나노분석의 기초 및 응용

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나노분석의 기초 및 응용

- 나노분석의 기초

- 구조분석(SEM, TEM, FIB)

- 표면분석(XPS, AES, SIMS)

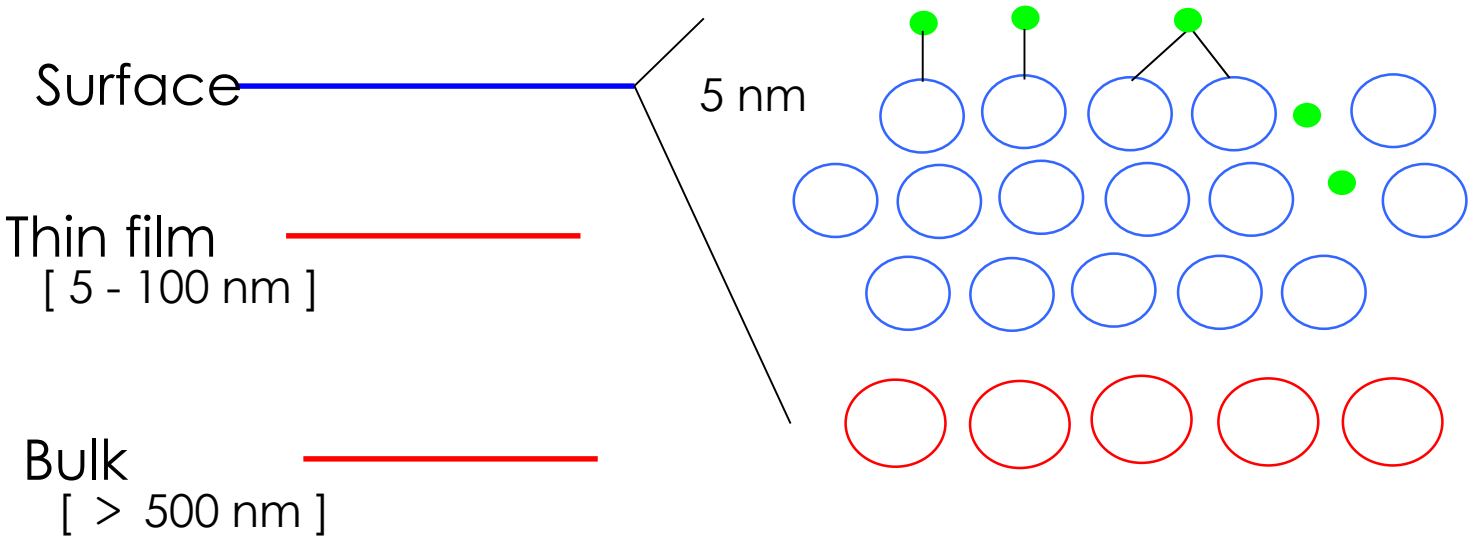
● 나노분석 기술

나노물질, 소재, 소자 등 시료의 형상을 나노미터 또는 원자 수준에서 관찰하거나, 구조 및 성분을 분석하고, 또는 물성 또는 특성을 계측하는 기술로서 측정 목적에 따라서 아래와 같이 분류할 수 있음

기술분류	기술개념
형상 및 구조 분석	형상 및 구조를 나노미터 또는 원자 수준에서 측정하고 분석하는 기술
성분 및 화학 분석	나노표면의 성분 및 화학 분석기술, 극미량 성분 분석기술
물성 및 특성 분석	광학특성 측정기술, 전기특성 평가기술, 역학특성 분석기술

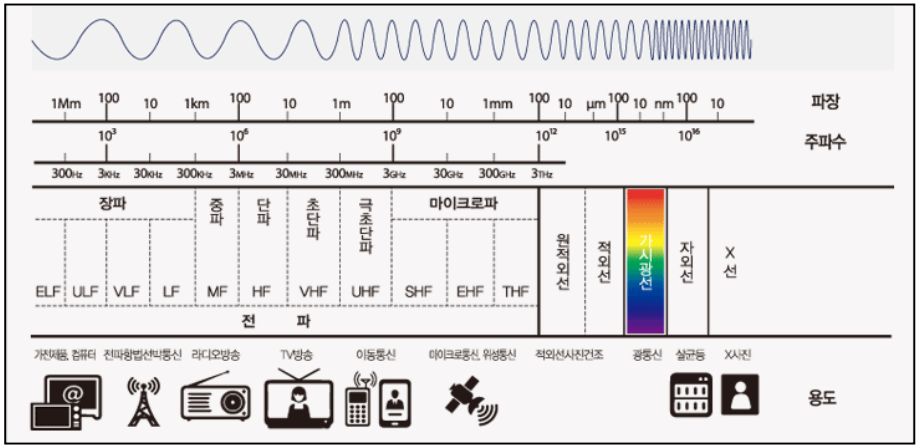
측정분석 대상

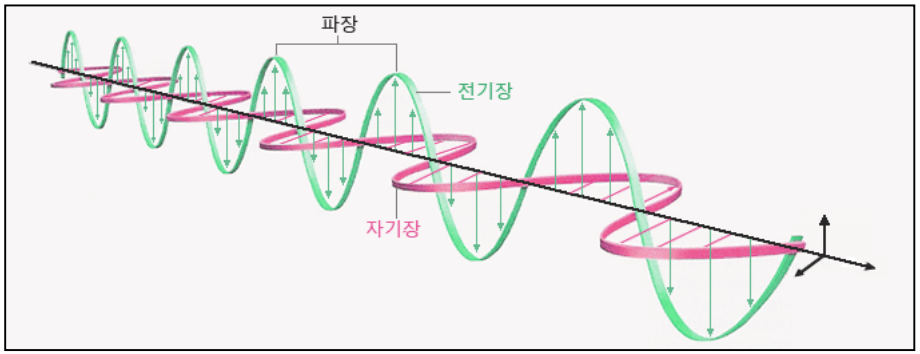
- **Solid**
  - Crystalline / Amorphous
  - Conductor / Insulator
  - Organic / Inorganic
  - Surface / Thin film / Bulk
- **What is it ?** Morphology & Structure
  Elemental & Chemical composition
- **Where is it ?** Spatial x-y, Depth z distribution



광원 : 전자기파(電磁波) & 물질파(De Broglie Wave)

전자기파





$E = h\nu = h(c/\lambda) = hc/\lambda$, $\lambda = hc/E$

E(빛의 에너지)
h(플랑크 상수) = $6.626\ 070\ 040 \times 10^{-34}$ J•s
ν(빛의 진동수)
c(빛의 속도) = 3×10^8 m/s
λ(빛의 파장)

물질파@전자(, 이온 등)

Rest mass		m_0	=	9.1091×10^{-31} kg
Charge		e	=	-1.602×10^{-19} C
Kinetic energy		E	=	eU
				$1\ \text{eV} = 1.602 \times 10^{-19}$ J
Velocity of light		c	=	2.9979×10^8 m s^{-1}
Rest energy		E_0	=	$m_0c^2 = 511$ keV
Spin		s	=	$h/4\pi$
Planck's constant		h	=	6.6256×10^{-34} J s
Nonrelativistic	$(E \ll E_0)$			Relativistic $(E \sim E_0)$
Newton's law	$F = \frac{dp}{d\tau}$	F	=	$\frac{d}{d\tau}(mv)$ (2.7)
Mass	$m = m_0$	m	=	$m_0/\sqrt{1 - v^2/c^2}$ (2.8a)
Energy	$E = eU = \frac{1}{2}m_0v^2$	mc^2	=	$m_0c^2 + eU = E_0 + E$ (2.9)
		m	=	$m_0(1 + E/E_0)$ (2.8b)
Velocity	$v = \sqrt{2E/m_0}$	v	=	$c\sqrt{1 - \frac{1}{(1 + E/E_0)^2}}$ (2.10)
Momentum	$p = m_0v = \sqrt{2m_0E}$	p	=	$\sqrt{2m_0E(1 + E/2E_0)}$ (2.11)
			=	$\frac{1}{c}\sqrt{2EE_0 + E^2}$
Wavelength	$\lambda = \frac{h}{p} = h/\sqrt{2m_0E}$	λ	=	$h/\sqrt{2m_0E(1 + E/2E_0)}$ (2.12)
			=	$hc/\sqrt{2EE_0 + E^2}$

$\lambda = h/p = h/mv$

E_0	electrons	He ions	Ga ions
100 keV	~ 4 pm	~ 0.05 pm	~ 0.01 pm
10 keV	~ 10 pm	~ 0.1 pm	~ 0.03 pm

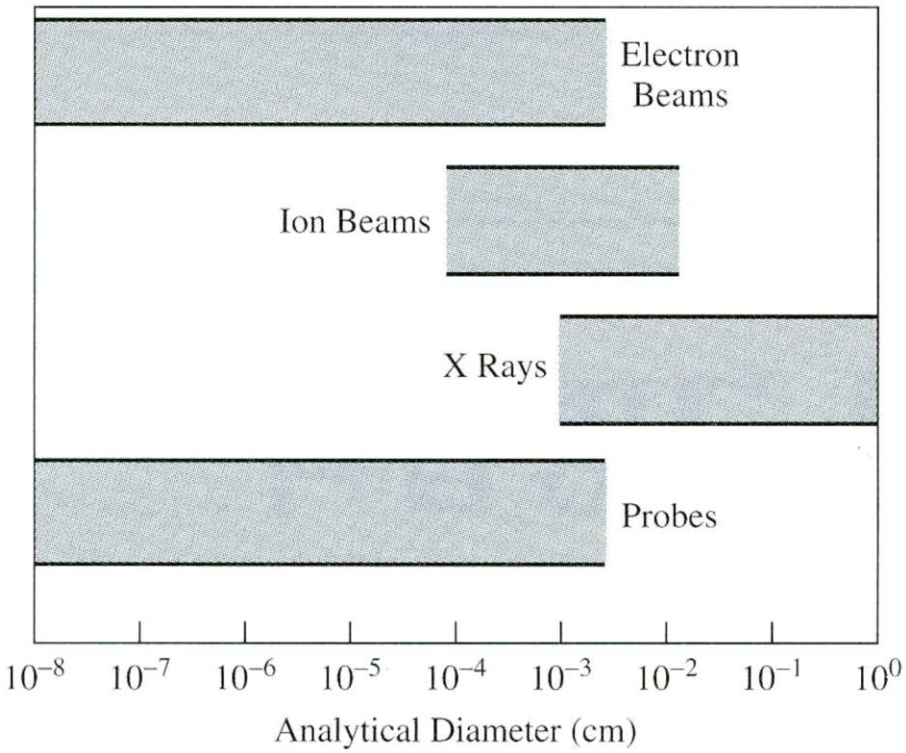
Beam & Probe (광원 & 탐침)

A beam : a directed flow of particles or waves that carries energy and information (amplitude, phase, frequency…)

- **Light (전자기파)**
  - Electromagnetic radiation
  - Visible rays : 380~770 ㎚
  - Infrared rays
  - Ultraviolet rays
  - LASER

- **Probe (탐침)**
  - Extremely sharp tip (3-50 nm radius of curvature)

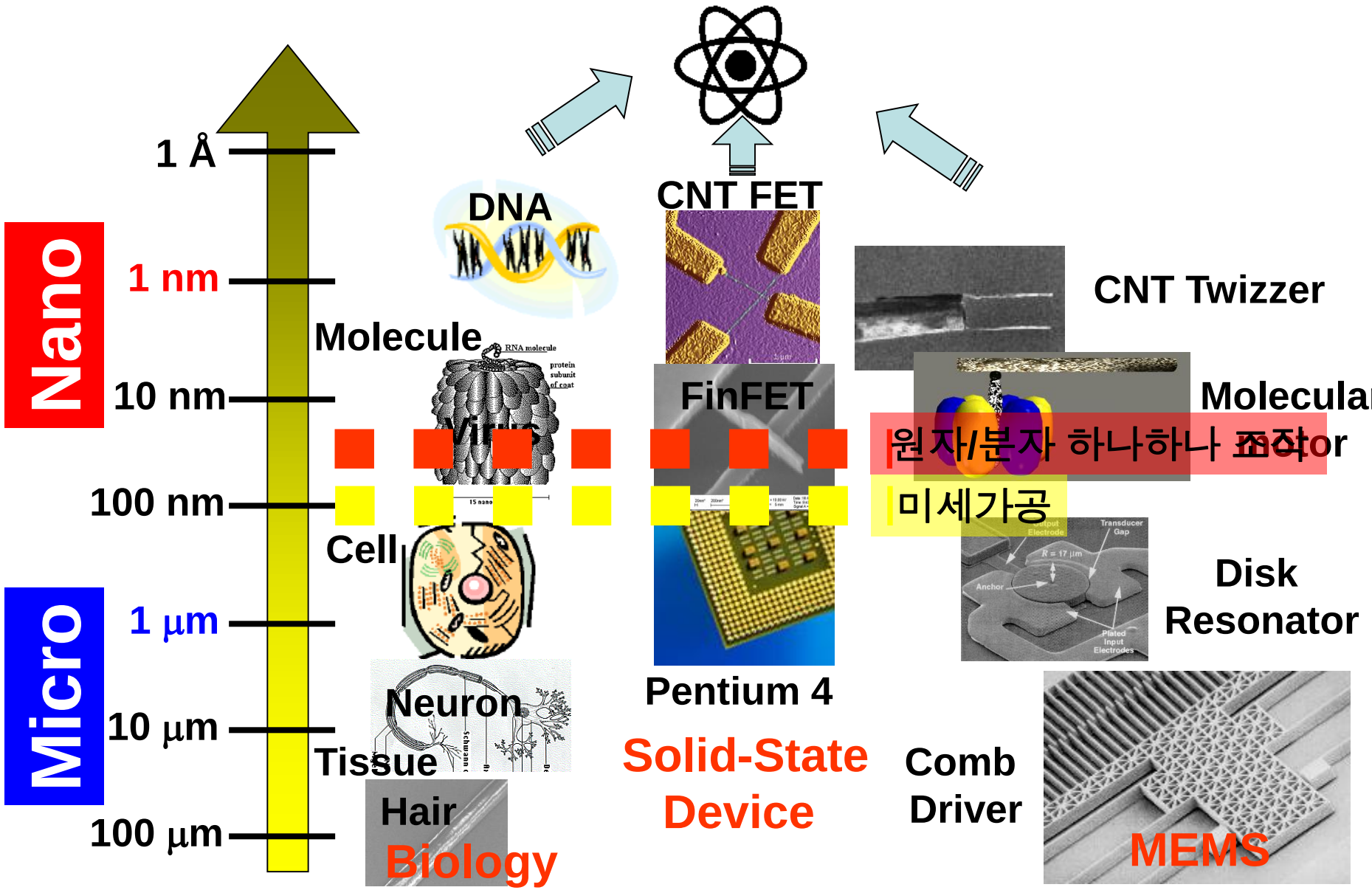
- **X-Ray (전자기파)**
  - Electromagnetic radiation
  - Wavelength : about 100 Å to 0.01 Å
  - X-rays by the transitions of inner electrons (light by the transitions of outer electrons)



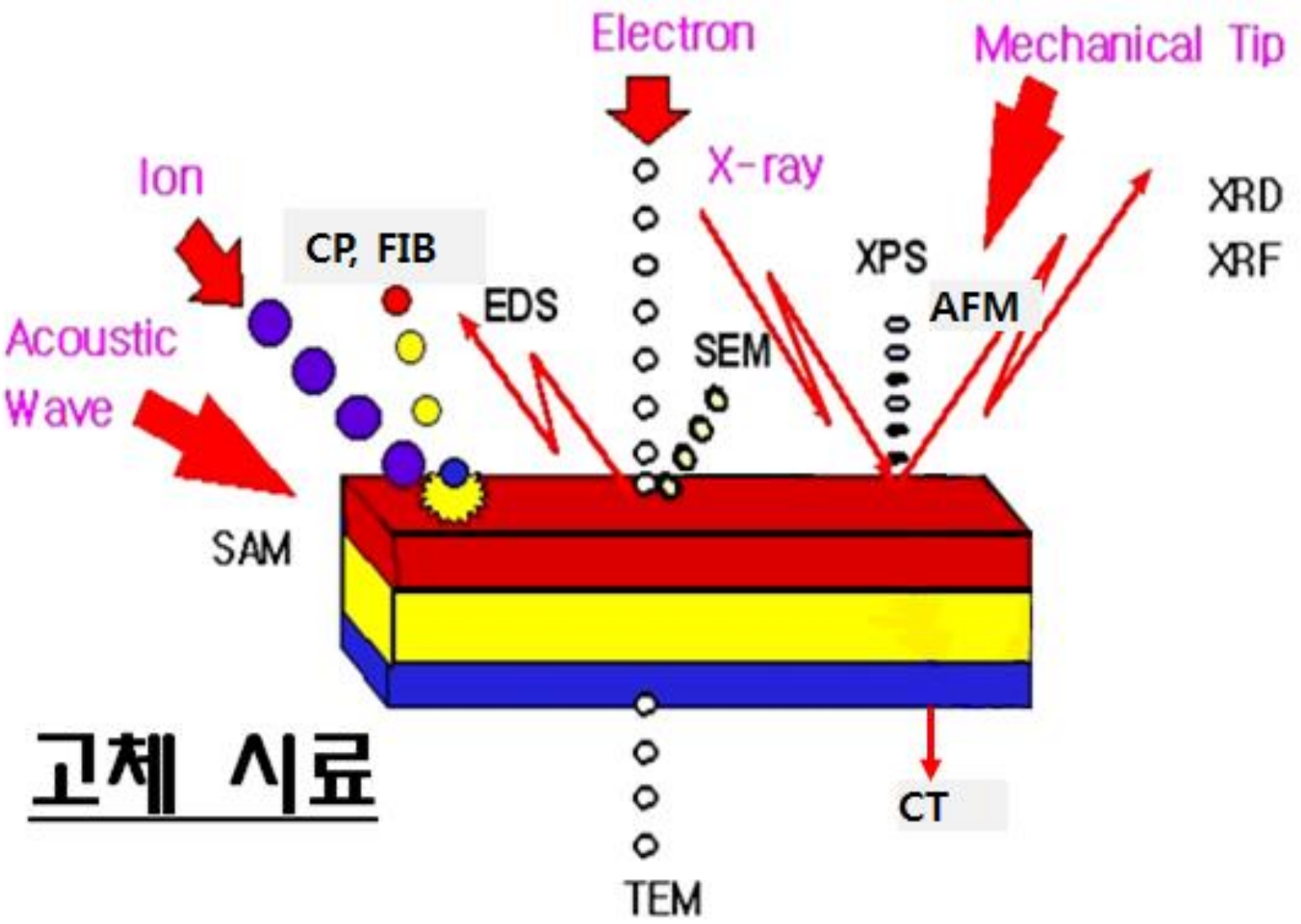
- **Ion (물질파)**
  - Particle formed when a neutral atom gains or loses one or more electrons
  - Cation : an atom that loses an electron
  - Anion : an atom that gains an electron

- **Electron (물질파)**
  - Negatively charged particle (-1.602×10^{-19} C)
  - Mass : 9.107×10^{-28} g

나노기술(Nanotechnology)



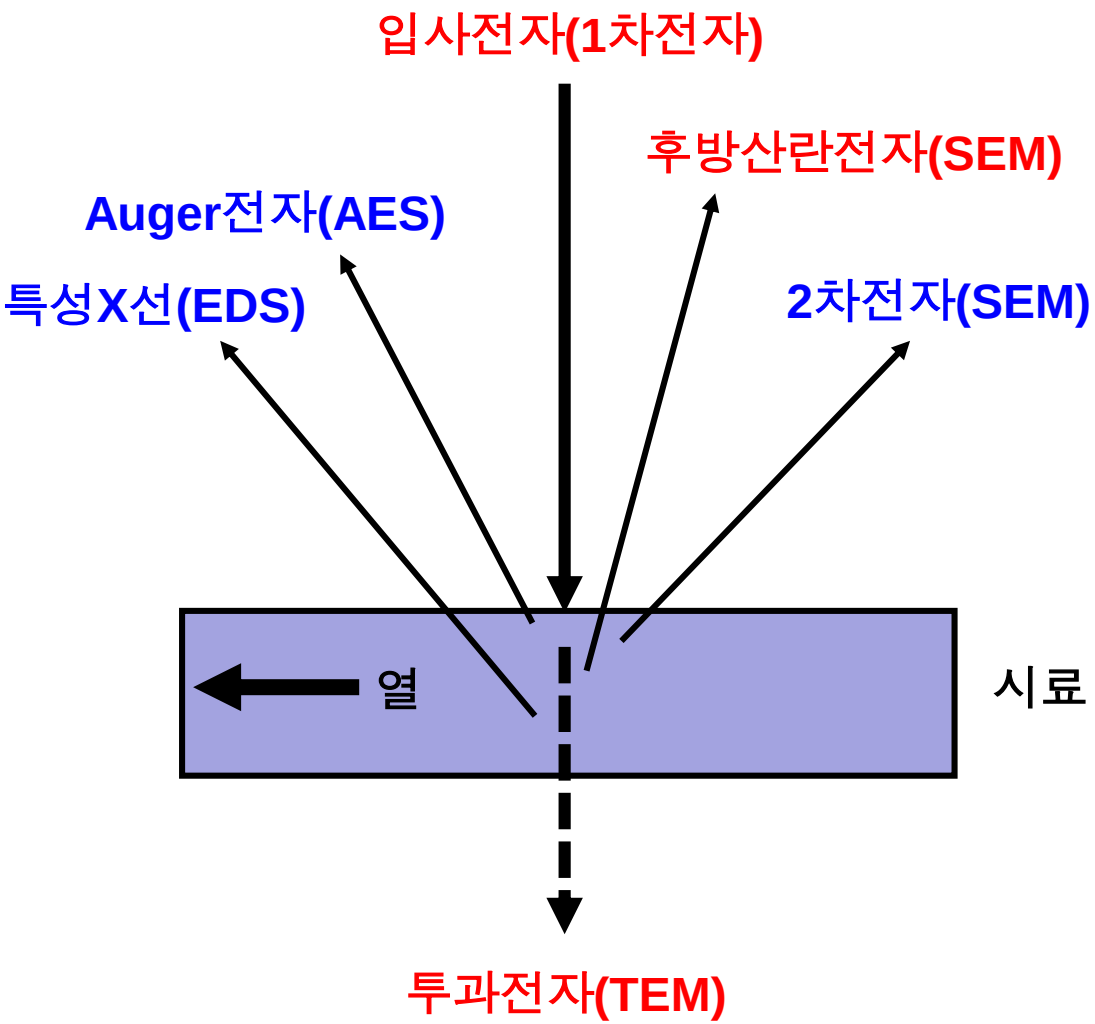
다양한 분석기법



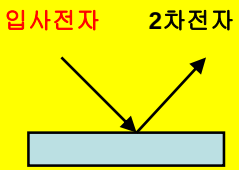
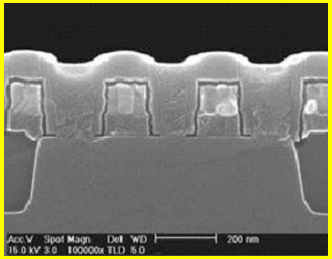
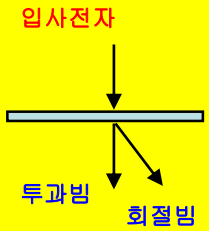
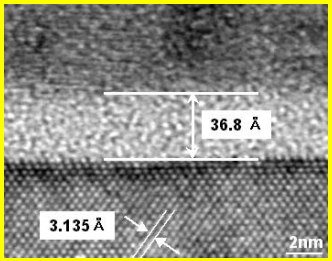
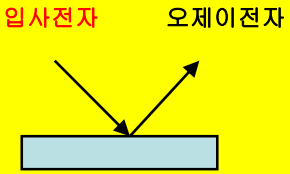
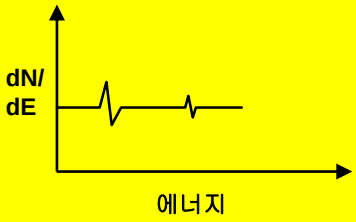
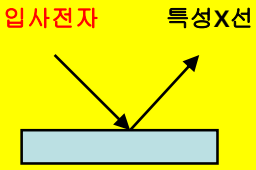
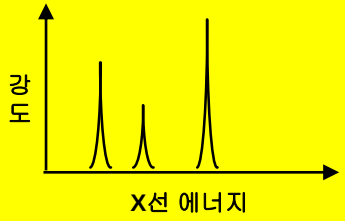
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전자를 이용한 분석기술

전자와 고체의 상호작용

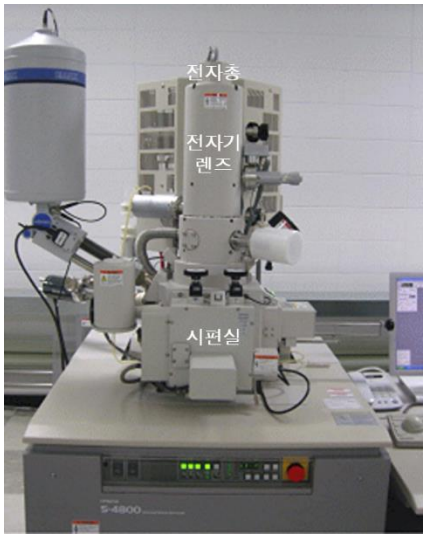

입사전자(1차전자)
후방산란전자(SEM)
Auger전자(AES)
특성X선(EDS)
2차전자(SEM)
열
시료
투과전자(TEM)

전자빔 분석기술의 원리와 특징

분석기법	원리	측정 방법	측정 Data	측정 항목
주사전자현미경 (Scanning Electron Microscopy)		•시료 표면에 1~30 kV의 전자를 입사시켜 이로부터 나오는 2차전자를 검출		•미세한 표면, 단면의 형상, CD 측정 •분해능 : ~1 nm (FEG)
투과전자현미경 (Transmission Electron Microscopy)		•100-1000 kV의 전자를 시료에 투과시켜 원자 상호간의 작용에 의한 투과와 회절되는 전자를 검출함		•미세한 결정결함, 결정구조, 결정입자의 크기, 원자 Lattice의 관찰 •분해능 : 2 Å
오제이전자분광법 (Auger Electron Spectroscopy)		•시료에 전자선을 입사시켜 여기 방출되는 원소 특유의 오제이전자의 에너지를 측정		•성분분석 •Sputtering을 통해 깊이 방향 분포 분석, 표면분석에 주로 사용 •Small probe size
에너지분산 X선분광법 (Energy Dispersive X-ray Spectroscopy)		•시료에 수 십, 수 백 keV의 전자빔을 입사시켜 방출되는 원소 고유의 특성X선 에너지를 측정		•성분분석 •공간분해능 : 수 십 nm @ SEM : ~1 nm @ TEM •Better for several atomic %

전자빔 광원을 이용한 분석장비

SEM



TEM



AES



표면형상

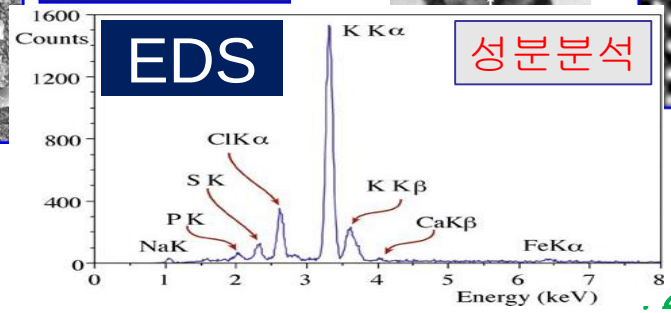


미세구조

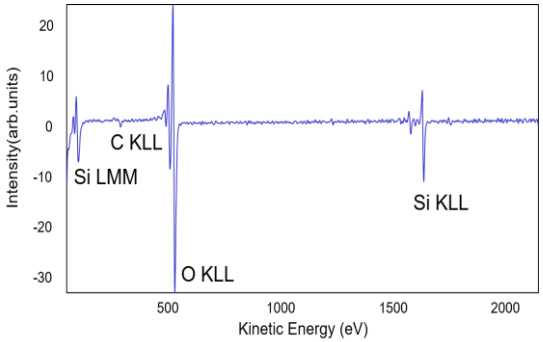


HRTEM of Au

EDS



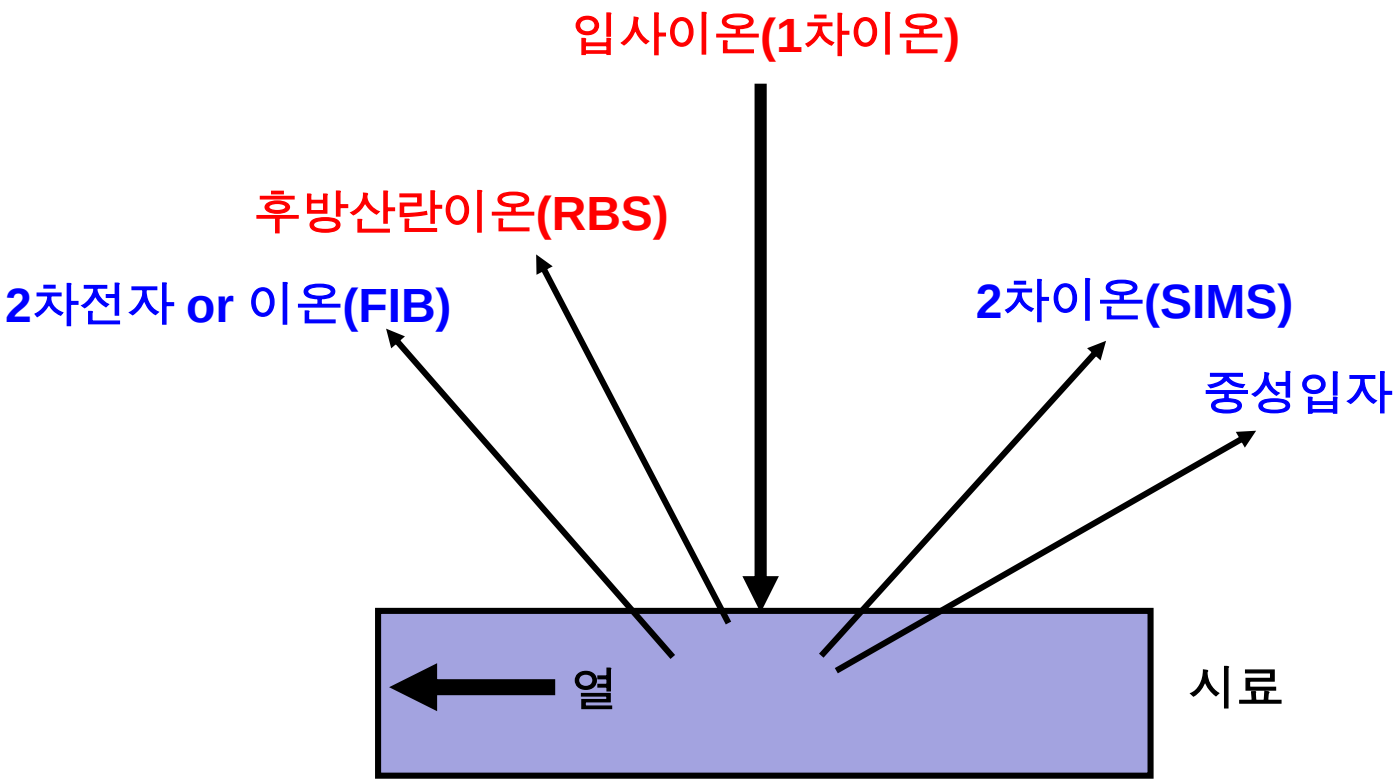
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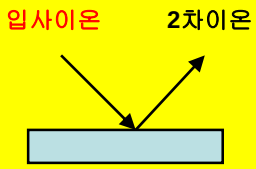
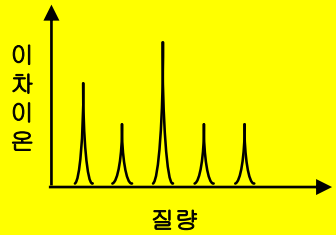
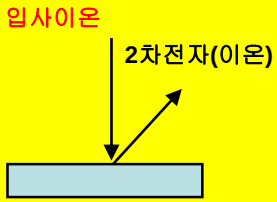
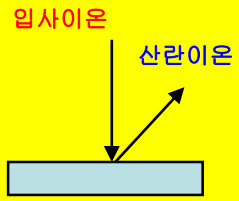
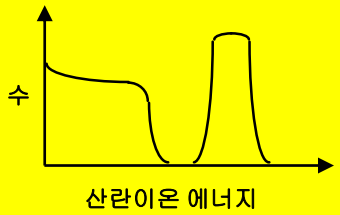
성분분석

이온을 이용한 분석기술

이온과 고체의 상호작용



이온빔 분석기술의 원리와 특징

분석기법	원리	측정 방법	측정 Data	측정 항목
이차이온 질량분석법 (Secondary Ion Mass Spectrometry)		•1-20 kV의 1차이온을 시료에 입사시켜 Sputter하여 방출되는 2차이온의 질량을 분석		•불순물의 질량 분석과 조성의 측정, 깊이 방향의 불순물 분포 측정(Si 중의 Boron의 경우 $10^{17}/cm^3$ 정도까지 검출가능) •Composition Standards •Best Sensitivity
집속이온빔 (Focused Ion Beam)		•수십 keV로 가속된 이온을 시료에 입사시켜 방출되는 전자 및 이온을 검출하여 영상을 형성하거나 Milling을 함		•특정 위치의 단면관찰 및 불량분석 •반도체 회로수정 •EDS 등을 이용한 성분 분석 •특정위치 TEM 시편제작
러터퍼드 후방산란분광법 (Rutherford Backscattering Spectroscopy)		•수 MeV로 가속된 이온을 시료에 입사시켜 후방산란되는 이온의 에너지를 측정		•조성과 불순물의 깊이 방향 분포 결정(Channeling법과 조합시켜 사용) •Precise atomic % without standards •Nondestructive, simultaneous •Depth and composition

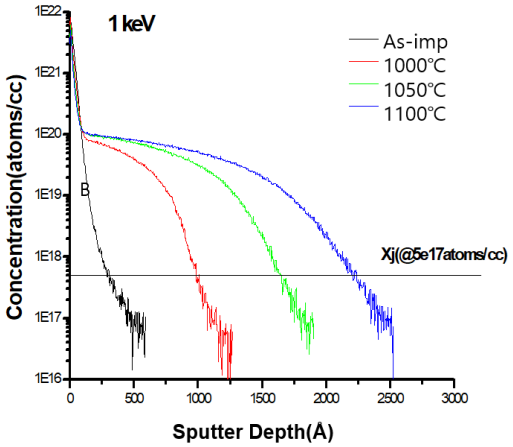
이온빔 광원을 이용한 분석장비

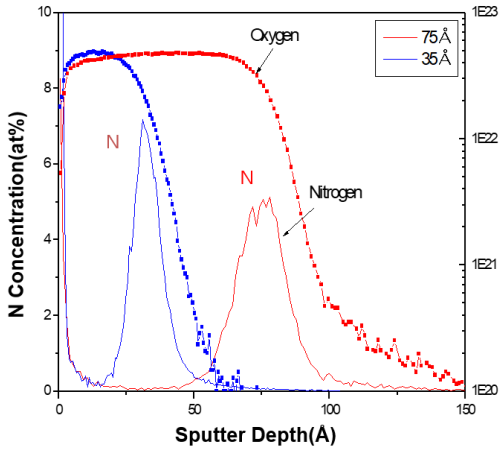
SIMS



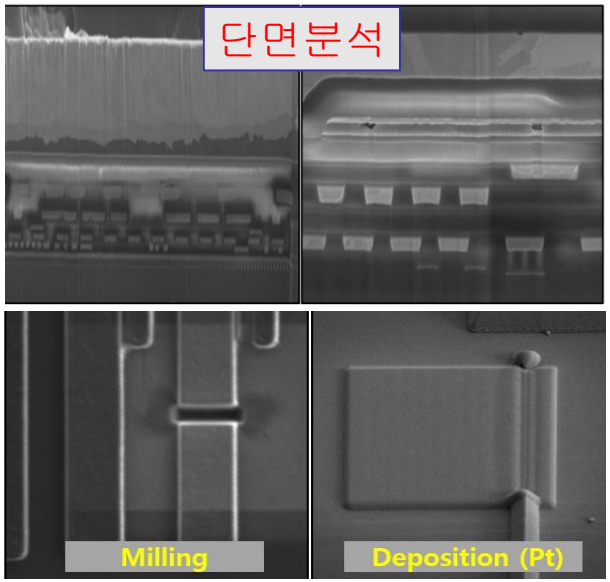
FIB





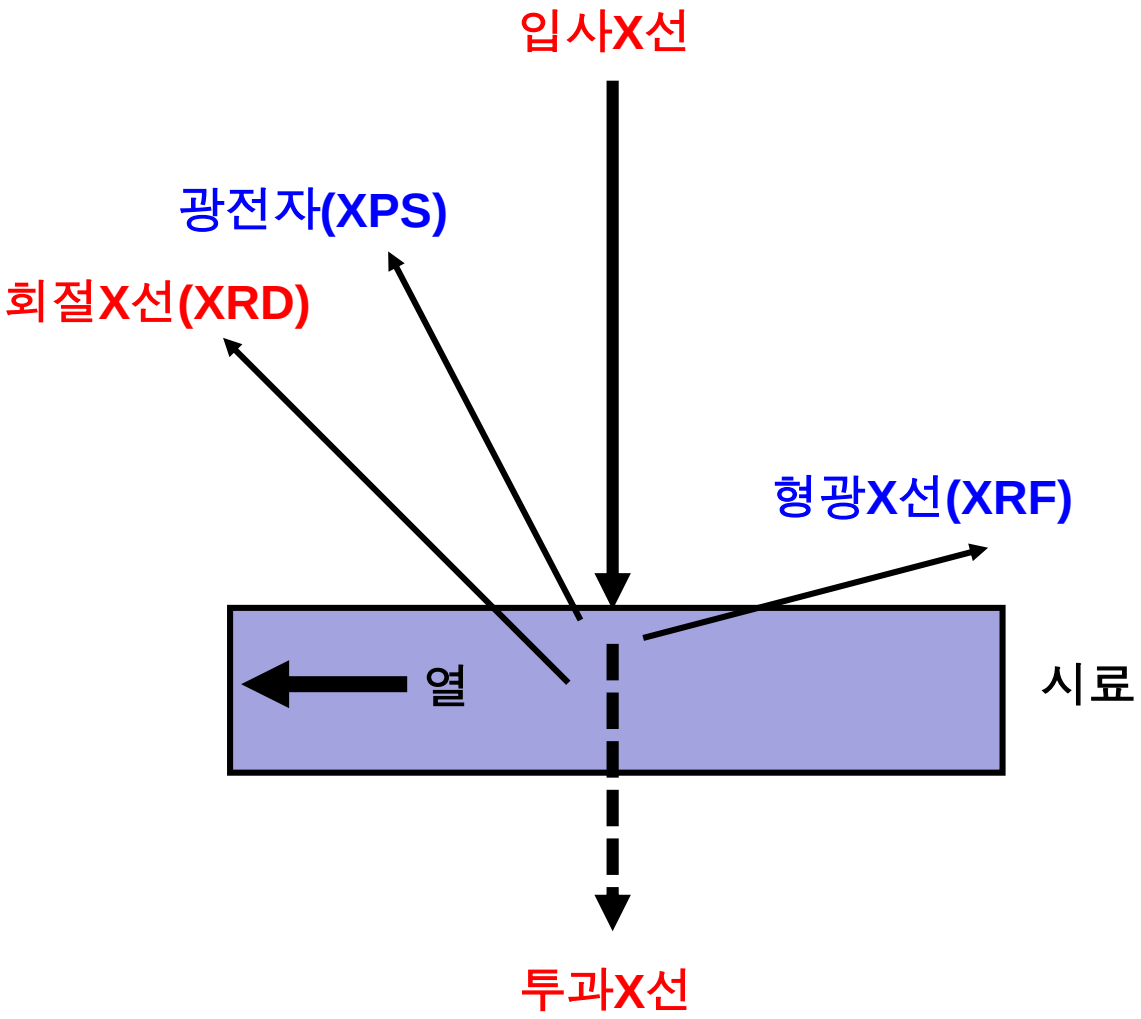


미량 성분분석

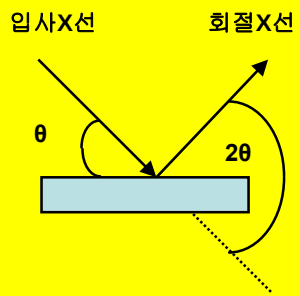
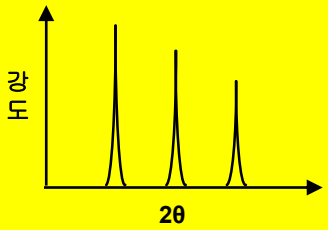
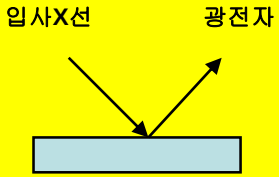
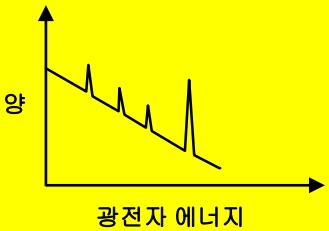
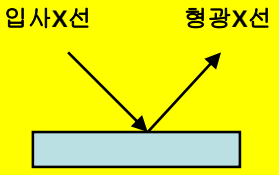
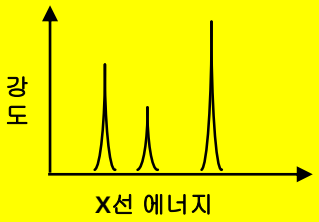


X선을 이용한 분석기술

X선과 고체의 상호작용



X선 분석기술의 원리와 특징

분석기법	원리	측정 방법	측정 Data	측정 항목
X선회절법 (X-ray Diffraction)		•시료에 X선을 입사시켜 Bragg 조건을 만족하는 X선을 검출		•비파괴 분석 •결정구조, 결정입자의 크기, 결정 Stress, 결정 우선방위의 결정
X선광전자분광법 (X-ray Photoelectron Spectroscopy)		•X선에 의해 여기된 광전자의 에너지를 측정		•성분분석 •Sputtering을 통해 깊이 방향 분포 분석 •화학결합 상태를 알 수 있음 •Chemical bonding
X선형광분석법 (X-ray Fluorescence Analysis)		•시료에 수 십 keV의 X선을 입사시켜 방출되는 원소 고유의 특성X선의 에너지를 측정		•비파괴 분석 •넓은 분석 범위(ppm ~ %) •시료준비 용이 •신속한 분석

X선 광원을 이용한 분석장비

XRD

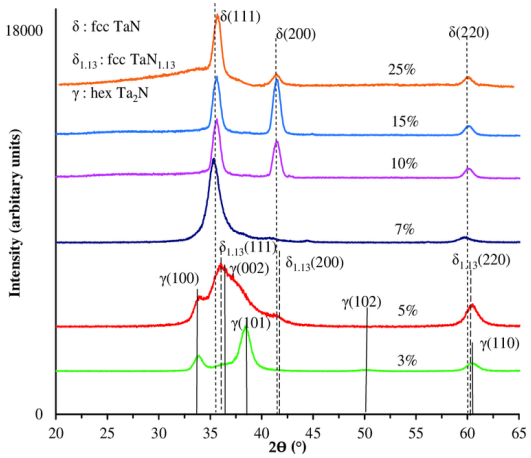


XPS

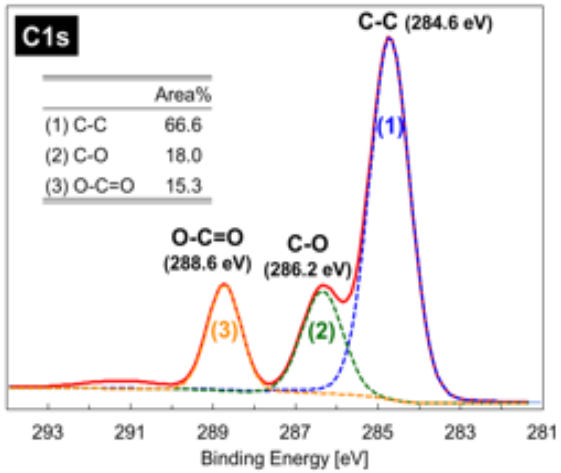


XRF

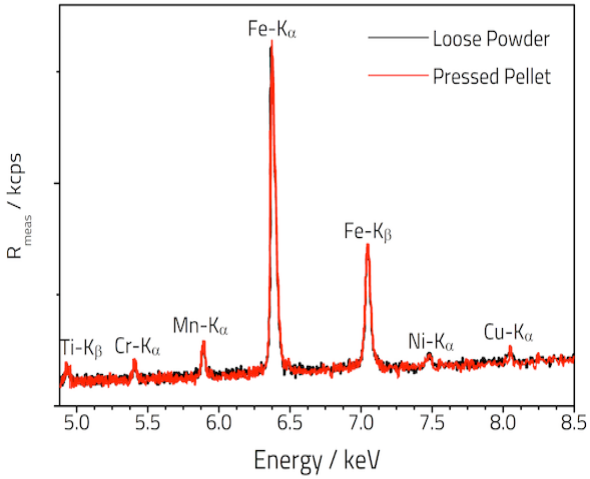




결정구조



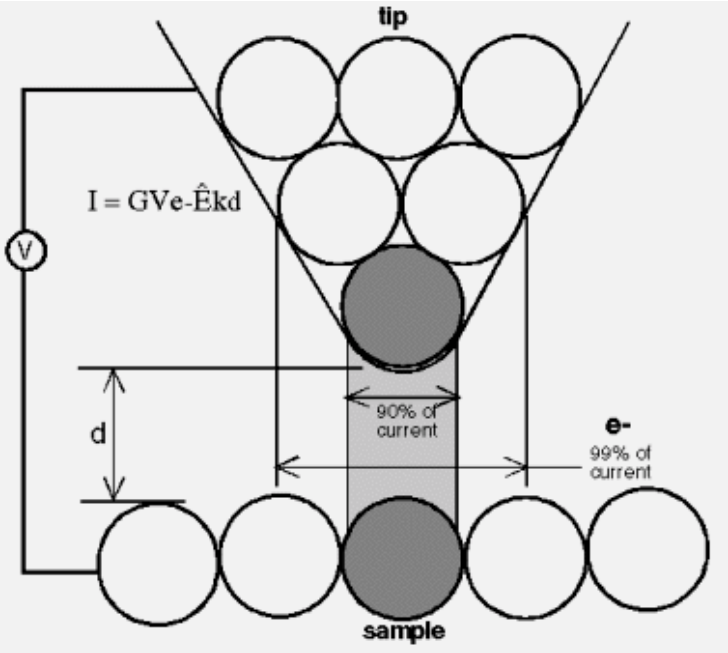
성분분석

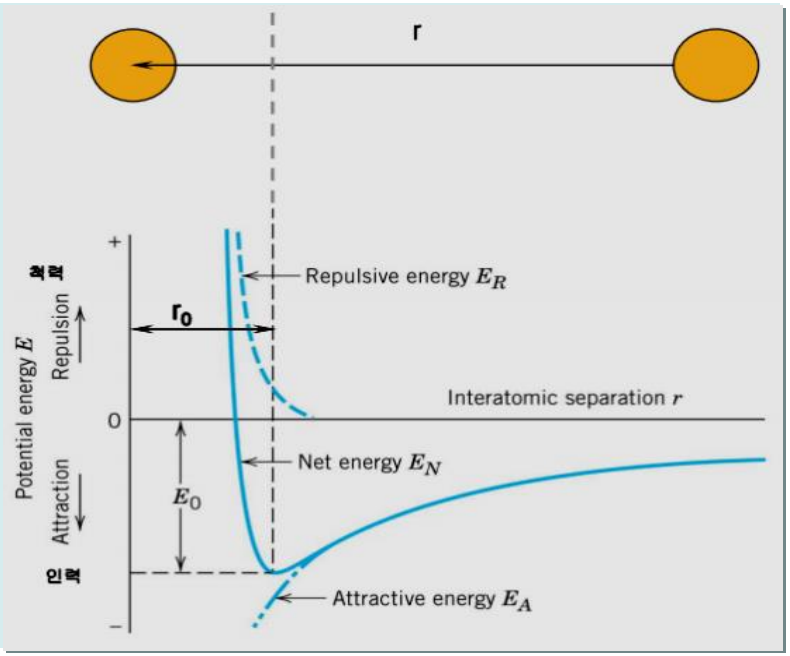


성분분석

탐침을 이용한 분석기술

탐침과 고체의 상호작용

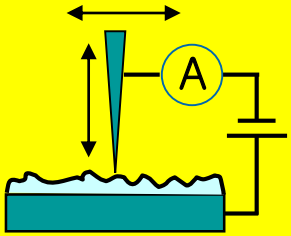
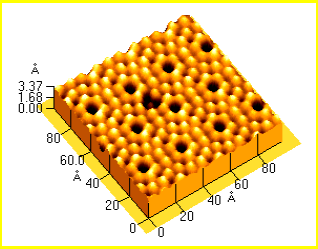
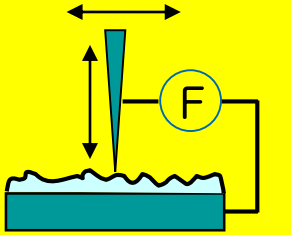
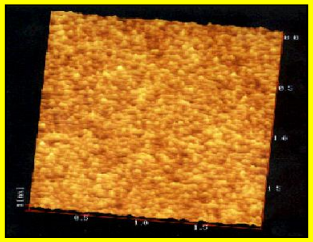
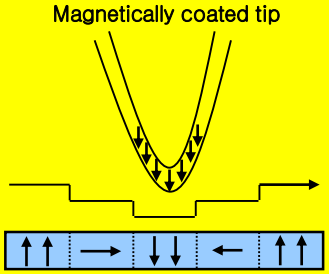
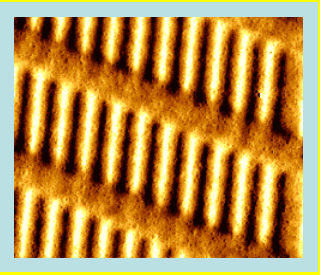
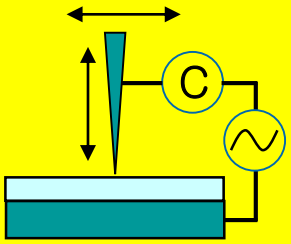
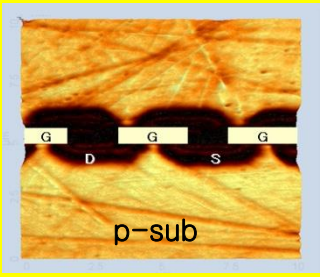




- Conductor Sample
- Distance ↓⇒ Tunneling Current ↑

- Atomic Force
- 탐침과 시료 사이의 거리가 가까워지면 인력이 증가하다가 매우 가까워지면 척력이 급격이 증가

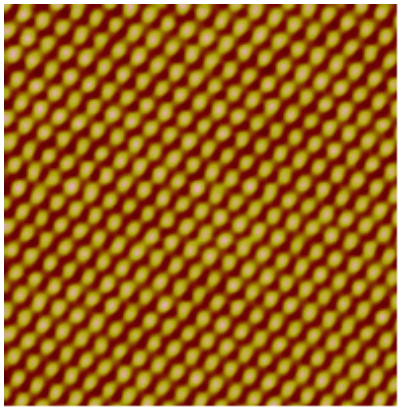
주사탐침현미경(SPM) 분석기술의 원리와 특징

분석기법	원리	측정 방법	측정 Data	측정 항목
주사터널링현미경 (Scanning Tunneling Microscopy)		•금속 탐침과 시료(도체, 반도체) 사이의 터널링 전류를 이용하여 표면의 높낮이를 측정		•Surface morphology •자기조립박막의 molecular imaging •Atomic imaging
원자력현미경 (Atomic Force Microscopy)		•탐침과 시료 원자간의 상호 작용 힘을 이용하여 접촉식 혹은 비접촉식 방법으로 국소적인 표면의 높낮이 변위를 측정		•Surface morphology •Grain size measurement •Line CD measurement
자기력현미경 (Magnetic Force Microscopy)		•자성체가 입혀진 탐침과 시료 표면의 자기적 성분에 의한 캔틸레버의 휨을 측정함으로써 자기 분포도를 관찰		•하드디스크 자기분포 관찰 •자성박막의 특성분석
주사정전용량현미경 (Scanning Capacitance Microscopy)		•Capacitance sensor를 이용하여 탐침과 시료 사이의 capacitance 변화를 측정함으로써 표면의 carrier나 charge density 분포를 관찰		•반도체 표면의 carrier 농도 분포 관찰 •반도체 소재의 결함분석

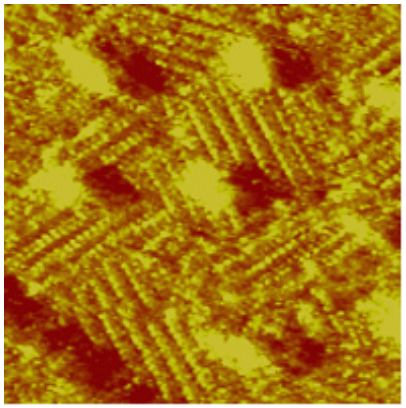
주사탐침현미경 분석

STM (Scanning Tunneling Microscopy)

1-Merpato-2-propanol SAMs on Au (111)



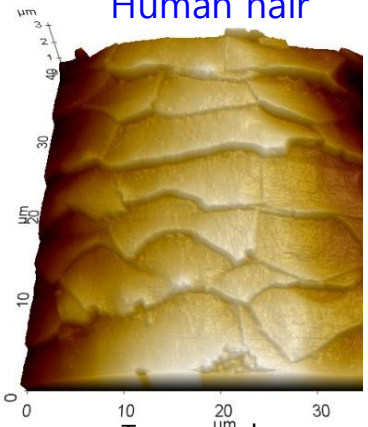
Au (111), 5nm x 5nm



SAMs on Au (111), 20nmx20nm

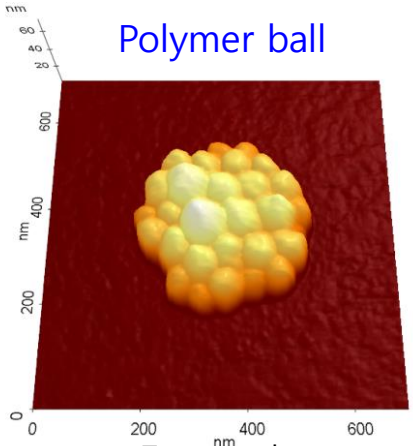
AFM (Atomic Force Microscopy)

Human hair



Topography

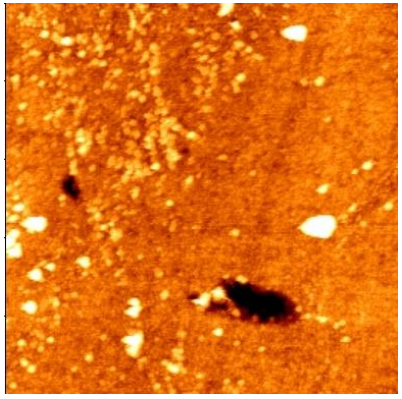
Polymer ball



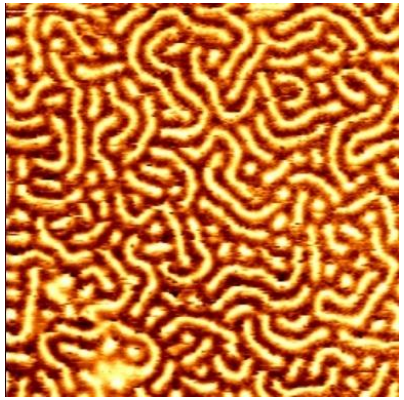
Topography

MFM (Magnetic Force Microscopy)

Alloy of Co/Pt



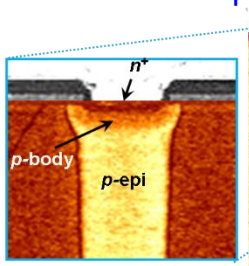
Topography (5umx5um)

