Nanomaterials and Device Characterization

(나노소재 및 소자 분석)

Prof. Tae-Wook Kim

Department of Flexible and Printable Electronics
Jeonbuk National University

Syllabus

Course Description

This course is designed to teach the principles and concepts of most important techniques available for micro and nanoscale materials characterization and chemical analysis necessary for the development of new materials.

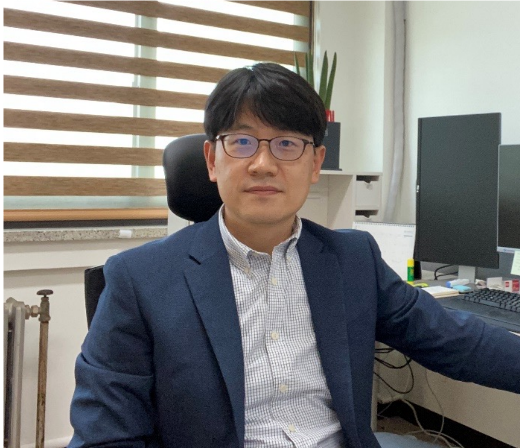
The topics include

- basic principles of characterization,
- instrumentation development
- application platforms to image and probe structural, electrical, magnetic, and optical characterizations as well as chemical analyses at nanometer scale.

Course Goal/Guide

- describe fundamental principles of operation of diverse materials characterization and chemical analysis techniques
- develop the ability to analyze and evaluate the characterization data (plots, graphs, figures, images, and so on)
- develop innovative and creative approach towards design and development of new materials and analysis of their properties.

About Professor



김태욱; 金兌昱; TAE-WOOK KIM

유연인쇄전자공학과

연구실: 공대 5호관 313호

063-270-4827

E-mail: twk@jbnu.ac.kr

Handout will be uploaded right before the class at the website of Applied Nanomaterials and Devices Lab. (www.appnd.org)

강의자료는 Applied Nanomaterials and Devices Lab. 의 홈페이지에 업로드 될 예정임. (www.appnd.org)

About Professor

김태욱; 金兌昱; TAE-WOOK KIM

유연인쇄전자공학과

연구실: 공대 5호관 313호

063-270-4827

E-mail: twk@jbnu.ac.kr

Handout will be uploaded right before the class at the website of Applied Nanomaterials and Devices Lab. (www.appnd.org)

강의자료는 Applied Nanomaterials and Devices Lab. 의 홈페이지에 업로드 될 예정임. (www.appnd.org)

Website (www.appnd.org)

KIM RESEARCH GROUP

전북대학교
JEONBUK NATIONAL UNIVERSITY

Applied Nanomaterials & Devices LAB.

Electronics & Probes by Materials Engineering

연구의 즐거움; PLAY RESEARCH

HOME RESEARCH PROFESSOR MEMBER PUBLICATIONS FACILITES LECTURE PHOTO CONTACT US

Welcome to visit Applied Nanomaterials & Devices Lab.

Our research group is interested in engineering of organic and inorganic functional materials to study novel functions from materials engineering in nanoscale and to probe their functions for potential electronic applications. Recently, we are focused on tailoring size, shape, dimension, and functionality of metallic nanomaterials and integration of emerging electronic devices with ultimate softness for wearable electronics.

Click

Click

KIM RESEARCH GROUP

전북대학교
JEONBUK NATIONAL UNIVERSITY

Applied Nanomaterials & Devices LAB.

Electronics & Probes by Materials Engineering

연구의 즐거움; PLAY RESEARCH

HOME RESEARCH PROFESSOR MEMBER PUBLICATIONS FACILITES LECTURE PHOTO CONTACT US

2026-01

- Please visit following website for online class (https://ieilms.jbnu.ac.kr/)

- 온라인 강의 수강은 다음의 웹사이트를 이용하세요 (https://ieilms.jbnu.ac.kr/)

- **Surface Analysis of Nanomaterials (나노소재표면분석):** JBNU-KIST 산학연융합학과
  - Lecture note 1
  - Lecture note 2
  - Lecture note 3
  - Lecture note 4
  - Lecture note 5
  - Lecture note 6
- **나노소재 및 소자분석:** 신소재공학부 전자재료전공
  - Lecture note 1
  - Lecture note 2
  - Lecture note 3
  - Lecture note 4
  - Lecture note 5
  - Lecture note 6
  - Lecture note 7
  - Lecture note 8

Syllabus

Textbooks

- **Main**: Lecture notes, Related review papers and recently published hot-journal papers
- **Sub**: A Guide to materials characterization and chemical analysis /edited by John P. Sibilia. New York, N.Y. :VCH Publishers, Materials Characterization: Introduction to Microscopic and Spectroscopic Methods/ Yang Leng John Wiley & Sons.

Evaluation

- Midterm exam (30%), Final exam (40%), Homework (20%), Attendance and participation (10%)

Office hours: Anytime by e-mail appointment

Contact: twk@jbnu.ac.kr

Attendance

확진자 등 발생 시 조치방안 및 출석 인정 안내

□ 조치방안 및 출석 인정 기간

대상		조치방안	출석인정 기간
확진자		– 증상 유무에 관계없이 검체채취일로부터 7일(접종력과 무관) · 등교중지 · 7일 후 재검사 결과 음성 시 수업진행·참여 가능	검체채취일로부터 7일
접촉자	동거인	– (접종 완료자') 7일 수동감시 · 진단검사(PCR) 결과 확인 전까지 등교 중지 · 진단검사 결과 음성인 경우 수칙'을 지키며 등교 가능 * 감시기간 동안 본인 건강상태 모니터링, 조금이라도 증상이 있는 경우 검사받기, 마스크 착용하기, 다중이용시설 방문 자제 등	(접종 완료자) 진단검사(PCR) 결과 확인 전까지 출석인정 ※ 진단검사(PCR) 결과 음성일 경우 등교
		– (접종 미완료자'') 7일 공동격리 · 등교중지 · 7일 후 재검사 결과 음성 시 수업진행·참여 가능	(접종 미완료자) 검체채취일로부터 7일
	확진자와 같은 수업에 참여한 경우	– (증상이 있는 경우) 선별진료소 진단검사(PCR) 실시 – (증상이 없는 경우) 신속항원검사(개인용) 실시 · 신속항원검사(개인용) 결과 음성이면 등교 가능 양성인 경우, 진단검사(PCR)를 실시하고 검사결과 확인 전까지 등교중지 ※ 검사결과 음성이라도, 마스크 착용 등 방역수칙 반드시 준수	①의심증상이 있거나 ②신속항원검사(개인용) 결과 양성인 경우, PCR검사를 실시하고 검사결과 확인 전까지 출석인정
해외 입국자			입국 후 7일(자율 격리기간)
백신 접종	접종당일		접종당일
	접종 다음날		접종에 따른 이상반응 발생 시 의사 소견서 등을 참고 하여 대학(원)장이 출석인정 기간 결정

□ 출석인정 절차: 해당 학생이 출석인정원(별도서식)과 증빙서류를 소속 학과에 제출 → 학장 허가 → 출석인정허가서를 교과목 담당교원에게 제출
(증빙서류: 예방접종증명서, 진단서, 진료확인서, 처방전, 출입국사실증명서, 항공기 탑승권 등)

- 2회 결석까지 인정, 출석 부를 때 자리에 있어야 함.
- 3회 지각은 1회 결석으로 생각
- 코로나바이러스 등의 이유로 결석시 메일 필수 (문자 캡쳐)

Detailed Lecture Plan

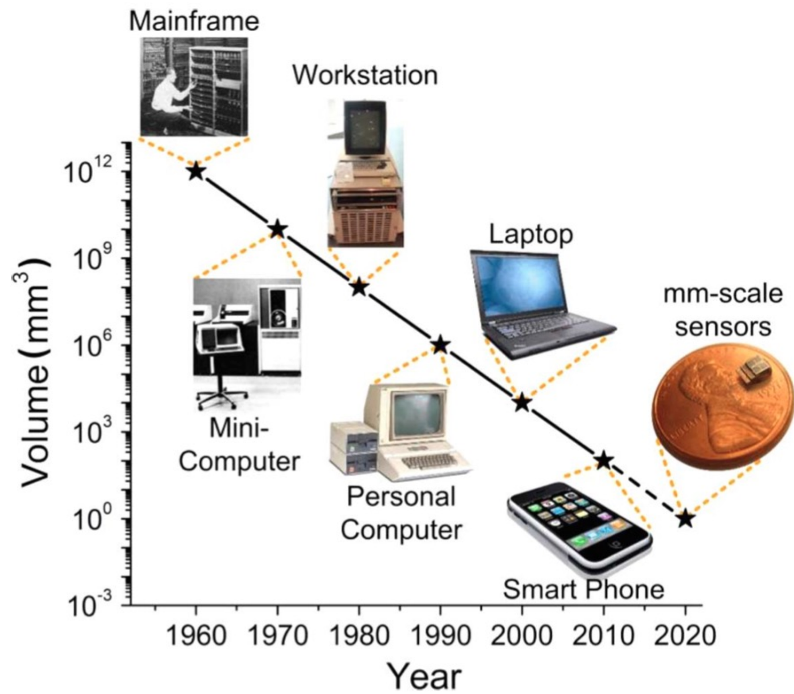
- **1st week**: Introduction to instrumental analysis
- **2nd-3rd weeks**: SPM (STM, AFM, EFM)
- **4th-5th weeks**: x-ray diffraction and x-ray fluorescence
- **6th-7th weeks**: SEM (image, chemical analysis: EDS & WDS)
- **8th week**: mid-term exam
- **9th week**: TEM (image, chemical analysis: EDS & EELS, diffraction analysis)
- **10th-11th weeks**: surface analysis I (electron spectroscopy: AES, XPS)
- **11th-12th weeks**: surface analysis II (ion spectroscopy: RBS, SIMS)
- **13-14th weeks**: optical characterization (UV-VIS spectroscopy, PL, Raman spectroscopy, FTIR)
- **15th week**: final exam

Introduction-Importance of Characterization

NOBEL PRIZE Awards

- Physics 1914, Max von Laue: X-ray diffraction
- Chemistry 1922, Francis Aston: Mass-spectroscopic separation of isotopes ("SIMS")
- Physics 1924, Manne Siegbahn: X-ray spectroscopy
- Physics 1930, Venkata Raman: The scattering of light, Raman-effect
- Physics 1981, Kai Siegbahn: HR photoelectron (ESCA)
- Physics 1986, G. Binnig, H. Rohrer: Scanning tunneling microscopy
- Physics 1986, Ernst Ruska: Electron Microscopy
- Chemistry 1991 Richard Ernst: Development of NMR spectroscopy
- Physics 1994, Clifford Shull: Neutron diffraction

Introduction: Technology Development



Phone call

message

Web surfing

Email

camera

Music, movie

Health care

…

Introduction: More from Less

"**More from less**, for more and more"

Moore's Law – The number of transistors on integrated circuit chips (1971-2018)

Our World in Data

Moore's law describes the empirical regularity that the number of transistors on integrated circuits doubles approximately every two years. This advancement is important as other aspects of technological progress – such as processing speed or the price of electronic products – are linked to Moore's law.

Transistor count

Data source: Wikipedia (https://en.wikipedia.org/wiki/Transistor_count)
The data visualization is available at OurWorldinData.org. There you find more visualizations and research on this topic.

Licensed under CC-BY-SA by the author Max Roser.

Introduction

The First Transistor – December 23, 1947

Walter Brattain & John Bardeen's pnp point-contact germanium transistor operated as a speech amplifier with a power gain of 18 on December 23, 1947.

In January23 1948 William Shockley documented his idea for the Junction Transistor.

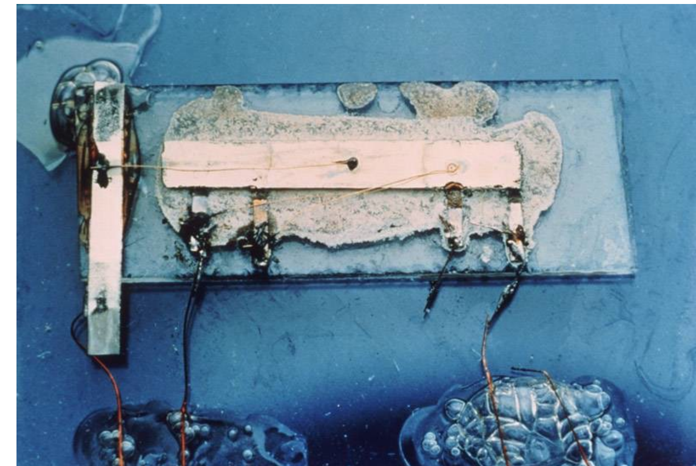
1956 Nobel Prize for Physics to William Shockley, John Bardeen & Walter Brattain for "Discovery of the Transistor Effect."

Jack Kilby (source: TI Website) – Integrated Circuit

- Jack Kilby arrived at TI (Dallas) in May 1958.
- Kilby's lab book for July 24, 1958 contain the following":

"The following circuit elements could be made on a single slice: resistors, capacitors, distributed capacitor, transistor."

- Kilby demonstrated 1st integrated circuit on September 12, 1958, and filed for patent in February 1959.
- **Awarded Nobel Prize for Physics 2000**



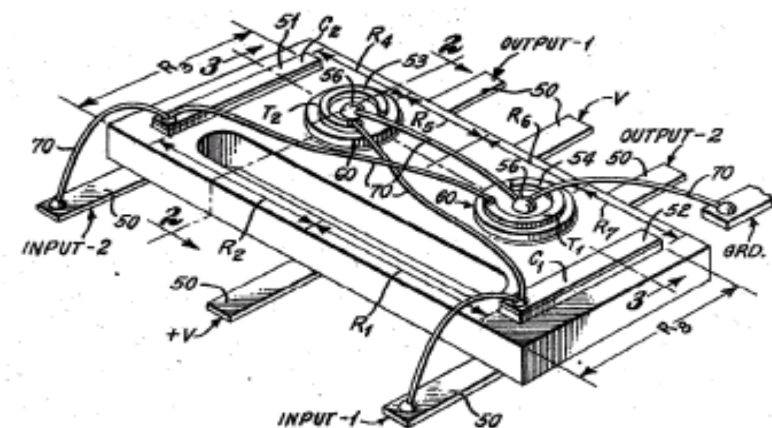
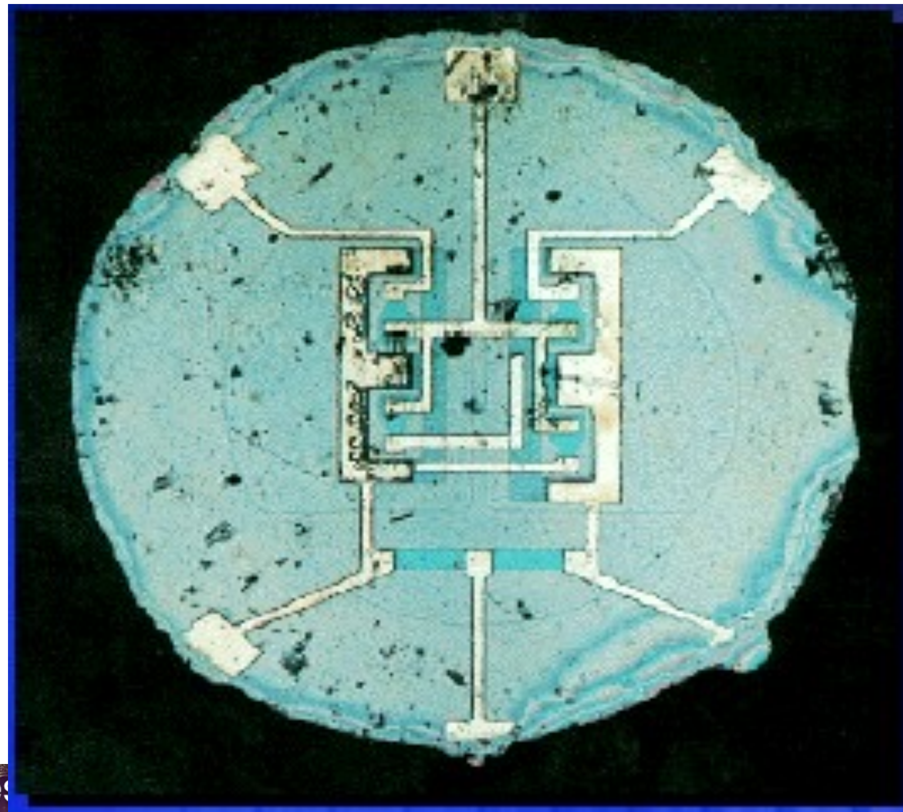


