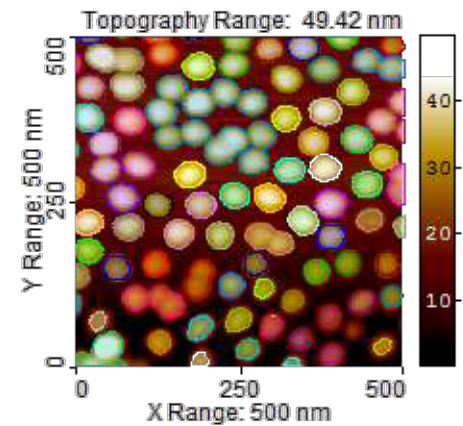
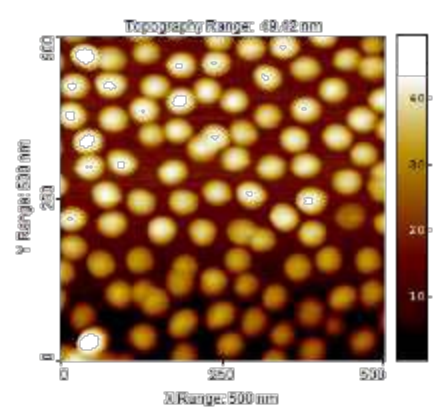
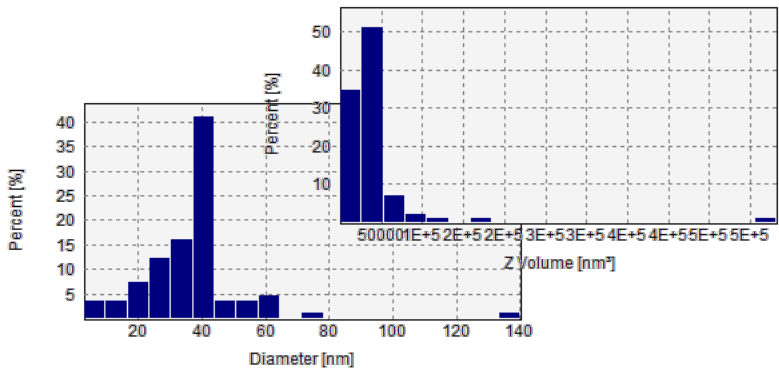
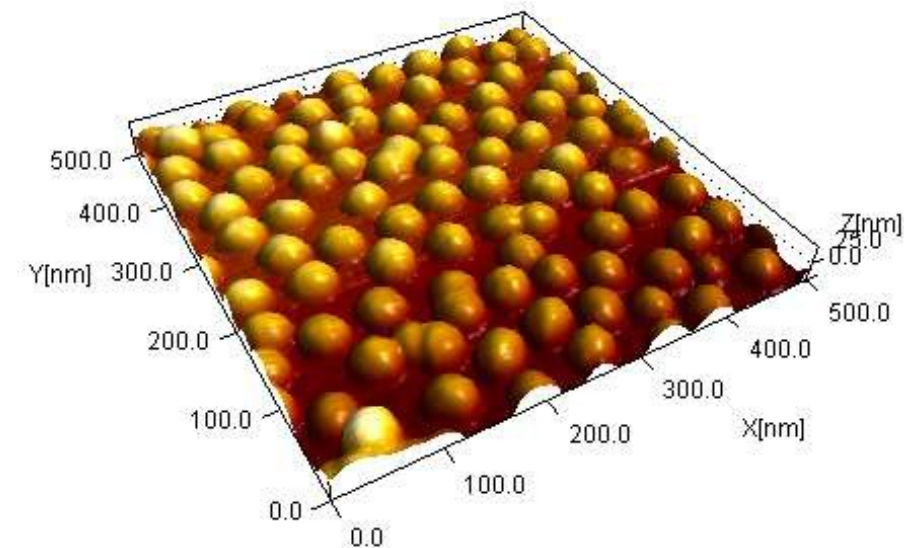
# Particle Analysis

AFM can easily discern nanometer size particles/additives

**Consumer Goods:** Cosmetics, Hair Care, Paints/Dyes

**Medical:** Drug Delivery, Pill fillers, Active ingredients

**Industrial:** Structural additives



# Nanotubes

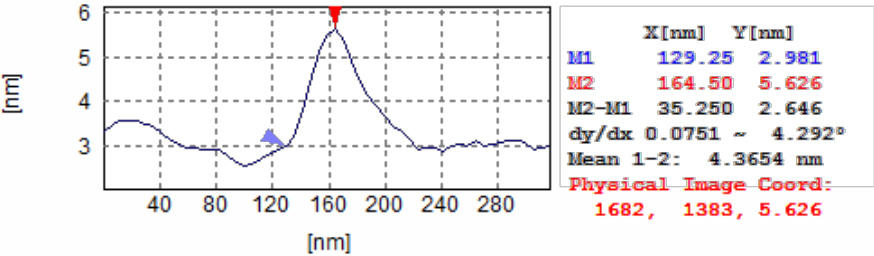
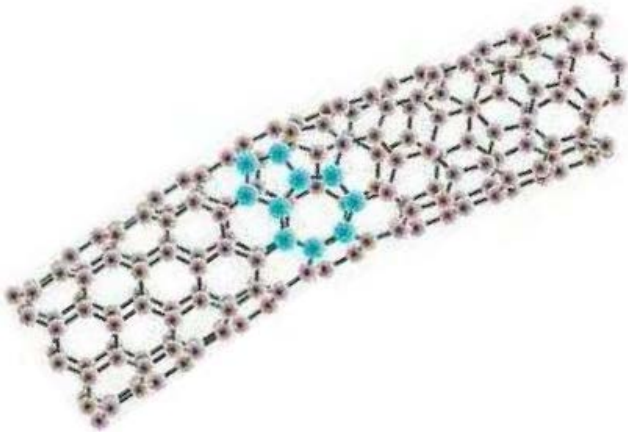
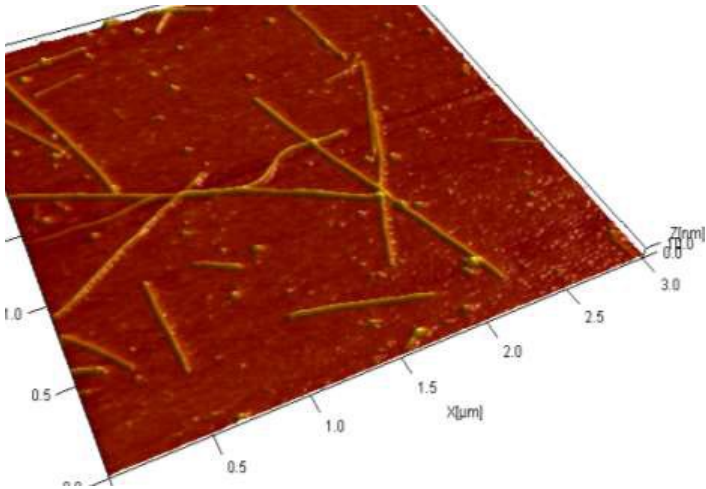
AFM is the technique of choice for characterizing carbon and other nanotubes. Length, diameter, and density can easily be determined.

**Structural:** Windmills, Car frames

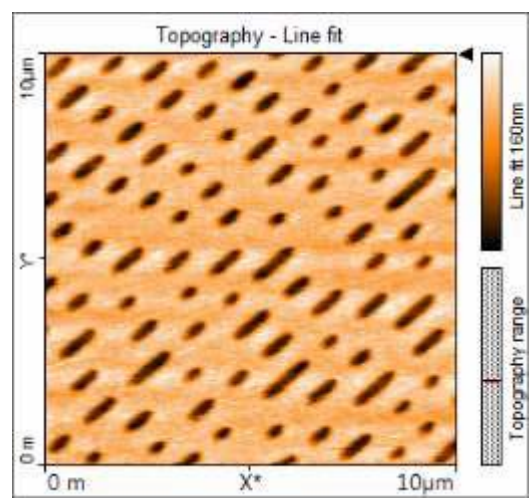
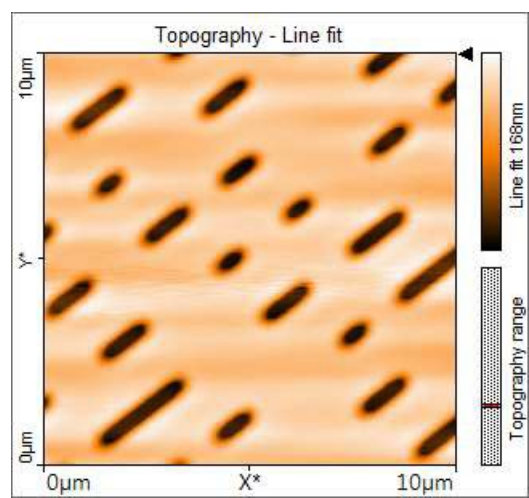
**Biological:** Pathogen detection, Bone scaffolding

**Energy:** Windmills, Solar cells

**Consumer:** Flexible electronics



# Why are all the AFM images “orange”?







Speaking of Color...

