

 Institut foton

Fonctions Optiques pour les
Technologies de l'informatiON

 PERA

Université
de Rennes

INSA

Optical characterizations of epitaxial materials: from crystal defects to optoelectronic properties

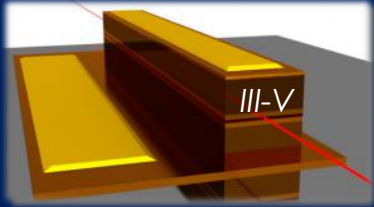
C. Cornet, P. Huillery and Y. Léger.

Univ Rennes, INSA Rennes, CNRS, Institut FOTON – UMR 6082, F-35000 Rennes, France

Introduction : Optical properties of materials

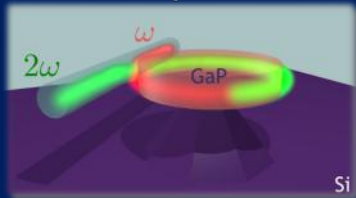
Photonic applications

Active photonics



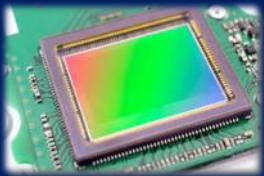
- Telecommunications, computing, medical imaging, defence, surgery, ...

Passive photonics



- Optical communications, on-chip signal processing...

Sensors



- Defense, agriculture, environment, gas detection, health...

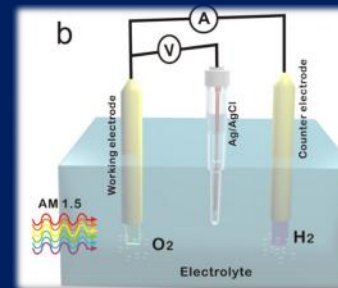
Energy applications

Photovoltaics



- Sustainable low CO₂ emissions energy production, conversion of sunlight into electricity. ...

Solar fuels



- Solar hydrogen production through water splitting, sun-assisted atmospheric carbon conversion into fuel...

Outline

I-Bandstructure of semiconductors, crystal defects and optical processes

- Bandstructures and semiconductors
- Crystal defects and their impact on optoelectronic properties

II-Characterizing light emission properties

- Photoluminescence
- Electro- & Cathodo-luminescence

III-Characterizing light absorption properties

- Absorbance measurements
- Ellipsometry & Photo-current

IV-Toward single photons sources

- micro-photoluminescence, $g(2)$



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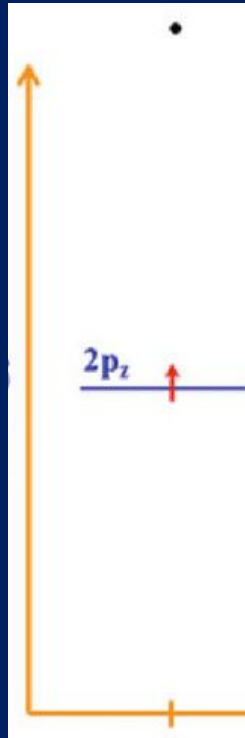


What is a bandstructure ?

→ Linear chain of carbon atoms



Energy



Conduction Band CB (empty)

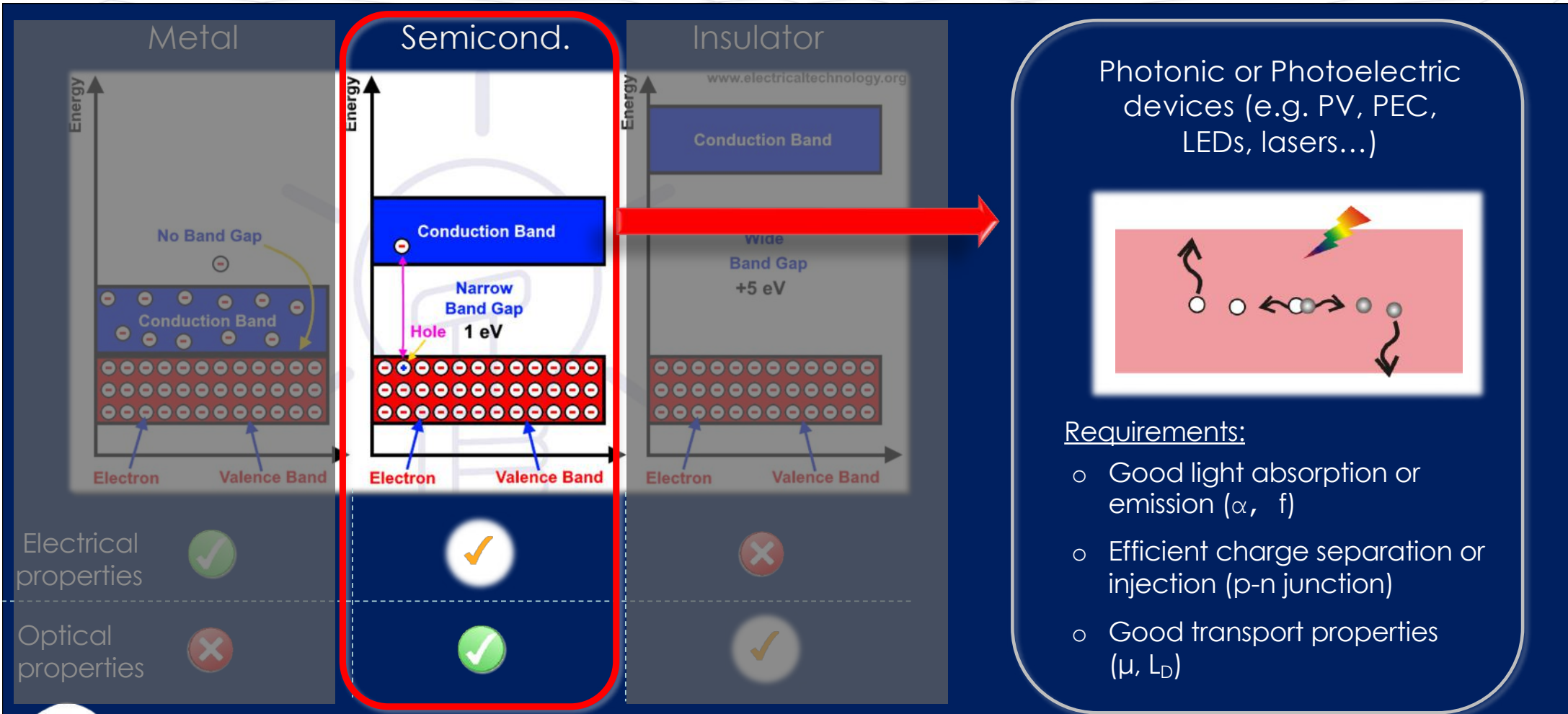
Bandgap

Valence Band VB (filled)

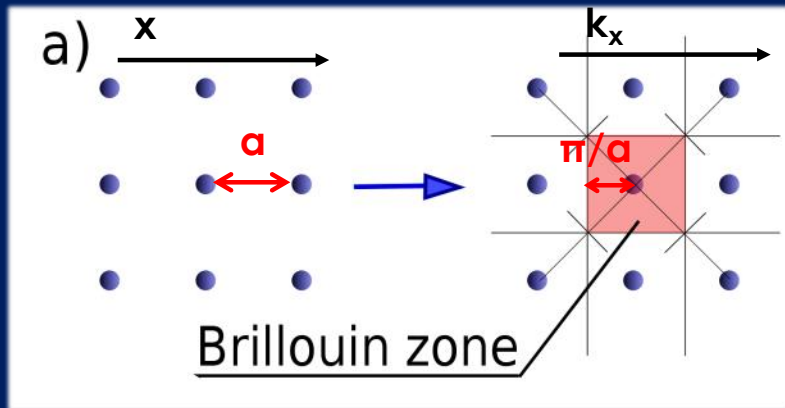
No. of carbon atoms



Band structure classifications



2D square lattice



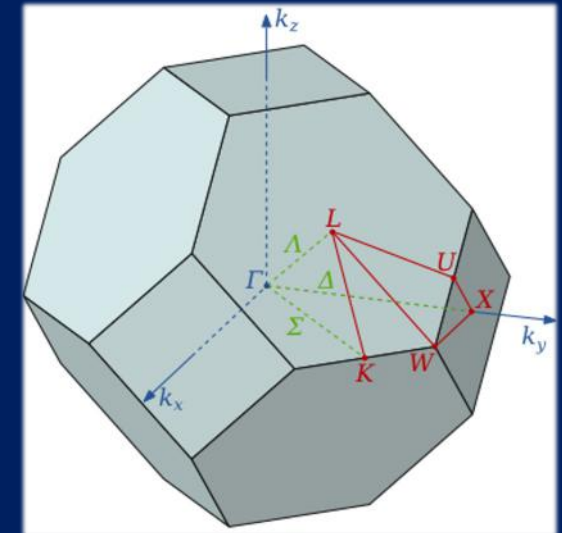
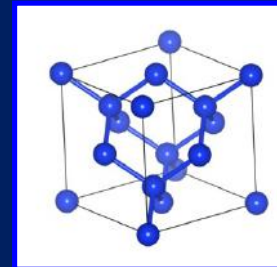
$$\psi(x + na) = \psi(x)$$

$$\psi(x + a) = e^{ika} \psi(x)$$

k = wavevector associated to phases of regular atomic orbitals in the crystal

3D crystal

Silicon : a diamond
FCC structure

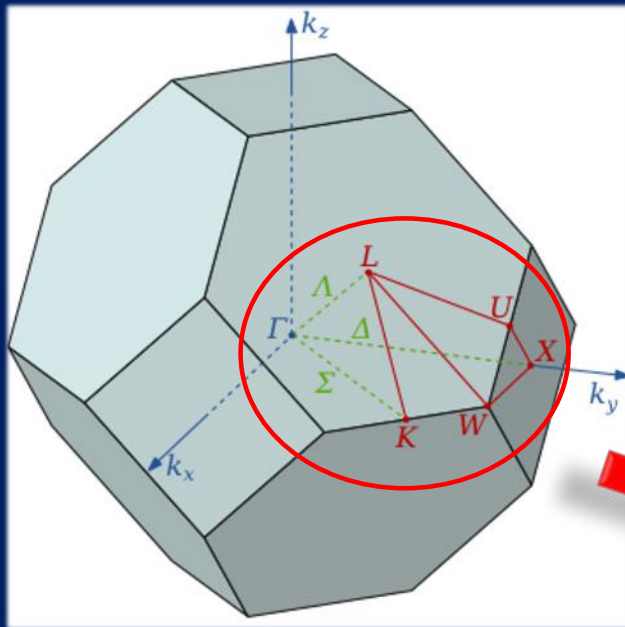


Different planes in the reciprocal space
→ various periodicities of the 3D crystal



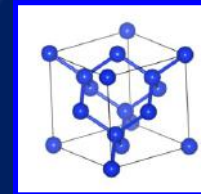
Any change of the lattice ordering, interatomic distance, nature of atoms, will change the properties in k -space and associated Energy levels.