Figure from Jack Kilby's US Patent

Introduction

1st Planar Integrated Circuit (Robert Noyce) 1961

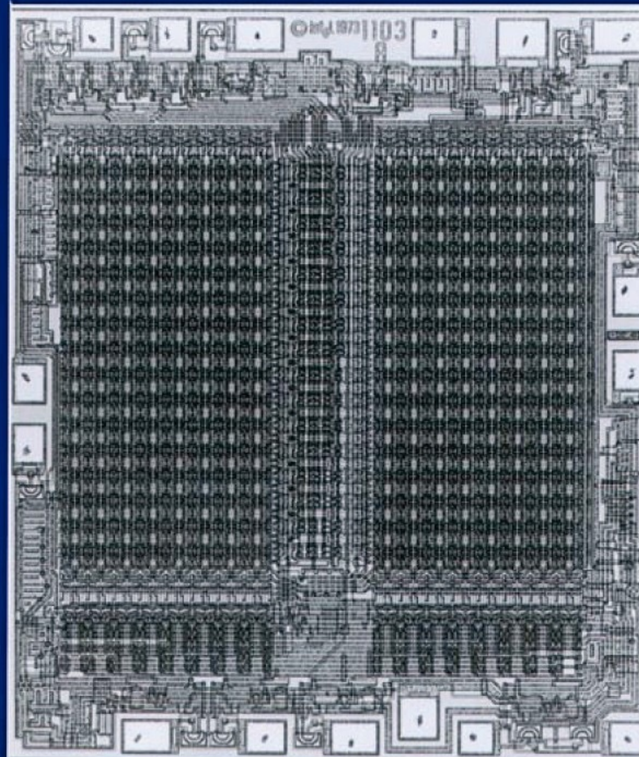
It is thin film interconnect! (at Fairchild)



Introduction

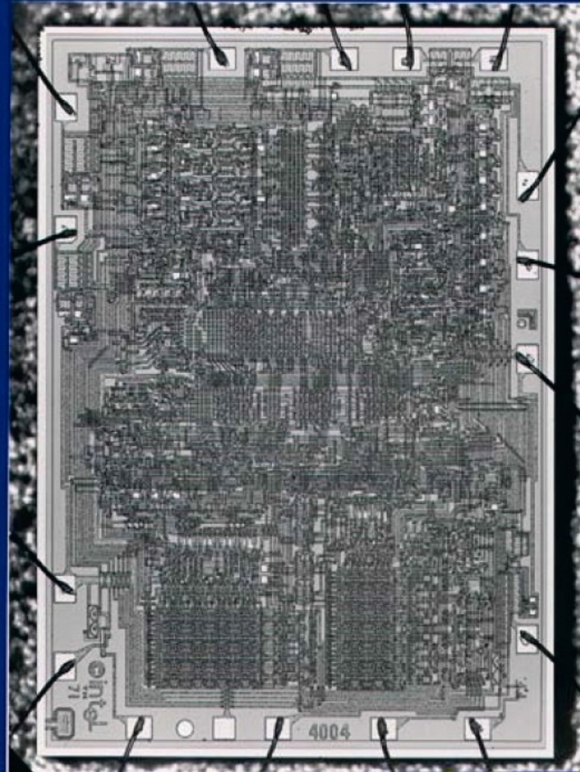
FIRST DRAM

(1103 by Intel 1970)



Introduction

FIRST MICROPROCESSOR
(4004 by Intel 1971)



2300 transistors

60kHz clock speed

Moore's Law enables a significant fraction of all human effort:

•Digital Content & Services
•>>$3-4 Trillion

Software & Services
$3-4 Trillion

Electronic Systems
-$1 Trillion

SemiConductor Chips
$280 Billion

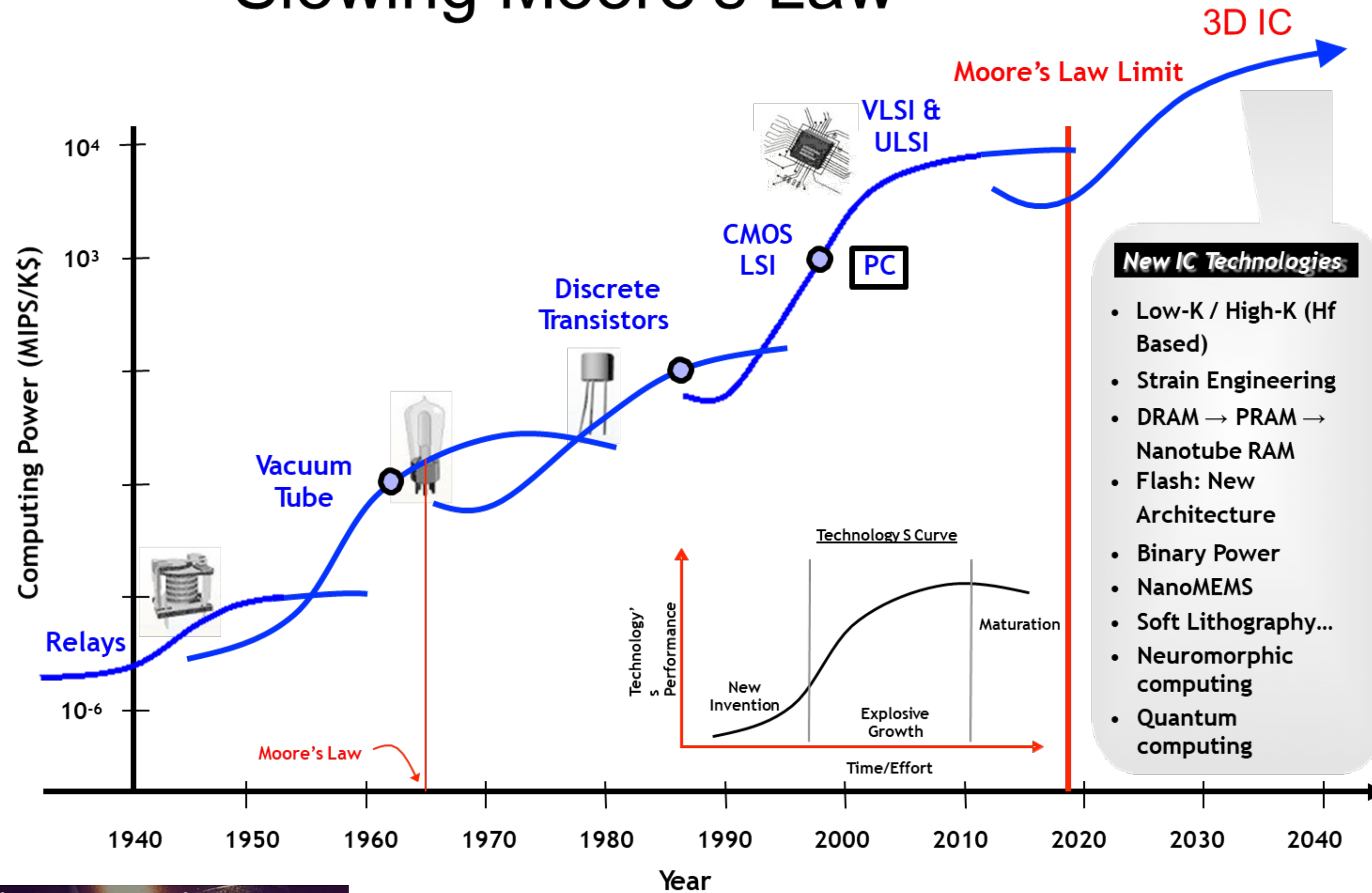
Research &
Development

The Technology Pyramid:

The future

- The major development in the near future is consumer electronic products. The need of nanoelectronic devices will grow.
- Continuous R&D efforts to extended electronics.
- 3D IC by closer integration of Si technology and packaging technology.
- Heterogeneous integration of other technology with Si, such as opto-electronics and MEMS/NEMS and nanomaterials.

Introduction



Introduction

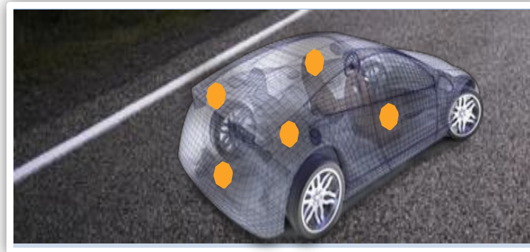
The Remote Control For Your Life

IN THE FUTURE YOUR MOBILE PHONE WILL ACT AS YOUR DIGITAL “6TH SENSE”

Internet of Things (IoT)

EVERYTHING AROUND US IS BECOMING INTELLIGENT & CONNECTED

Automotive



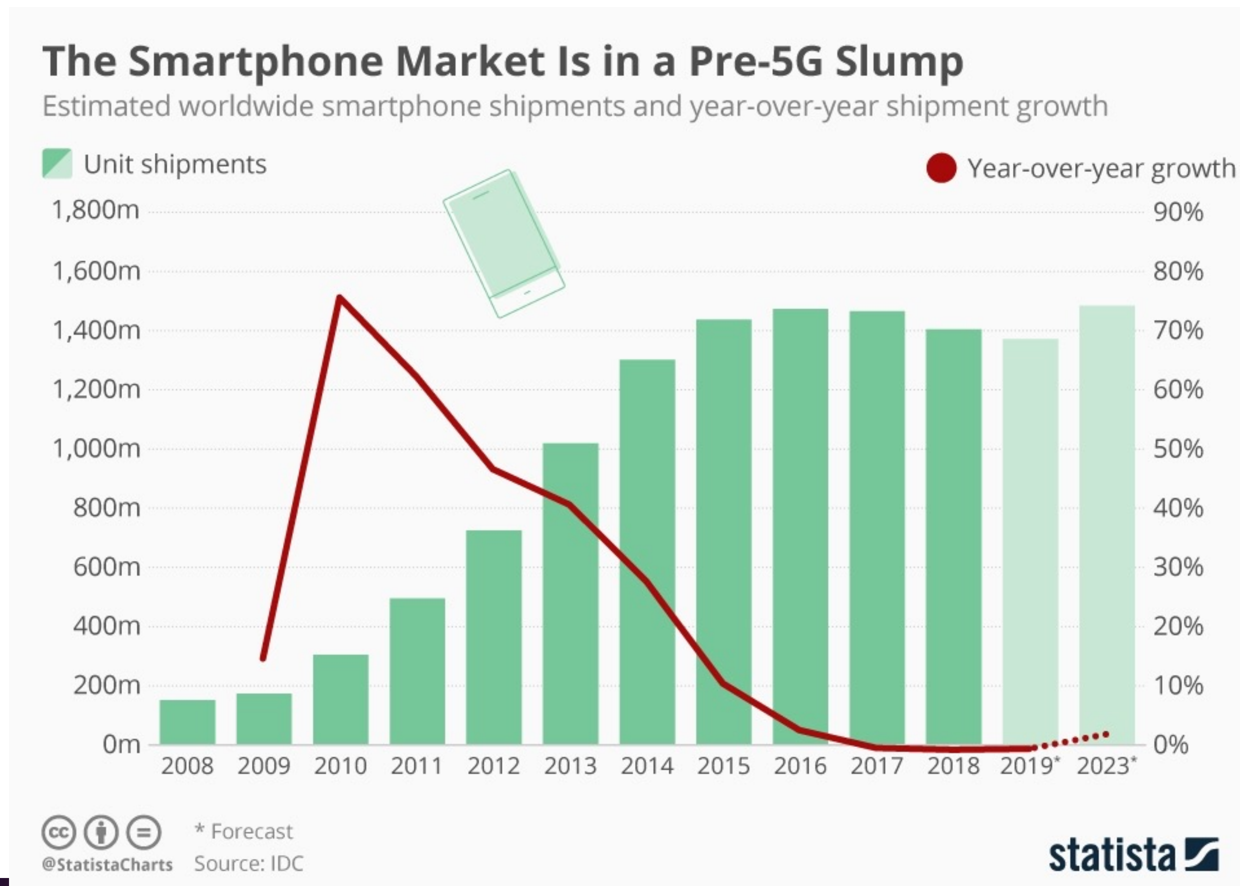
Industrial

Health & Fitness

Home

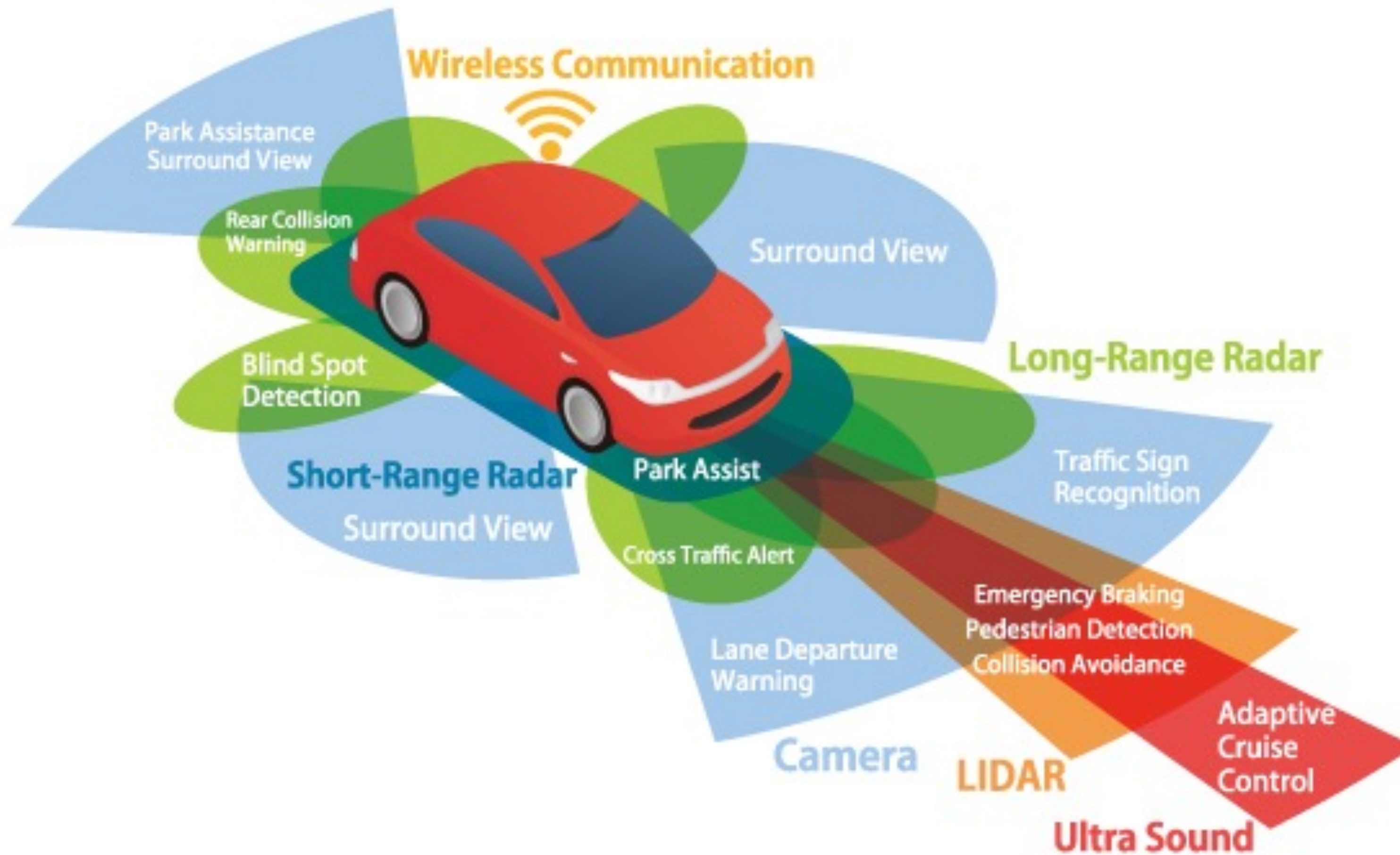
Increasing Machine-to-Machine (M2M) Communication