A Matching Game...

Match the AFM  
image to the Disk

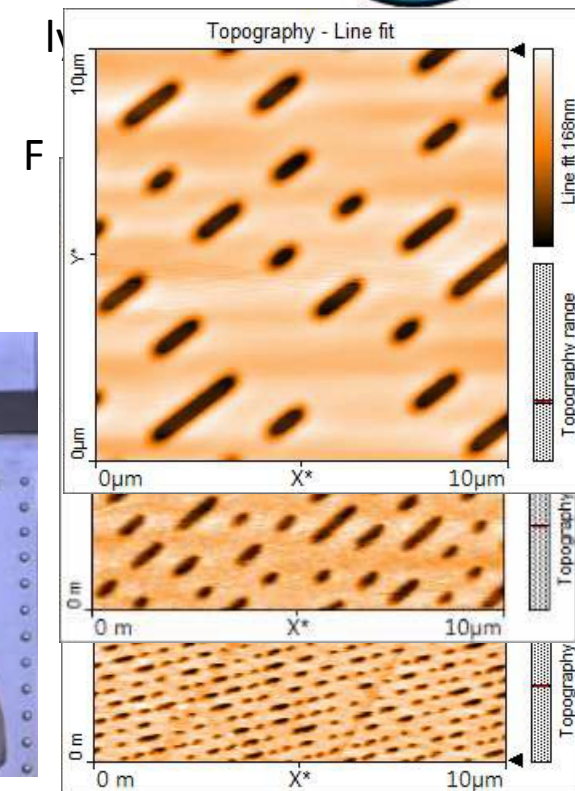


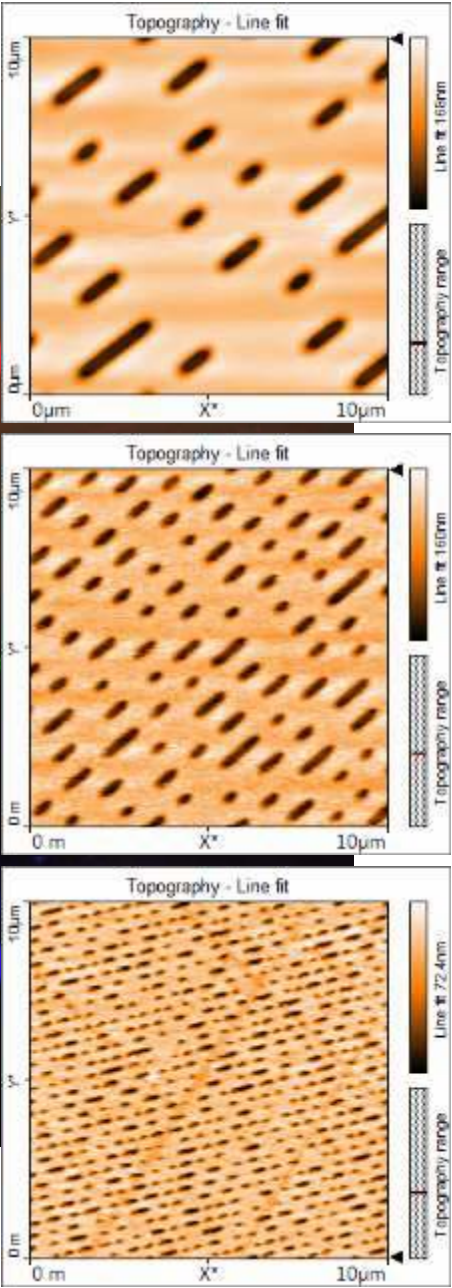
Facebook profile for Mike Davis. The profile picture shows a man in a blue shirt holding a book with a flame. The cover photo shows a periodic table of elements. The profile name is Mike Davis. The bio says "Mike Davis". The profile has 161 photos, 67 mutual friends, and 67 mutual friends. The post is titled "AFM Images I took in my basement." and is updated about a month ago. The post text says: "15 years ago, if you told me I would have an AFM in my basement, I would have been pissed as hell. 'Can't I leave that damn thing at the lab!'. Now I think its pretty awesome, and I would give almost anything for one of the hundreds of crappy samples I lost or threw away in grad school." Below the text are three AFM images, each labeled "Topography - Line fit". The images show a grid of dark, elongated features on a lighter background. The first image has a scale bar from 0µm to 10µm. The second image has a scale bar from 0 m to 10µm. The third image has a scale bar from 0 m to 10µm.





- ✓ CD..
- ✓ ... DVD...
- ✓ ... Blue Ray..
- ✓ A **Blue** Ray?
- ✓ Like a Ray Gun?
- ✓ Like a LASER Ray Gun?
- ✓ Like "Light Amplification by Stimulated Emission of Radiation"?







# Five Microscopes

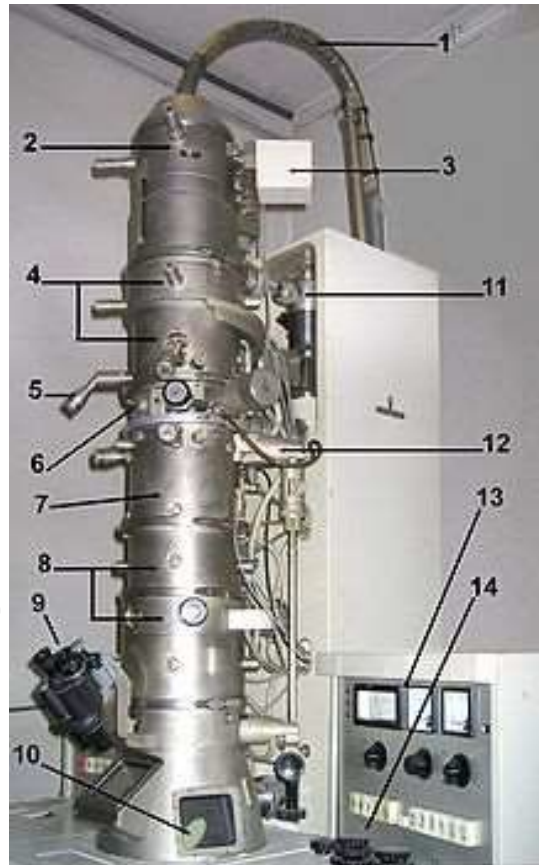
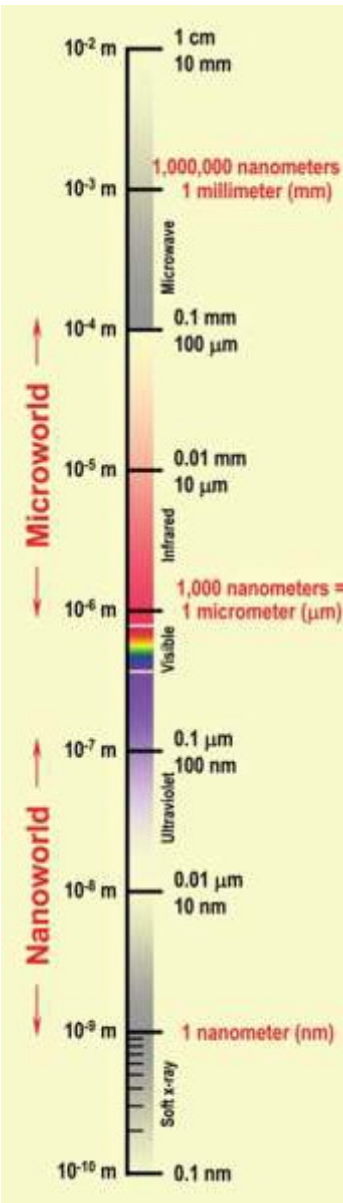
to fit the scale of things