From Brillouin zone ...

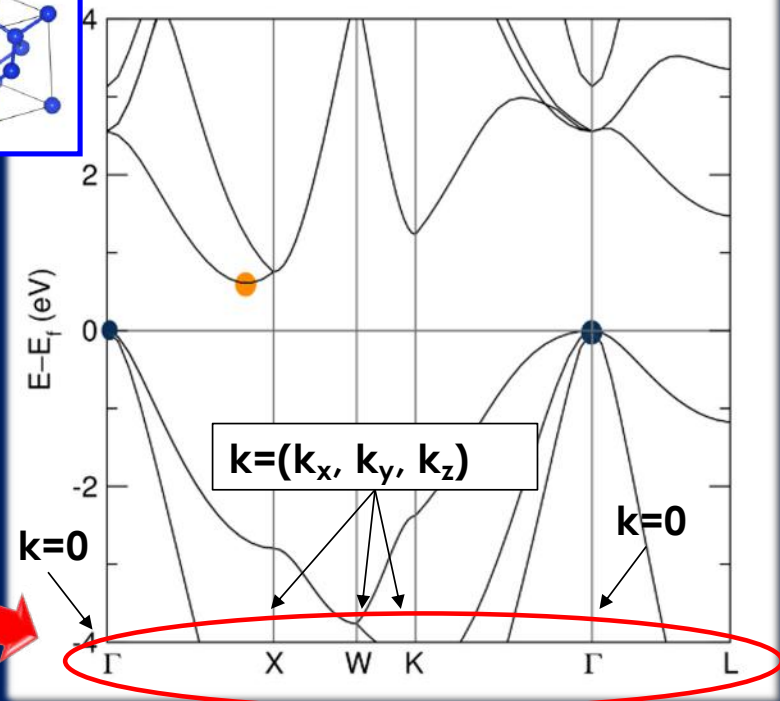


First Brillouin zone of FCC lattice, a truncated octahedron, showing symmetry labels for high symmetry lines and points

...To bandstructure



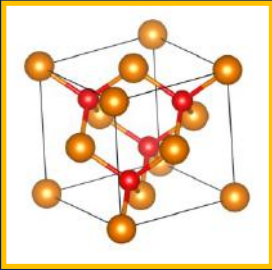
Si



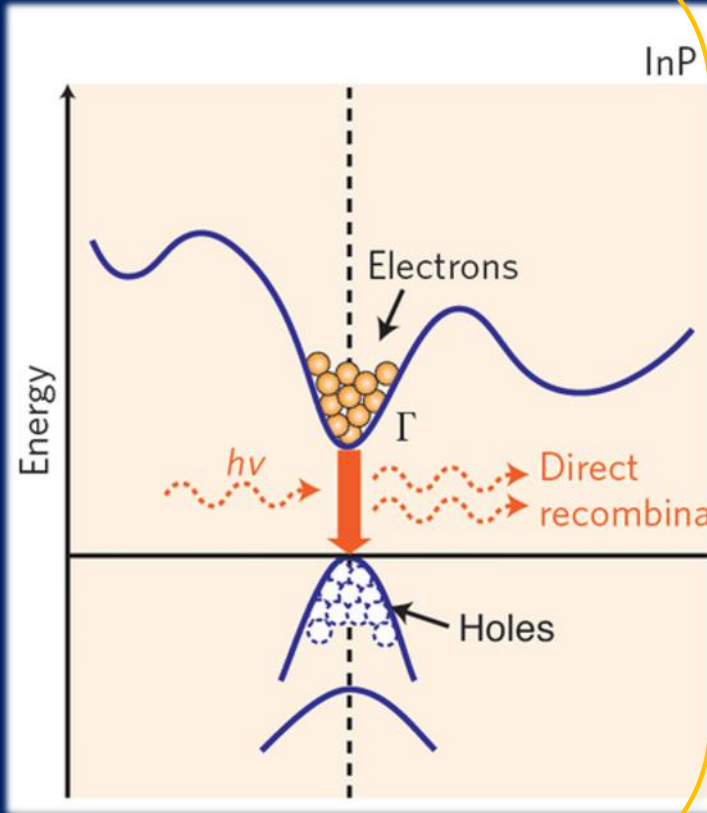
Bandstructures : specific paths in k-space

Direct vs Indirect bandgaps

Zinc-Blende structure

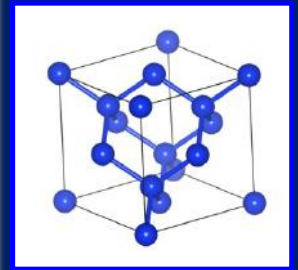


III-V FCC



Direct = optically efficient

Diamond structure



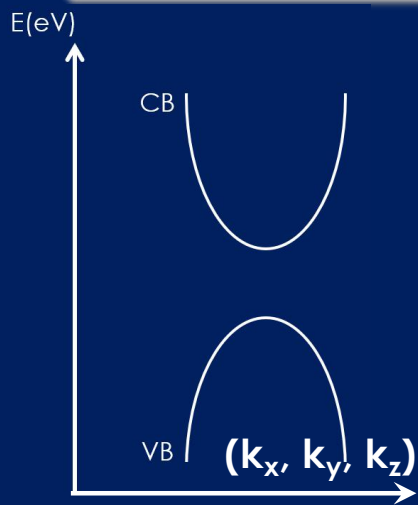
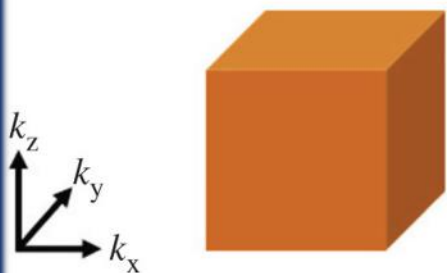
Si FCC

Indirect = optically limited

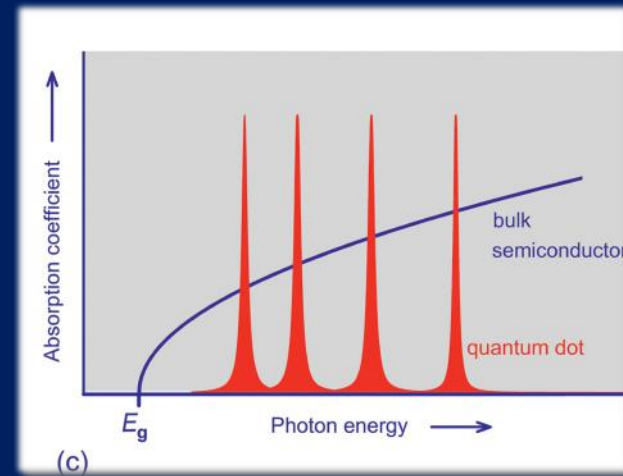
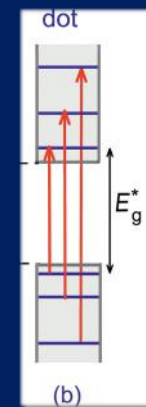
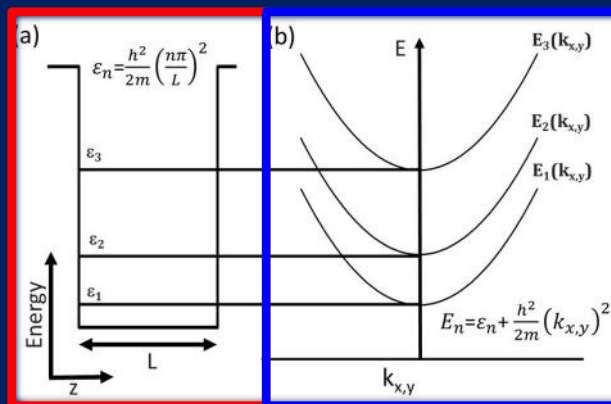


Quantum confinement & dimensionality

no confinement
bulk
(3D materials)



(k_z) (k_x, k_y) (k_x, k_z) (k_y)



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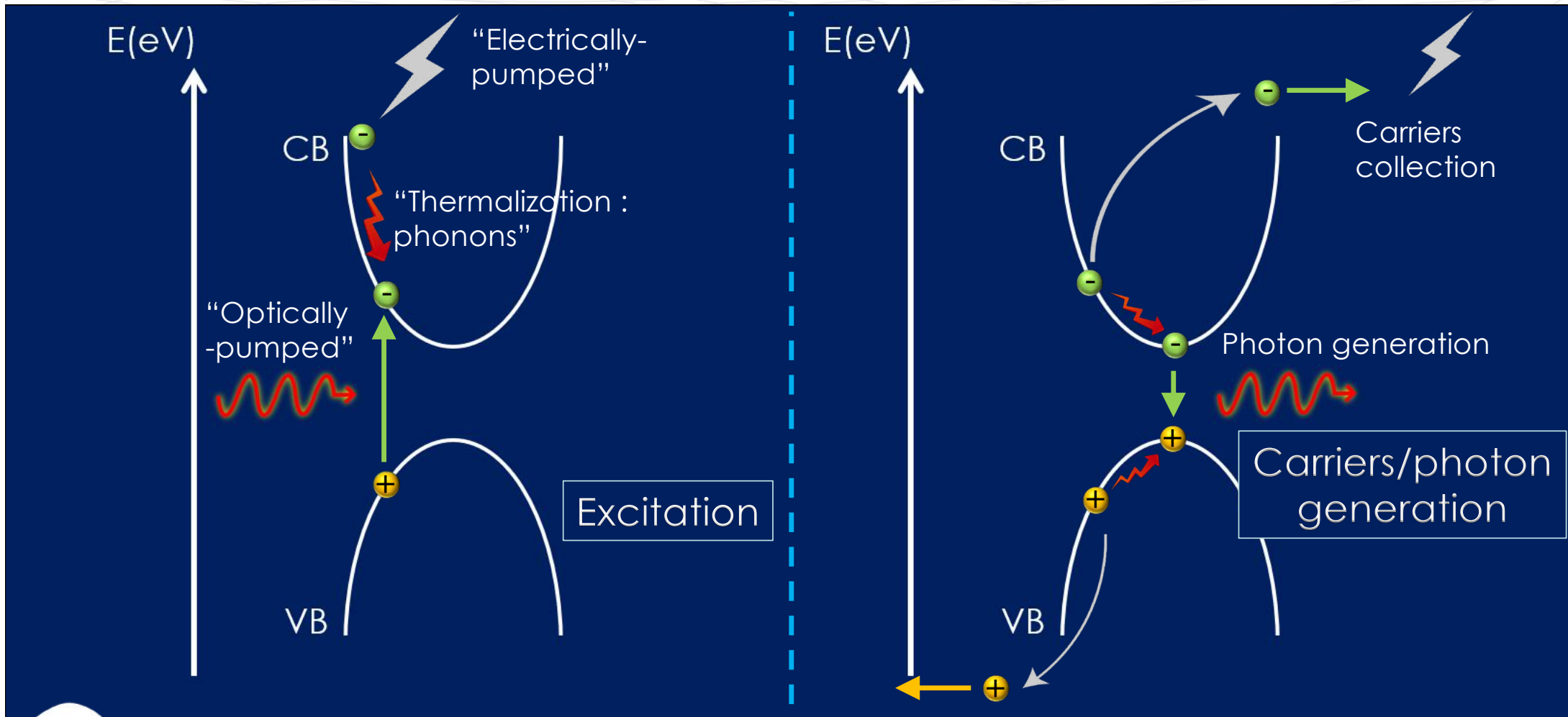
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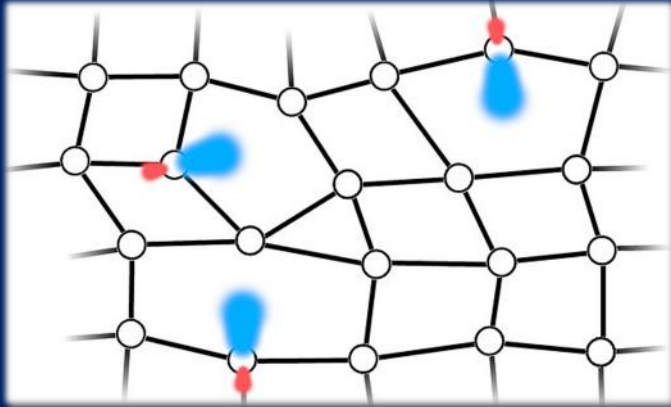
Ideal light-matter interactions in SC