Continued Smartphone Momentum



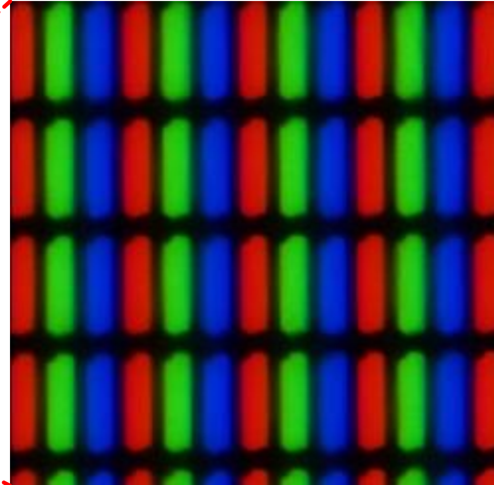
Introduction

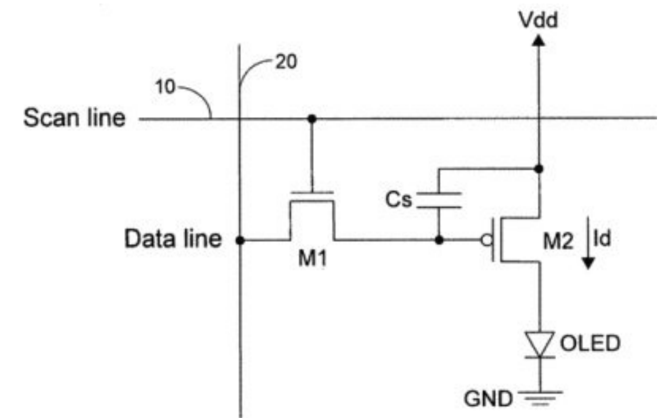
New market through self-driving car industry

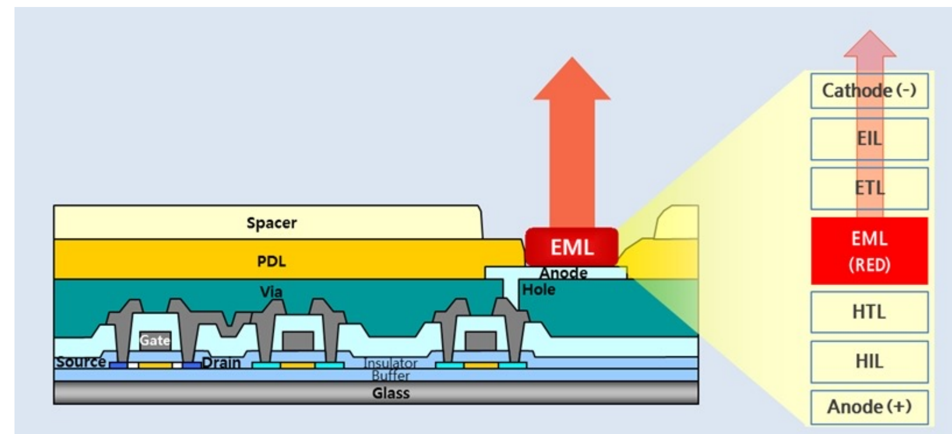


Introduction: Importance of Nanomaterials/Devices

iPhone 12



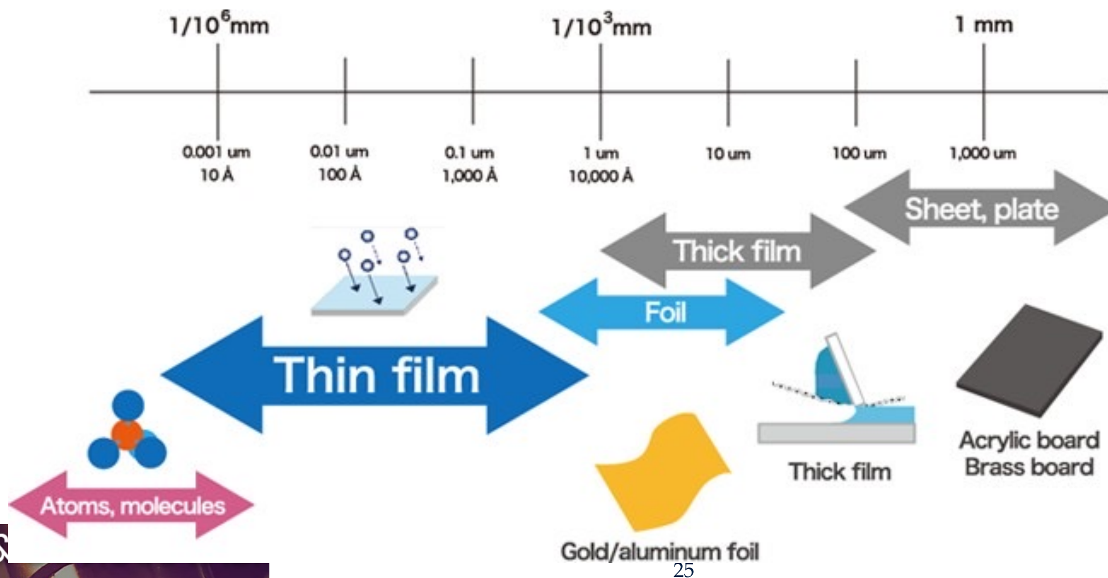




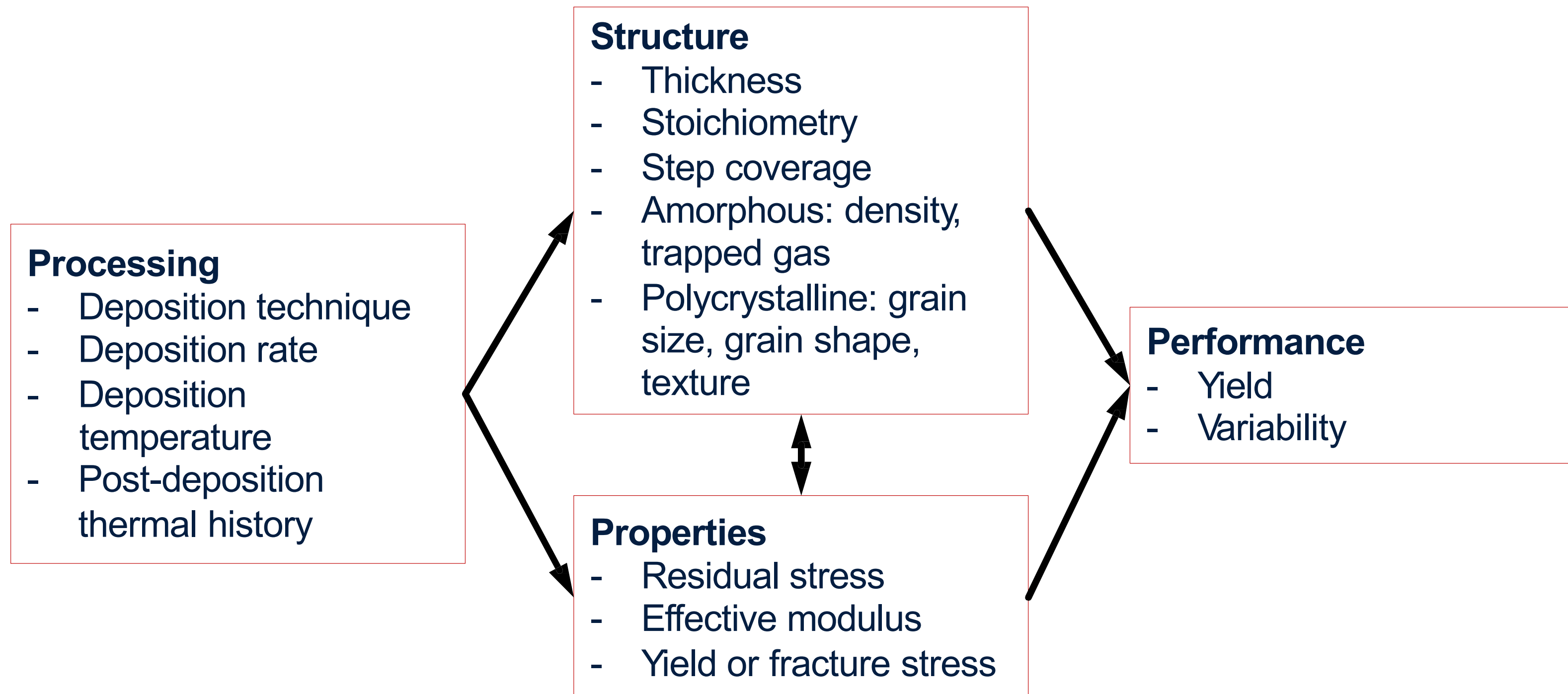
Thin-film

Thin film: a layer of material ranging from fractions of a nanometer (monolayer) to several micrometers in thickness

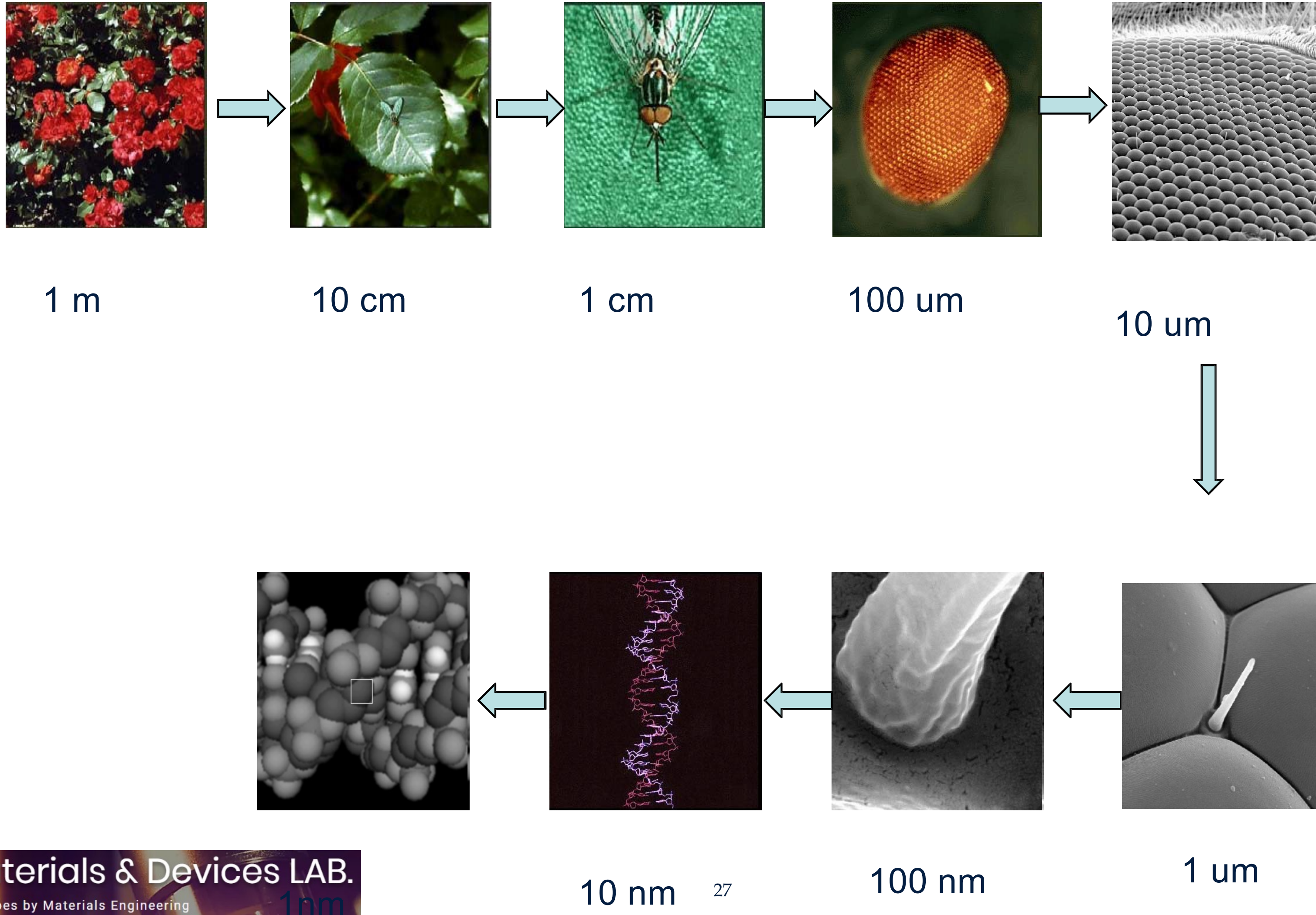
Thin-film deposition: the controlled synthesis of materials as thin films, which is a fundamental step in many applications



Structure and properties of thin-films

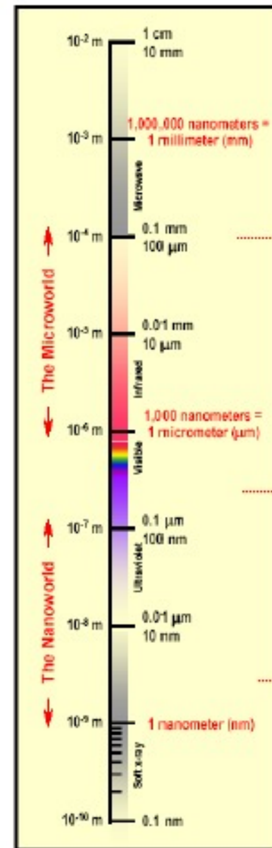


Nanomaterials: What is nano?

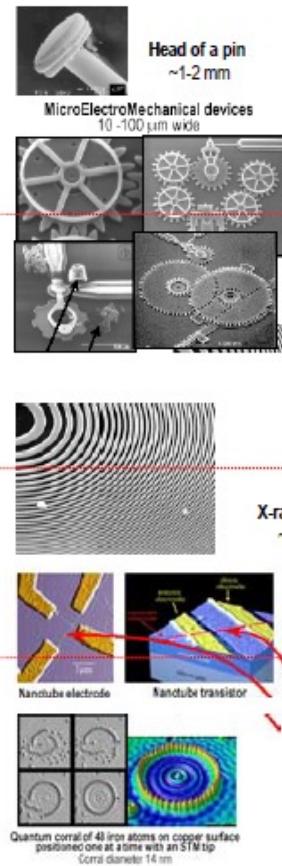


Scale of Things

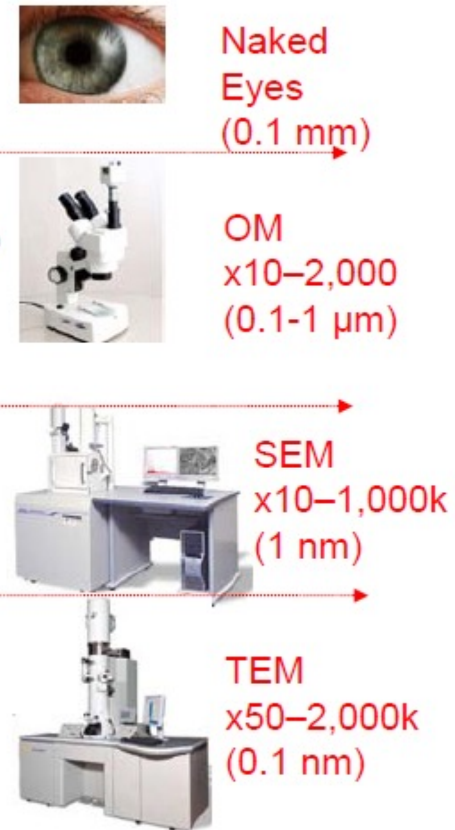
Things Natural



Things manmade

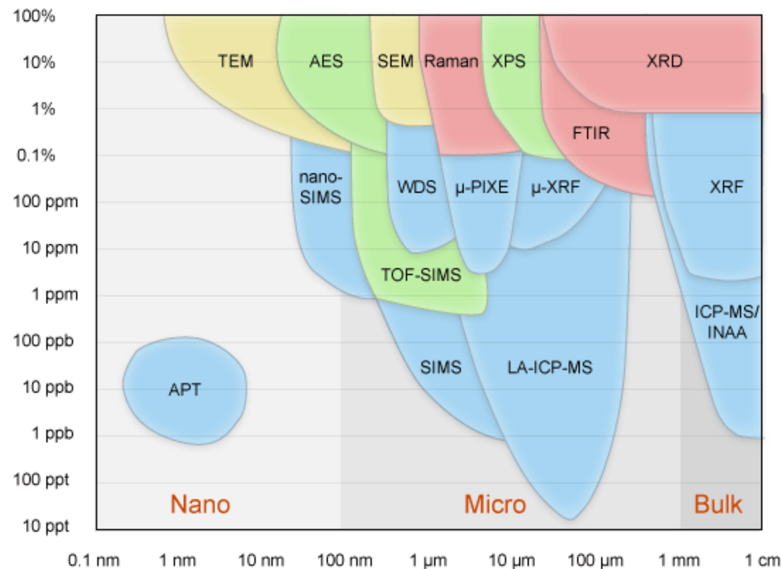


Observation Tools

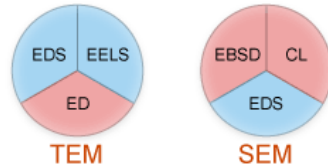


Material Analysis Techniques

Resolution vs sensitivity diagram

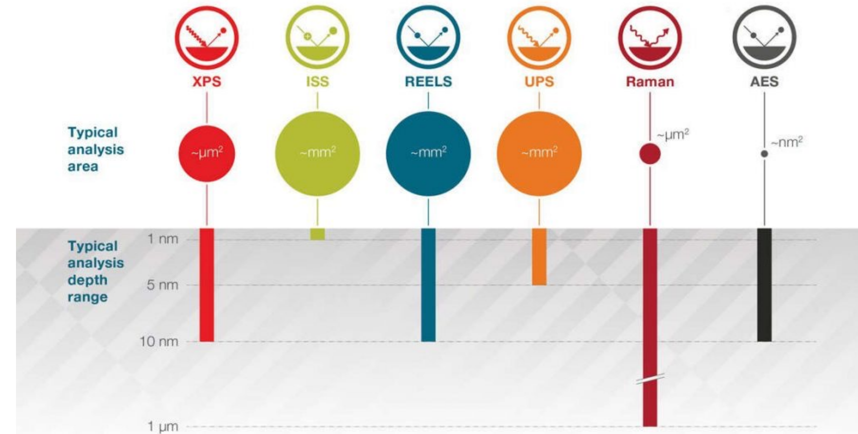


- Elemental composition
- Structural information
- Surface and thin film analysis
- SEM and TEM host multiple techniques



- The most important considerations are spatial resolution and the detection limit.

