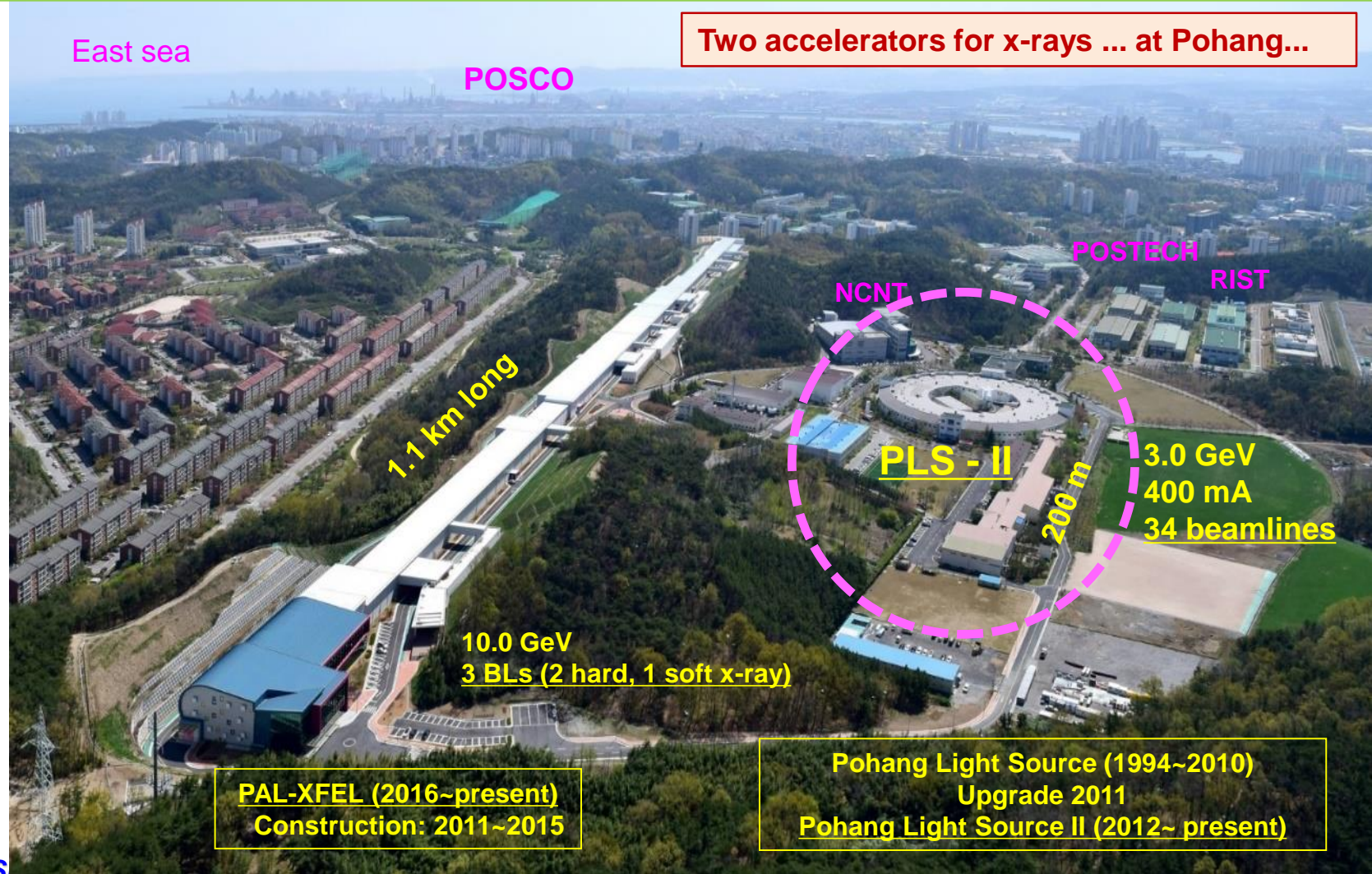


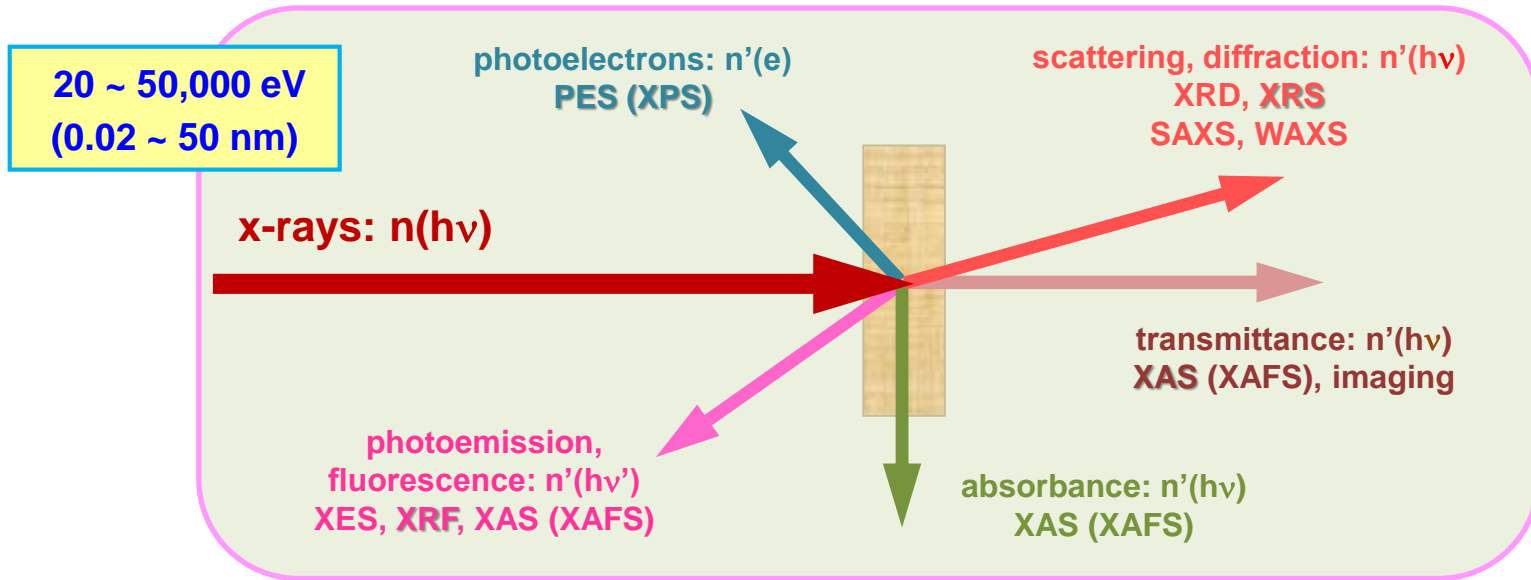
# Pohang synchrotron radiation facility (PLS-II) and its application activities

신 현 준

방사광연구단, 포항가속기연구소



# Use of x-rays; a probe based on light-matter interaction...



**PES:** photoemission/ photoelectron spectroscopy  
**XPS:** X-ray photoemission/ photoelectron spectroscopy  
**XRD:** X-ray diffraction  
**XRS:** X-ray scattering  
**SAXS:** Small angle x-ray scattering  
**WAXS:** Wide angle x-ray scattering  
**XAS:** X-ray absorption  
**XAFS:** X-ray absorption fine structure  
**XES:** X-ray emission spectroscopy  
**XRF:** X-ray fluorescence

