TAIWAN Photon Source

X-ray Nanoprobe Beamline for Nanoscale Physics Research

> Tseng, Shao-Chin 2019/09/26

National Synchrotron Radiation Research Center X-ray Image Group Experiment Facility Division NSRRC

www.nsrrc.org.tw

Outline

- Synchrotron Light Source
- Application of Synchrotron Light
- X-ray nano probe at TPS



"Light" is indispensable to man's exploration of nature.











Invisible light: IR and X-ray...



http://www.hko.gov.hk/prtver/html/docs/education/ed u02rga/radiation/radiation_02-c.shtml



http://www.yingfukeji.com/appl/grid.html



http://www.uv-teck.com/Ttwjy

Rontgen used a simple accelerator to discover X-rays







Hand mit Ringen (Hand with Rings): Wilhelm Rontgen's first "medical" X-ray, of his wife's hand, taken on 22 December 1895 and presented to Ludwig Zehnder of the Physik Institut, University of Freiburg, on 1 January 1896.

http://en.wikipedia.org/wiki/X-ray



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http://snallabolaget.com/?page_id=666





Photon energy



Electromagnetic wave from a moving charge



同步加速器光源的原理

- ▶ 電子在電子槍(1)內產生,經過直線加速器(2)加速至能量為5,000萬電子伏特。
- > 電子束進入增能環(3)後,繼續增加能量至15億電子伏特(1.5GeV),速度非常接近光速 (0.99999995倍)。
- ▶ 電子束經由傳輸線(4)進入儲存環(5)。
- 當儲存環累積足夠的電子束後,經由各個磁鐵的導引與聚焦,電子束在偏轉磁鐵(6)及插件磁鐵(7)發出同步加速器光源,經由光束線(8)將光源引導至實驗站(9)進行實驗。
 - · 電子束在發出同步加速器光源後,要靠高頻腔(10)來補充失去的能量。





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A Synchrotron Step by Step



A brief history

• First observed:

1947, General Electric, 70 MeV synchrotron

• First user experiments:

1956, Cornell, 320 MeV synchrotron

• First insertion Device:

1979, 7 pole wiggler, SSRL



- 1st generation light sources: high energy physics synchrotrons and storage rings used parasitically for synchrotron radiation – eg DESY (Germany), INS-SOR (Tokyo), SPEAR (USA), (1960's, 1970's)
- 2nd generation light sources: purpose built synchrotron light sources, eg Photon Factory, NSLS, Daresbury (1980s onwards)
- 3rd generation light sources: optimised for high brilliance with low emittance and Insertion Devices; SPRing-8,ESRF, APS, Diamond, ...(1990's onwards)
- Free Electron Laser sources: FLASH (Germany), LCLS (USA), SACLA (Japan), FERMI (Italy) ... (2000's)
 Generations of Synchrotron Light Source

Next??

2015 Cheiron School



Generations of Synchrotron Light Source







2nd generation: dedicated source

3rd generation: low emittance with ID's 4th generation: free electron laser



Third Generation Sources: Undulator Insertion Devices



Angular distribution of synchrotron radiation emitted from various magnets







Unique Features of Synchrotron Light Source

- High intensity
- **Continuous spectrum**
- **Excellent collimation**
- High polarization
- **Pulsed-time structure**

以X光為例,同步加速器光源在 這個波段的亮度比傳統X光機還 要強百萬倍以上!過去需要幾個 月才能完成的實驗,現在只需幾 分鐘便能得到結果。以往因實驗 光源亮度不夠而無法探測的結構, 現在藉由同步加速器光源,都可 分析得一清二楚,也因此得以開 發新的研究領域。



光亮度:指單位時間內通過單位立體角的單位頻寬光子數。



Next Step - X-ray Lasers? Yes → FELs





Linac-Based Free Electron Laser Self-Amplified Spontaneous Emission (SASE)











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Calculations. in vacuum Neutze et al., Nature 2000





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Beamline



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with ultra-fine particles of aluminum oxide)

not so high (alloy of tungsten)

If an optical component is irradiated by too much power

One user opened FE slit excessively. **2kW** Melted



Slit : "Too much is as bad as too little"



Overview of x-ray focusing devices

Diffraction	focus size, focal length [energy]	energy range	aberration -coma -chromatic -figure error
Fresnel Zone Plate	12 nm, f = 0.16 mm [0.7 keV], 30 nm, f = 8 cm [8 keV]	soft x-ray hard x-ray	-coma small -chromatic exist -figure error small
Sputter sliced FZP	0.3 μm, f = 22 cm [12.4 keV], 0.5 μm, f = 90 cm [100 keV]	8-100 keV	-coma small -chromatic exist -figure error large→small
Bragg FZP	2.4 μm, f = 70 cm [13.3 keV]	mainly hard x-ray	-coma small -chromatic exist -figure error small
Multilayer Laue Lens	16 nm(1D), f = 2.6 mm [19.5 keV], 25nm × 40nm, f=2.6mm,4.7mm [19.5 keV]	mainly hard x-ray	-coma large -chromatic exist -figure error small