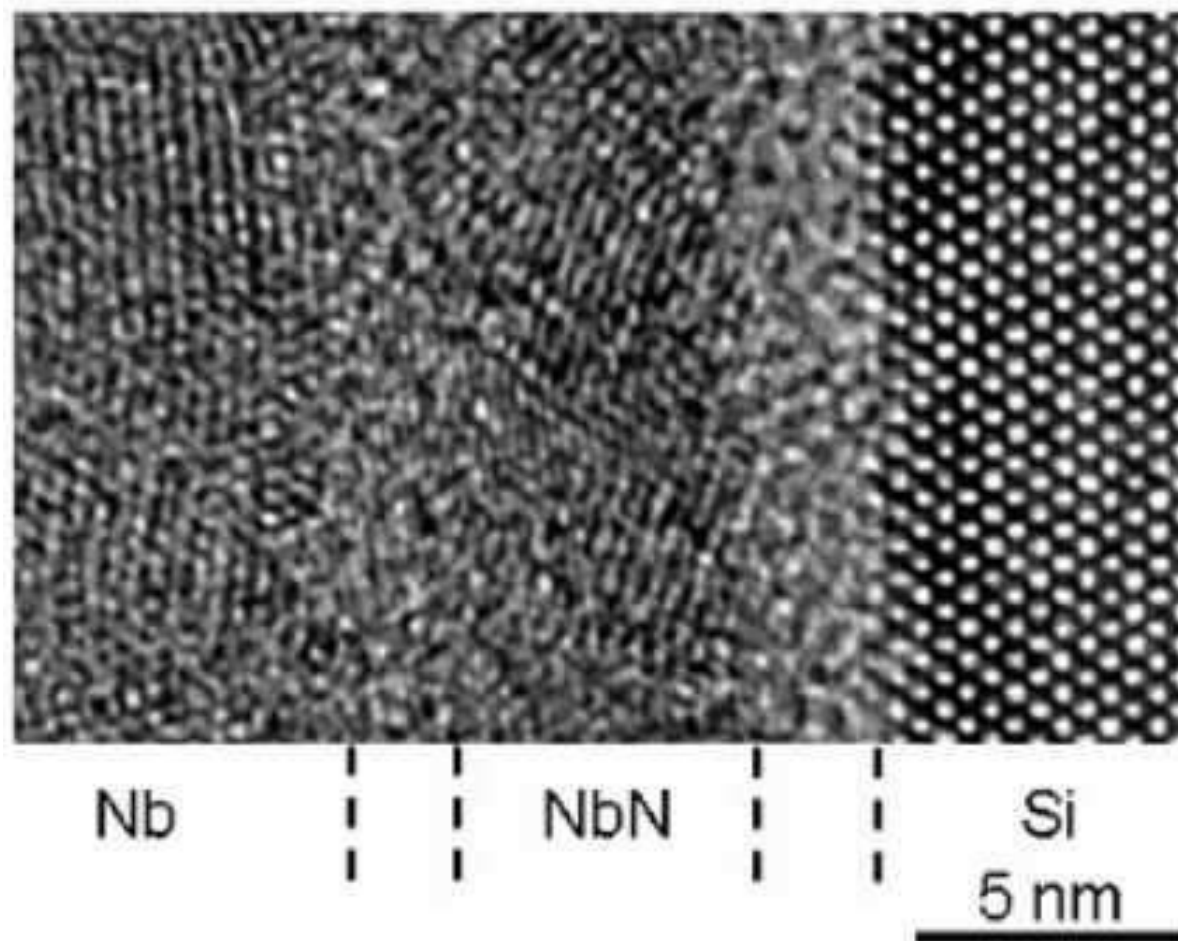
# Transmission Electron Microscope

## Scanning Electron (SEM)

With each instrument, I'll try to communicate:

- What it does.
- How it works, on a high level.
- Where it's used.
- What subjects can be taught with it.
- What skills your students can learn from it. (*this is subjective*)

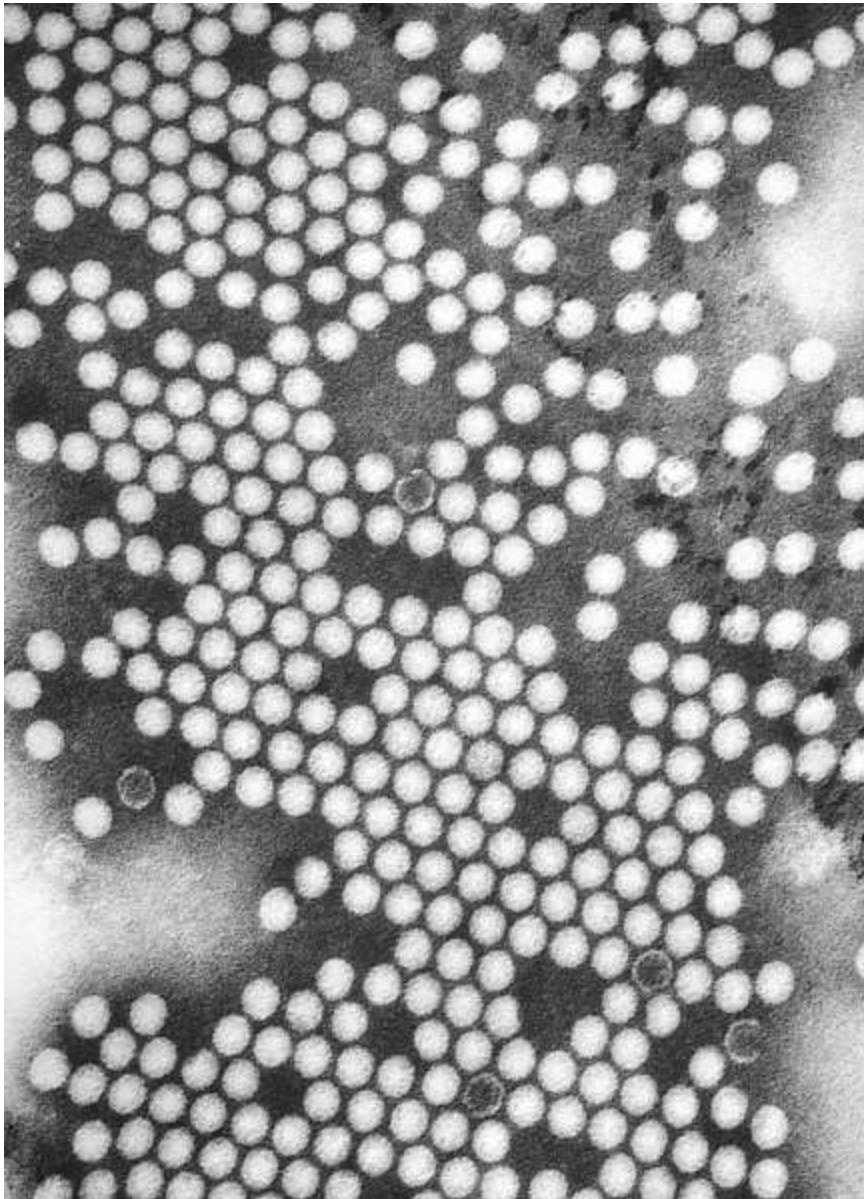




Layer structure on a silicon substrate:

(Photo by: Siegel/Gerthsen).

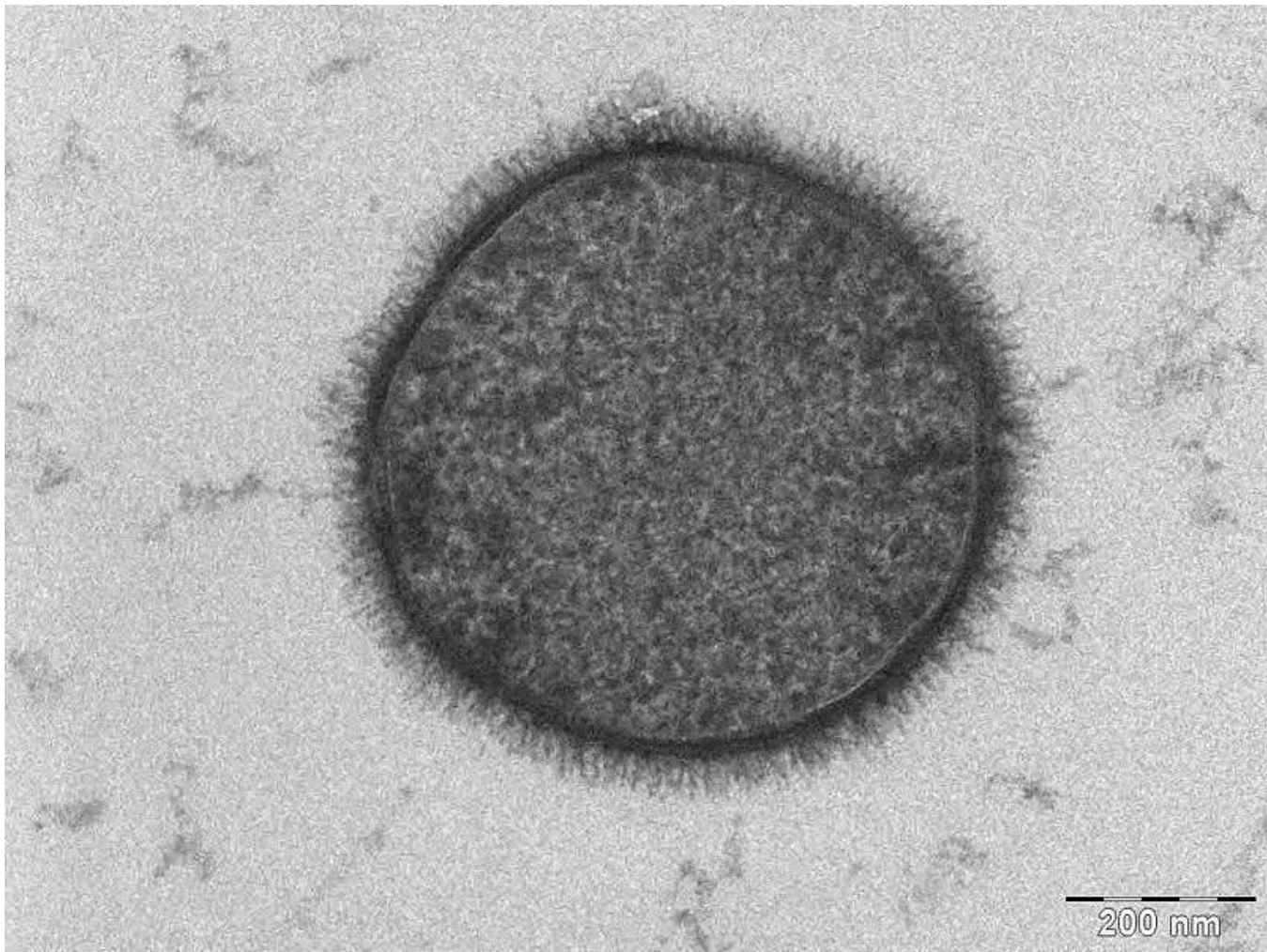
[http://www.kit.edu/visit/1839\\_156.php](http://www.kit.edu/visit/1839_156.php)