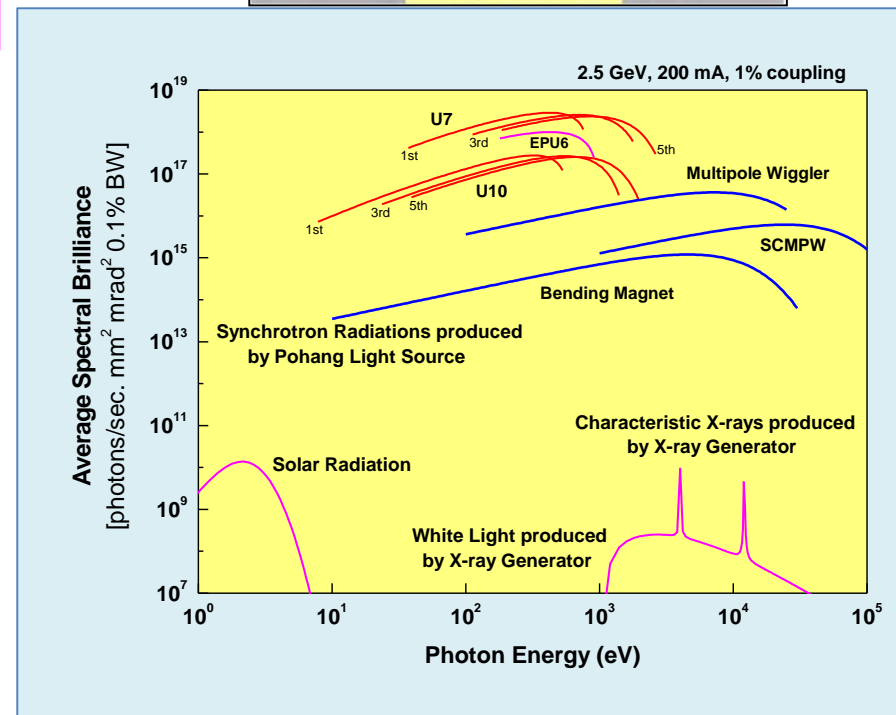
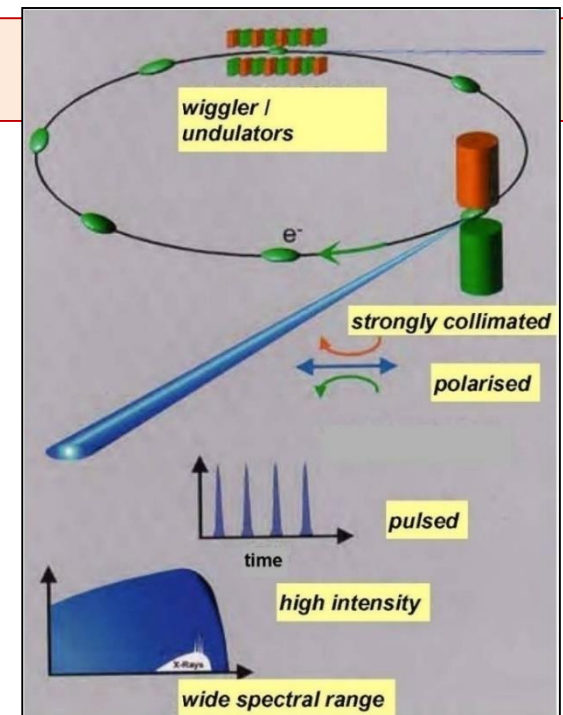
# Synchrotron-radiation X-rays (soft – hard) !!!

## PLS-II storage-ring parameter;

- Electron beam energy: 3 GeV
- E-beam current: 300 ~ 400 mA, top-up fill (3-5 min.)
- **User beam time: 190 days/year**
- Emittance: 5.8 nm.rad
- Straight sections for ID: 20
- Circumference: 281.8 m, 12 Cells, DBA
- Linac: 170 m, 3GeV

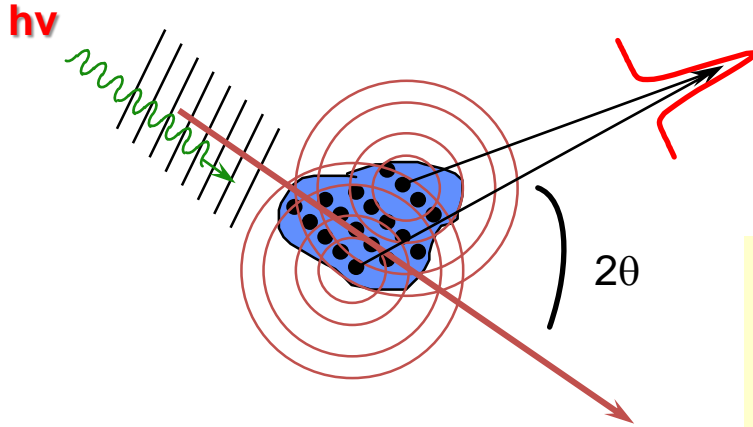
## Compared to other x-ray sources;

- **Intense and bright**
- **Wide spectral range: tunable...**
- **Stable**
- **Pulsed x-rays: ~ 2 ns, ~ 30 ps**  
(1 bunch ~ 500 MHz, 1 turn ~ 1 MHz)
- **Small source size: coherent...**
- **Polarized x-rays: linear, circular**



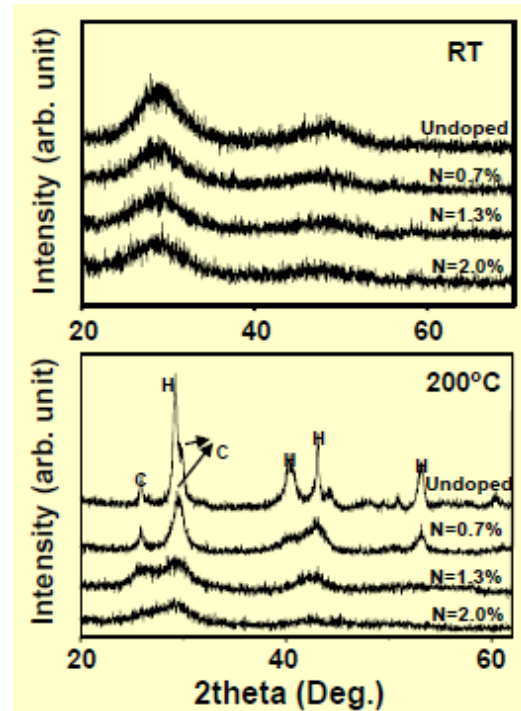
# x-ray scattering (XRS), x-ray diffraction (XRD)

Constructive interference  
at Bragg condition



$$2d \sin(\theta) = \lambda \quad \Rightarrow \quad d = \lambda / 2 \sin(\theta)$$

$$\theta : 0.01^\circ \sim 90^\circ \quad \Rightarrow \quad d : 1 \mu\text{m} \sim 0.02 \text{ nm}$$



**Science** HAAAS HIGHLIGHTS OF 2015

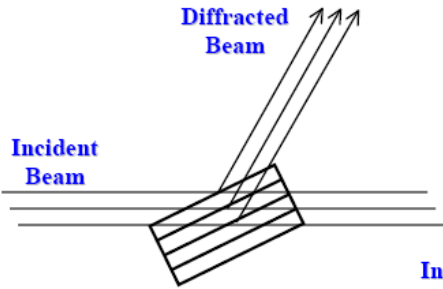
**CO<sub>2</sub> capture from humid flue gases and humid atmosphere using a microporous copper silicate**

Shuvojit Datta et al.  
Science 2015, 350, 302-306  
서강대학교, 윤경병

PLSII 2D Supramolecular Crystallography

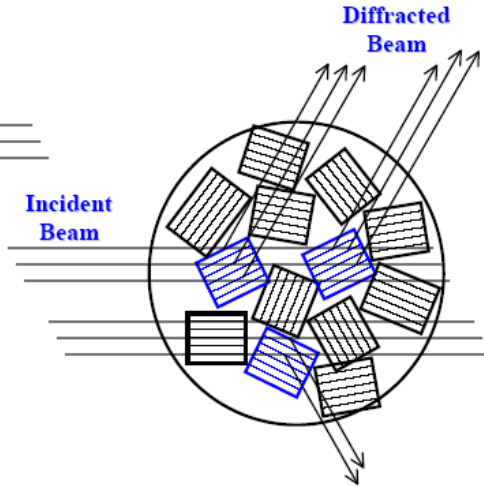
# Powder Diffraction

## Single Crystal Diffraction

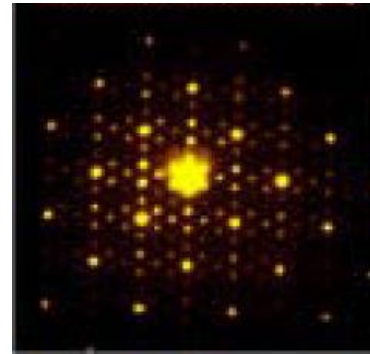


In powder diffraction only a small fraction of the crystals (shown in blue) are correctly oriented to diffract.