Refraction	focus size, focal length [energy]	energy range	aberration -coma -chromatic -figure error
Pressed Lens	1.5 μm, f = 80 cm [18.4 keV], 1.6 μm, f = 1.3 m [15 keV]	mainly hard x-ray	-coma small -chromatic exist -figure error large
Etching Lens	47nm × 55nm, f = 1cm, 2cm [21 keV]	mainly hard x-ray	-coma small -chromatic exist -figure error small

Reflection

Kirkpatrick-Baez Mirror	7 nm × 8nm, f=7.5cm [20 keV]	soft x-ray hard x-ray	-coma large -chromatic not exist -figure error small
Wolter Mirror	0.7 μm, f = 35 cm [9 keV]	<10 keV	-coma small -chromatic not exist -figure error large
X-ray Waveguide	95 nm, [10 keV]	soft x-ray hard x-ray	-coma large -chromatic not exist -figure error large







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absorption coefficient



Figure 2.16 (a) The x-ray absorption coefficient μ for beryllium (red curve), silicon (yellow) and lead (blue) as a function of the photon energy. (b) Sharp increases in the absorption occur when the photon energy is just sufficient to eject the electron from the electronic orbital to the continuum. The x-ray absorption (left) and atomic orbital labellings (right) are shown.



Transmission X-ray Microscope (TXM)





Photoemission Spectroscopy

Energy Distribution Curve (EDC)



 $KE = hv - BE - \phi$

$$\frac{d\sigma}{d\Omega} \propto \sum \left| \left\langle \Psi_f \right| A \cdot P \left| \Psi_i \right\rangle \right|^2 \cdot \delta \left(E_f - E_i - h\nu \right)$$

Selection rule: $\Delta l = \pm 1$ $\Delta m_l = 0$ (linearly polarized) $\Delta m_l = \pm 1$ (L. circularly polarized) $\Delta m_l = \pm 1$ (R. circularly polarized)



HAXPES = Hard X-ray photoelectron spectroscopy







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(石膏) crystals

"Crystals"

GaN (hexagonal) crystal cluster grown by hydride vapor phase epitaxy



AlN pyramids grown by MBE





Bragg Law - X-ray reflected by the (hkl) planes



In terms of diffraction, two key characteristics of a set of crystal planes : 1/d and orientation



Single Crystal Diffraction - Laue Diffraction

- •Method: stationary
- •Light source: a polychromatic 'pink' beam (e.g. $\Delta E < 1 \text{ keV} @ 10 \text{ keV}$)
- Applications: orient single crystals, determine their crystal quality, dynamical studies of transient crystalline states (time-resolved study)
 Disadvantage: not well-suited for determining the full atomic structure of a crystal area detector






Synchrotron light source is a powerful tool for basic and applied studies in physics, chemistry, materials, biology and medicine, and their many subfields.



Condensed-Matter Physics



Electronic properties of novel materials can be revealed with X-ray scattering.



(Source: website of Railway Technical Research Institute, Japan)

 $\mathrm{La}_{0.5}\mathrm{Sr}_{1.5}\mathrm{MnO}_4$



Energy Science











structure $\leftarrow \rightarrow$ electrochemical properties of electrode



develop novel electrode materials.

Biological structure: protein crystallography

















TLS Experimental Hall





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掃描式光電子能譜顯微術

(Scanning Photoelectron Microscopy; SPEM)





為何發展光電子能譜顯微術?

小尺度結構分析:STM,TEM,SEM...等

SPEM

小尺度成份分析:







0

Si2s Si2p

0 1000 200800 600 400 Binding Energy (eV)

Survey Spectrum of Silicon Wafer



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典型 ESCA 能譜

光電子發射之特性:

1.非破壞性 2.表面分析技術 3.具化學鍵結分辨能力



Universal Curve





hv e e e⁻ nnne-nnne-nnne-~µm





Provides information about

- Kind of atom
- Number of atoms
- Chemical shift



Rev. Mod. Phys., 54(1982)709



6 Å SiO_2 grown on Si(100)









Parallel Imaging for Chemical State Mapping (PICSM)