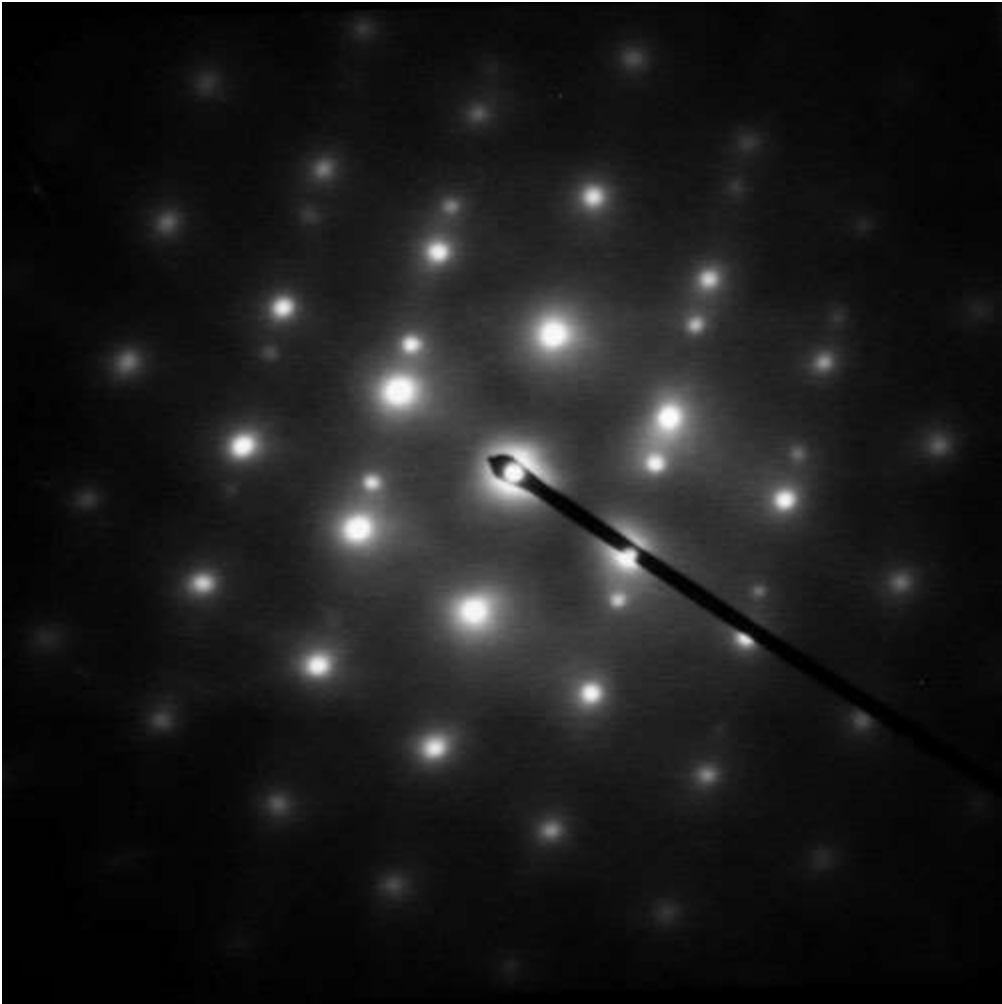
A TEM image of the [polio](#) virus.  
The polio virus is 30 [nm](#) in size.

[http://en.wikipedia.org/wiki/Transmission\\_electron\\_microscopy#cite\\_note-1](http://en.wikipedia.org/wiki/Transmission_electron_microscopy#cite_note-1)

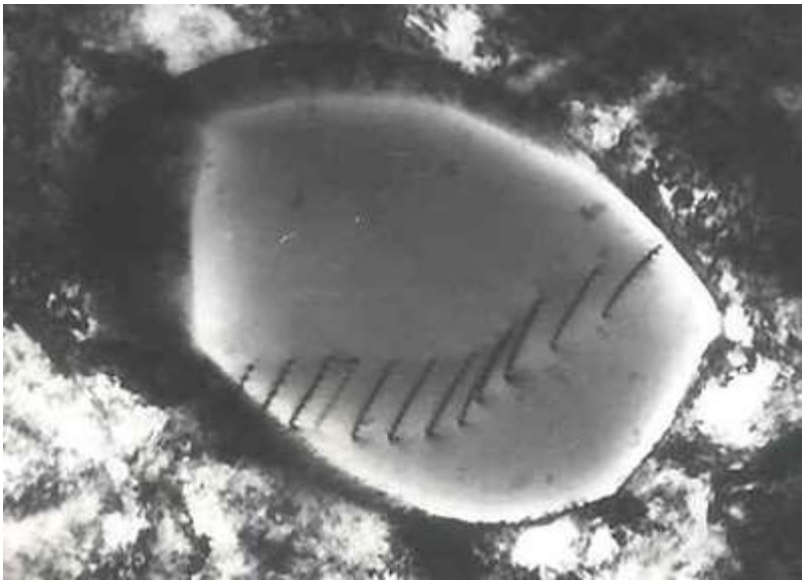




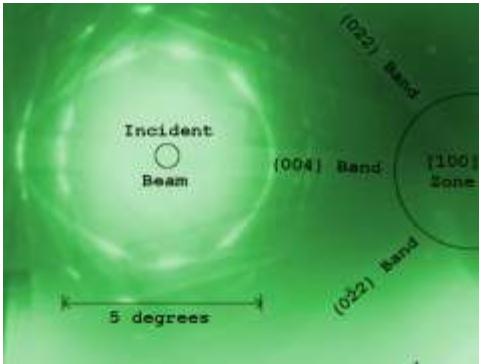
A section of a cell of [\*Bacillus subtilis\*](#), taken with a Tecnai T-12 TEM. The scale bar is 200 nm.