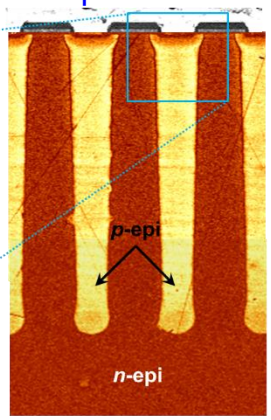


MFM

SCM (Scanning Capacitance Microscopy)

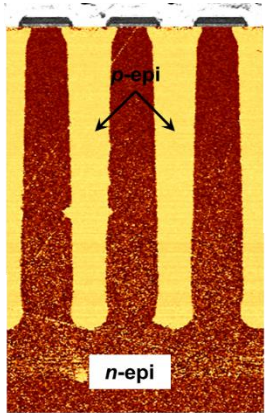
Dopant profile of MOSFET





(50um x 50um)

SCM Amplitude



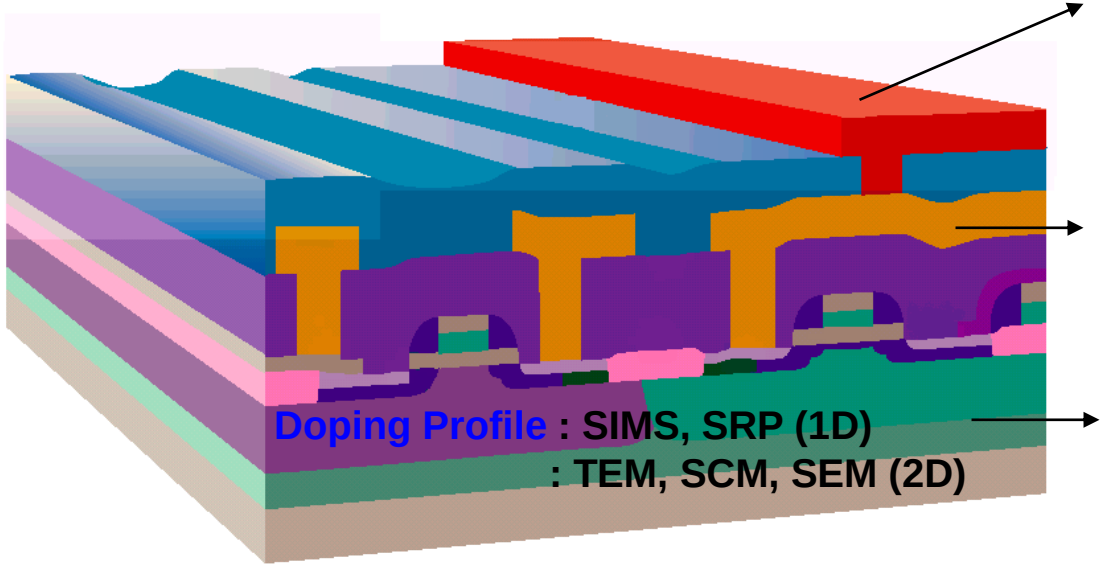
SCM Phase

Semiconductor Analysis Techniques

Morphology / Topography / Structure : SEM, TEM, AFM

Crystal Structure / Texture : XRD, ED

Particle Analysis : AES, XPS, SIMS, EDS (SEM, TEM), EELS (TEM)



Surface Impurity : SIMS, AES, XPS

Film Composition & Impurity :
SIMS, RBS, AES, XPS, EDS, EELS

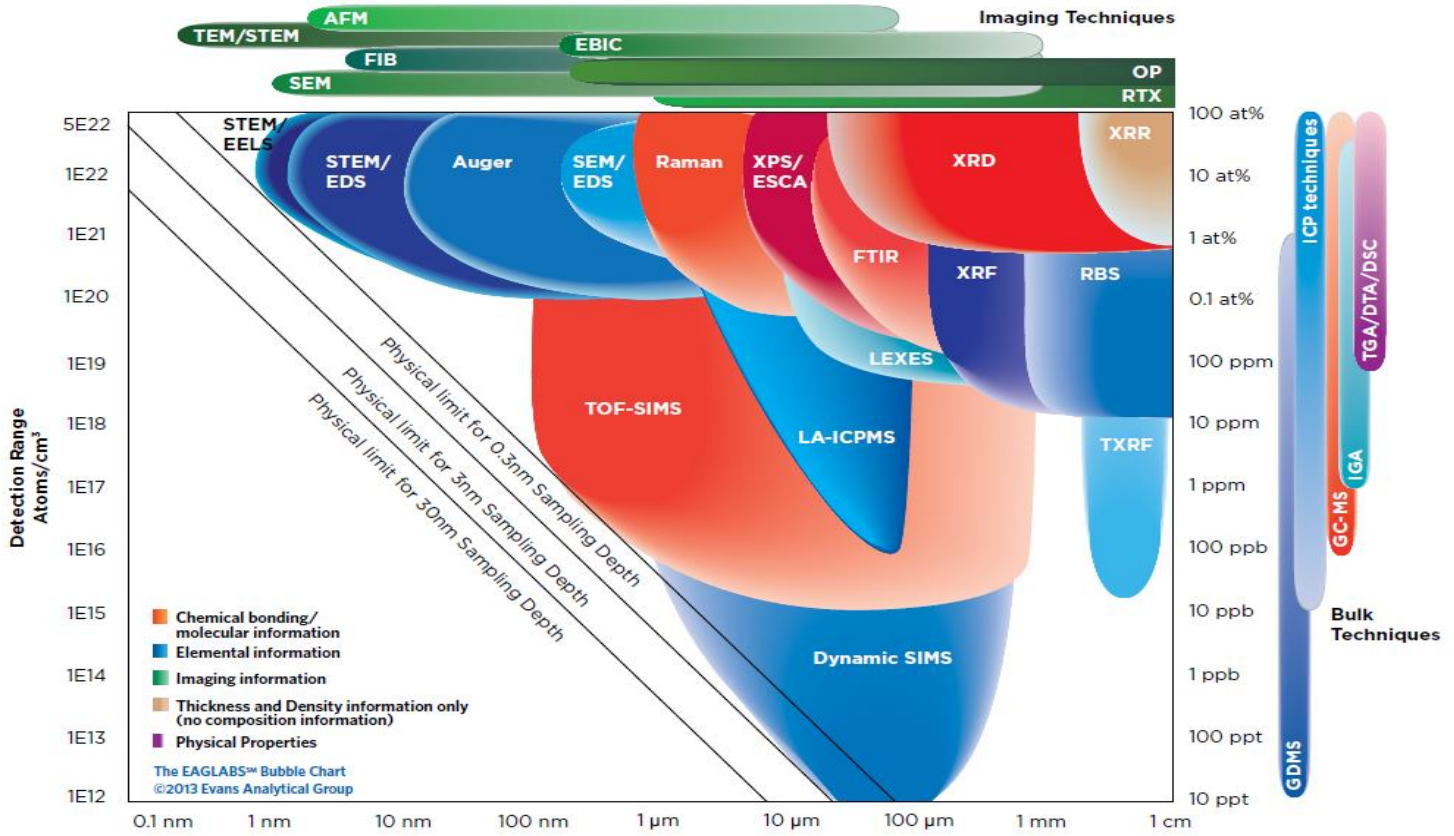
Doping Profile : SIMS, SRP (1D)
: TEM, SCM, SEM (2D)

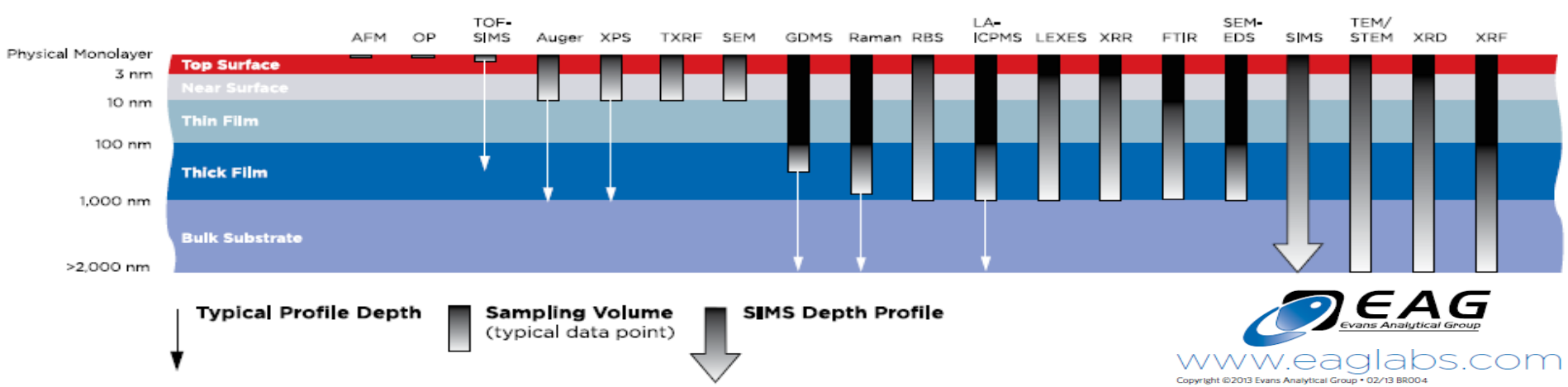
Bulk Impurity : SIMS

Chemical Structure : XPS, EELS

Stoichiometry : RBS

Analytical Resolution vs. Detection Limit





EAG Evans Analytical Group

www.eaglabs.com

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Summary

Analysis Method	Source	Energy	Signal	Type of Information	Resolution	Remarks
•SEM	Electron	•0.5-30 keV	Secondary Electron	•Morphology	•SR: ~1 nm	•Easy sample preparation
•TEM /STEM		•> 80 keV	Transmitted Electron	•Morphology •Crystal Structure	•SR: ~2 Å	•High resolution
•AES		•0.5-10 keV	Auger Electron	•Elemental/Chemical •Li-U	•SR: < 15 nm •DR: < 2 nm	•Small probe size
•SEM/EDS		•0.3-30 keV	Characteristic X-ray	•Elemental •B-U	•SR: ~0.1 ㎛	•Better for several atomic %
•STEM/EDS		•> 80 keV			•SR: ~1 nm	•Nanoscale analysis
•M-SIMS	Cs, O Ion	•1-15 keV	Secondary Ion	•Elemental •All elements detect	•SR: > 30 ㎛ •DR: 2-30 nm	•Composition analysis •Best sensitivity
•FIB	Ga Ion	•1-40 keV	Secondary Electron or Ion	•Morphology	•SR: > 5 nm	•Milling, Deposition, TEM sample prep.
•RBS	He Ion	•> 1 MeV	Scattered Ion (He)	•Elemental •Li-U	•SR: 2 mm •DR: 1-10 nm	•Precise atomic % without standards •Non destructive, simultaneous depth and composition
•XRD	X-ray	•60 kV	Diffracted X-ray	•Crystal Structure		•Texture, residual stress •Grain size measurement •Etc.
•XPS		•> 1 keV	Photoelectron	•Elemental/Chemical •Li-U	•SR: 10 ㎛ •DR: 1-10 nm	•Chemical bonding •Valence States
•XRF		•수십 keV	Characteristic X-ray	•Elemental •B-U		•비파괴분석 •EDS에 비해 미량 성분 검출
•STM	Probe (SPM)		Tunneling Current	•Surface Topography	•LR: < 0.1 nm •DR: 0.01 nm	•금속 Tip, 금속시료
•AFM			Atomic Force	•Surface Topography	•LR: < 10 nm •DR: 0.01 nm	•가장 많이 이용됨
•MFM			Magnetic Force	•Domain Structure	•LR: < 20 nm	•시료 표면의 미세 자기 구조

Summary

형상 및 구조분석 장비	성분 및 화학분석 장비	물성 및 특성분석 장비
주사전자현미경 **(Scanning Electron Microscope, SEM)** •시료에 1~20 kV의 전자를 입사시켜 시료표면에서 나오는 2차전자를 검출 • 시료표면의 형상을 1 nm 수준에서 관찰 • EDS 부착하여 미소영역의 성분분석	**오제이전자분광기** **(Auger Electron Spectrometer, AES)** •시료에 전자선을 입사시켜 여기 방출되는 원소 특유의 오제이전자의 에너지의 양을 측정 •조성과 불순물의 분석&표면분석 •깊이방향의 성분분포 분석	**전기특성 측정시스템** **(Electrical Characterizer)** • I-V, C-V 측정 등 • 접합과도용량분광법(DLTS) – 시료의 깊은 준위 및 활성화 에너지 측정 • 시료의 결함특성 분석
투과전자현미경 **(Transmission Electron Microscope, TEM)** •수백 keV의 전자를 시료에 투과시켜 투과전자와 회절되는 전자를 이용하여 영상화 • 시료내부의 구조를 0.1 nm 수준에서 관찰 • EDS & EELS를 이용한 나노영역 성분분석	**X선광전자분광기** **(X-ray Photoelectron Spectrometer, XPS)** •시료에 X선을 입사시켜 X선에 의해 여기된 전자의 에너지의 양을 측정 •조성과 불순물의 분석 & 화학결합 상태 분석 •깊이방향의 성분분포 분석	**나노프로빙시스템** **(Nano-probing System)** •전자현미경을 이용한 나노스케일 영역에서 전기적 특성을 분석 • I-V, C-V 측정 등 •시료의 결함 특성 분석
집속이온빔시스템 **(Focused Ion Beam System, FIB)** •수십 keV의 집속된 이온으로 시료를 식각하고 방출되는 2차전자&이온으로 영상화 • 특정 불량위치의 단면관찰 •EDS 부착하여 관심 영역의 성분분석	**X선형광분석기** **(X-Ray Fluorescence Analyzer, XRF)** •시료에 수십 keV의 X선을 입사시켜 방출되는 원소 고유의 특성X선을 검출 •비파괴적이며 신속한 분석 & 시편준비 용이 •넓은 분석 범위(ppm~%)	**4단자탐침기** **(4-point Probe)** •4개의 직렬 탐침을 사용하며 내부 2탐침에서 전위차와 외부 2탐침에 흐르는 전류를 측정 •박막의 면저항, 비저항 측정
주사터널링현미경 **(Scanning Tunneling Microscope, STM)** •금속탐침과 시료(도체, 반도체) 사이의 터널링 전류를 이용하여 표면의 구조분석 •표면형상 측정(LR: <0.1 nm, DR: 0.01 nm) •원자&분자 영상	**2차이온질량분석기** **(Secondary Ion Mass Spectrometer, SIMS)** •1-20 kV의 1차이온을 시료에 입사시켜 방출되는 이차이온의 질량을 분석 •불순물의 질량 분석과 극미량 조성의 측정 •깊이 방향의 불순물 분포 측정	**광발광 분광기** **(Photoluminescence Spectrometer, PL)** •입사광에 의해 여기된 전자가 재결합하는 과정에서 방출되는 빛스펙트럼을 분석 • 시료 또는 불순물의 전자구조적 특성 분석 • 시료의 결함특성 및 발광특성 분석

Summary

형상 및 구조분석 장비	성분 및 화학분석 장비	물성 및 특성분석 장비
원자력현미경 **(Atomic Force Microscope, AFM)** •탐침과 시료의 원자간 힘의 세기를 접촉 및 비접촉 방법으로 측정 •표면형상 측정(LR: <10 nm, DR: 0.01 nm) •결정립 크기, 배선 CD 등 측정	**러터퍼드 후방산란분광기** **(Rutherford Backscattering Spectrometer, RBS)** •수 MeV로 가속된 이온을 시료에 입사시켜 방출되는 이온의 에너지의 양을 검출 •재료의 조성과 불순물의 깊이 방향 분포 결정 •표준시편 없이 절대정량 분석 가능	**나노압입시험기** **(Nanoindentor)** •압자를 소재에 압입하면서 하중과 변위를 연속적으로 측정하고, 압흔의 면적을 유추 •시료의 경도&탄성계수 측정 •잔류응력&유동곡선 측정
X선회절기 **(X-Ray Diffractometer)** •시료에 X선을 입사시켜 Bragg 조건을 만족하는 X선을 검출 • 결정구조, 우선방위, 잔류응력 측정 • 박막두께 및 밀도&거칠기(XRR 기법)	**3차원 원자현미경** **(3D-Atom Probe)** •탐침시료에 레이저 등을 이용하여 증발시킨 원자의 무게 및 위치를 분석 •3차원적으로 원자의 종류 및 위치 분포 측정 •Sub nm 수준의 공간분해능, 10 ppm 수준의 검출한계	**자외-가시선 분광광도계** **(UV-Visible Spectrophotometer)** •UV-Vis 영역의 빛을 조사, 원자 혹은 분자에서 전자전이 과정 중 특정파장의 흡광도 측정 •원자 및 분자의 전자구조, 조성 분석 •흡광도&원자 및 분자의 농도 분석
공초점현미경 **(Confocal Microscope)** •레이저광원과 핀홀을 이용해 초점이 맞은 영상과 공간의 정보로부터 절편상을 재현 • 비파괴적으로 세포나 조직의 단층상 분석 • 3차원으로 시료의 위치나 내부구조 분석	**라만분광기** **(Raman Spectrometer)** •레이저 이용한 단일 파장의 입사광에의한 분자의 진동으로 에너지가 변화된 산란광을 검출 •분자의 결합종류 및 작용기 분석 •유기 및 무기 불순물 또는 반응생성물 분석	**나노입자분석기** **(Nanoparticle Size Analyzer)** •동적광산란법(DLS)을 이용하여 나노입도 측정(습식) •콜로이드나 단백질 등 나노입자 입도, 무게, 분포, 집합도 분석(측정범위: 1~수천 nm)
타원편광해석기 **(Ellipsometer)** •빛이 반사, 투과한 후 편광상태 변화를 측정하여 빛의 파장에 물질의 굴절률 측정 • 박막의 두께를 0.1 nm 수준으로 측정 • 굴절률과 유전함수 측정, 비파괴적 측정	**푸리에변환 적외선분광기** **(Fourier Transform Infrared Spectrometer, FT-IR)** •시료에 적외선 광원을 입사시켜 각 파장의 흡광도를 얻고 주파수에 따른 스펙트럼으로 분석 •분자의 결합종류 및 작용기 분석 •미량 불순물 분석	**레이저회절 입도분석기** **(Laser Scattering Particle Size Analyzer)** •레이저회절을 이용하여 입도측정(건식, 습식) •각종 분체 및 액체상의 입자 크기 및 입도 분포 측정(측정범위: 0.1~수천 μm)

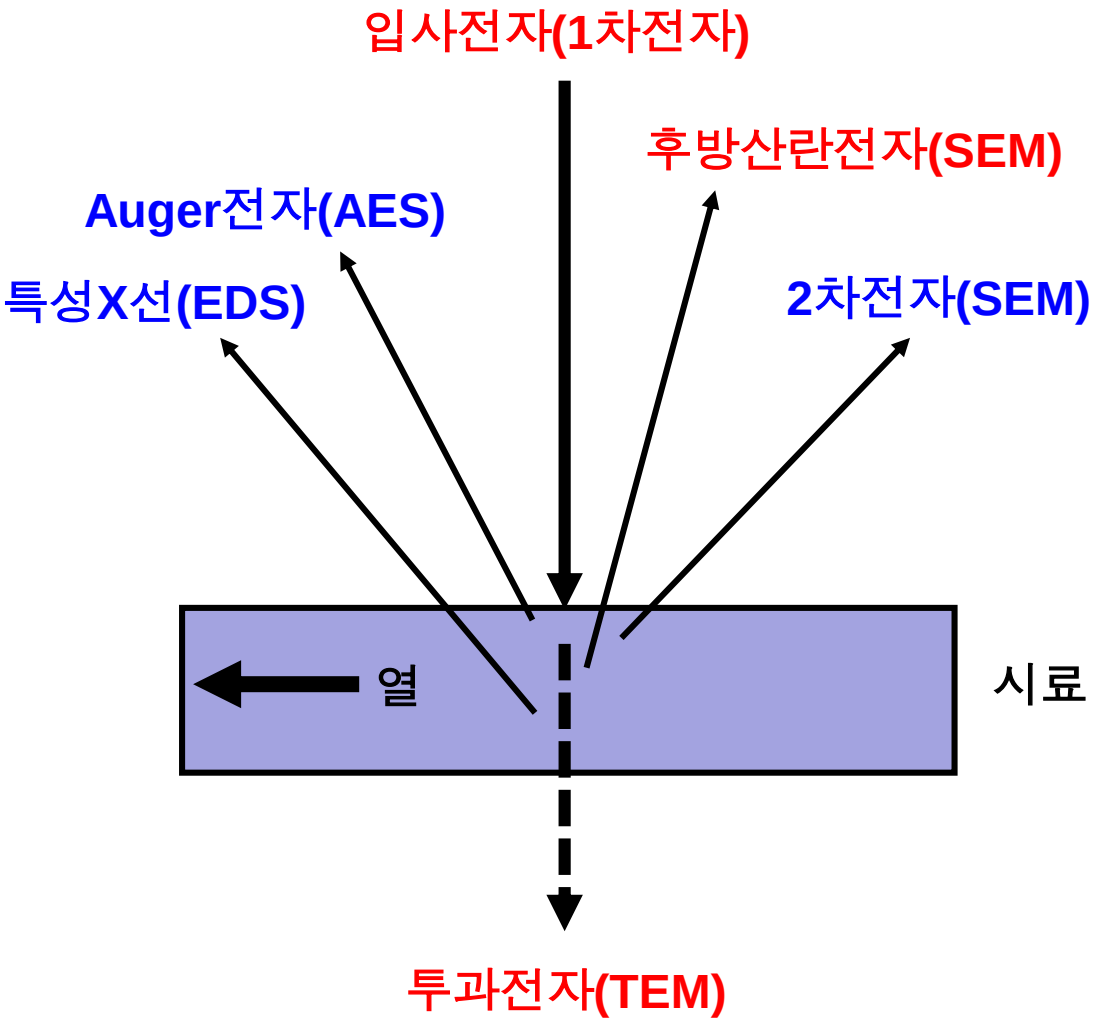
나노분석의 기초 및 응용

- 나노분석의 기초

- **구조분석(SEM, TEM, FIB)**

- 표면분석(XPS, AES, SIMS)

전자와 고체의 상호작용

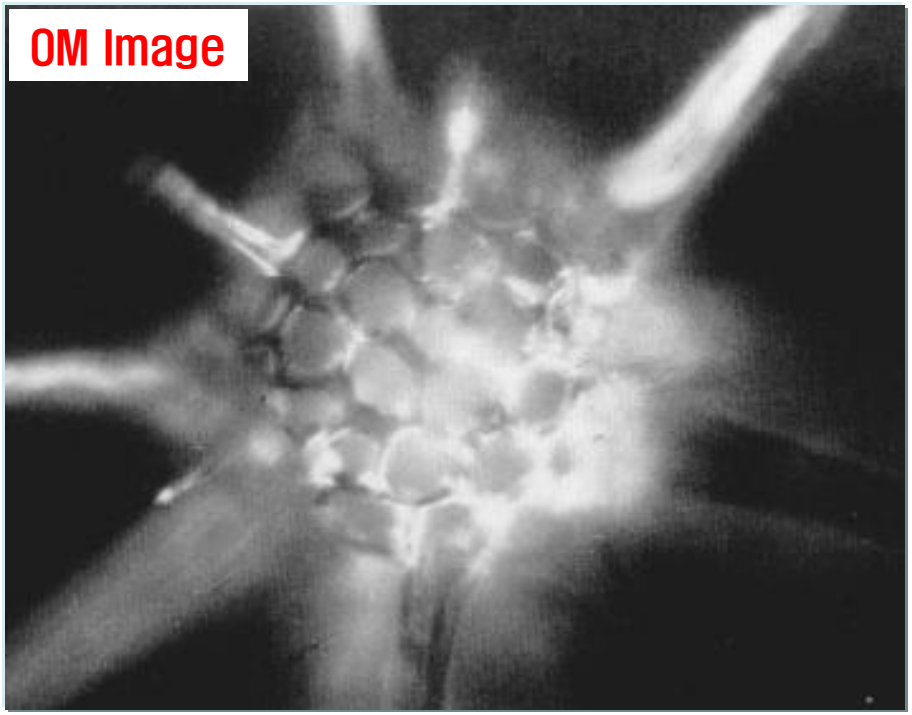

입사전자(1차전자)
후방산란전자(SEM)
Auger전자(AES)
특성X선(EDS)
2차전자(SEM)
열
시료
투과전자(TEM)

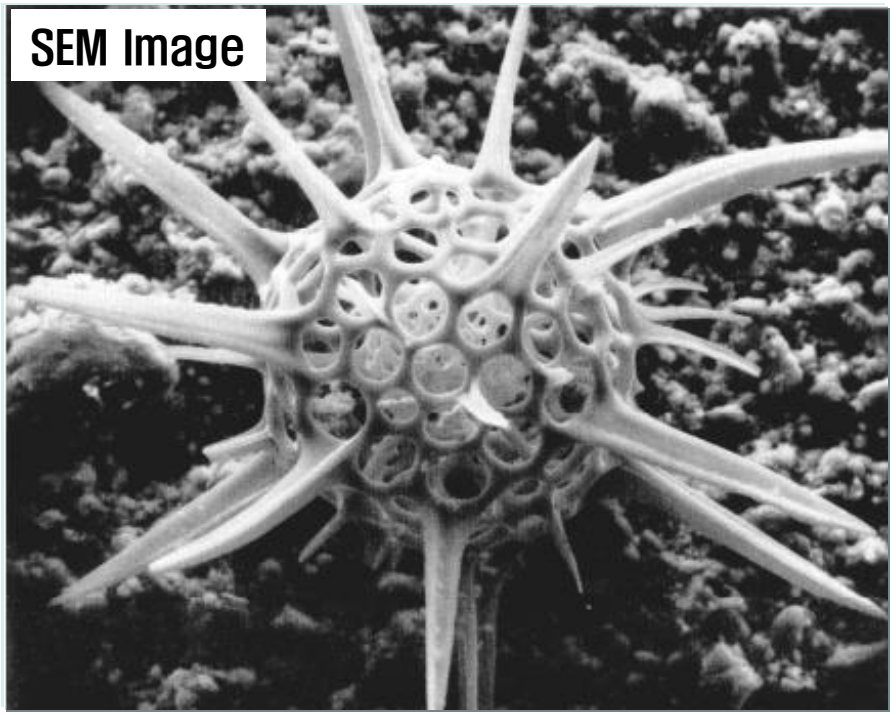
走査電子顕微鏡 분석

Scanning Electron Microscopy (SEM)

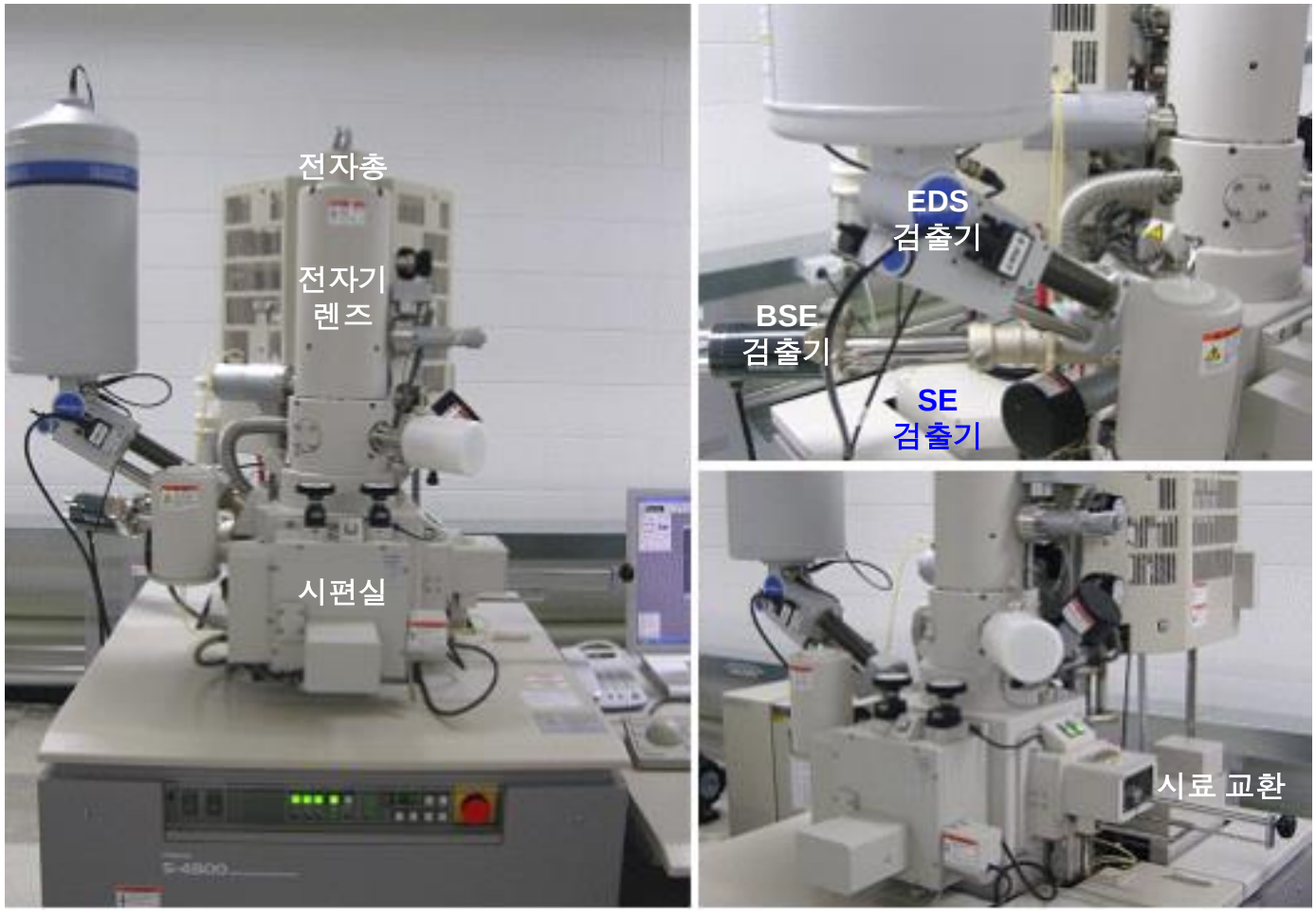
주사전자현미경(SEM)의 개요

- **광학현미경(Optical Microscope)**
  - 분해능이 좋지 않음
  - 초점심도가 좋지 않음
- **주사전자현미경(Scanning Electron Microscope)**
  - 분해능이 우수
  - 초점심도가 좋음





주사전자현미경(SEM) 분석의 개요 : 장치



- **SE (Secondary Electron, 이차전자)**
  : 입사 전자빔이 시편과 충돌하여 전자빔과 시편의 비탄성 충돌에 의해 시편에서 발생하는 50 eV 이하의 에너지를 갖는 전자

- **BSE (Backscattered Electron, 후방산란전자)**
  : 입사 전자빔이 시편과 충돌하여 입사전자가 후방으로 방출되는 전자

주사전자현미경(SEM)의 개요

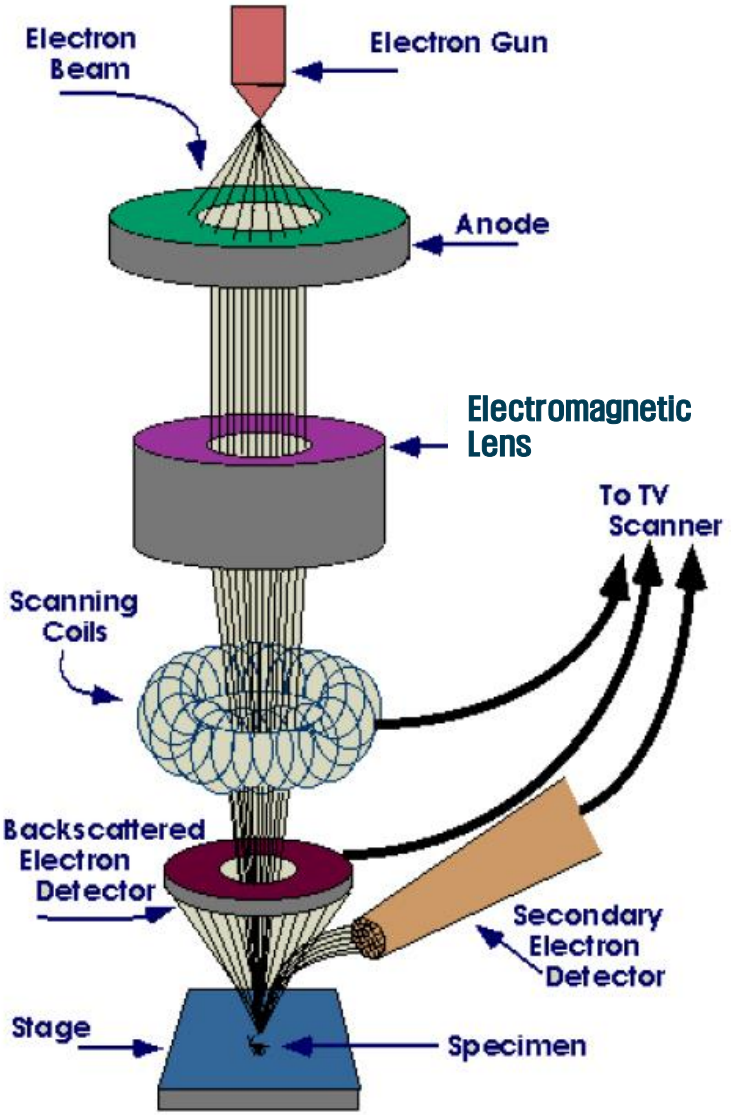
분석 특징 및 분석 기법	분석능	배율
• 표면 형상 및 구조의 관찰 • SE와 BSE 영상 • EDS 원소분석 (Point, Line, Mapping)	• SEM의 공간분해능: ~1 nm • EDS 원소 분석의 검출 범위 : C-U	• SEM의 배율 $M = L_{CRT} / L_{Specimen}$

재료 및 나노소자의 **형상 및 형태 관찰**

미세 Particle 및 오염물질의 **위치 추적 및 형상 분석**

EDS를 이용한 **물질 및 박막의 조성 분석**

주사전자현미경(SEM) 분석의 개요 : 장치



전자총에서 전자발생

↓

수십 keV의 에너지로 가속
(~수백 eV ~30 keV)

↓

전자기렌즈로 집속

↓

시료표면에 조사 및 주사

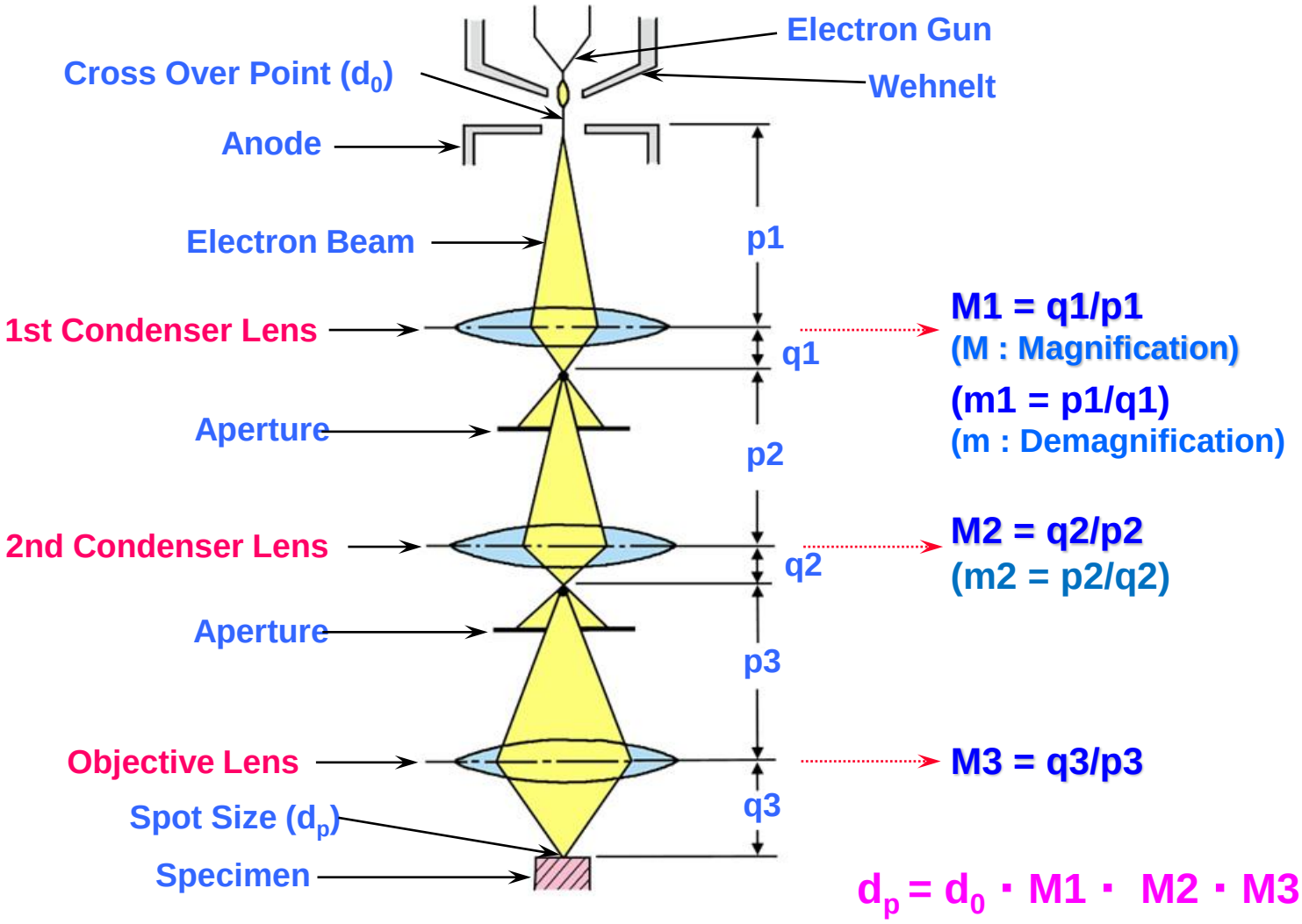
↓

검출기에서 SE/BSE 신호 검출
(X선, 발광, 전류 등도 검출)

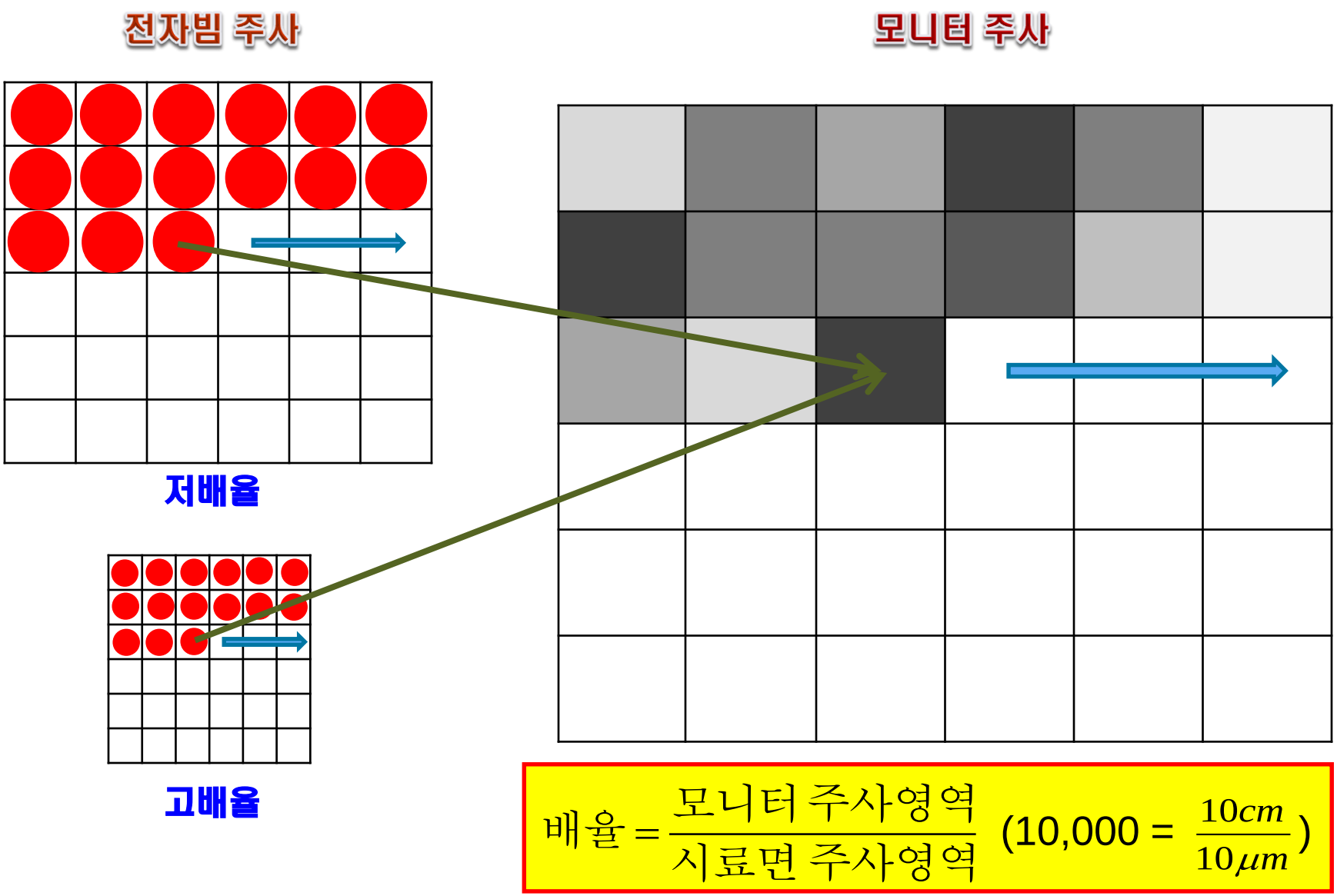
↓

증폭된 전류신호를 모니터와
동기화시켜 영상 형성

전자렌즈계

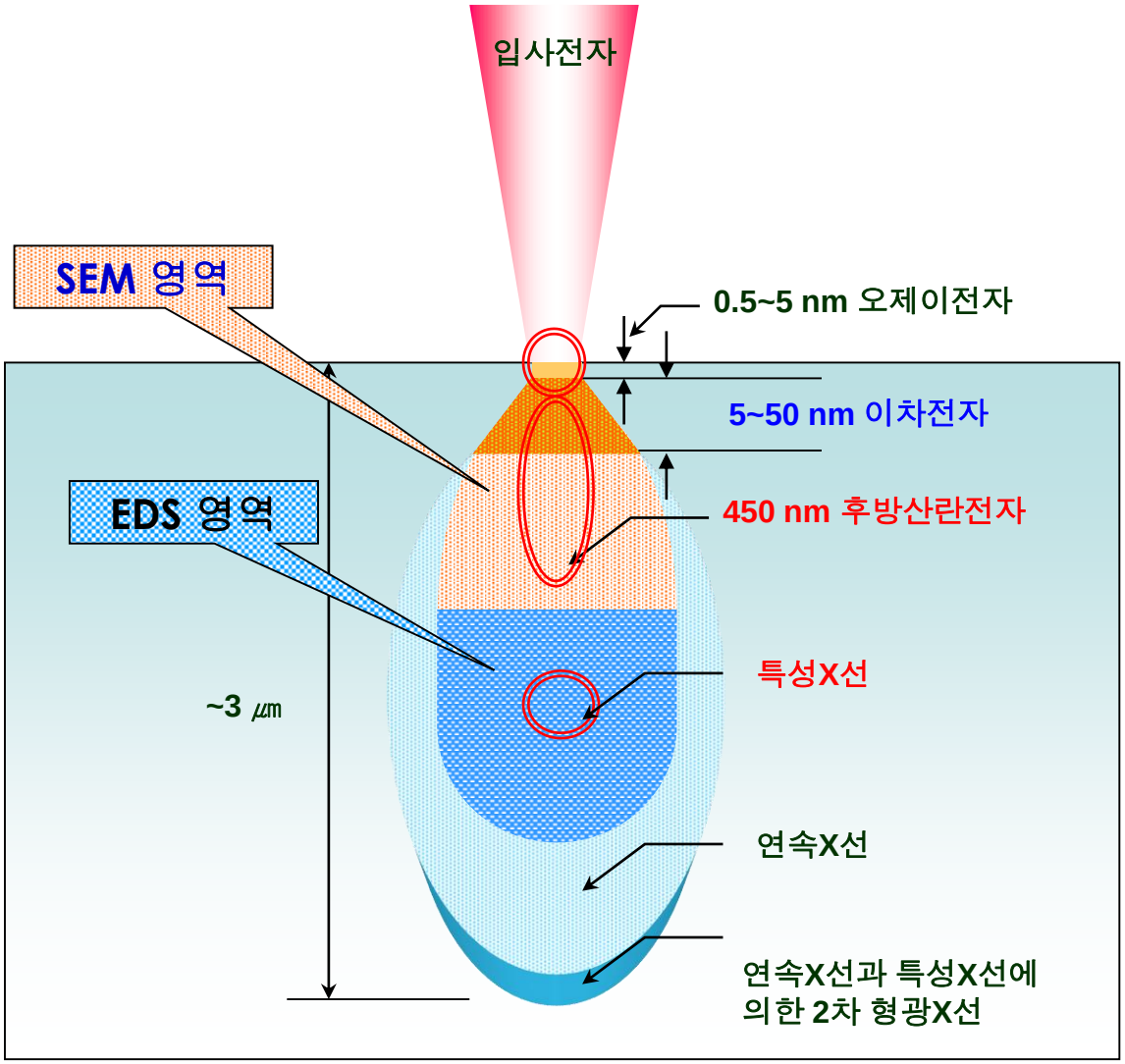

Electron Gun
Wehnelt
Cross Over Point (d_0)
Anode
Electron Beam
p1
1st Condenser Lens
q1
M1 = q1/p1
(M : Magnification)
(m1 = p1/q1)
(m : Demagnification)
Aperture
p2
2nd Condenser Lens
q2
M2 = q2/p2
(m2 = p2/q2)
Aperture
p3
Objective Lens
M3 = q3/p3
q3
Spot Size (d_p)
Specimen
$d_p = d_0 \cdot M1 \cdot M2 \cdot M3$

주사전자현미경(SEM)의 배율 확대 원리



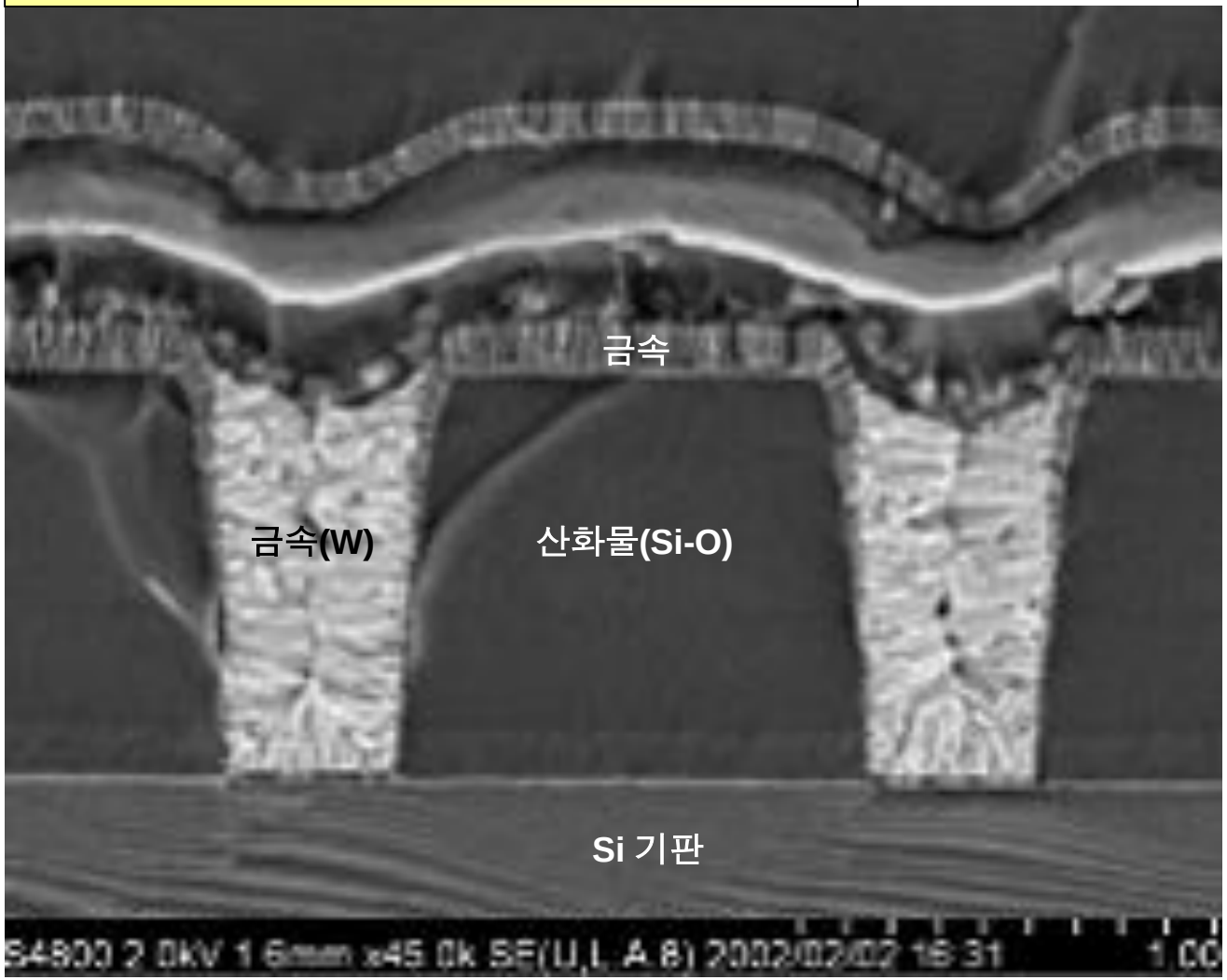
$$\text{배율} = \frac{\text{모니터 주사영역}}{\text{시료면 주사영역}} \quad \left(10{,}000 = \frac{10cm}{10\mu m}\right)$$

상호작용 부피와 신호


입사전자
SEM 영역
0.5~5 nm 오제이전자
5~50 nm 이차전자
EDS 영역
450 nm 후방산란전자
특성X선
~3 ㎛
연속X선
연속X선과 특성X선에
의한 2차 형광X선

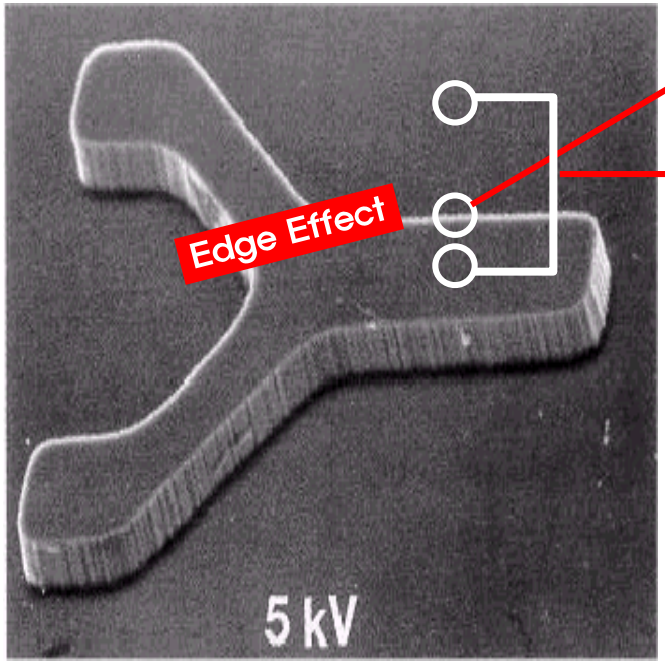
이차전자(SE) 발생차이에 의한 영상 콘트라스트

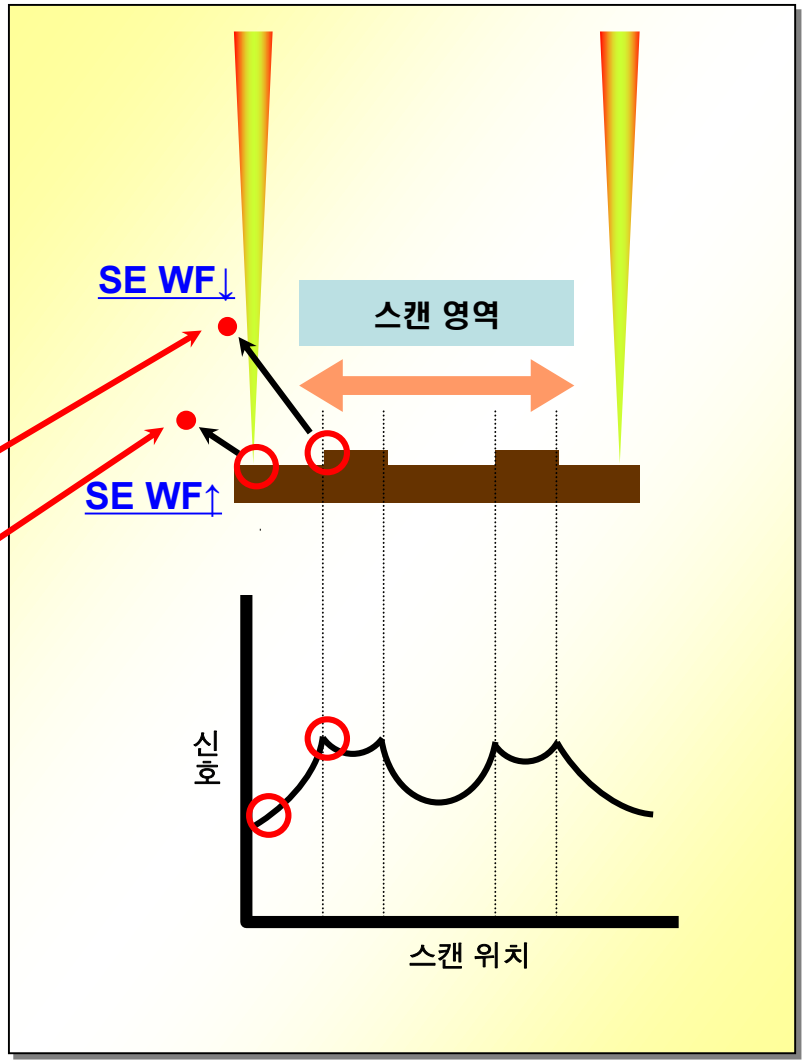
● 이차전자(SE)의 신호는 외부 방출 일함수에 의해 좌우됨



표면형상 관찰의 원리

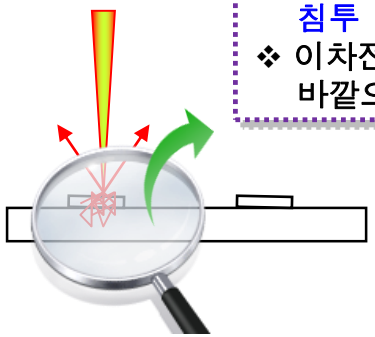
- 이차전자(SE)의 신호는 외부 방출 일함수에 의해 좌우됨
- SEM 영상의 형태적인 정보는 시편 표면에서 이차전자 방출 일함수(WF) 차이, 엣지효과(Edge Effect)에 따른 신호의 절묘한 형태화임