Surface analysis techniques



Measurement technique	Energy range	Primary beam and secondary signal	Application
SEM	0.3 - 30KeV	Electron - Electron	Surface morphology
TEM	100 - 400KeV	Electron – Electron	High resolution structure
AES	500eV - 10KeV	Electron – Electron	Surface layer composition
LEED	20 - 200eV	Electron – Electron	Surface structure
EMP (EDX)	1 - 30KeV	Electron – X-Ray	Surface region composition
STEM	100 - 400KeV	Electron – Electron	X-ray analysis, imaging
EELS	100 - 400KeV	Electron – Electron	Local small area composition
SNMS	1 - 15eV	Ion - Atom	Trace composition vs depth
ISS	0.5 – 2KeV	Ion - Ion	Surface composition
PIXE	1KeV	Ion - X-Ray	Trace composition
SIMS	1 – 15KeV	Ion - Ion	Trace composition vs depth
SIM	5 – 20KeV	Photon - Electron	Surface characterization
XPS	> 1KeV	Photon - Electron	Surface composition
XRD	> 1KeV	Photon – X-Ray	Crystal structure
RBS	> 1MeV	Photon - Ion	Composition vs depth
XRF	> 1KeV	Photon - X-Ray	Composition (1μm)

****Abbreviation:*** *SEM- Scanning Electron Microscopy, TEM- Transmission Electron Microscopy, AES- Auger Electron Spectroscopy, LEED- Low-Energy Electron Diffraction, EMP- Electron Microprobe, STEM- Scanning TEM, EELS- Electron Energy Loss Spectroscopy, SNMS- Secondary Neutral Mass Spectroscopy, ISS- Ion-Scattering Spectroscopy, PIXE- Particle-Induced X-Ray Emission, SIMS- Secondary Ion-Mass Spectroscopy, SIM- Scanning Ion Microscopy, XPS- X-Ray Photo Electron Spectroscopy, XRD- X-Ray Diffraction, RBS- Rutherford Backscattering, XRF- X-Ray Fluorescence* [**86**].

Surface Analysis of Nanomaterials

		Output signal (emission)		
		electron	photon ($h\nu$)	particle
Input source (excitation)	electron	AES, EELS, HREELS, LEED, RHEED, SEM, TEM	EDS, WDS, APS	ESD
	photon ($h\nu$)	XPS (ESCA), UPS	XRF, FTIR, XRD	PSD
	Ion	INS	PIXE	SIMS, RBS, ISS
	energy	FEM, STM		FIM

- **Analytical techniques**

(1) Electron Microscopy : SEM, TEM, STEM, FEM

(2) Diffraction : LEED, RHEED, XRD, TEM

(3) Analytical methods using electron beams : AES, SAM, EPMA(XMA), EELS, HREELS, LEED, RHEED, SEM, TEM

(4) Analytical methods using photons : XPS(ESCA), UPS, FTIR, XRD, XRF

(5) Analytical methods using ion beams : SIMS, RBS, ISS

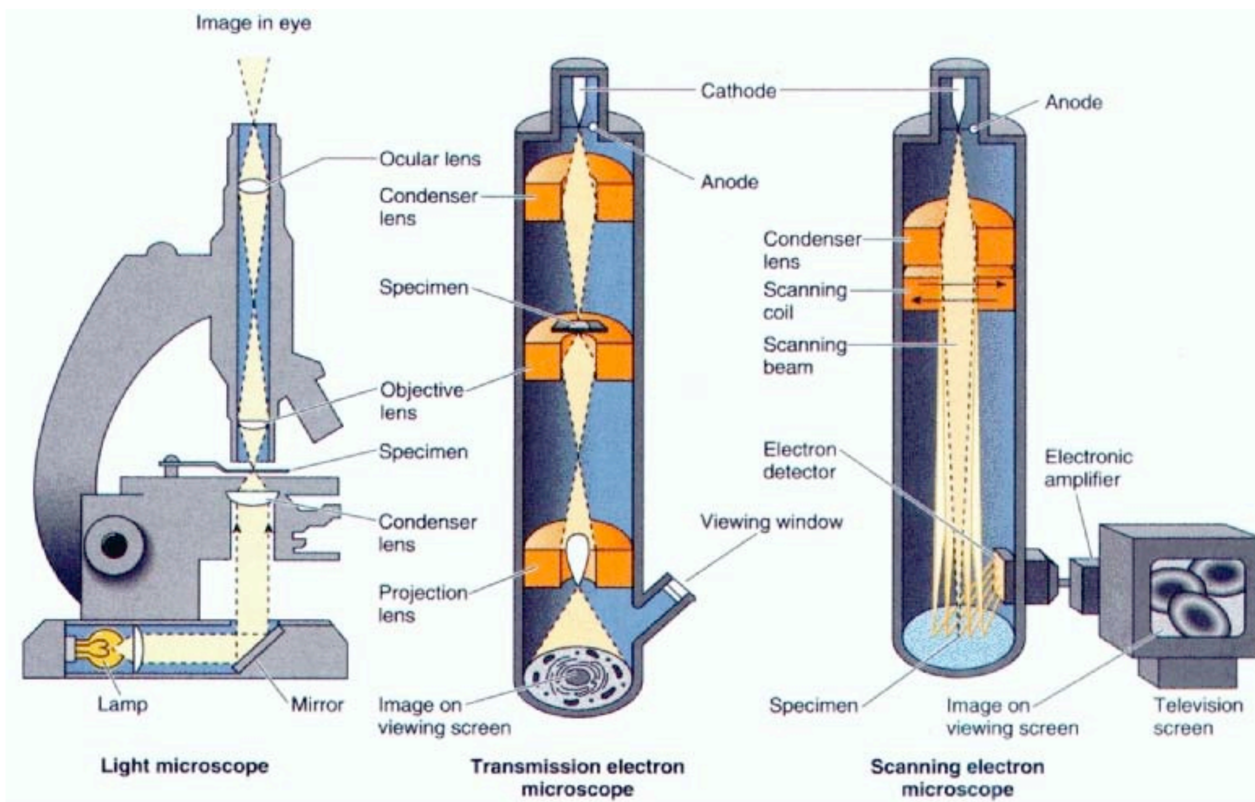
Abbreviations of surface analysis techniques	
AEM	Analytical Electron Microscopy
AES (SAM)	Auger Electron Spectroscopy (Scanning Auger Microscopy)
APS	Appearance Potential Spectroscopy
ARXPS	Angle-Resolved X-ray Photoelectron Spectroscopy
EDS, EDX	Energy Dispersive X-ray Spectroscopy
EELS	Electron Energy Loss Spectroscopy
EPMA	Electron Probe Micro-analysis (=XMA), EDS & WDS
ESCA	Electron Spectroscopy for Chemical Analysis (=XPS)
ESD	Electron Stimulated Desorption
EXAFS	Extended X-ray Absorption Fine Structure
FEM	Field Emission Microscopy
FIM	Field Ion Microscopy
HREELS	High Resolution Electron Energy Loss Spectroscopy
ISS	Ion Scattering Spectroscopy
INS	Ion Neutralization Spectroscopy
PIXE	Particle Induced X-ray Emission
PL	Photoluminescence Spectroscopy
PSD	Photon Stimulated Desorption
RAMAN	Raman Spectroscopy
RBS	Rutherford Backscattering Spectroscopy
RHEED	Reflection High Energy Electron Diffraction
SEM	Scanning Electron Microscopy
SIMS	Secondary Ion Mass Spectroscopy
STM	Scanning Tunneling Microscopy
TEM	Transmission Electron Microscopy
UPS	Ultra-violet Photoelectron Spectroscopy
WDS	Wavelength Dispersive X-ray Spectroscopy
XPS	X-ray Photoelectron Spectroscopy (=ESCA)
XRD	X-ray Diffraction
XRF	X-ray Fluorescence
XMA	X-ray Microanalysis (=EPMA), EDS & WDS

Comparison of Microscopy

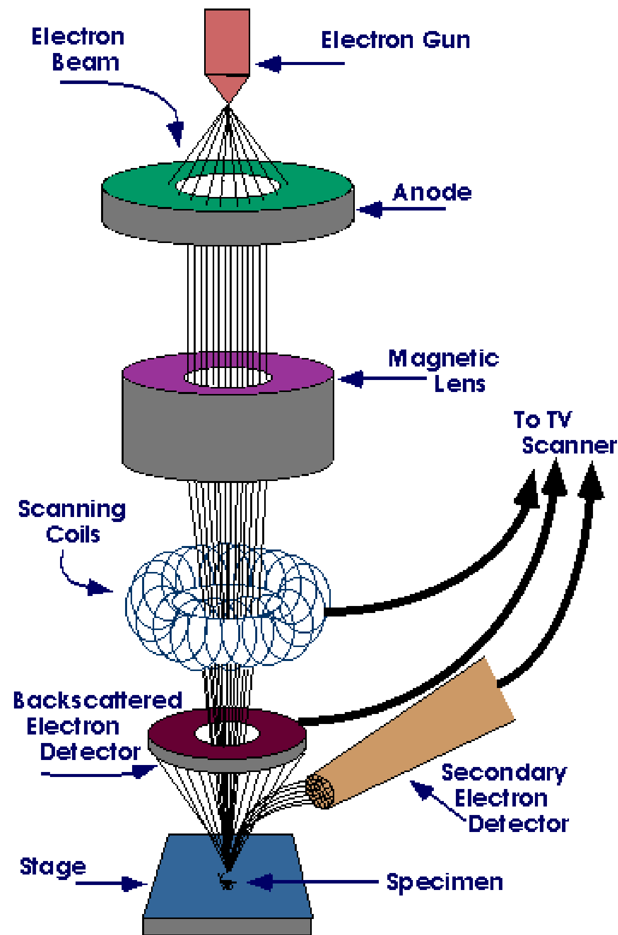
OM (Optical Microscopy)

TEM (Transmission Electron Microscopy)

SEM (Scanning Electron Microscopy)



SEM (Scanning Electron Microscopy)

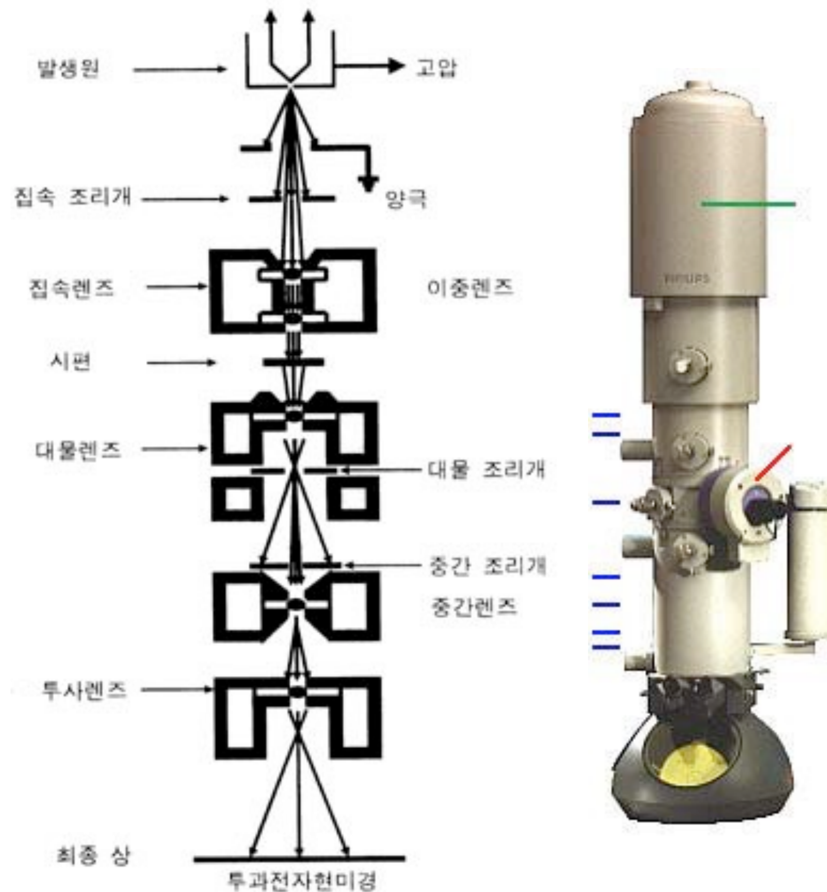


- A type of electron microscope capable of producing high resolution images of a sample surface
- X-ray microanalysis (composition analysis): X-rays, which are produced by the interaction of electrons with the atoms in the sample, may be detected in an SEM equipped

- Energy-dispersive X-ray spectroscopy (EDS, EDX, EDXS)

- Wavelength dispersive X-ray spectroscopy (WDS, WDX, WDXS).

TEM (Transmission Electron Microscopy)



An imaging technique

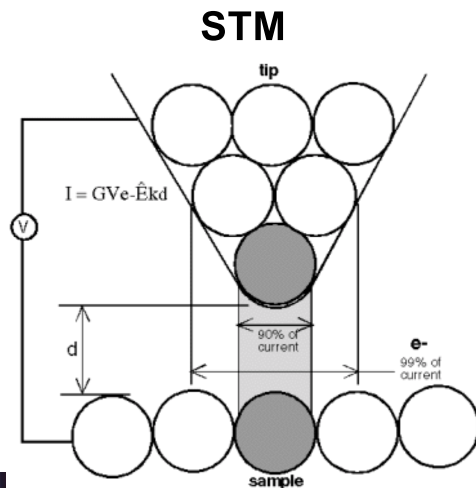
- A beam of electrons (100~1000KeV) is transmitted through a specimen
- an image is formed, magnified
- directed to appear either on a fluorescent screen or photographic film, or to be detected by CCD camera.