 $12 \ \mu\text{m} \times 12 \ \mu\text{m}, 0.1 \ \mu\text{m/pixel}$



* *

* *









新型態同步輻射顯微術簡介







conventional ARPES on a large, pure single crystal





most of the momentum information is lost

as our spot size is much larger than the grain size.

conventional ARPES of polycrystalline graphite

Fermi Surface

nanoARPES of polycrystalline graphite

we can recover all the momentum information by sampling one grain at a time













NSRRC is constructing a low-emittance synchrotronbased light source, Taiwan Photon Source (TPS)

TL

3 Gev 518.4 m 500 mA 1.6nm-rad

TPS



國家同步輻射研究中心 National Synchrotron Radiation Research Center TPS provides opportunities for scientists to reveal electrons, spins, and lattices, covering a wide range of applications.





Comparison of Brilliance between TLS and TPS

The X-ray spectrum (photon energy 8 keV - 70 keV): the brightness of bending magnet >10². the brightness of IDs: $4 \sim 6$ orders of mag.







Groundbreaking 2010-02-07



2010-02-10

2010-05-21

2010-08-04

2011-02-05



2011-09-27

2012-05-14

2013-01-16



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Civil Construction of TPS







Taiwan Photon Source (TPS)

台灣光子源第一道光芒 2014.12.31







The user operation of the TPS will begin in 2015.







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Nanoprobe project is in the first phase at TPS

05A Protein μ-crystallography

09A Temporally <u>Coherent</u> XRD

45A Sub-μm soft X-ray Spectroscopy 21A X-ray NanoDiffraction

> 23A X-ray Nano-probe

TPS Phase-I beamlines

25A Coherent X-ray Scattering

41A Soft X-ray Scattering
X-ray Methods: With tens-nm resolution (incoherent)

Beamline specification

Energy range : 4 - 15 keV

 $10^{10} \sim 10^{11}$ photons/sec

 $< 2 \times 10^{-4}$ with Si(111) crystals

 \sim 40 nm at 10 keV (H \times V,

Energy scanning capabilities.

Energy resolution :

High-order harmonic

contamination:

TAIWAN TPS 23A

Photon flux :

Beam size :

FWHM)

 $\leq 1 \times 10^{-4}$

- nano-XRF (x-ray fluorescence)
 - Element-specific nanoimaging
- nano-XAFS (x-ray absorption fine structures)
 - Local electronic structure
 - Local chemical environments
 - Element-specific, averaged over nano-size area
- nano-XEOL (x-ray excited optical luminescence)
 - X-ray-to-visible down-conversion efficiency in nano phosphor
- nano-PXM (projection x-ray microscopy)
 - Absorption and phase contrast x-ray images

Beyond sub-ten-nm resolution (coherent)

- •nano-CXDI (coherent x-ray diffraction imaging)
- Bragg-ptychograpgy
 - strain dynamics in nano-devices

Other than X-rays

- •SEM (SE, EDS, CL with high spatial resolution)
- •Fly scanning



- •Nanomotors (optional)
- •Sample environment -heat, electric, L-He (optional)

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Optical Layout

Expected Beamline performance

- Energy range : 4 15 kev
- Photon flux : 10¹⁰~10¹¹ photons/sec
- Energy resolution : $< 2 \times 10^{-4}$ with Si(111) crystals
- Beam size : ~ 40 nm at 10 keV (H × V, FWHM)

Screen 4

Slits 5

HFM 2

Slits 6

- → High-order harmonic contamination : $\leq 1 \times 10^{-3}$
- Energy scanning capabilities.
- ✤ Vacuum environment 1x10⁻⁶ torr

Screen 5

Focus

Nested K-B

(Montel) mirrors

Features

Undulator

- ≻2-stage Horizontal focusing
- ➤Horizontal DCM
- ≻Short in length (<70 m)
- ➤Windowless
- ➤Vertically coherent

Montel optics



Slits 3

Slits 4

Slits 2



1. nano-optics: Montel mirrors





No crack at edge is observed by Optical microscope



Delivered in June 2015 by J-Tec

Mirror holders



A1	Х	Optical encoder		
A2	Υ	Optical encoder		
A3	Z	Optical encoder		
A4	Pitch	Laser interferometer		
A5	Roll	Laser interferometer		
A6	Yaw	Laser interferometer		
A7	Тор-Ү	Pre-aligned		
A8	Тор-Z	Pre-aligned		
A9	Top-Pitch (X)	Pre-aligned		
A10	Top-Roll (Y)	Pre-aligned		