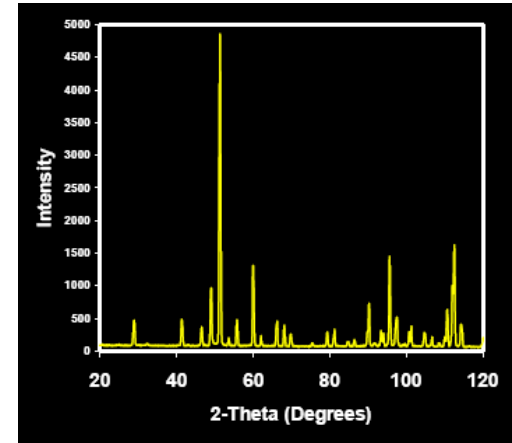
## Powder Diffraction



Courtesy of Dr. D. C. Ahn



Single Crystal Diffraction (3 dimensional)



The ideal "powder" sample contains tens of thousands of randomly oriented crystallites

$$I_{hkl} \propto |F_{hkl}|^2$$

$$F_{hkl} = \sum_{j=1}^m N_j f_j \exp[2\pi i(hx_j + ky_j + lz_j)]$$

**nature** HIGHLIGHTS OF 2015

A zeolite family with expanding structural complexity and embedded isorecticular structures

Structure prediction and successful synthesis of the targeted zeolites

	RHO-01 (RHO)	RHO-02 (PAU)	RHO-03 (ZSM-25)	RHO-04 (PST-25)	RHO-05 (PST-05)
Scaffold					
Embedded cages					
Entire framework					

Na-ECR-18, Na-RHO, Na-ZSM-25

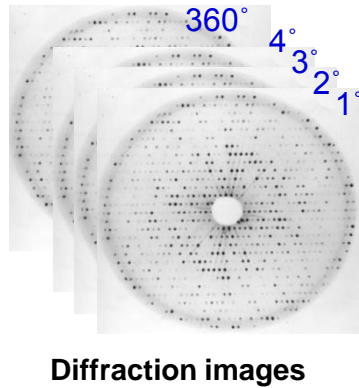
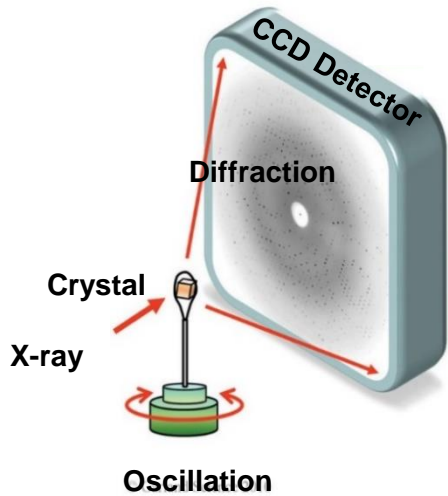
Uptake (mmol/g)

Time (min)

"Good selective CO<sub>2</sub> adsorption properties of new zeolite"

Jiho Shin et al. Nature 524, 74-78 POSTECH, Suk Bong Hong

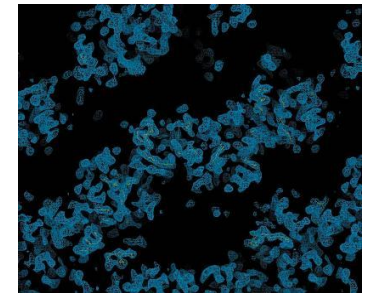
BL9B HRPD (High Resolution Powder Diffraction)



**Fourier transform**



HKL2000  
Phenix  
CCP4



Electron density map

**Model building  
refinement**



**Cell** HIGHLIGHTS OF 2016

Structure of Human DROSHA

DROSHA (Human)  
Dicer (Giardia)

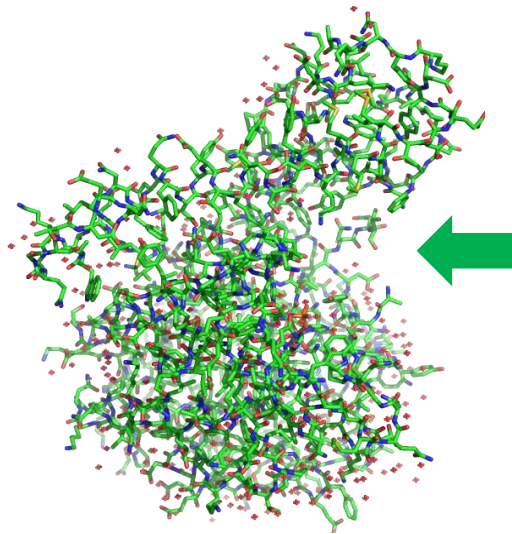
DGCR8 C-terminal tail  
RliIDb  
Catalytic sites  
Connector  
Platform  
DROSHA  
PAZ-like

DGCR8  
~11 bp (~28 Å)  
"Bump"