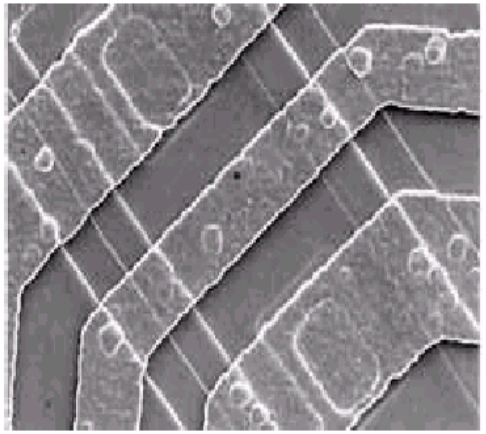


입사전자와 시편의 각도에 따른 이차전자 신호의 변화

❖ 전자빔이 시편 내로 깊게 침투
❖ 이차전자들이 시편 바깥으로 나오기 어려움

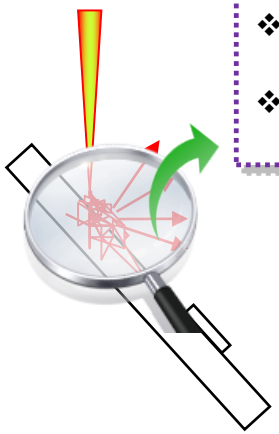


신호가 적음

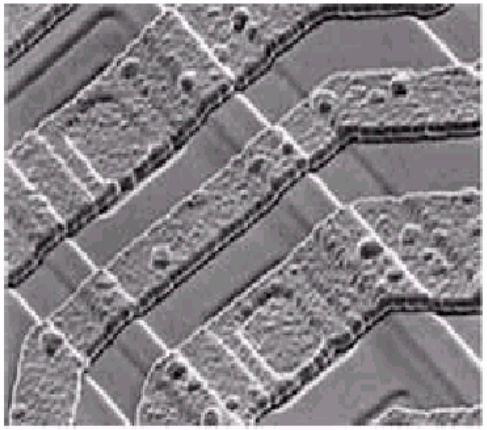


(a) Tilt angle: 0°

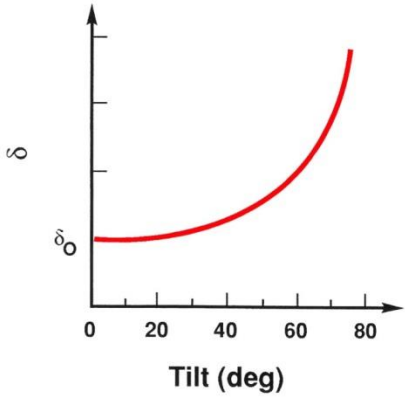
❖ 전자빔이 시편 내로 깊게 침투하지 못함
❖ 많은 수의 이차전자들이 시편 바깥으로 나옴



신호가 큼



(b) Tilt angle: 45°

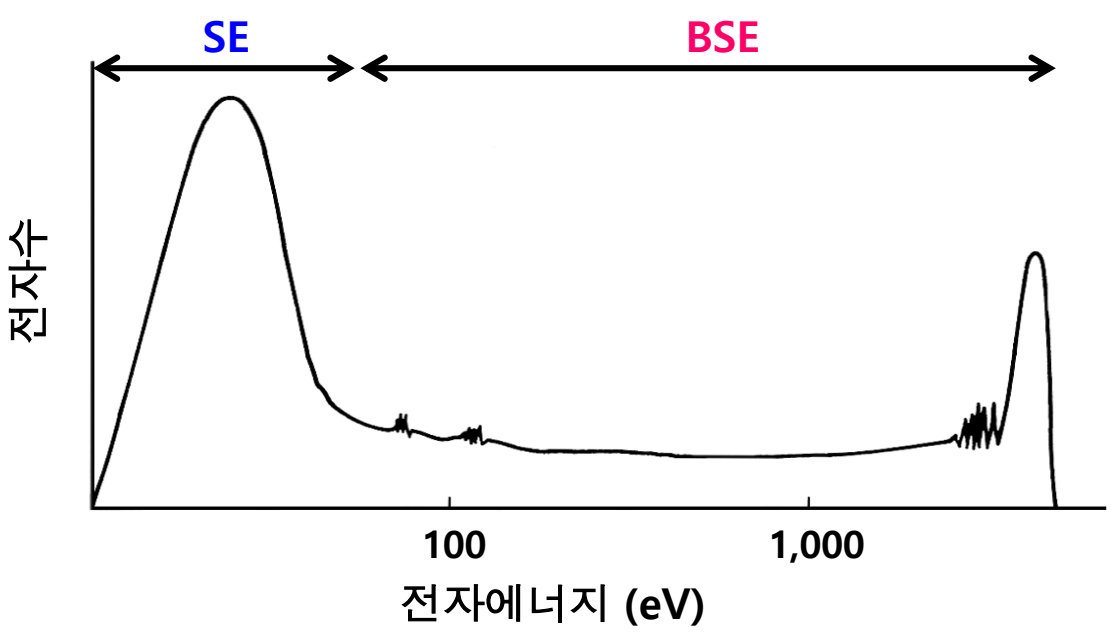


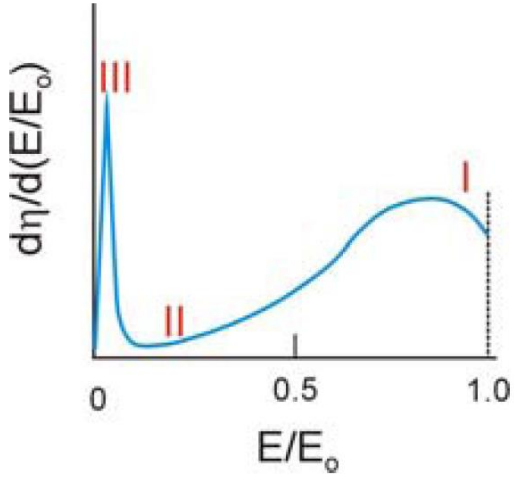
- 이차전자(SE)의 생성 및 탈출은 전자빔 경로에 비례
- 기울어진 경로에서 더 많은 SE가 발생 밝게 보임

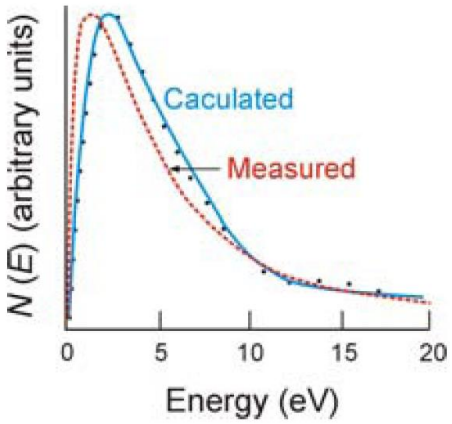
이차전자(SE)와 후방산란전자(BSE)

● **이차전자(SE)**
: 입사 전자빔이 시편과 충돌하여 전자빔과 시편의 비탄성 충돌에 의해 발생하는 50 eV 이하의 에너지를 갖는 전자

● **후방산란전자(BSE)**
: 입사전자가 시편에 조사될 때 시편과 충돌하여 되돌아 나오는 전자



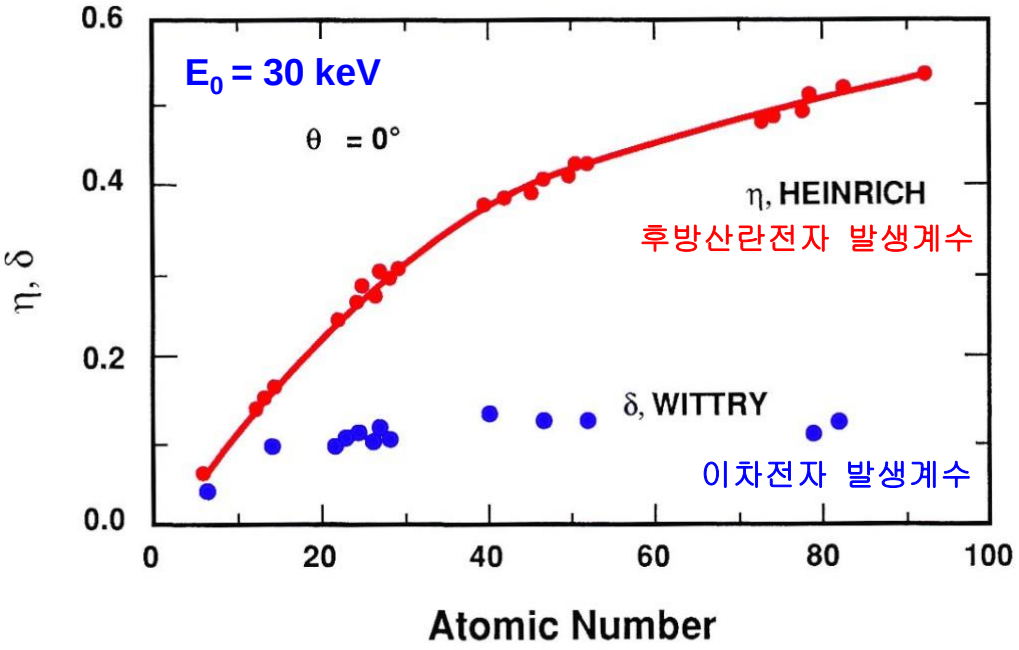




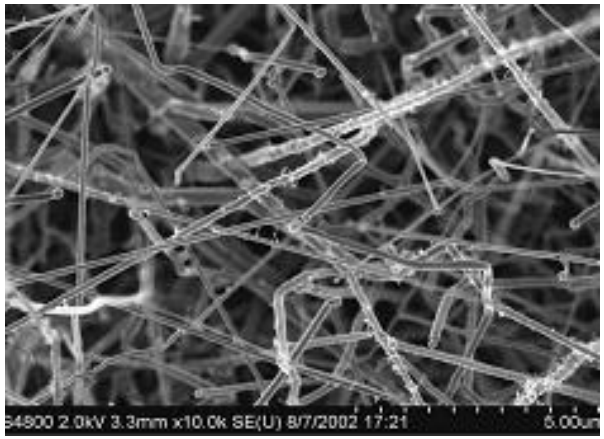
- **SE : 0 ~ 50 eV (2~5eV, SE의 90% 이상이 10 eV 이하)**
- **BSE : 0 ~ 가속전압**

- **SE 방출 최대 깊이는 평균자유행로(λ)의 5배 정도임**
- **일반적인 금속의 1λ = 1 nm, 절연체 1λ = 10 nm**
- **SE 이탈 깊이는 BSE 이탈 깊이의 1/100 수준임**

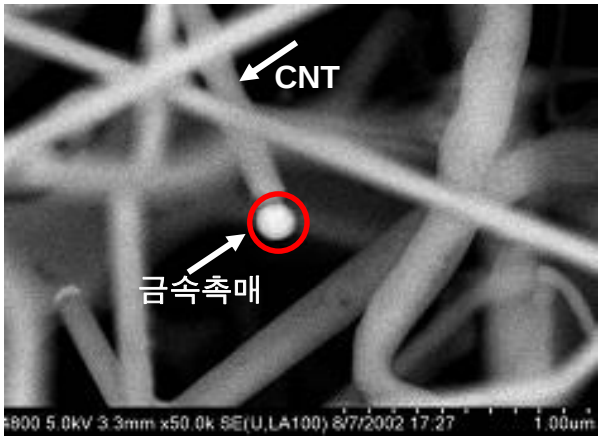
이차전자(SE)와 후방산란전자(BSE)



- SE는 원자번호 증가에 따라서 발생계수 차이 거의 없음
- BSE는 원자번호 증가에 따라서 발생계수 증가하는 경향
  : 원자번호(Z) 콘트라스트를 보임



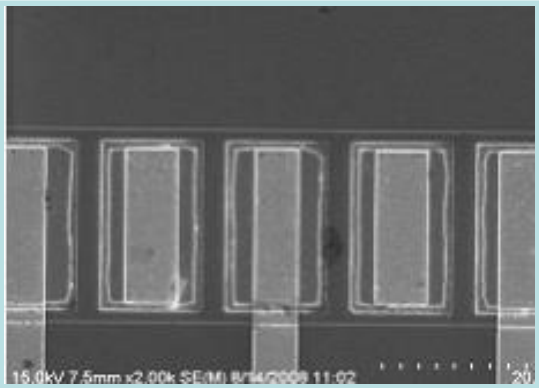
이차전자 영상

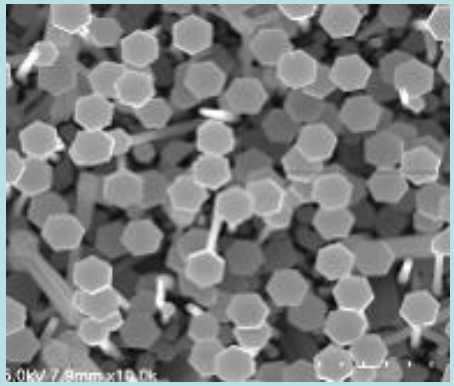


후방산란전자 영상

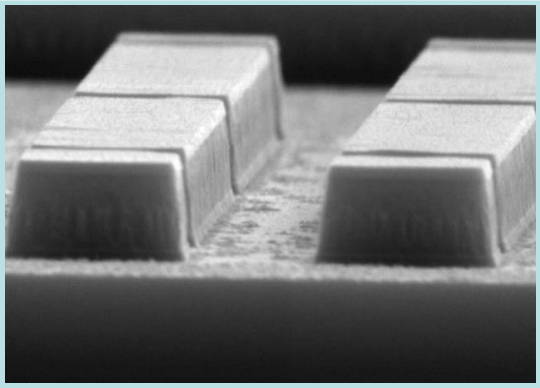
주사전자현미경(SEM)을 이용한 관찰

● **형상 관찰 및 크기 측정**

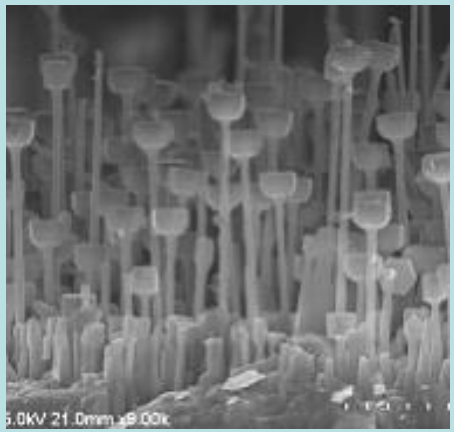




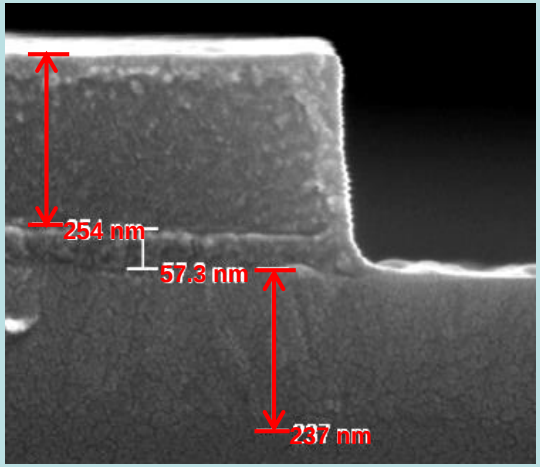
표면 형상의 관찰







단면 구조의 관찰



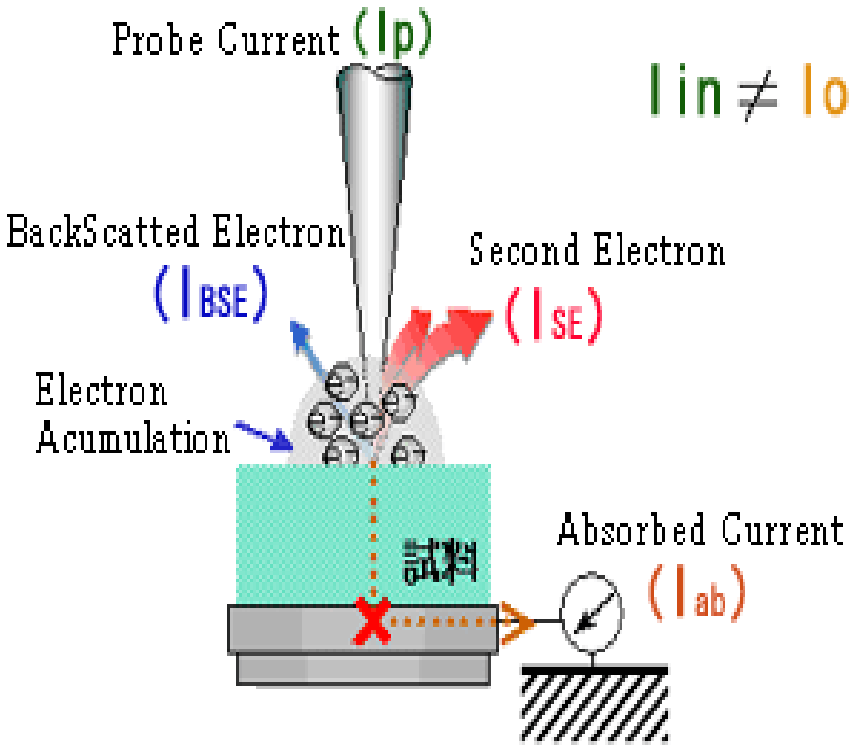
두께의 측정

대전효과 (Charging Effect)

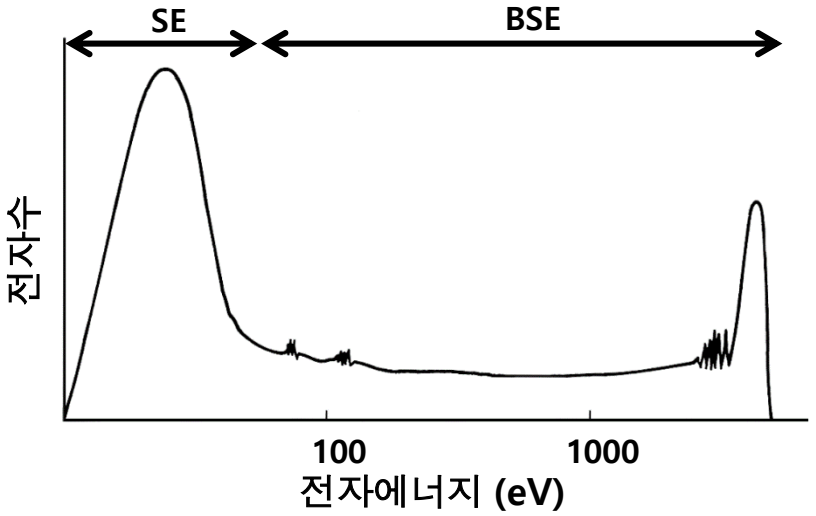
$I_{in} = I_p$

$I_{out} = I_{SE} + I_{BSE} + I_{ab}$

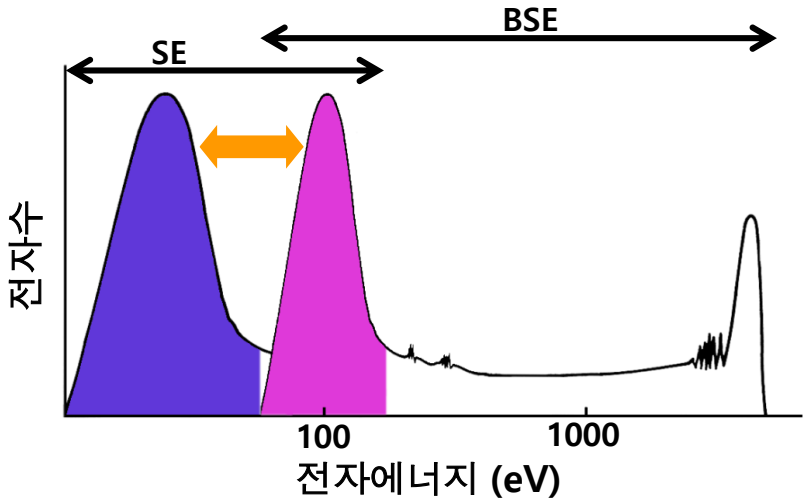
$I_{in} \neq I_{out}$



Energy of SE and BSE



Charge-up sample의 경우

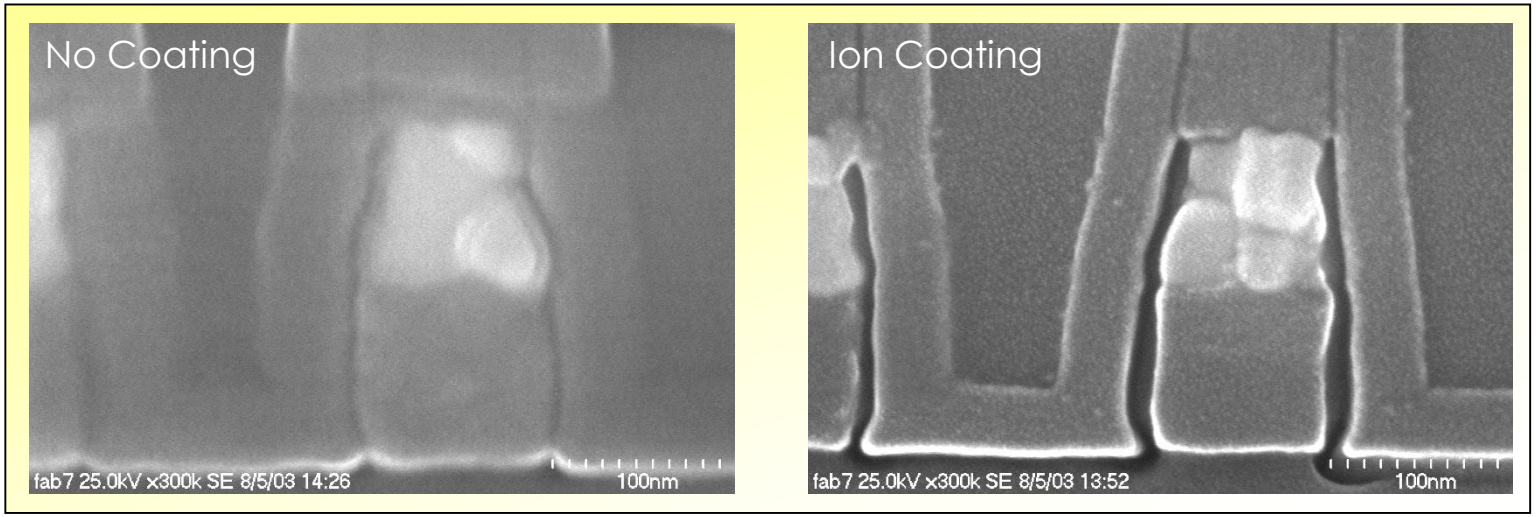


대전효과를 줄이는 방법

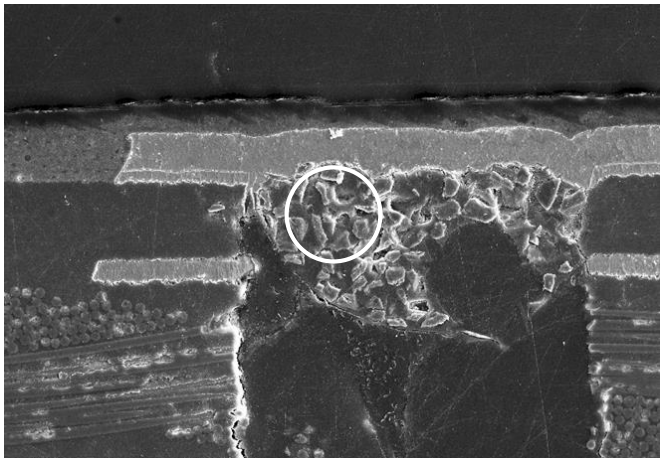
- **BSE mode or Lower detector**
  - BSEs have high energy so image is not sensitive to charge-up
- **Capture in fast scan (Picture integration function)**
- **Decrease I_p → I_{in}↓**
- **Decrease V_{acc} → I_{in}↓**
- **Coating (Pt, Cr, Au, C, Au/Pd etc.)**

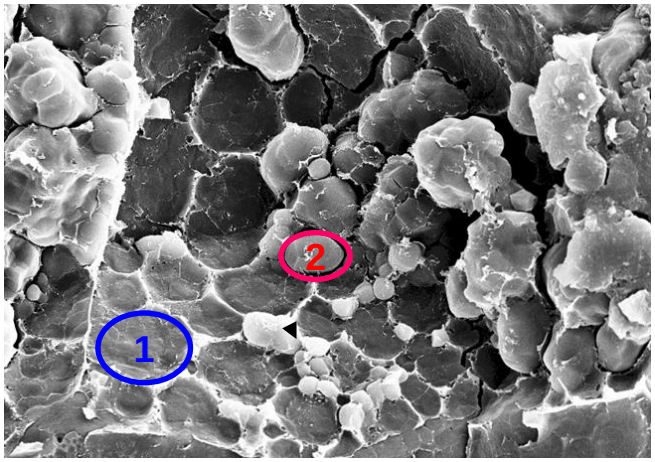
이온코팅을 통한 대전효과 방지 : 영상의 개선

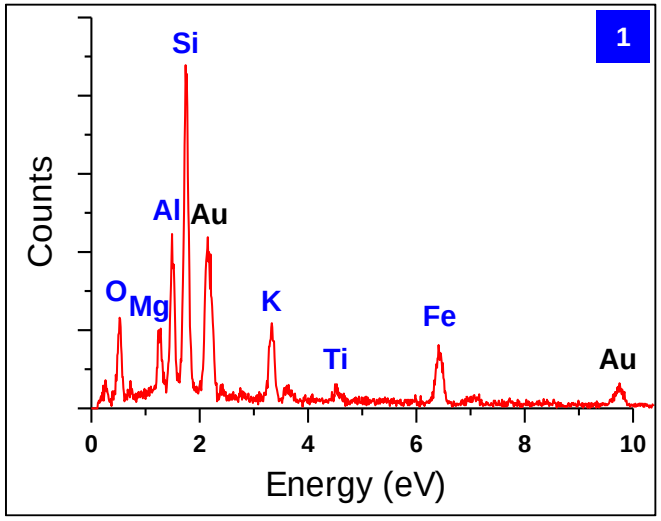
- **시편의 처리 방법 : 이온 코팅 – 시편의 대전(charging) 효과 방지를 위한 시편 표면의 코팅**

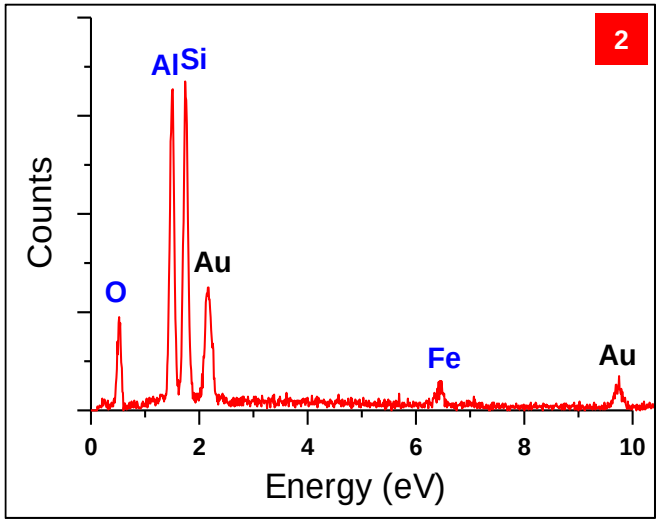


SEM-EDS 성분분석 : 점분석



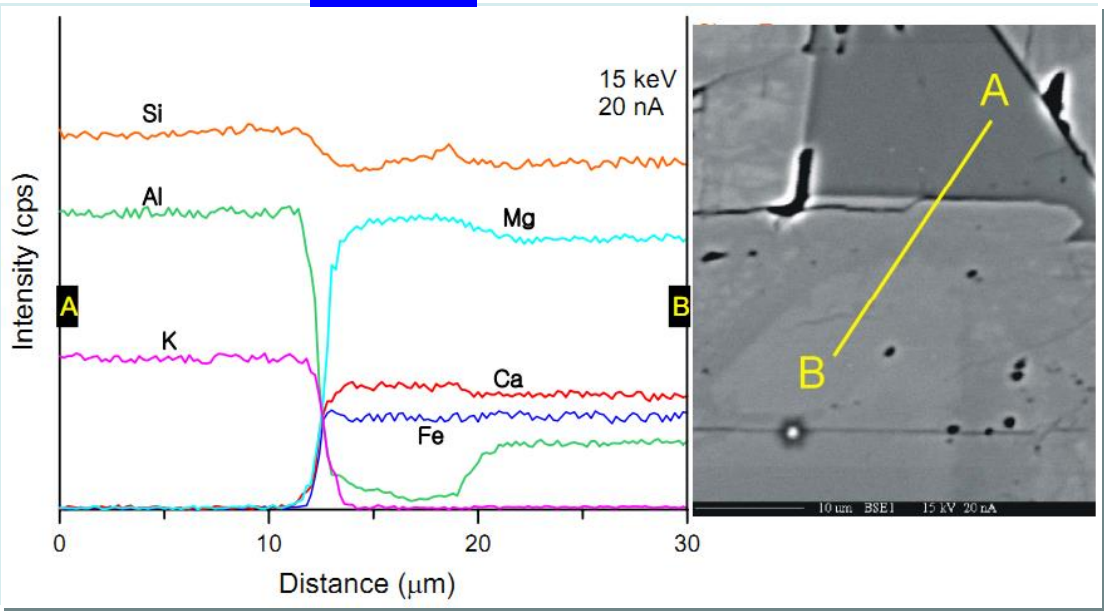




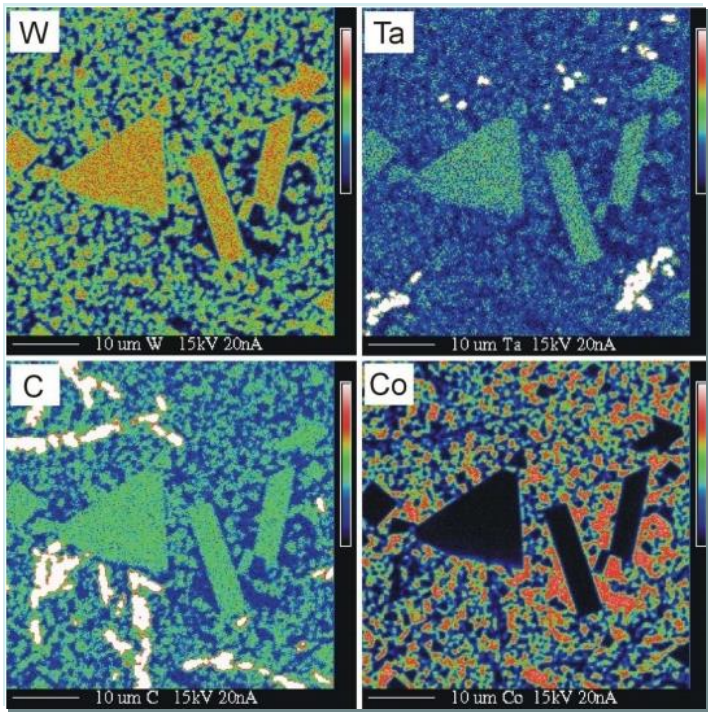


SEM-EDS 성분분석 : 선분석, 면분석

선분석



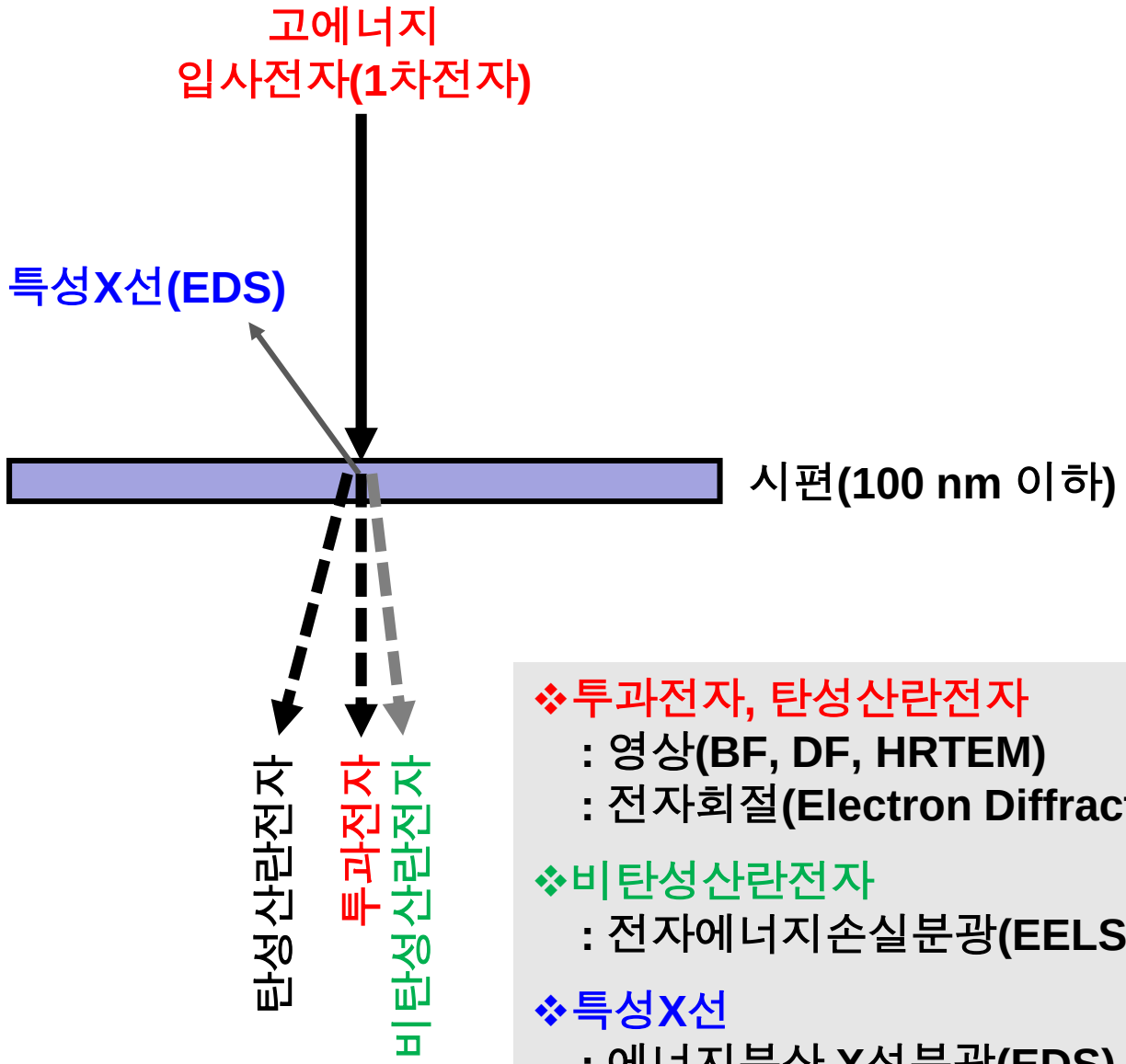
면분석



透過電子顯微鏡 분석

Transmission Electron Microscopy (TEM)

투과전자현미경(TEM) 분석의 개요



❖투과전자, 탄성산란전자
: 영상(BF, DF, HRTEM)
: 전자회절(Electron Diffraction)

❖비탄성산란전자
: 전자에너지손실분광(EELS)

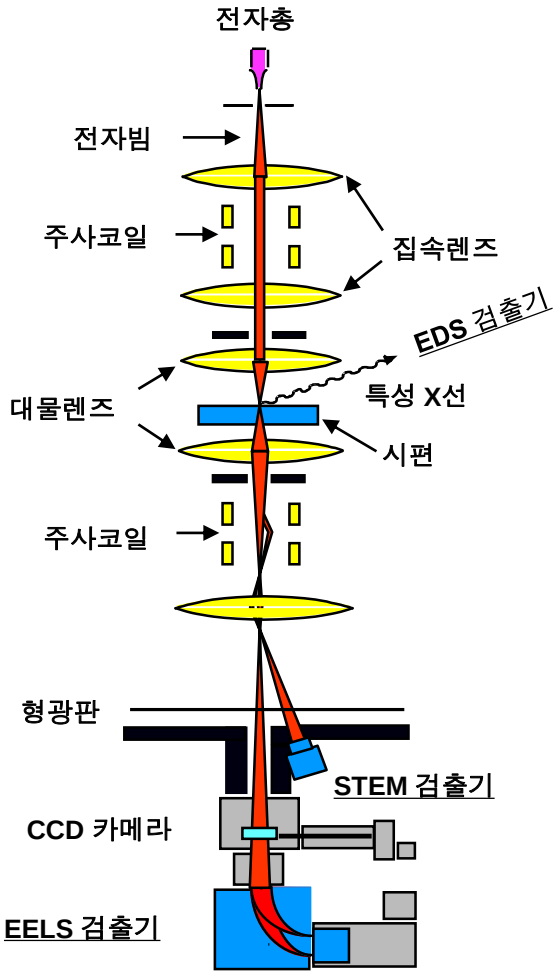
❖특성X선
: 에너지분산 X선분광(EDS)

나노스케일의 미세영역에서 HR-Image, Diffraction Pattern, EDS & EELS Spectrum을 모두 얻을 수 있음

투과전자현미경(TEM) 분석의 개요 : 장치

- **전자빔이 얇은 시편을 투과하며 발생하는 투과전자와 탄성산란 전자를 이용하여 재료의 미소영역을 확대·관찰하는 장치**
  : 물질의 미세구조, 격자결함, 결정구조, 원자배열 상태 등 조사
- **시료와 전자빔과의 상호작용으로 발생하는 특성X선을 EDS로 검출하거나,**
  시료를 투과한 비탄성 산란전자의 에너지를 EELS로 측정하면 미소영역의 조성을 분석할 수 있음





TEM 분석의 개요

영상분석

● 미세구조 분석
- 명시야상(BF)
- 암시야상(DF)
- HRTEM
- STEM(BF, DF, HR)

전자회절

● 결정구조 분석
- 제한시야 전자회절(SAED)
- KiKuchi 회절
- 나노빔&수렴성 빔 전자회절

성분분석

● 화학성분 분석
- 에너지분산 분광법(EDS)
- 전자에너지손실 분광법(EELS)

1. 원자 수준 분해능에서 물질의 구조분석
2. 전자회절을 이용한 물질의 결정구조 및 박막의 배향성 분석
3. EDS 원소분석(Point, Line, Mapping)
4. EELS 원소분석(Point, Line, Mapping)

- TEM의 점분해능 : ~0.2 nm
- EDS 분석의 공간분해능 : 나노미터 수준
- EELS 분석의 공간분해능 : 나노미터 수준

Å level의 분해능에서 재료 및 나노소자의 형상 관찰 및 박막의 두께 측정

Defect (lattice defect, dislocation 등) 관찰

결정립의 크기, 형태 등의 관찰

전자회절을 이용한 특정 영역의 미세 구조 및 방위 분석

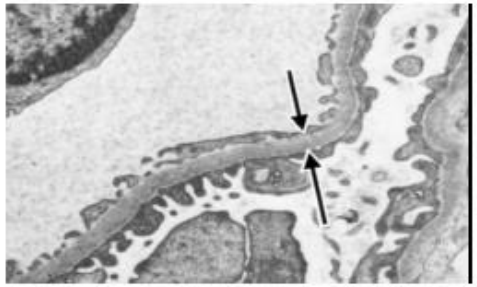
결정 및 계면의 원자 배열 상태 관찰 (HRTEM)

박막 및 nm scale 미소 영역의 조성 분석 (EDS, EELS)

TEM 분석의 개요

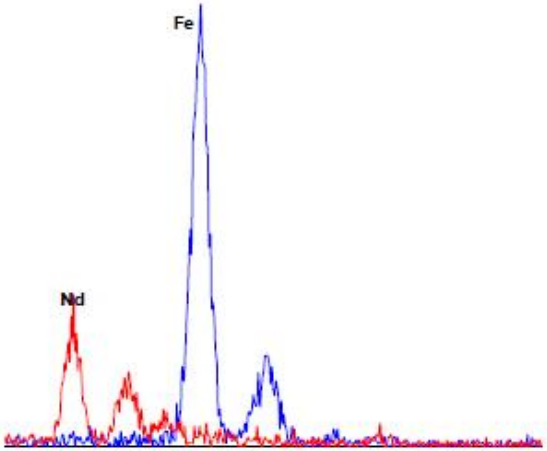
❖ Types of Information

➢ Image



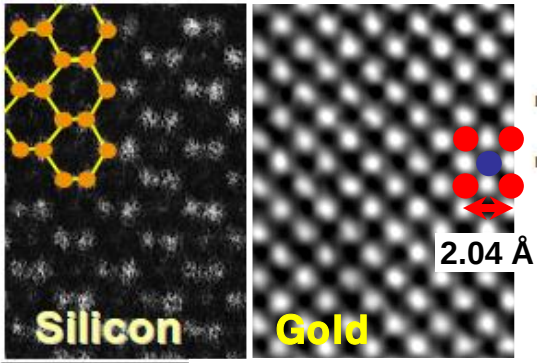
- TEM BF/DF Image
- STEM BF/DF Image

➢ Chemistry

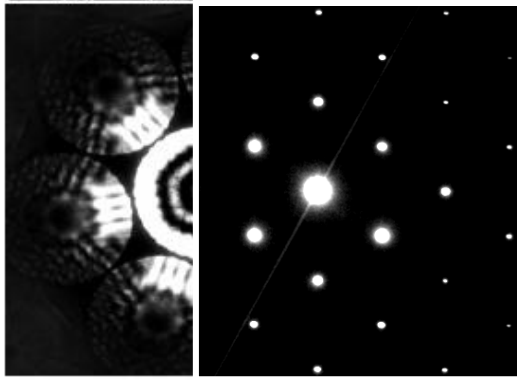


- EDX
- EELS
- GIF Element
- Mapping

➢ Structure



- HRTEM Image
- HAADF Image



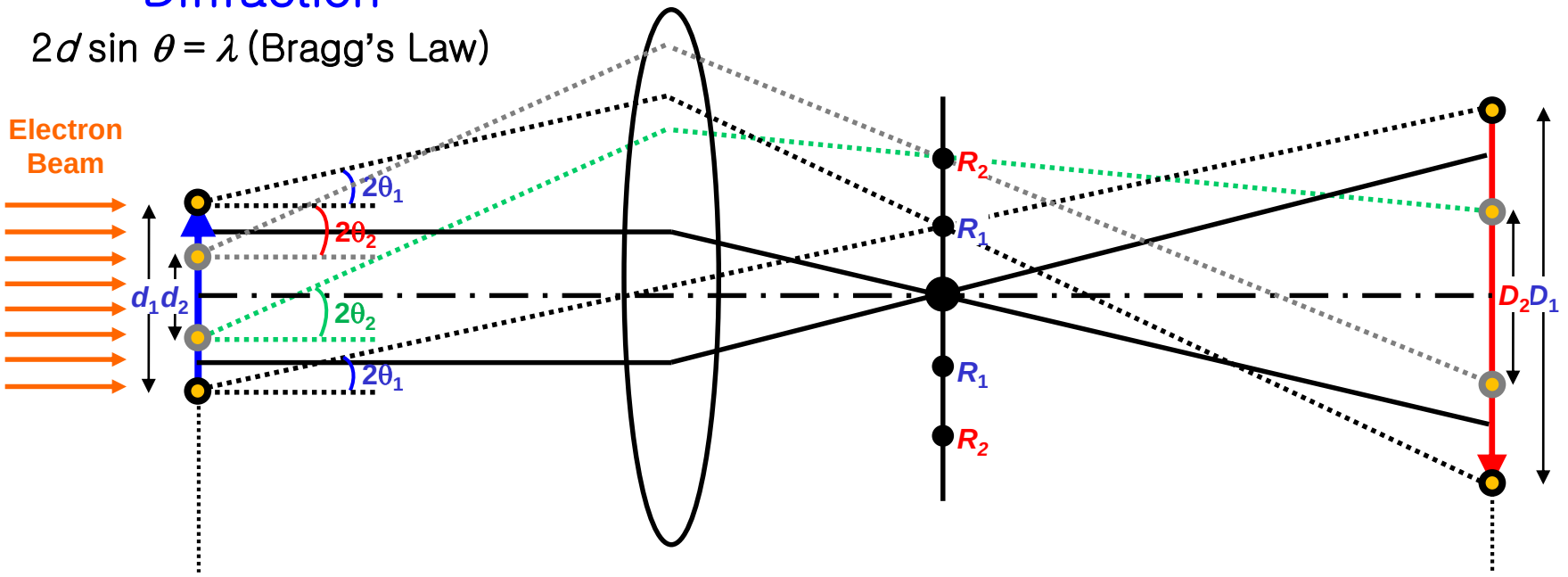
- Diffraction Pattern
- CBED
- Kikuchi Line

www.raonoptics.co.kr/chtml/board.php?

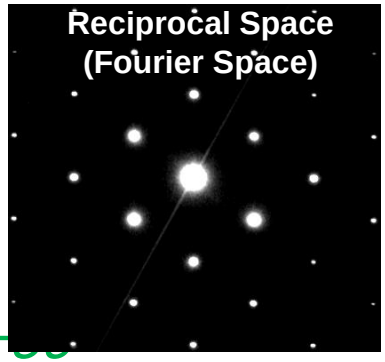
TEM 영상 및 전자회절의 원리

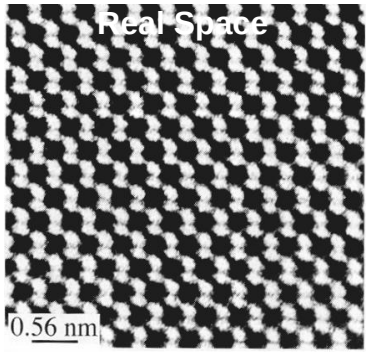
Diffraction

$2d \sin\theta = \lambda$ (Bragg's Law)

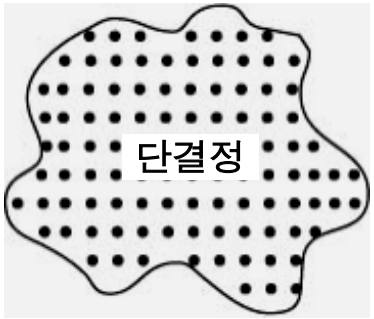


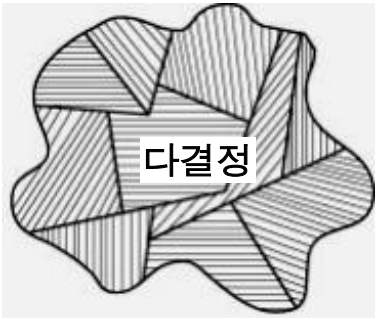


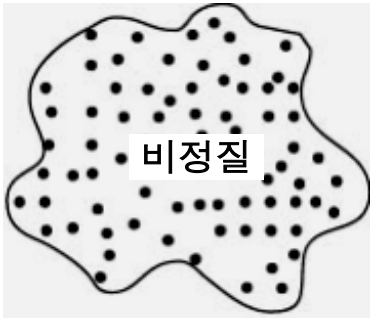


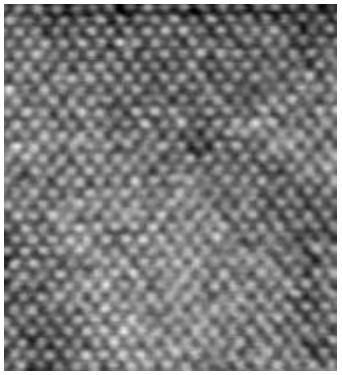


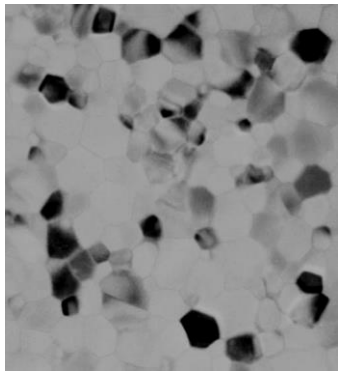
결정성에 따른 회절도형의 변화

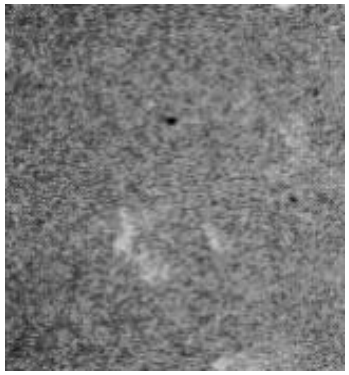


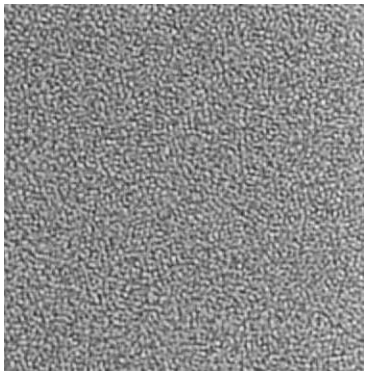


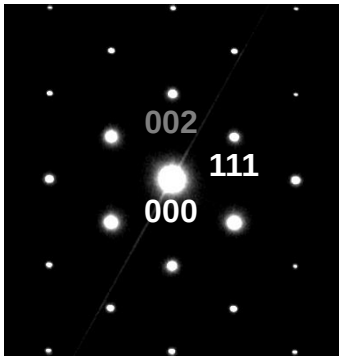




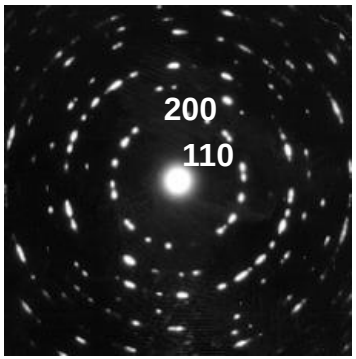




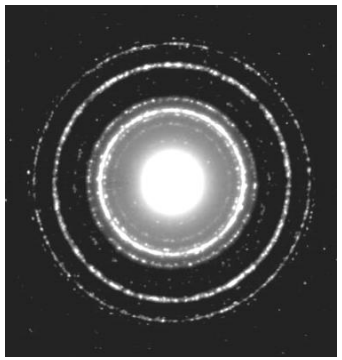




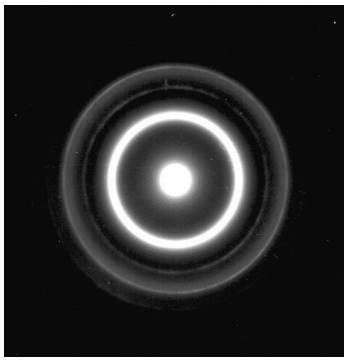
Si [110] 입사,
단결정



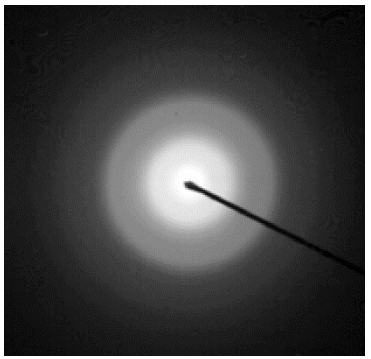
W, 다결정,
결정입도 大



ZrO_2, 다결정,
결정입도 小



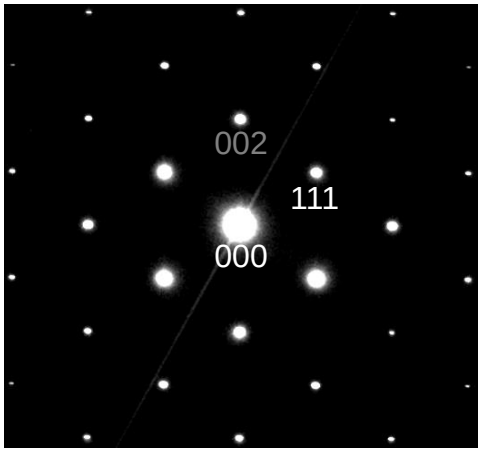
결정립도
매우 작음



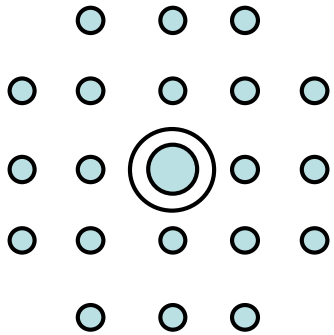
비정질

● TEM의 영상관찰 기법

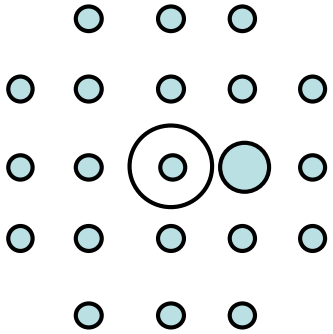
- **명시야상 : 흡수, 회절 콘트라스트(진폭 콘트라스트)**
  - 투과빔을 대물조리개로 선택함에 의해 결상
- **암시야상 : 흡수, 회절 콘트라스트(진폭 콘트라스트)**
  - 특정의 회절빔을 대물조리개로 선택함에 의해 결상
- **HRTEM 영상 : 위상 콘트라스트**
  - 투과빔과 회절빔의 합성(간섭)에 의해 결상
- **회절도형 : 역격자 공간에서 결정의 대칭성 및 결정면의 방향 및 거리에 대한 정보**