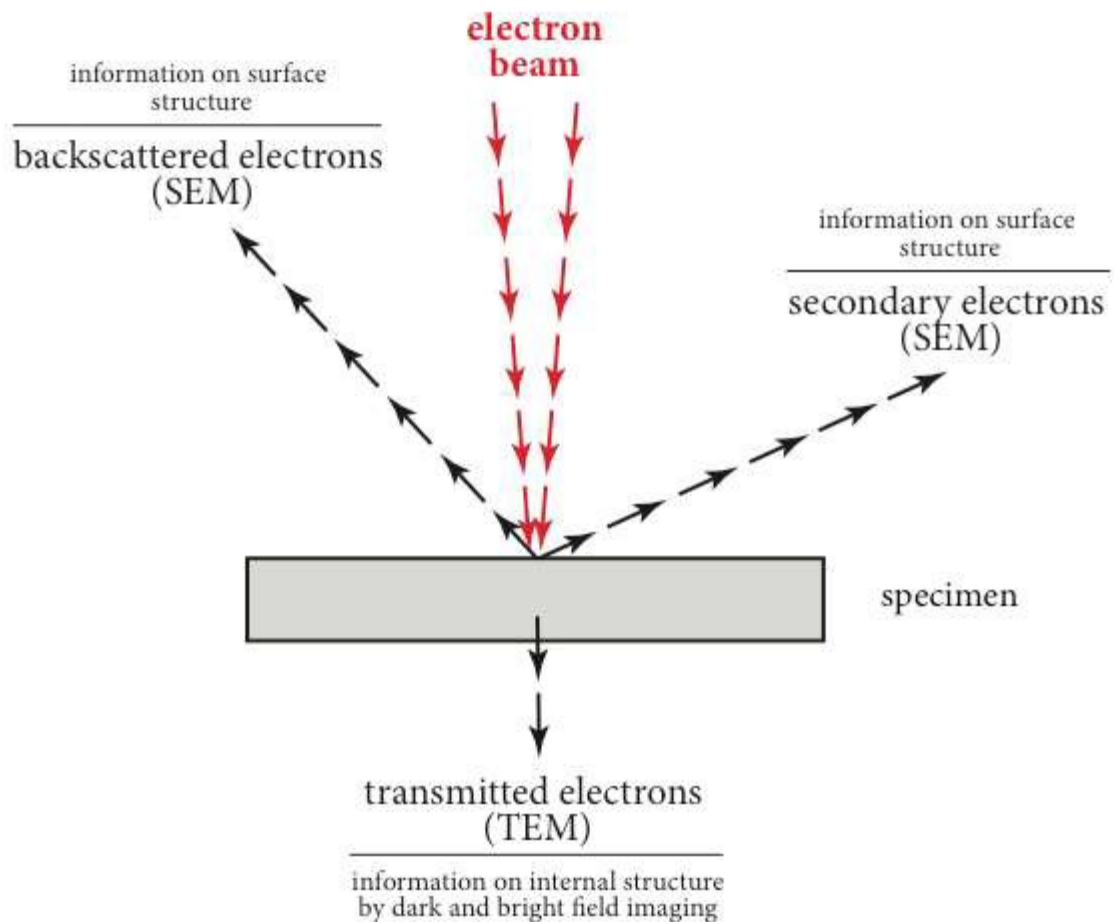
Crystalline diffraction pattern from a twinned grain of FCC Austenitic steel

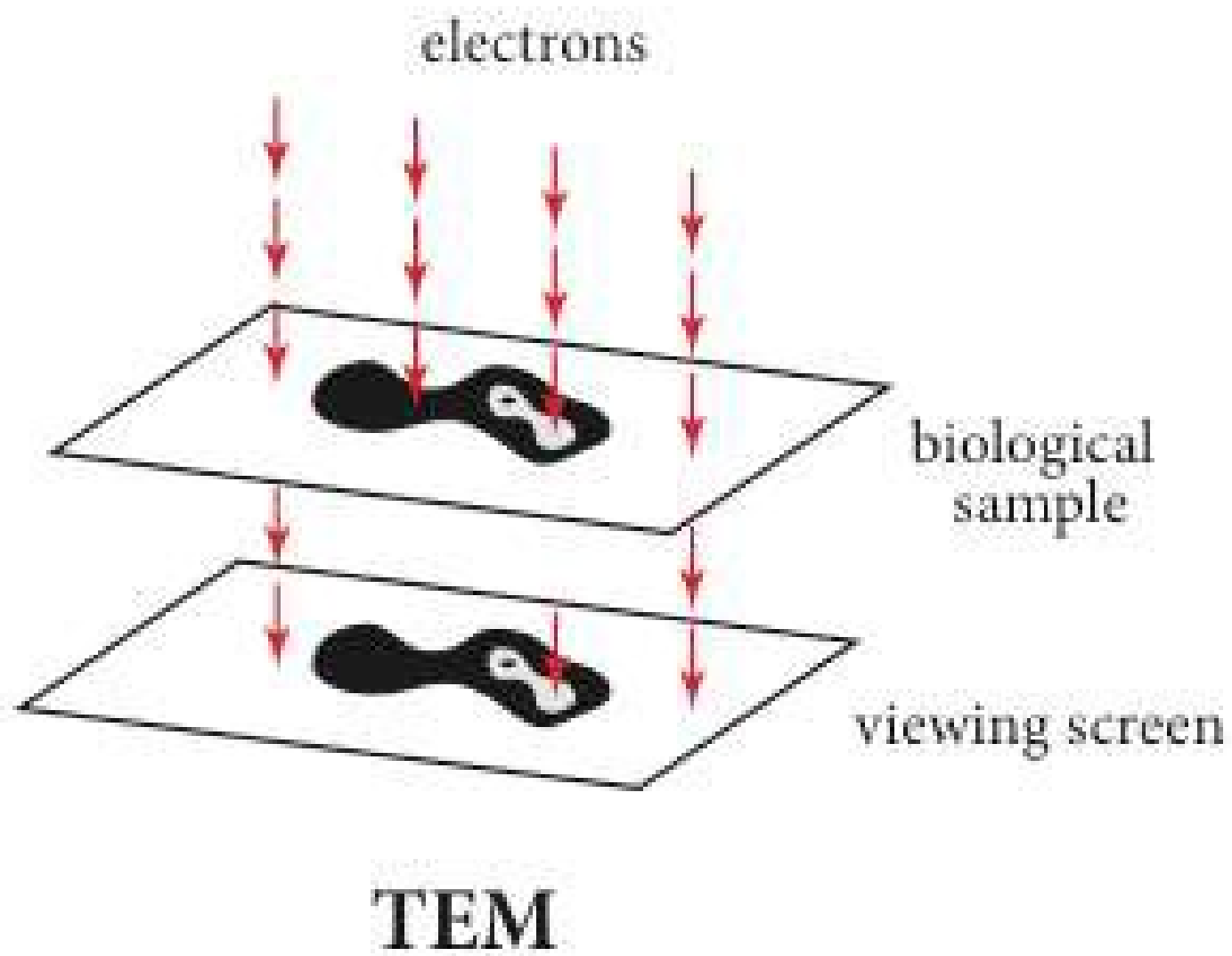


Transmission electron micrograph of [dislocations](#) in steel

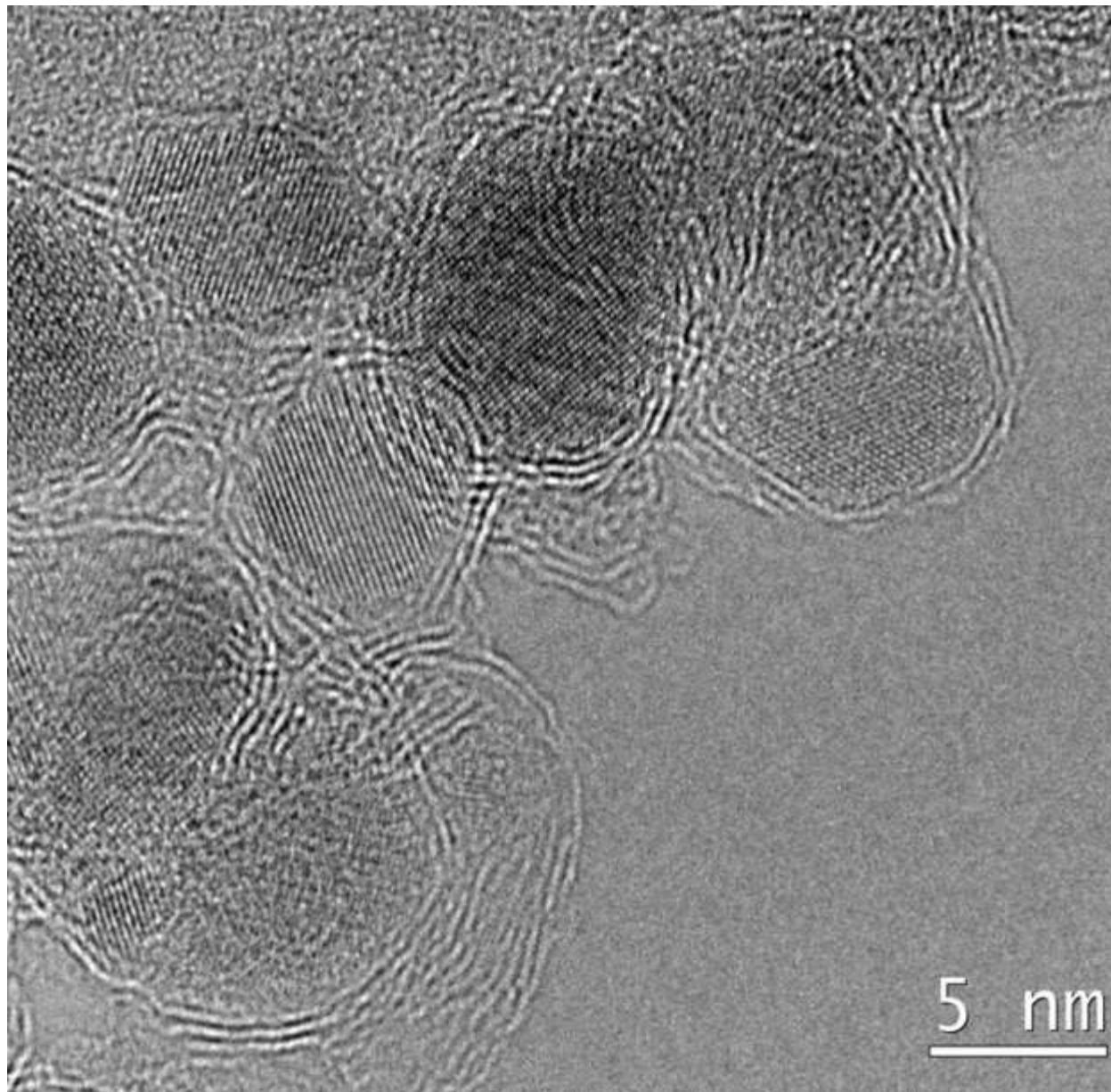


Convergent-beam Kikuchi lines from silicon, near the [100] [zone axis](#)