미세한 결정결함, 결정구조, 결정입자의 크기, 원자 **Lattice**를 관찰

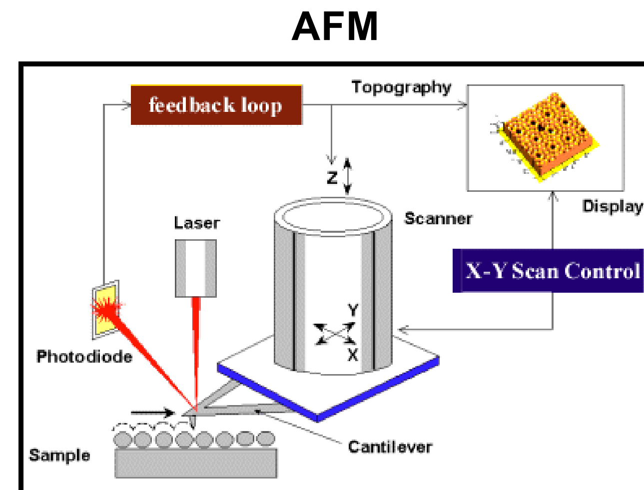
SPM (Scanning Probe Microscope)

- Scanning Tunneling Microscopy(STM): topography, local DOS
- Atomic Force Microscopy (AFM): topography, force measurement
- Lateral Force Microscopy (LFM): friction
- Magnetic Force Microscopy (MFM): magnetism
- Electrostatic Force Microscopy (EFM): charge distribution
- Near-field Scanning Optical Microscopy (NSOM): optical properties with spatial resolution (R) smaller than wavelength of light (R ~50nm)

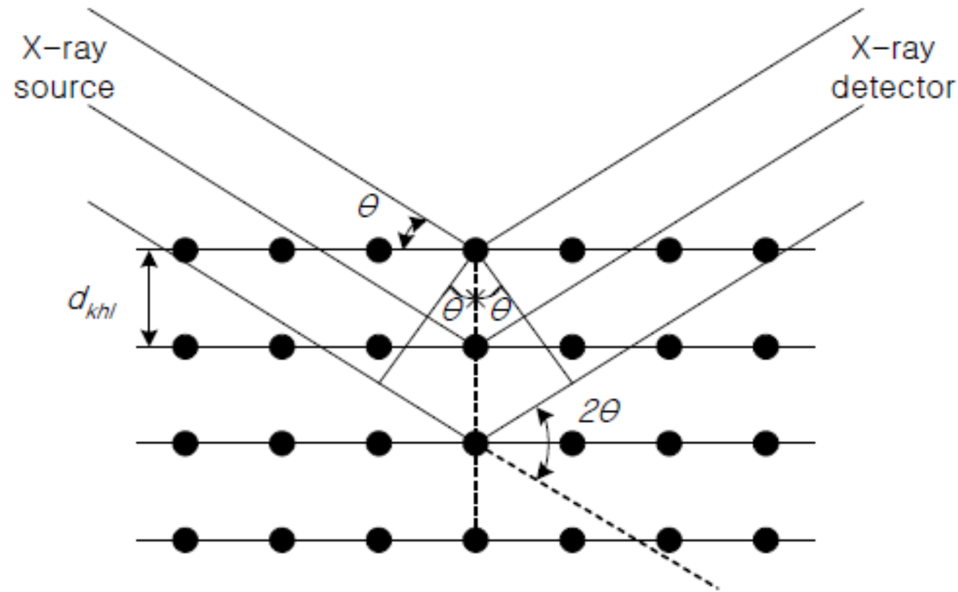
STM



AFM



XRD (X-ray Diffraction)



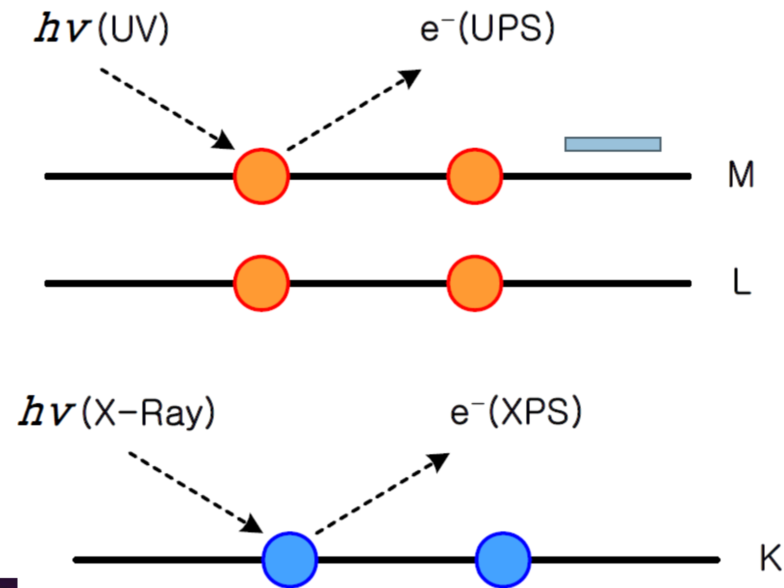
$$2d\,\sin\theta = n\lambda$$
$$(n=1, 2, 3, \ldots)$$

- 결정면에 X-선을 주사하여 일어나는 회절현상을 이용하여 결정구조를 분석.
- 결정구조 분석, 박막구조 해석, 결정결함 분포

ESCA (Electron Spectroscopy for Chemical Analysis)

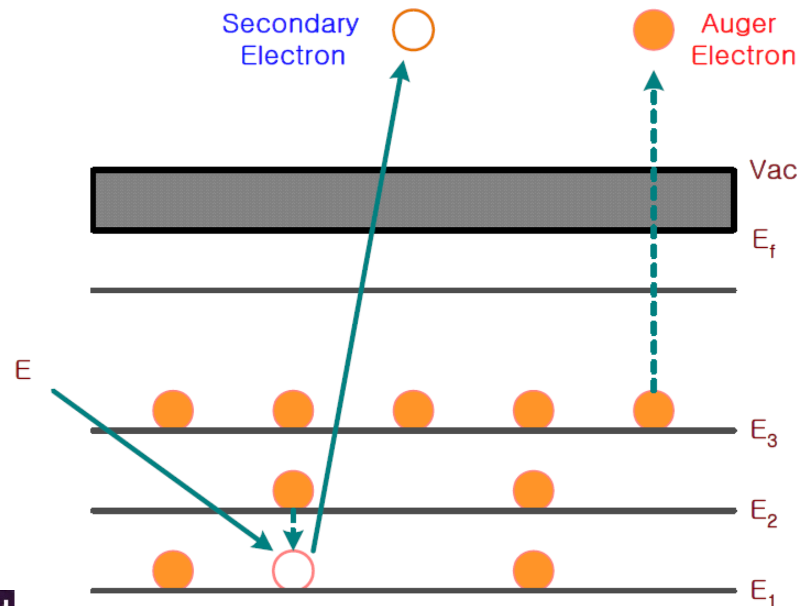
Electron spectroscopy for chemical analysis (ESCA)

- X-ray photoelectron spectroscopy (XPS): The most common type is based upon the irradiation of the sample surface with monochromatic X-radiation.
- Ultraviolet photoelectron spectroscopy (UPS): A monochromatic beam of ultraviolet radiation causes the ejection of electrons form the analyte.



AES (Auger Electron Spectroscopy)

- Auger electron spectroscopy (AES) is a surface-sensitive spectroscopic technique used for elemental analysis of surfaces.
- Underlying the spectroscopic technique is the Auger effect, as it has come to be called, which is based on the analysis of energetic electrons emitted from an excited atom after a series of internal relaxation events.



Analysis Approaches: Input-Output

Input rad.

Output detected

	Electron	Ions	Photons	X-rays
Electron	AES, SEM, TEM			XPS
Ions	ESD	SIMS, RBS, FIB		
Photons			RL, UV/VIS, Raman	
X-rays	EDS			XRD, XRF

AES: Auger Electron Spectroscopy
SEM: Scanning Electron Microscopy
TEM: Transmittance Electron Microscopy
SIMS: Secondary Ion Mass Spectroscopy
RBS: Rutherford Backscattering Spectroscopy
XPS: X-ray Photoelectron Spectroscopy

XRD : X-ray diffraction
XRF : X-ray fluorescence
EDS : Energy Dispersive x-ray Spectroscopy
PL : Photoluminescence
UV/VIS-spectroscopy
Raman-spectroscopy

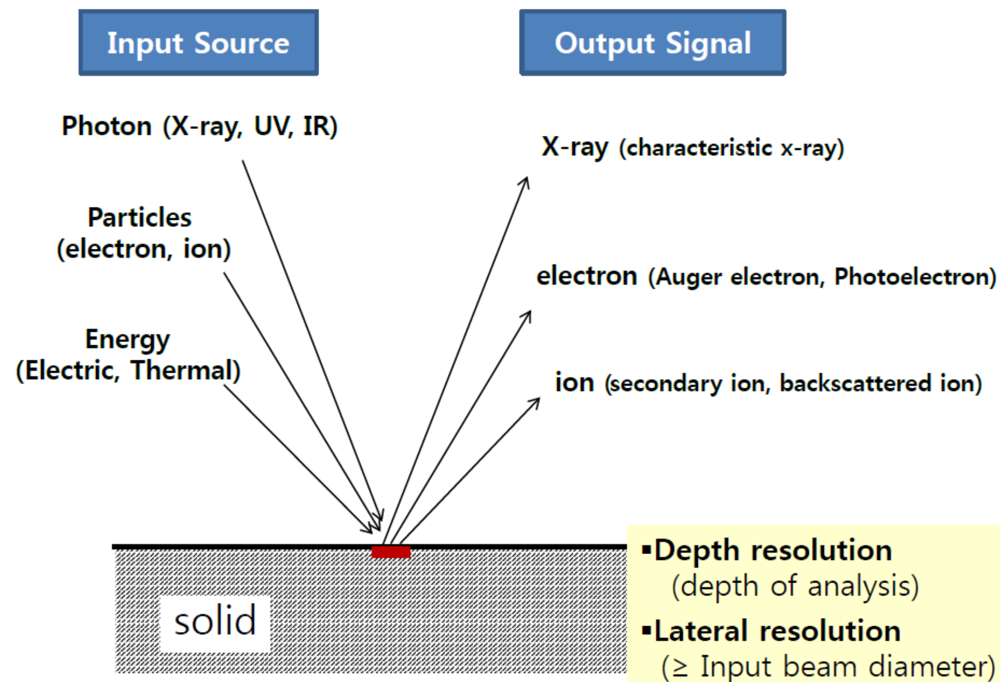
Some Important Techniques for Surface Analyses

Technique		Input source	Output signal	Depth resolution	Lateral resolution	Trace capability	Vacuum
	EDS, WDS	Electron	X-ray	0.1~5 μm	0.1~5 μm	0.1% 0.01%	HV
	XRF	X-ray	X-ray	10~100μm	~1 mm	0.01~0.1%	Air
Surface spectroscopy	AES	Electron	Electron (Auger electron)	0.5~5 μm	0.005~1μm	0.1~1%	UHV
Surface spectroscopy	XPS, ESCA	X-ray	Electron (Photoelectron)	0.5~5 μm	~1 mm micro~3μm	0.1%	UHV
Surface spectroscopy	SIMS	Ion	Ion (secondary ion)	0.3~3 nm	~0.1 μm	ppb~a few %	UHV
Surface spectroscopy	RBS	He^+	He	1~20 nm	~1 mm micro~1μm	0.1~10%	HV

- **Input source is related with lateral resolution**
- **Output signal is related with depth resolution**

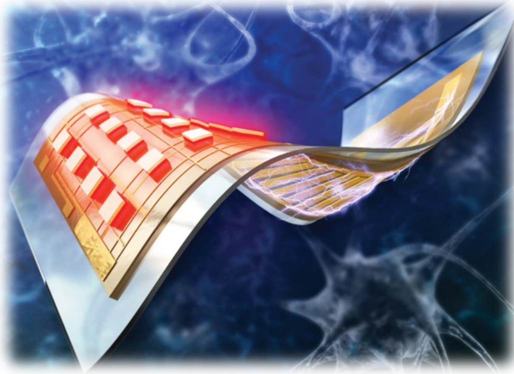
New Tools for Nanoscale Characterizations

- Structural analysis: SEM, TEM, XRD, SPM
- Chemical analysis: AES, XPS, SIMS, EDS, SPM
- Electronic, optical analysis: UV/VIS spectroscopy, PL, SPM
- Magnetic analysis: *SQUID, SPM



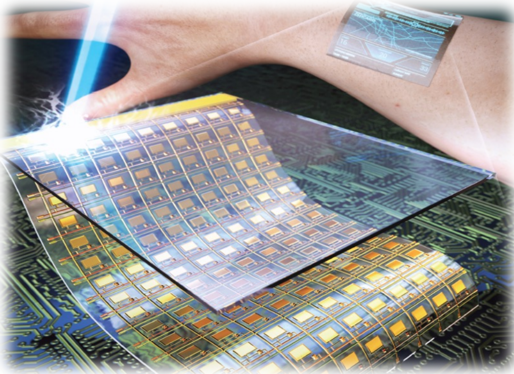
Material Analyses in Recent Researches

1. III-V/III-N-Based Flexible MicroLEDs

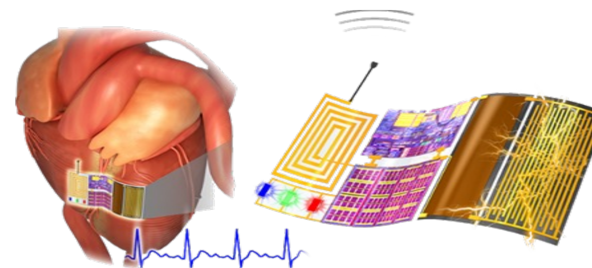


2. Micro/Nano-fabrication

3. Flexible/Wearable Electronics

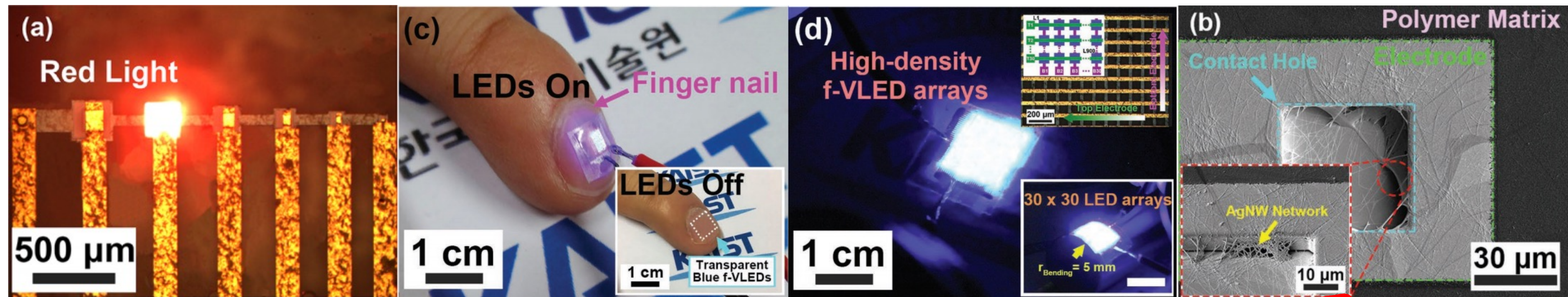


4. Biomedical Applications

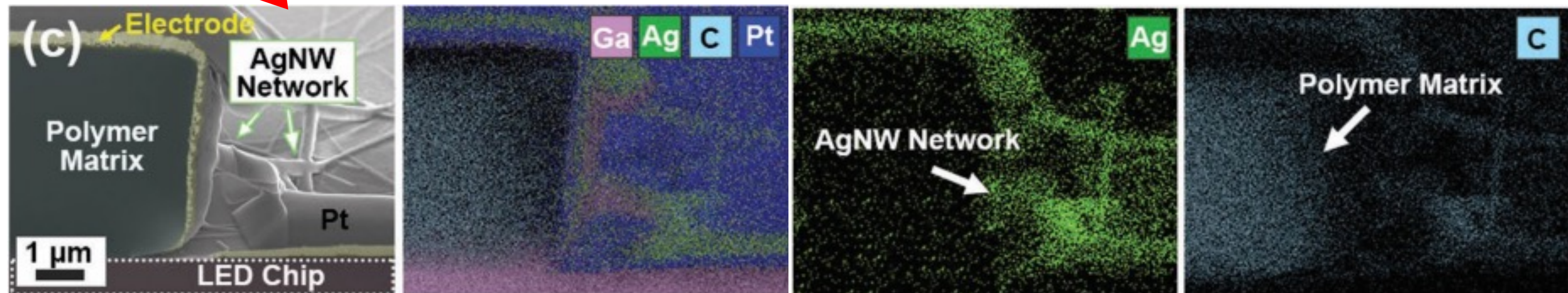


Material Analyses in Recent Researches

Flexible MicroLED (III-N/III-V)



Cross-sectional

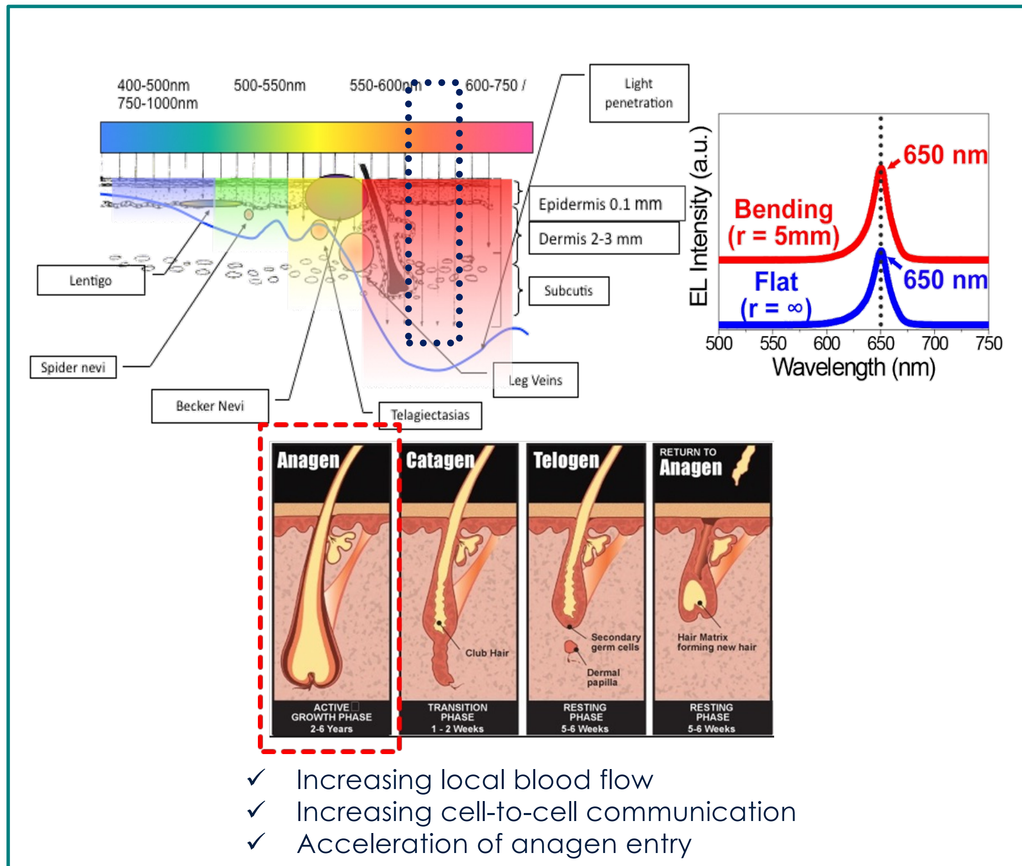


EDX
(Energy Dispersive x-ray Spectroscopy)

Lee et al., *ACS nano* 12.9 (2018): 9587-9595.
Lee et al., *Advanced Materials* 30.28 (2018): 1800649.