Montel Mirrors in the holder

Simulation of Focus Spot

Simulation at 10 keV, average reflection=0.802, by ray tracingSource size $12.5 \ \mu m \ x \ 12.5 \ \mu m$ Source divergence $6 \ \mu rad \ x \ 6 \ \mu rad$ FHWM $25 \ nm \ x \ 25 \ nm,$



Experimental Station





Experimental Station









Sample environment and Preparation Chamber





The Design of the L-He Cooling System



→FEM simulation results



In-Situ Gas and Liquid Cell

























nano-XRF (x-ray fluorescence)

Element-specific nano-imaging





High resolution XRF tomography

Pixel size: 100 nm, sample rotation 0-180° (4.5° / image)



Silversmit et al., Anal. Chem. 81 (2009)

nano-XRD (x-ray Diffraction)

nano-Crystalline









nano-XAFS (x-ray absorption fine structures)

- Local electronic structure
- Local chemical environments
- Element-specific, averaged over nano-area









nano-XEOL (x-ray excited optical luminescence) & CL (cathodoluminescsnce)





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XEOL: 1.nano-area 2.multi-layer, buried layer

Doping species, Band gap, Defect band, Charge transport Adv. Mater. 2014.

DOI: 10.1002/adma.201304345

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ZnO microwire SEM and CL mapping



nm



nano-XRF testing





nano-XRF testing



Nation

nano-XRF testing





Defect (Cu)



Ptychograpgy Testing For strain measurement





Time-Resolved X-ray Excited Optical Luminescence (TR-XEOL)

Photoexcitation dynamics in solution-processed formamidinium lead iodide perovskite thin films for solar cell applications

Hong-Hua Fang¹, Feng Wang², Sampson Adjokatse¹, Ni Zhao², Jacky Even³ and Maria Antonietta Loi¹





www.nature.com/lsa

nano-PXM (projection x-ray microscopy)

– Absorption and phase contrast x-ray images







TRENDS in Cell Biology



Adv. Mater. **2014**, DOI: 10.1002/adma.201304345

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X-ray Bragg projection ptychography from thin film heterostructures



Summary

X-ray methods at 23A of TPS

- nano-XRF (x-ray fluorescence)
 - Element-specific nanoimaging
- nano-XRD (x-ray diffraction)
 - Nano-Crystalline
- nano-XAFS (x-ray absorption fine structures)
 - Local electronic structure
 - Local chemical environments
 - Element-specific, averaged over nano-size area
- nano-XEOL (x-ray excited optical luminescence)
 - X-ray-to-visible down-conversion efficiency in nano phosphor
- nano-PXM (projection x-ray microscopy)
 - Absorption and phase contrast x-ray images
- nano-CXDI (coherent x-ray diffraction imaging)
- Bragg-ptychograpgy
 - strain dynamics in nano-devices







Applications

Solid state physics

- Strongly correlated electron systems YbAl3, YbInCu4, YbCu2Si2 La1-xSrxMnO3 (LSMO)
- Spintronics
- LaVO3, LaAlO3, Fe3-xMxO4(M=Mn, Zn)
- Compound semiconductors InGaZnO, GaCrN, InN, ZnMgO





M. Chu, et al., Annu. Rev. Mater. Res. 39 (2009) 203-229

High-k gate stacks



FIG. 1. Diagram of the W/HfO₂/GeON/Ge stack.



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房價

每間單人住宿價為NT\$700/天,雙人住宿價為NT\$1,000/天;四人房價為NT\$1,500/天。

房型	寶景	間數	房間坪數	收費	備註
二張單人床		30	6	1,000元/晚	單人住宿一律 NT \$700 元
四張單人床		1	12	1,500元/晚	
交誼廳		4	10		一至四樓
餐廳		2	7		三至四樓





March 2014 Physics Today



「科學」期刊(104年1月30 日)

Taiwan unveils new synchrotron

「自然光子學」期刊(104年5 月專文)

'FR **First light for** the TPS

THE HEPTECH NETWORK







「亞太物理學會聯合會刊」 期刊(104年4月號封面)





X-ray Nanoprobe Construction Team

Project Leader: Prof. J. Raynien Kwo (NTHU)

Construction Team

Leader

Mau-Tsu Tang Beamline-



Shih-Hung Chang and Beamline Group Endstation-

Gung-Chian Yin, optical design and overall system integration.

Bo-Yi Chen, Mechanical engineer.

Chien-Yu Lee, Electronic engineer.

Huang-Yeh Chen, Mechanical design and experiment assistant.

Jian-Xing Wu, Software programmer, GUI and control panel design.

Beamline Scientist and Manager-

Shao-Chin Tseng, Sample preparation, experiment design Bi-Hsuan Lin, Experiment design, XEOL, XRD.















Thanks for your attentions