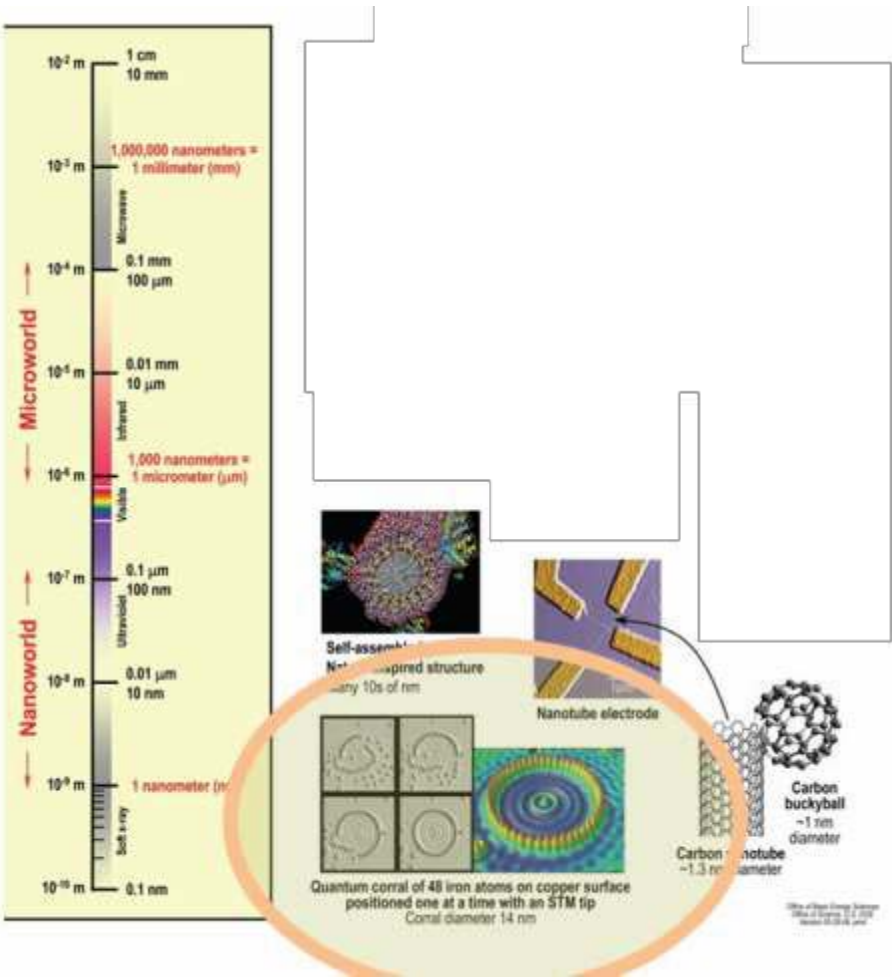
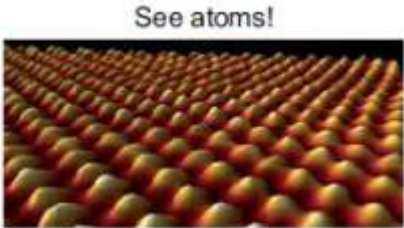
**HRTEM image of a  
Fe/LiF/C anode for  
lithium ion batteries**

*R. Prakash and C. Kübel et al., J. Power  
Sources, 2011, 196, 5936-5944.*



# SCANNING TUNNELING MICROSCOPE

- What it does:
  - Measures and creates a visual representation of very smooth, atomic level, surfaces. Image atoms.

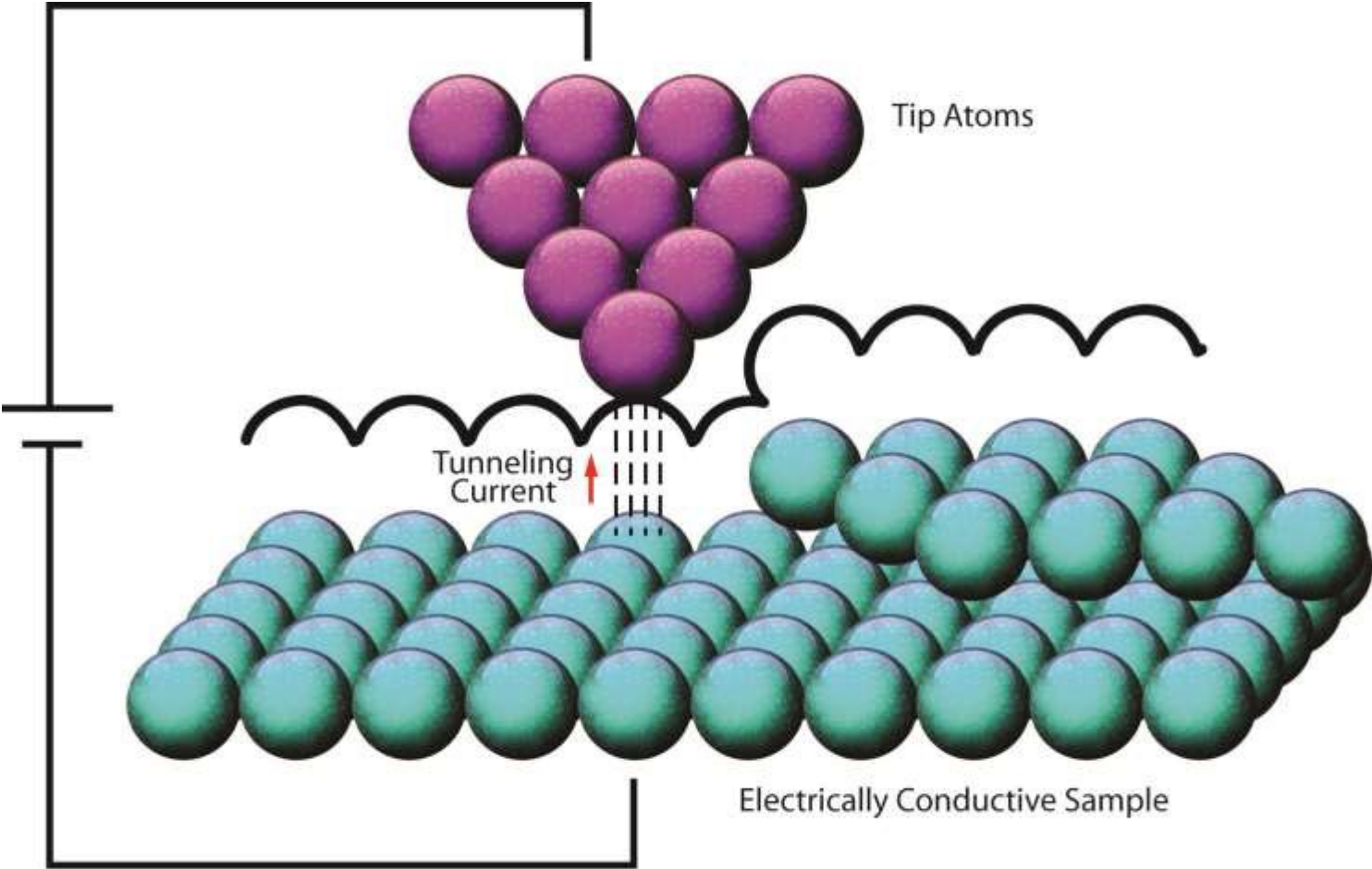


Iconic image of “Nano” from IBM in the 90’s - “Quantum Corral”