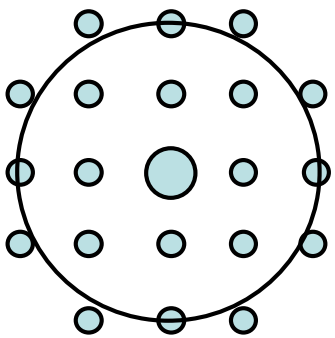
Si [110] 입사의 회절도형



명시야상

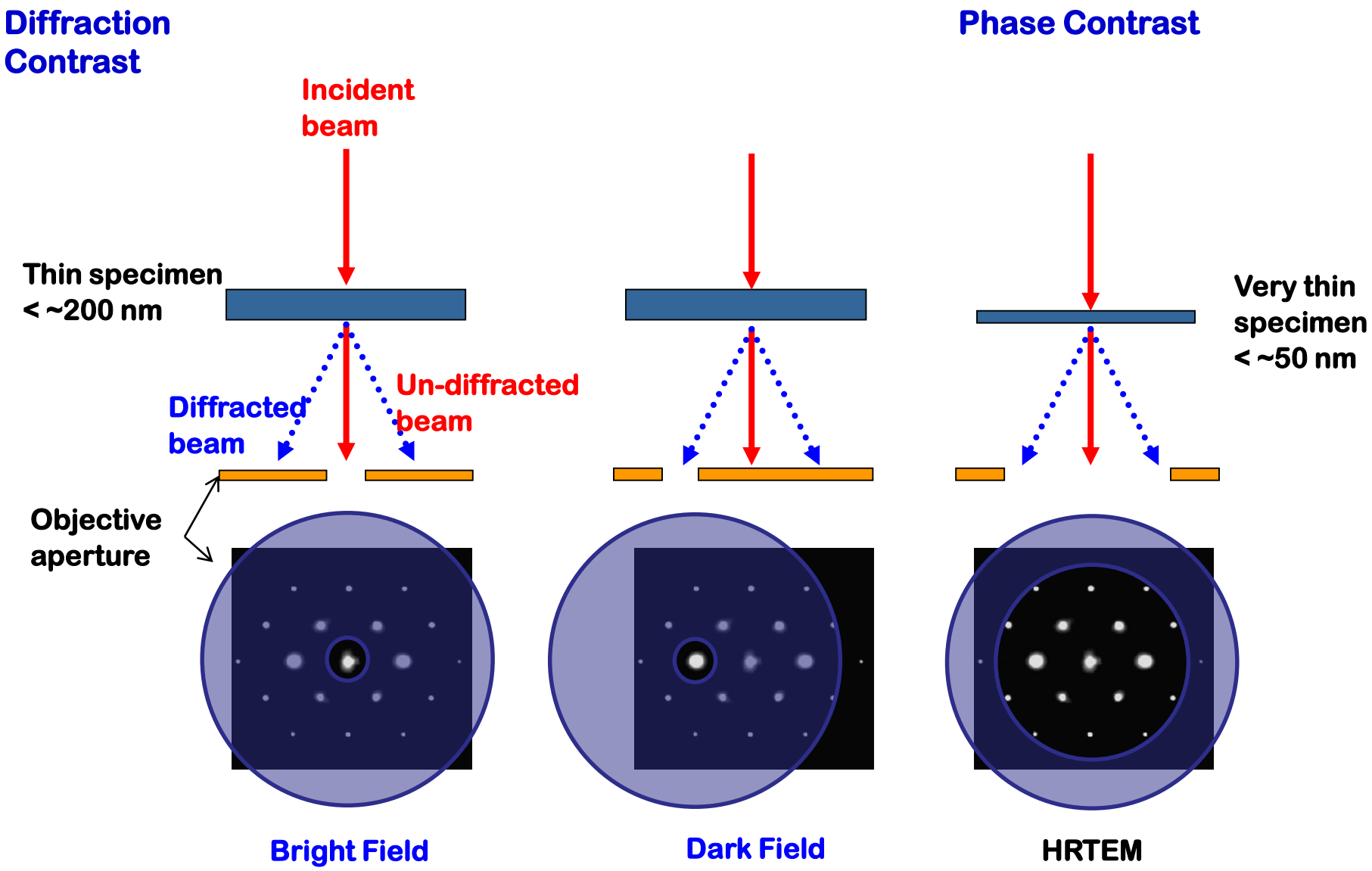


암시야상



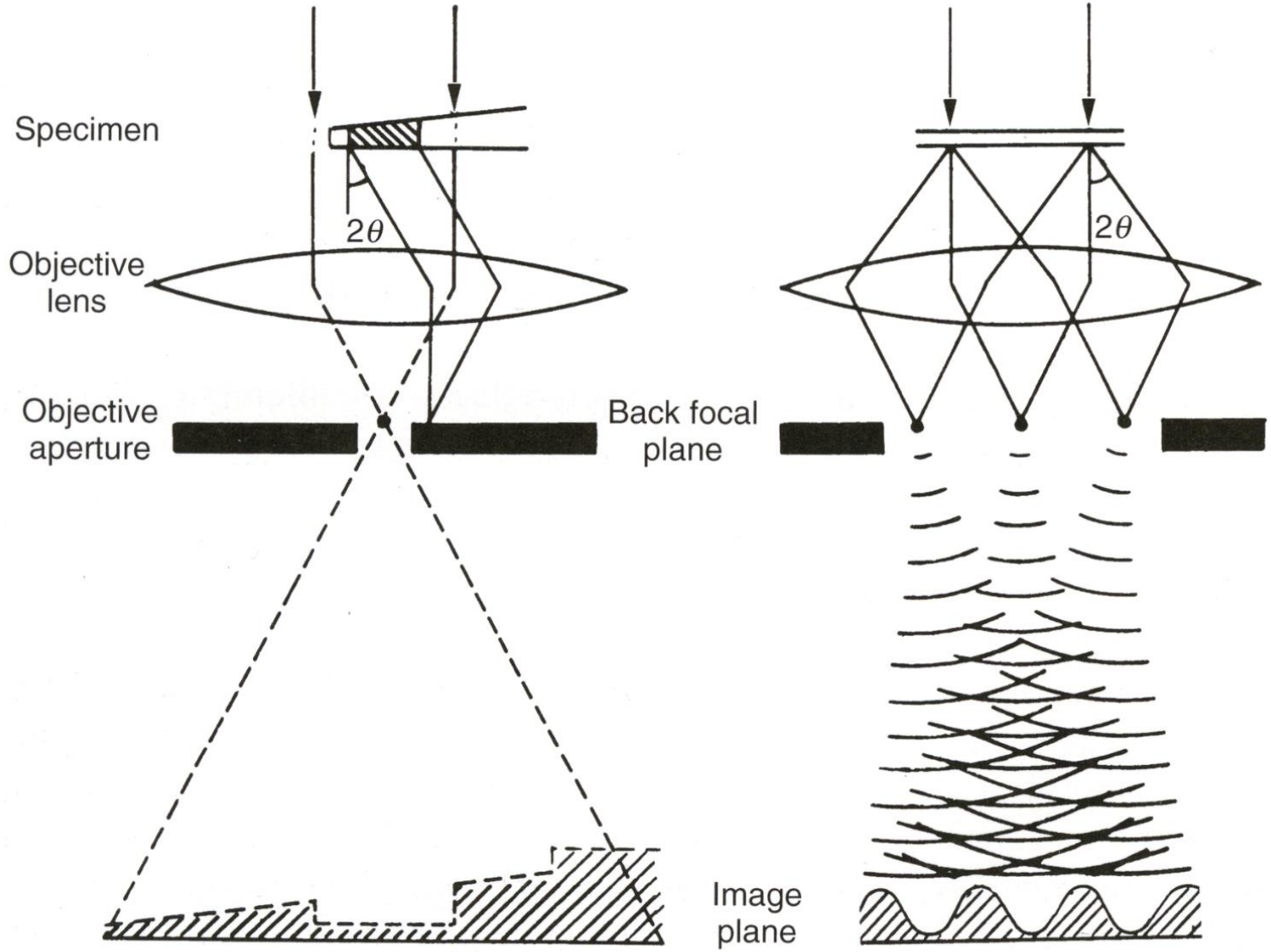
HRTEM 영상

TEM Imaging Mode



D. B. Williams et al., Transmission Electron Microscopy: A Textbook for Materials Science

TEM Imaging Mode



진폭 콘트라스트

Bright Field & Dark Field Images

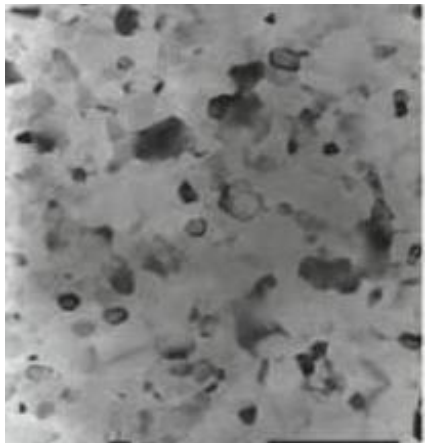
위상 콘트라스트

HRTEM Images

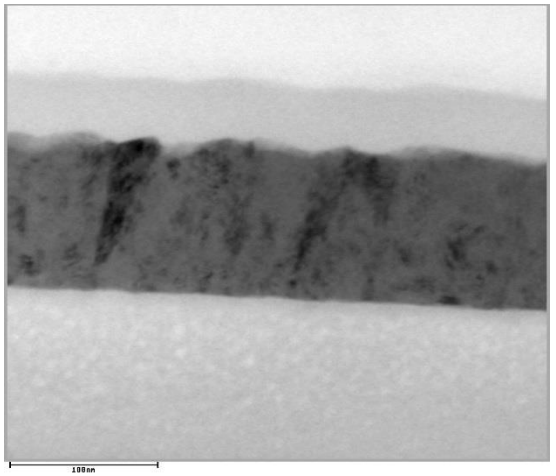
BF Image *vs* DF Image

- Bright field mode: 투과파를 objective aperture로 선택함에 의해 결상
- Dark field mode: 특정의 회절파를 objective aperture로 선택함에 의해 결상

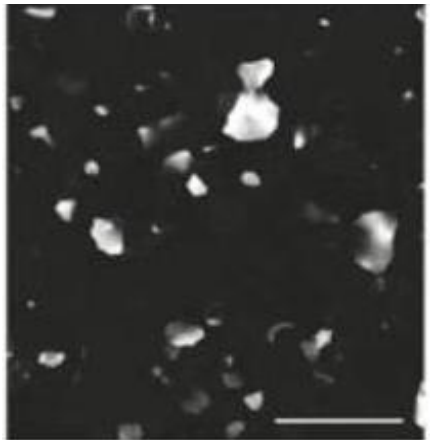
BF Image

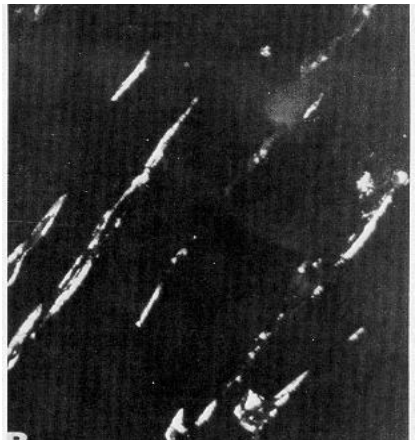






DF Image

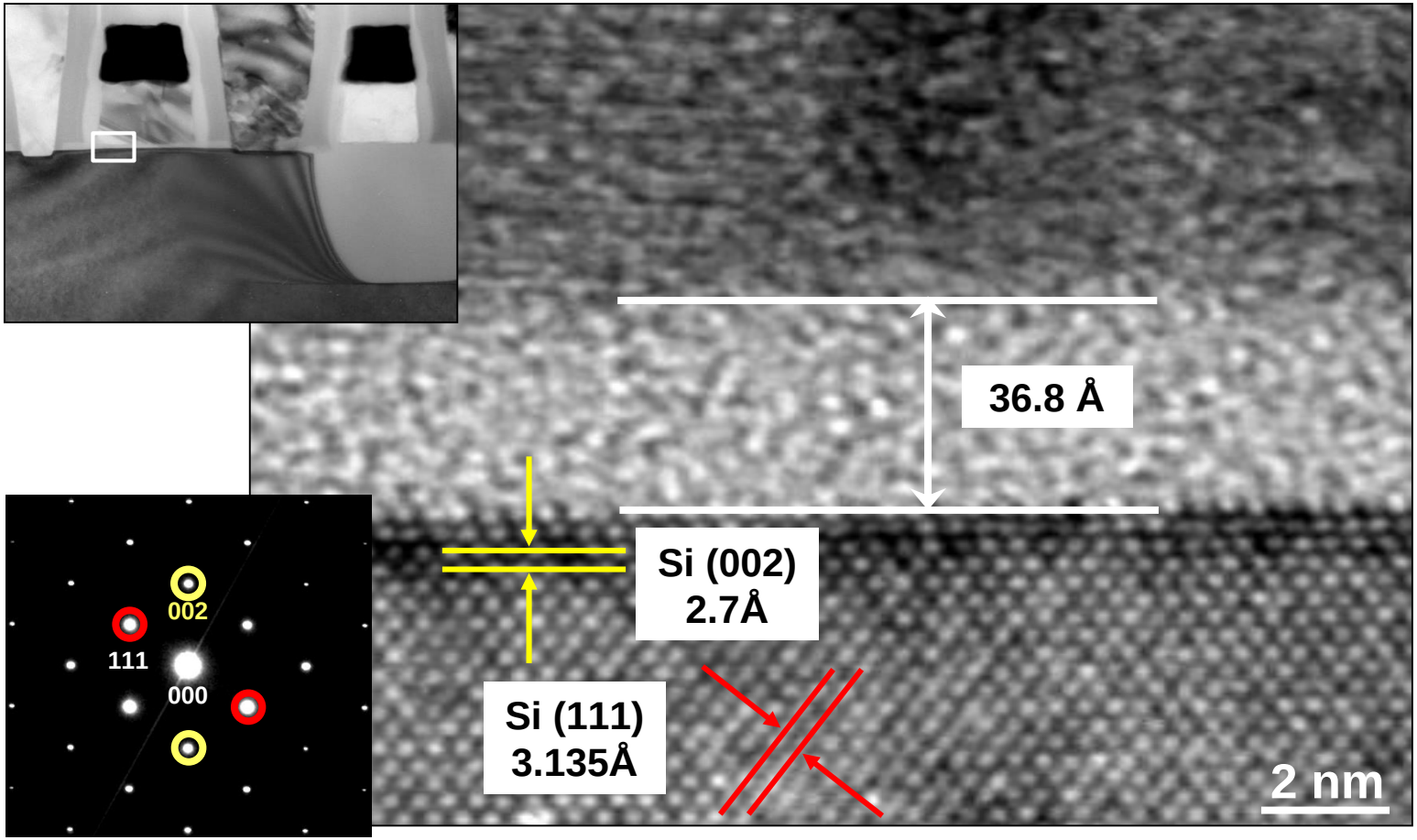






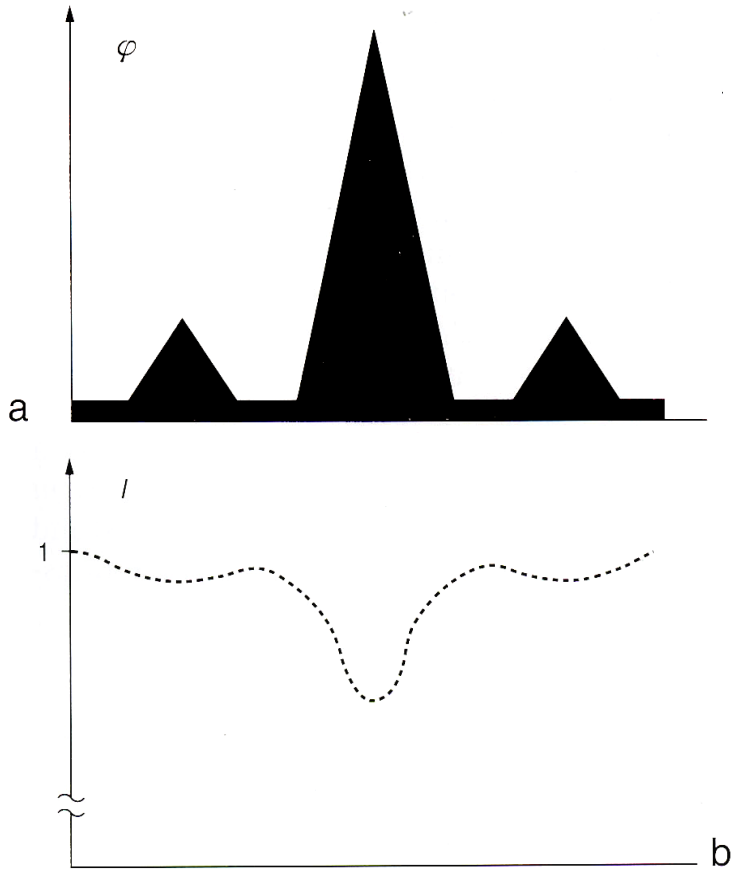
HRTEM Image : Lattice Imaging

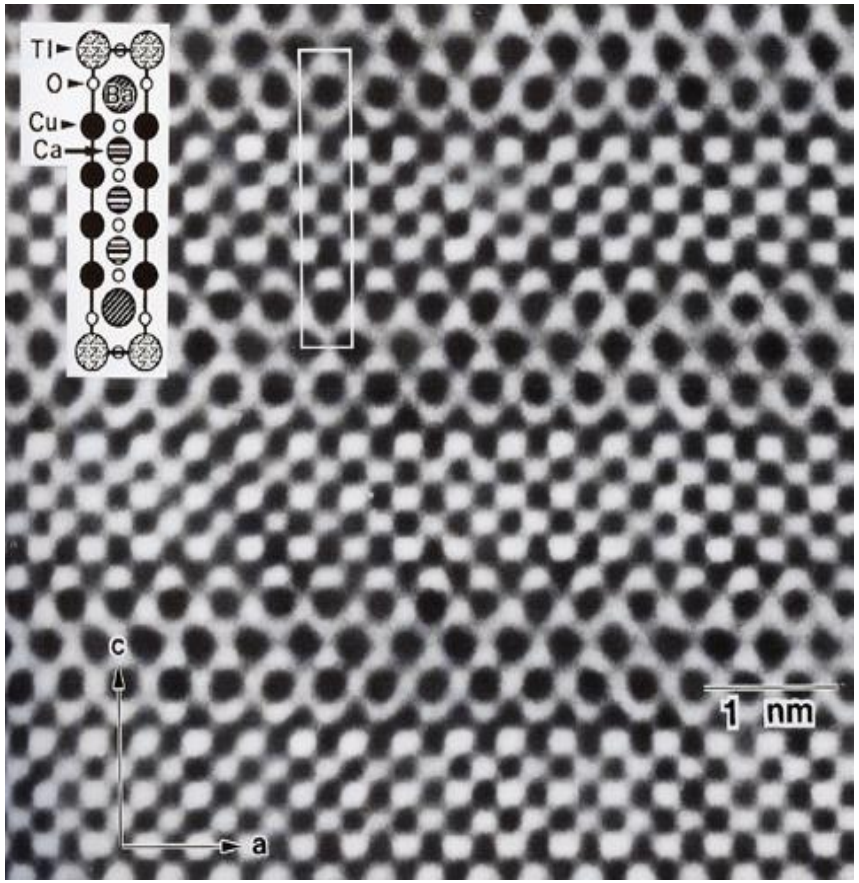
- HRTEM mode: 투과빔과 회절빔의 합성(간섭)에 의해 결상
- Phase contrast by the interference among beams



Si [110] zone

HRTEM Image : Structure Imaging





(a) 결정의 내부 **potential**

(b) **HRTEM Image**의 **Intensity**

$$I(x,y) \approx 1 - 2\sigma\varphi(-x,-y)\Delta z$$

Tl계 초전도산화물의 **HRTEM Image**

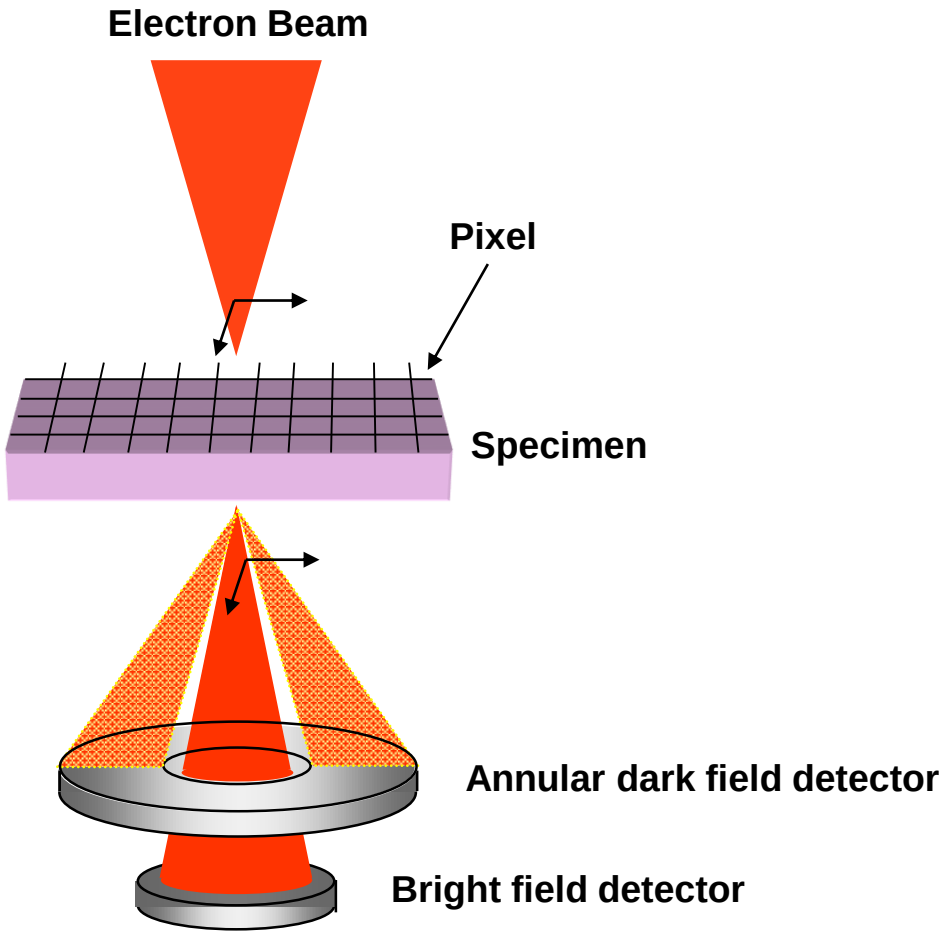
D. Shindo et al., High-Resolution Electron Microscopy for Materials Science

TEM 기법을 이용한 나노분석

Nanoanalysis by TEM Techniques

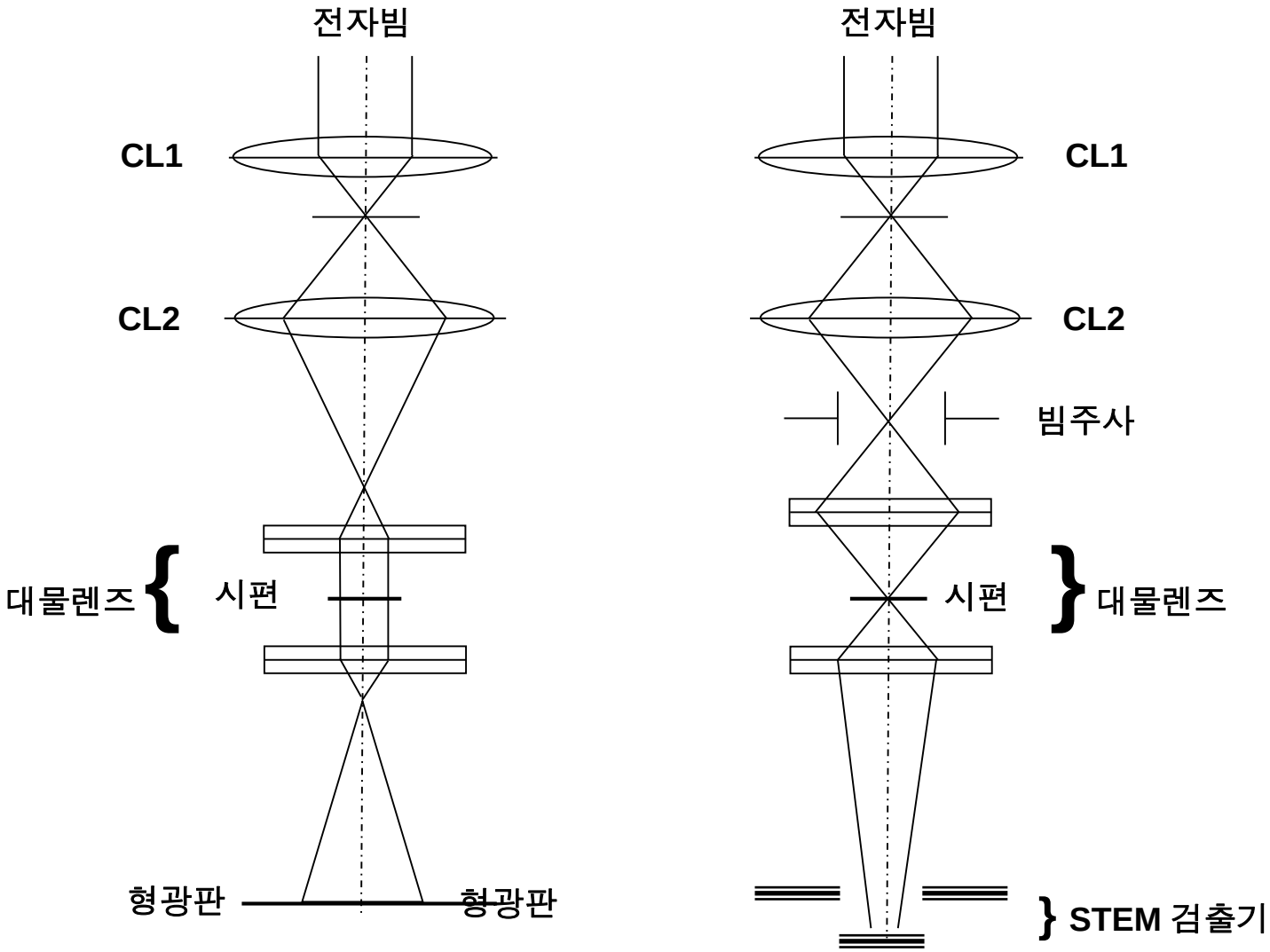
STEM의 원리 및 응용

Scanning Transmission Electron Microscopy (STEM)의 원리

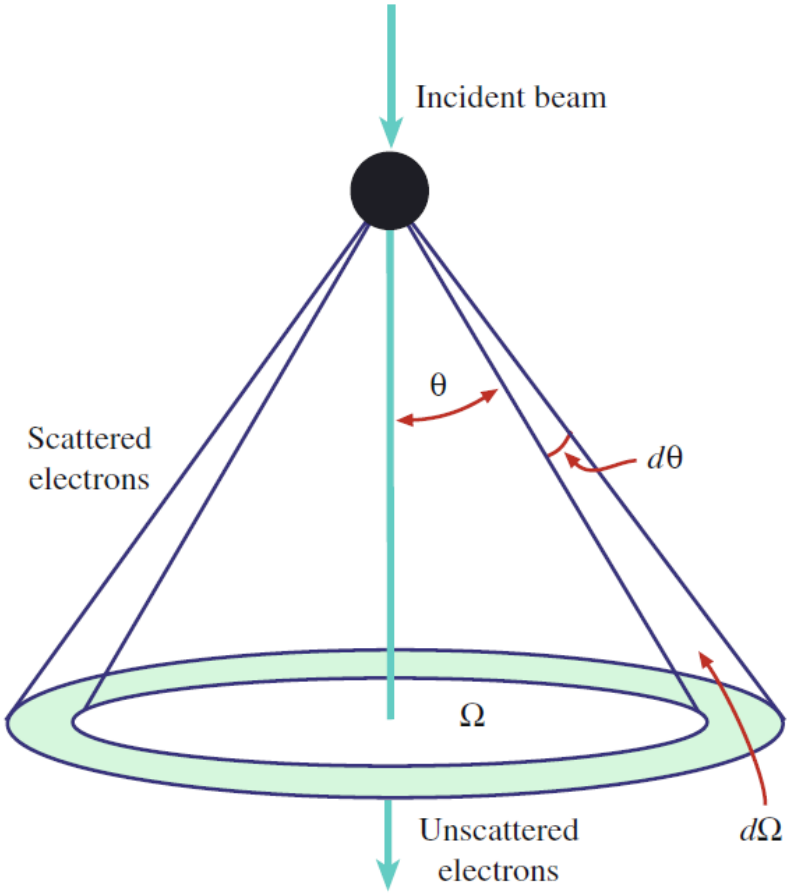


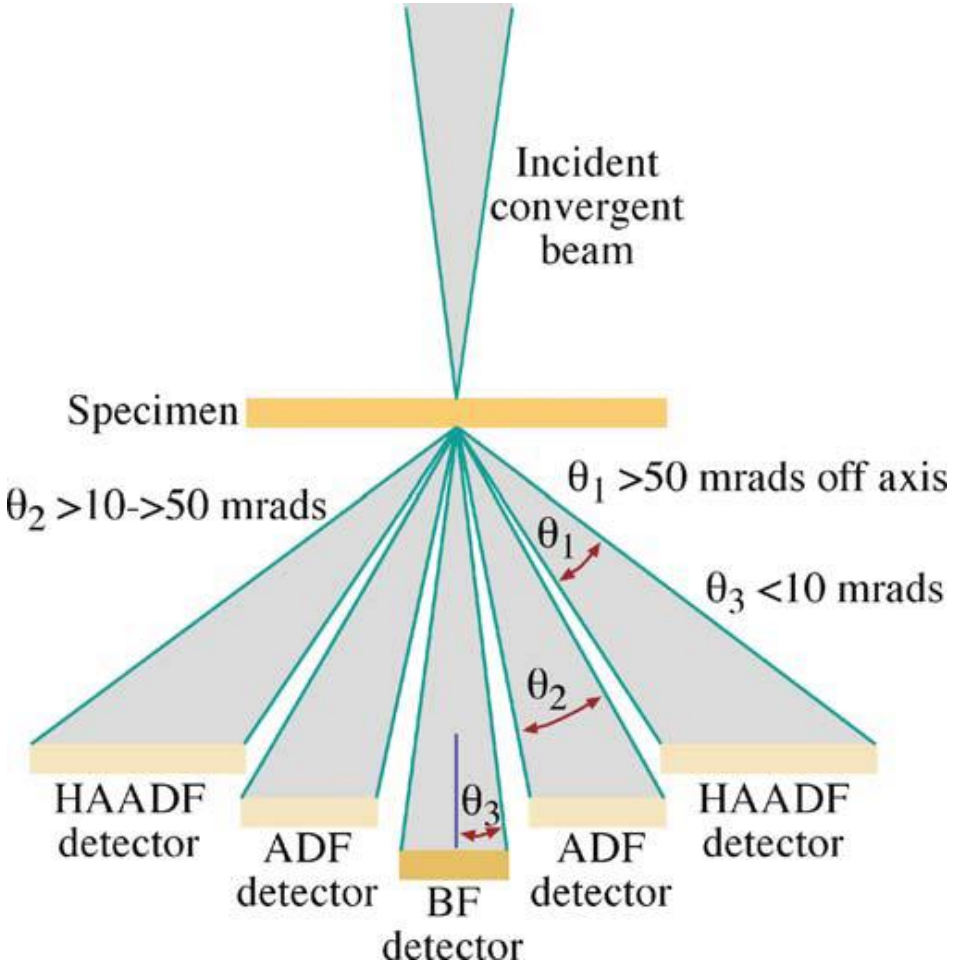
- 대물렌즈로 시편에 쪼여지는 전자빔을 더욱 집광시키고 좁은 영역으로 퍼지는 투과빔을 검출하여 영상화시킴
- 2차원적인 영상을 만들기 위해서 시편에 형성되는 작은 탐침을 2차원적으로 주사시키면서 검출기에도 동일한 모드의 주사전류를 가하여 투과빔의 세기를 영상화시킴
- EDS 및 EELS와 결합하여 미소영역의 분석에 유용하게 이용됨

CTEM *vs* STEM


전자빔
CL1
CL2
대물렌즈
시편
형광판
형광판
CTEM
전자빔
CL1
CL2
빔주사
시편
대물렌즈
STEM 검출기
STEM

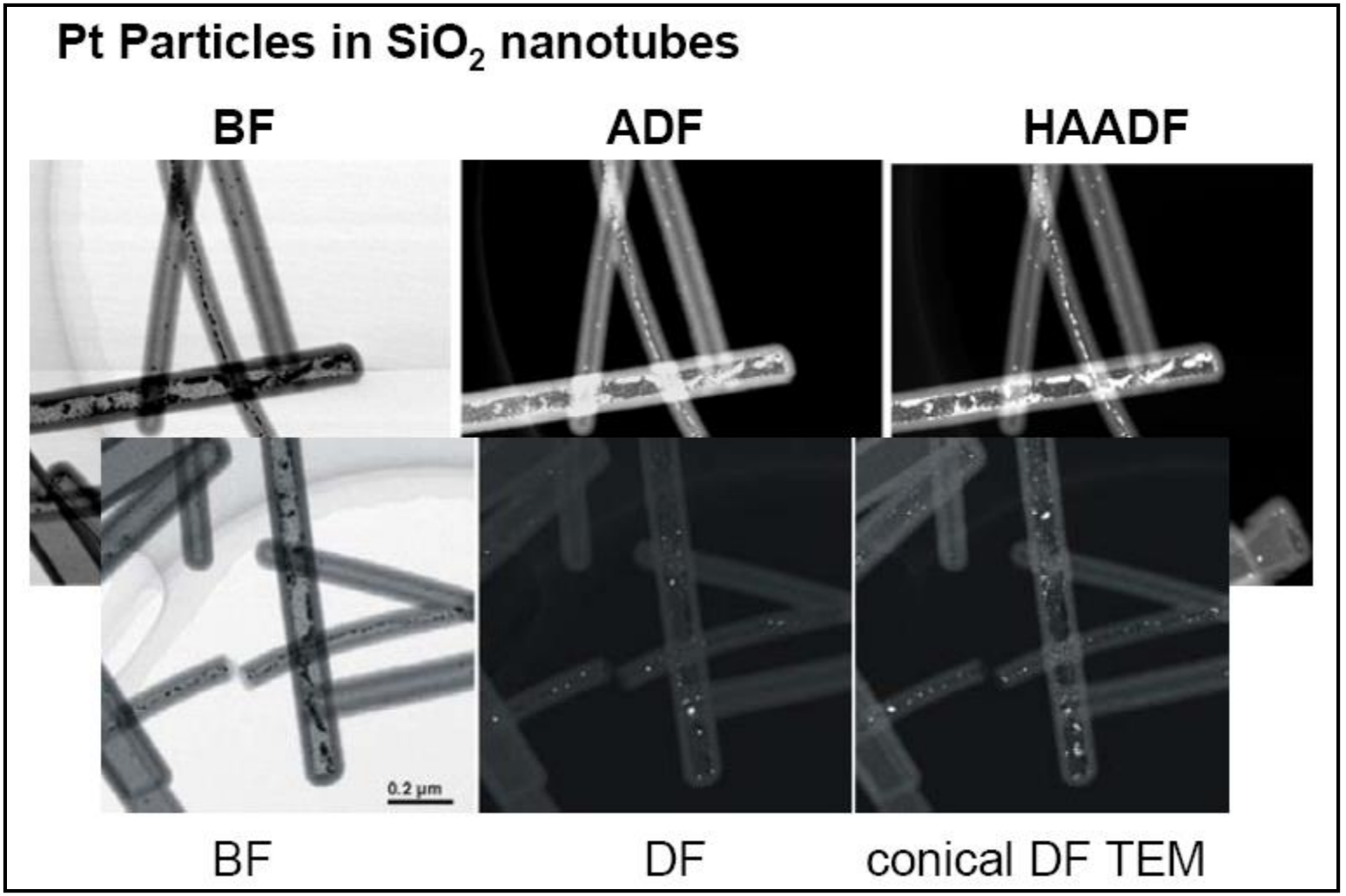
STEM Detectors



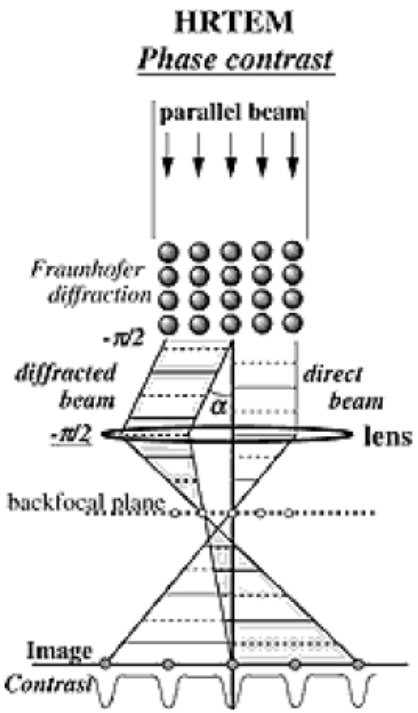


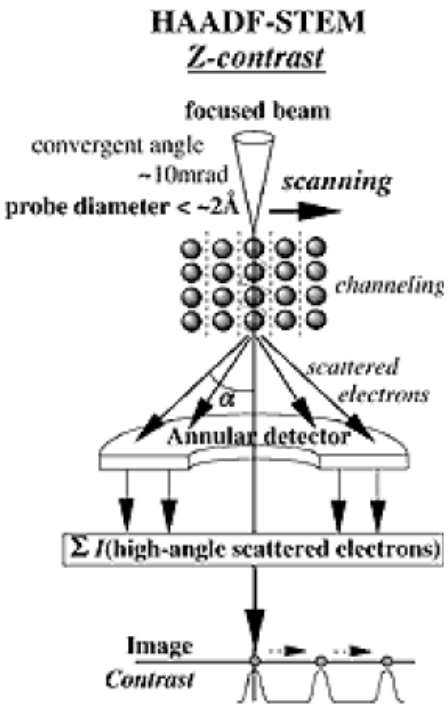
D. B. Williams et al., Transmission Electron Microscopy: A Textbook for Materials Science

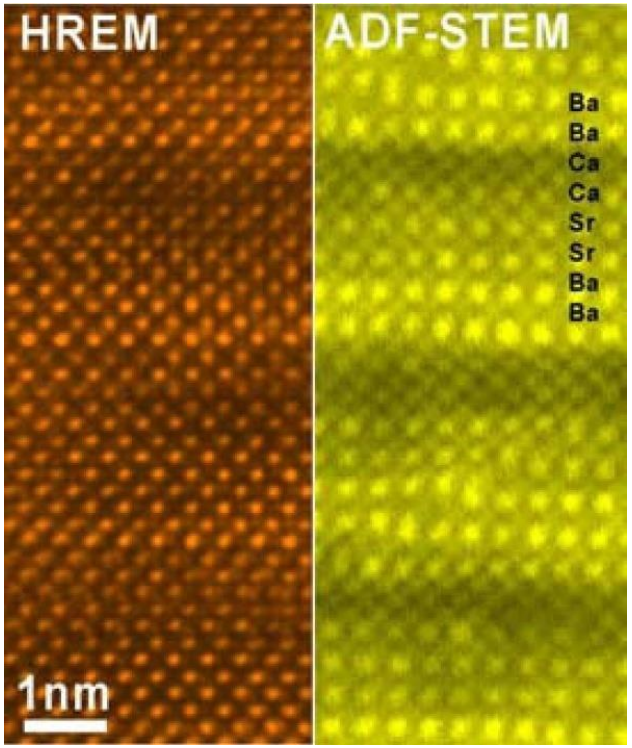
Z-Contrast Imaging with HAADF Detector



HRTEM vs HRSTEM







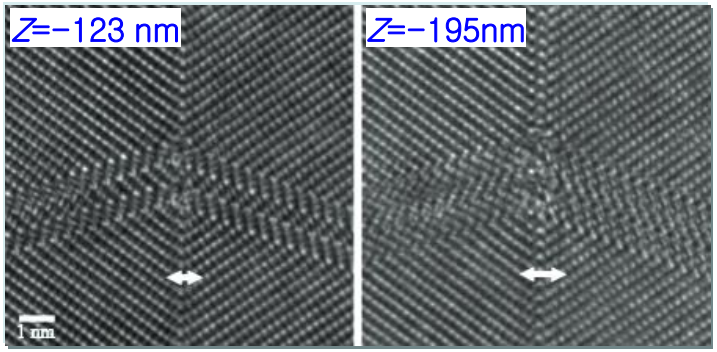
$BaTiO_3/SrTiO_3/CaTiO_3$ superlattice

1. Contrast

- High-resolution TEM (HRTEM) image is a phase contrast image (indirect image). The contrast depends on defocus.
- STEM image is a direct atomic column image (average Z-contrast in the column).

2. Delocalization Effect

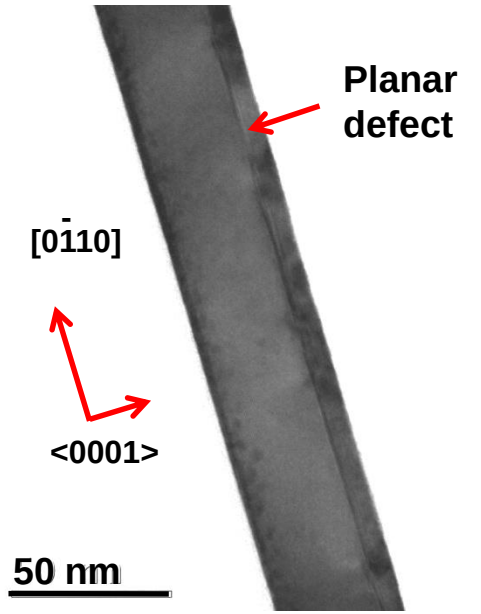
- High-resolution TEM image from FEG has delocalization effect.
- STEM image has no such an effect.



Twin boundaries in $BaTiO_3$, Jia et al. 1999

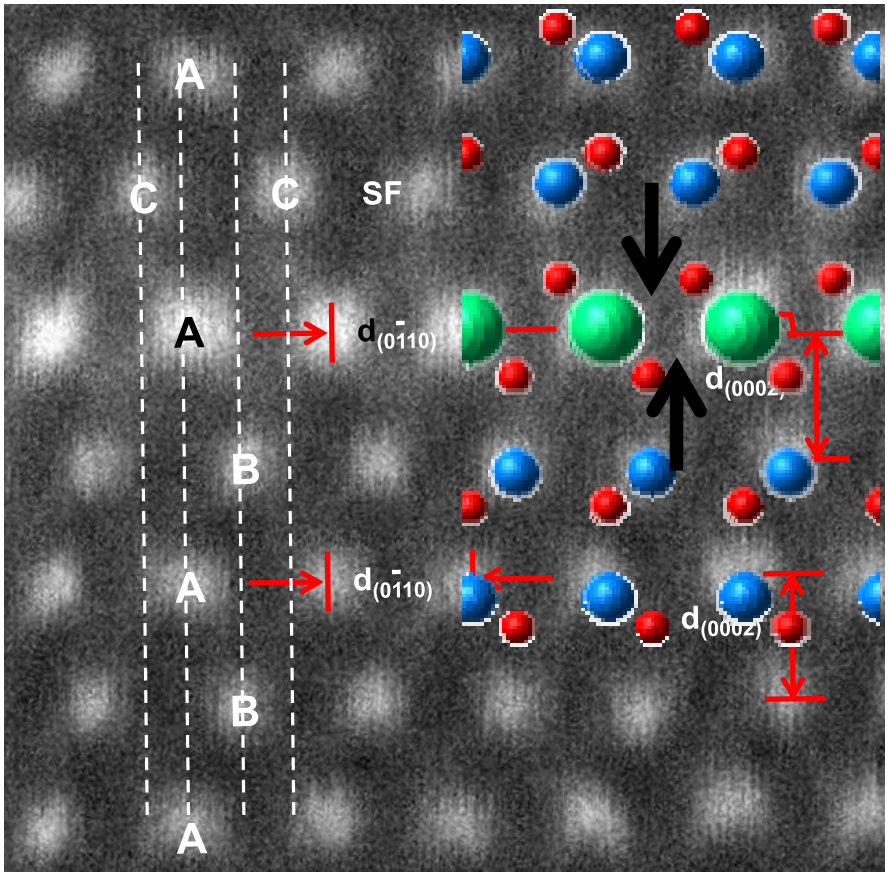
Cs-Corrected STEM Analysis

Synthesis : Sn-doped ZnO



Stacking Fault

Type 1(b) : ABAB|ACAC



HAADF Angle: 90 ~ 170 mrad

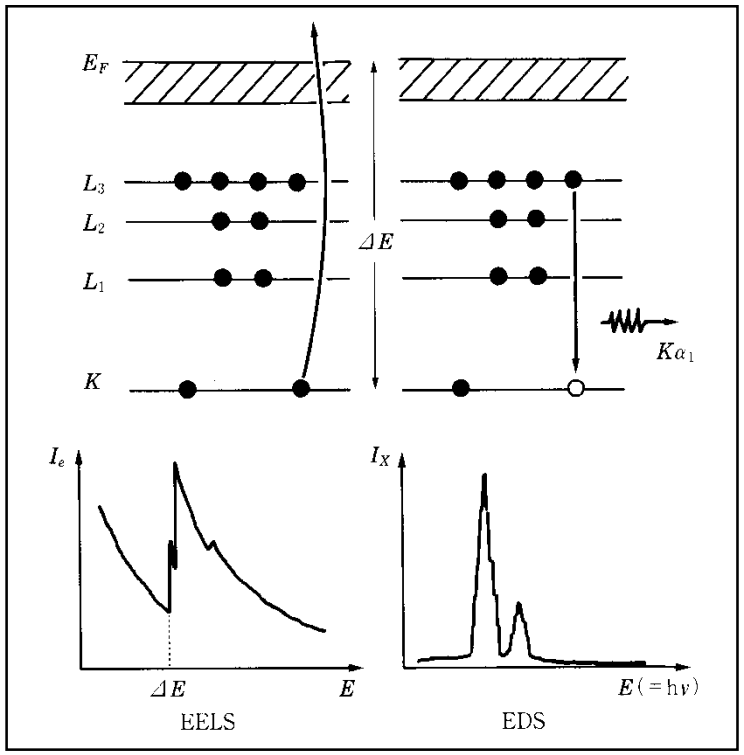
EDS & EELS의 원리 및 응용

TEM을 이용한 성분분석

● TEM을 이용한 성분분석 원리

- **EDS (Energy-dispersive X-ray Spectroscopy)**
  - 점분석(point analysis), 라인프로파일(line profile), 원소매핑(elemental mapping)

- **EELS (Electron Energy-Loss Spectroscopy)**
  - 점분석(point analysis), 라인프로파일(line profile), 원소매핑(elemental mapping)



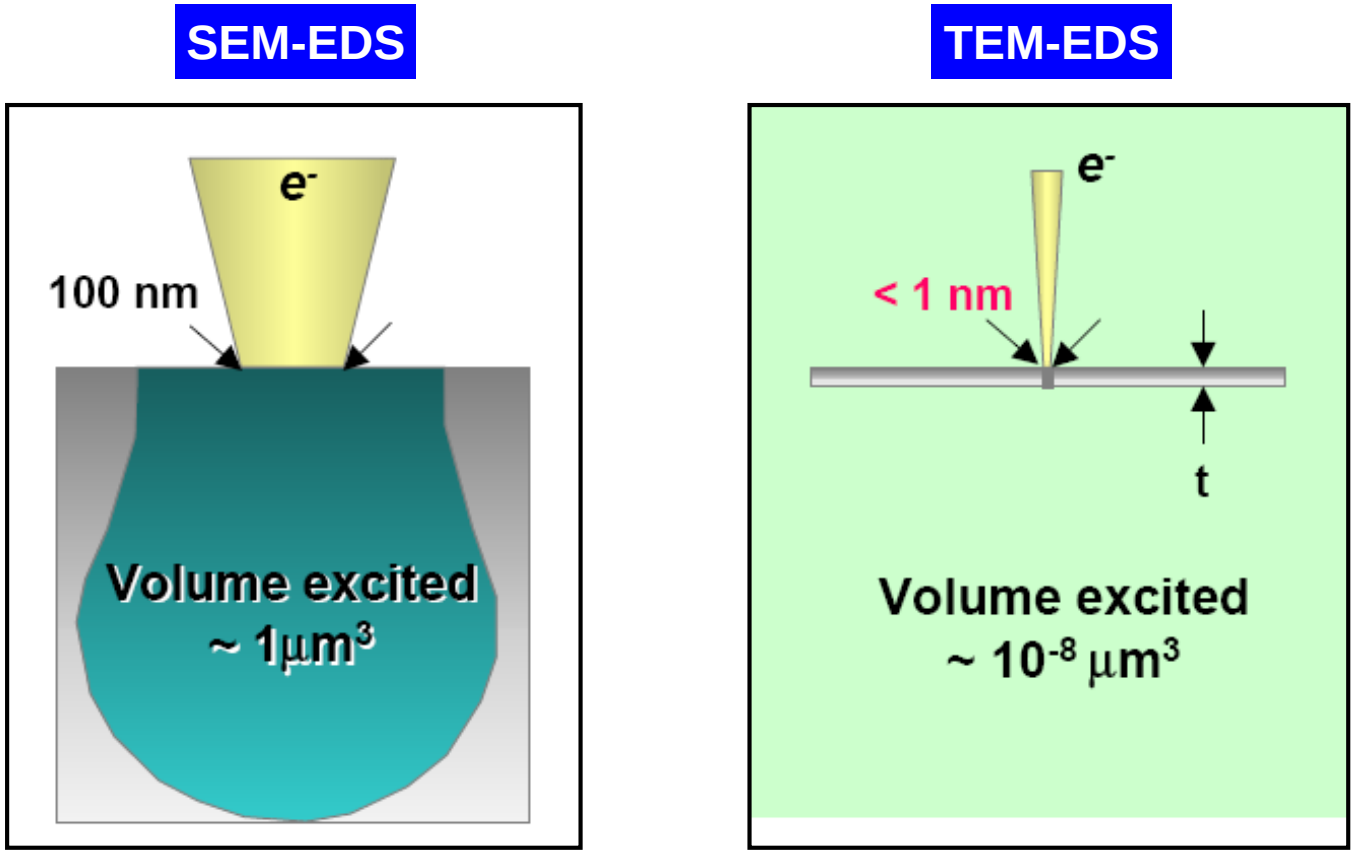
- EDS: $E = 0.013\ (Z\text{-}1)^2$ (keV)
  1. 내각전자가 여기된 후 원래의 위치로 천이되는 과정에서 특성 X선 발생
  2. 원소 구별의 척도

- EELS
  1. 입사전자가 내각 전자와 충돌하여 임계 이상의 에너지를 손실하였을 때 peak 형성 (입사전자의 비탄성 산란 이용)
  2. 원소 구별의 척도가 되며, peak의 chemical shift 또는 형태변화로 화학결합 상태 조사

EDS와 EELS 분석의 비교

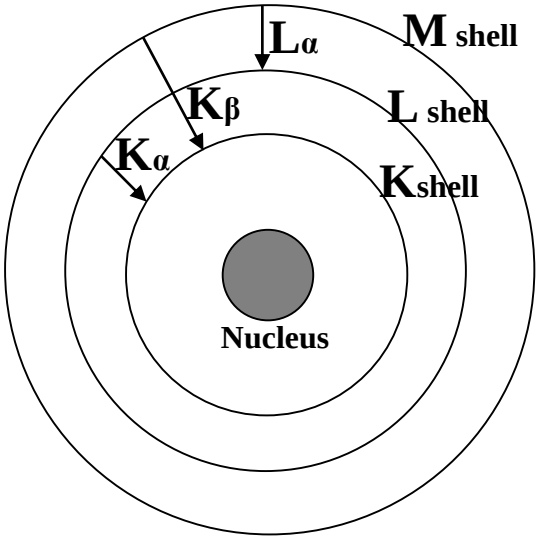
	EDS	EELS	비고
검출 신호	특성X선	에너지손실 전자	EDS : 2차여기 이용 EELS : 입사전자 이용
측정 효율	낮음	높음	EELS의 경우 측정시간이 짧음
분석원소	B - U	H - U	EDS : 중원소에 유리 EELS : 경원소에 유리
검출한계	수 wt%	수 at%	
Energy 분해능	약 120 eV	0.7 eV@T-FEG 0.3 eV@C-FEG meV 수준@Monochromator (chemical shift 측정)	EDS는 background의 강도가 낮아 정량분석에 유리
원소 **map**	STEM (다원소 동시 분석)	Energy filtering (Background 제거)	EDS : 다원소 map EELS : energy filtering

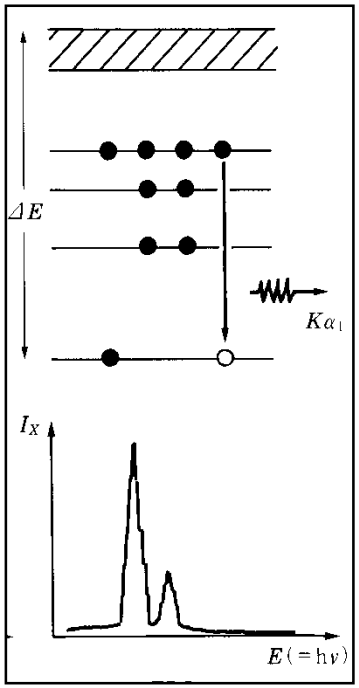
SEM-EDS와 TEM-EDS의 비교 : Beam Broadening

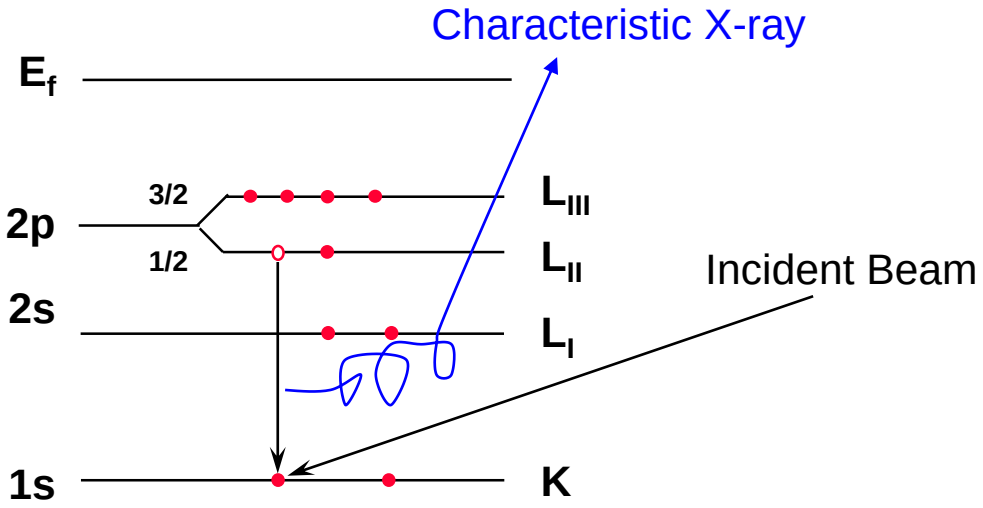


EDS 성분분석

특성X선 (Characteristic X-Ray)



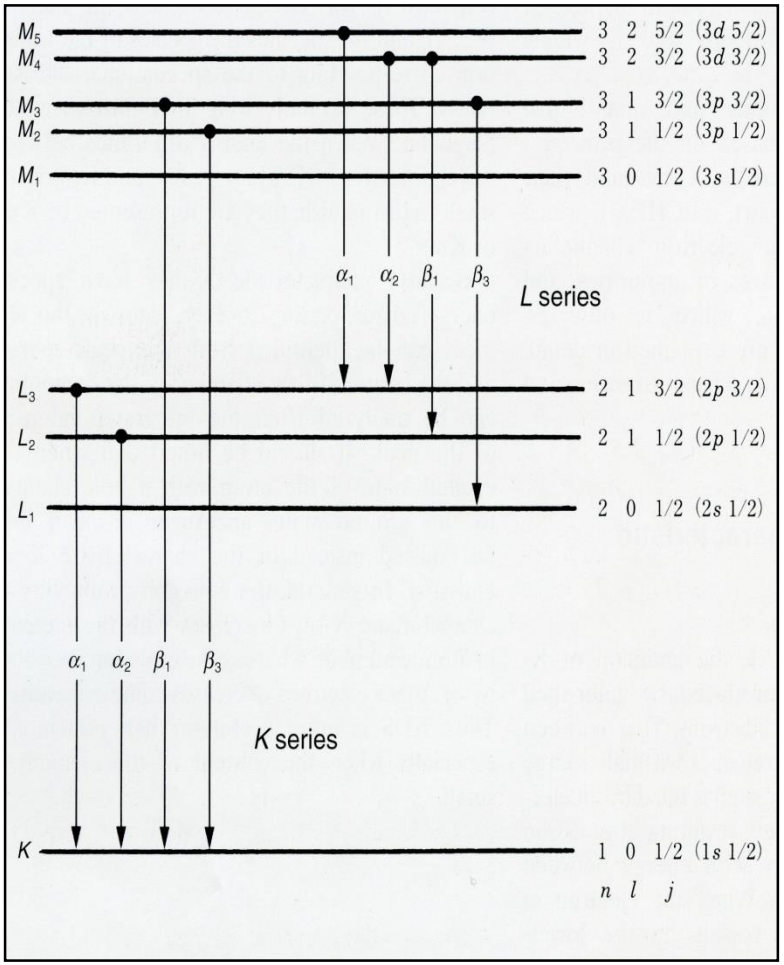


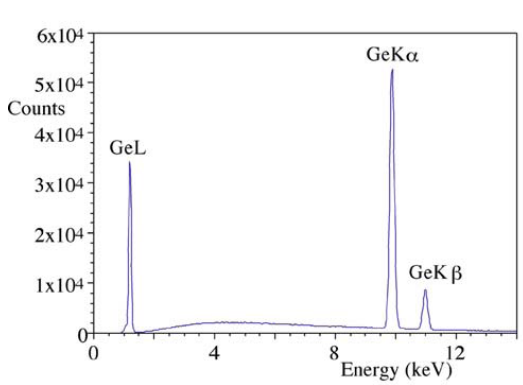


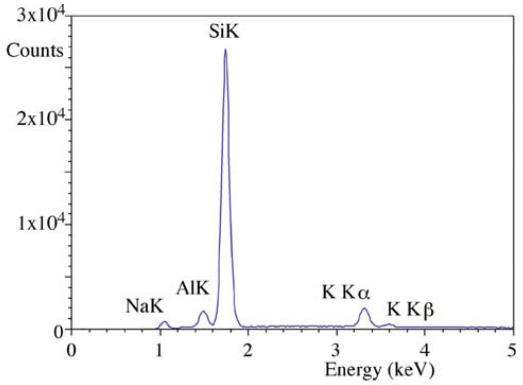
- $E = 0.013\,(Z-1)^2$ (keV)

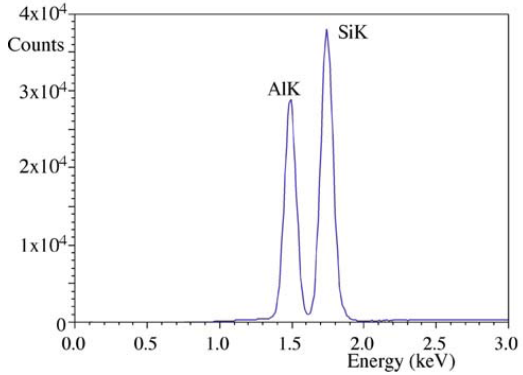
1. 내각전자가 여기된 후 원래의 위치로 천이되는 과정에서 특성 X선 발생
2. 원소 구별의 척도