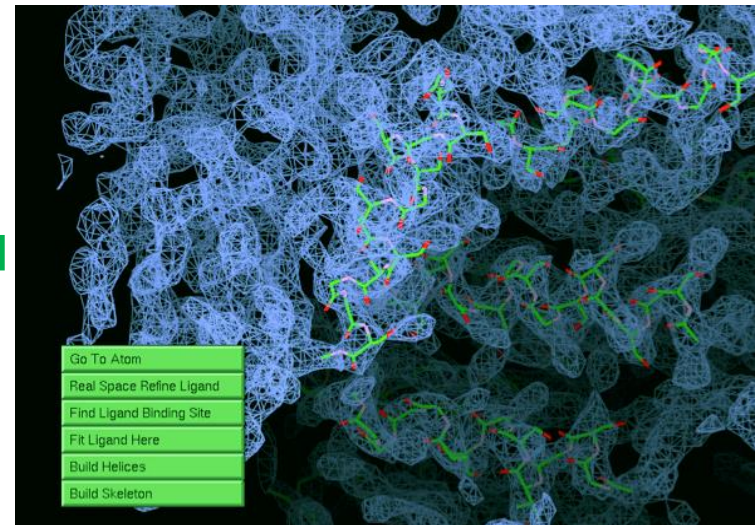
DROSHA  
~25 bp (~65 Å)  
Giardia Dicer

단백질 결정학 비라인 sc

CELL 2016, 164, 81-90  
IBS RNA연구단, 김빛내리 교수



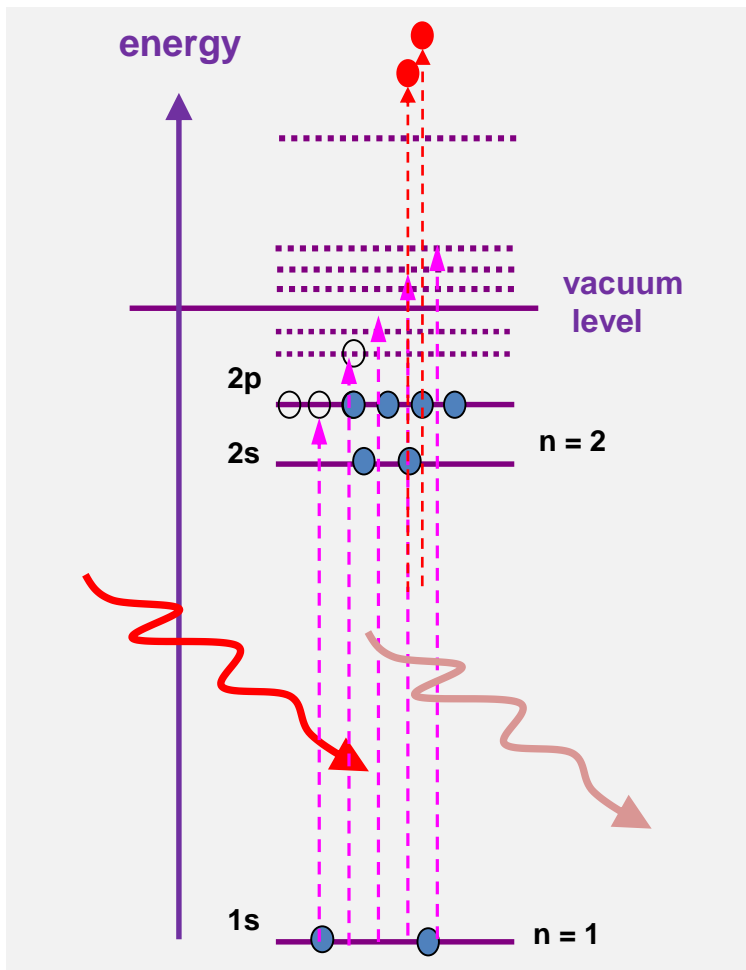
Final molecular model



Courtesy of Dr. Y. Kim

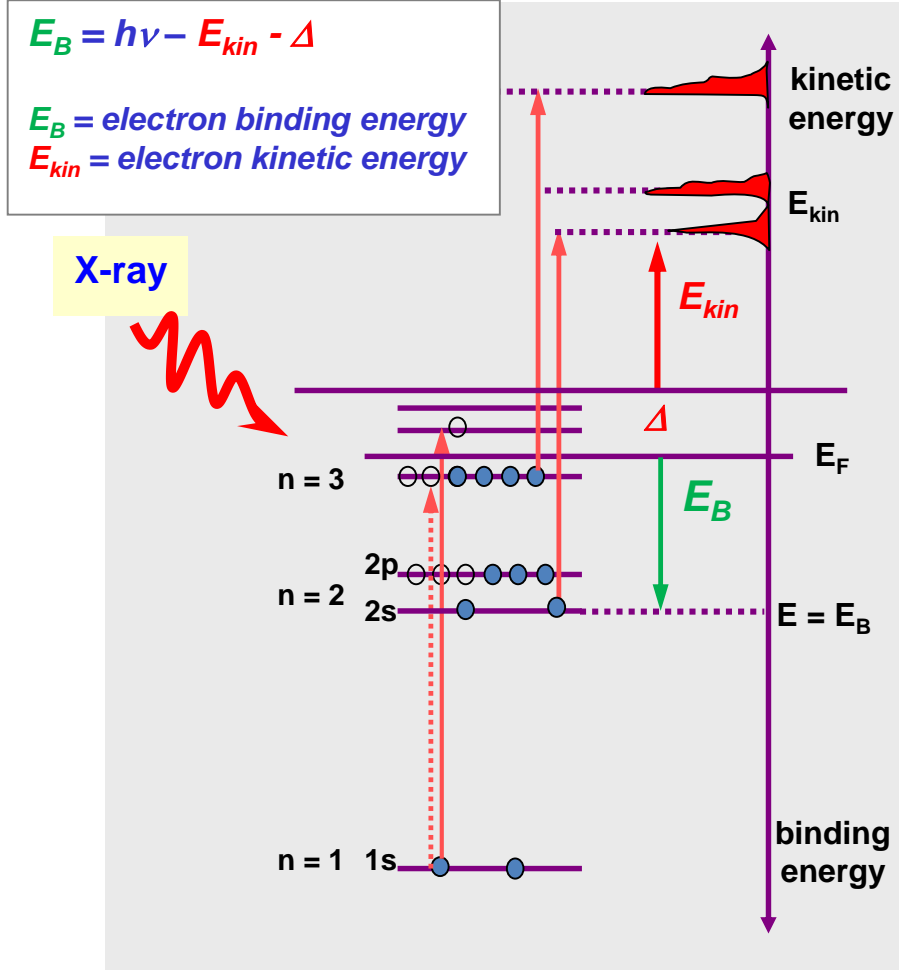
**XAS**

**unoccupied DOS**

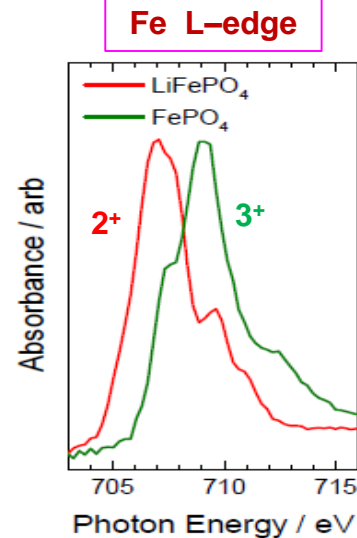
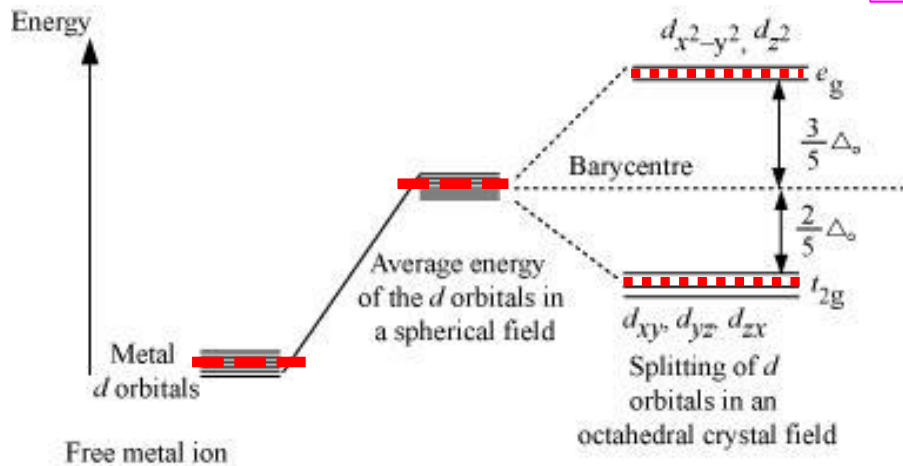


**XPS**

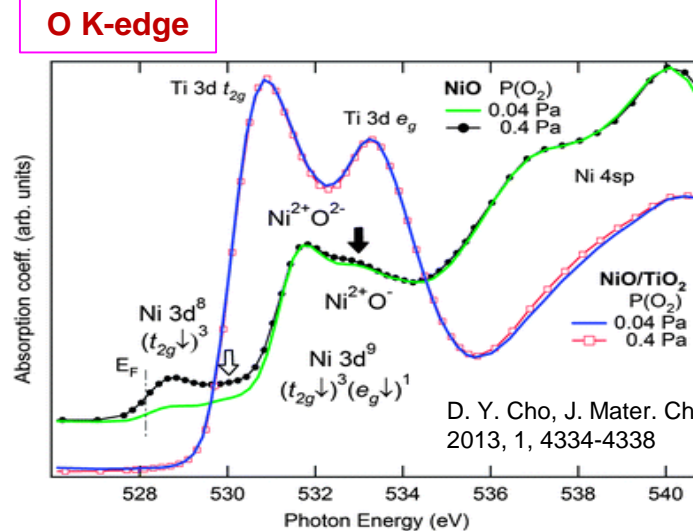
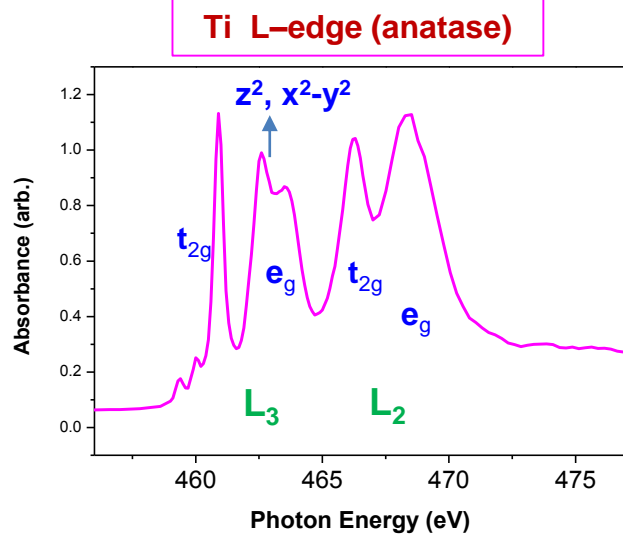
**occupied DOS**



# X-ray absorption spectroscopy (XAS): practical use of soft x-rays...



W. C. Chueh et al,  
Sandia Report, 2012.

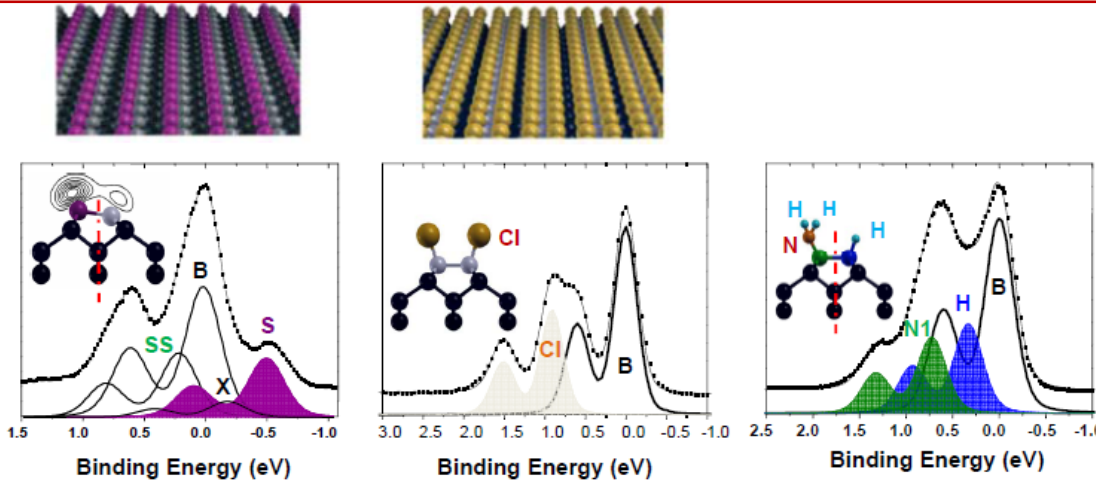
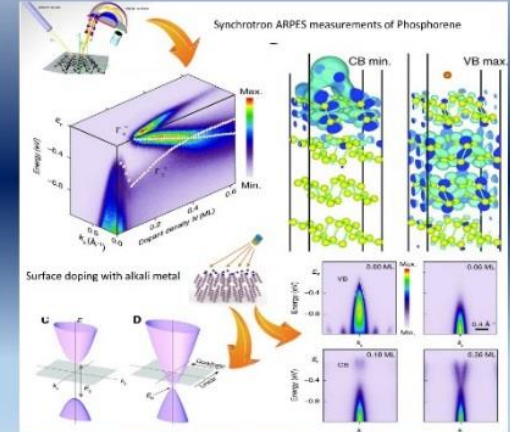


D. Y. Cho, J. Mater. Chem. C.  
2013, 1, 4334-4338

element, crystal structure, oxidation state, chemical states, magnetic moment,  
electronic structure, ...