Proposed classification of crystal defects

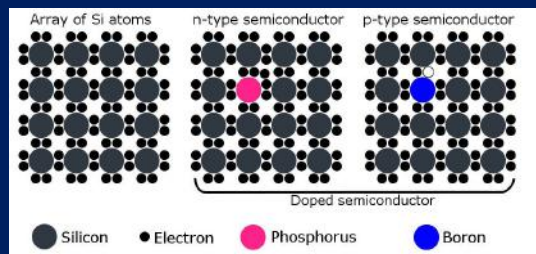
1

A dangling bond in a material is an unsatisfied valence on an immobilized atom



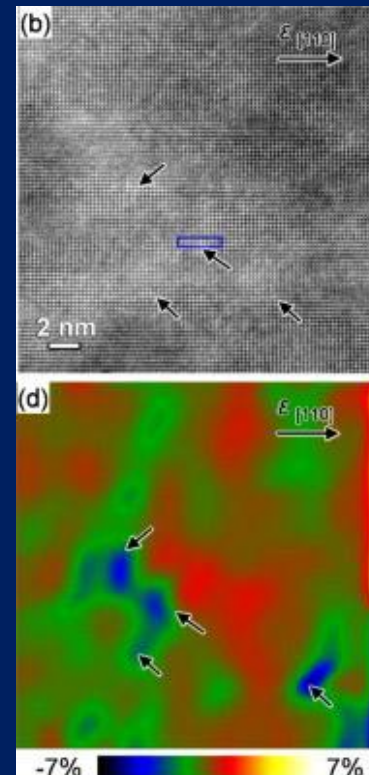
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Insertion of a new atom with a different valence induces charge fluctuations :



2

Local fluctuations of the potential



Fluctuations of the stoichiometry induces fluctuation of the band positions

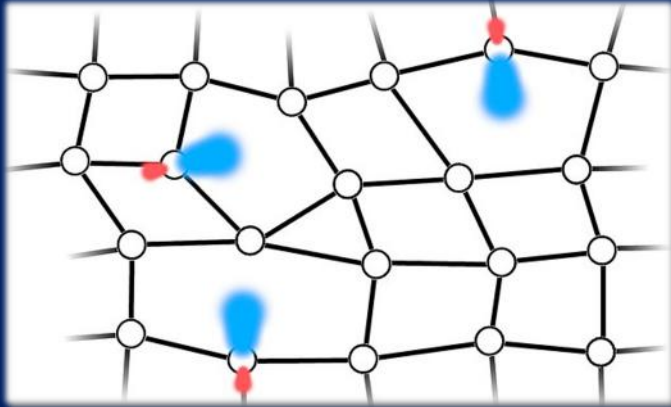


Proposed classification of crystal defects

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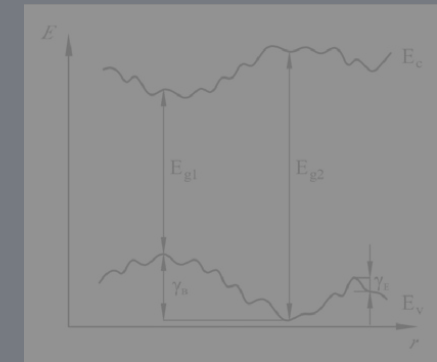
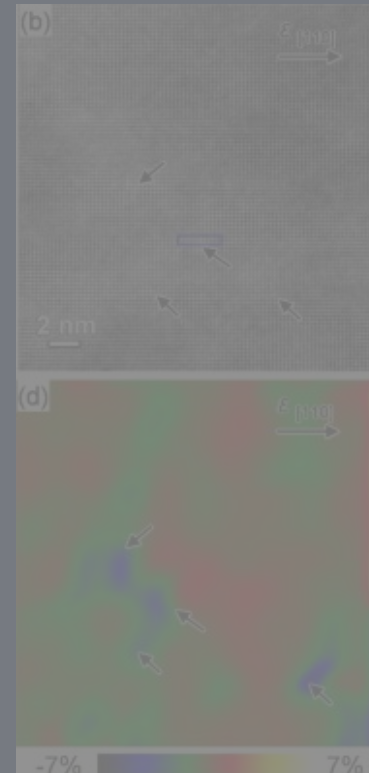
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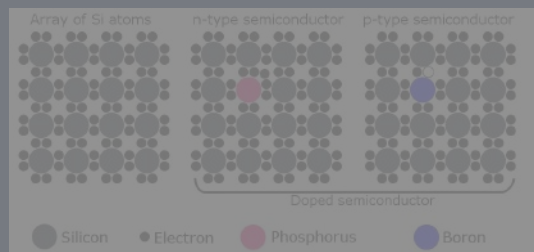
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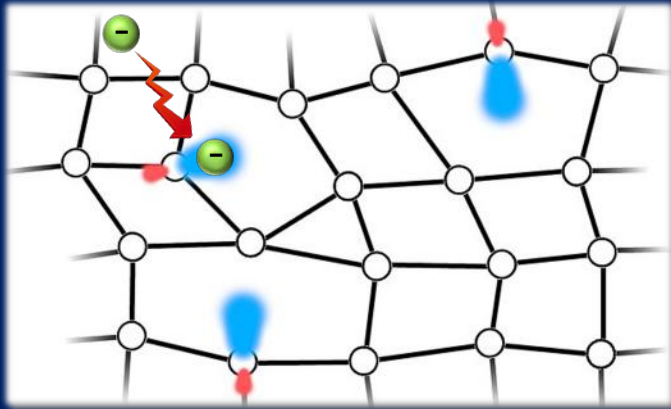
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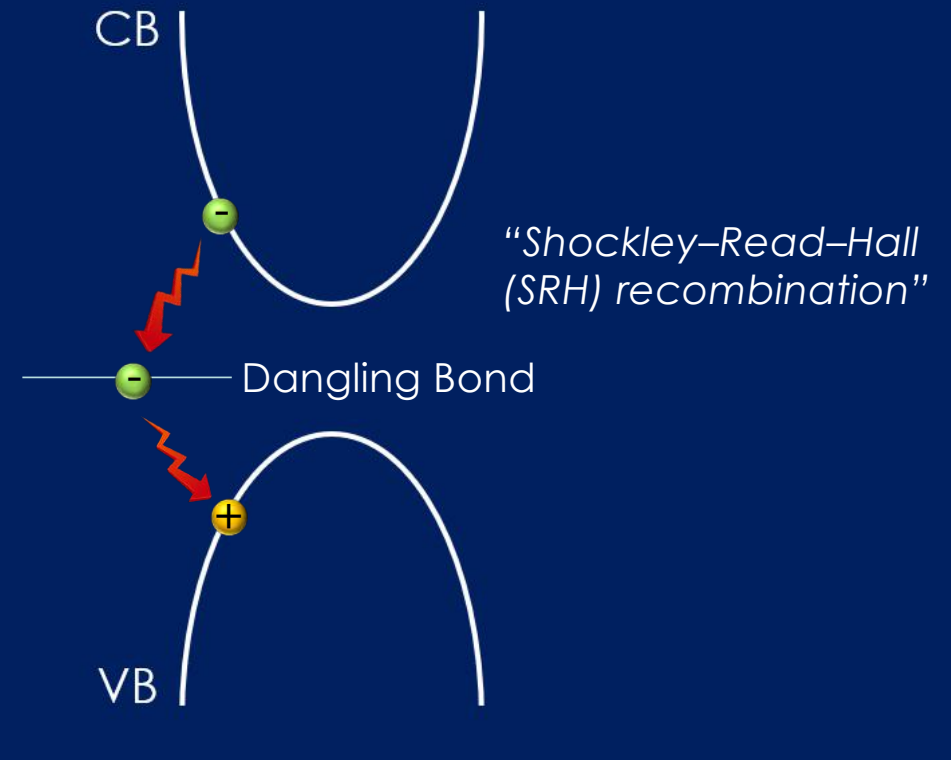
Dangling bonds and optical properties

Trapping of carriers in DBs



→ Dangling bonds will capture carriers in the conduction band, which won't be available for light emission or carriers collection.