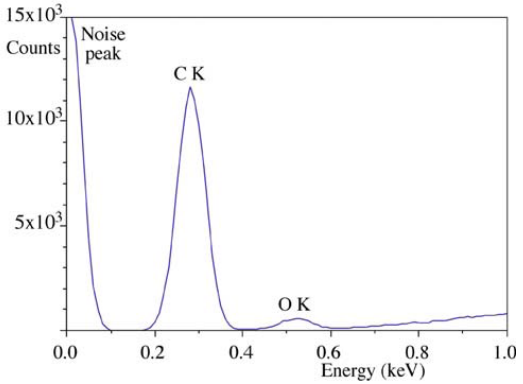
Energy Dispersive X-Ray Spectroscopy (EDS)의 개요

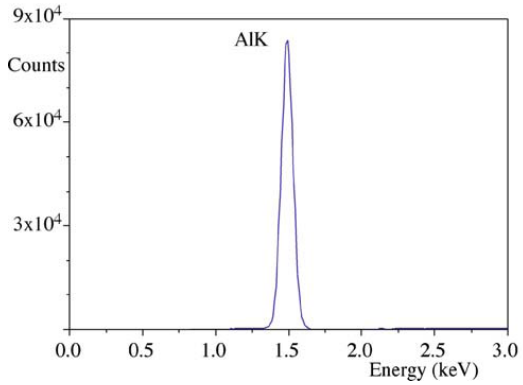


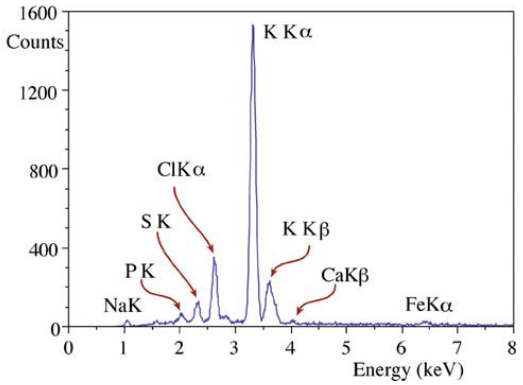












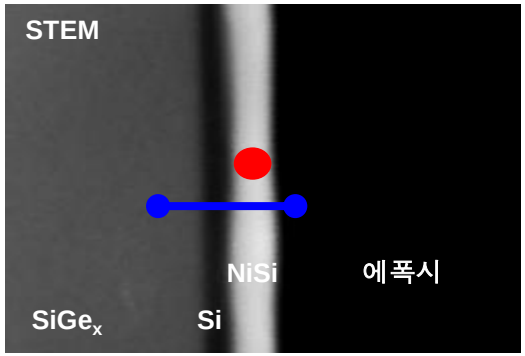
- Selection Rule
  - $\Delta l = \pm 1$

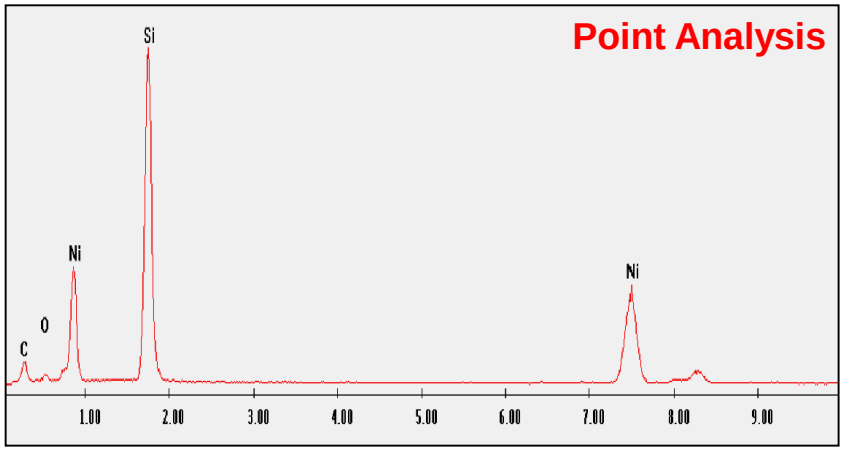
D. Shindo et al., Analytical Electron Microscopy for Materials Science

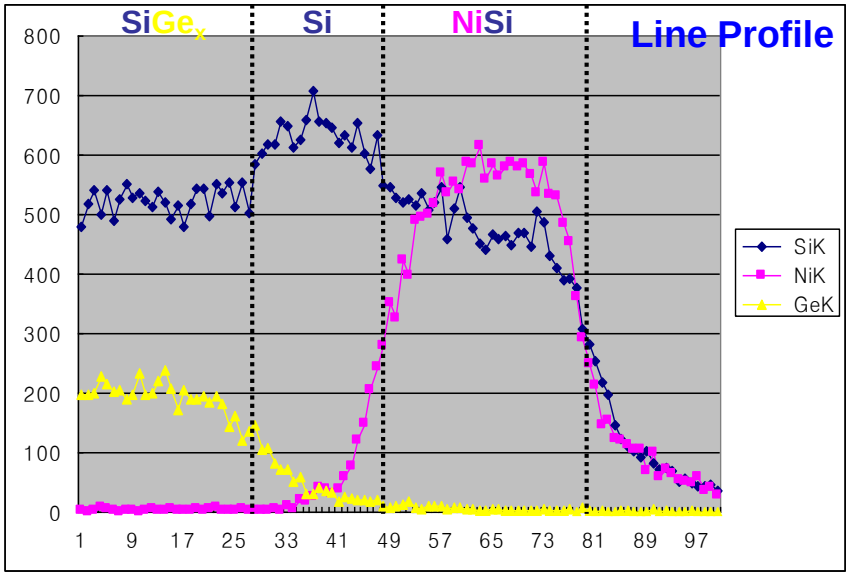
D. B. Williams et al., Transmission Electron Microscopy: A Textbook for Materials Science

EDS 분석 사례

- **EDS 분석 사례 : Point 분석 및 Line 프로파일**

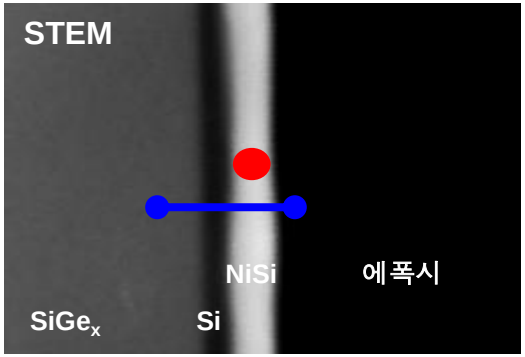


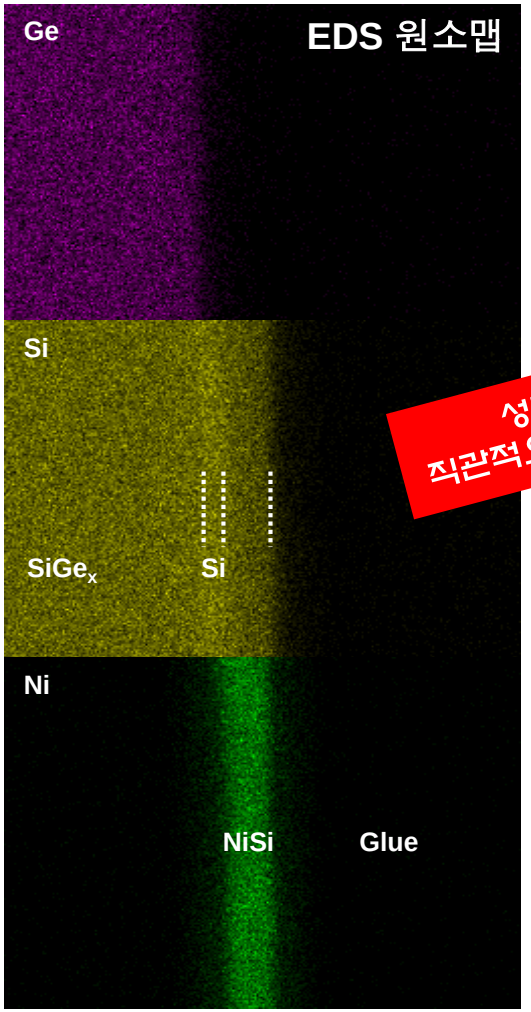




EDS 분석 사례

● EDS 분석 사례 : 원소 Map

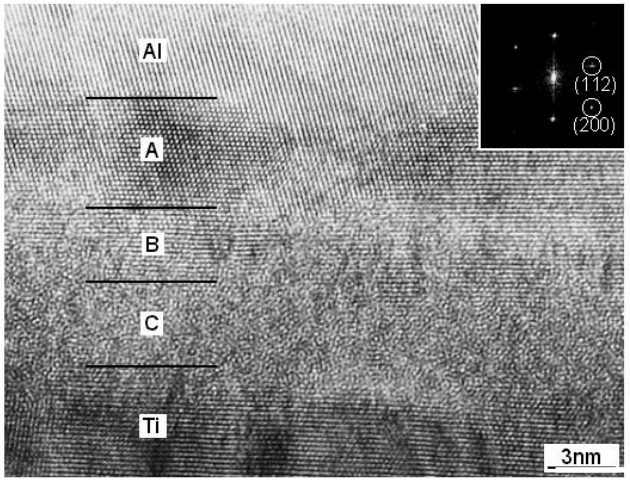


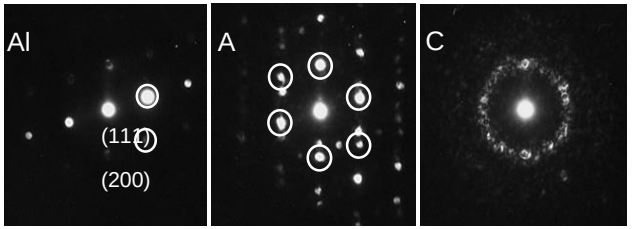


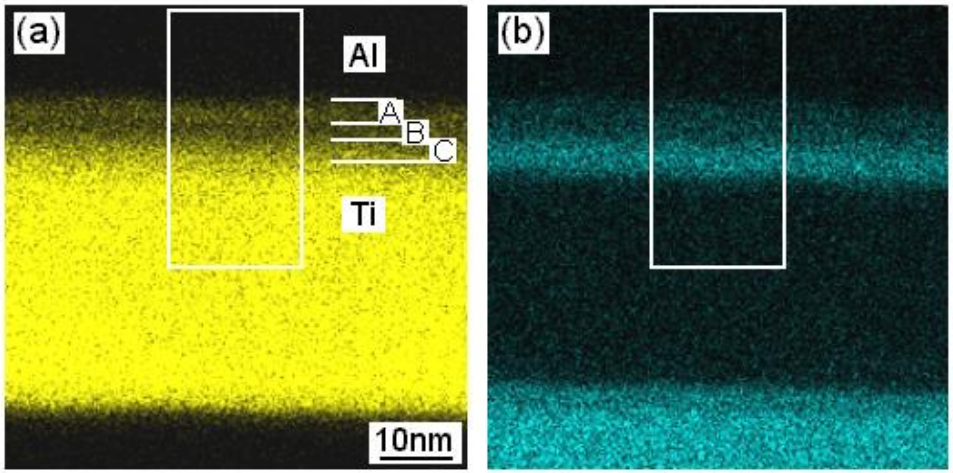
성분 분포
직관적으로 확인 가능

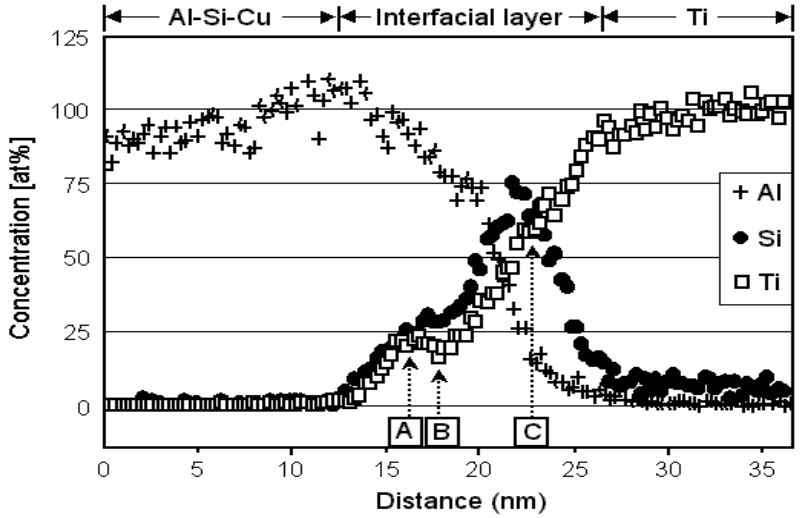
FE-TEM / HRTEM & EDS 분석

● Al-Si-Cu/Ti Film 계면의 나노스케일 계면구조 분석 : HRTEM + EDS







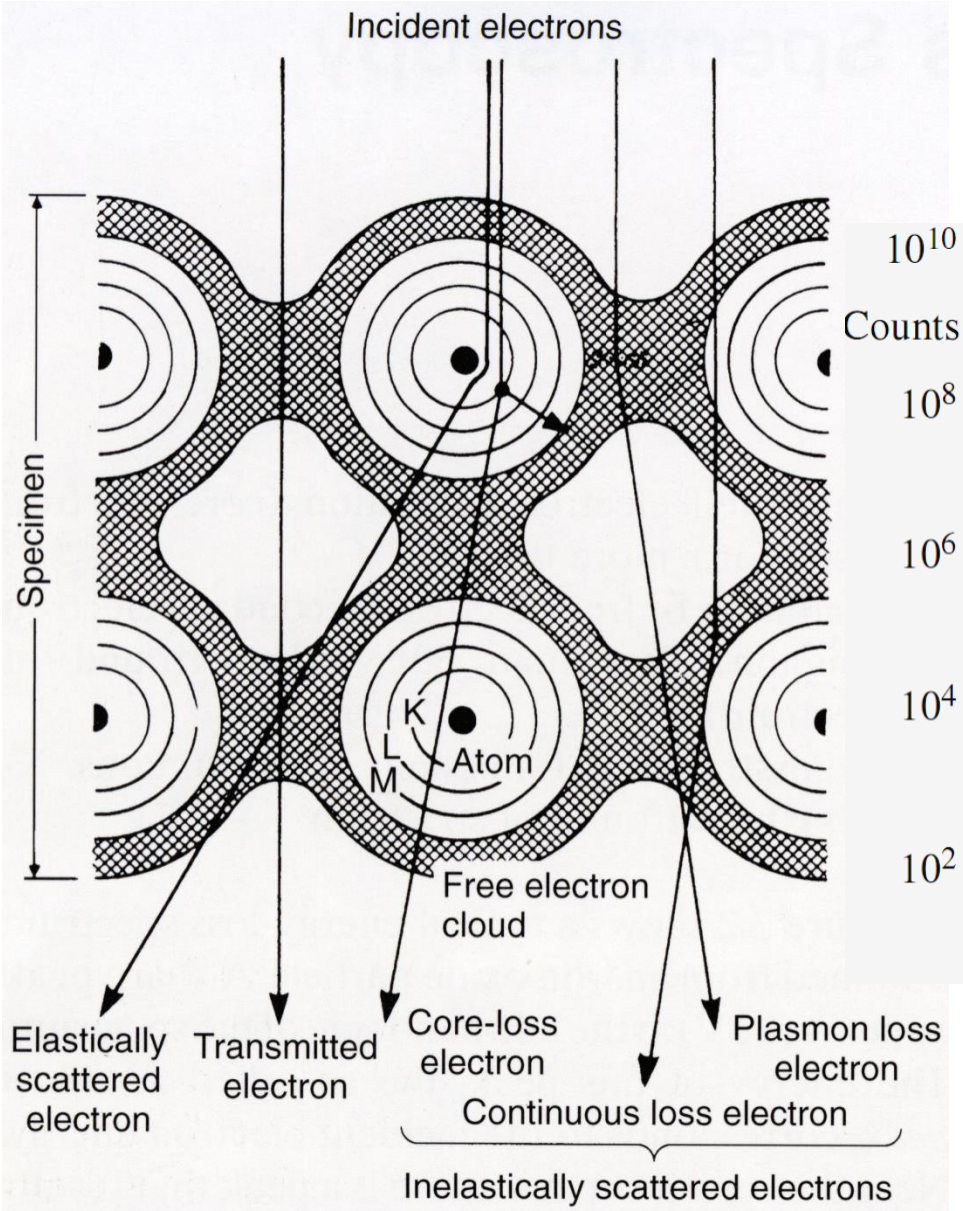


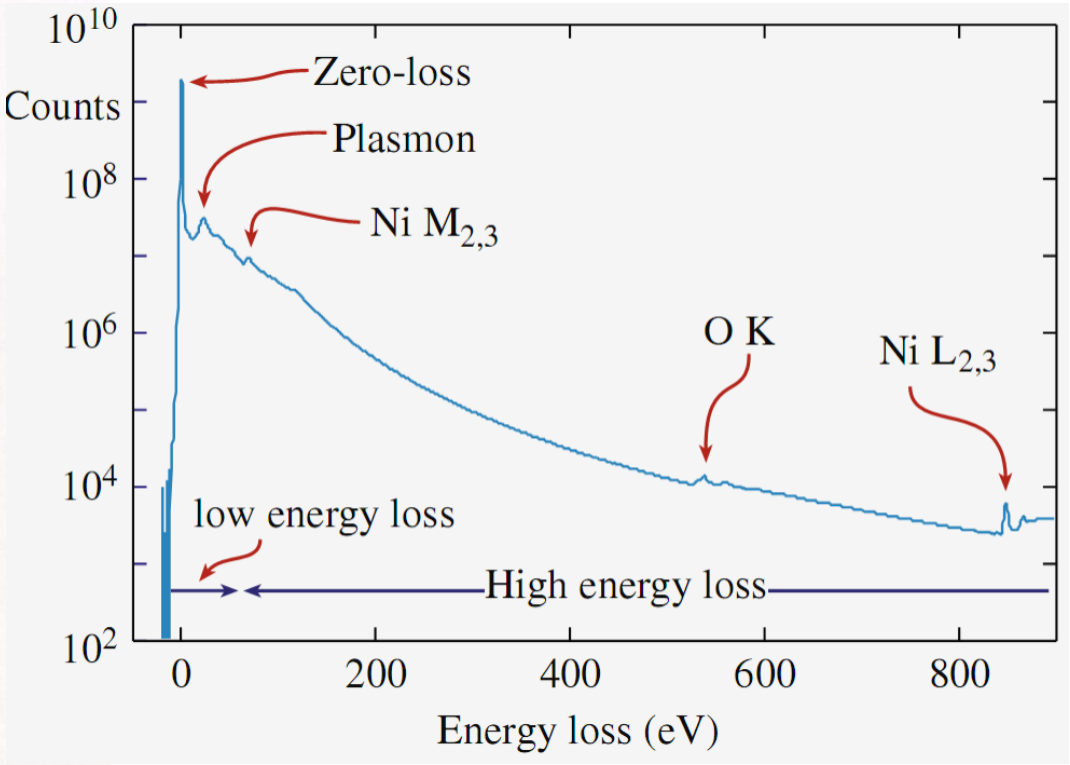
Results

- A층의 조성 Ti:Al:Si=1:3:1
- C층의 조성 Ti:Si=1:1

EELS 성분분석

EELS 원리

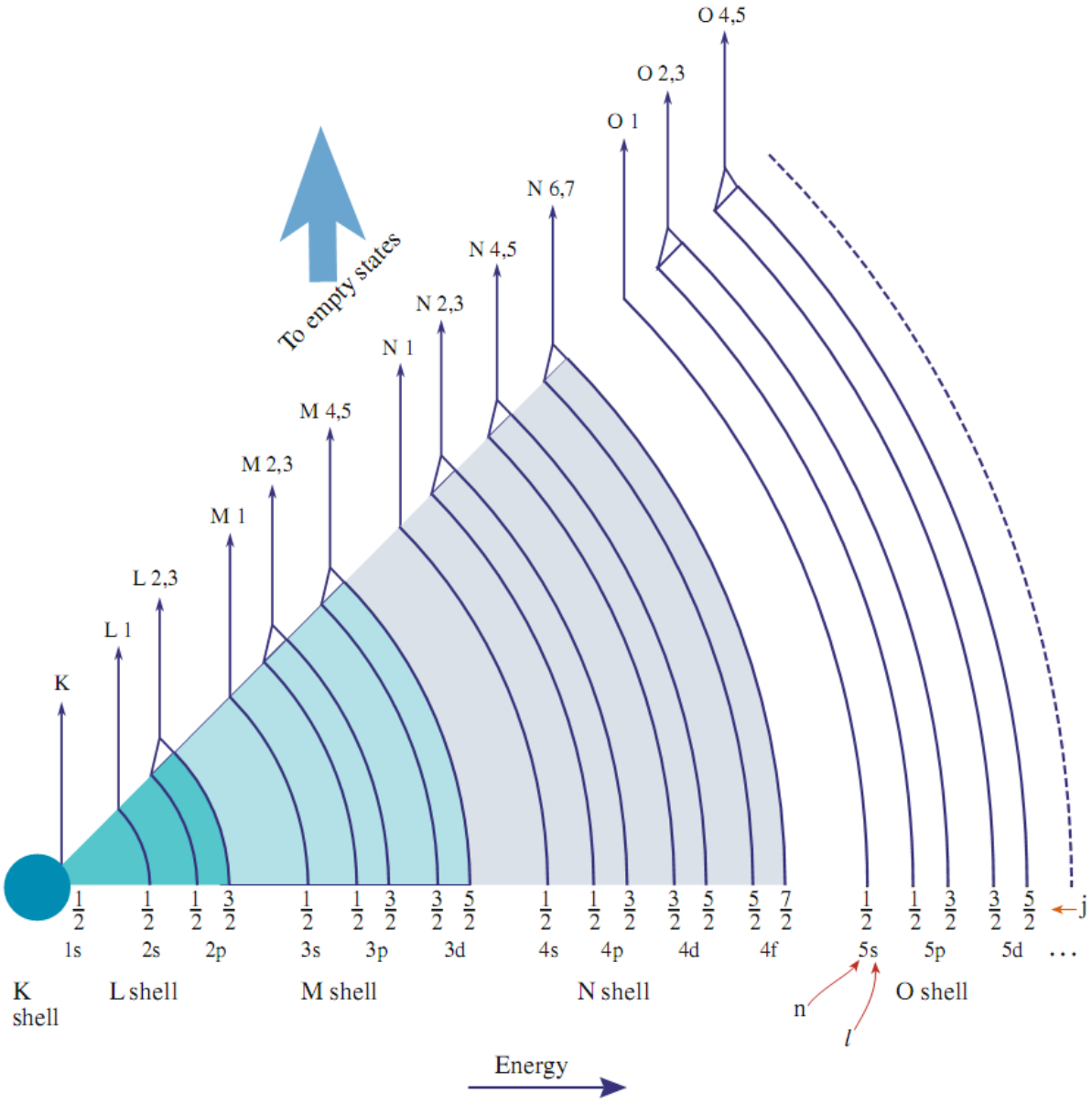




D. B. Williams et al., Transmission Electron Microscopy:
A Textbook for Materials Science

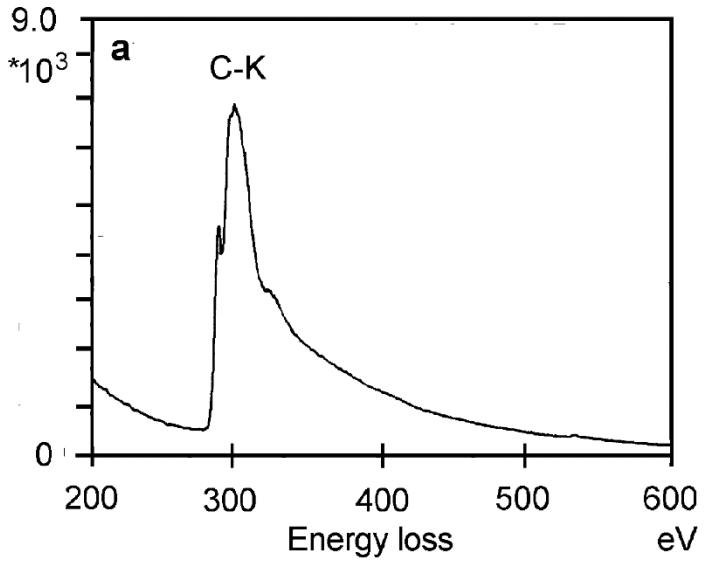
D. Shindo et al., Analytical Electron Microscopy for Materials Science

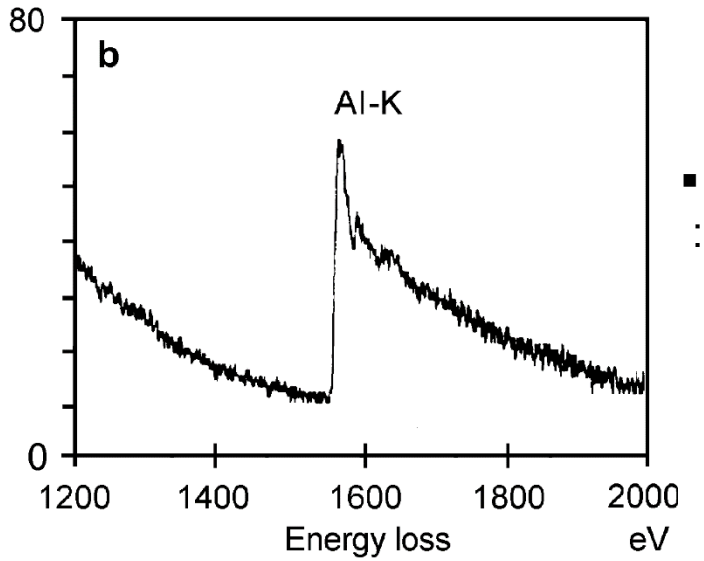
EELS Peak of High Energy Loss



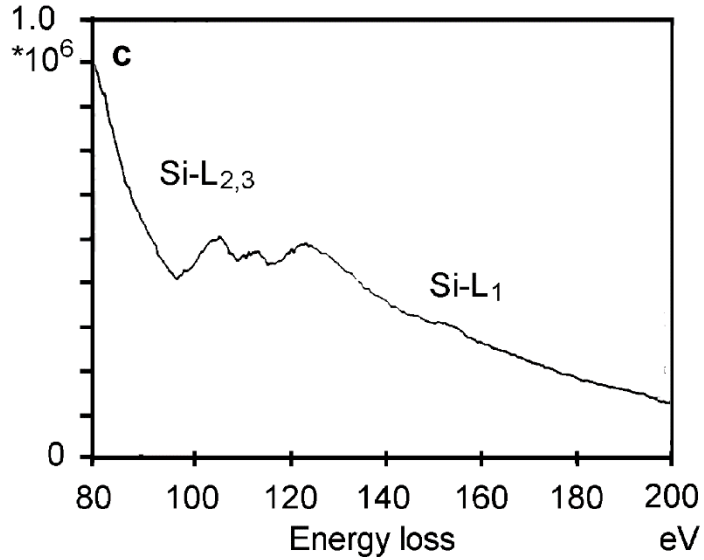
D. B. Williams et al., Transmission Electron Microscopy: A Textbook for Materials Science

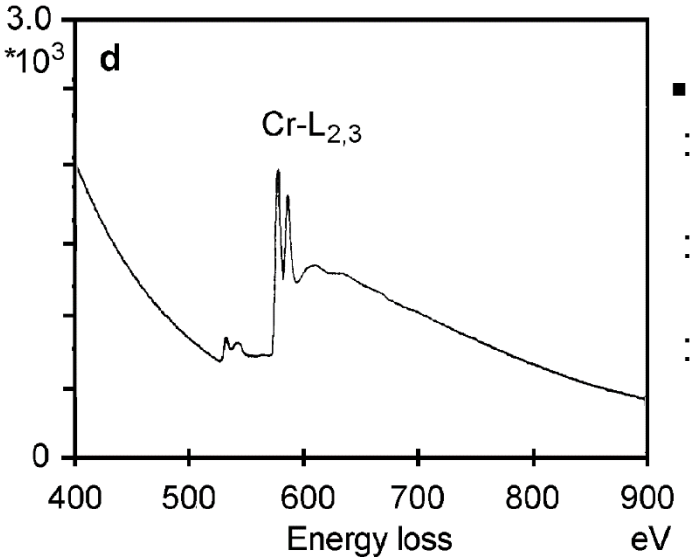
EELS Spectrum : Peak Shape of High Energy Loss





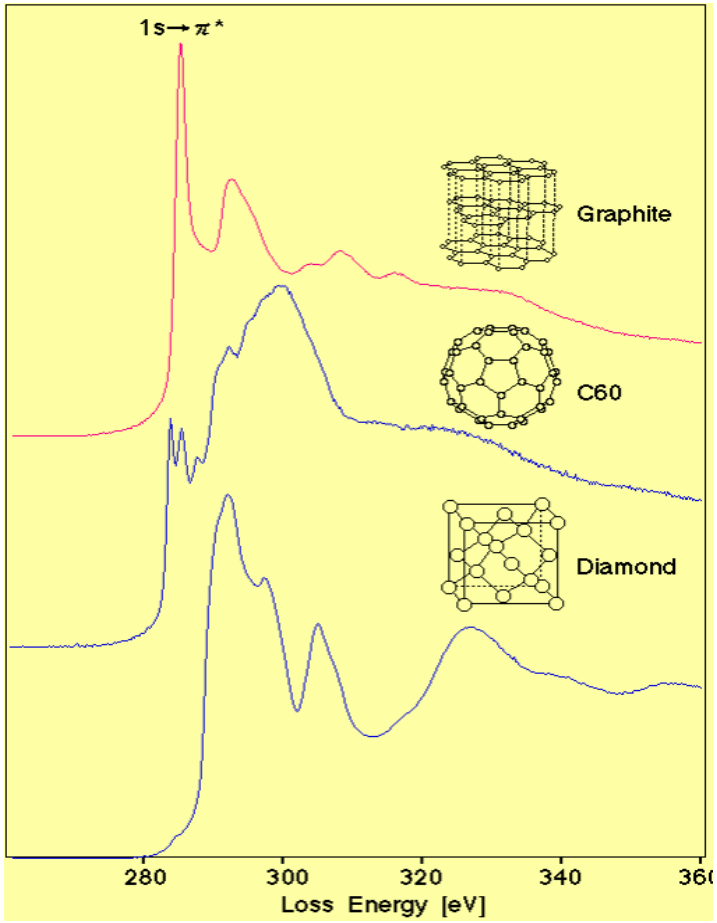
- **K 이온화(K Edge)**
  : Sawtooth Shape

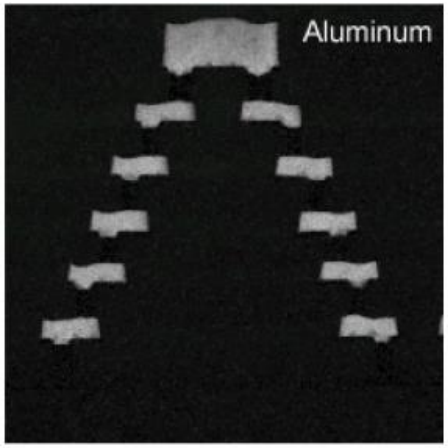


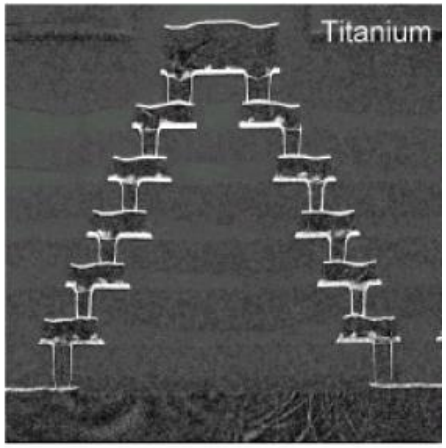


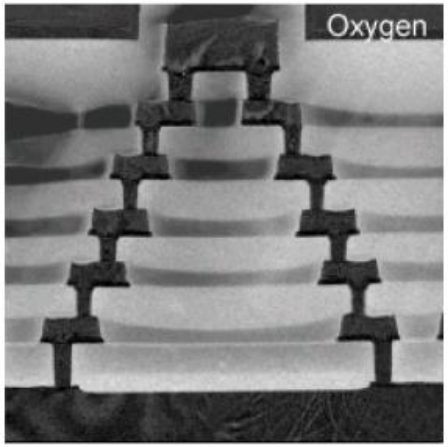
- **L_{23} 이온화(L Edge)**
  : Delayed Maximum
  (Si - Cl)
  : White Line
  (K - Cu)
  : No White Line
  (Cu - Br)

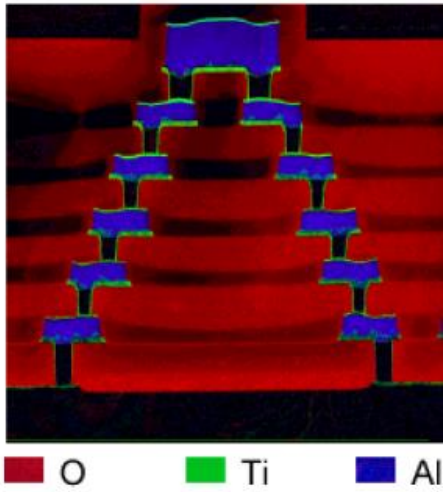
● EELS 분석 사례


1s→π*
Graphite
C60
Diamond
280
300
320
340
Loss Energy [eV]

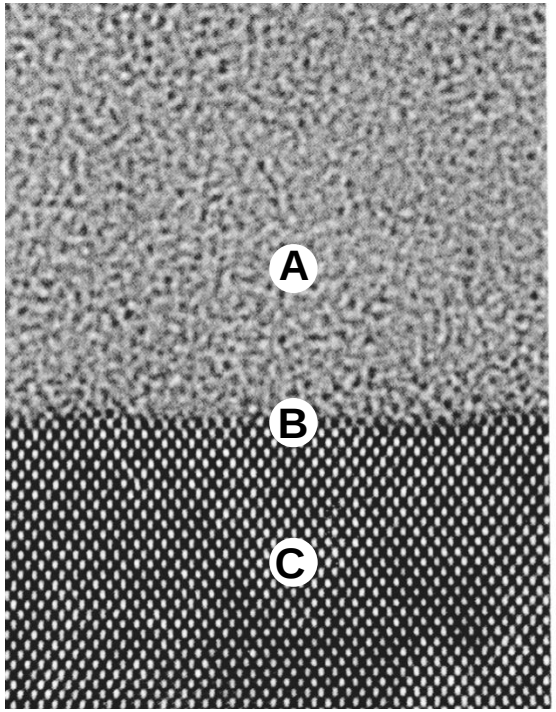

Aluminum

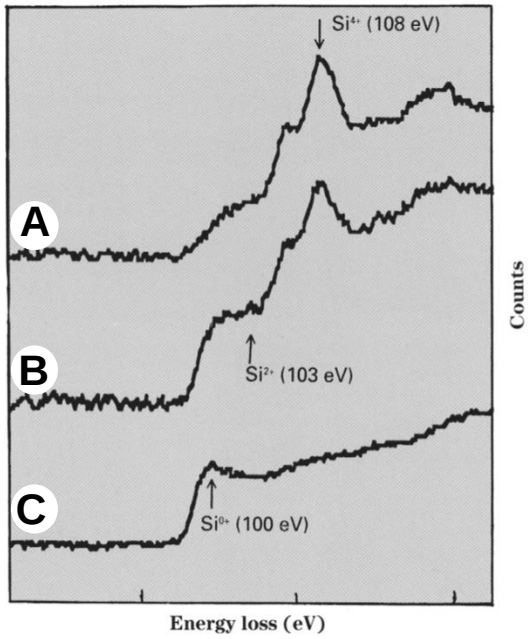

Titanium


Oxygen


O
Ti
Al

● **EELS 분석 사례**



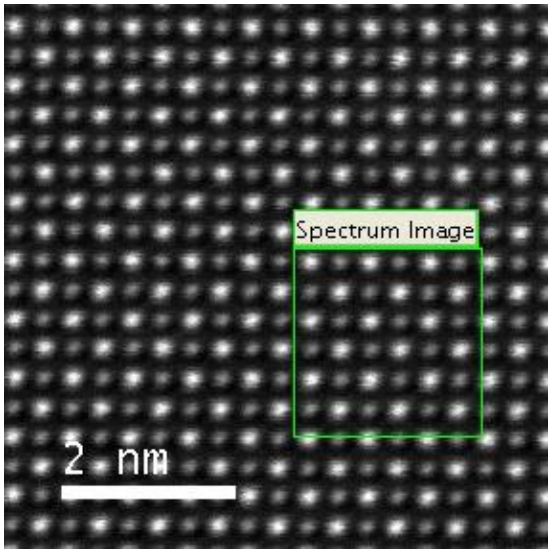


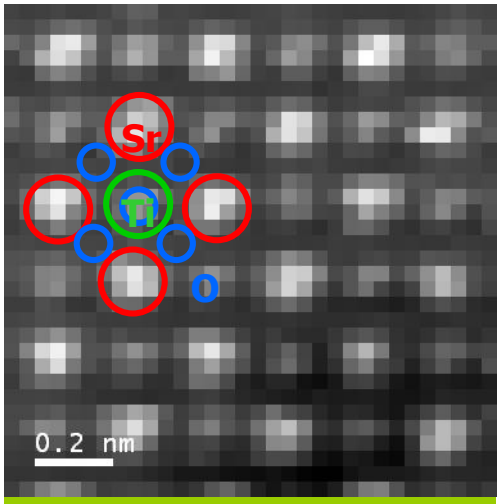
Amorphous Si (A), boundary (B) and Si-crystal (C)

Si^{0+}	Si^{2+}	Si^{4+}
Si | Si–Si–Si | Si	Si | O–Si–Si | O	O | O–Si–O | O

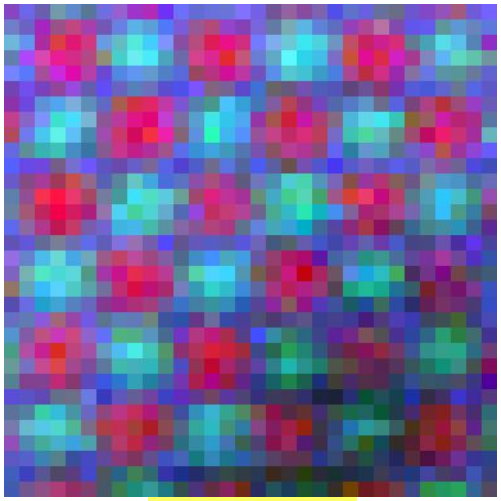
- Core Loss Spectrum의 미세구조
  - ELNES (energy-loss near-edge structure): edge peak에서 약 50 eV의 영역으로서 비점유 상태밀도가 반영된 영역

Atomic column EELS Map ; Specimen : $SrTiO_3$

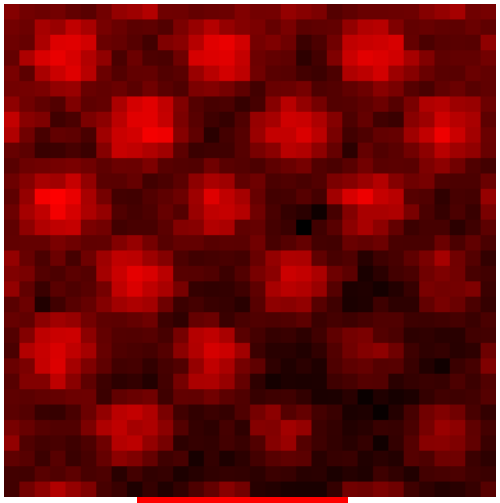




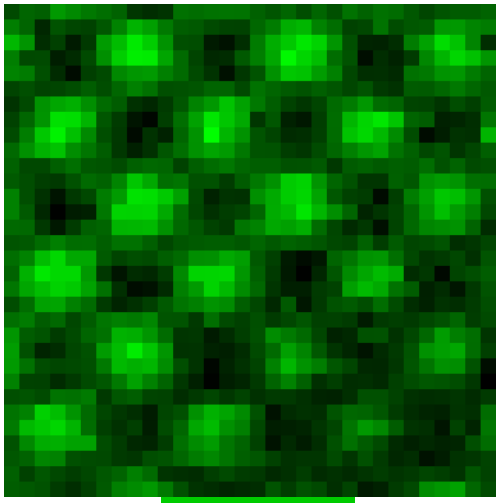
HAADF signal(acquired simultaneously)



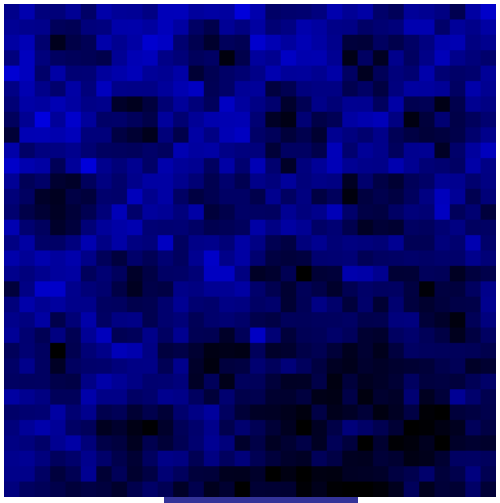
RGB map



Sr-M map



Ti-L map

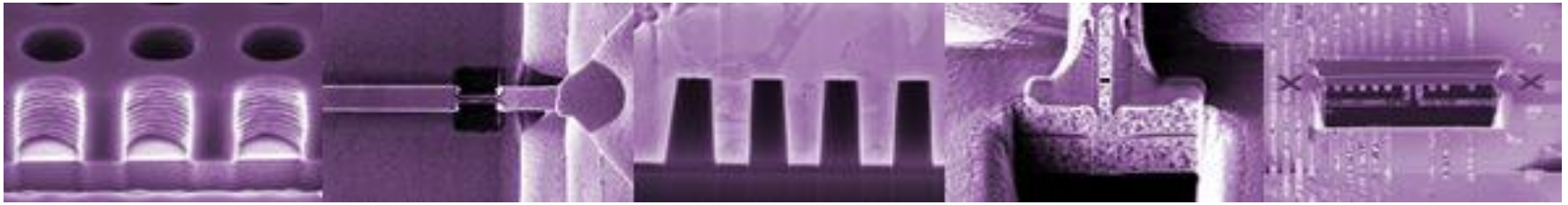


O-K map

集束이온빔 분석

Focused Ion Beam (FIB)

Focused Ion Beam (FIB)

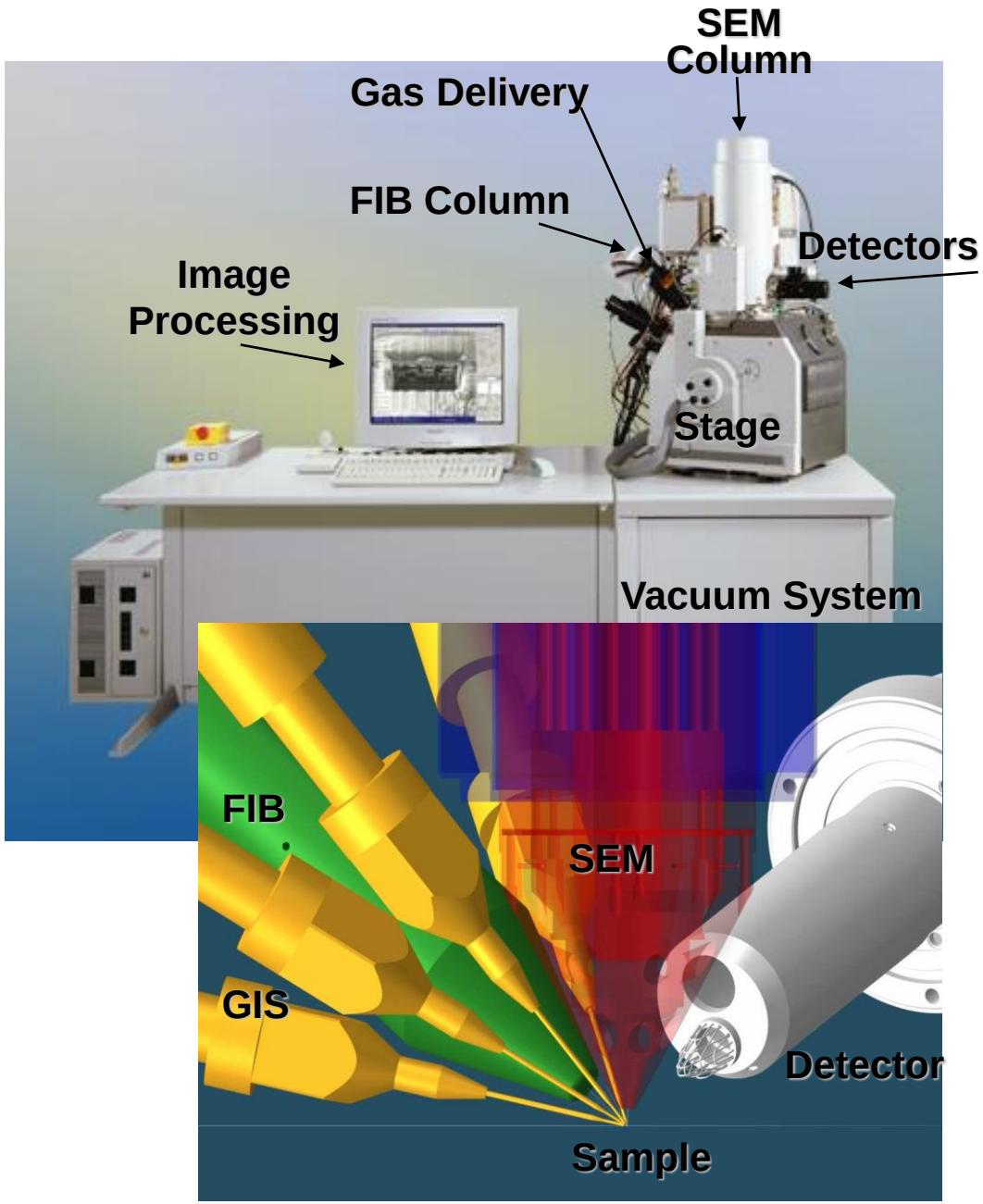


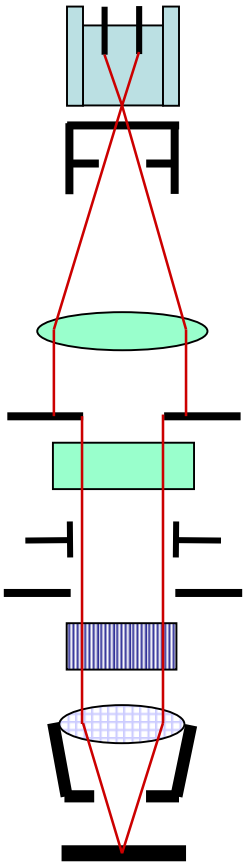
- ***Focused Ion Beams can image an IC's surface as well as add and remove conductors and insulators.***

- Focused Ion Beam (FIB) technology allows the capability to *locally etch* materials by milling a sample surface with a highly focused ion beam. This same beam can also be used to *locally deposit conductors and insulators*. These operations can be made with submicron precision and give rise to a number of useful applications.

- Typically FIB is utilized in on-chip *circuit modification* (redesign/repair, device isolation, probe pad formation) and advanced *circuit diagnostics* for failure analysis (cross-sectioning and TEM / SEM sample preparation).

Dual Beam FIB System





Ga^+ LMIS

Extractor Cap

Beam Acceptance Aperture (BAA)

Condenser Lens

Beam Defining Aperture (BDA)

Quadrupole

Beam Blanking Plate

Beam Blanking Aperture

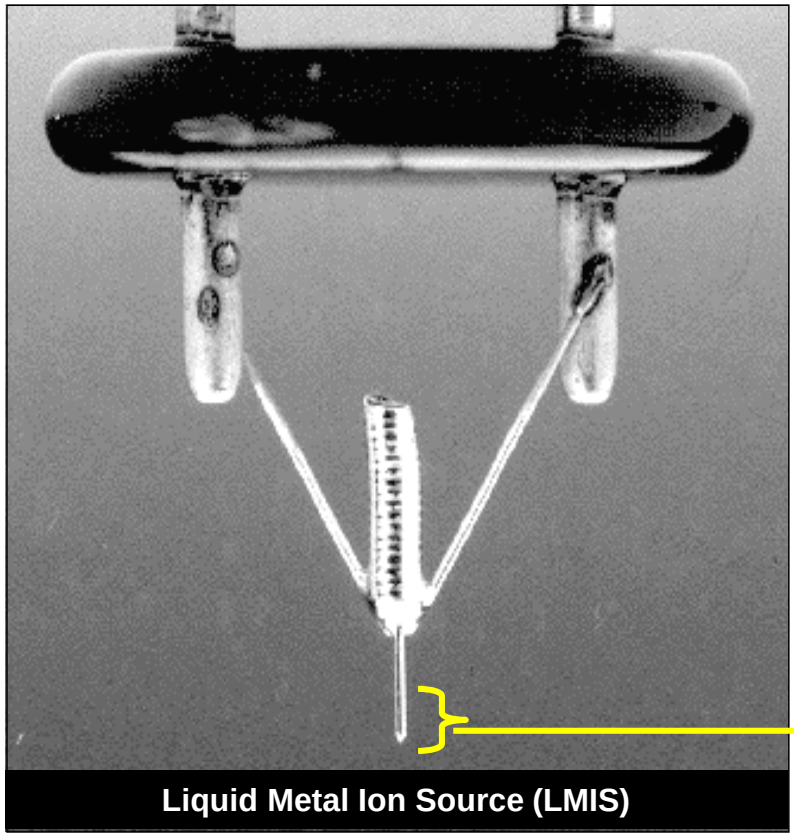
Deflection Octopole

Objective Lens

Sample

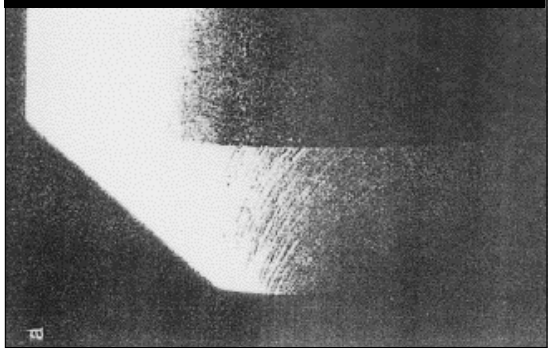
Ion Column for FIB

Liquid Metal Ion Source (LMIS) - Gallium

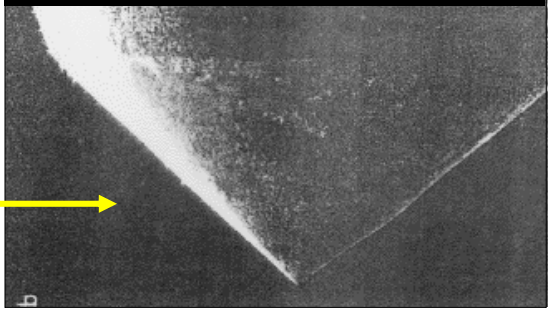


Liquid Metal Ion Source (LMIS)

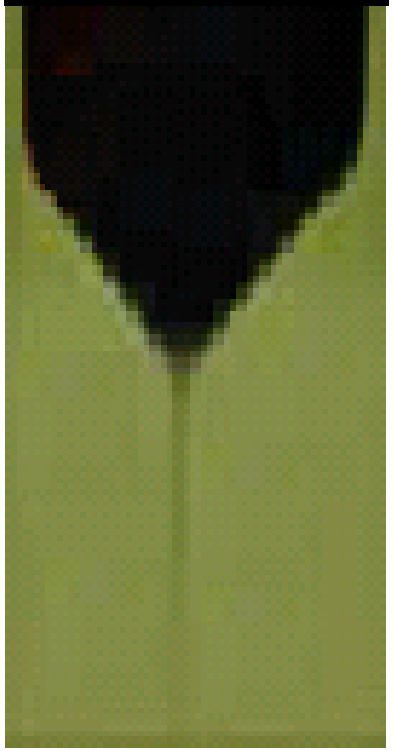
Tungsten tip without Gallium



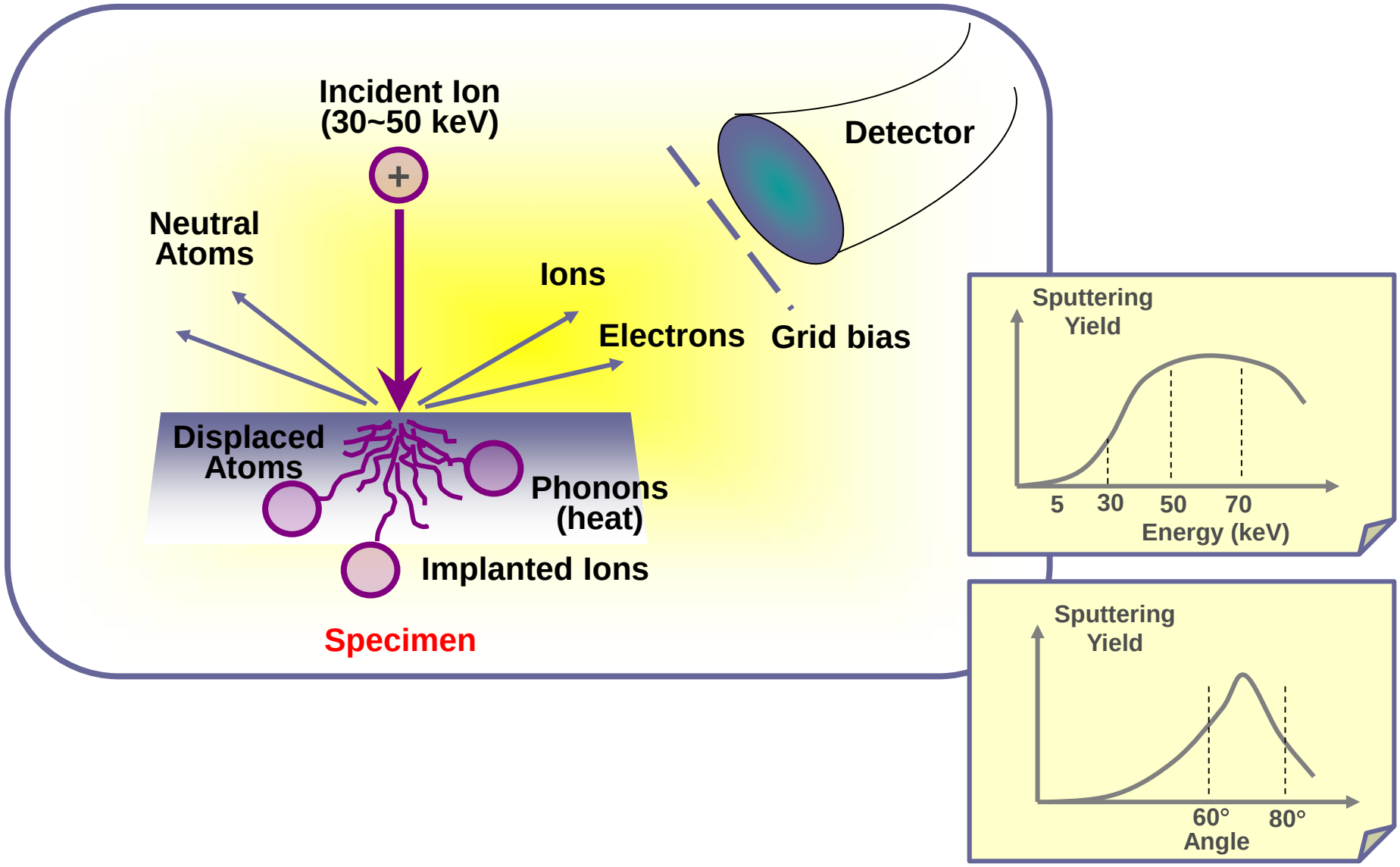
Tungsten tip with Gallium forming a “Taylor Cone”