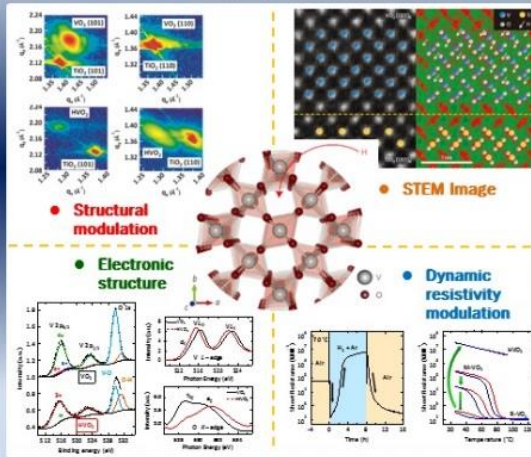
## Observation of tunable band gap and anisotropic Dirac semimetal state in black phosphorus



Moon et al., Adv. Mat. 19, 1321 (2007)  
Appl. Phys. Lett. 91, 193104 (2007)

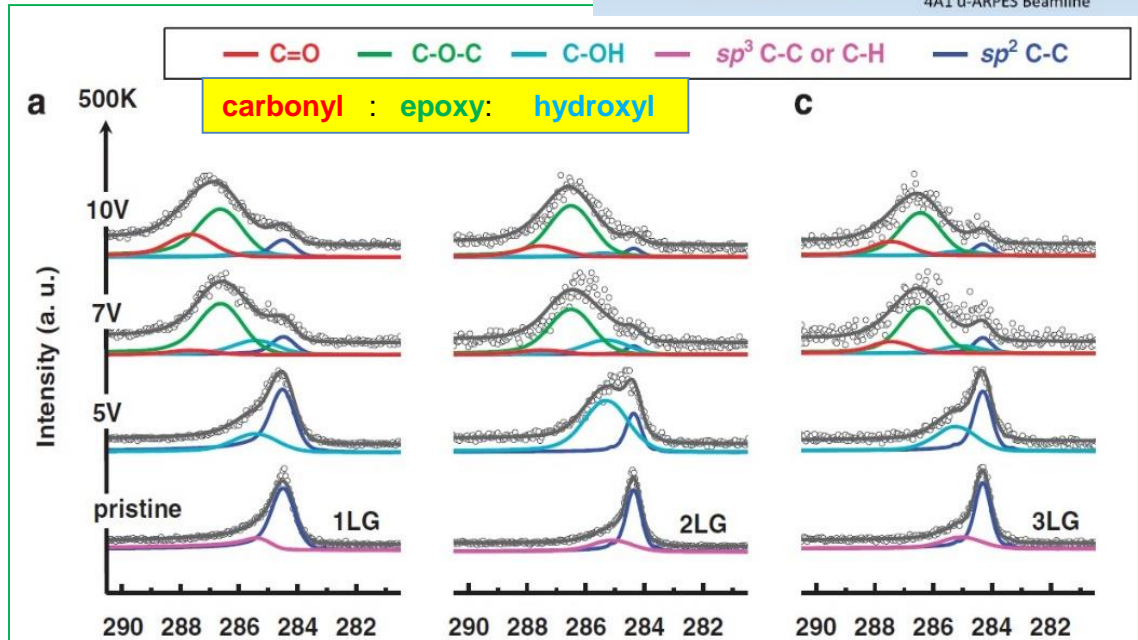
nature materials

## Reversible phase modulation and hydrogen storage in multivalent VO<sub>2</sub> epitaxial thin films

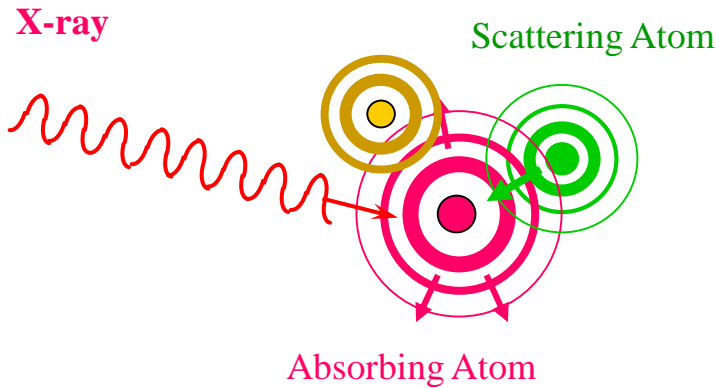


Nature materials, 15, 1113 (2016)  
포항공대, 손준우 교수

I.-S. Byun, NPG Asia Mat. (2014)

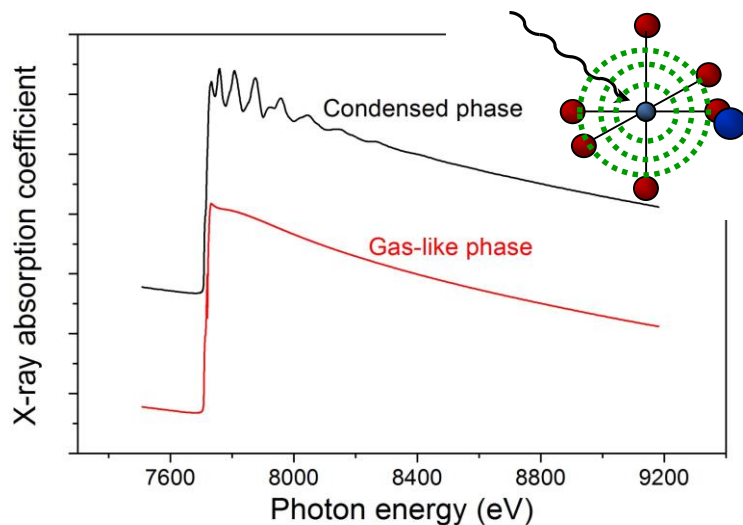


# Extended X-ray Absorption Fine Structure (EXAFS)



$$E_{(\text{photon})} - E_{o(\text{electron binding})}$$

$$= K.E. = \frac{h^2 k^2}{2m_e}$$



REPORT

SOLAR CELLS

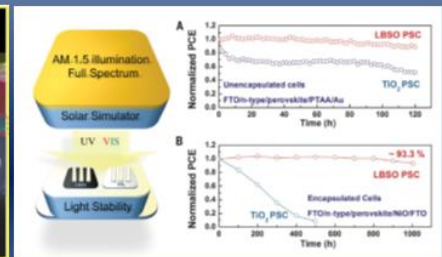
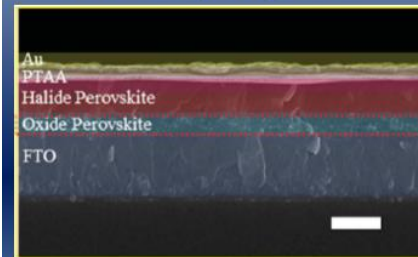
Colloidally prepared La-doped BaSnO<sub>3</sub> electrodes for efficient, photostable perovskite solar cells



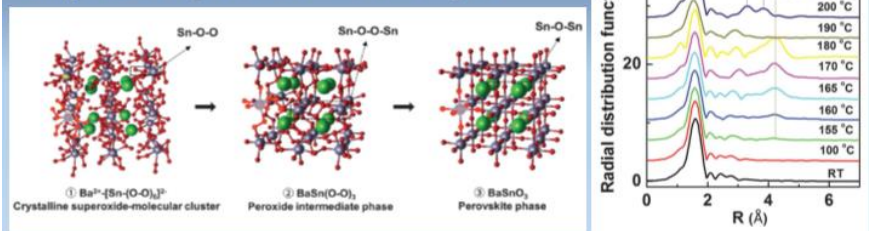
## Perovskite solar cells (PSCs)

Lanthanum (La)-doped BaSnO<sub>3</sub> (LSBO) perovskite as an electron-transporting layer :

a steady-state power conversion efficiency of 21.2%, versus 19.7% for a mp-TiO<sub>2</sub> device