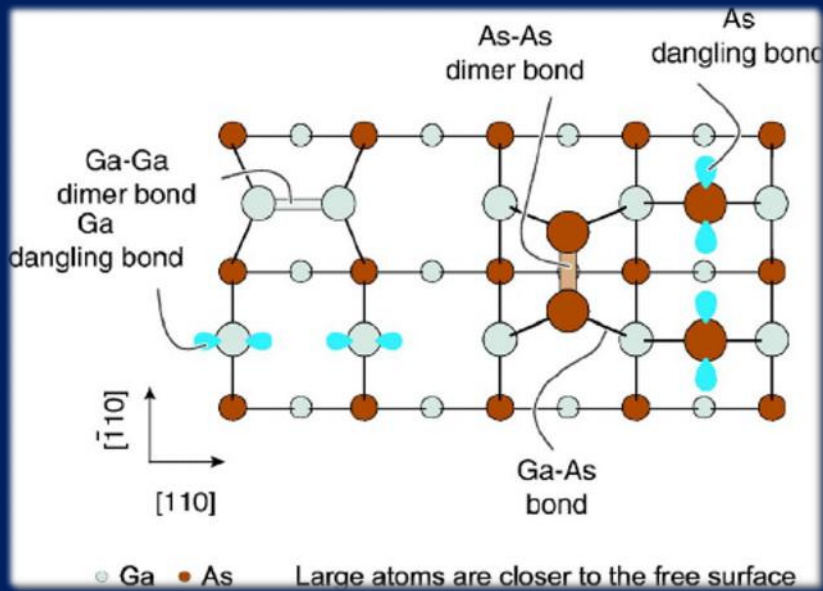
E(eV)



Dangling bonds in surfaces (2D)

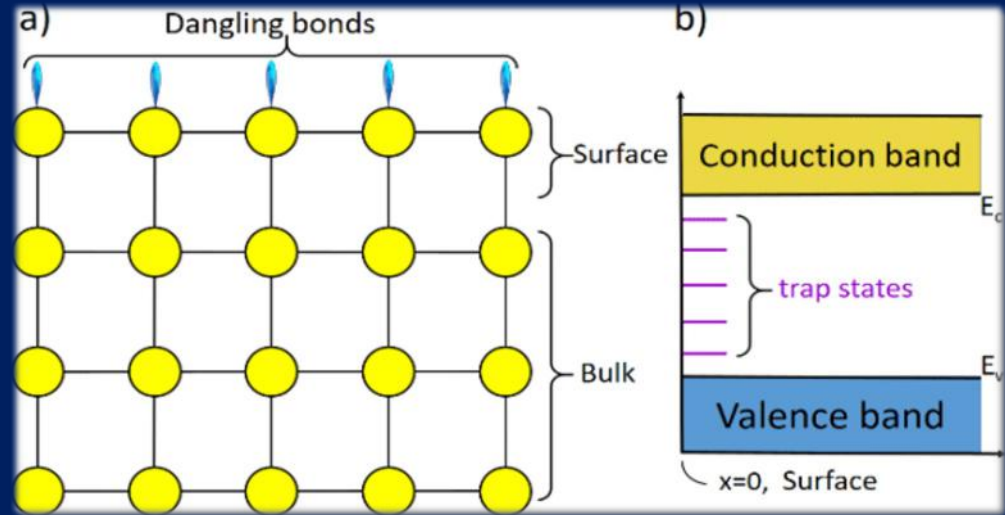
DBs at surfaces

- The free surface of a SC is usually full of Dangling Bonds !



Top view of the GaAs (001) surface.

"Surface states"



Mid band-gap states = surface states

- Decrease of optical efficiency, or carrier collection efficiency
- Surface passivation strategies (ex: Sulfur)

Misfit dislocations & dangling bonds

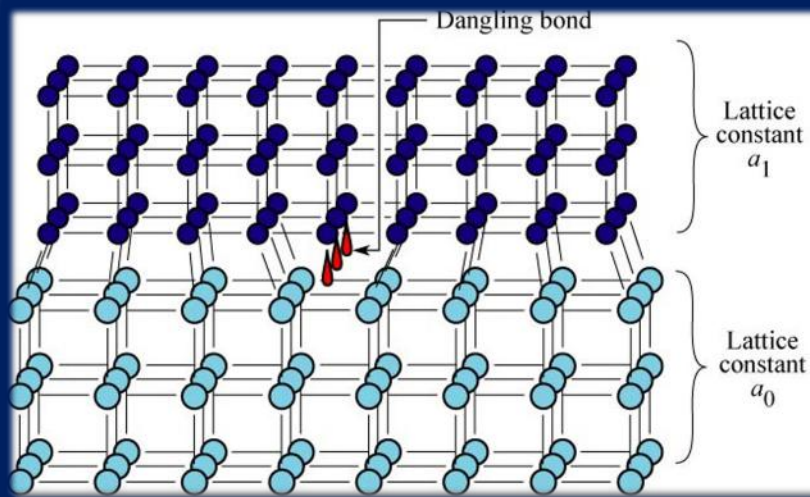
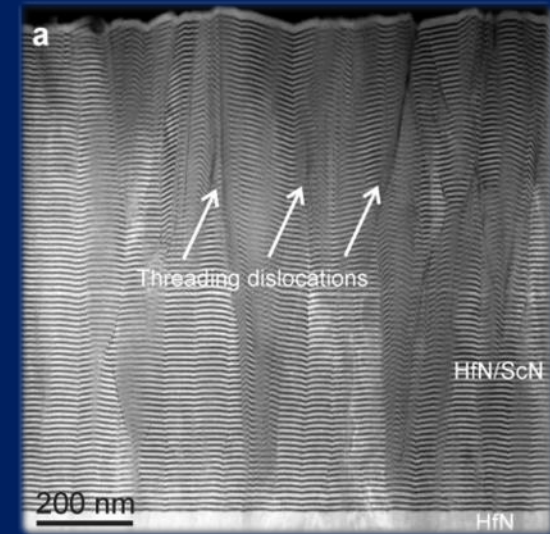


Fig. 7.12. Illustration of two crystals with mismatched lattice constant resulting in dislocations at or near the interface between the two semiconductors.

E. F. Schubert
Light-Emitting Diodes (Cambridge Univ. Press)
www.LightEmittingDiodes.org

The lattice mismatch between different layers will generally lead to misfit dislocations.

Propagation

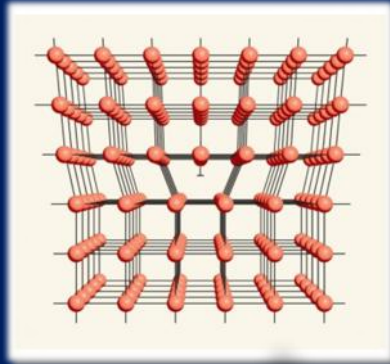


Dislocations density depends on the lattice mismatch, and are known to propagate through the entire sample

➡ Decrease of optical efficiency, or carrier collection efficiency (known as a "device killer")

Dislocations management

Lattice mismatch

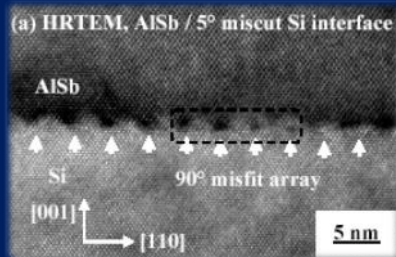


Dislocations : a non-radiative defect that propagates

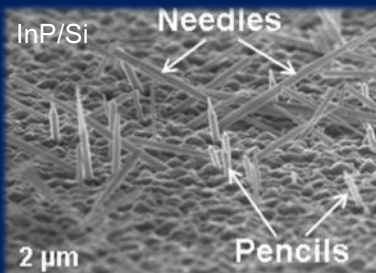
Pseudomorphic approach



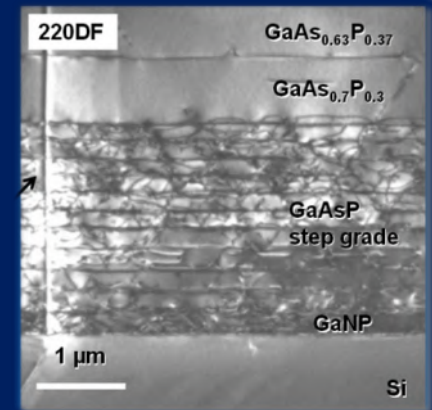
Localized dislocations



Nanowires

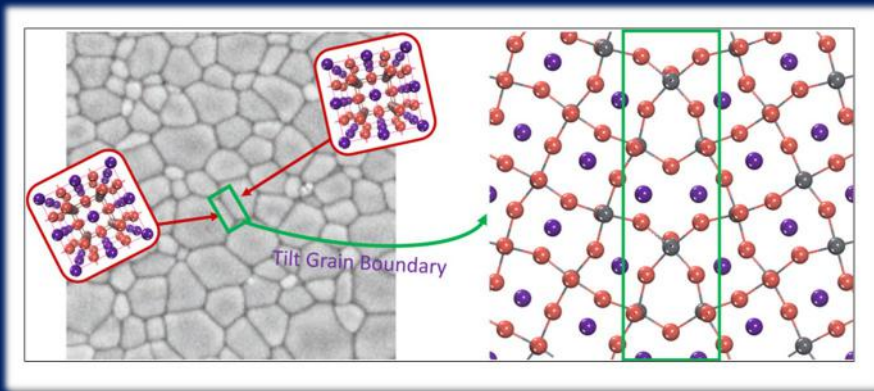


Graded buffers



Grain Boundaries : Epitaxy or not ?

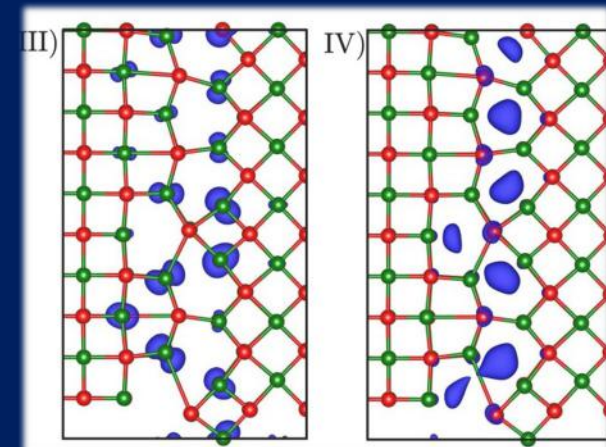
- Polycrystalline materials, or bad quality epitaxial materials can present grain boundaries (originating from the coalescence)



Ex : Perovskites

Grain Boundaries and Dangling bonds

- Grain boundaries are usually full of DBs (trap states)

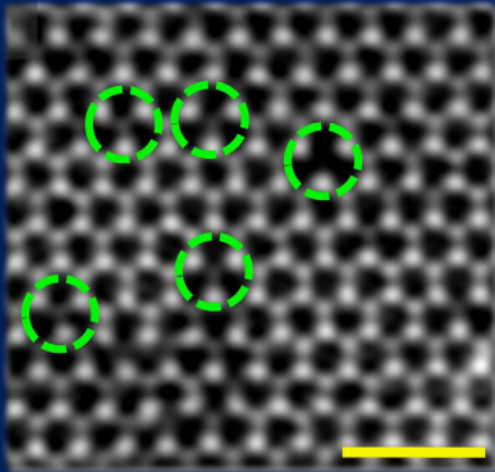


Ex: MgO

➔ Also known as a "device killer", grain boundaries can be counter-passed e.g. in perovskites by potential fluctuations

Vacancies

- Vacancy = a missing atom in the crystal

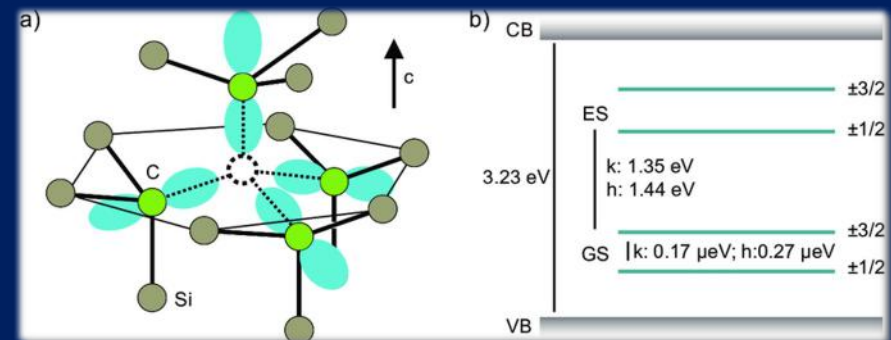


Ex : MoS₂

→detrimental for devices, but depends on the density.