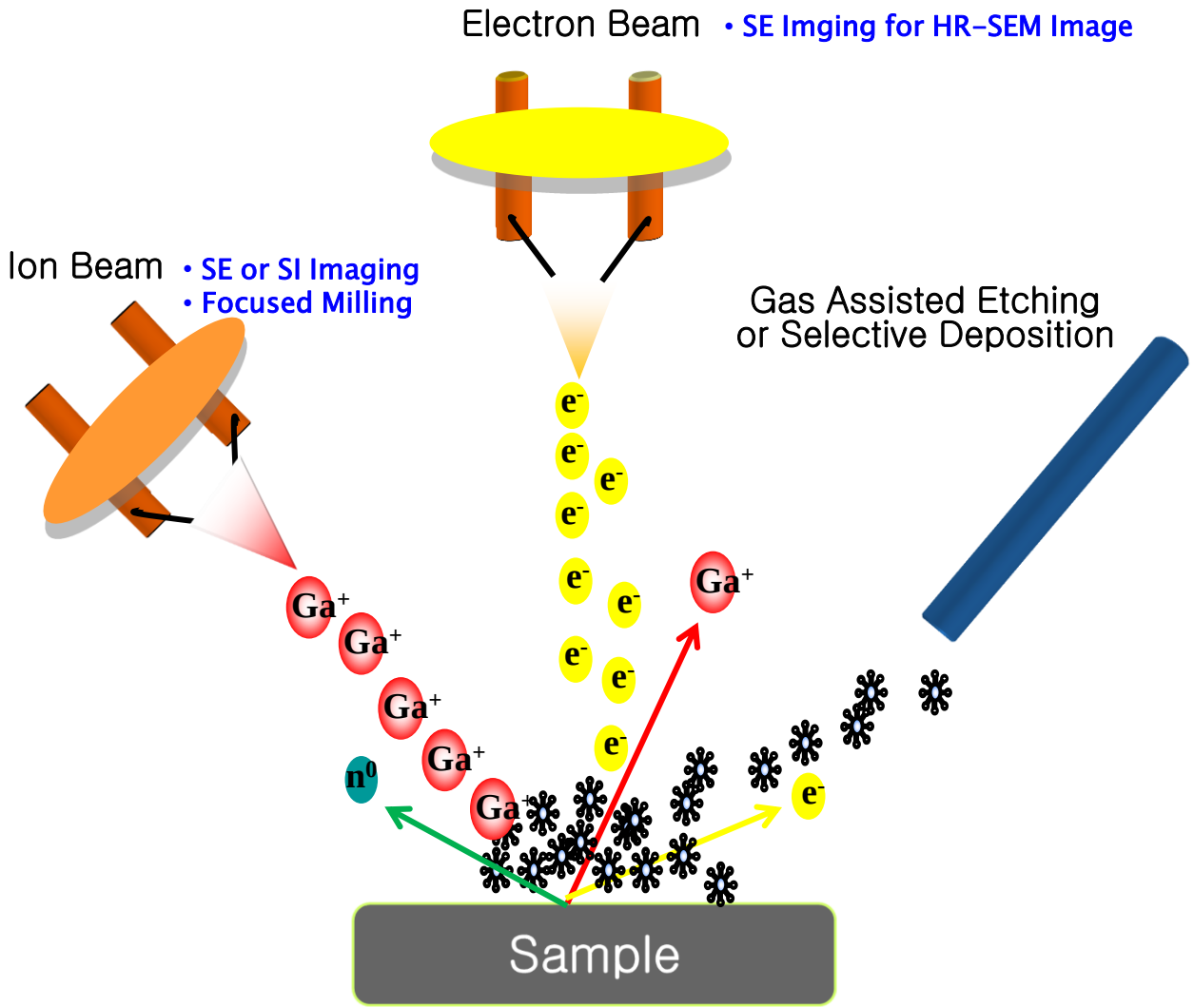
Ion Emission from the LMIS “Taylor Cone”



Focused Ion Beam (FIB)

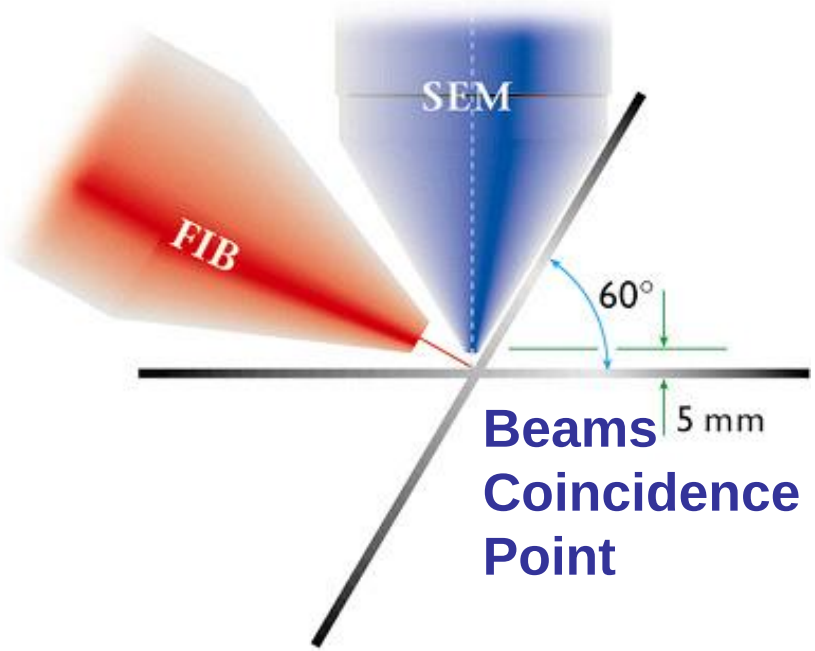


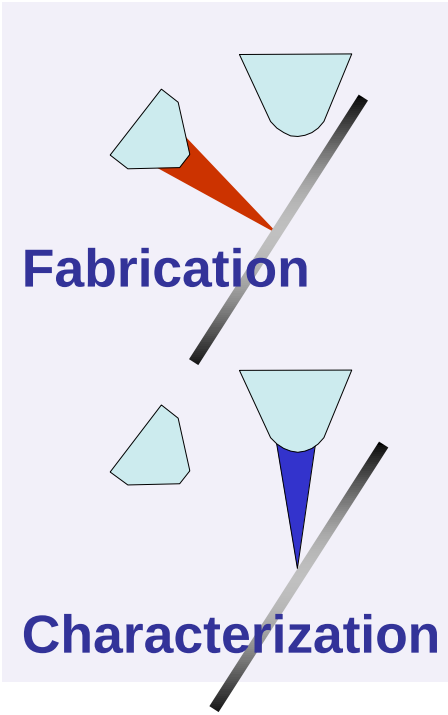
Dual Beam FIB System



Dual Beam Geometry

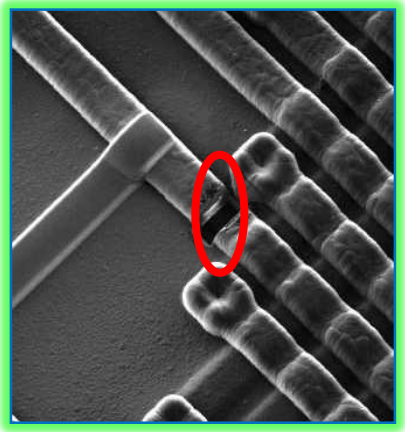
- **Dual Beam = SEM + FIB**
- **FIB (Focused Ion Beam) = (Ga^+, 1 pA – 20 nA, > 5 nm spot)**
- **SEM = ~1 nm resolution**
- **Options : EDS, EBSD, STEM**



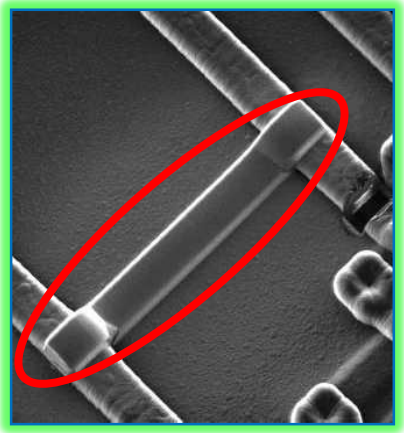


Applications of FIB Techniques

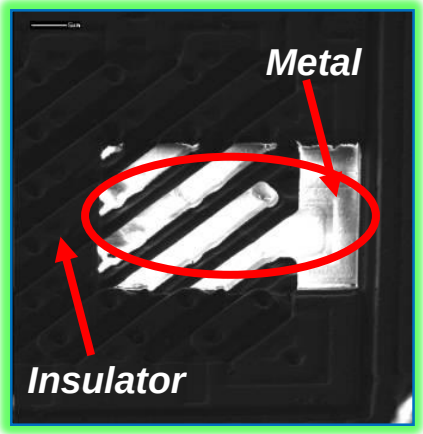
Milling



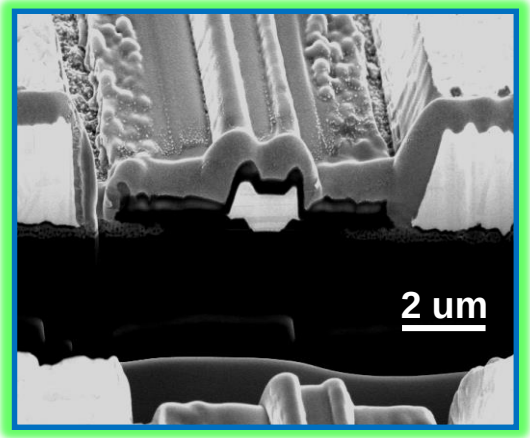
Metal Deposition (Pt)

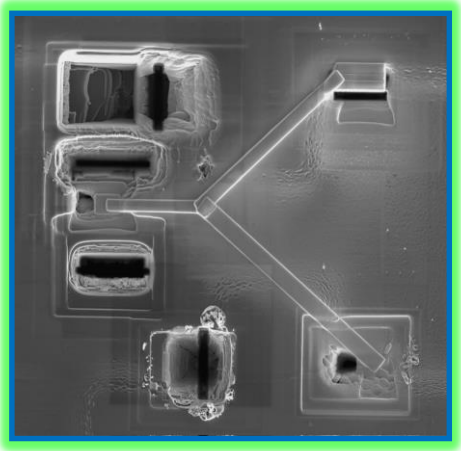


Enhanced Etch



Cross Section

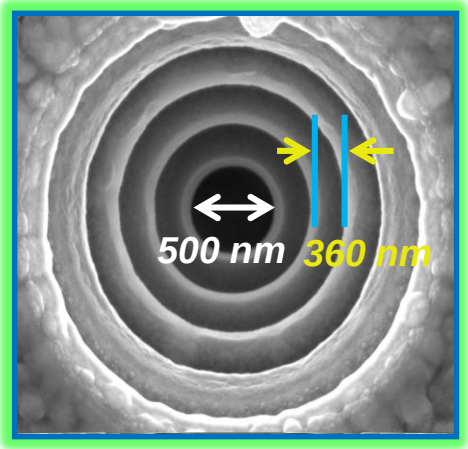




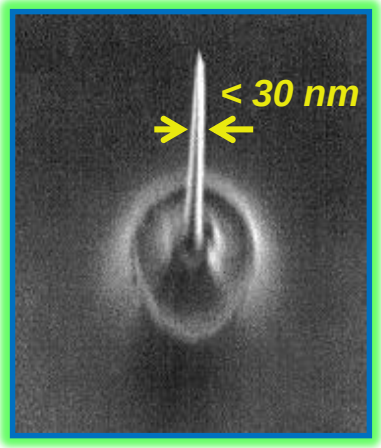
회로수정



미세 **Hole** 가공



Step Hole 가공



SPM Tip 가공

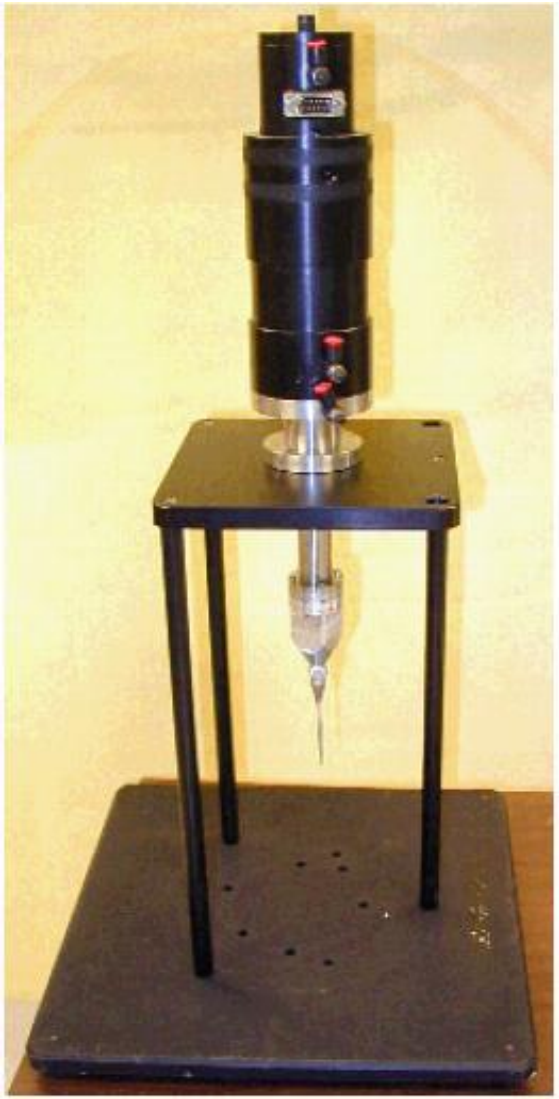
Depositing & Etching Gas Delivery Systems

Deposition Gas Injector Systems (GIS)

1) **Platinum**
2) **Tungsten**
3) **Carbon**
4) **Insulator Deposition**

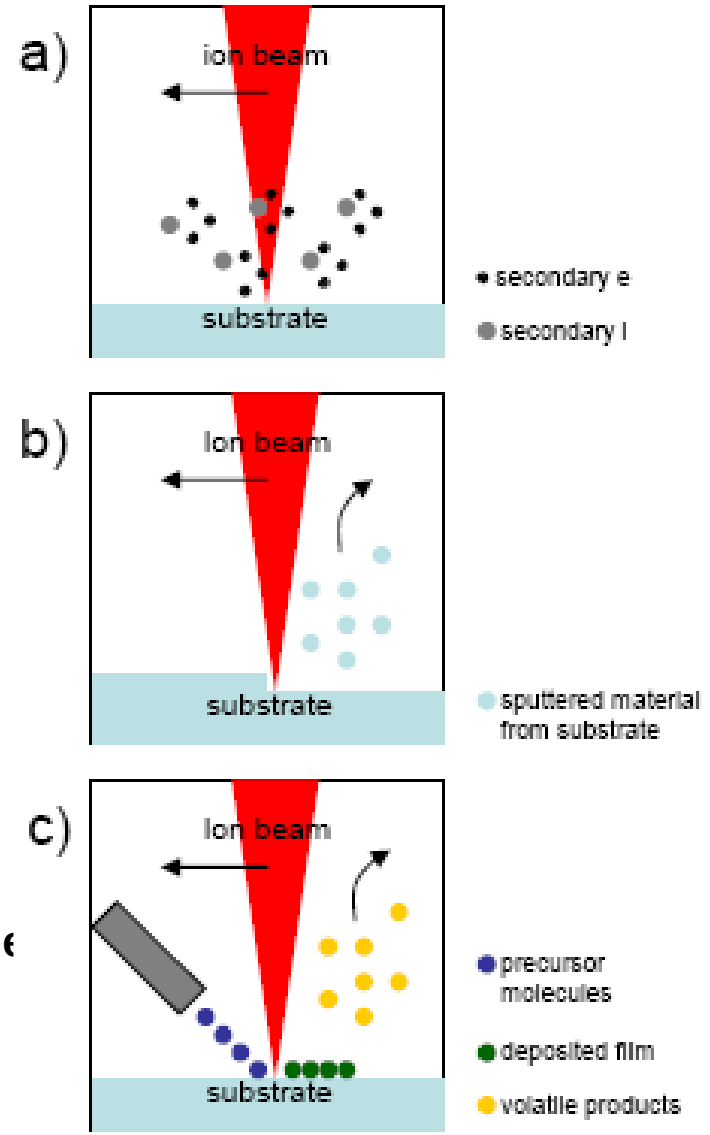
Reactive Gas Injector Systems (GIS)

5) **Enhanced Etch -“EE”**
6) **Insulator Enhanced Etch -“IEE”**
7) **Delineation Etch - “DE”**
8) **Selective Carbon Mill - “SCM”**
9) **CoppeR_x**



Operating Modes of FIB

- **Emission of Secondary Ions and Electrons (a)**
  - **FIB imaging**
    Low ion current
- **Sputtering of Substrate Atoms (b)**
  - **FIB milling**
    High ion current
- **Chemical Interactions (Gas Assisted) (c)**
  - **FIB deposition**
  - **Enhanced (preferrential) etching**
- **Other Effects**
  - **Ion implantation**
  - **Displacement of atoms in the solid : Induced damage**
  - **Emission of photons : Heating**

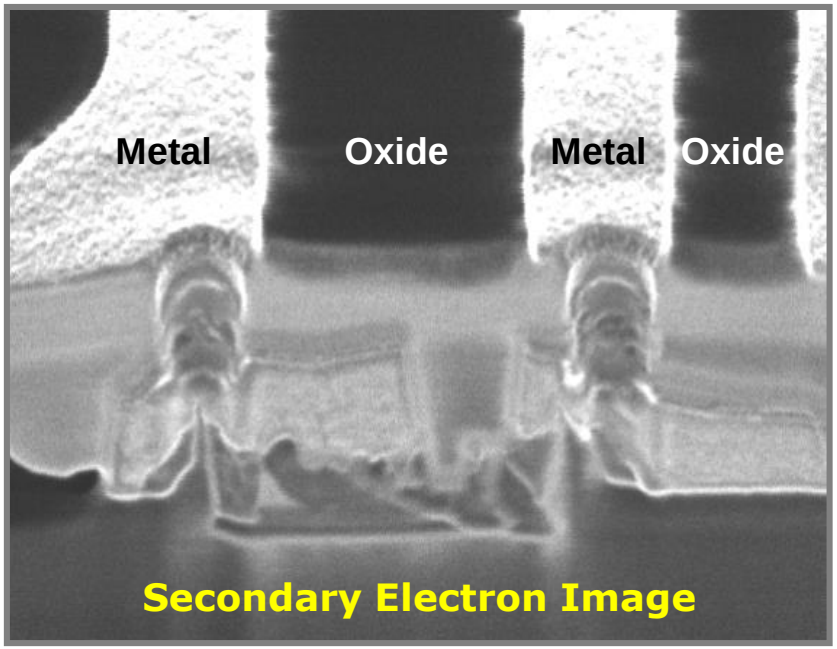


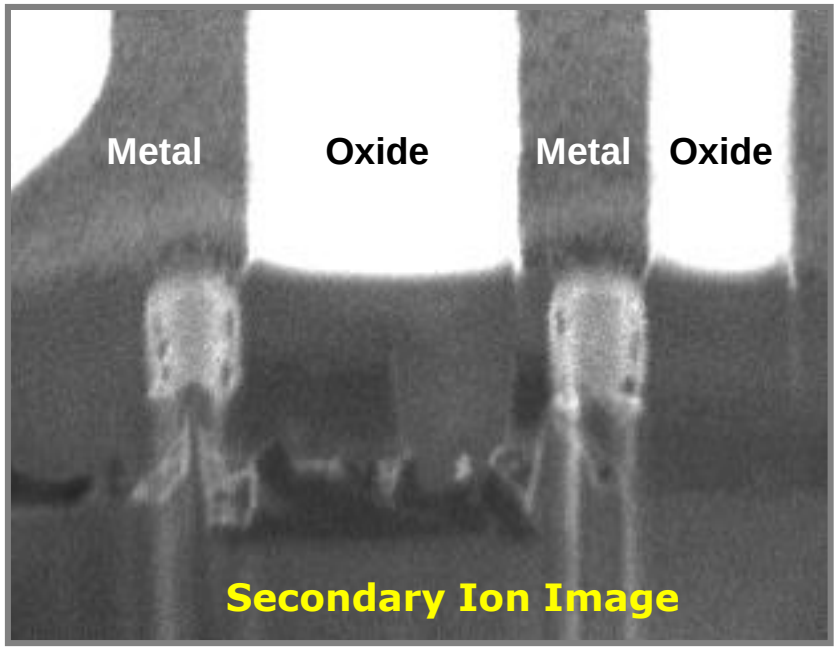
Dual-beam FIB의 응용

- **Imaging**
- **Milling, Deposition, Etching**
- **Cross-section Analysis**
- **Circuit Modification**
- **TEM Sample Preparation**
- **Micro-machining**
- **Nano-fabrication**

FIB Imaging (Scanning Ion Microscopy)

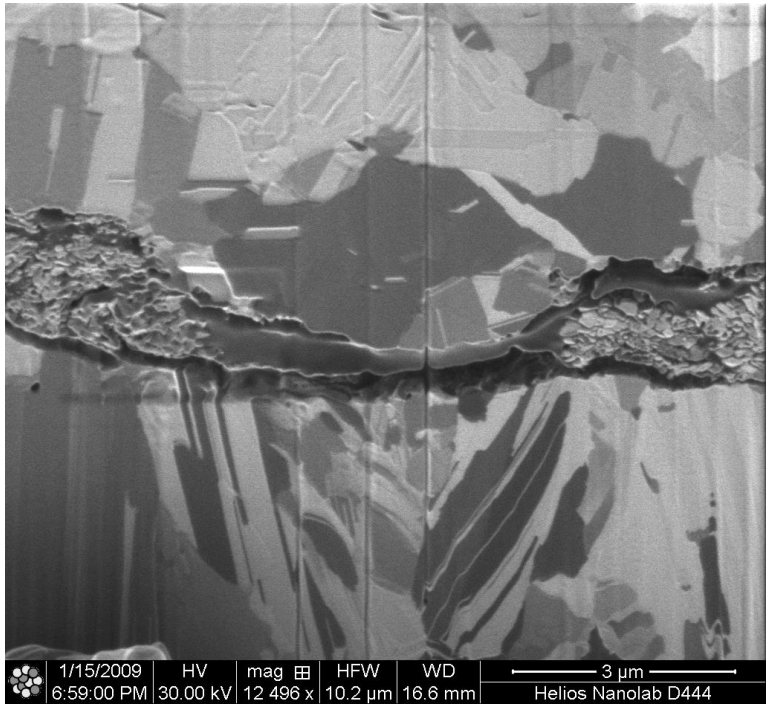
- **Morphology : Topology & Grain boundary**
- **SE(이차전자) : Topology effect ; High-resolution**
- **SI(이차이온) : Ion channeling effect ; Grain boundary contrast**



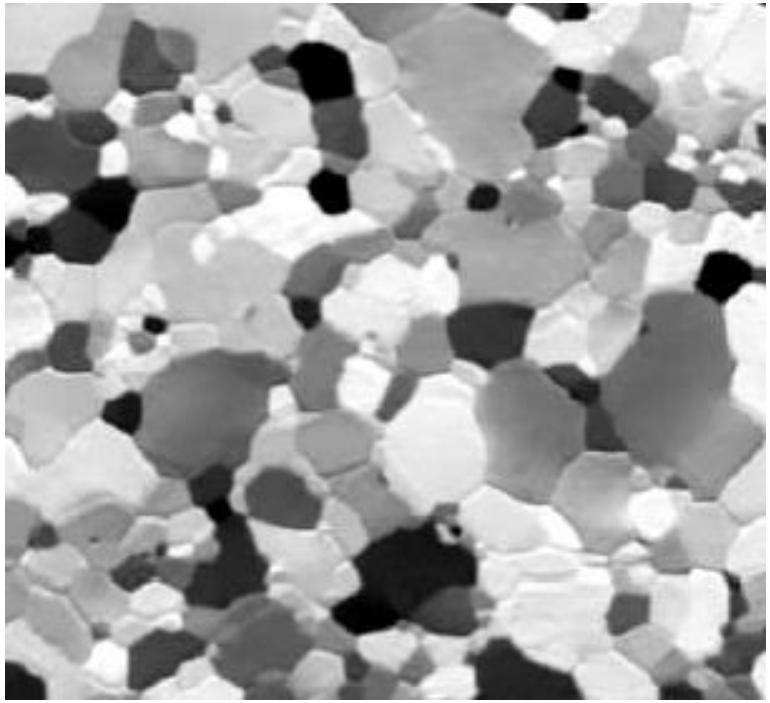


FIB Imaging (Scanning Ion Microscopy)

- **Morphology : Topology & Grain boundary**
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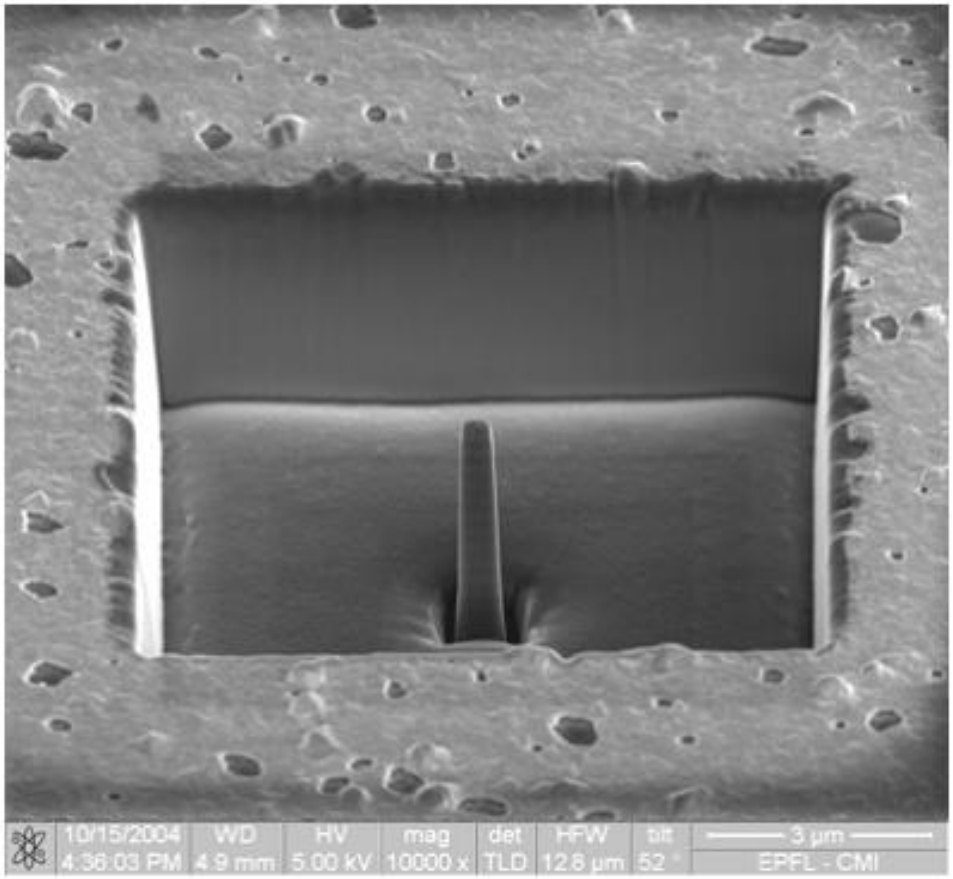


Ni-Cu



Al film

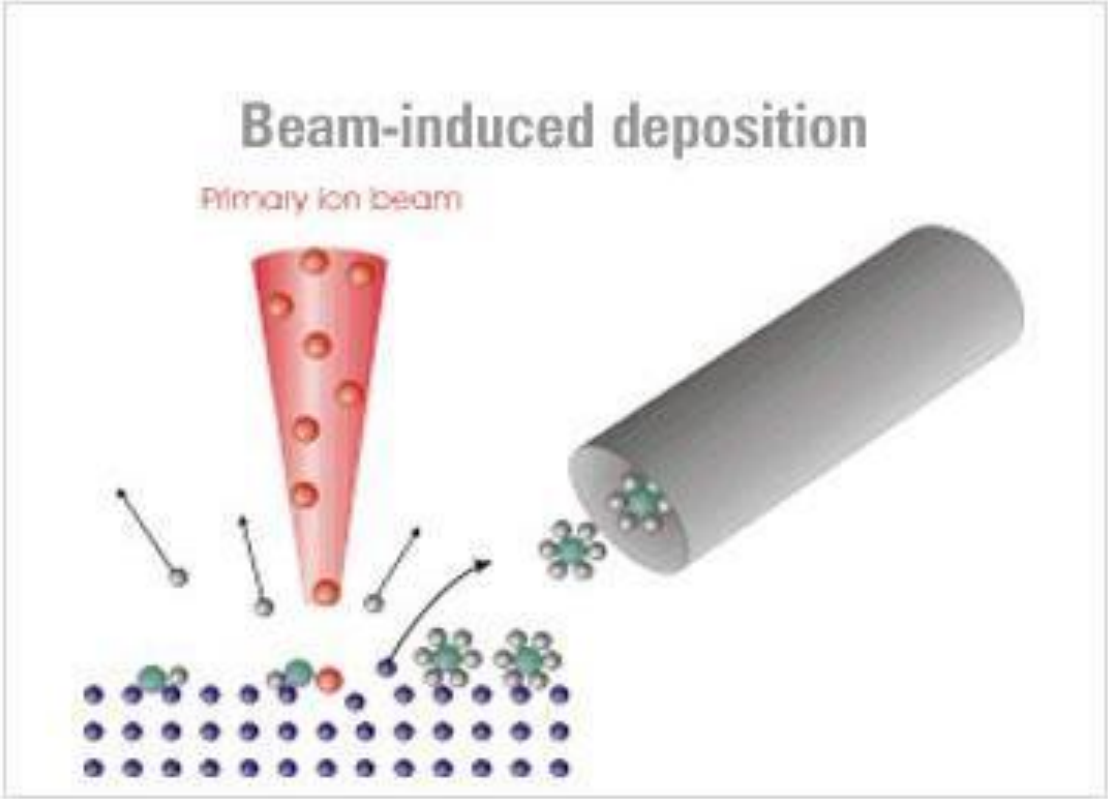
FIB Milling

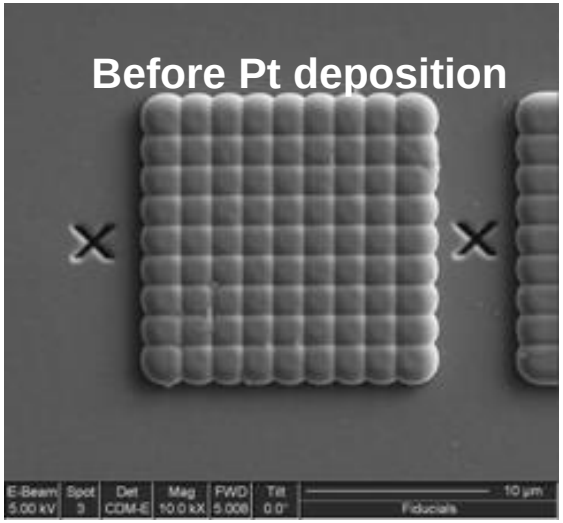


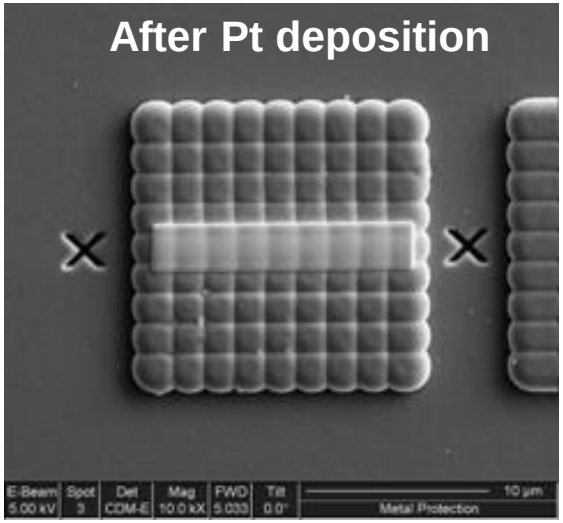
Material	Sputterrate [µm³/nC]
Si	0.27
Thermal Oxide	0.24
TEOS	0.24
Al	0.3
Al2O3	0.08
GaAs	0.61
InP	1.2
Au	1.5
TiN	0.15
Si3N4	0.2
C	0.18
Ti	0.37
Cr	0.1
Fe	0.29
Ni	0.14
Cu	0.25
Mo	0.12
Ta	0.32
W	0.12
MgO	0.15
TiO	0.15
Fe2O3	0.25
Pt	0.23
PMMA	0.4

Metal Deposition

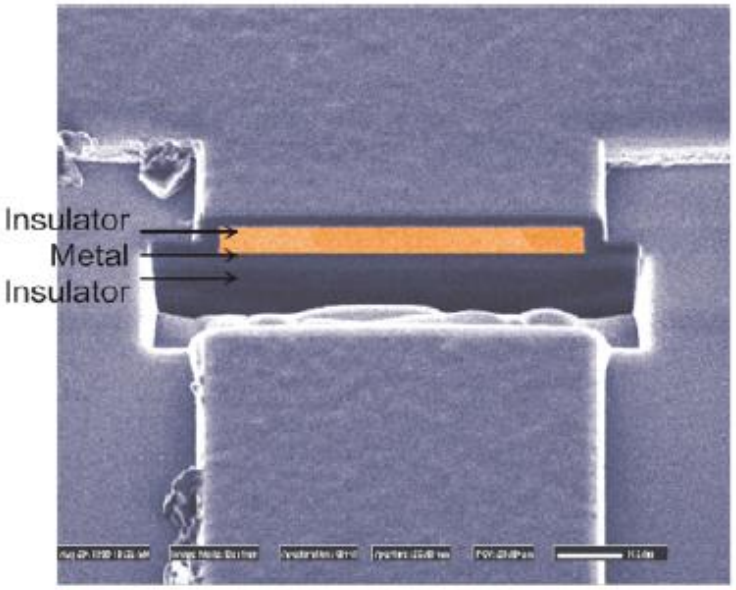
A heated capillary is inserted close to the surface and injects a precursor gas (PT-based metallorganic molecules). These molecules adsorb onto the sample surface. When the incident beam comes in contact with them, it breaks their weak hydrocarbon bonds. The free hydrocarbons are then carried away by vacuum, leaving the metal behind.

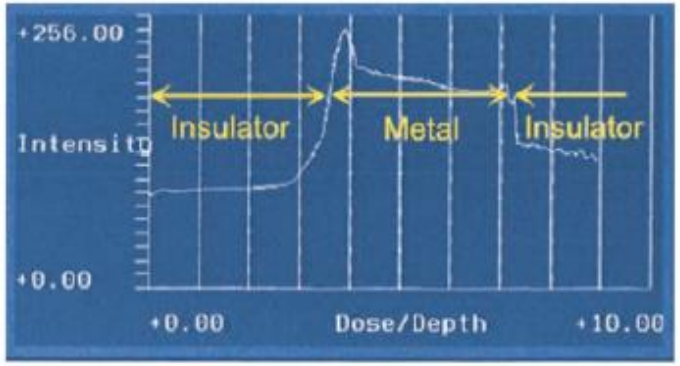


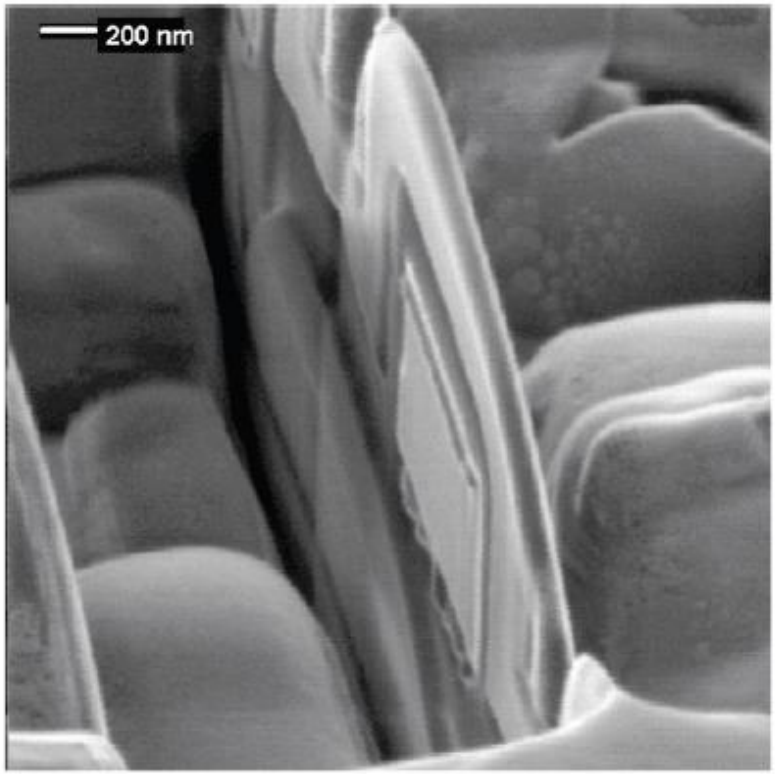




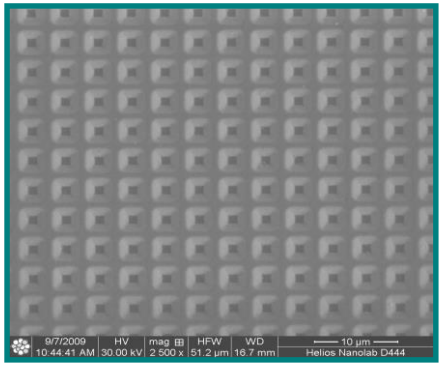
Etching


Insulator
Metal
Insulator

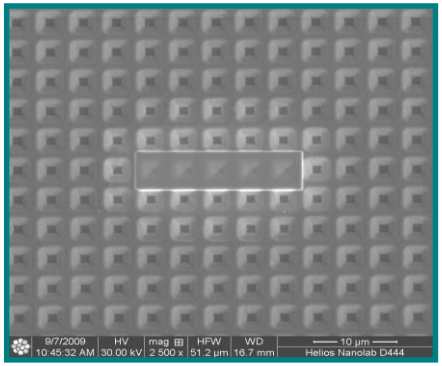

+256.00
Intensity
+0.00
Insulator
Metal
Insulator
+0.00
Dose/Depth
+10.00


200 nm

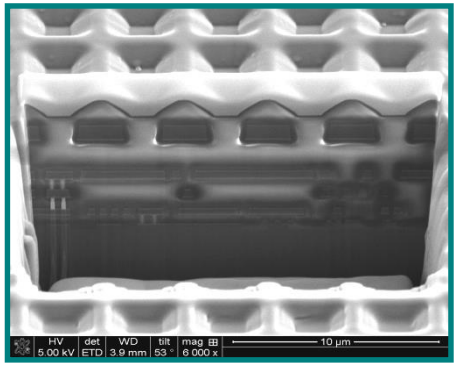
Cross-section Analysis



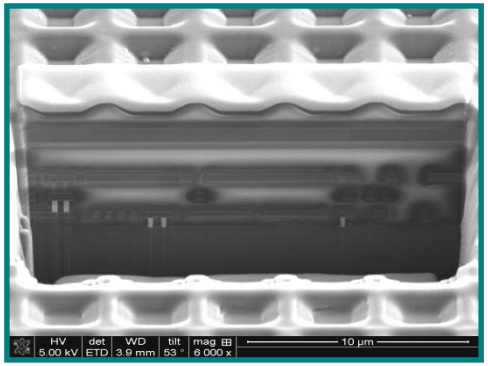
1. 분석위치 찾기



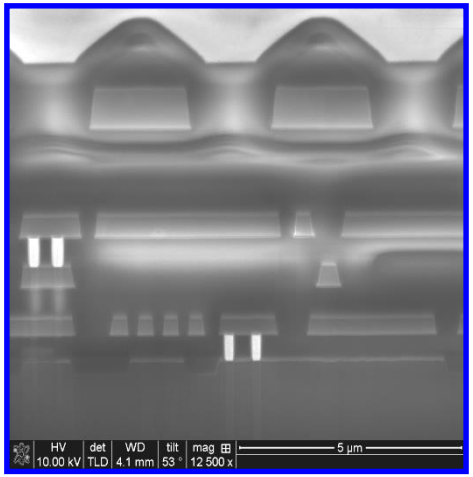
2. 표면보호를 위해 증착



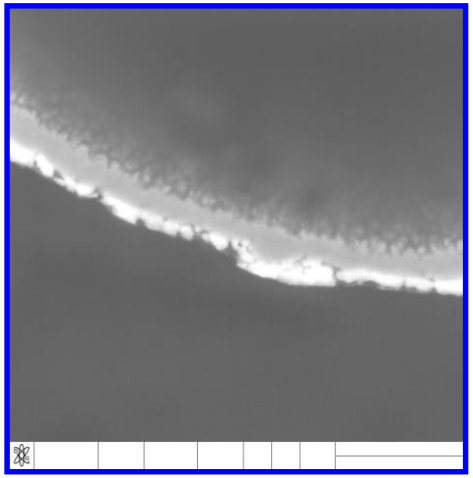
3. Rough-milling



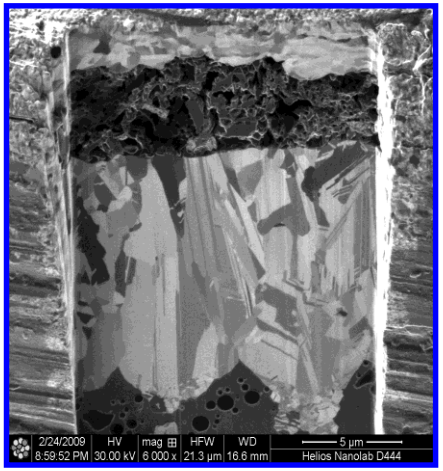
4. Polishing후 단면관찰



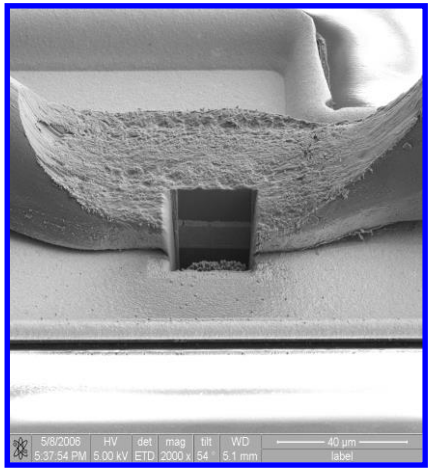
Device



Au/Ni/Polymer Ball

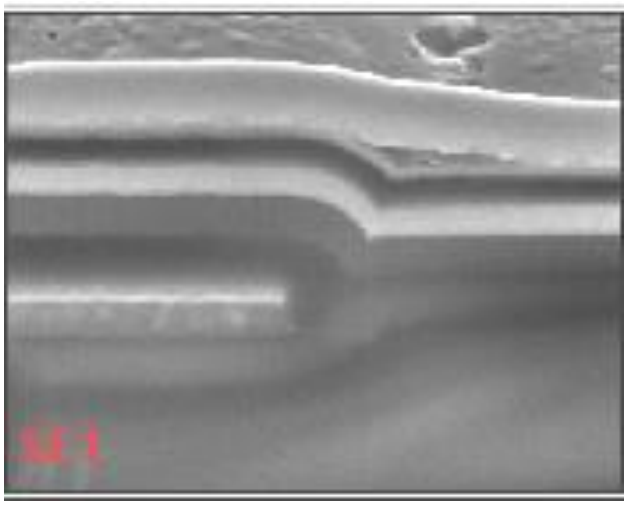


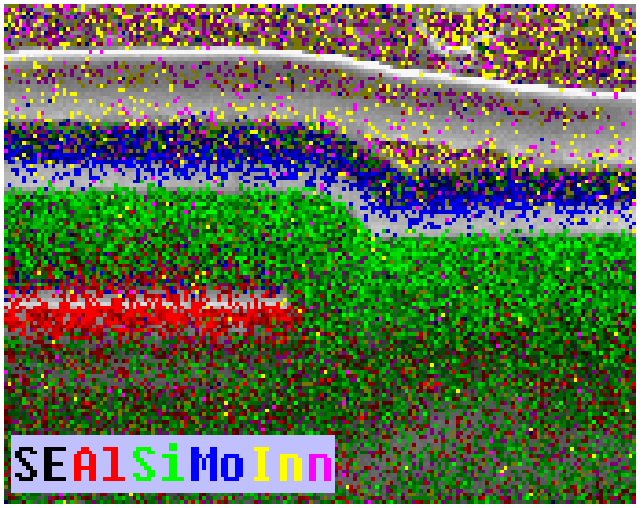
PCB 기판

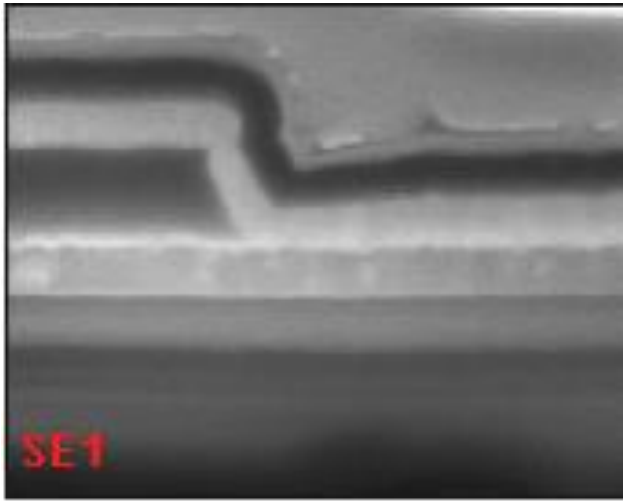


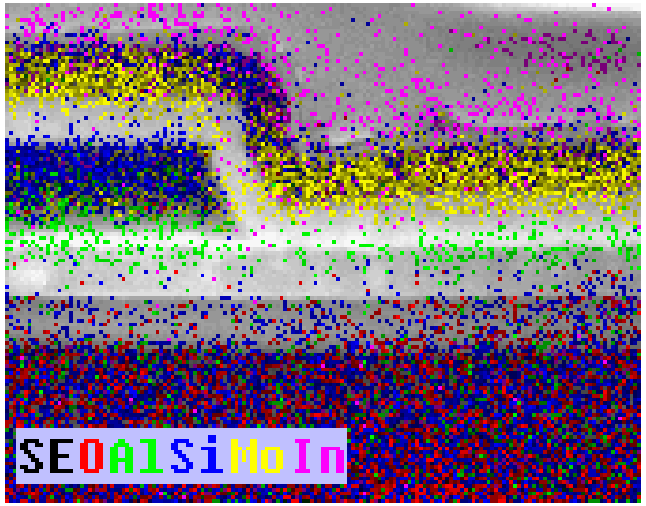
Wire Bonding

Failure Analysis with the Cross-sectioned Specimen



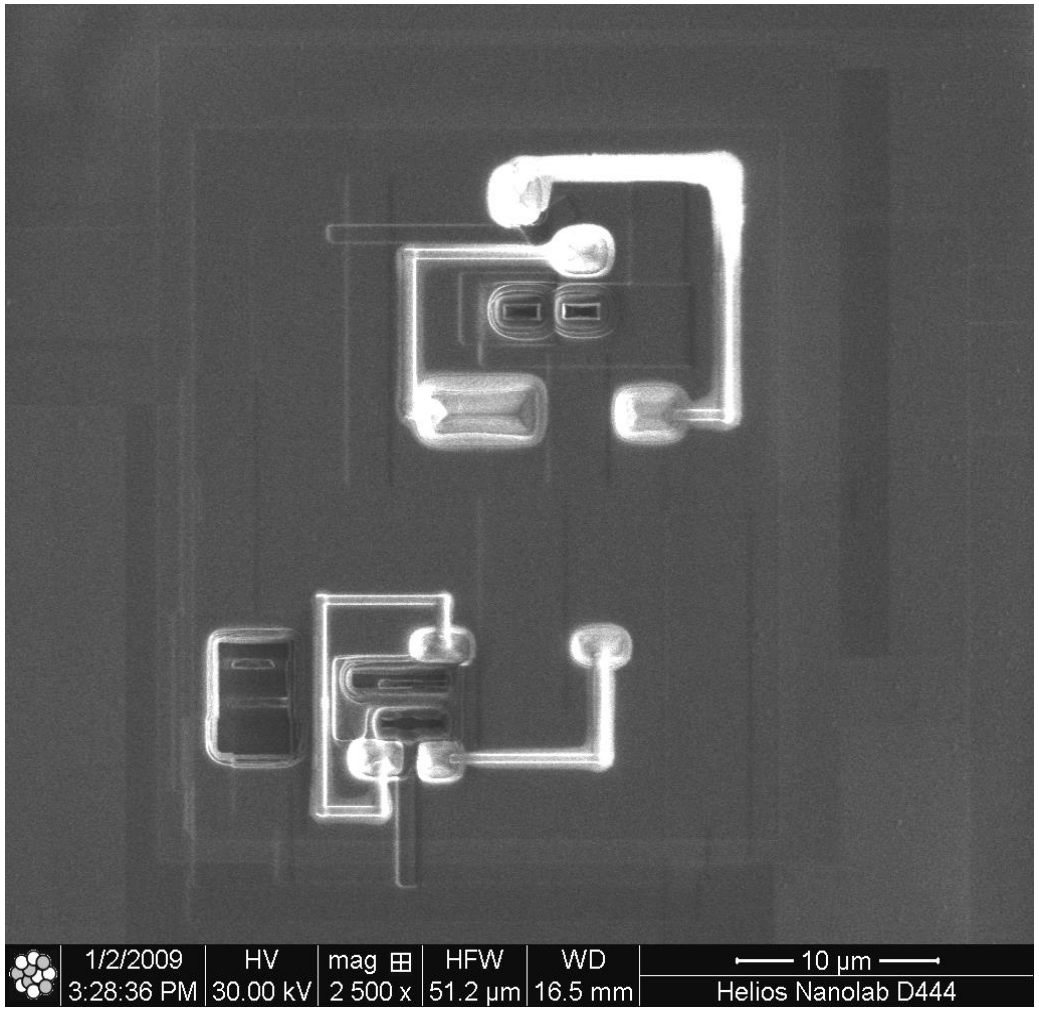




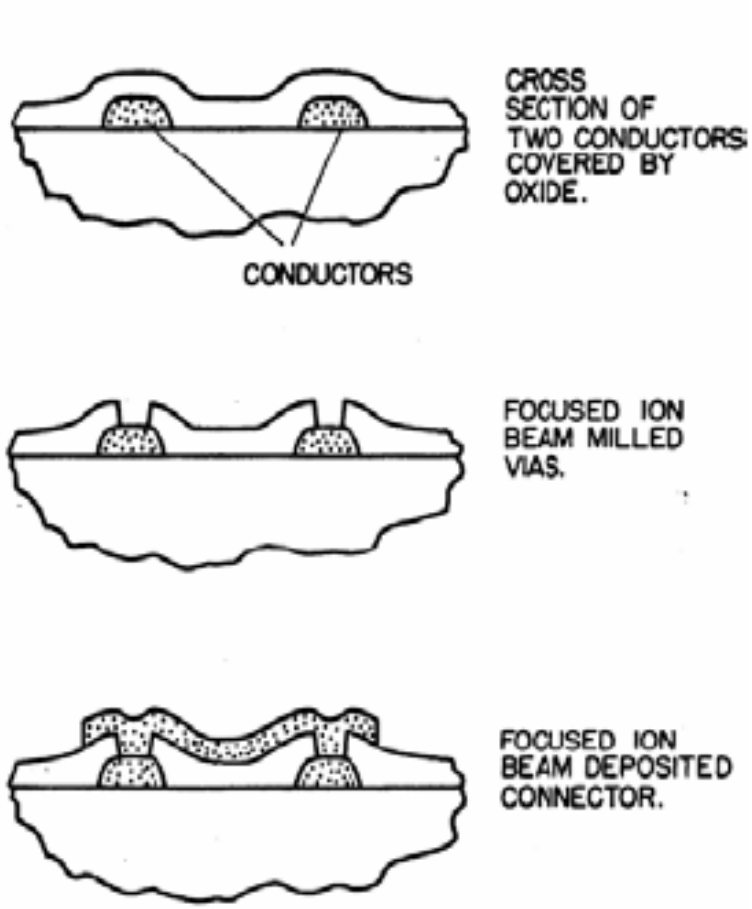


LCD 단면구조 분석 및 성분분석

Circuit Modification

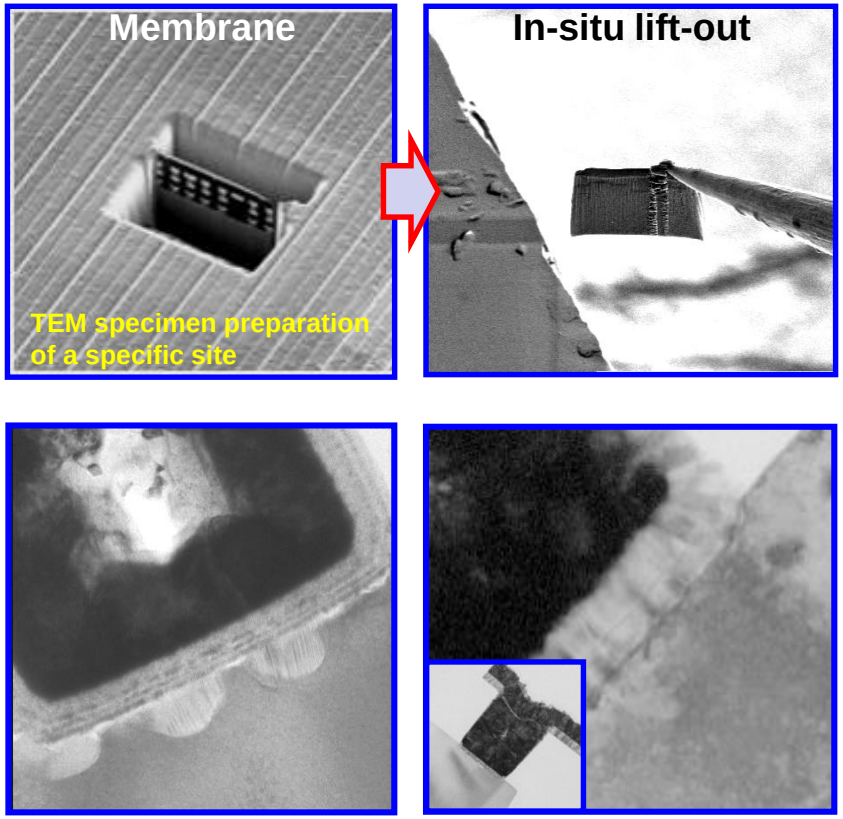


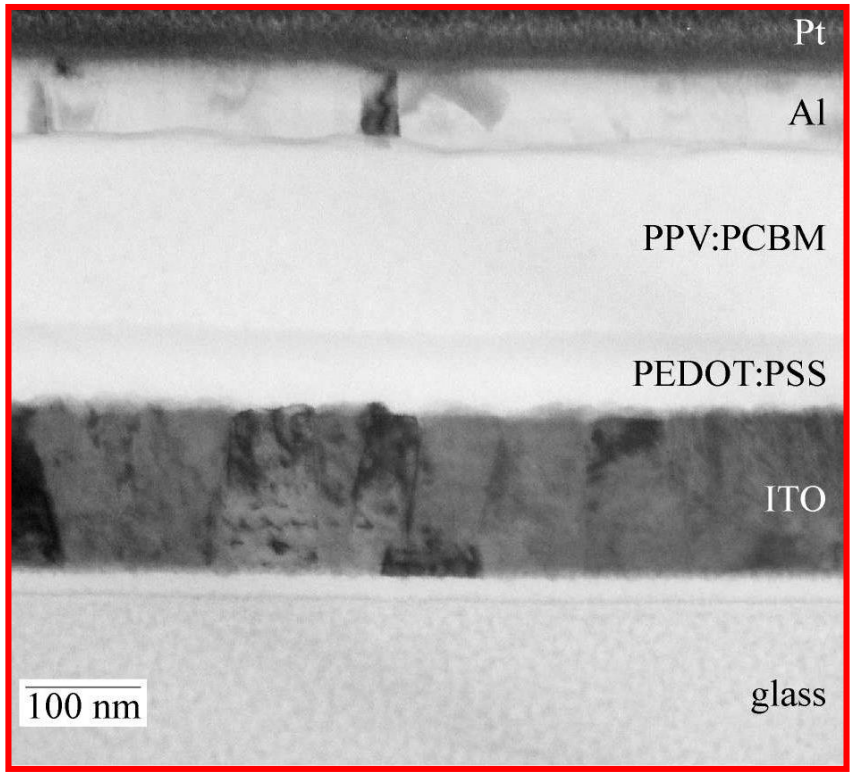
Metal3까지 공정된 제품으로
Top metal과 M2, M1 cutting 및 deposition