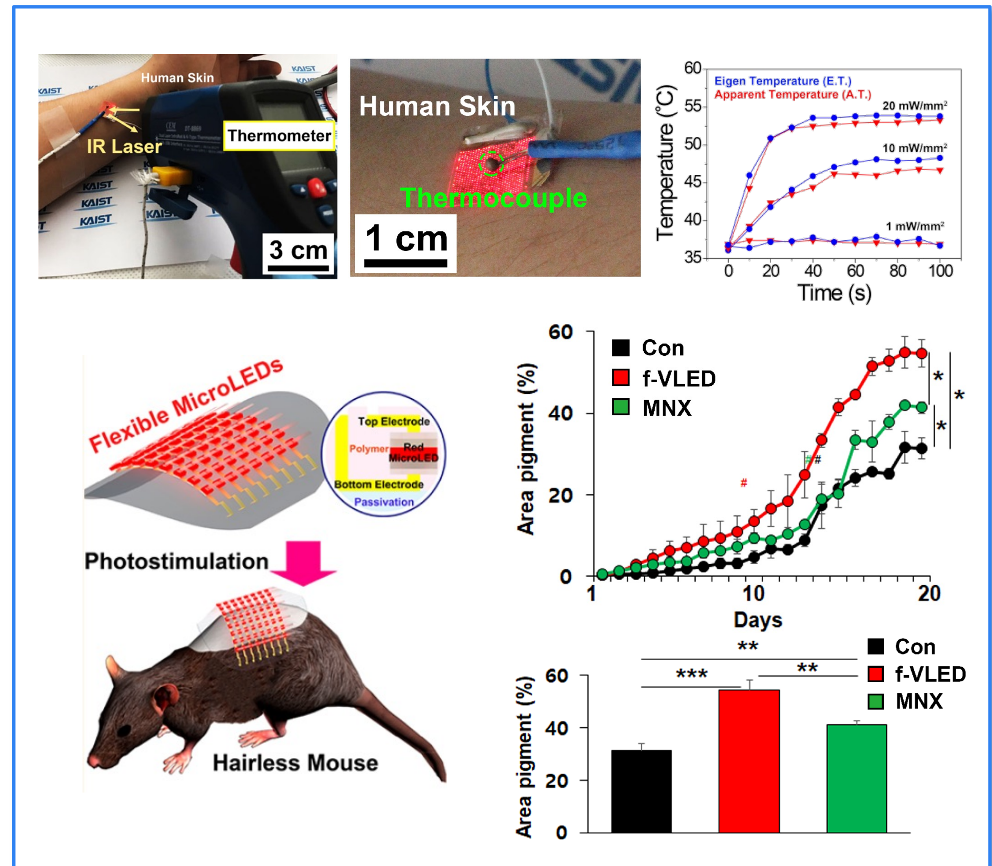
Flexible Vertical MicroLED for Hair-Regrowth

Photostimulation



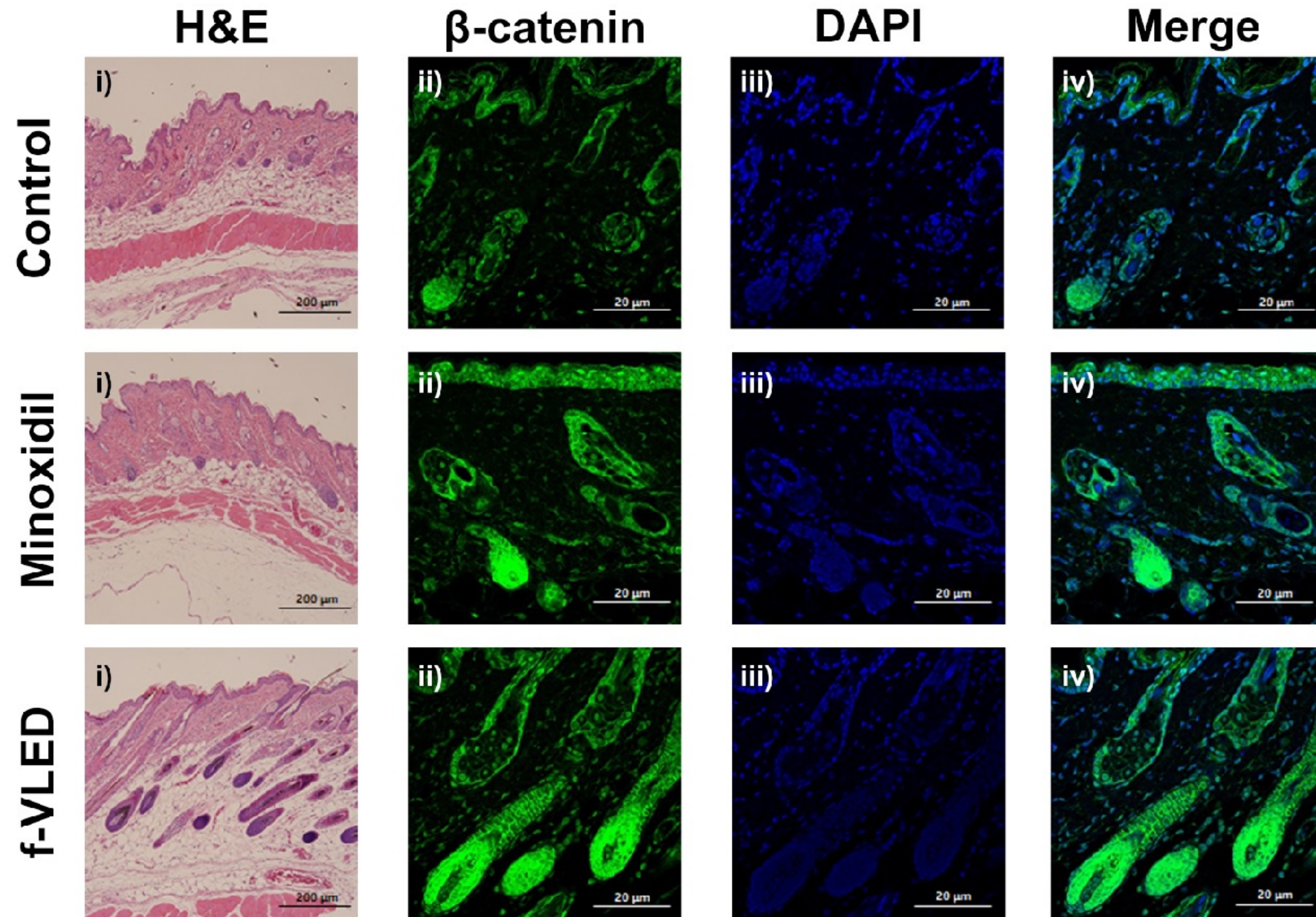
- ✓ Increasing local blood flow
- ✓ Increasing cell-to-cell communication
- ✓ Acceleration of anagen entry

Hair-Regrowth by f-VLED



Lee et al., ***ACS nano*** *12.9 (2018): 9587-9595.*

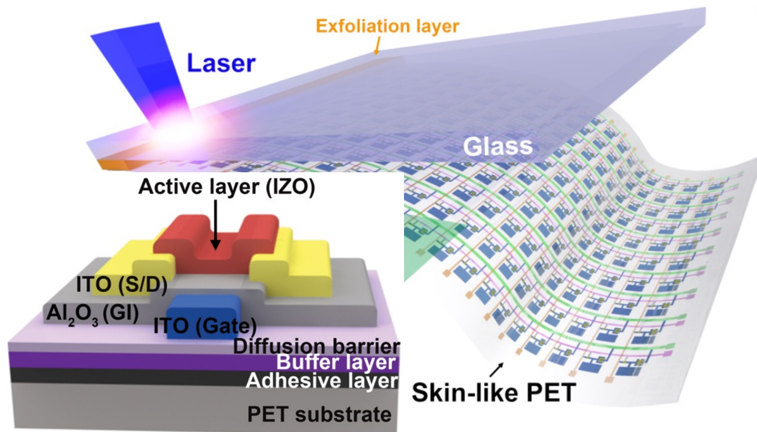
Hair-Regrowth by Red MicroLEDs

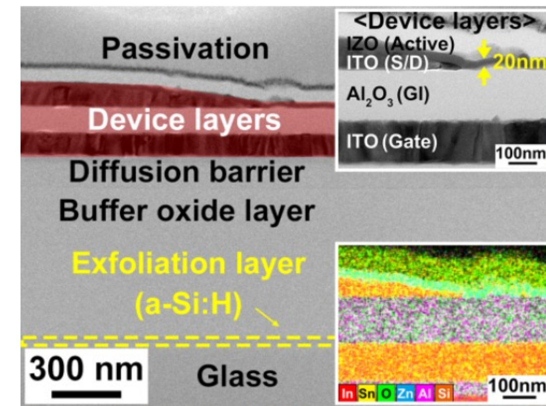


Lee et al., *ACS nano* 12.9 (2018): 9587-9595.

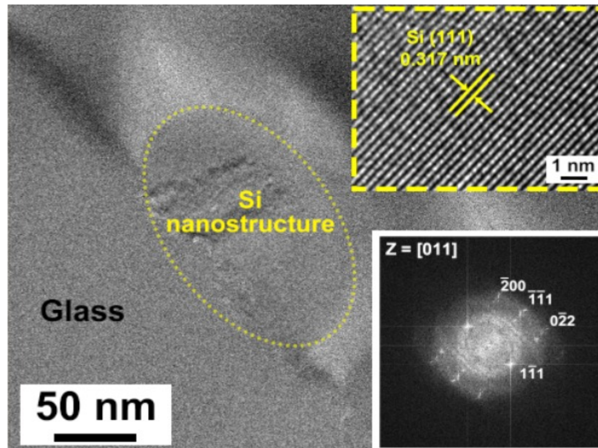
Material Analyses in Recent Researches

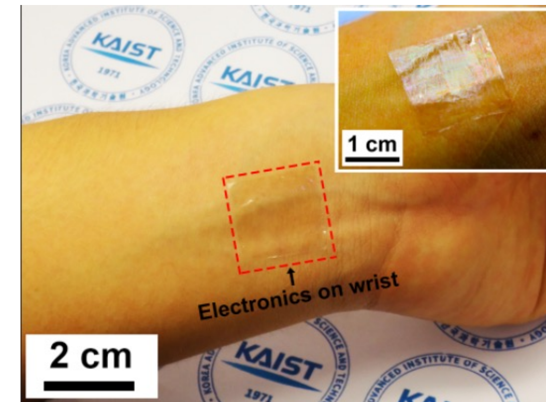
Flexible TFT





EDX

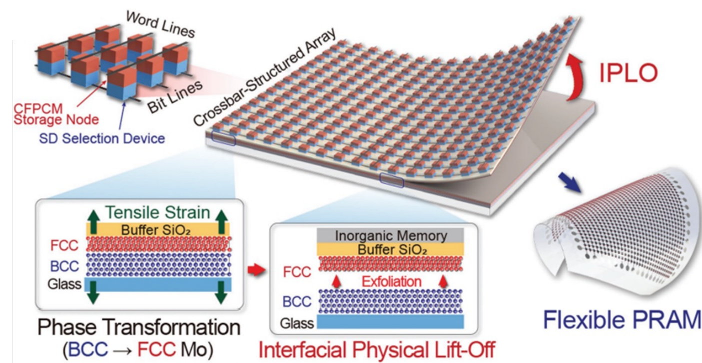


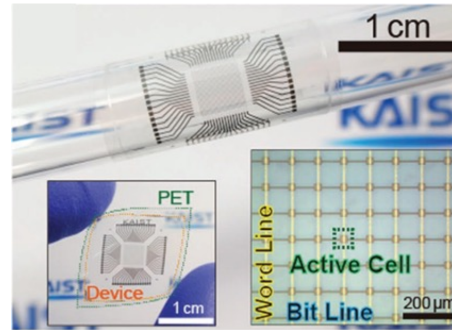


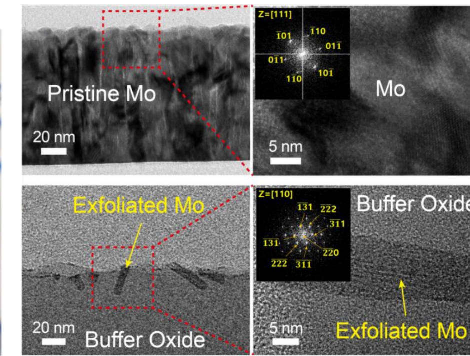
Lee et al., *Adv. Funct. Mater.*, 26 (34), 6170, 2016

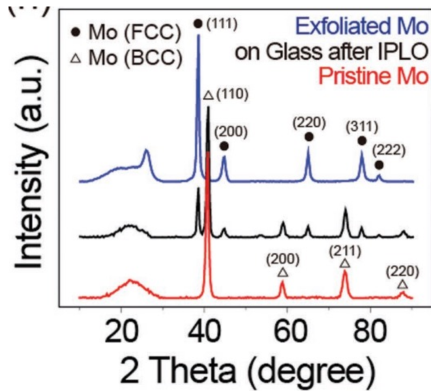
Material Analyses in Recent Researches

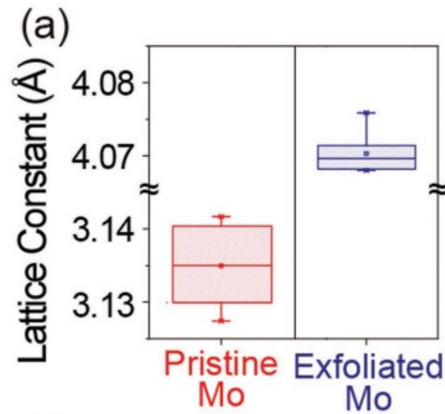
Flexible Memory

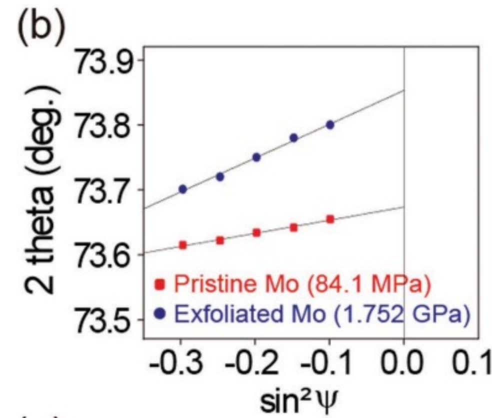


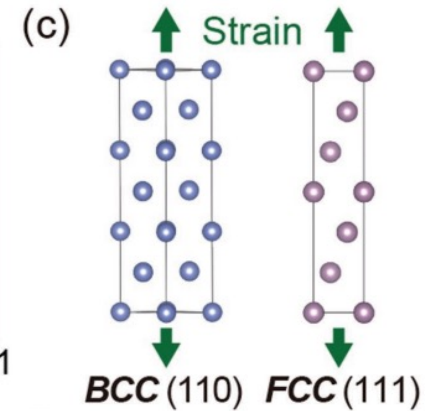










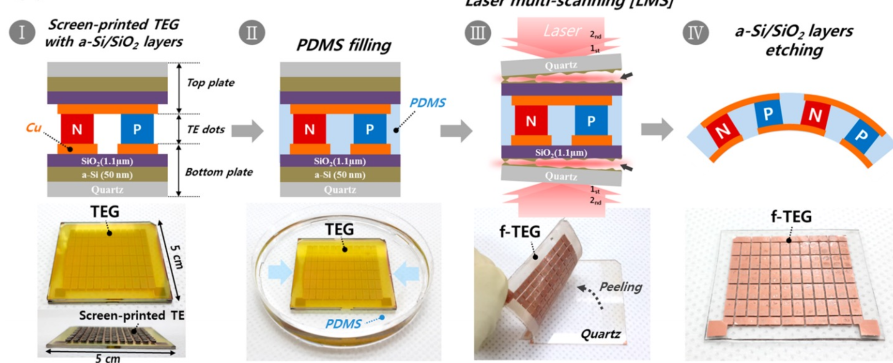


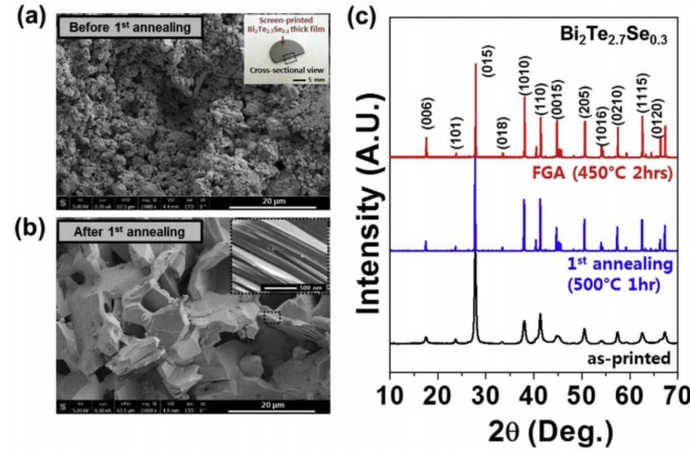
XRD

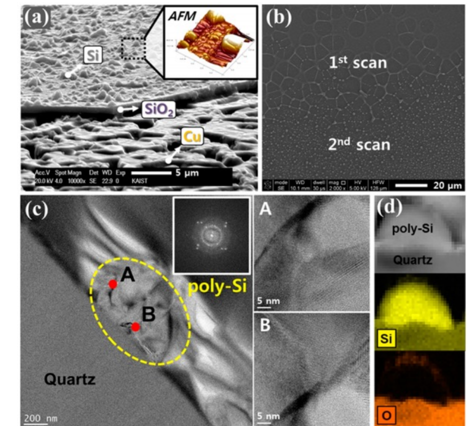
Lee et al., *Adv. Funct. Mater.*, 29 (6), 1806338, 2019