Vacancy : is it really a defect ?

- Vacancy = a 0D nanostructure, with quantized energy levels.



Ex : Si Vacancy in SiC

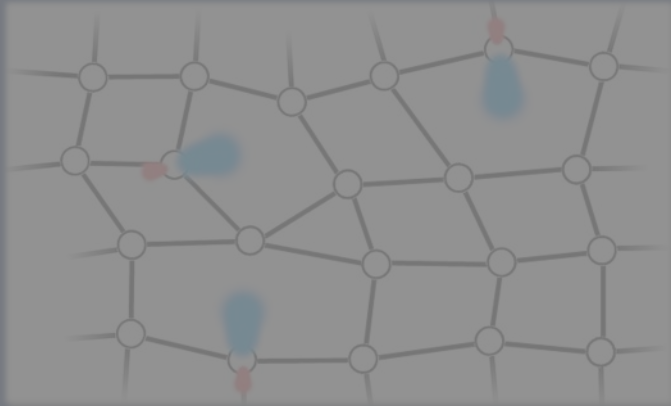


A real potential for quantum technologies ? (N-V centers)

Proposed classification of crystal defects

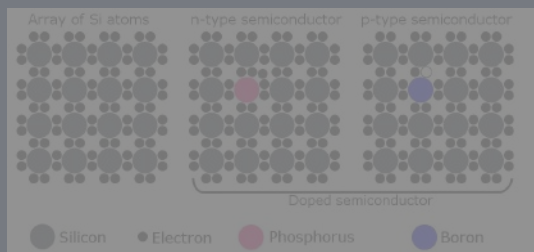
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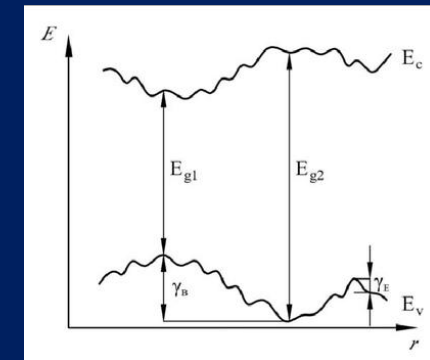
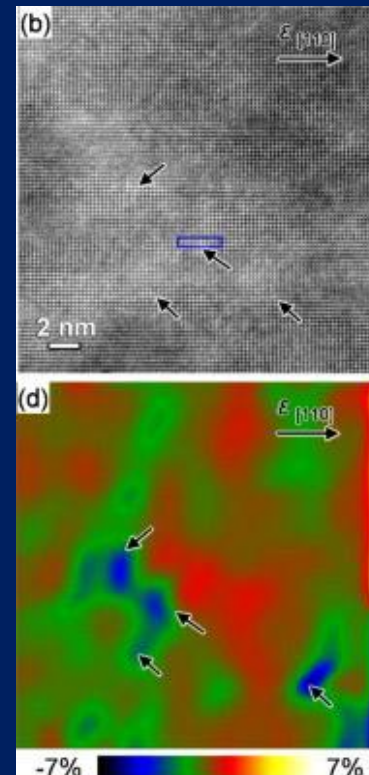
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Insertion of a new atom with a different valence induces charge fluctuations :



Local fluctuations of the potential

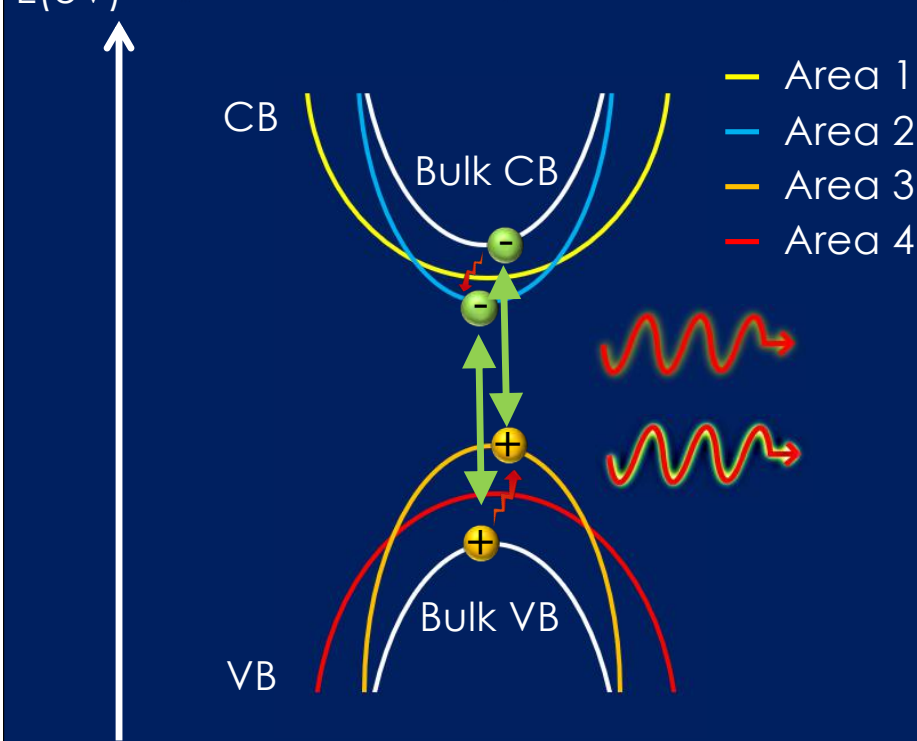
2



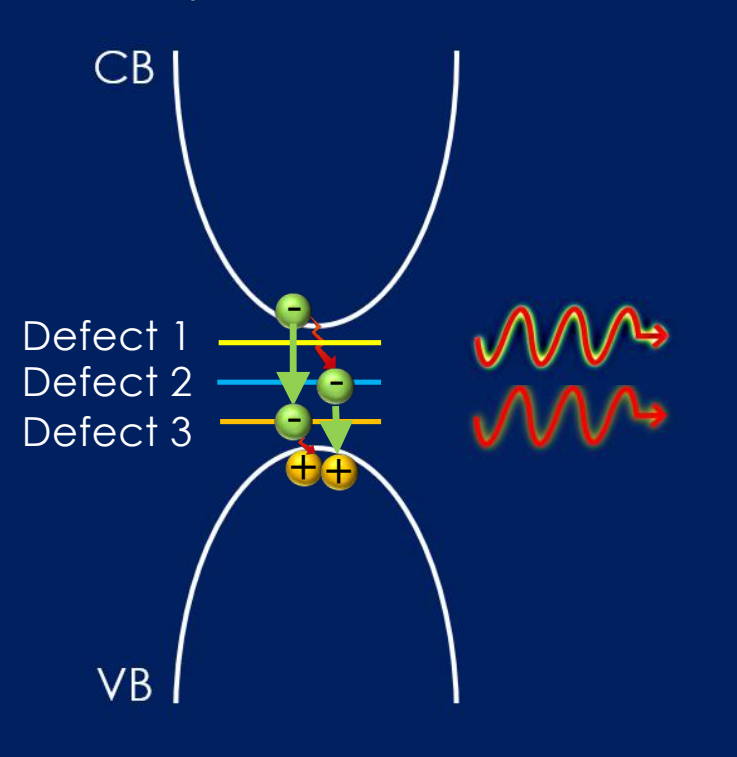
Fluctuations of the stoichiometry induces fluctuation of the band positions

Local fluctuations of the Potential

Local modification of the potential in 1D, 2D or 3D defects



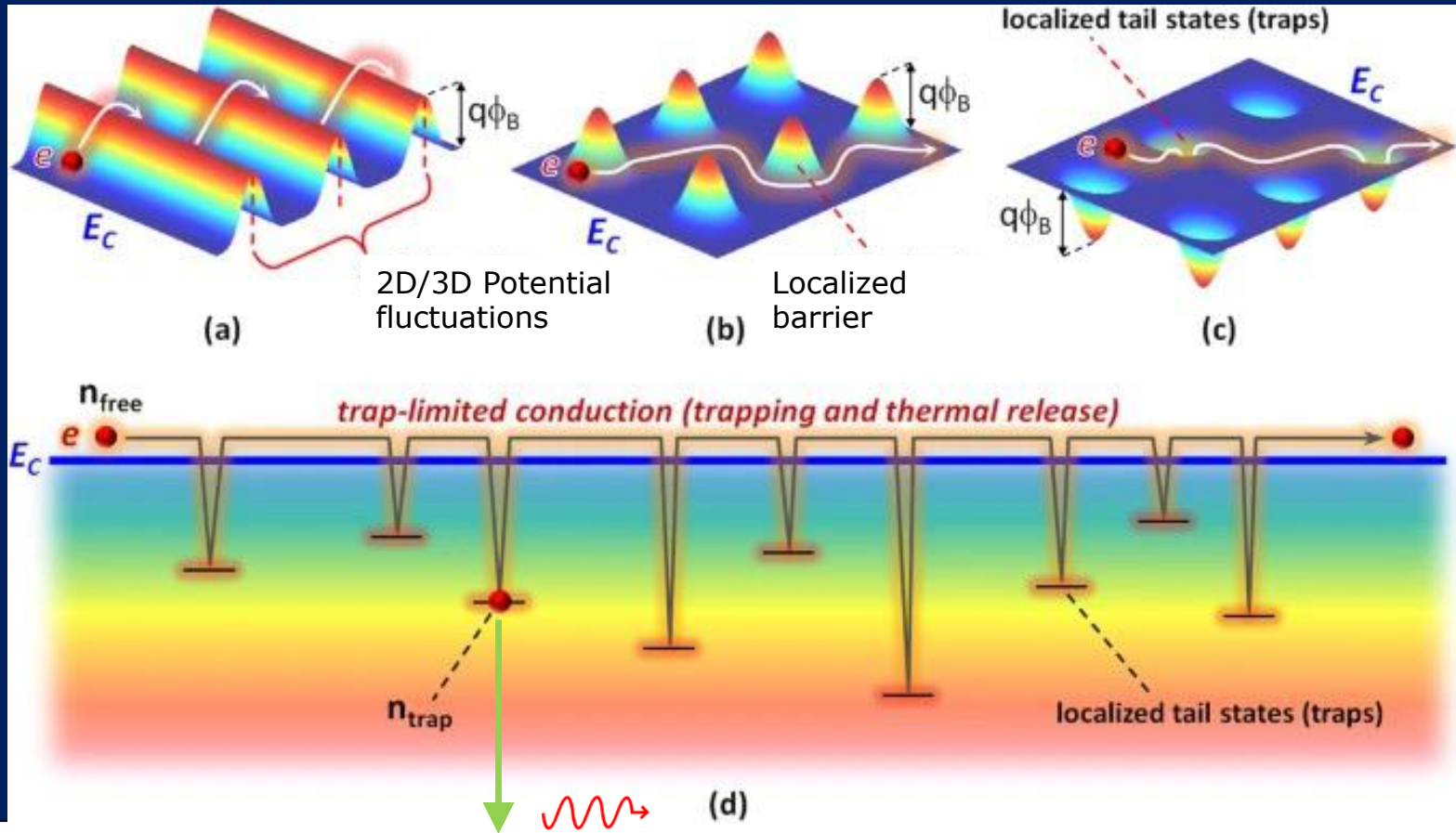
Local modification of the potential in a 0D defect



→ Some defects will modify locally the bandstructure, leading to localization or barriers for transport.