TEM Specimen Preparation

- 특정 위치에서의 **TEM** 시편 제작
- 이온 밀링법으로 가능하지 않은 **TEM** 시편 제작
  - 밀링 속도의 차이가 큰 물질로 구성된 다층시료: 금속/비금속 계면 등
  - 균일한 두께의 **TEM** 시편 제작이 필요한 경우: **Electron Holography** 등





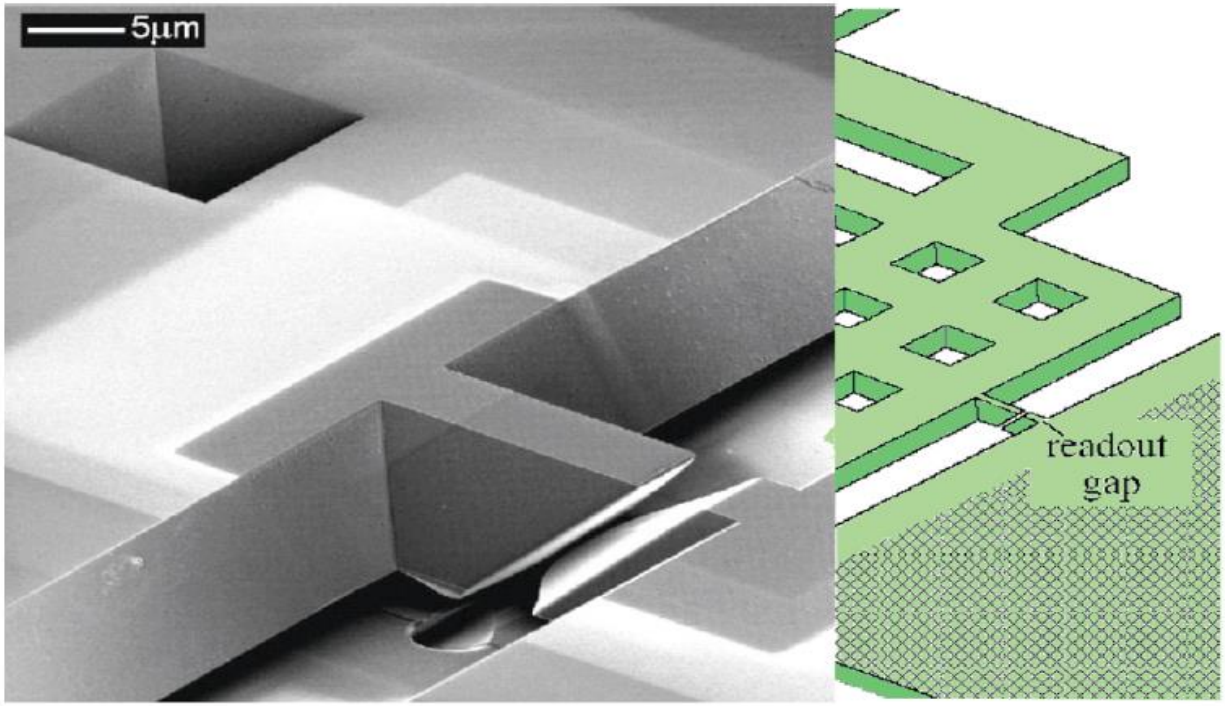
Sample with high milling rate difference

Micromachining

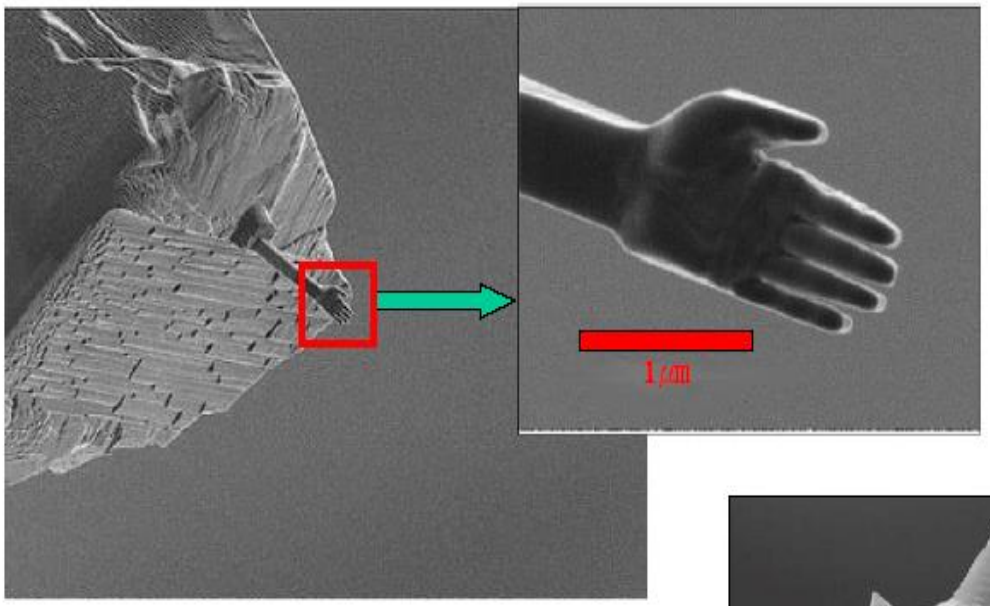
- direct-write technique
- high spatial resolution (≈10 nm) in combination with a five-axis motorized stage
- possibility to mill *and* deposit materials

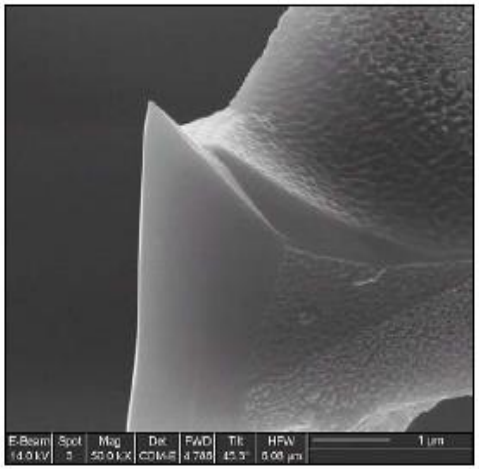
→ FIB is powerful tool for maskless micromachining

- subtractive (milling) and additive (deposition) machining are possible in the same machine



Nanofabrication





AFM Tip Application



Nano-Mechanics Application

Download: FEI Company web site

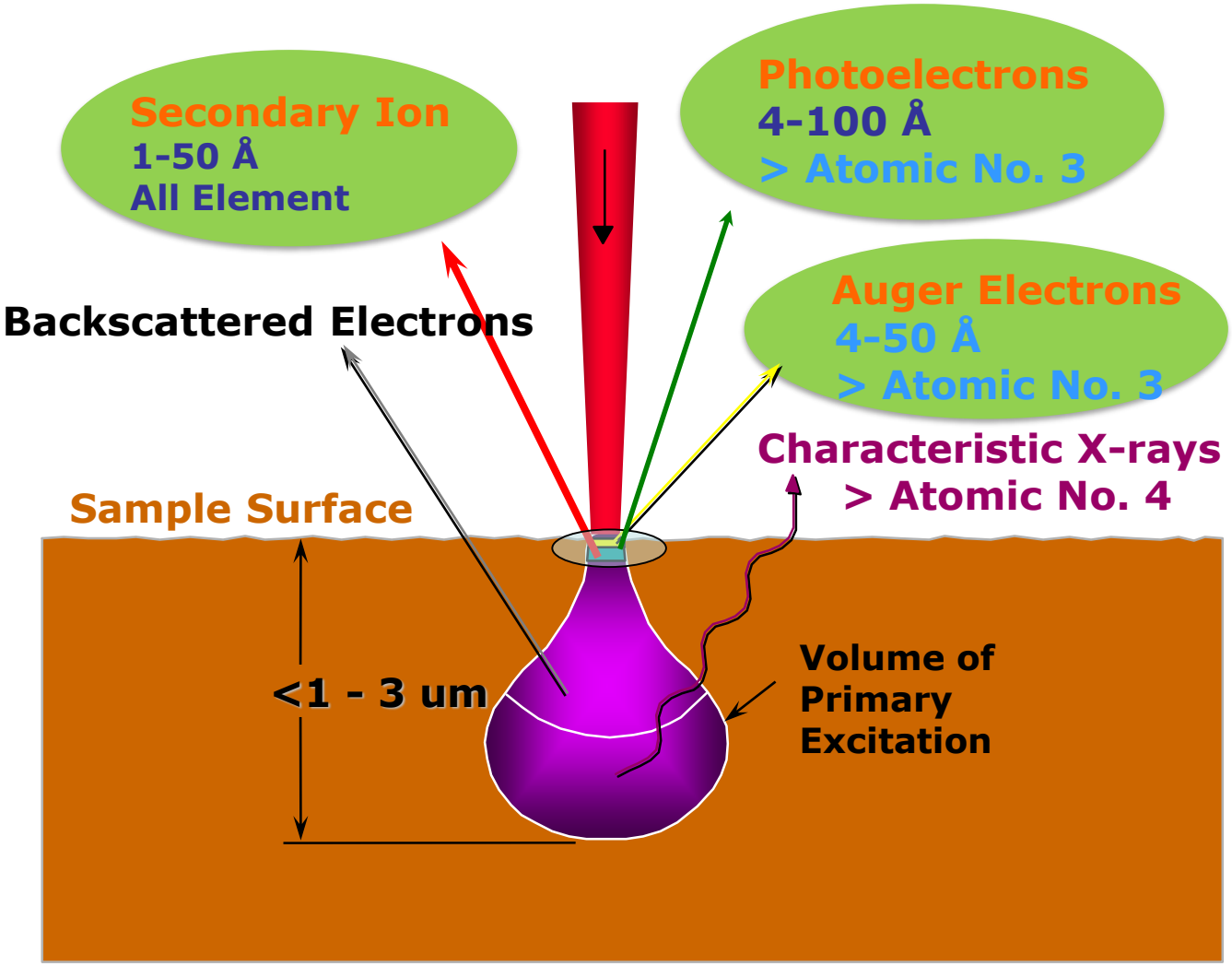
나노분석의 기초 및 응용

- 나노분석의 기초

- 구조분석(SEM, TEM, FIB)

- **표면분석(XPS, AES, SIMS)**

Interaction Volume : Electron & Ion & X-ray Beams



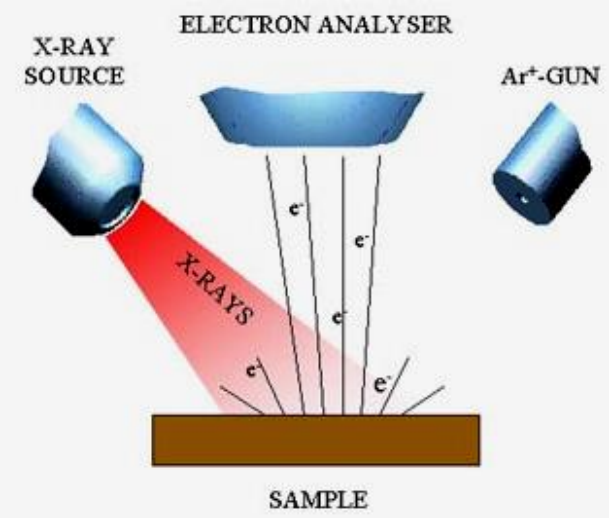
Surface Analysis Techniques

- Electron Spectroscopy - Analysis Signal : Electrons
  - X-ray Photoelectron Spectroscopy - XPS
    (also known as ESCA, Electron Spectroscopy for Chemical Analysis)
  - Auger Electron Spectroscopy - AES
- Ion Spectroscopy - Analysis Signal : Ions
  - Ion Scattering Spectroscopy - ISS
  - Secondary Ion Mass Spectrometry - SIMS

X선光電子分光 분석

X-Ray Photoelectron Spectroscopy (XPS)

X-Ray Photoelectron Spectroscopy (XPS)

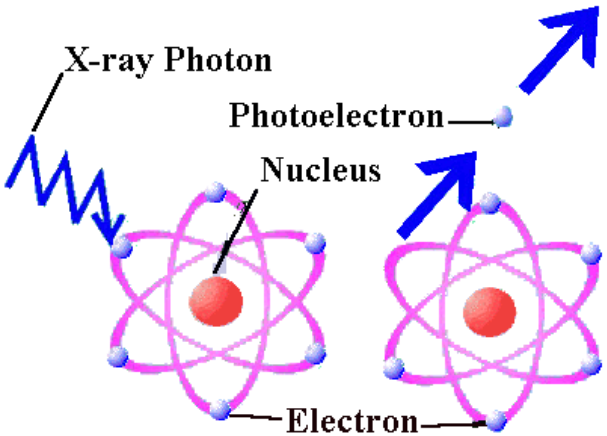


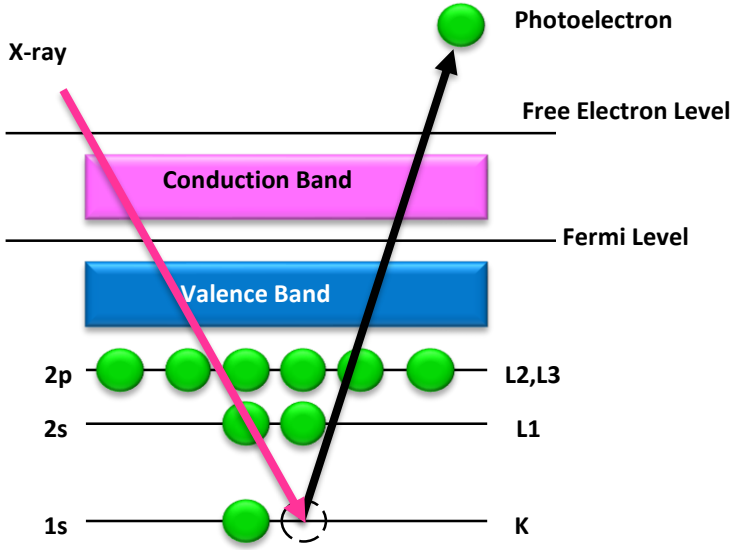
XPS 측정 개략도

- 결합에너지 측정, 화학적 정보 제공
- 50 Å의 분석 깊이를 갖는 표면분석 기술
- 정량적
- Mapping과 깊이 분포로부터 공간적 분포 제공

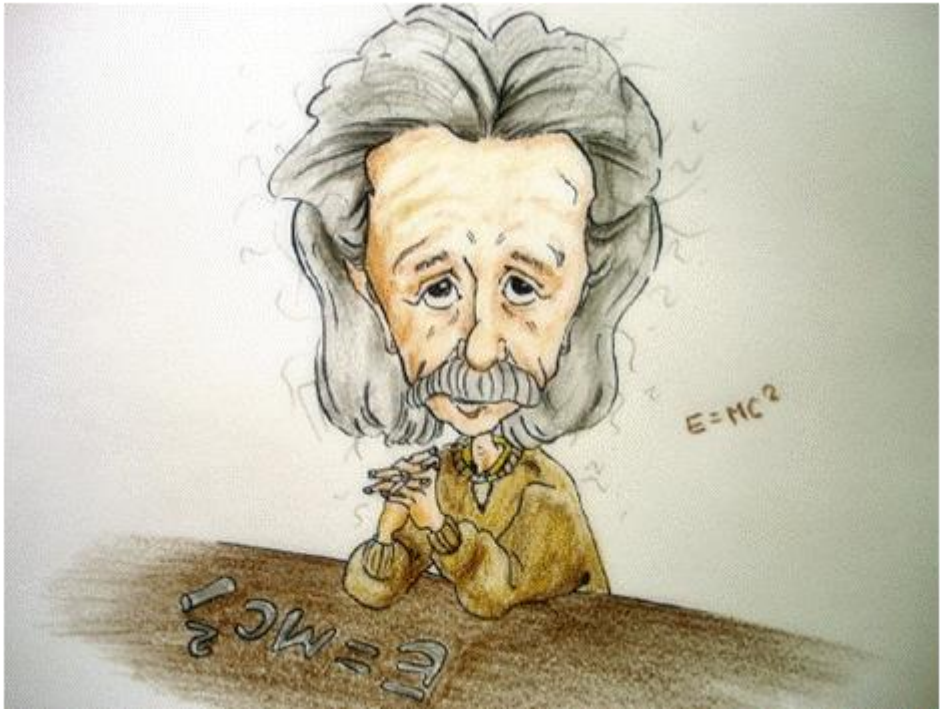
$$E_K = h\upsilon - E_b - \phi_S$$

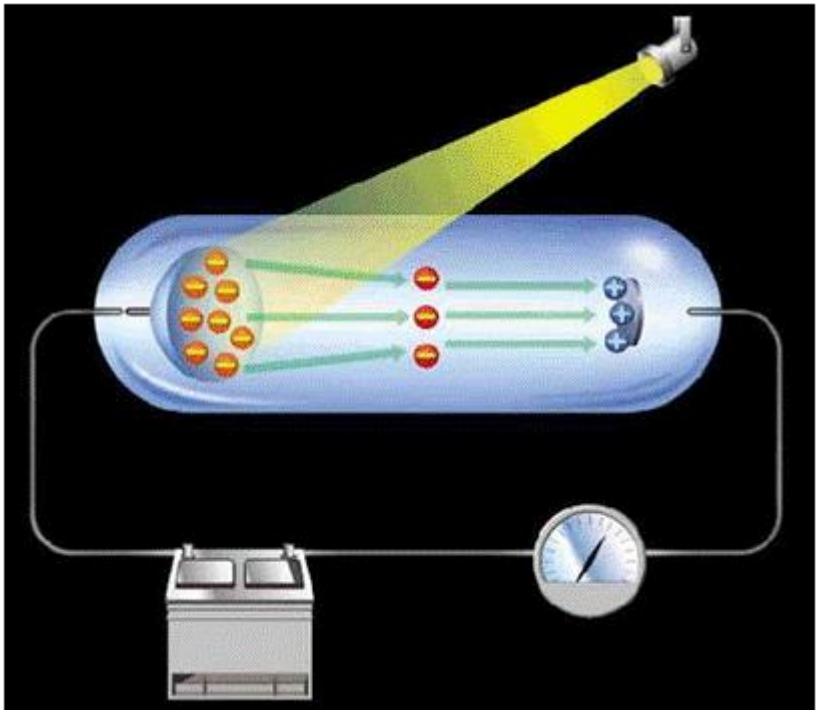
E_K : 방출된 광전자의 운동에너지
hυ : X-ray photon의 특성에너지
E_b : 내각준위의 결합에너지
φ_S : 검출기의 일함수





아인슈타인과 노벨상





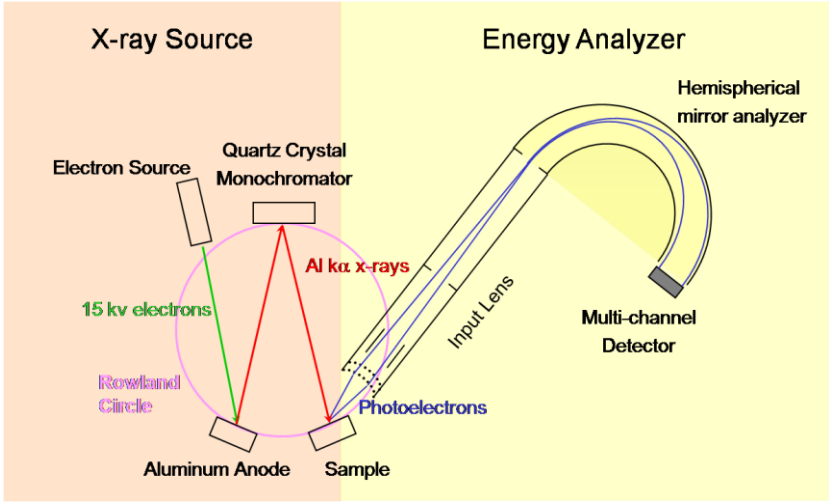
- 1910년 오스트발트에 의해서 특수상대성이론으로 노벨상 후보로 처음 지명
- 제1차세계대전을 거치면서 계속 노벨상 후보로 추천되는 빈도가 계속 증가
- 1920년 광전효과에 대한 업적으로는 처음으로 노벨상 후보 등록
- 1921년 노벨상 위원회가 아인슈타인의 업적을 인정, 이월된 1921년의 노벨물리학상을 1922년에 수상
- 수상 근거 : 그의 주된 업적인 상대성이론이 아니라, '이론물리학 특히 광전효과의 해석에 대한 기여'로 한정해서 발표

X-Ray Photoelectron Spectroscopy (XPS)

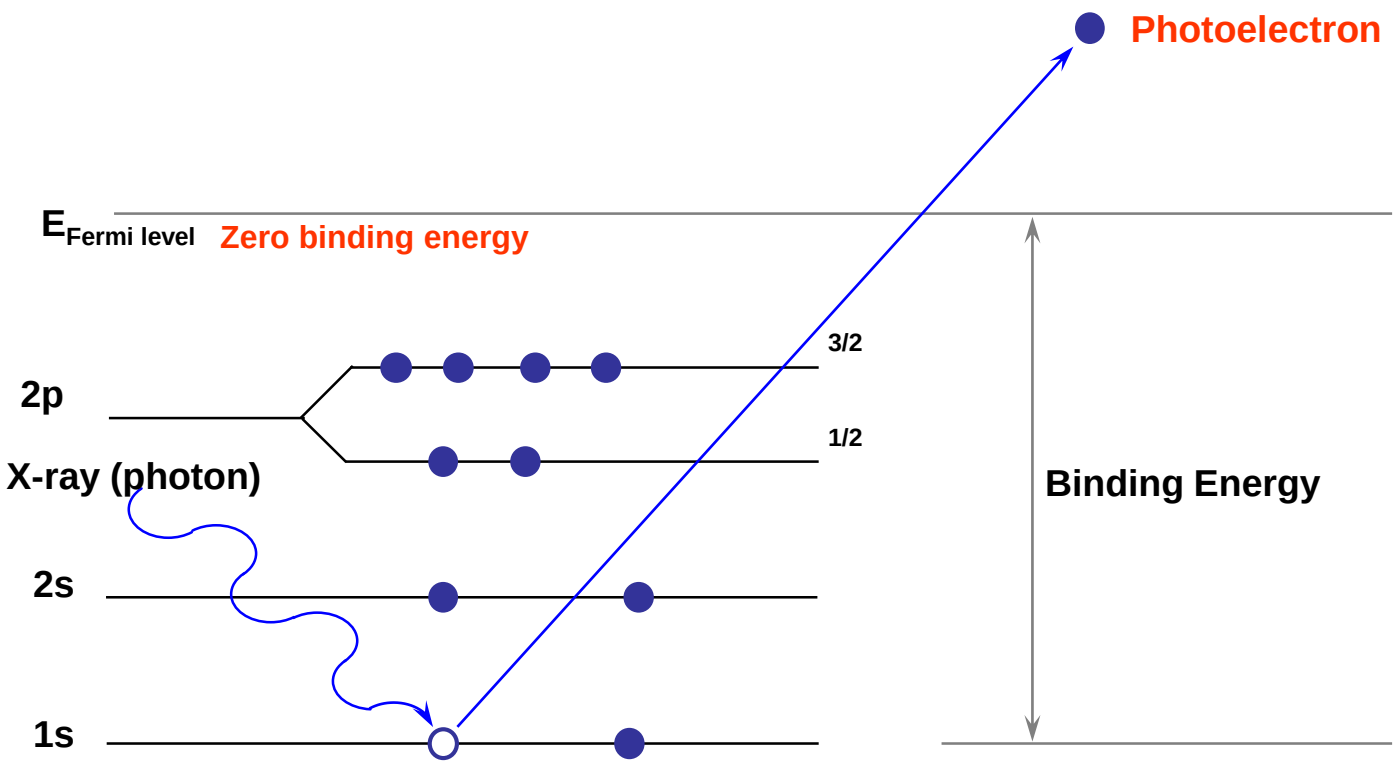
- XPS (ESCA) is an analytical technique that provides compositional information from the top few atom layers of a sample by probing it with a mono-energetic X-ray beam.

  - Detect : all elements above He
  - Detection limits : 0.1 - 1.0 at%
  - Analysis depth : 5 - 75 Å
  - Spatial resolution : ≧ 10 μm probe size





Photoelectric Effect



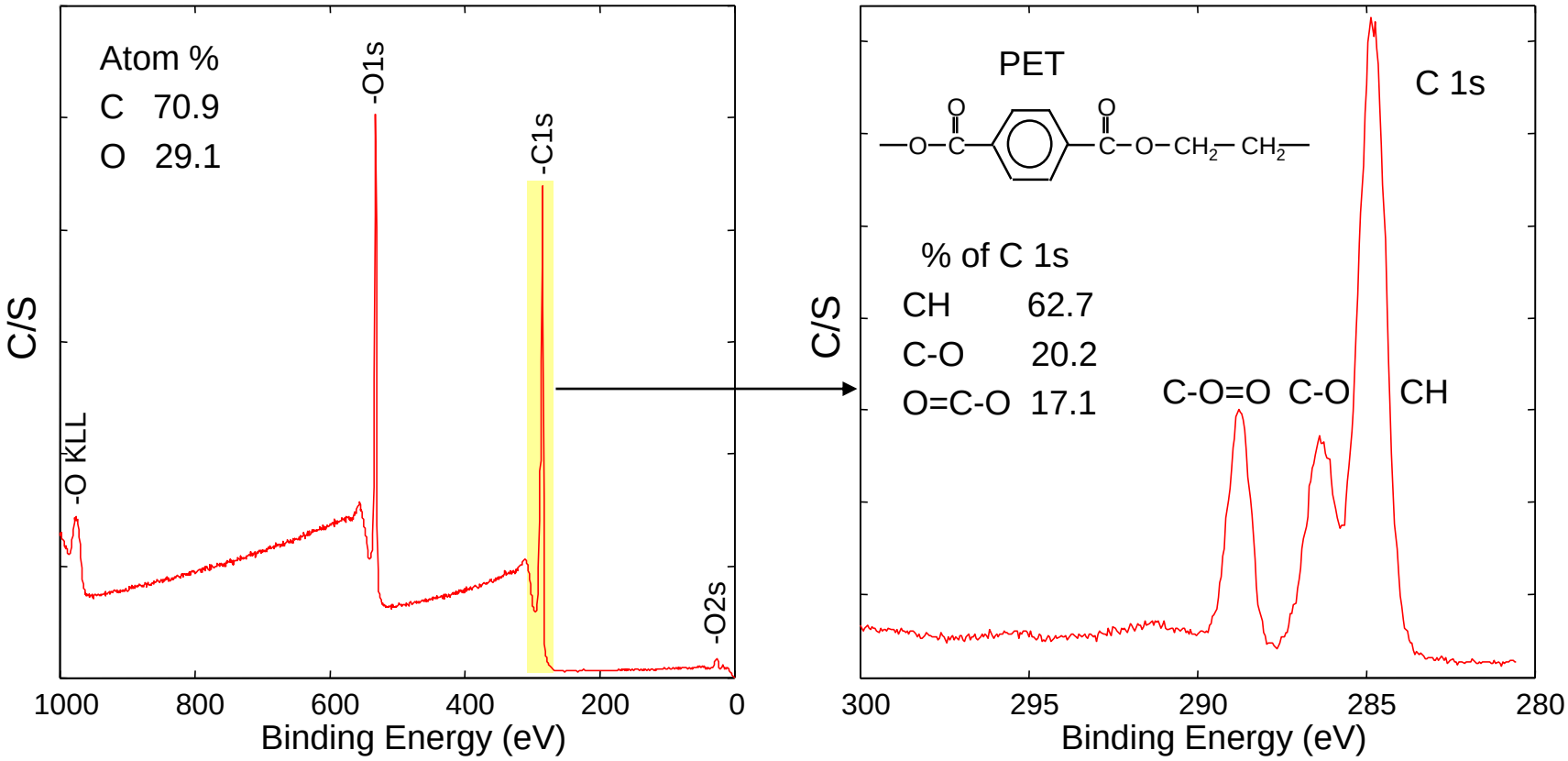
Kinetic Energy of P. E. = X-ray Energy – Binding Energy – W. F. of Spectrometer

$$E_K = h\upsilon - E_b - \phi_S$$

Binding Energy = X-ray Energy – Kinetic Energy of P. E. – W. F. of Spectrometer

Typical XPS Spectrum

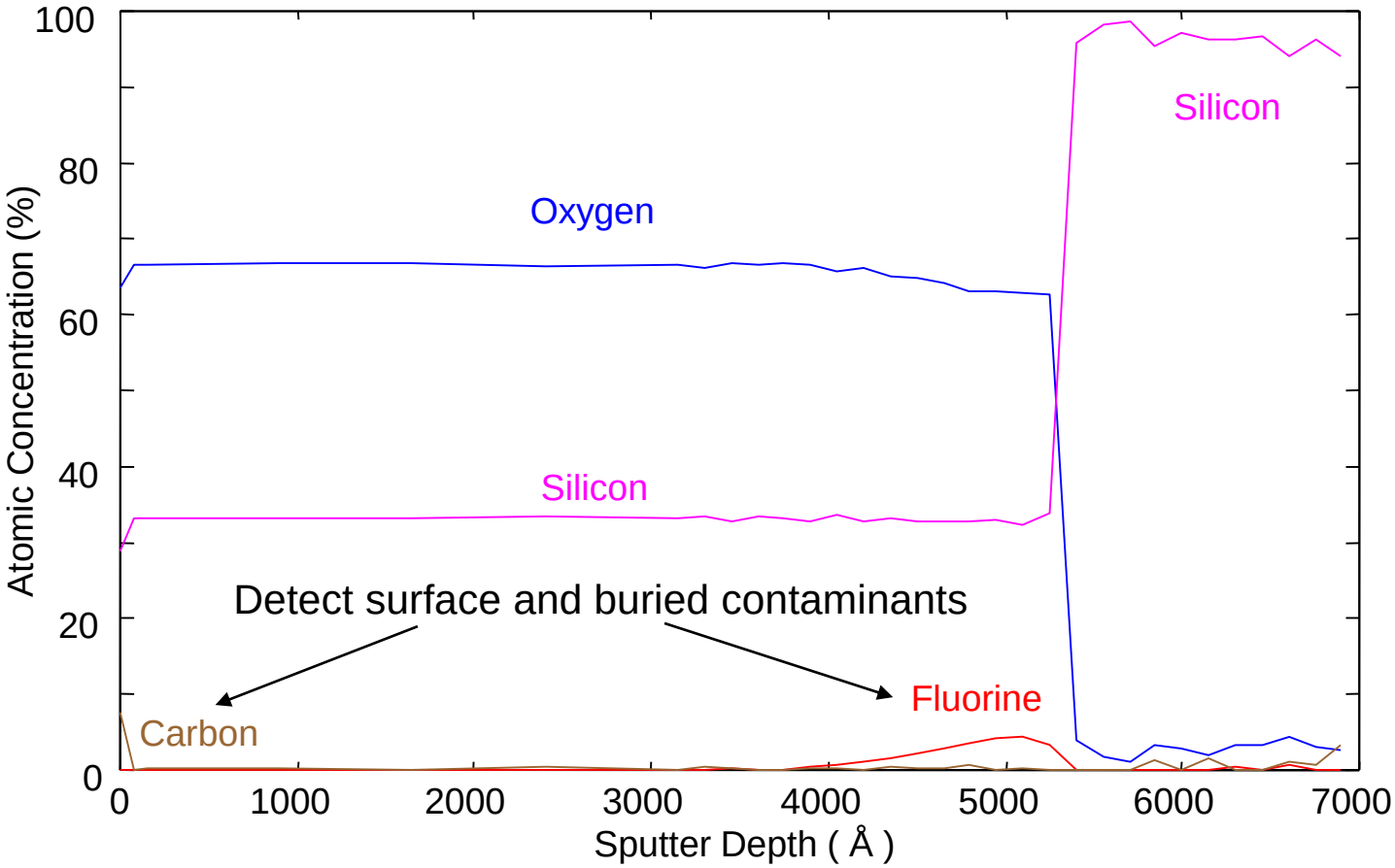
Poly(ethylene terephthalate)



XPS survey spectra provide quantitative elemental information

High-resolution XPS spectra provide quantitative chemical state information

XPS Sputter Depth Profiling



The analysis of thin film structures to verify composition or search for impurities at interfaces

XPS Characteristics

Advantages

- Determination of valence states and/or bonding environment of atoms near the surface
- Convenient and quick elemental analysis of surfaces
- Identifies organic functional groups in polymer or other organic surfaces
- Characterization of very thin surface layered structures
- Depth profiling or both conducting and insulating materials

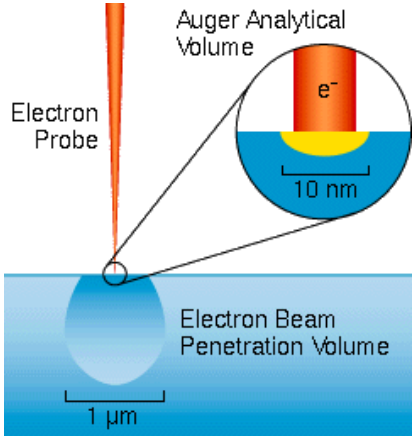
Disadvantages

- Spatial resolution is low
- Cannot detect hydrogen or helium
- Requires that samples be small and compatible with high vacuum
- Elemental quantification by XPS depends on instrumental, chemical and sample related factors

오제이電子分光 분석

Auger Electron Spectroscopy (AES)

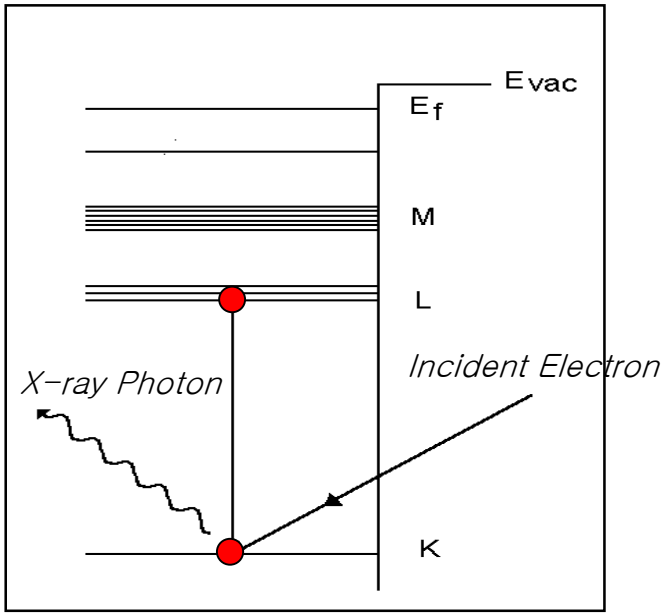
Auger Electron Spectroscopy (AES)



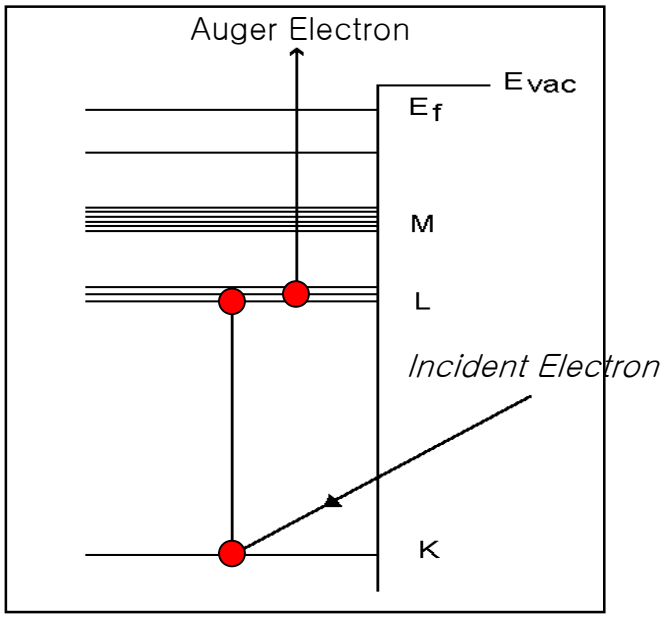
Auger 전자와 X-ray 방출 비교

Relaxation of excited state by the transition of a higher lying electron to fill the core hole and the simultaneous emission of an Auger electron or X-ray photon

X-ray Fluorescence



Auger Electron Emission

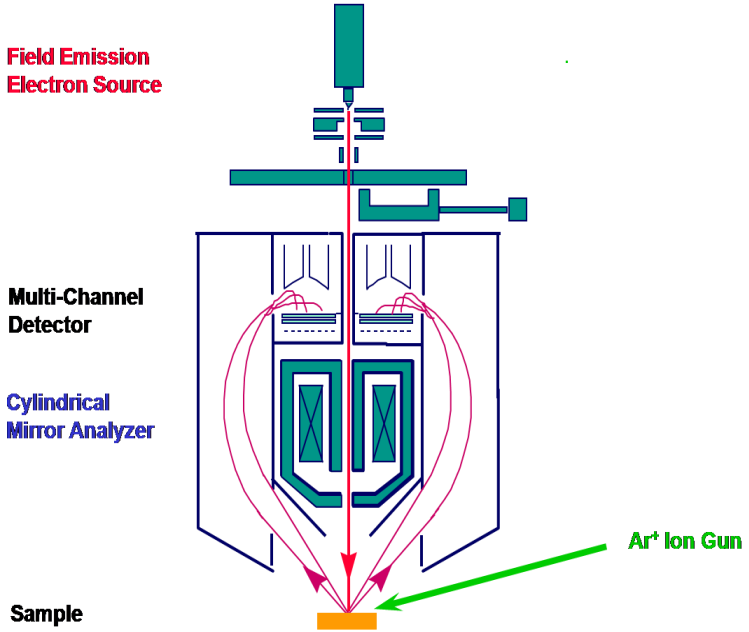


Auger Electron Spectroscopy (AES)

- AES is an analytical technique that provides compositional information on the top few monolayers of material.

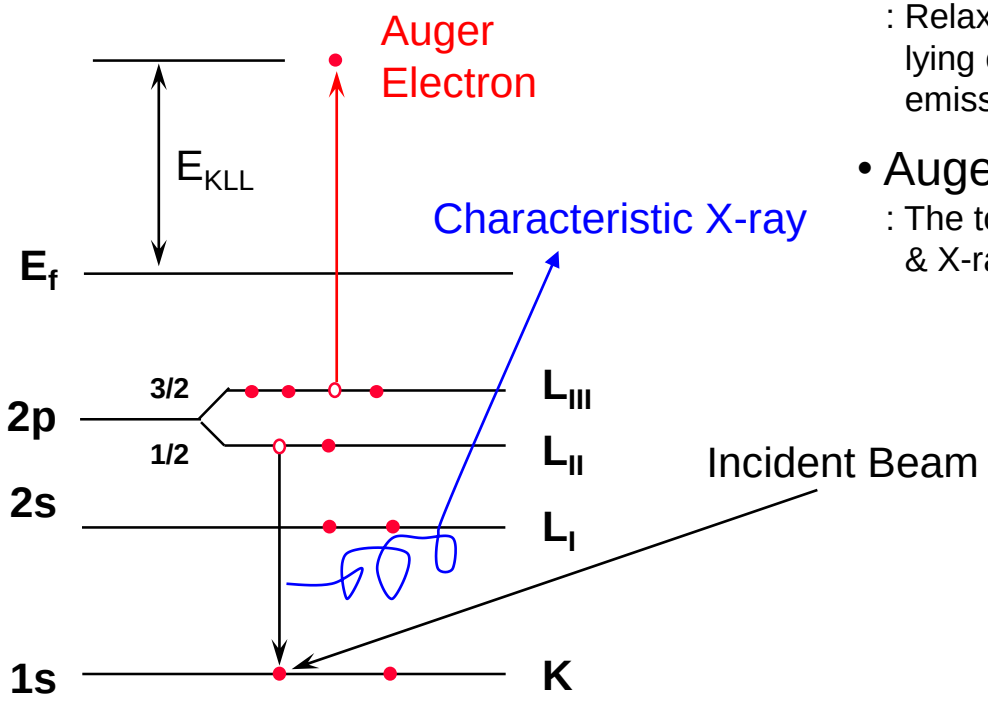
- Detect all elements above He
- Detection limits : 0.1 - 1.0 at%
- Surface sensitive : 4 - 50 Å
- Spatial resolution : ≧ 10 nm probe size





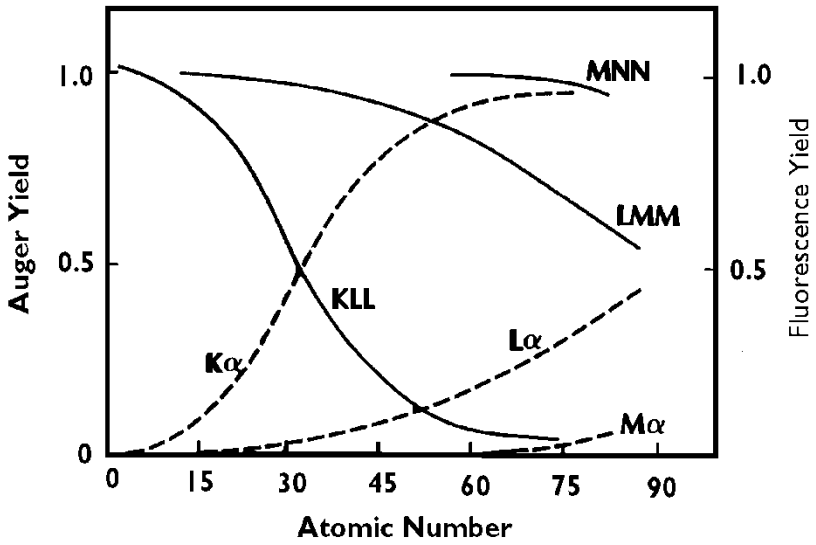
Auger Electron Emission Process

- Auger electron emission results from a 2 electron Coulombic readjustment to an initial core hole.



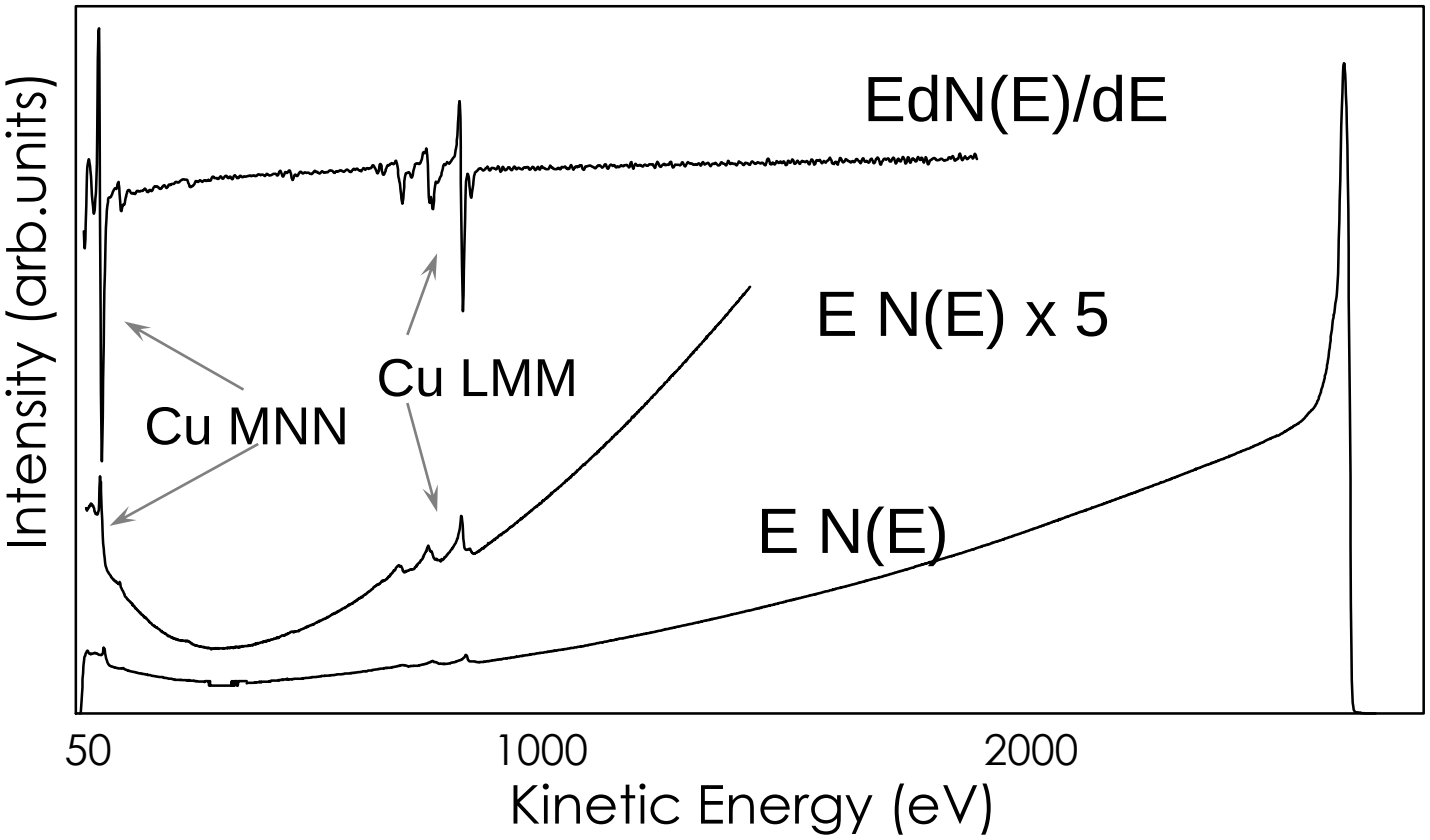
$$E_{KLL} = E_K - E_{LII} - E_{LIII}$$

- Auger & X-ray Emission Comparison
  : Relaxation of excited state by the transition of a higher lying electron to fill the core hole and the simultaneous emission of an Auger electron or X-ray photon.
- Auger and Fluorescence
  : The total Auger probability is given by the Auger emission & X-ray fluorescence curves such that $\rho_A + \rho_X = 1$.

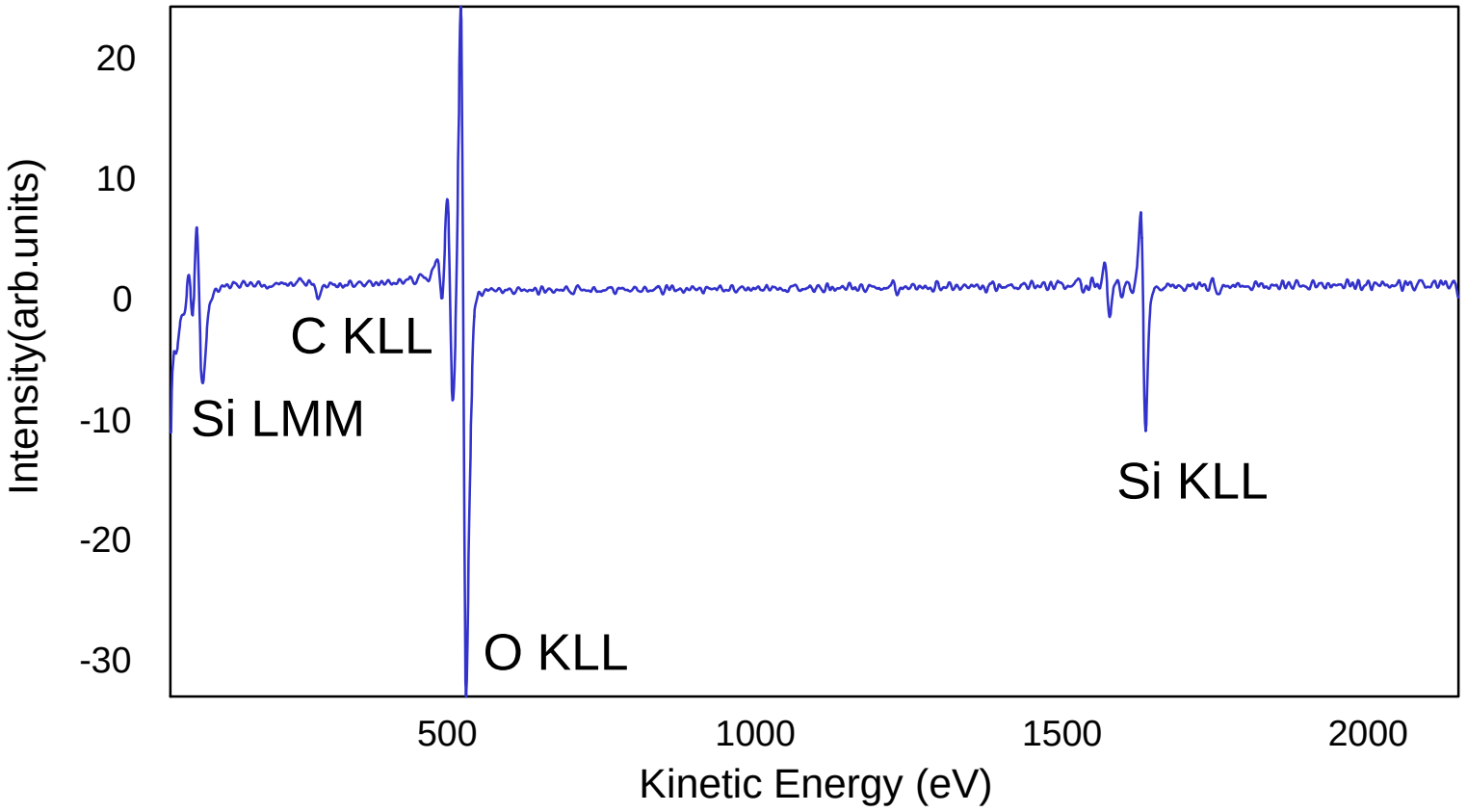


Typical AES Spectra

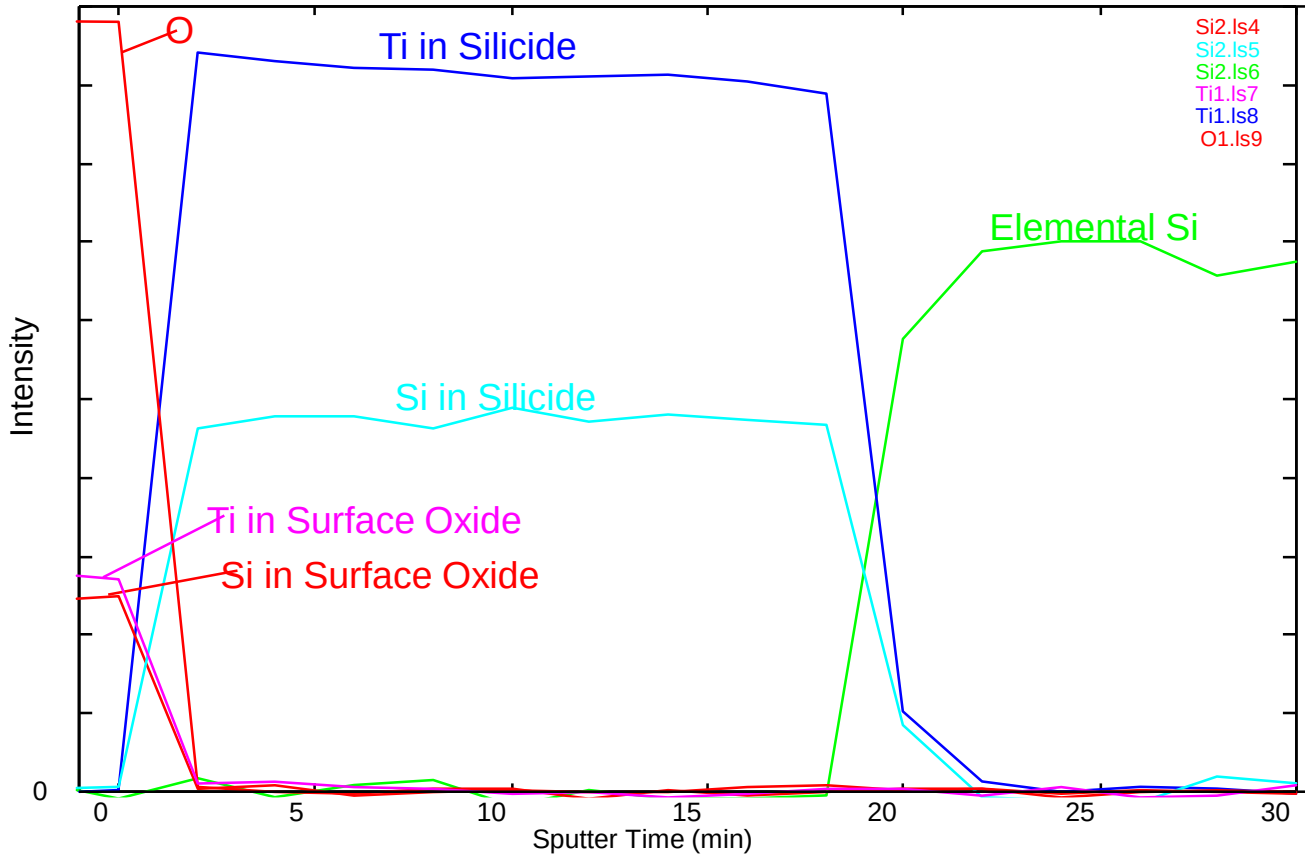
- Direct and differentiated Auger spectra



Typical AES Spectra : SiO_2 Survey



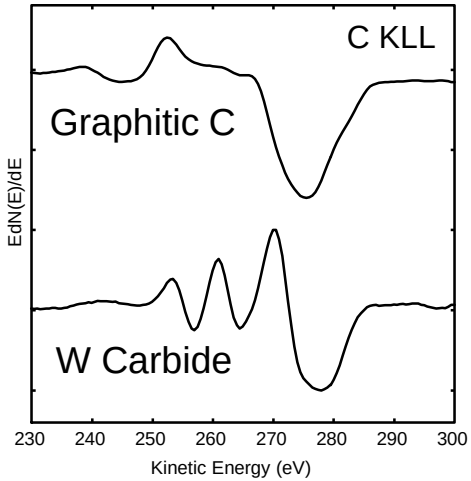
Typical AES Spectra : $TiSi_2$/Si Depth Profile



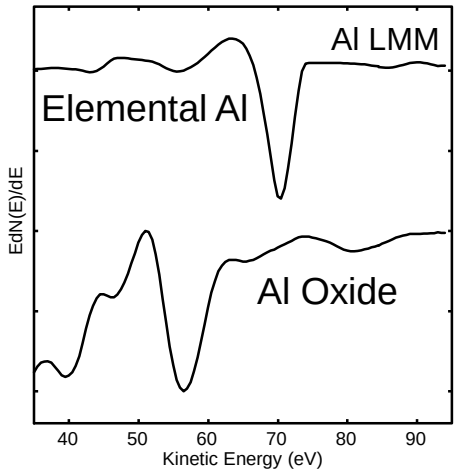
Chemical Information

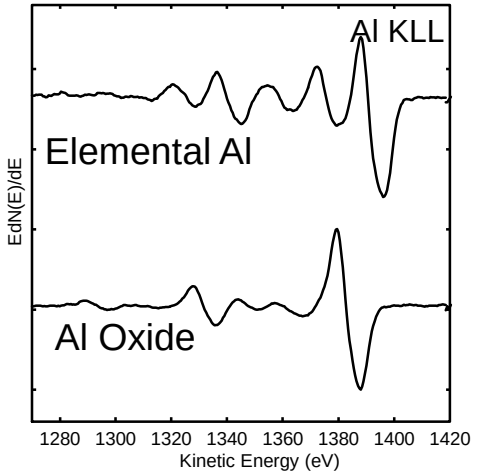
- Variations in an element's chemical state are reflected in the Auger spectrum