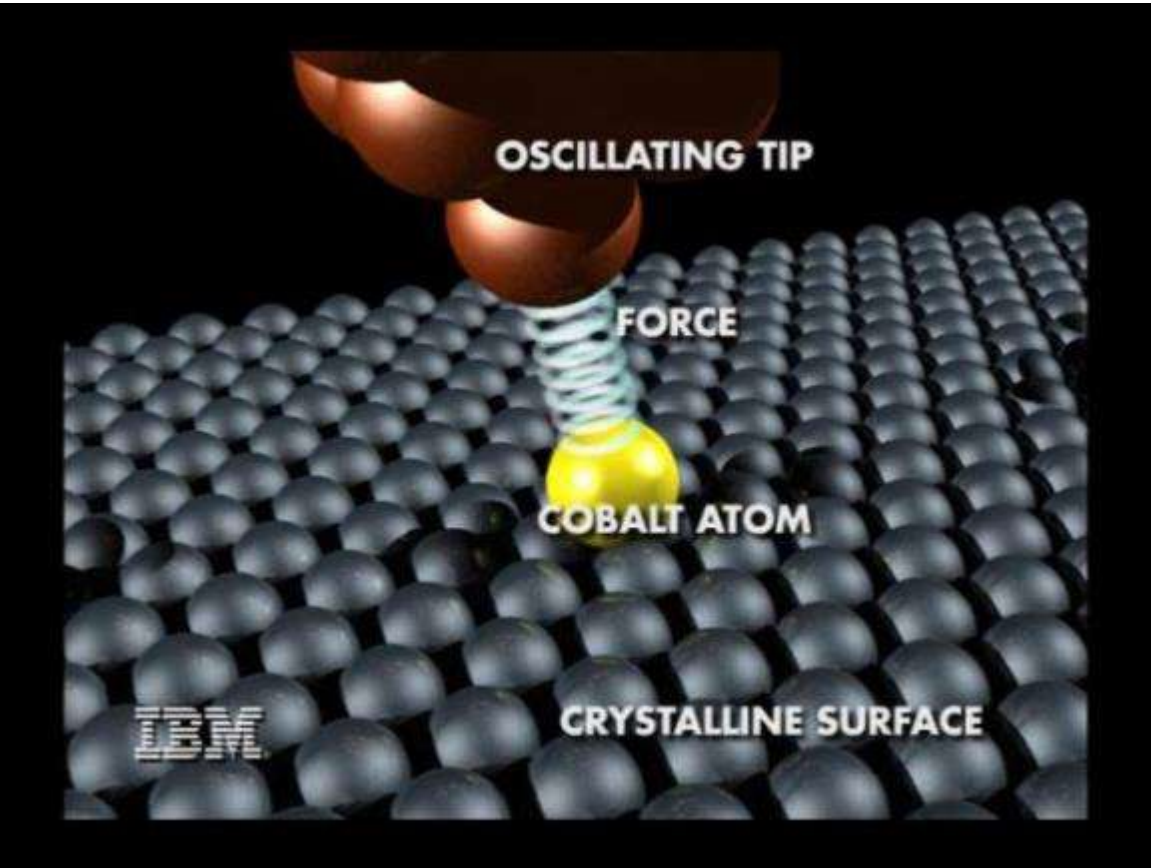
# **A Boy and His ATOM – World's Smallest Movie (IBM)**

<http://www.youtube.com/watch?v=oSCX78-8-q0>

# Quantum Tunneling



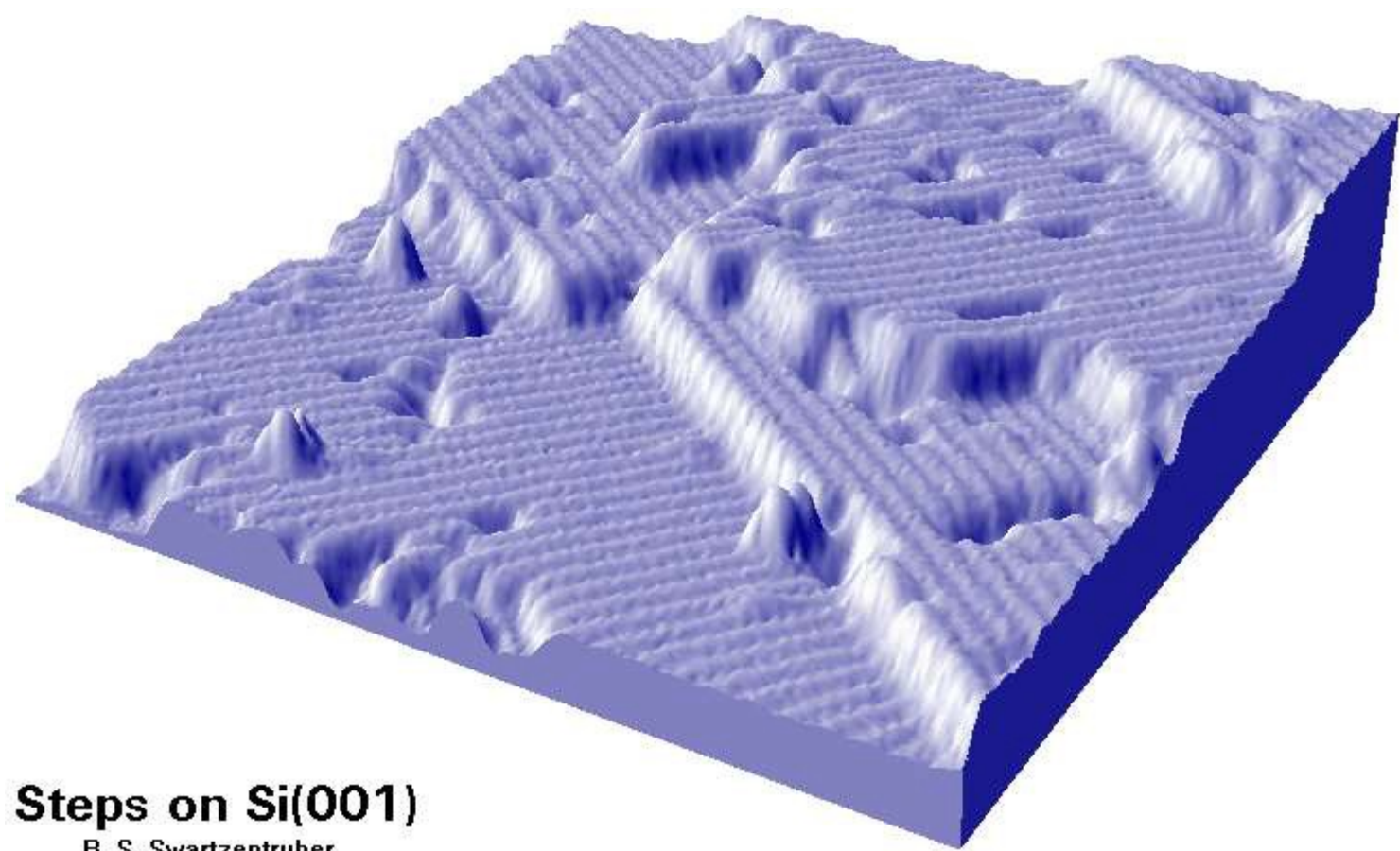
Artist's impression of the IBM microscope tip



<http://physicsworld.com/cws/article/news/2008/feb/27/feeling-the-force-on-a-single-atom>

Where it's used:

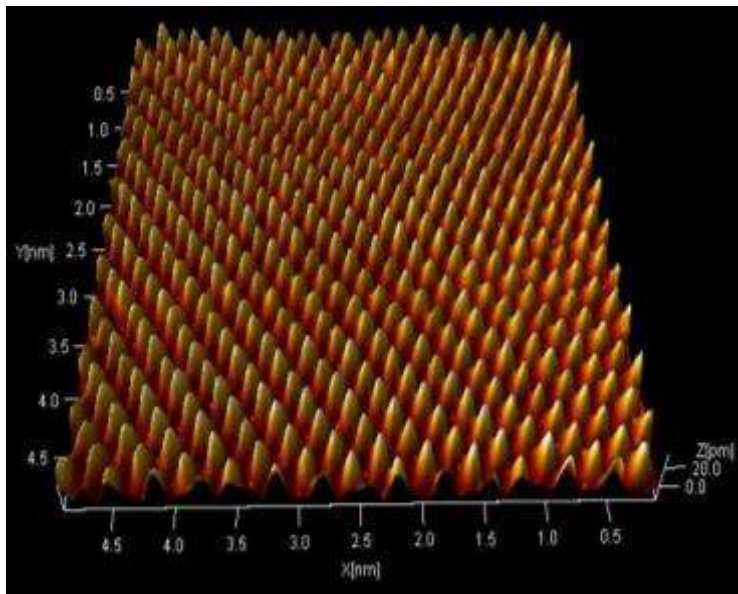
- Advanced Research Labs
- *Teaching and Outreach*
- *Any other industrial uses, audience?*



