



# Luz de Radiación Sincrotrón, su uso y algo más

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Seminarios Institucionales Facultad de Ciencias en Física y Matemáticas UNACH 28 de febrero de 2019

## Los aceleradores de partículas en el mundo



CICLOTRON, BEVATRON, SINCROTRON, LINAC,... XFEL

AREAS DE LA MATERIAL											
Física del estado solido	Ciencia de los materiales	Física Atómica	Física molecular	Química	Foto-química	Física nuclear	Estructura biomolecular	Biología celular	Geología	Fabricación industrial	TÉCNICAS UTILIZADAS
Х	Х	Х	Х		Х		х		Х		Espectroscopia absorción/reflexión
Х	Х	Х	Х		Х		х		Х		Espectroscopia de emisión
Х	Х			Х				Х			Espectromicroscopia
Х	Х	Х	Х	Х	Х		х		Х		Espectroscopia de foto-electrones
Х	Х	Х	Х	Х	Х				Х		Espectroscopia de fotoiones
Х	Х	Х	Х	Х	Х		Х	Х	Х		EXAFS, XANES
Х	Х		Х								Holografía
Х	Х		Х								Difracción de foto-electrones
Х	Х		Х								Desorción fotoestimulada
Х	Х		Х	Х				Х			Dicroísmo circular
Х	Х	Х	Х	Х	Х		х	Х	Х		Análisis por fluorescencia de RX
Х		Х	Х	Х							Fotones inelásticamente dispersados
Х	Х			Х	Х		х		Х		Difracción/dispersión de RX
Х	Х			Х							Ondas estacionarias de RX
	Х			Х	Х		х		Х		Dispersión Difusa de RX
	Х			Х	Х		х		Х		Dispersión de RX de ángulo rasante
							х	Х	Х		Microscopia de rayos X suaves
	Х									Х	Litografía de RX
									Х		Tomografía
				х				Х			Microtomografía
Х	Х	Х	Х				х				Interferometría de RX
								Х	х		Imageología de RX
					Х					Х	Procesamiento asistido por fotones
										Х	Microfabricación
						Х					Dispersión inversa de Compton

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## La historia

Г

1895	Lenard. Electron scattering on gases (Nobel Prize).	< 100 keV electrons.			
19 <mark>1</mark> 3	Franck and Hertz excited electron shells by electron bombardment.	w misnurst-type machines.			
1906	Rutherford bombards mica sheet with natural alphas and develops the theory of atomic scattering.	Natural alpha particles of			
1911	Rutherford publishes theory of atomic structure.	several file v			
1919	Rutherford induces a nuclear reaction with natural alphas.				
	Rutherford believes he needs a source of many M the nucleus. This is far beyond the electrostatic ma	MeV to continue research on achines then existing, but			
1928	Gamov predicts tunnelling and perhaps 500 k	eV would suffice			
1928	Cockcroft & Walton start designing an 800 k Rutherford.	vV generator encouraged by			
1932	Generator reaches 700 kV and Cockcroft & Walton 400 keV protons. They received the Nobel Prize in	split lithium atom with only n 1951.			

## El ciclotrón (~1930's)



https://www.researchgate.net/figure/The-4-inch-cyclotronvacuum-chamber-showing-the-single-dee-andelectrostatic\_fig2\_242117937



http://www2.lbl.gov/Science-Articles/Archive/rev-idea.html

# EL CICLOTRÓN



## **CICLOTRONES**



"Los sincrotrones son descendientes de otro tipo de colicionadores circulares llamados ciclotrones. Los ciclotrones aceleran particulas e un patron esperial, empezando desde su centro."

Los ciclotrones son frecuentemente utilizados para crear grandes cantidades de un tipo especifico de particulas, tales como muones o neutrons. También son populares en investigaciones medicas porque tiene el rango de energía e intensidad que producen isotopos medicos. 7

## Bevatrón



El **Bevatrón** o **Bevatron** (atom smasher) fue un acelerador de partículas -concretamente, un sincrotrón de focalización débil- del Lawrence Berkeley National Laboratory que comenzó a operar en 1954.