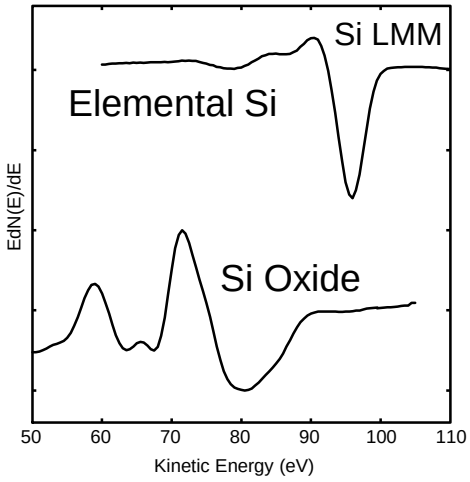
Energy position
Line shape
Loss Structure

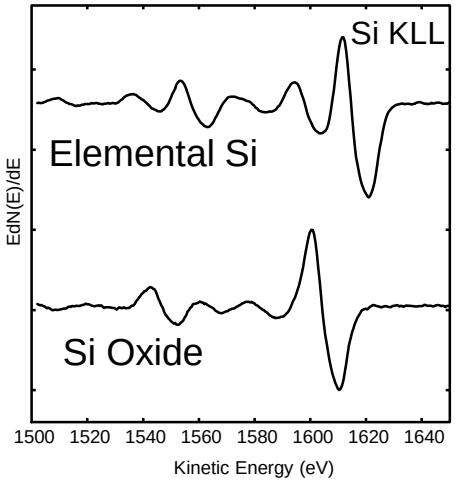


C KLL
Core-Valence-Valence



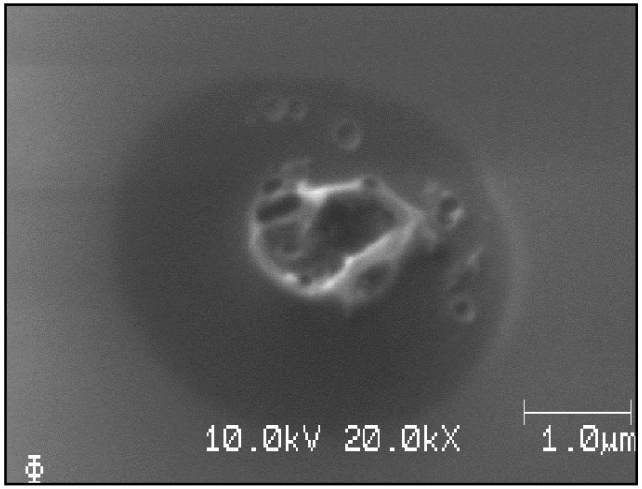


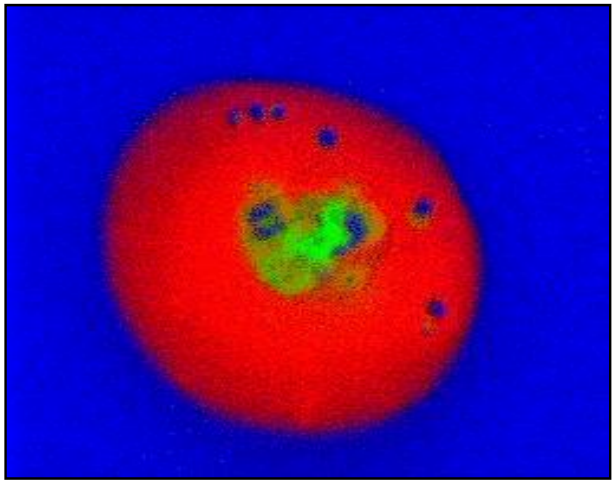




AES Line Scan and Scanning Auger Microscopy

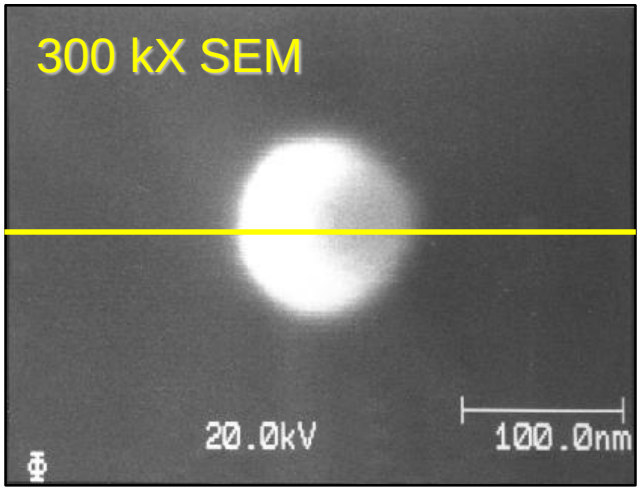
- Analysis of a Complex Defect after Poly-Si Etch

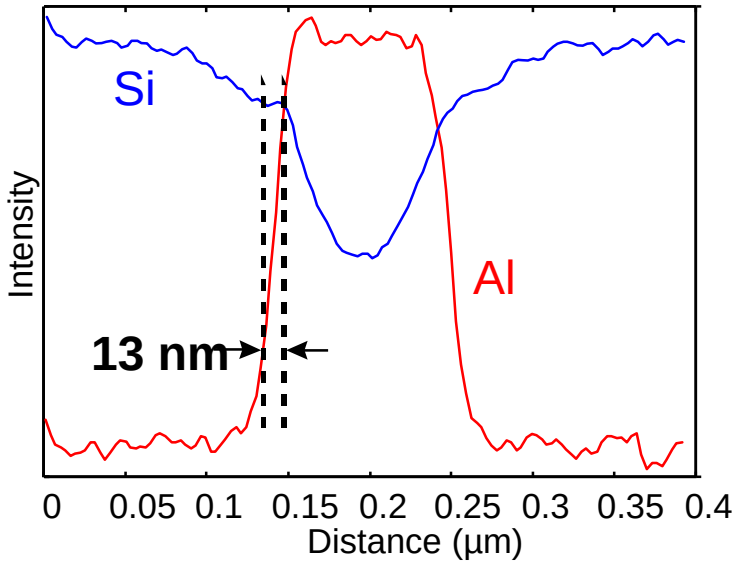




Red=C
Blue=Si
Green=S

- Analysis of 0.1 mm Al Particle on Si





AES Characteristics

Advantages

- Spatial resolution is high
- Analysis is relatively rapid
- It is sensitive to light elements (except H and He)
- Chemical information is available in some cases
- It provides reliable semiquantitative analysis

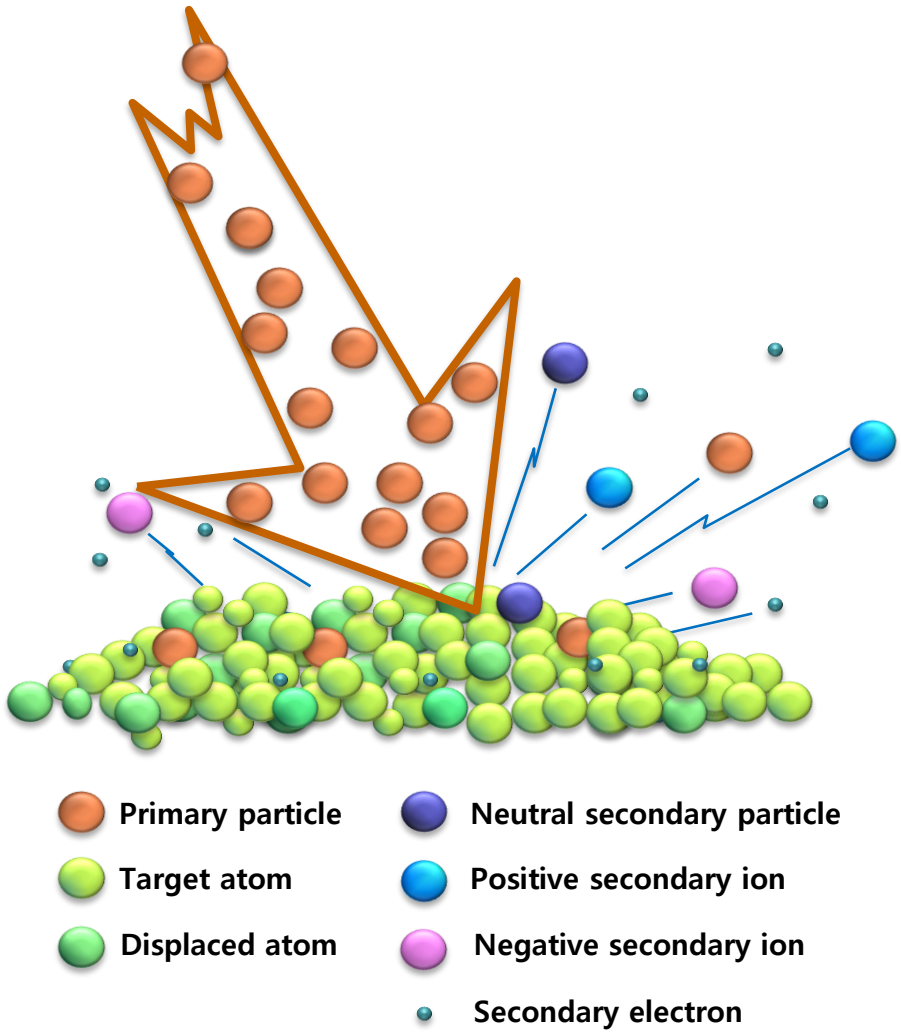
Disadvantages

- UHV compatible sample (Cannot use volatile sample)
- Insulators are difficult to study due to surface charging (organic ≥ 5 nm, oxide ≥ 400 nm)
- Sensitivity is modest (0.1 to 1.0 at%)
- Elemental quantification by AES depends on instrumental, chemical and sample related factors

이차이온質量 분석

Secondary Ion Mass Spectrometry (SIMS)

이차이온질량분석(SIMS) 기술의 개요



Secondary Ion Mass Spectrometry

(이차이온 질량 분석기, SIMS)

- 고체 표면에 에너지를 갖는 일차이온 빔을 충돌시키면 고체표면으로부터 이차입자들(Ion, Electron, Photon, Neutron 등)이 튀어나오는데, 이때 **sputter**된 입자 중 전하를 갖는 이차이온이나 분자 이온의 질량과 검출량을 측정하는 방법
- 고체표면에 분포하고 있는 물질의 성분 및 깊이 방향으로 각 성분에 대한 농도분포를 분석

- **Best Detection Limit, ppb 수준까지 측정**
- **Si 내에 이온주입된 B 원소를 $10^{17}/cm^3$ (ppm) 정도까지 검출 가능**
- **H 원소의 농도도 측정 가능**

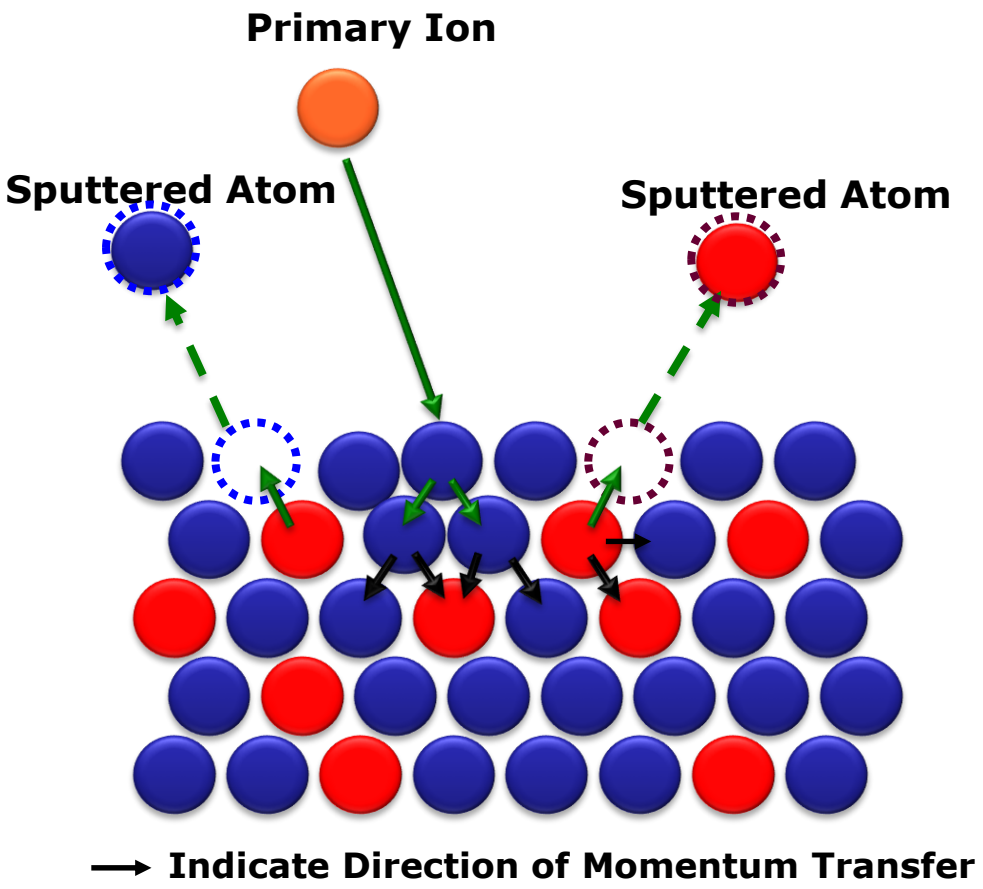
- Sputtering Yield (Y_0)

$$Y_0 \propto \frac{E_{impact}}{U_s}$$

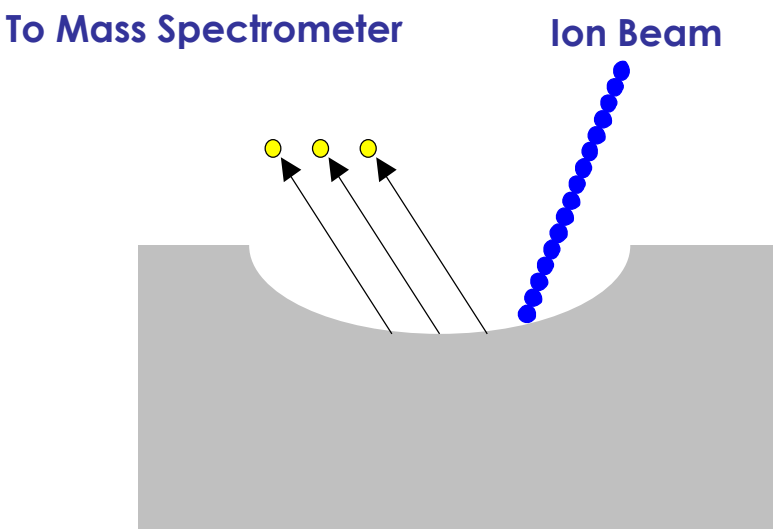
U_s : binding energy of surface atoms

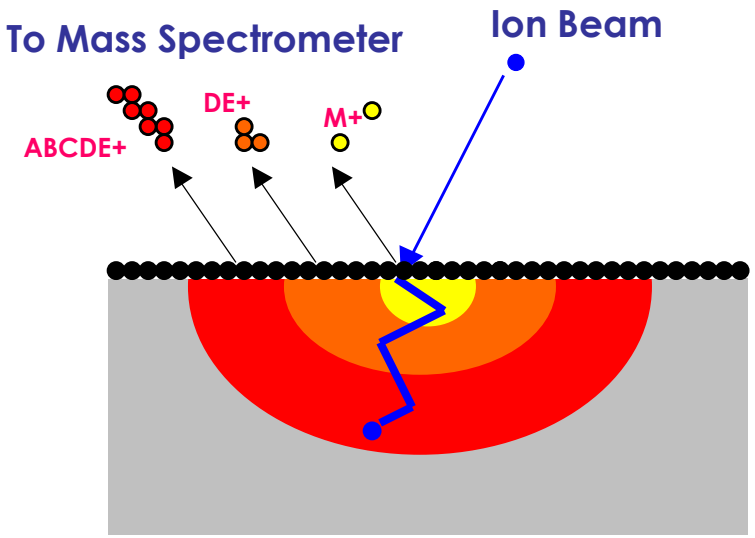
E_{impact} : primary ions impact energy

✓ As a consequence of the collision, Atoms have a velocity component greater than 90° will be ejected from the surface.



Dynamic and Static Modes





Dynamic SIMS

- Material removal
- Elemental analysis
- Profiling

Static SIMS

- Ultra surface analysis
- Elemental or molecular analysis
- Analysis complete before significant fraction of molecules destroyed

Spectrometer | **Magnetic Sector** | **Time of Flight**

- Ion Yield(Y_i) $$Y^{\pm} = Y \bullet \alpha^{\pm}$$

Y : sputtering yield
α : Ionization efficiency

High sensitivity of SIMS instruments requires an optimization of the Useful Yield = Y_i x T

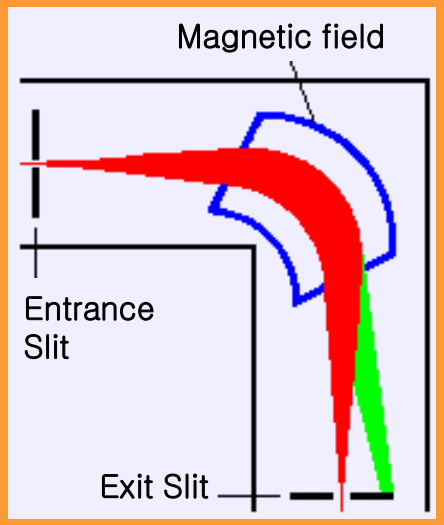
◆ Ionization yield Y_i

- ✓ Use of reactive primary ions
- ✓ O_2^+ or O^- enhances ionization of electropositive species (metals, alkalis, …)
- ✓ Cs^+ enhances ionization of electronegative species (C, O, S, Cl,…)

Basic Principle of SIMS

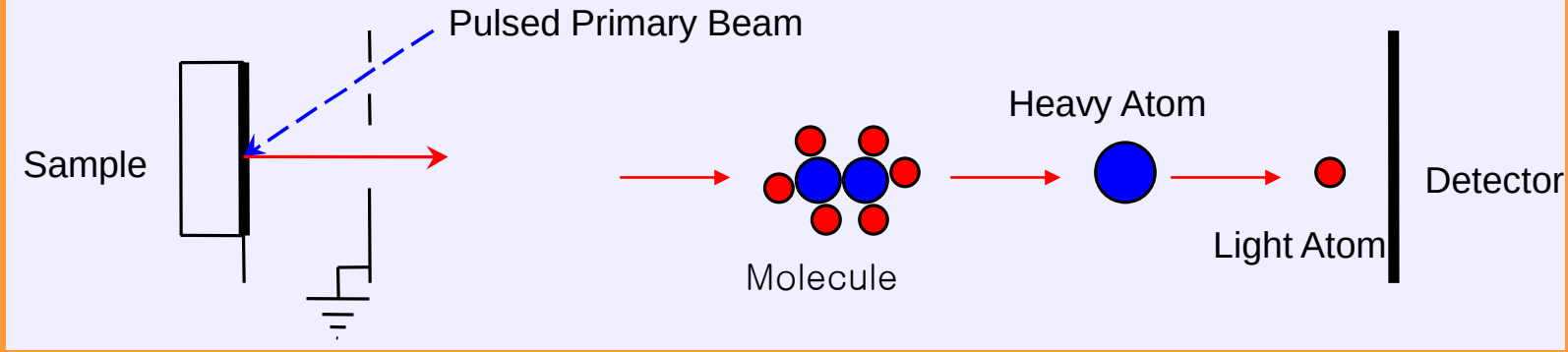
- **Magnetic Sector type**

$$\frac{mv^2}{r} = \frac{eHv}{c}$$ (원심력=구심력)



Magnetic sector type Analyzer

- TOF(Time-of-Flight)



Time-of-Flight type analyzer

$$eV = \frac{1}{2}mv^2 \qquad v = \frac{l}{t} \qquad m = \left(\frac{2eV}{l^2}\right) \bullet t^2$$

SIMS Instrumentation and Capabilities

	Strengths	Weaknesses
Magnetic Sector (Dynamic-SIMS)	• High transmission (~40%) • High mass resolution (M/ΔM = ~10,000) • **Best detection limit**	• Primary beam incidence angle is dependent on primary beam energy (21°~65°)
Time of Flight (Static-SIMS)	• High transmission • High mass resolution (M/ΔM > 10,000) • Excellent lateral resolution • Parallel mass detection • **Organic molecule mass analysis**	• Quantification limited • Depth profiling limited

Detection Limits @ M-SIMS

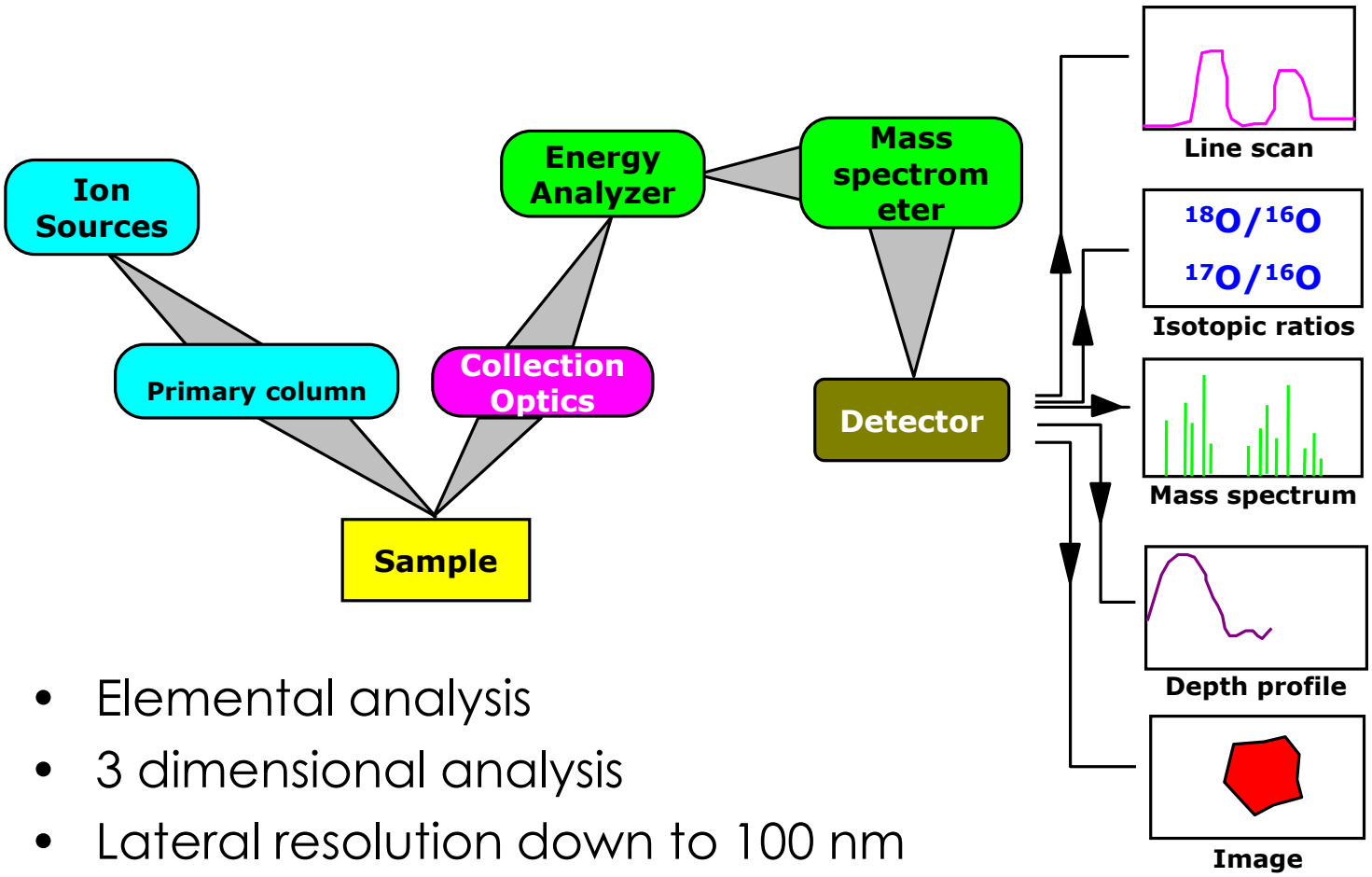
- ✓ The detection limit of an element depends on :
  - count rate limited
  - residual atmosphere (for H, C, O and N)
  - mass interference
  - contamination of the analysis chamber

In GaN under Normal Depth Profiling Condition

O_2/SIMS Positive Secondary Ion Detection		Cs/SIMS Negative Secondary Ion Detection		Cs/SIMS Positive Secondary Ion Detection (CsM+)	
Element	DL (atoms/cm³)	Element	DL (atoms/cm³)	Element	DL (atoms/cm³)
Be	1E+14	H*	8E+16 - 2E+17	Mg	5E+15
Li	1E+14	C*	5E+15 - 2E+16	Zn	1E+16
B	1E+15	O*	1E+16 - 3E+16		
Na	5E+14	Si	3E+15		
Mg	5E+14	As	5E+15		
Al	1E+15				

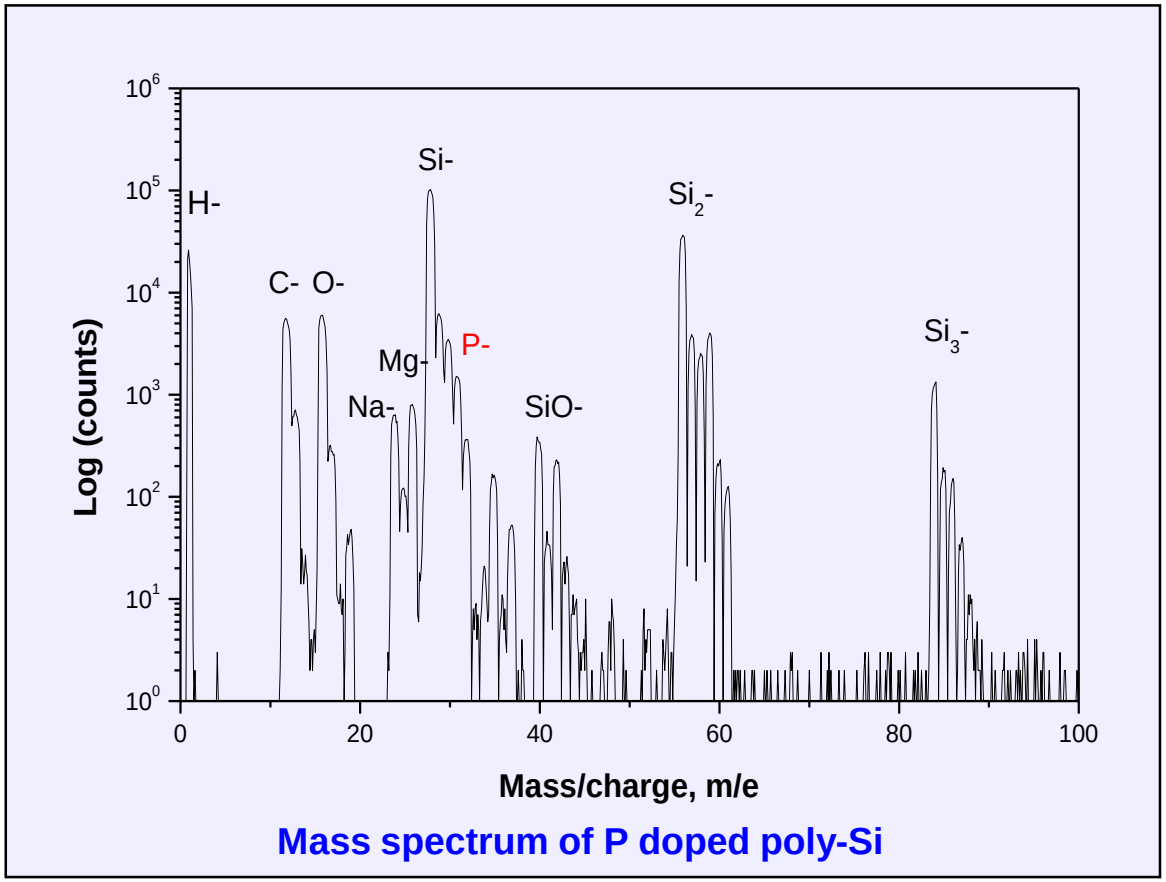
*Varies with vacuum conditions

SIMS Analysis Method



- Elemental analysis
- 3 dimensional analysis
- Lateral resolution down to 100 nm
- Depth resolution down to 2 nm
- Detection limits from ppm to ppb
- Quantitative analysis with standard samples

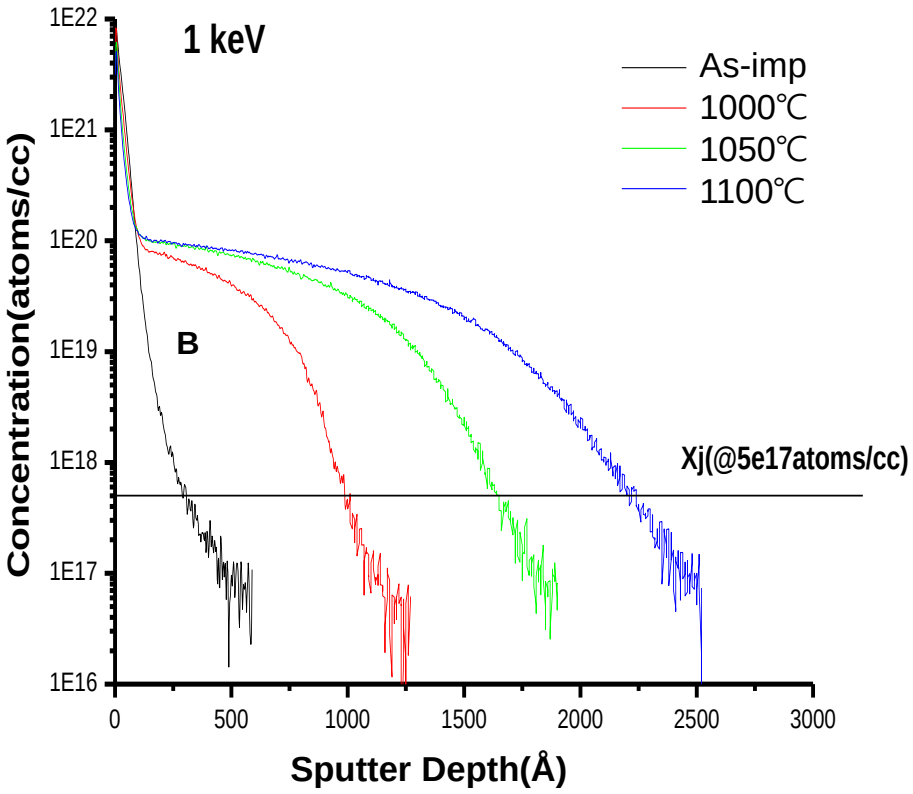
Mass Spectrum



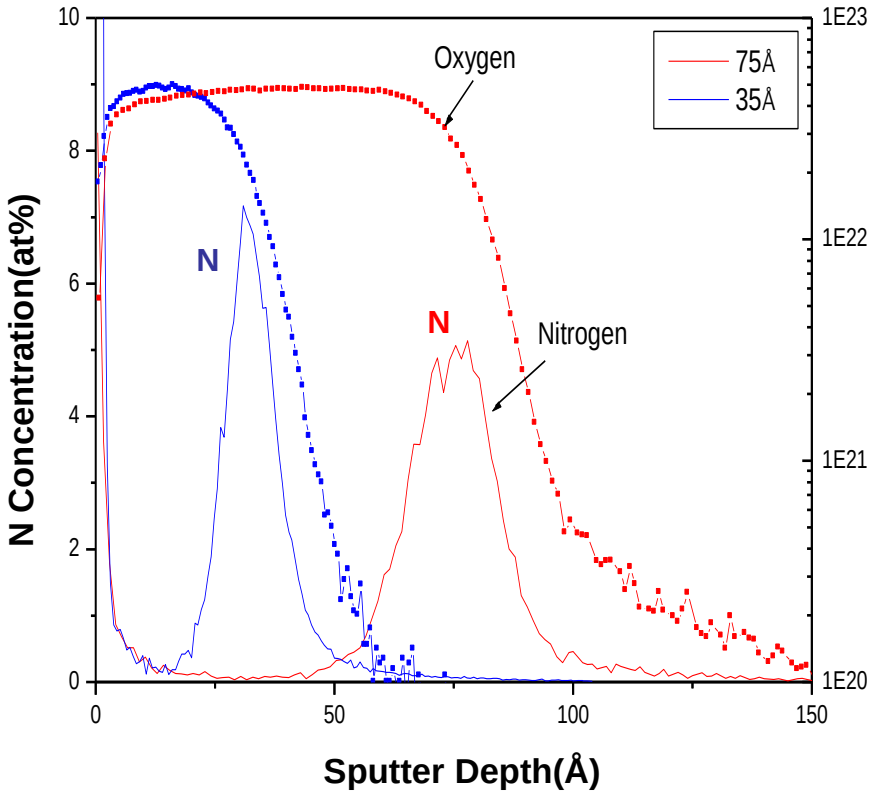
Mass spectrum of P doped poly-Si

- Mass Spectra는 미리 선택된 Mass range를 Mass Spectrometer가 scan하는 동안 Sample을 Sputtering해서 형성된 ion signal를 monitoring함으로써 sample surface에 존재하는 성분을 분석하는 방법
- Mass Spectra에 나타난 Ion intensity는 element의 isotopic abundance를 나타냄

Depth Profile : Si 반도체의 SIMS 분석



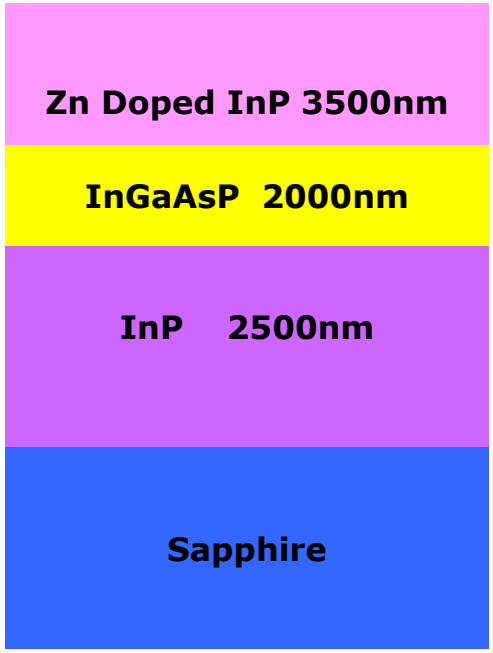
Si 반도체에서의 **B** 이온주입과 열처리에 따른 **B** 깊이 프로파일의 평가

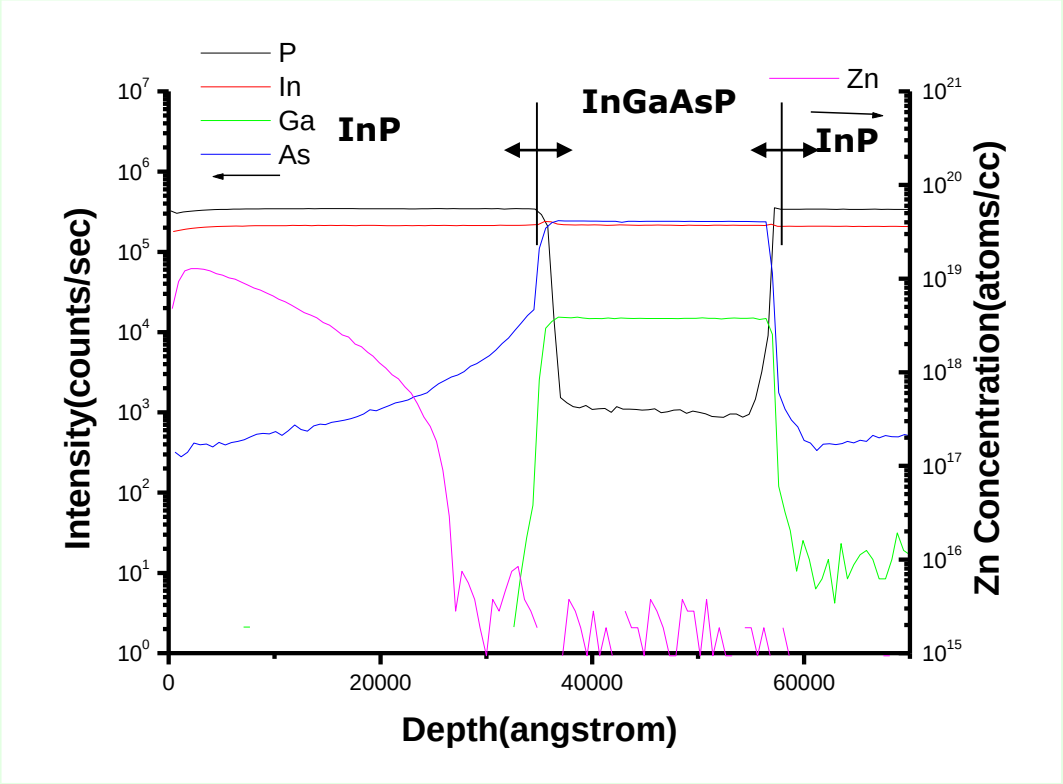


산화박막(**7.5 nm**, **3.5 nm**)에서의 질소 농도 분석

5um

Depth Profile : 화합물반도체

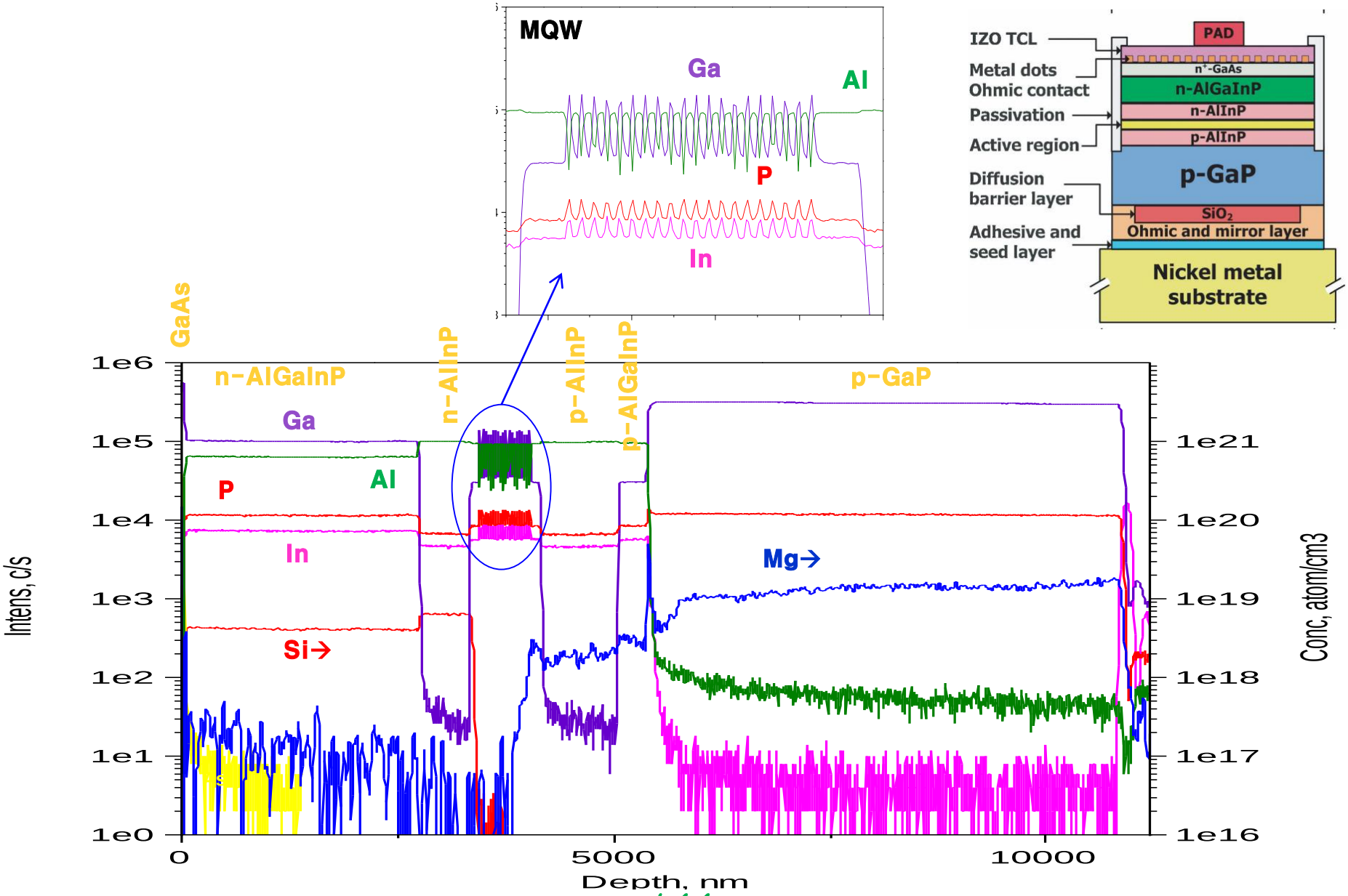




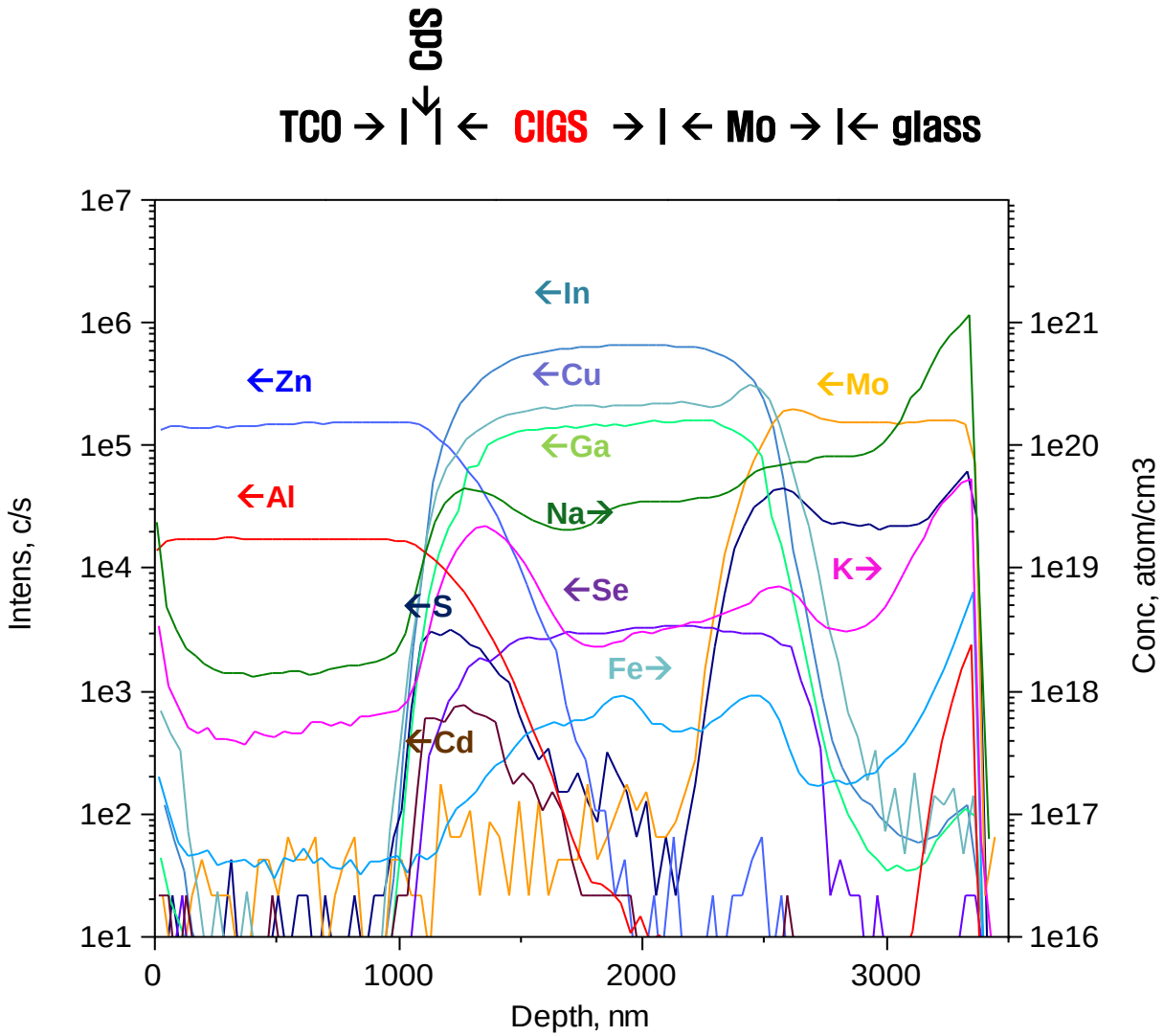
Multi-layer depth profile

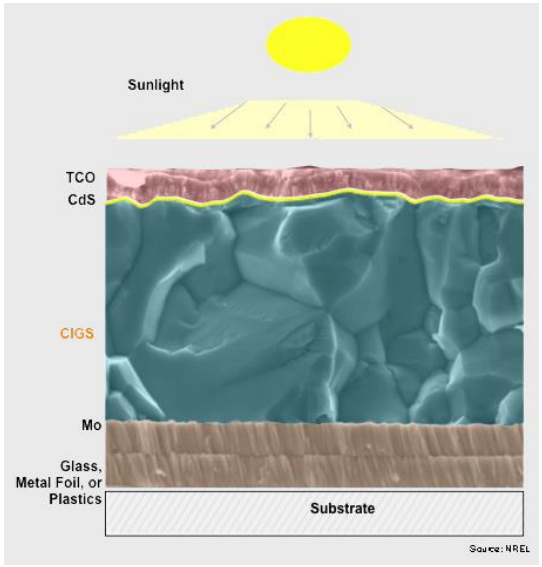
- 특정 Element 들에 대해 깊이별 농도 분포를 확인하기 위한 분석 Mode
  : 깊이에 따른 원소 분포 및 Film interface 부근에서의 Impurity pile-up 확인 가능

Depth Profile : LED Chip의 SIMS 분석



Depth Profile : CIGS 태양전지의 SIMS 분석





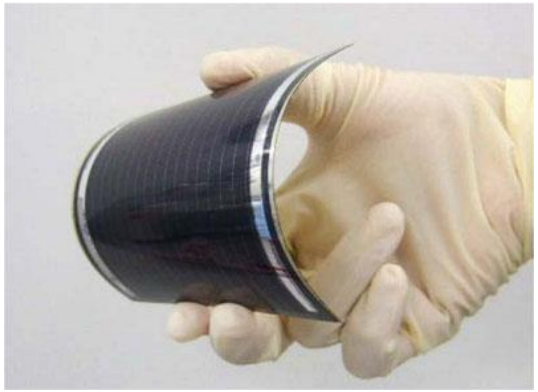
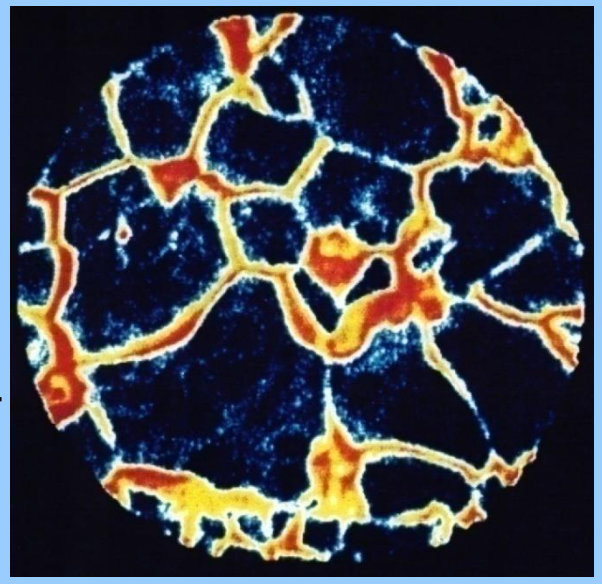
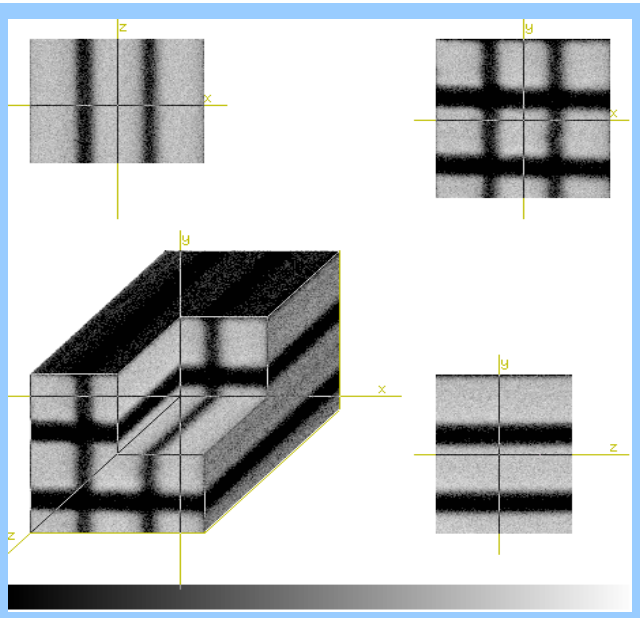


Image Acquisition





3D mapping

- Sample surface의 위치에 따른 Secondary ion intensity를 나타내는 분석 방법
- 특정 Area에 대해 Sputtering하면서 깊이별로 Mapping하여 3D 분석가능

SIMS Characteristics

Advantages

- Detection limits of ppm for most elements and ppb for favorable elements
- All elements detectable
- Isotopes can be distinguished (ex. ^{10}B & ^{11}B)
- Depth resolution of 2~5 nm possible, and 10~20 nm typical

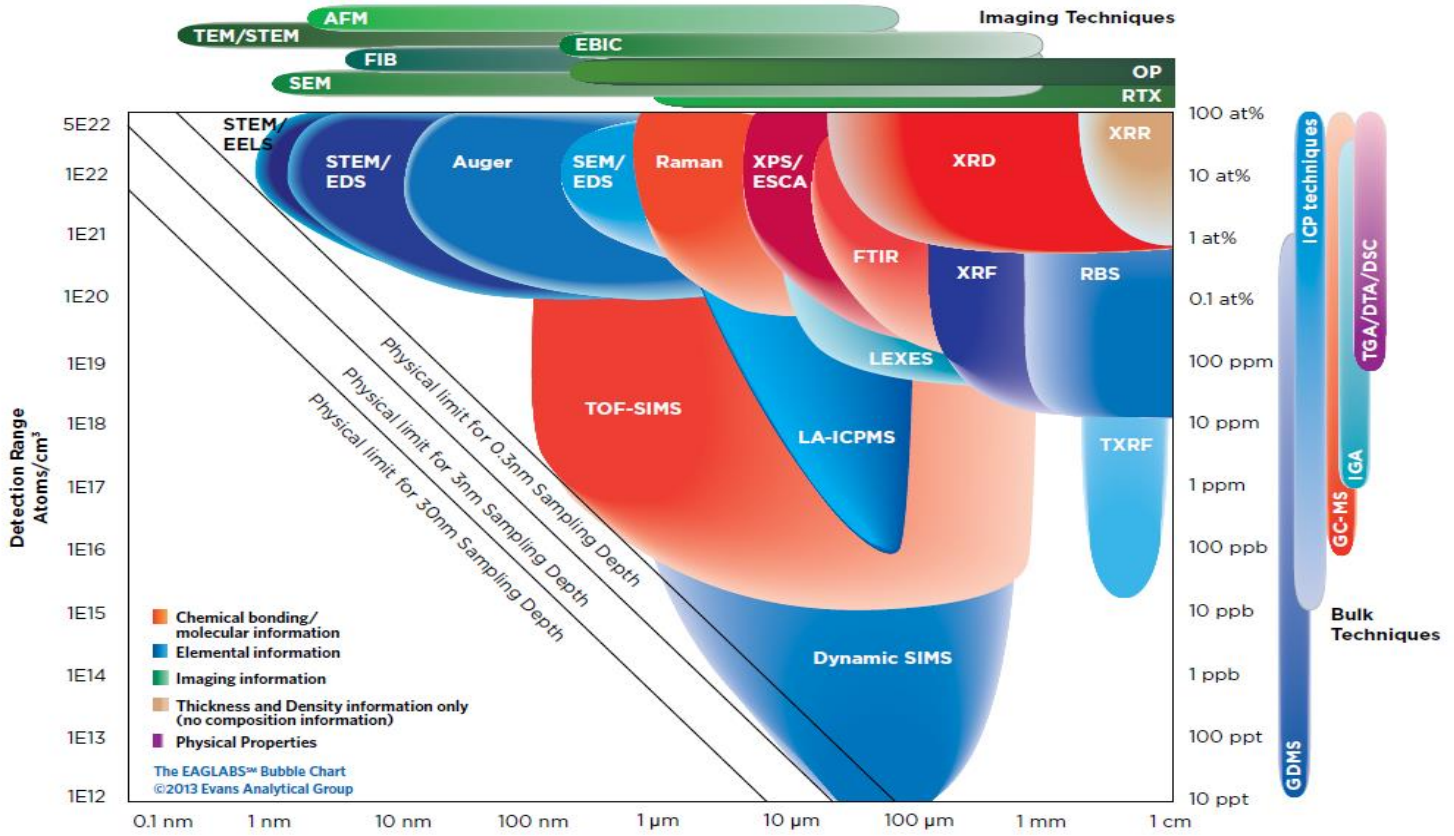
Disadvantages

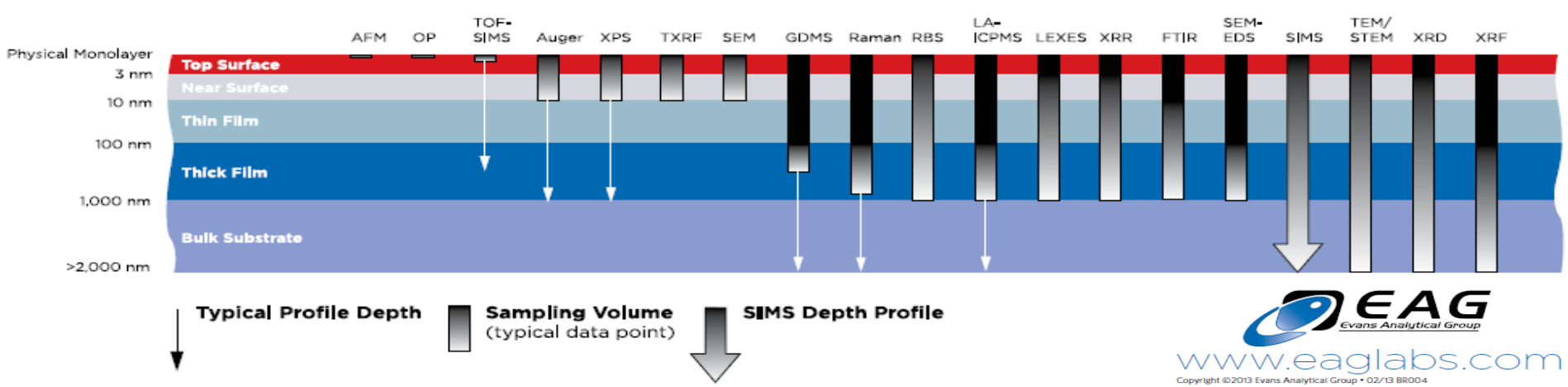
- Mass interference (ex. ^{31}P *vs* $^{30}Si^{1}H$)
- Secondary ion yields are often matrix dependent
- Flat surface required for best depth resolution
- Destructive analysis

Comparison of Surface Analysis

	Typical Application	Signal Detected	Elements Detected	Detection Limits	Depth Resolution	Lateral Resolution
XPS	Surface analysis of organic and inorganic materials, depth profiling	Photoelectrons	Li~U Chemical bonding information	0.1~1.0 at%	2~20 nm (Profiling mode) 1~10 nm (Surface analysis)	10 μm~ 2 mm
AES	Surface analysis, microanalysis, micro area depth profiling	Auger electrons	Li~U	0.1~1.0 at%	2~20 nm (Profiling mode) 3 nm (Surface analysis)	≥ 10 nm
SIMS	Dopant and impurity depth profiling, surface and microanalysis, insulator analysis	Secondary ions	H~U	10^{12}~10^{16} at/cm^3 (ppb~ppm)	1~30 nm	≥ 30 μm (Depth profiling) 1 μm (Imaging mode)

Analytical Resolution vs. Detection Limit





EAG Evans Analytical Group

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