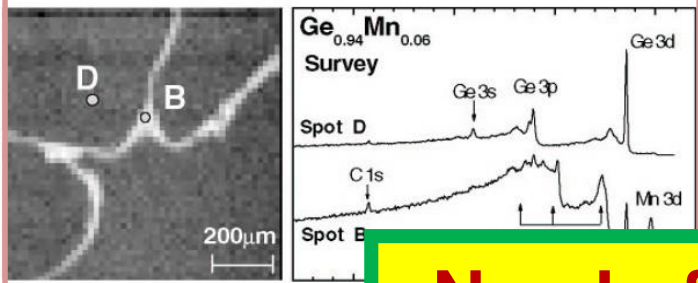


## Structural Evolution of Cubic Lanthanum (La)-doped BaSnO<sub>3</sub> (LSBO) perovskite through Low temperature synthesis below 200 degree



Science, 356, 167–171 (2017)

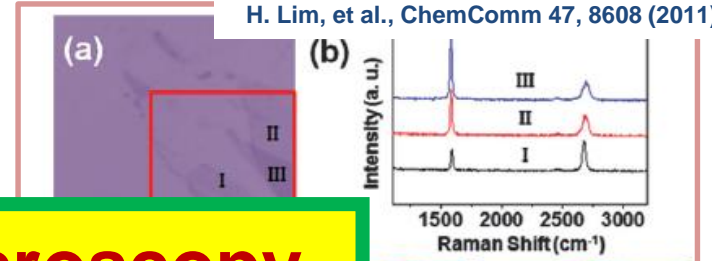
유니스트, 석상일교수



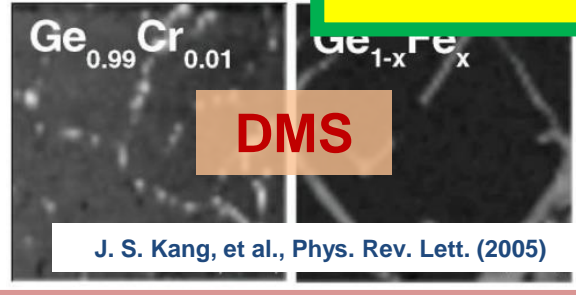
**Optoelectronic device**

K.J. Kim et al., Adv. Mater. 20, 3589 (2008)  
H. Lim, et al., ChemComm 47, 8608 (2011)

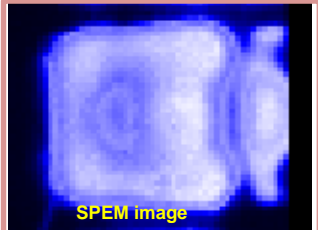
**Need of spectro-microscopy**



**Graphene**



Appl. Phys. A 78, 623 (2004)

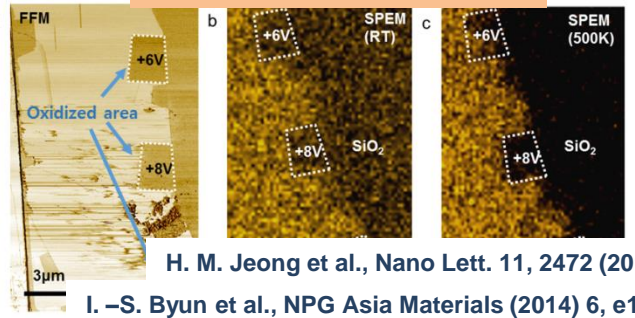
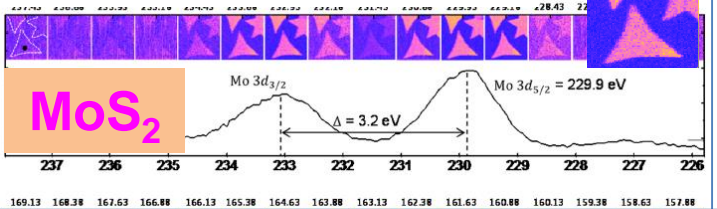


**PDP**



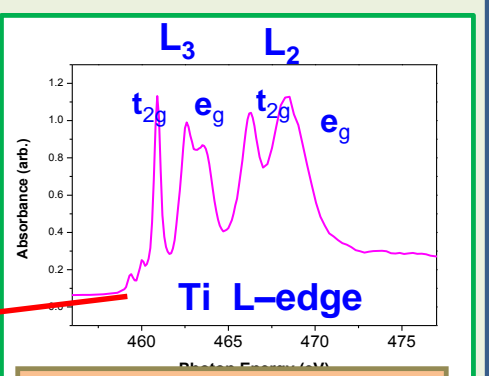
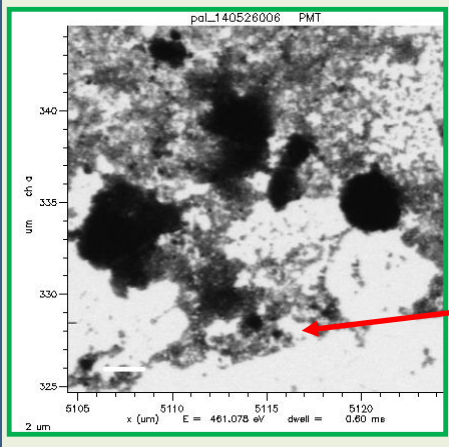
JJAP 44, 861 (2005)

I. Song et al., Angew. Chem. Int. Ed. 53, 1266 (2014)  
W. Park et al., ACS Nano 8, 4961 (2014)

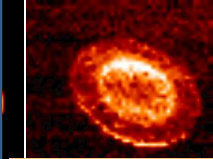


H. M. Jeong et al., Nano Lett. 11, 2472 (2011)

I. -S. Byun et al., NPG Asia Materials (2014) 6, e102



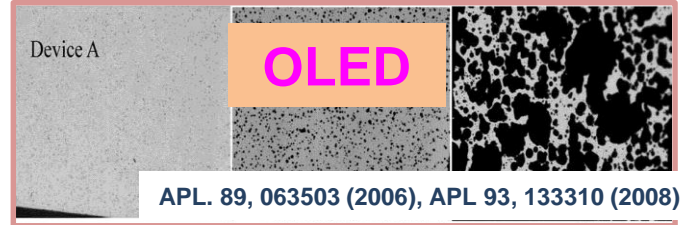
**Anatase TiO2**



**Kevlar**



**Polymer**



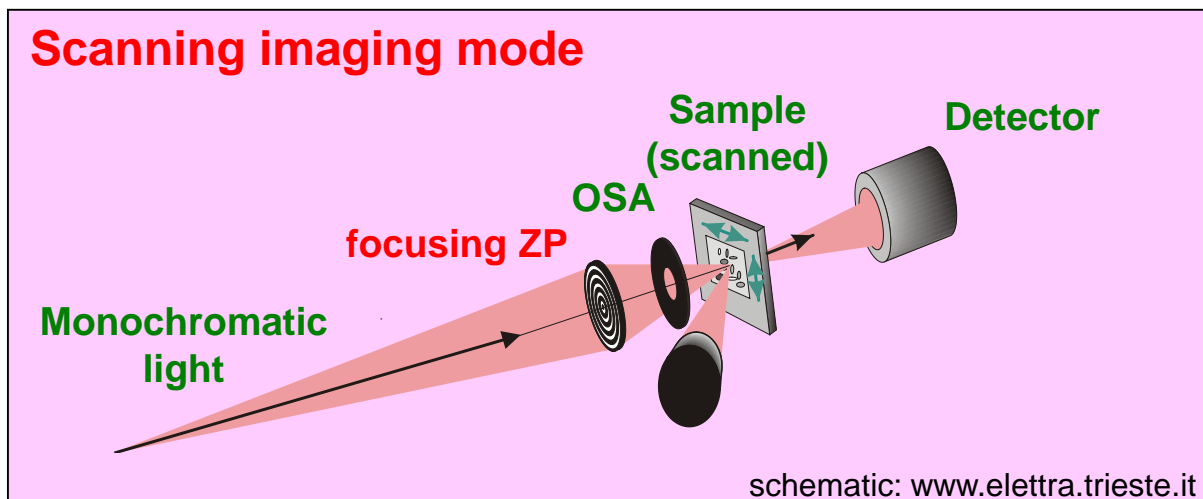
**OLED**



**PLED**

J. Chung et al., Org. Electron. 9, 869 (2008)

## Microscopy-spectroscopy /// Spectro-microscopy (Nanoscopy)

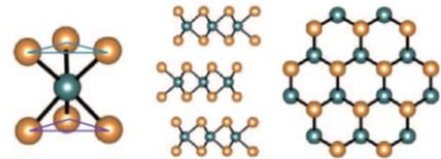


- **improvement of focal power/ focusing lens ...**
  - **minimal variation of focal position ...**
  - **detectors for fast imaging ...**
  - **nice softwares ...**