**Steps on Si(001)**  
B. S. Swartzentruber



# Imaging atoms and beyond....



- What subjects can be taught with it.
  - See ATOMS!
  - Physics
- What skills your students can learn from it. (*this is subjective*)
  - *Conceptualization*
  - *Understanding data, and how it was collected*
  - *Operating delicate equipment*
  - *Not quite a resume skill yet.*

# TEST... Match the image group with the Scope



*3D Optical Microscope*



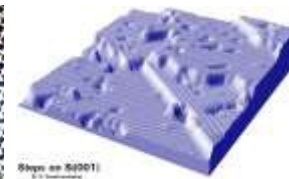
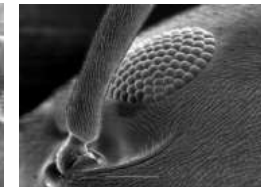
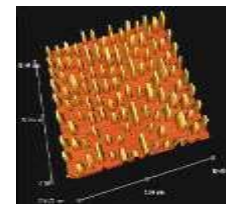
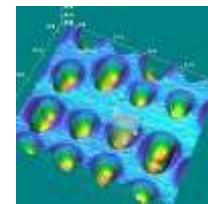
*SEM*



*AFM*



*STM*



# Is it Practical?

Classroom Integration – Square Peg,  
Round Hole?

## Five Microscopes



to fit the scale of things

**3D Optical**

Wavelength of Light



**Scanning Electron (SEM)**

Wavelength of Electron



**Atomic Force (AFM)**

Atomic Force



**Transmission Electron (TEM)**

Electron Interaction  
through a material



**Scanning Tunneling (STM)**

Quantum Force





your clean room

isn't like





isn't  
like



Making microprocessors is a tricky business. The tiniest speck of dust is the equivalent to a two-ton boulder around our microscopic transistors. This is why our clean rooms are 10,000 times cleaner than a hospital operating room. It's also why our workers must wear these silly-looking outfits. Learn more at [sponsorseftomorrow.com](http://sponsorseftomorrow.com).



Sponsors of Tomorrow™

# Practical Considerations

When considering investing  
in instrumentation...



- **Fast – to use – classrooms have a boxed time**
  - A 20 min “get ready” time per instrument is not realistic

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  - Power supply – into the wall.
  - Pumps – basic. No dangerous chemicals. No special wet or fume hood for venting.



# Practical Considerations

When considering investing in instrumentation...



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  - You’re a professional teacher, not an amateur technician
- **No infrastructure requirements**
  - Power supply – into the wall.
  - Pumps – basic. No dangerous chemicals. No special wet or fume hood for venting.
- **Easy-to-use “real” scientific instrument**
  - Not a model; not ‘dumbed down’ equipment; a real instrument made intuitive; think “iPad”
  - Capable of the same type of experiments in research institutions and industry; measures in the same way



Is it Practical?  
That's not the question...  
Is it do-able?  
Answer for yourself...



shreya @TeachNano now  
Students at Wheeling High School working with the Zeta 3D Optical profiler! @lisa\_delmuro pic.twitter.com/rfNXuUpu5c  
Hide photo Reply Delete Favorite More





# The Scale of Things – Nanometers and More



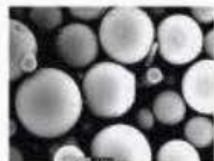
## Things Natural



Dust mite  
200  $\mu\text{m}$



Ant  
~ 5 mm

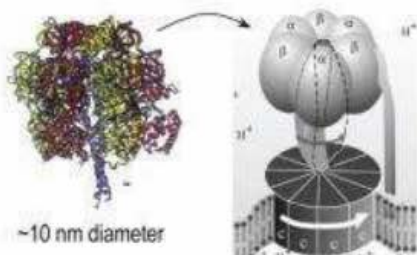


Fly ash  
~ 10-20  $\mu\text{m}$



Human hair  
~ 60-120  $\mu\text{m}$  wide

Red blood cells  
(~7-8  $\mu\text{m}$ )



~10 nm diameter

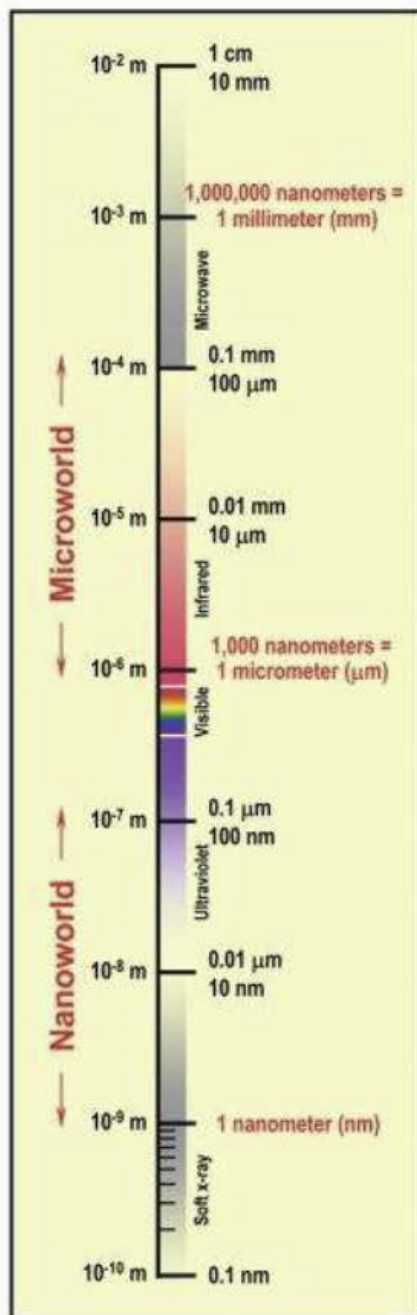
ATP synthase



DNA  
~2-1/2 nm diameter



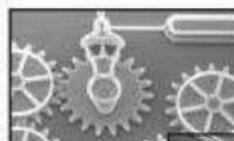
Atoms of silicon  
spacing 0.078 nm



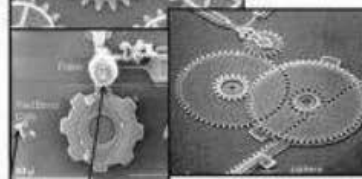
## Things Manmade



Head of a pin  
1-2 mm

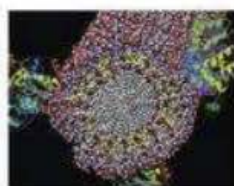


MicroElectroMechanical (MEMS) devices  
10 -100  $\mu\text{m}$  wide



Pollen grain  
Red blood cells

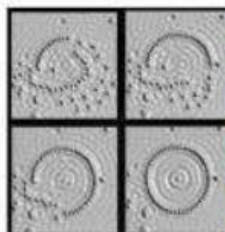
Zone plate x-ray "lens"  
Outer ring spacing ~35 nm



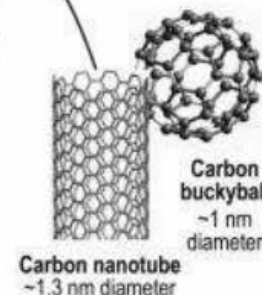
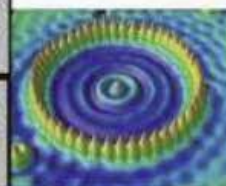
Self-assembled,  
Nature-inspired structure  
Many 10s of nm



Nanotube electrode



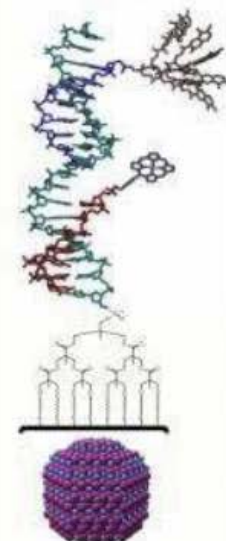
Quantum corral of 48 iron atoms on copper surface  
positioned one at a time with an STM tip  
Corral diameter 14 nm



Carbon nanotube  
~1.3 nm diameter

Carbon  
buckyball  
~1 nm  
diameter

## The Challenge



*Fabricate and combine  
nanoscale building  
blocks to make useful  
devices, e.g., a  
photosynthetic reaction  
center with integral  
semiconductor storage.*



# Questions?

Please type all questions into the Chat Box





# Webinar Recordings

To access this recording, slides, and handout visit  
[nano4me.org/webinars.php](http://nano4me.org/webinars.php)



Thank You!

Thank you for attending the  
NACK Network webinar

**Fundamentals of Metrology and  
Characterization for Nanotechnology**