En los años siguientes al descubrimiento del antiprotón (1955), se efectuaron numerosos trabajos pioneros usando haces de protones extraídos del acelerador, haciéndolos colisionar contra blancos en los que se generaban haces secundarios de partículas elementales, no solamente de protones sino también de neutrones, piones, "partículas extrañas" y otras muchas

## SINCROTRONES



"Los sincrotrones son aceleradores de particulas al mas alto nivel de energía, en el mundo. El gran colicionador de hadrones es el que se encuentra en lo mas alto de la lista, con su abilidad de acelerar particulas a una energia de 6.5 trillions de electronvolts antes que colicionen con particulas de igual energía, viajando en dirección opuesta. "

## Sincrotrones



https://es.wikipedia.org/wiki/Sincrotr%C3%B3n



https://www.gleeble.com/products/specialty-systems/gleeble-synchrotron.html

https://www.slideserve.com/rafael-curry/e-matias-canadian-light-source-university-of-saskatchewan



http://www.everystockphoto.com/photo.php?imageId=21126715



## Acelerador Lineal (LINACS)



"For physics experiments or applications that require a steady, intense beam of particles, linear accelerators are a favored design. SLAC National Accelerator Laboratory hosts the longest linac in the world, which measures 2 miles long and at one point could accelerate particles up to 50 billion electronvolts."

## LINAC



https://portal.slac.stanford.edu/sites/lcls\_public/headlines/Pages/Headlines-Vol3No2.aspx



https://www.researchgate.net/figure/Part-of-the-beam-line-in-the-Stanford-Linear-Accelerator-Centre-SLAC-with-Linac\_fig3\_292748244

## Diseñando ATLAS para el HLC





## HLC



## **EL SINCROTRON**

## El sincrotrón



https://www.youtube.com/watch?v=URZYBwWRqA8

## El sincrotrón El espectro de energías y el estudio de la materia



## X-rays can probe smaller objects

Dr. Johanna Nelson Weker

In 1920s X-rays placed in the Electromagnetic spectrum



- Shorter wavelength than visible light
  - Probe smaller objects
- Higher energy than visible light



SLAC

## ¿Que tanta energía tenemos?



https://als.lbl.gov/about/about-the-als/



#### http://photon-

science.desy.de/research/students teaching/sr a nd fel basics/fel basics/tdr spectral characteristi cs/index eng.html

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Energy [eV]

## La generación de la luz sincrotrónica



- 1. Linac (60% or 0.6c)
- 2. Booster Synchrotron (99.999994%)
- 3. Storage Ring,
- 4. Undulators and Wigglers
- 5. Beamline,
- 6. Experiment Station,
- 7. RF System

#### ¿Tenemos haz?



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## Técnicas básicas

#### **Espectroscopía**

Es utilizada para estudiar las energías de las partículas que son emitidas o absorbidas por muestras que son expuestas al haz de luz y es comunmente utilizada para determiner las caracteristicas de los enlaces molegulares y el movimiento de electrones.

#### Microscópía/imageología

Estas técnicas utilizan el haz de luz para obtener imagines con resolución especial muy fina, de las muestras bajo estudio y son usadas en diversas areas de investigación, tales como la biologia cellular, litografía, microscopia infraroja, radiología y tomografía de rayos-X.

#### **Dispersión/Difracción**

Estas técnicas utilizan los patrones de luz producidos cuando los rayos-X son deflectados por la red de atomos estrechamente espaciada en los solidos y tambien para determiner la estructura de de cristales y moleculas grandes, tales como proteinas.

## Que podemos hacer y como?

- Explorar las propiedades de los materiales
- Analizar muestras por trazas de elementos
- Demostrar la estructura de atomos y moleculas
- Estudio de especimenes biologicos
- Investigar reacciones químicas
- Crear accesorios y maquinas.



10.32

11.32

## Categorías por programas científicos

Beamlines are organized into six scientific programs, based on the research techniques they offer.







https://www.bnl.gov/ps/



Complex Scattering

Diffraction & In-Situ Scattering





Soft X-ray Scattering & Spectroscopy



Structural Biology

#### **Program Beamlines**



#### Soft Inelastic X-ray Scattering

The SIX beamline enables researchers to study electron correlation and structure in complex materials with ultrahigh energy resolution and sensitivity. Researchers use

SIX to investigate high-temperature superconductors, topological insulators, and emergent phenomena in novel materials, and to understand the underlying physics of these materials and their potential applications in energy science.



21-ID-2

ESM

#### X-ray Fluoresence Electron Spectro-Microscopy 1, 2

The ESM beamlines offer two versatile experimental stations for spectroscopic and microscopic investigations of novel materials. Using ESM's high energy resolution, small spot size, and wide range of photon energies, researchers can uncover the fundamental physics and chemistry of newly synthesized materials and incredibly small crystals with high precision.

Frontier Synchrotron Infrared Spectroscopy



structure and behavior of materials under extreme conditions. FIS offers researchers the possibility to mimic the temperature and pressure found deep inside of

planets, and to investigate the properties and reactions of materials and condensed matter in these special environments using infrared electronic and vibrational spectroscopy.