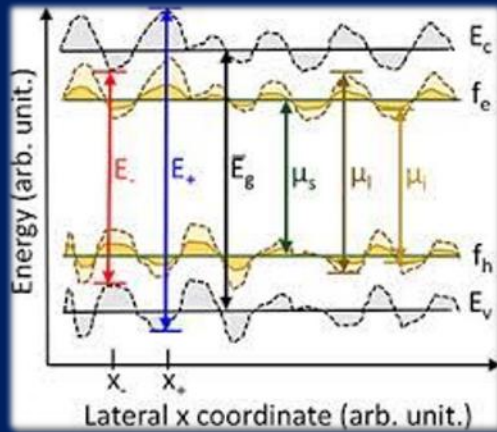
Potential landscape seen by electrons

Potential landscape seen by an electron in a material with 0D, 1D, 2D or 3D defects



Potential fluctuations due to disorder (3D)

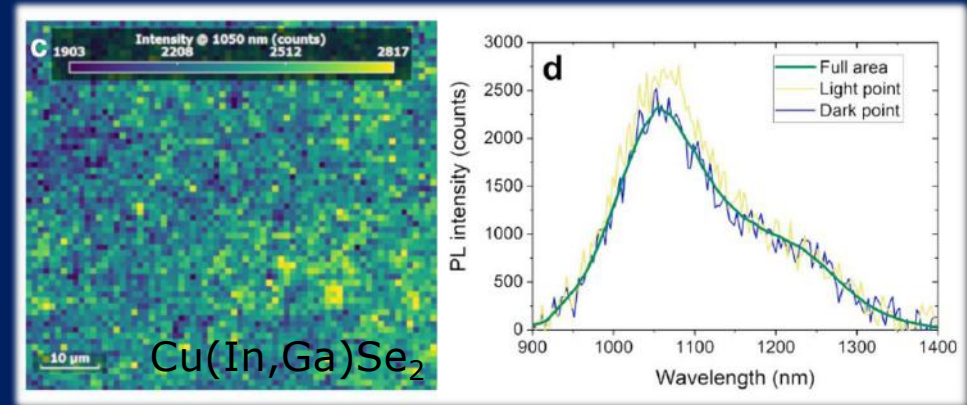
Entropic composition fluctuations of alloys: $A_xB_{(1-x)}$



→ small variations of the composition during the growth can change the bandstructure

Consequences on optical properties

- PL mapping
- PL spectra



→ Broadening of spectral width, inhomogeneity

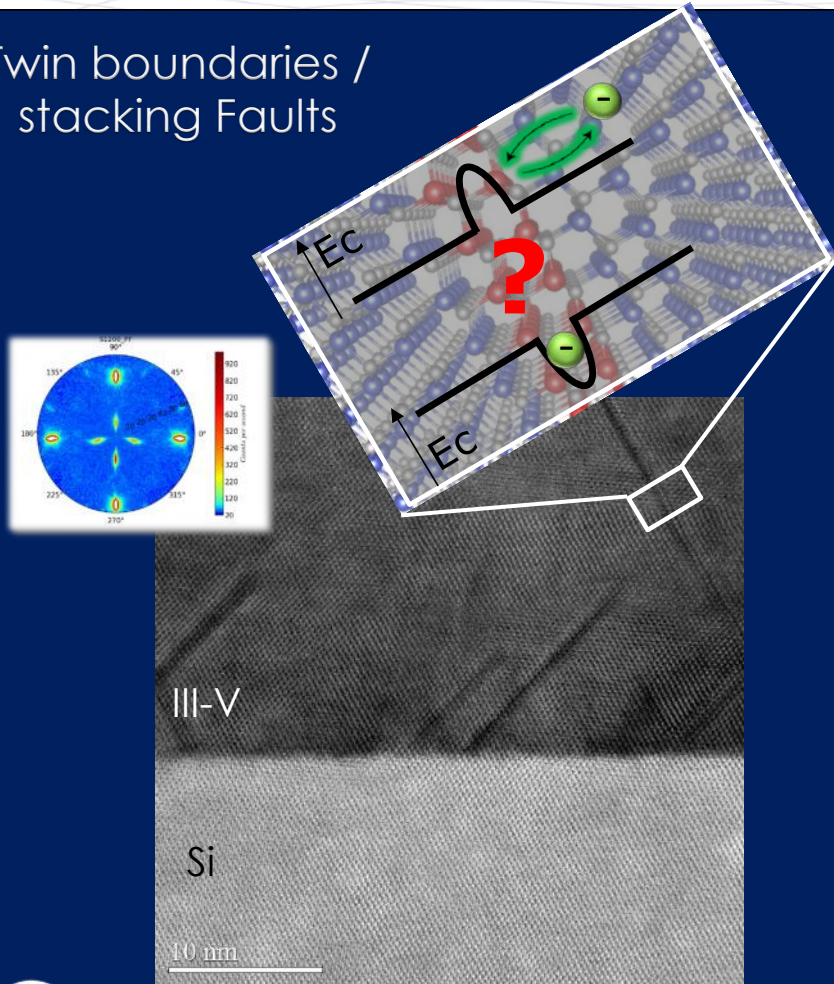
➔ CB and VB will fluctuate over a scale typically >500 nm

Potential fluctuations due to MT or APBs (2D)

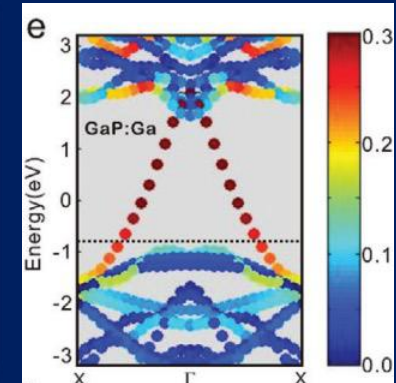
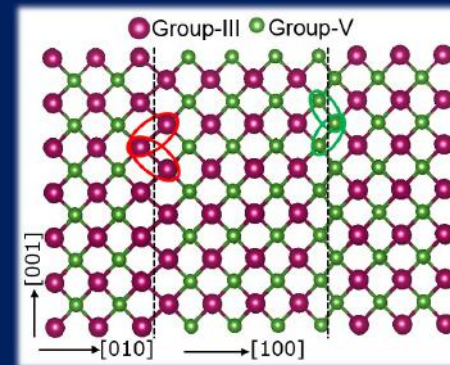
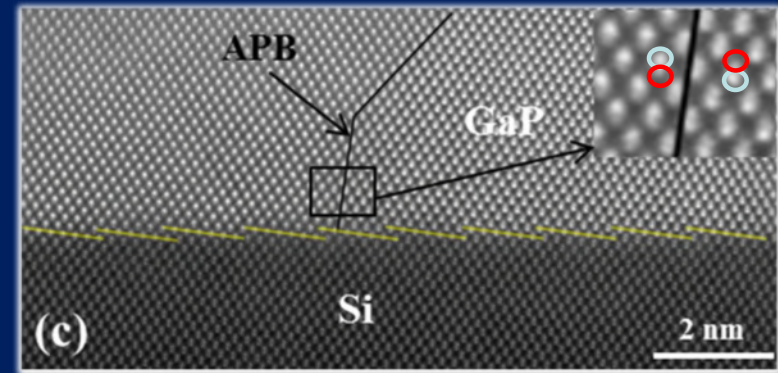
Optoelectronics of crystal defects

2 Potential fluctuations defects

Twin boundaries / stacking Faults



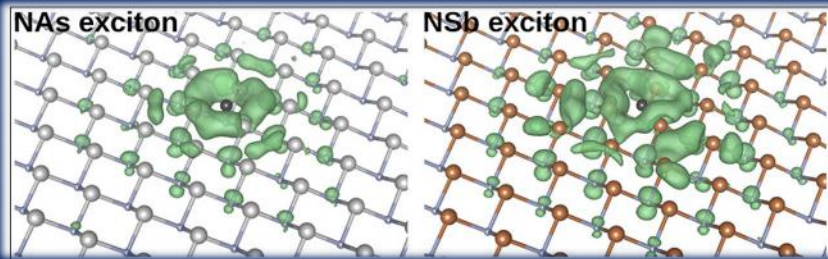
Antiphase boundaries



Strong modifications of the bandstructure : metallic inclusions ! (shortcuts)

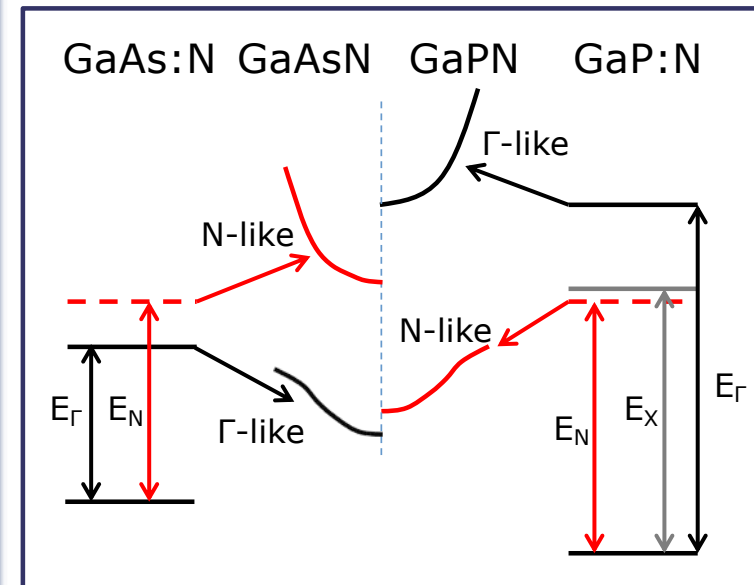
Diluted (metastable) alloys ($x < 5\%$)