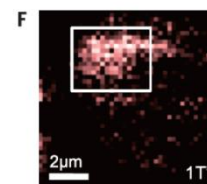
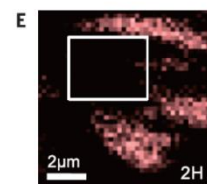
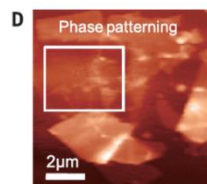
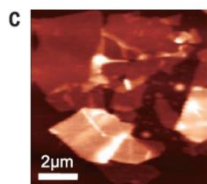
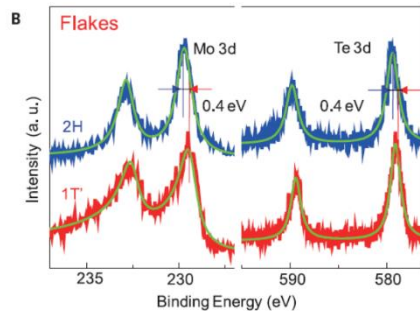
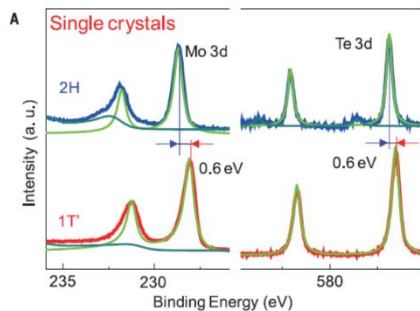
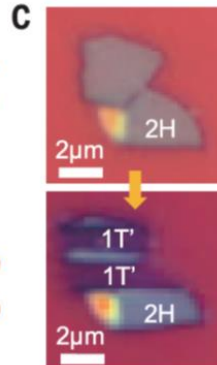
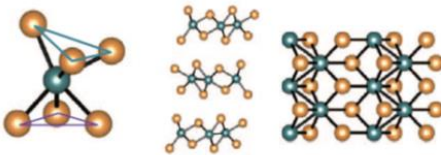
# MoTe<sub>2</sub>

## Heterophase in homojunction

hexagonal  
2H



metallic  
monoclinic  
1T'



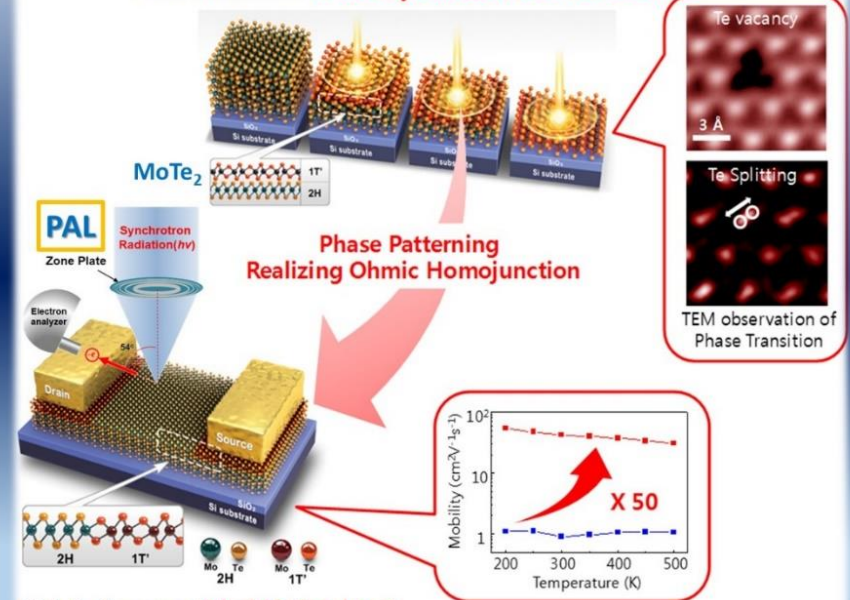
Different lattice symmetry  
No MoO<sub>3</sub>, TeO<sub>2</sub>, other element..

Science  
AAAS

HIGHLIGHTS OF 2015

# Phase patterning for ohmic homojunction contact in MoTe<sub>2</sub>

Laser Irradiation : Te-vacancy Induced Phase Transition



High Performance "Ideal 2D Transistor"

Suyeon Cho et al. *Science* 349, pp625-628

IBS Center for Integrated-nanostructure Physics,  
Sungkyunkwan Univ., Heejun Yang, Young Hee Lee

Scanning Photoelectron Microscopy (SPEM) @ 8A1



**and ... ..**

- 개발 여지가 많음: 정밀도 (에너지분해, 공간분해, 편광 정도), 첨단화 (사용의 편의성, 효율, 자동화, 실시간, *operando*), 새로운 원리 (편광, 검출기, 시분해, *two photon*), ...

Status: beamline map..

# ~ 34 PLS II Beamlines

<http://pal.postech.ac.kr>

Public Beamline: 23	■ ID
Agreement Beamline: 7	■ BM
Exclusive Beamline: 4	** Construction

fs-THz (Technical Building II)