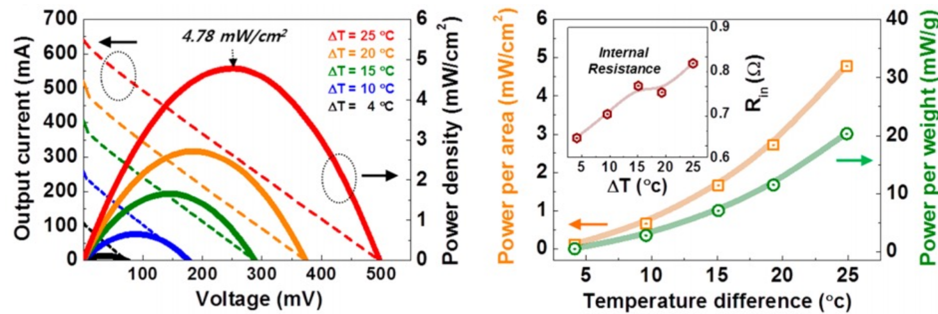
Material Analyses in Recent Researches

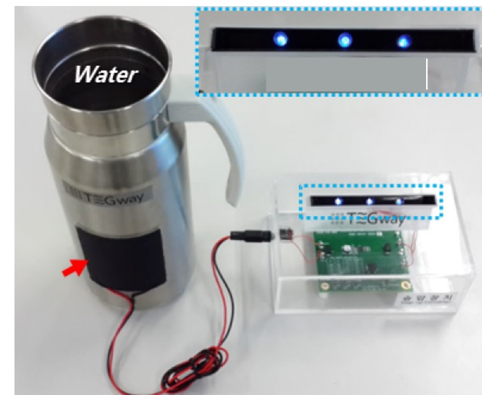
Thermoelectric Power Generator

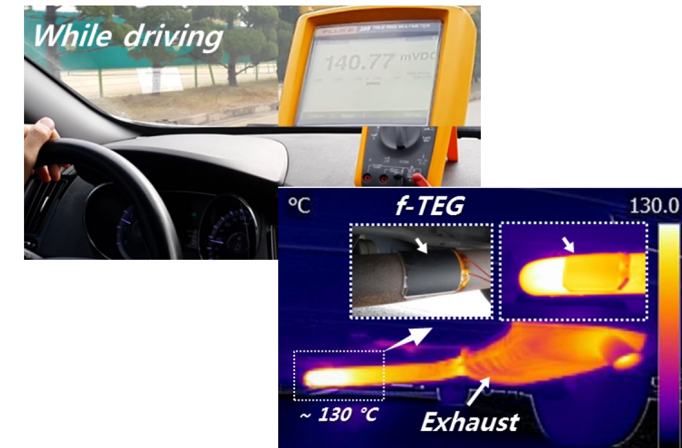








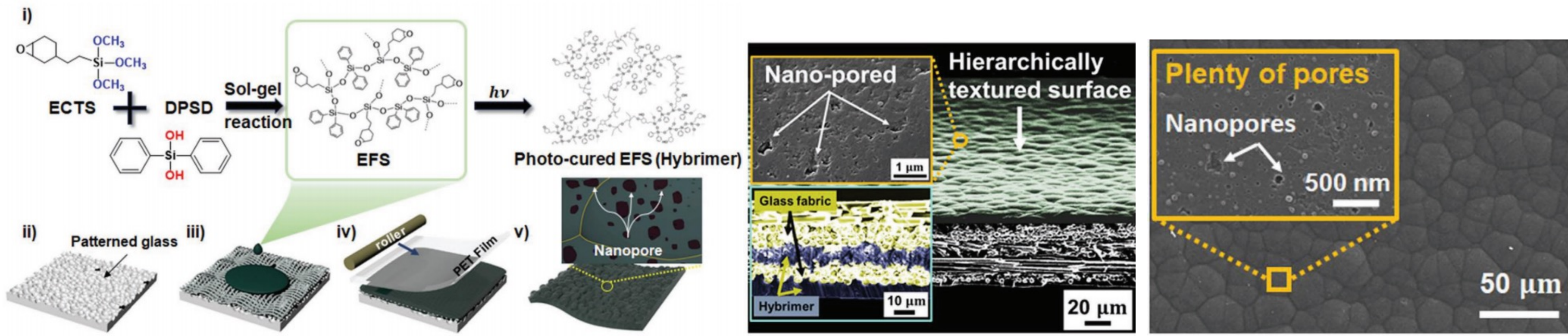


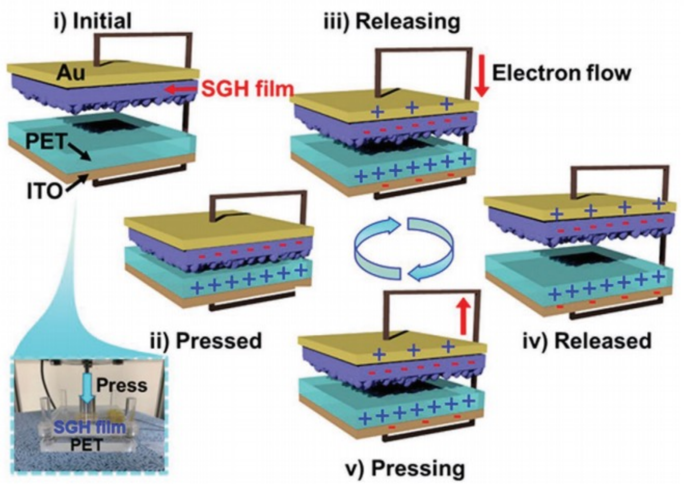


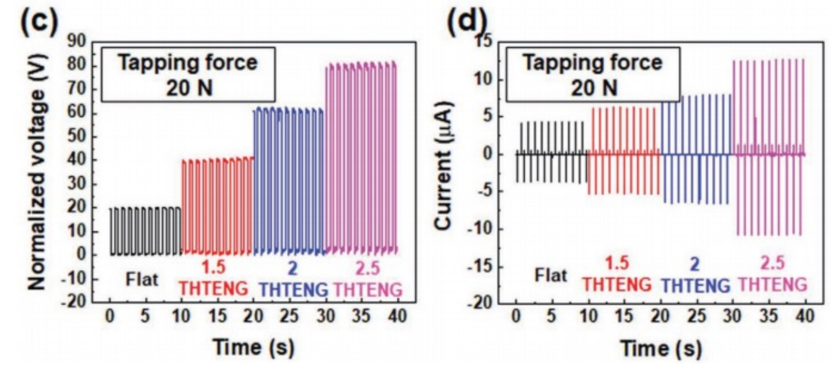
Lee et al., ***ACS nano***, 10 (12), 10851, 2016
Lee et al., ***Nano Energy***, 31, 258, 2017

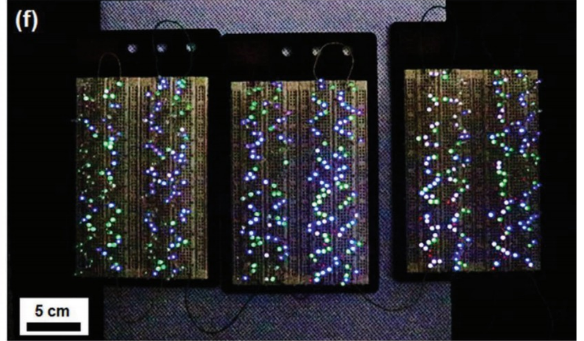
Material Analyses in Recent Researches

Triboelectric Power Generator





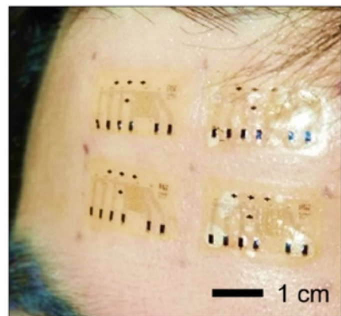


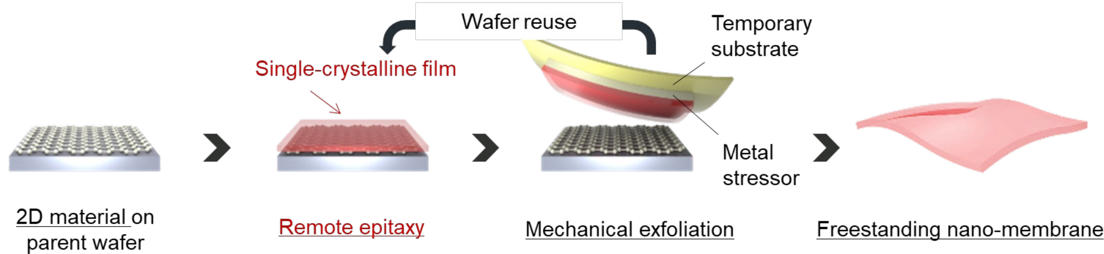


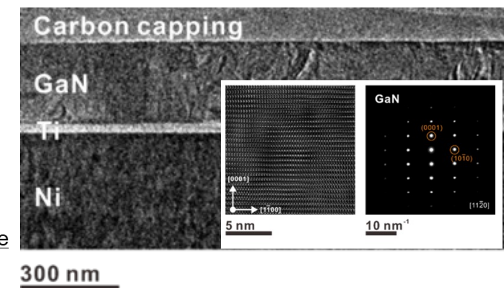
Lee et al., ***Adv. Funct. Mater.***, 30 (49), 2005610, 2020

Material Analyses in Recent Researches

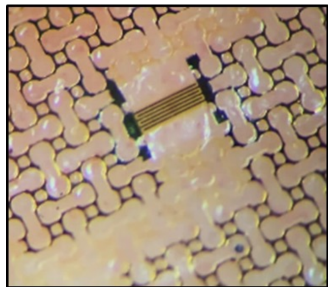
Single-Crystalline Physiological Sensors

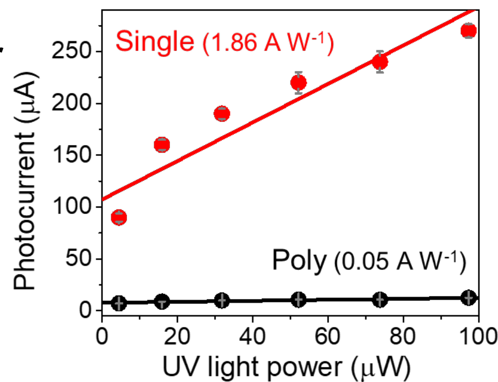




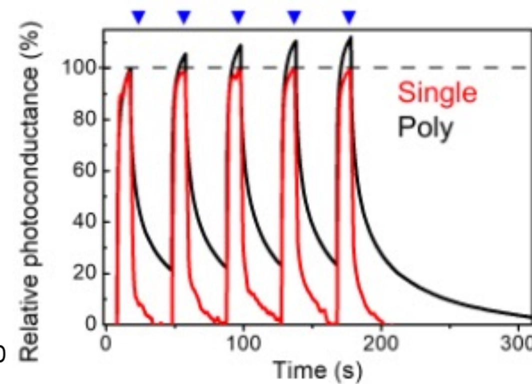


UV & Strain Sensor

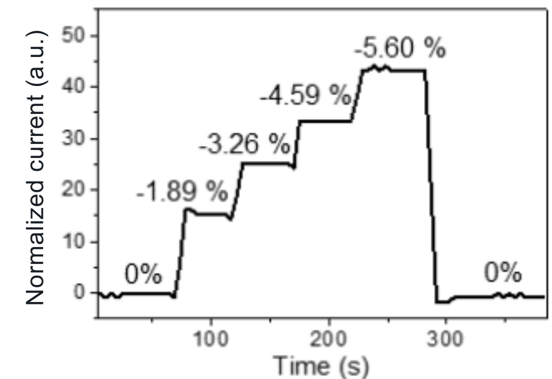




High UV response (37x)



Fast switching (9x)