- o Incorporation of some atoms with very different valence configurations in a crystal (e.g. N or Bi in III-V semiconductors)



Possible strong localization around the added atoms

Hybridization of Localized states



Ex : GaP(N) and GaAs(N)

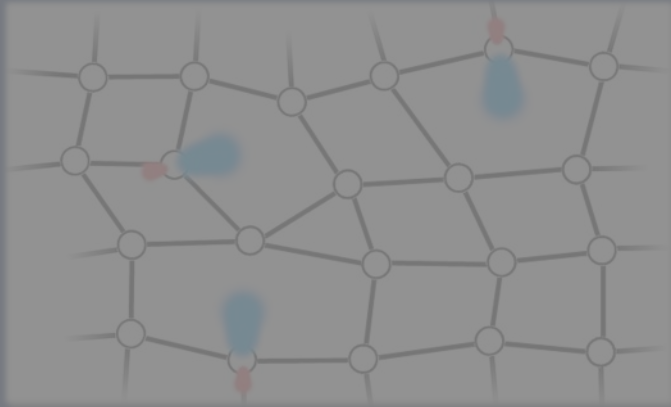


A unique nitrogen level interacting with the conduction band of the host material

Proposed classification of crystal defects

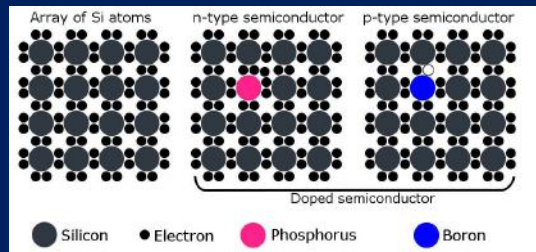
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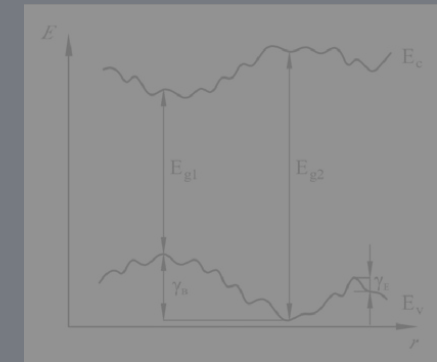
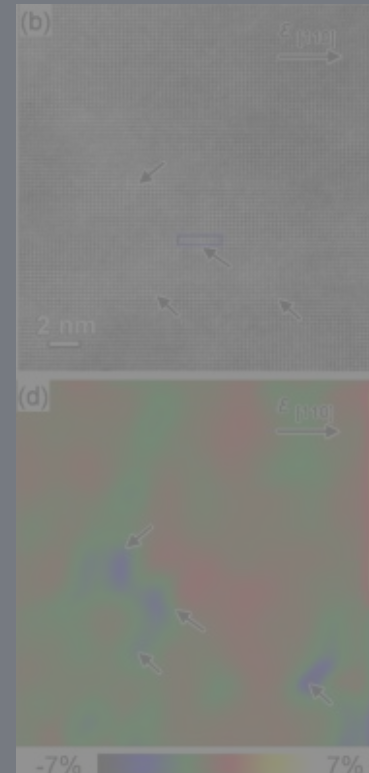
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2

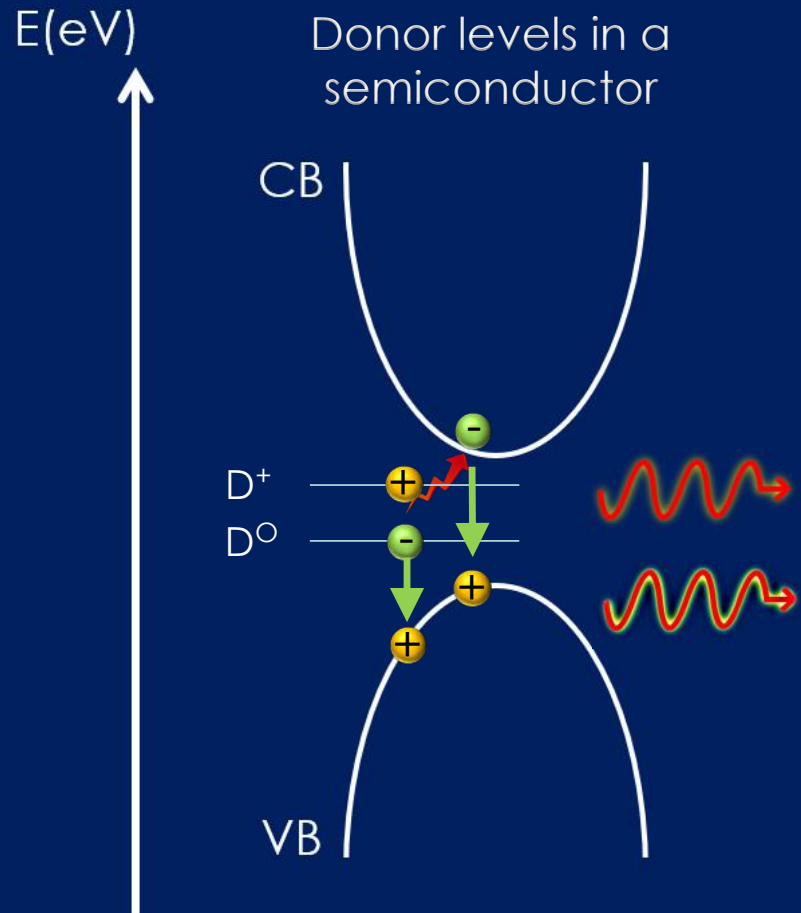
Local fluctuations of the potential



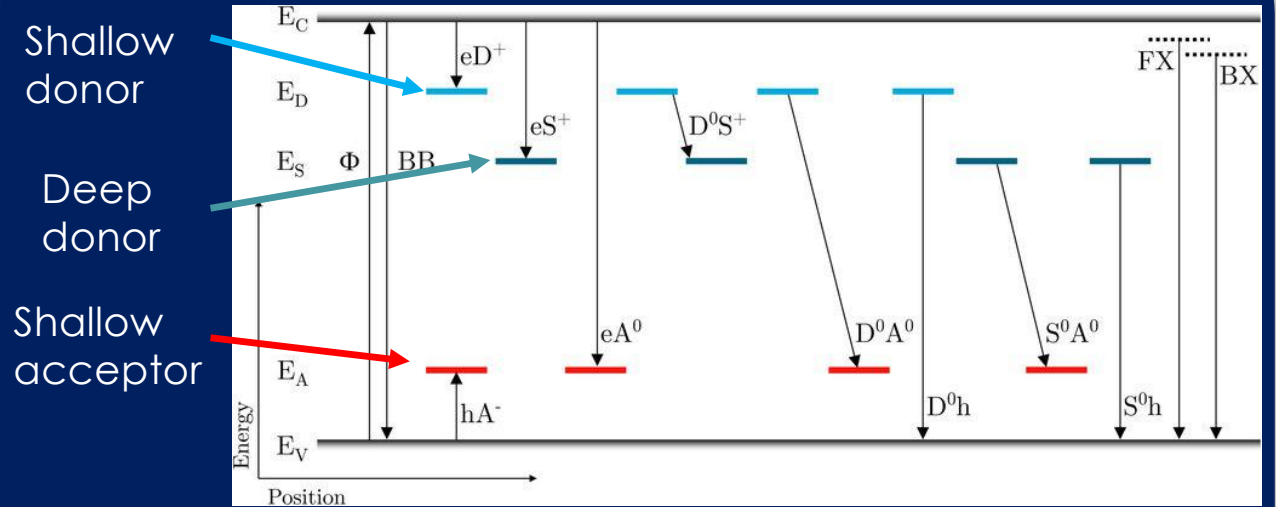
Fluctuations of the stoichiometry induces fluctuation of the band positions



Charge variations due to donors/acceptors (dopants 0D)



The acceptors/donors radiative properties



➔ Many optical transitions enabled by the presence of donors or acceptors in a semiconductor

Crystal defects in semiconductors

1

Crystal defects with Dangling Bonds

Ex : Surfaces, Dislocations, grain boundaries, vacancies

→ Non-radiative recombination defects (loss of carriers)

2

Potential fluctuations in the Crystal

Ex : Alloy disorder, MT, APBs, diluted alloys, type II SCs.

→ Modification of transport properties and of the bandgap

3

Charge fluctuations in the Crystal

Ex : Doping, impurities, donors, Acceptors.

→ Modification of transport properties and of the bandgap



In any cases, a strong impact on photoelectric properties of devices (volumic density and dimensionality of defects matters !)