#### Magnetospectroscopy, Ellipsometry and Time-resolved Optical Spectroscopies

The MET beamline is a dedicated and versatile spectroscopy tool for studies on condensed matter under diverse experimental conditions, including low

temperatures and high magnetic fields. Using the beamline's advanced capabilities, researchers can study the electronic structures and emergent phenomena of novel materials, including multiferroics, topological insulators, and high-temperature and conventional superconductors.



IOS

#### Coherent Soft X-ray Scattering

The CSX beamline offers researchers state-of-the-art soft xray scattering and imaging tools with world-leading, coherent, and high photon flux for investigating the electronic texture and dynamics of composite materials.

This unique combination of spectroscopic, microscopic, and imaging tools enables researchers to explore the correlation between electronic behavior and emergent phenomena in novel materials.

#### In situ and Operando Soft X-ray 23-IR-2 Spectroscopy.

The IOS beamline offers researchers specialized tools for in situ and operando spectroscopy on heterogeneous

catalysis and other energy systems. Researchers can study complex chemistry and energy conversion under ambient and elevated pressure of various gases. By offering this new capability, IOS bridges the long-standing pressure gap problem in catalysis between ideal surface science experiments and industrial catalytic processes.



## ¿Como identifico las energías-materia?

#### **X-RAY DATA BOOKLET**

Center for X-ray Optics and Advanced Light Source

Lawrence Berkeley National Laboratory

## Orange book X-ray



http://xdb.lbl.gov/xdb.pdf

## Range to cover: Beamline flux curve



Fig. 1.2 Extended range flux curve of BL 532

http://www2.lbl.gov/Science-Articles/Archive/sabl/2005/August/assets/docs/STXM\_Beamline\_5-3-2\_Manual.pdf

# Spectroscopic Imaging: absorption

## contrast

Electron Binding energies:  $E_{K} > E_{L} > E_{M}$ 



**Binding energy** = energy required to kick an electron completely out of the atom (characteristic to the element)

Photons with energy matching an electron binding energy are more likely to be absorbed



# LAS TÉCNICAS

## Full Field – Scanning microscopy Benefits/downsides of each microscopy



#### Scanning

#### Pros

- Low dose
- Florescence or electron detection modes
- Variable field of view ("zoom in")
- Compatible with mirror-based optics

#### Full field

#### Pros

• Fast (3D, dynamics, ...)

#### Cons

- High dose
- Field of view fixed by optic

#### Cons

• Slow

## Microscopy and spectroscopy $\rightarrow$ STXM











## Técnicas hibridas (AFM-FTIR)

https://microscopy-analysis.com/editorials/editorial-listings/afm-synchrotron-light-probes-molecular-chemistry

## AFM-synchrotron light probes molecular chemistry





## Microscopia y tomografía InfraRojo

MS1

MS2

MS3 Jug 45

45

1 15 2 25 3 35 ×10-3

microns

15

45

microns

05 1 15 2 25 3 35 ×10<sup>-3</sup>

B





microns

15 2 25 3 35 ×10-3 microns

15 2 25

Optical path normalized image of different microspheres (MS1–MS3). (A) Carbonyl ester bond at 1750 cm<sup>-1</sup> of PLGA; (B) CH<sub>2</sub> wagging at 1450 cm<sup>-1</sup> of PLGA; (C) amide I bond at 1656 cm<sup>-1</sup>; (D) amide II bond at 1545 cm<sup>-1</sup>.

https://doi.org/10.1016/j.apsb.2015.03.008

## ARPES

#### spatially-resolved Angle-Resolved Photoemission Spectroscopy

MAESTRO, the Microscopic and Electronic STRucture Observatory (ALS)

Cuando los fotones a una energía definida inciden sobre muestra. permiten una la medición de la energía cinetica ángulo el de salida, V información brindando así sobre el momento y el estado de la energía del electron en el material.