e<sup>-</sup>

\*\* 8A2\_AP-XPS

8A1\_Scanning Photoelectron Microscopy

8C\_Nanoprobe XAFS

7D\_XAFS

8D\_XRS/XAFS POSCO

7C\_X-ray Nano Imaging

9A\_Ultra-Small Angle X-ray Scattering

7A\_Structural Biology I

9B\_High Resolution Powder Diffraction

\* 9C\_Coherent X-ray Imaging

6D\_UNIST-PAL

9D\_X-ray Nano-Micro Machining

6C\_BioMedical Imaging

10A\_STXM\_Soft X-ray Nanoscopy

6A\_MPK, Middle energy Soft X-ray Spectroscopy

\*\* 10A2\_HR-PES II

5D\_X-ray Scattering GIST

10C\_Wide Energy XAFS

5C\_Structural Biology II

10D\_HR-PES I / XAS KIST

5A\_Material Science XRS

11B\_

4D\_Photoemission Spectroscopy

11C\_Micro Macromolecular Crystallography

4C\_Small Angle X-ray Scattering II

12D\_Infrared Spectroscopy

4B\_X-ray Microdiffraction

\*\* 1C\_Time-resolved X-ray Science

4A2\_Spin & Angle Resolved PES

4A1\_u-Angle Resolved PES

1D\_XRS KIST-PAL

3D\_X-ray Scattering

3C\_Small Angle X-ray Scattering I

3A\_Resonant X-ray Scattering

2A\_Magnetic Spectroscopy

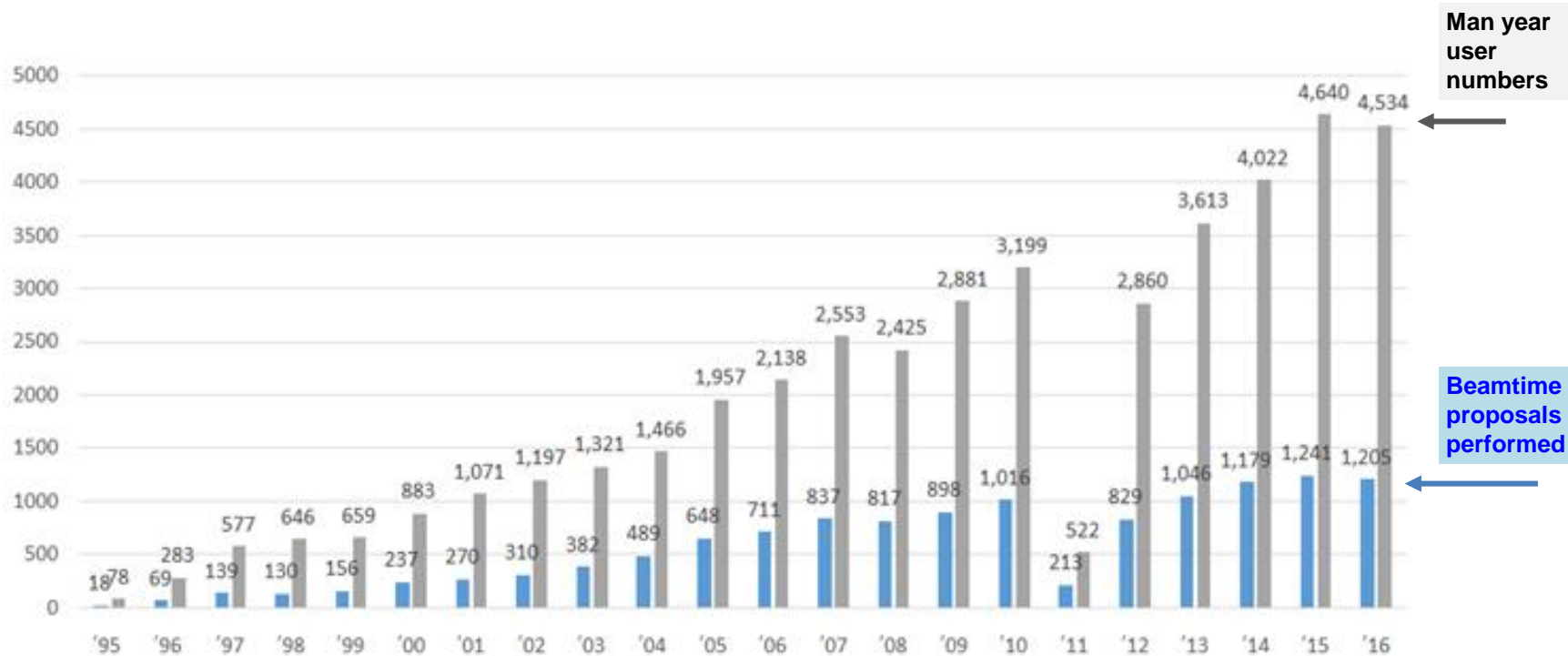
2D\_Supramolecular Crystallography

X-ray Scattering  
Protein crystallography  
SAXS

Photoemission  
Absorption spectroscopy

Other

## Performed beamtime proposals and user numbers ... publication status...



### Number of publication and average IF.

구분	'96	'97	'98	'99	'00	'01	'02	'03	'04	'05	'06	'07	'08	'09	'10	'11	'12	'13	'14	'15	'16	합계
SCI 논문수	3	14	48	77	64	112	145	163	179	188	256	326	390	385	407	451	293	353	551	451	418	4,856
편당 I.F.	2.6	1.8	2.6	2.5	2.4	2.5	3.1	3.2	3.1	3.5	3.4	3.6	3.8	3.6	3.3	3.8	3.9	4.4	4.24	5.86	6.19	



## Beamlines:

**Agreement beamlines;** KIST (2), GIST, UNIST, KRIBB, POSTECH

**Exclusive beamlines;** POSCO, GIST, MPK, IBS

### **Beamline department subsections:**

Structural biology

Materials chemistry

Eco-friendly materials

Energy Materials

Nano-materials spectroscopy

Spectro-nanoscopy

ITCC

## Applications to :

- New materials: semiconductor, energy, bio, life-science, geoscience, natural resources, catalysts, battery, nano & bio, etc.
- putting an emphasis on industrial application.

## Analysis on beamline types...

**General purpose beamlines:** we are trying to maintain competitiveness over other worldwide techniques.

XRS, SAXS, WAXS, PX  
 PES (XPS), XAS, XAFS  
 u-probe (u-XRF, u-XAFS)

→ multimodal, in-situ techniques.

**Top notch science/technique beamlines:**

Uniqueness, world best, world first...

State of the art instrumentations.

Top notch scientific objectives (strongly correlated systems, vortex, ...)

SAR-PES, AP-XPS, XMCD & multimodal, XAS in medium energy,  
 CDI, PCXS, Ptychography  
 tr-XRS, tr-THz

nano-imaging, nano-XAS, nano-XPS

full automation, high throughput, ...

→ multimodal, in-situ techniques.

**Industrial application: \* ITCC \***

High throughput → FBDD (2018-2020 yr.)/ SAXS/ Imaging

High energy x-ray imaging for thicker samples.

Spectro-microscopy (u-XAFS, u-XRF, STXM, SPEM)

AP-XPS, XPS on 2D materials, semiconducting device materials

→ multimodal, in-situ techniques.

## **Beamlines under consideration**

→ Putting efforts on industrial application has become our new mission...

**5C PX FBDD (2018-2020):** → endstation to be upgraded.

Full automation and FBDD facility setup

**2C high energy x-ray science (HE – XRS) (MPW):** (2019 ? – 2021 ?) → to be constructed.

Hard x-ray imaging for tomographic information..

High energy extreme condition science;

includes high pressure science (strong user consortium is established)

### **Beamline renovation plan to attract industries:**

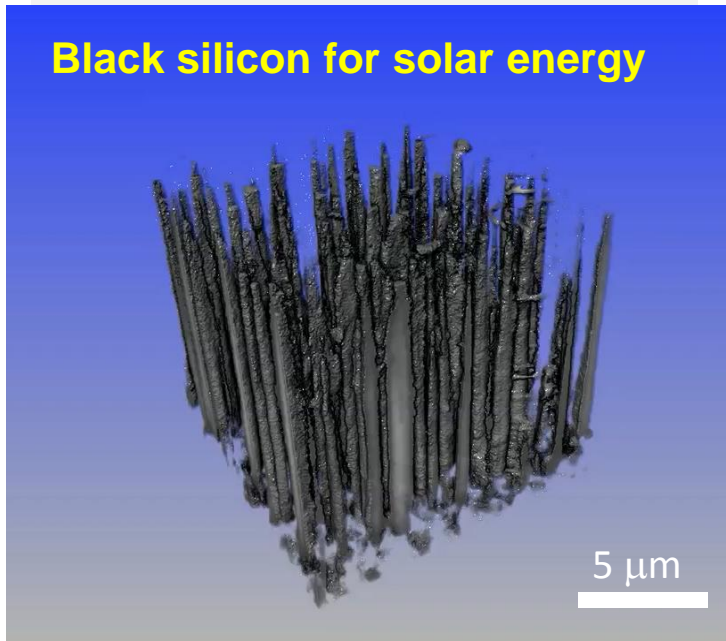
✓ A company is requiring an hard x-ray insertion device beamline for SAXS.

✓ Collaboration is on-going with POSCO and SKhynix, and other companies are showing interests...

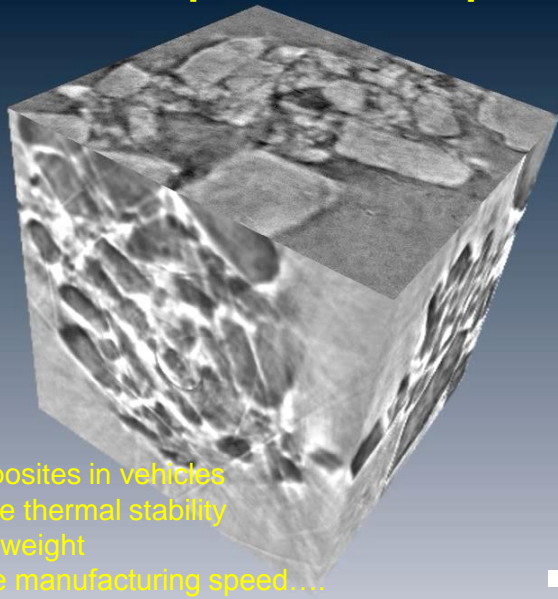
→ We may have to construct beamline(s), or renovate existing beamline(s), or build an efficient platform (comprised of several beamlines) for industrial application.

**3D nano structure inspection**

**Black silicon for solar energy**

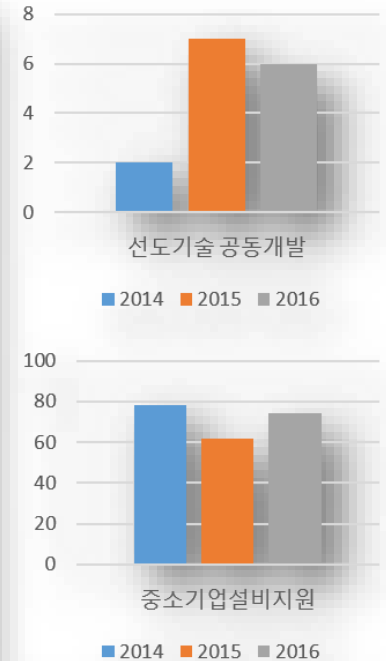
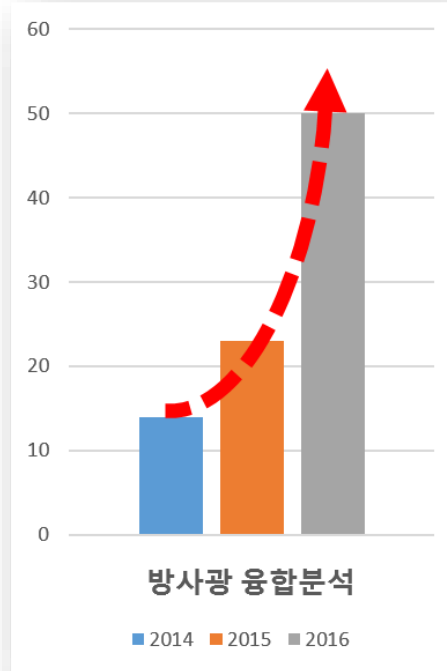


**Nano composite in bumper**



Nano composites in vehicles to enhance thermal stability to reduce weight to improve manufacturing speed...

**방사광 융합 분석 실적 추이**



항목	2014	2015	2016
방사광 융합분석	14	23	50 건
중소기업 설비지원	78	62	74 건
선도기술 공동개발 (기업 과제)	2	7	7 건
기업초청 및 현장방문세미나	27	19	15 회

**Thank you !**