TAIWAN Photon Source

X-ray Nanoprobe Beamline for Nanoscale Solid State Physics Research

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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)

Next??

2015 Cheiron School



Generations of Synchrotron Light Source

1st generation: parasitic 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

National Synchrotron Radiation Research Center





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





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

 $KE = hv - BE - \phi$

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

 $\Delta m_1 = 0$ (linearly polarized)

 $\Delta m_1 = +1$ (L. circularly polarized) $\Delta m_l = -1$ (**R. circularly polarized**)

Energy Distribution Curve (EDC)



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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.



La05Sr15MnO4

3y²- r²

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



Energy Science











structure ←→ electrochemical properties of electrode



develop novel electrode materials.

Biological structure: protein crystallography

















TLS Experimental Hall





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Parallel Imaging for Chemical State Mapping (PICSM)





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



x *

* *









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.





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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



2011-09-27

2010-02-10

1



2010-05-21

2010-08-04

2011-02-05









2013-01-16



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Civil Construction of TPS







Taiwan Photon Source (TPS)









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

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)

Slits 5

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

Features

- ▶2-stage Horizontal focusing
- ➢ Horizontal DCM
- \succ Short in length (<70 m)
- Windowless

Montel optics

 \succ Vertically coherent

Undulator



Nested K-B (Montel) mirrors

Slits 6

Focus



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




















X-ray Methods:

With tens-nm resolution (incoherent)

- 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

Beyond sub-ten-nm resolution (coherent)

•nano-CXDI (coherent X-ray diffraction imaging)

- Bragg-ptychograpgy
 - strain dynamics in nano-devices



TAIWAN TPS 23A

Beamline specification

- Energy range : 4 15 keV
- ♦ Photon flux : 10¹⁰ ~ 10¹¹ photons/sec
- Energy resolution : < 2×10⁻⁴ with Si(111) crystals
- Beam size : \sim 50 nm at 10 keV
- High-order harmonic contamination : ≦1 × 10⁻⁴
- Energy scanning capabilities.

Other than X-rays

SEM (SE, EDS, CL with high spatial resolution)
Fly scanning
In-Situ –electrical, gas, liquid

nano-XRF (X-ray fluorescence)

Element-specific nano-imaging

Pink beam mode at 12 KeV

Doping of ZnO NWs by transition metal Co. \blacktriangleright Pixel size : 25 x 25 nm² Application : Spintronic device Accumulation time : 0.5 sec/point Want to know: beam size : $60 \times 60 \text{ nm}^2$ (V x H) \geq Distribution Short structure order Doping method: • Elemental composition Ion implantation and thermal annealing (b Elemental map of XRF (C) SEM (d) (g) (f) Vapour-liguid-solid process Nano Lett. 2011, 11, 5322–5326



High resolution XRF tomography

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



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

X-ray Nano Probe for Plant of heavy metal residue







nano-XRF for Semiconductor industry





nano-XAFS (x-ray absorption fine structures)

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



X-ray Nano Probe for Lithium Battery (NTU-CCMS)







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





家同步輻射



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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In-situ Electrical Sample Holder





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Sn Blech Structure



Real-time X-ray Nanoprobe Study of Sn Whisker Growth







X-ray Fluorescence Mappings



- Low-temperature (50 °C) electromigration in micro joints was investigated (exsitu) using nano-XRF microscopy (beamline 23A, TPS).
- Significant electromigration of Cu in the Sn matrix and formation of Sn extrusions at the anode were well characterized using nano-XRF microscopy.



X-ray beam–induced current (XBIC): A focused synchrotron x-ray nanobeam over a single semiconductor nanowire heterostructure and simultaneously measured the current through the device and the emitted characteristic x-rays as a function of the incoming hard x-ray energy. With these results, it is possible to identify the compositional and molecular structure as well as localize the electrical fields present under typical working conditions. This information allows us to draw an energy band diagram consistent with the elemental distribution and a high-resolution chemical speciation map.



In-Situ Gas and Liquid Cell













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

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
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 - strain dynamics in nano-devices









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	L.	1	12	1,500元/晚	
交誼廳		4	10		一至四樓
餐廳		2	7		三至四樓



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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