## "tipos" de ARPES

			Resolu	ution	Temperature Range	Data Quality		
			Spatial Energy			best aspect	worst aspect	
ning Probe		μARPES	5 μm x 10 μm (H xV)	3 meV (nominal)	I 5-2300K	energy + k resolution, speed, LT	spatial resolution	
Scanr		nARPES	l 20 nm (50 nm to be added)	> 10 meV	"LT" (to be added)	spatial resolution	lack of speed	
field	PEEM	Imaging (real space)	35 nm	2.5 eV	100 1200/	speed,	energy + k- resolution	
Full-		ARPES (k-space)	4 µm	0.1 eV	100-1300K	x-ray absorption		

## Cristalografía de proteínas



http://www.labtimes.org/labtimes/method/m ethods/2011\_02.lasso



http://www.esrf.eu/home/UsersAndScience/Publications/Highlights/highlights-2015/structural-biology/sb10.html



https://en.wikipedia.org/wiki/X-ray\_crystallography

## SAXS (dispersión - angular)



Focused monochromatic

X-rays

It can be applied to samples that are either difficult or impossible to crystallise, may be complex or composite systems or materials with large scale self-organisation.

https://www.researchgate.net/figure/Combined-3D-PCCF-spectroscopyand-synchrotron-SAXS-WAXD-set-up\_fig3\_276071127

- Proteins & Biomaterials
- Polymers
- Environmental
- Colloids & Surfactants

1D WAXS 2D SAXS

https://www.diamond.ac.uk/industry/Techniques-Available/Small-Angle-X-ray-Scattering-SAXS.html

Synchrotron Small Angle X-ray Scattering Studies Reveal the Role of Neuronal Protein Tau in Microtubule Bundle Formation with Architectures Mimicking those Found in Neurons



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# SIXS (dispersión – superficie)

SixS (Surface Interface X-ray Scattering) is a beamline dedicated to the study of X-ray scattering from surfaces and interfaces of hard and soft matter in various environments in the 5-20 keV energy range. To be sensitive to the surface all the studies will be performed in grazing-incidence geometry.

dedicated to structural studies of interfaces (gassolid, solid-solid or solidliquid), as well as nanoobjects.



## 2D imaging can be misleading!<sub>SLAC</sub>



## Microtomografia (µCT)



# Microtomografia (µCT)



Geoderma, Volume 287, 1 February 2017, Pages 31-39 Soil pores and their contributions to soil carbon processes

Examples of <u>X-ray</u>  $\mu$ -CT images of (a) pieces of <u>particulate organic matter</u> (green) and <u>pores</u> (blue) identified within a soil macro-aggregate(6  $\mu$ m resolution); and b) water (blue) filled pores within solid matrix (brown) of soil sample (2  $\mu$ m resolution).

https://aip.scitation.org/doi/pdf/10.1 063/1.4952925

a) Schematic layout of the three-point bender inside the hot cell. b) 3D view of a notched Gilsocarbon nuclear graphite specimen fractured under load at 1000oC



## Microtomografia (µCT)

Using synchrotron X-ray microtomography to characterize the pore network of reservoir rocks: A case study on carbonates <u>Advances in Water Resources</u>, <u>Volume</u> <u>95</u>, September 2016, Pages 254-263.



Volume <u>renderings</u> of the laboratory sample Bolo5. In (a) the total volume of the sample is shown. We divided the entire volume in 3 portions: <u>Host Rock</u> (HR), Intermediate portion (Inter.) and the Compaction Band (CB) produced in laboratory. The <u>porosity</u> () decreases from HR to CB as shown in (b), (d) and (f). The connectivity also decreases from HR to CB as the size of the main backbone decreases from HR to Inter. to CB.

## Difracción de rayos-X de alta presión





https://als.lbl.gov/learning-from-roman-concrete/ Learning from Roman Seawater Concrete SEPTEMBER 25, 2013

Chin. Phys. B Vol. 25, No. 7 (2016) 076106

Electromagnetic Badiation

## XRF (Fluorescencia)





Synchrotron micro-XRF spectrum of BR and Wits Gold, and pyrite and quartz at 30 keV energy.

In situ XRF/XAS experiments at synchrotron radiation facilities (example: microfocus beamline ID22, European Synchrotron Radiation Facility, Grenoble).

## XRF - XAS - XANES



в 10 XANES Energy (keV) 210 012 2-k conuts 180 С 2.5 1.5 0.5 2.47 2.51 2.46 2.48 2.49 2.5 Photon Energy (KeV)

XRF mapping at Beam Line 10-2 (Figure 2B) revealed the distribution of Zn and K in healthy brain tissue, and brain tissue following ischemic stroke (Silasi et al. 2012). Sulfur K-edge XAS was performed at Beam Line 4-3 (Figure 2B), and revealed increased levels of taurine within grey matter (black line), relative to white matter (dashed line). This trend was only observed in non-fixed flash frozen brain tissue (Hackett et al. 2012). SLAC - STANFORD