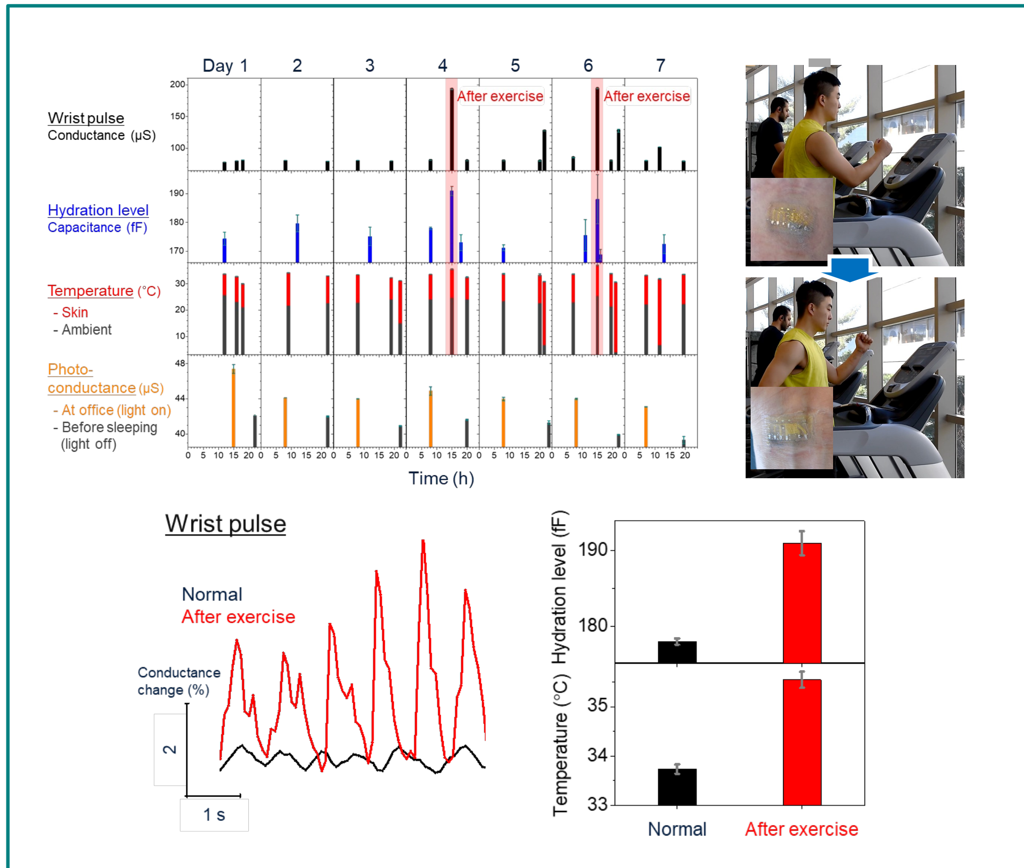


Low min. detectable strain

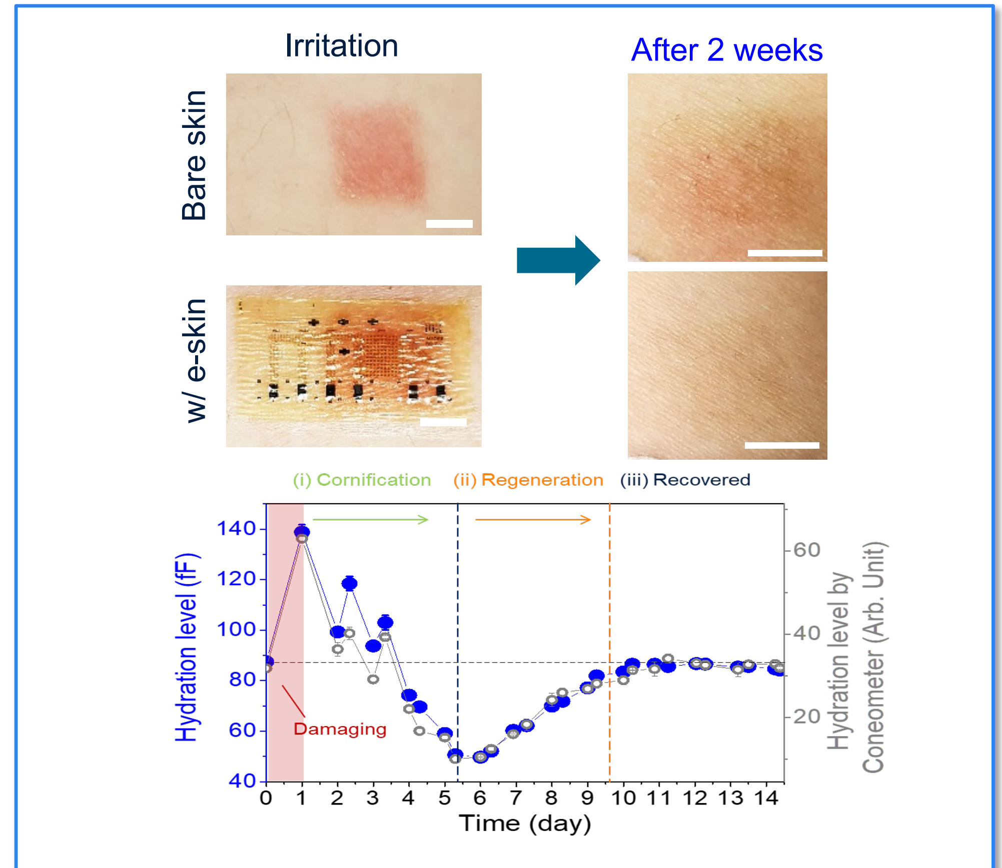
Lee et al. *Sci. Adv.*, 7 (27), eabg8459, 2021

Long-Term Accurate Monitoring via Perforated E-Skin

Daily Activity Checkup (1 Week)



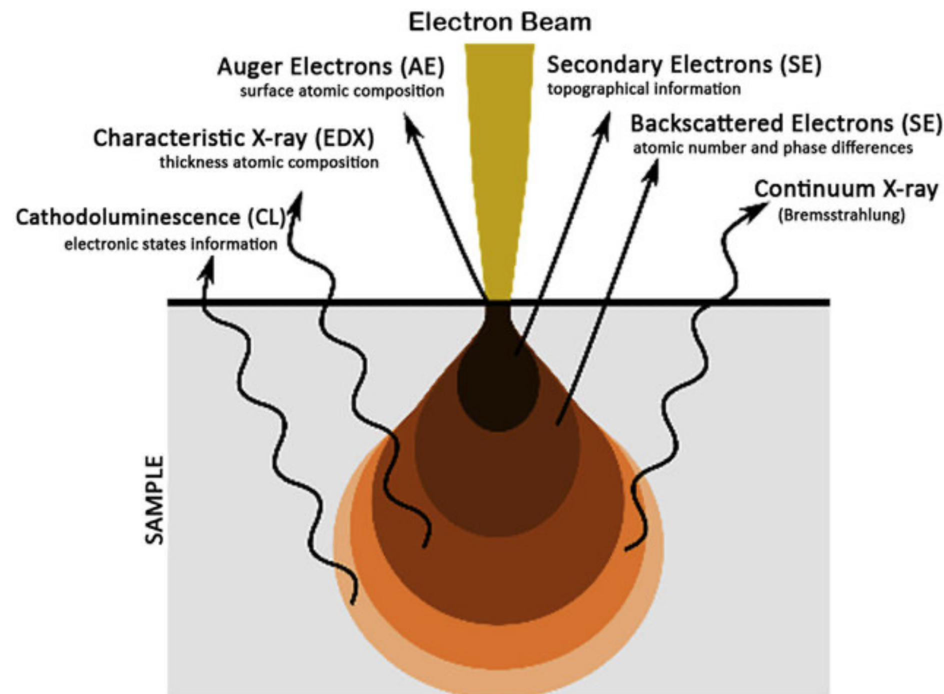
Monitoring Skin Regeneration (2 Weeks)

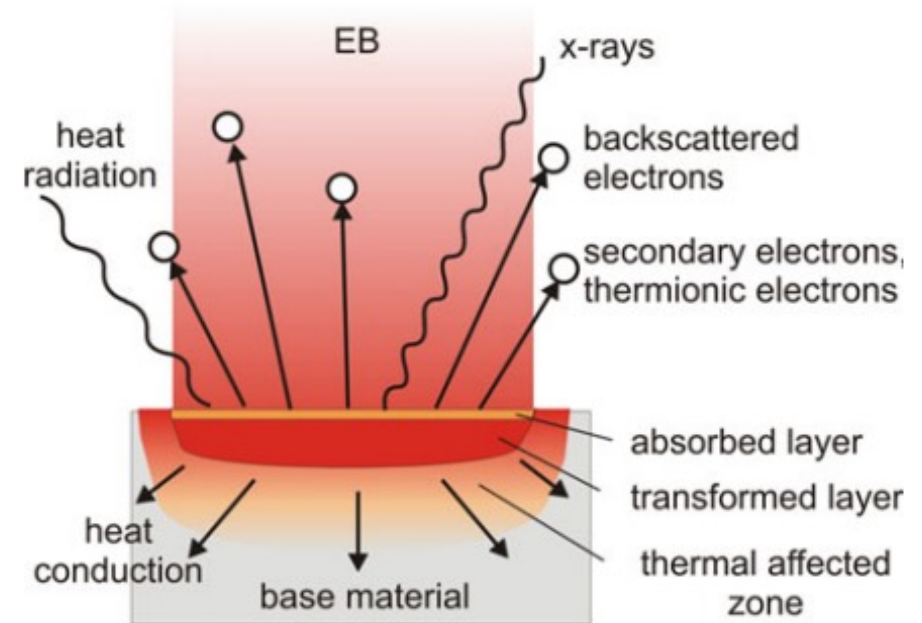


Lee et al. ***Sci. Adv.***, 7 (27), eabg8459, 2021

Electron Beams

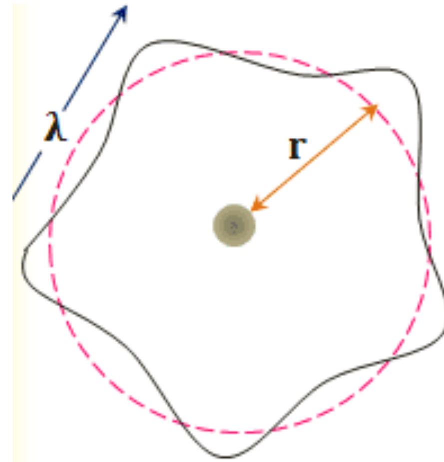
- Electron beams interact with matter in many ways:





Electron Beams

Louis de Broglie



de Broglie equation

$$\rho = \frac{h\upsilon}{c} = \frac{h}{\lambda}$$

ρ = particle's momentum
h = Planks constant (6.626×10^{-34}J·s)
λ = de Broglie wavelength
c = speed of light

- Major breakthrough in quantum physics

➔ Louis de Broglie— particle-wave duality.

- What does this mean?

➔ Simultaneously, a particle is both a material and a wave.

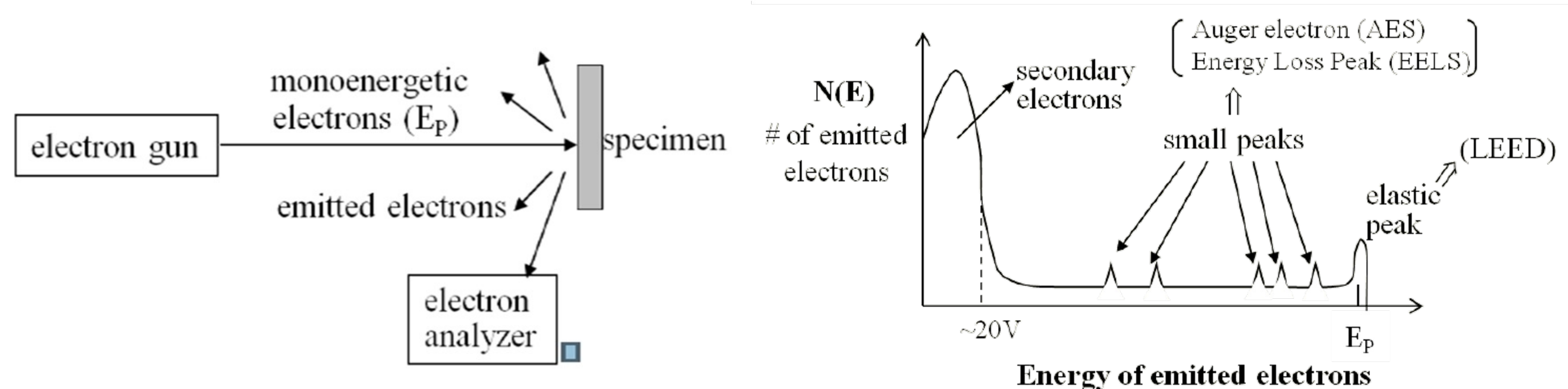
$$\lambda_e = \frac{h}{\rho} = \frac{h}{m_e \upsilon}$$

$$K.E. = \frac{1}{2} m_e \upsilon^2 = \frac{\rho^2}{2m_e^2} = eV$$

$$\rho = \sqrt{2m_e eV}, \qquad \lambda_e = \frac{h}{\sqrt{2m_e eV}}$$

m_e = rest mass for an electron (9.11×10^{-31}kg)
$e = 1.6022 \times 10^{-19}$C

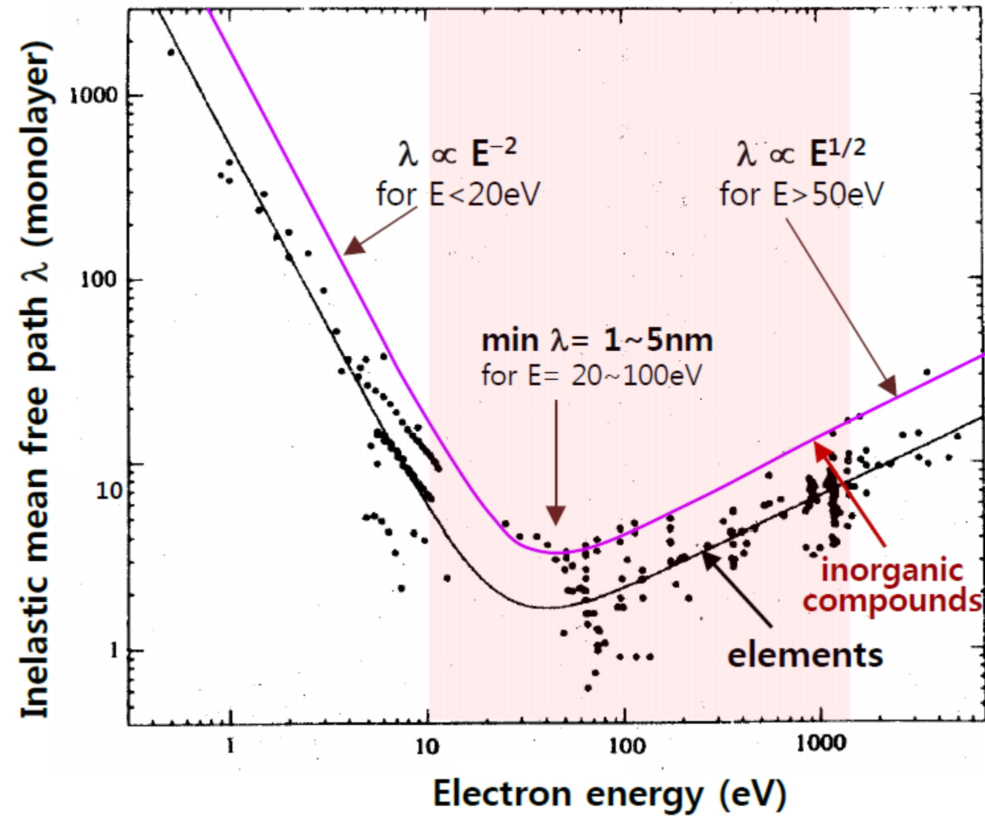
Electrons Emitted from a Solid Surface by an Incident Electron Beam



- Energy distribution of emitted electrons when a monoenergetic electron beam (E_P) is injected to the solid surface.

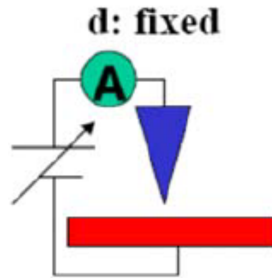
- Secondary electrons (≤~20eV) due to inelastic collision ⇒ SEM
- Elastic peak (a portion of the incident electrons) ⇒ LEED
- small peaks Auger electron peak ---- independent of E_P ⇒AES
- energy loss peak --------- dependent on E_P ⇒ EELS
- characteristic X-rays emitted from the sample ⇒ EPMA(EDS, WDS)

Inelastic Mean-Free-Path of Electrons in Solid vs Electron Energy



- λ of electron (E=10~1500eV) < a few nm in metals and a few tens of nm in ceramics ➔ Origin of surface sensitivity of electron spectroscopies

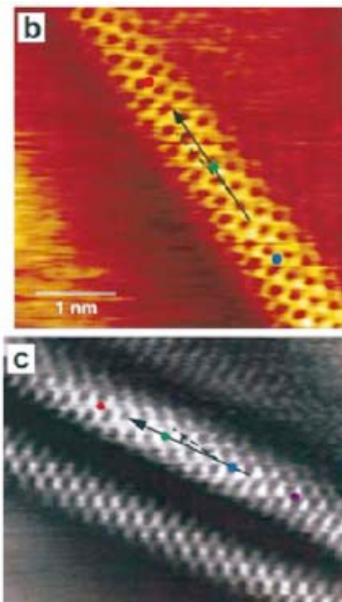
Example of Case Study: STM of Carbon Nanotubes

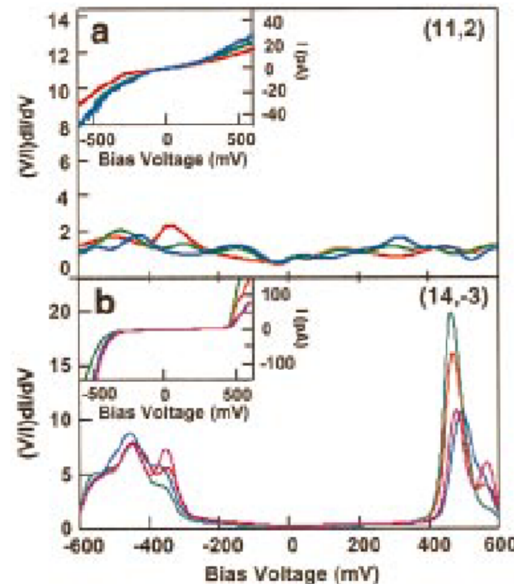


$I_t \sim \Sigma |\psi_{tip}|^2 |\psi_{sample}|^2 e^{-2\kappa d}$

$(Z)_{I,V}$ - x,y : Topography

dI/dV~ local electronic structure of sample surface
(LDOS: Local Density Of State)





Constant LDOS
Metallic SWNT

Semiconducting
SWNT

"Atomic structure and electronic properties of single-walled carbon nanotubes", Nature 391, 62-64 (1998).