Typical combined XRF/XANES analysis of a lymphocyte cell. A) XRF distribution map of S obtained with 3KeV incident beam (1m2 pixel size). B) XRF spectrum of the full map showing the fit for different elements performed using PyMCA [30]. The blue shadowed-box corresponds to the region of interest used to obtain the XRF intensity for S XANES. C) XANES spectrum obtained from the spot marked on A.

https://www-ssrl.slac.stanford.edu/content/science/highlight/2012-09-24/elements-stroke https://www.eurekaselect.com/138399/article

### **Differential phase contrast with segmented detector**

SLAC

#### freeze-dried cardiac myocyte



Now the shadow is diagonal from the top left to bottom right

### How a phase object changes the scattered beam



SLAC

## Quantitative phase gives thickness estimate



5 µm

Holzner, 2010 Doctoral dissertation Dept. of Physics and Astronomy, Stony Brook University

## X-FEL

X-ray Free Electron Laser (XFEL) is an X-ray combining the features of lasers at the free electron state. It may be the most promising light source for the next generation of scientific exploration and discovery.

The X-rays produced at <u>SPring-8 are ten billion</u> <u>times brighter than the sun</u>. However, the peaks and troughs of the light waves are not aligned. Laser light is light with its waves aligned. <u>The light produced by the XFEL will be</u> <u>a billion times brighter than SPring-8</u>. A brighter light will be a major step forward and enable us to observe faster movement in a smaller region.





http://xfel.riken.jp/eng/xfel/index.html





https://www.bbc.com/news/science-environment-41117442

## XFEL

- At the head of the XFEL, bunches of electrons are first sped up to near-light-speed in a super-cold, evacuated accelerator
- The particles are directed down long undulators magnetic systems that produce a slalom course for the electrons
- As they wiggle back and forth in the undulators, the fastmoving electrons emit very bright X-ray flashes
- The particles interact with this great sea of X-rays and begin to organise themselves into even tighter groupings
- This intensifies the brilliance of their emission and gives it coherence the X-rays are "in sync" and laser-like
- Having done their job, the electrons are siphoned off, leaving the X-ray flashes to hit their experimental targets

The machine will deliver trillions (1,000,000,000,000) of X-ray photons in a pulse lasting just 50 femtoseconds (0.000,000,000,000,05 sec), and it can repeat this 27,000 times a second. It allows for time-resolved investigations that are beyond what is possible in standard synchrotrons. For example, scientists will use a jet to stream their samples in front of the beam, priming them with another laser so that chemical reactions are triggered at just the right moment to be caught by the pulses.

"The huge hope for XFEL is that we will be able to do single particle imaging. So, you just put a stream of your protein complex or virus into the beam and you'd have enough photons that an individual biological entity would scatter those photons for you to get the shape of it,"

# CDI ideal for X-ray Free Electron Lasers (XFELS)



#### **Diffract before destroy!**

- For 3D data, many particles must be imaged
- Assumes all particles are identical
- Averages out difference
- Won't work for cells

Gaffney and Chapman, 2007, Science 316 1444

## Mapping strain in battery particle during cycling



SLAC

4 V, 8.167 Å

100 nm

## Bragg CDI at an XFEL: imaging acoustic phonons



Displacement

SLAC

La maps (M<sub>5</sub> edge @ 836 eV) (ALS BL 5.3.2.1)

## STXM vs. ptychography











## **Elemental mapping illuminates Fe contamination**



Resolution estimated  $\leq$  12 nm

 La: marker for La-exchanged ultrastable yttrium (USY) zeolite crystallites

- Distributed throughout
- Particle size 0.1 to 3  $\mu$ m<sup>2</sup>
- Fe: deposited at particle edge
  - Penetrates ~1 μm into particle
  - And outside of particle
  - Small nodules visible



SLAC

A.M. Wise et al. ACS Catal. (2016), 6, 2178-2181

## Ptychography with simultaneous fluorescence microscopy



Deng, et al. PNAS, 112, 8, 2314 (2015).

## Summary of X-ray microscopy SLAC

Scanning microscopy

- 1. Scanning transmission X-ray microscopy (STXM)
  - Fluorescence and electron detection
- 2. Differential phase contrast imaging/dark field



## ¿Y EN MÉXICO?





PROYECTO:

- ✓ MORELOS (2011)
- ✓ HIDALGO (2018)
- REDFAE
- ✤ REDTULS
- UNIVERSIDADES Y CENTROS DE INVESTIGACIÓN

## Diseñando ATLAS para el HLC









## iiGRACIAS!! Dr. Daniel Hernández Cruz dhernandezcruz@gmail.com

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