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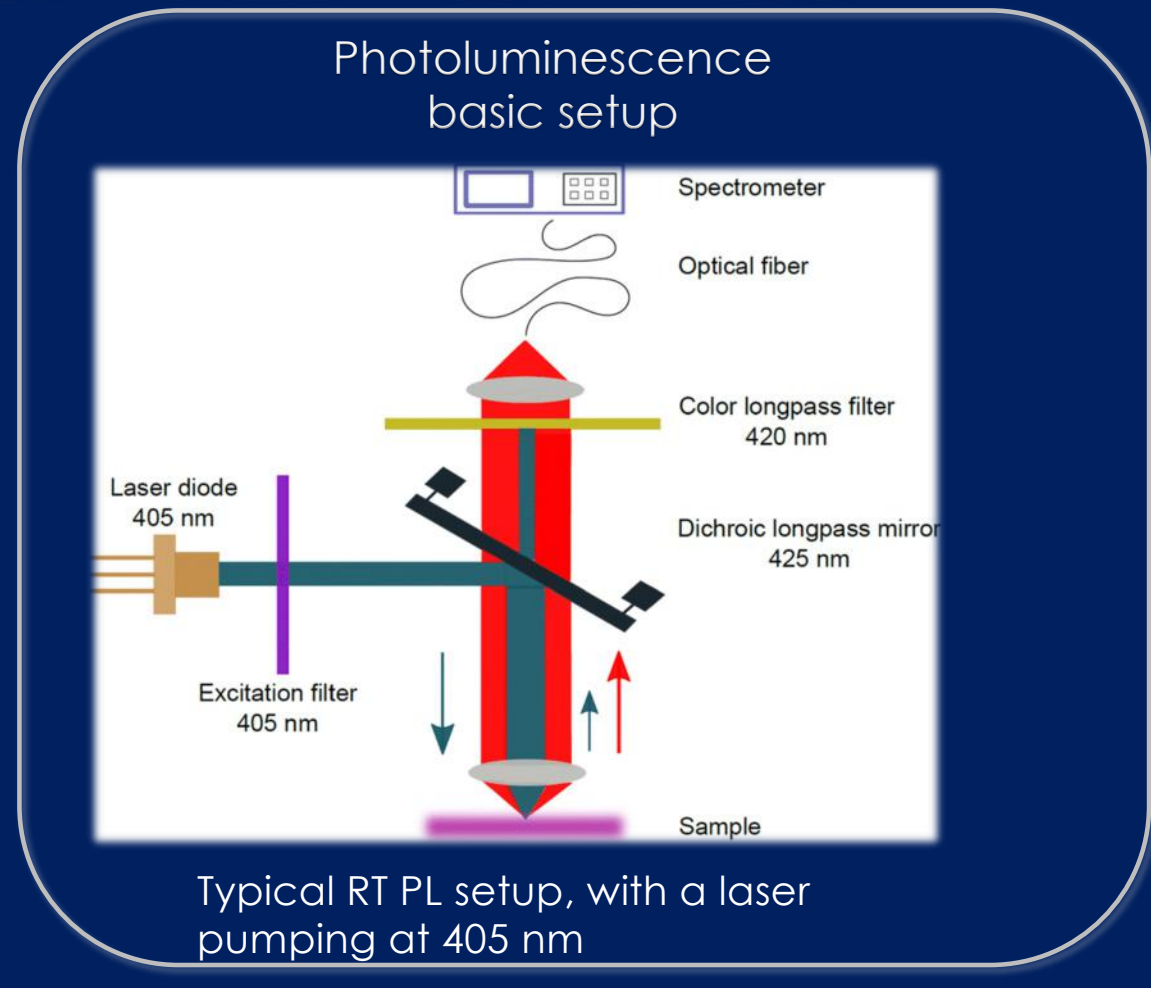
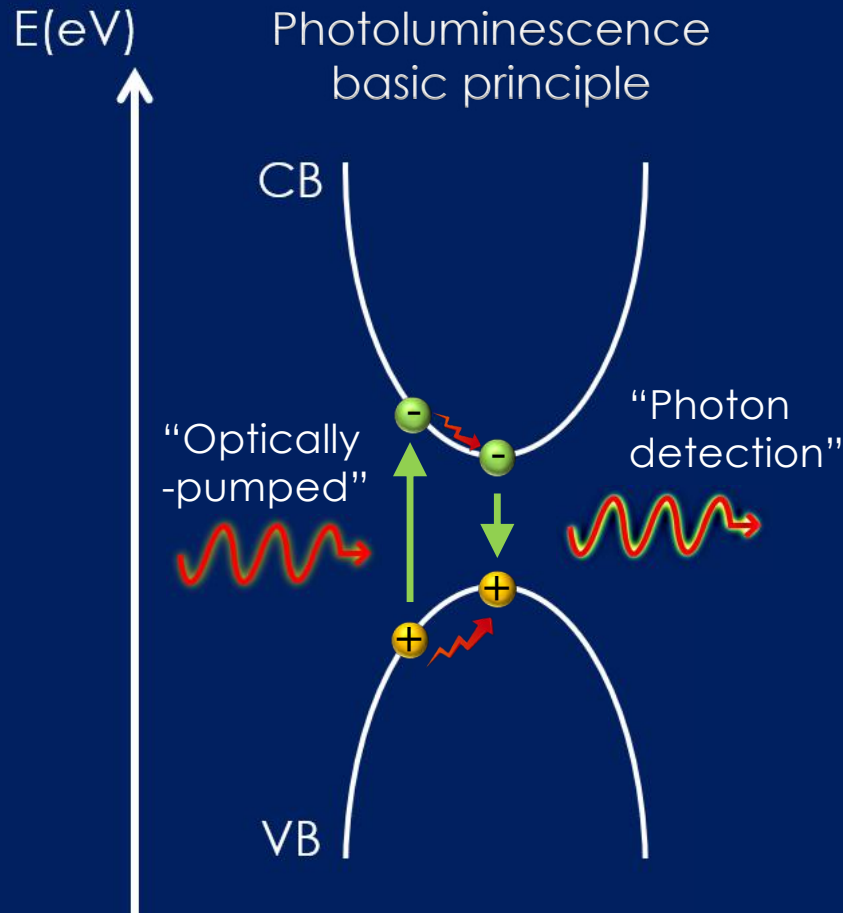
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PL basic principle and setup

Light emission properties

Photoluminescence



PL Experimental setups

Light emission properties

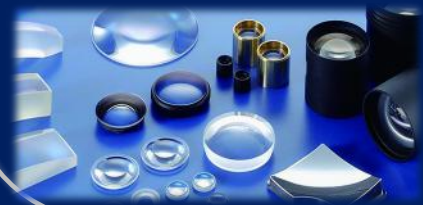
Photoluminescence

Starting first PL experiments, ...



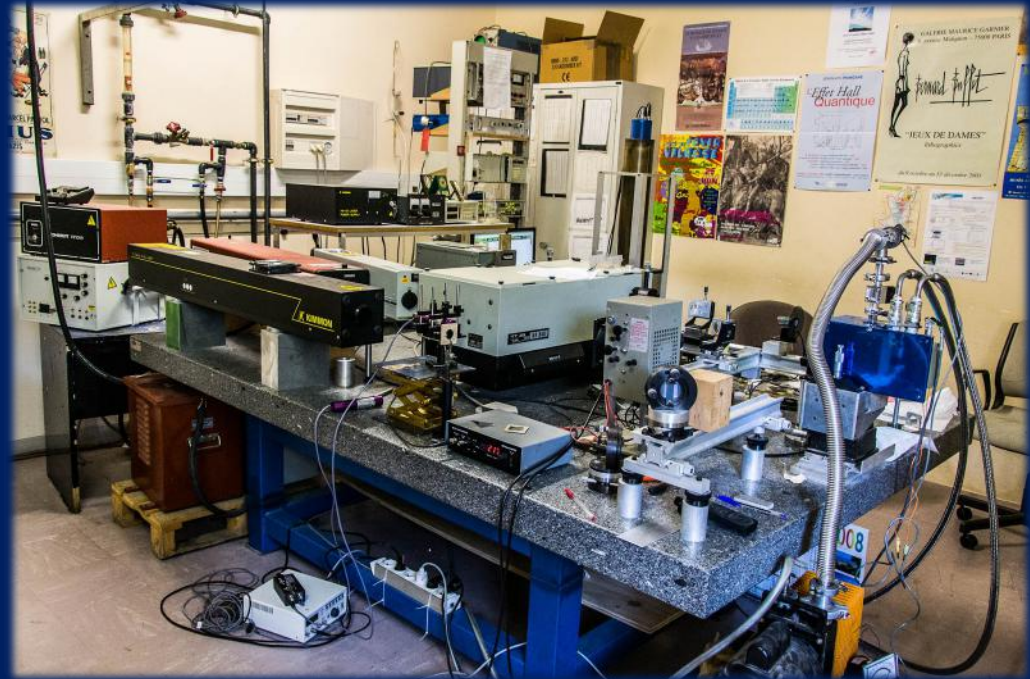
Excitation Laser

CCD spectrometer



Few optical components

...Few years later...



Power, temperature, time-resolved, μ -PL, mapping ...



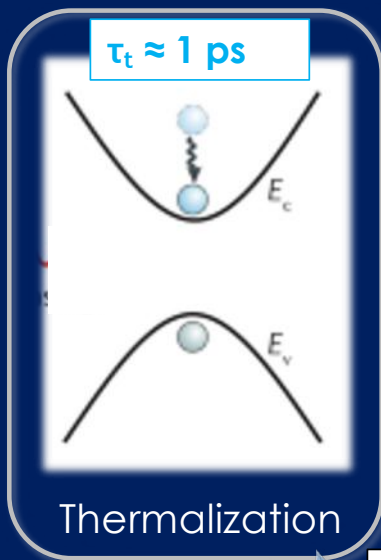
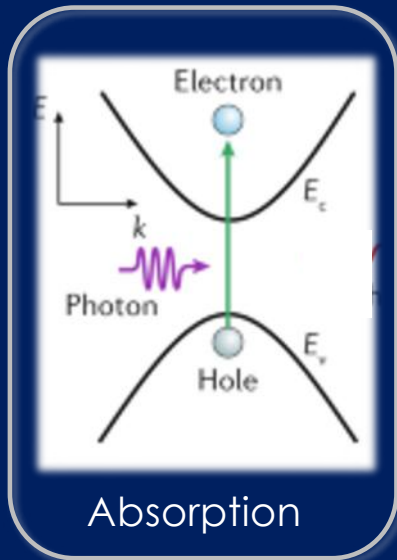
PL : Is it really simple ?

The PL processes

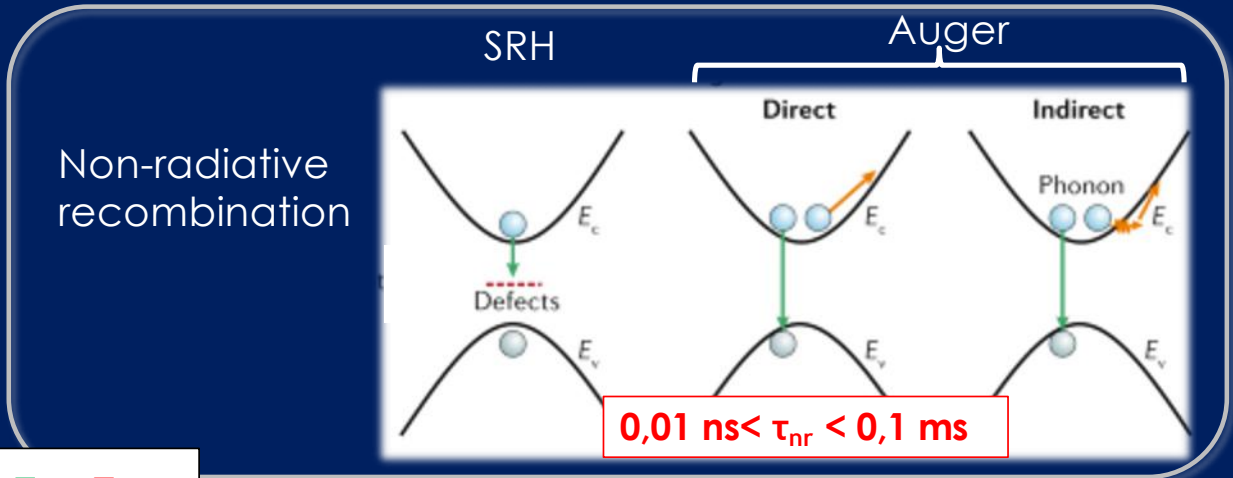
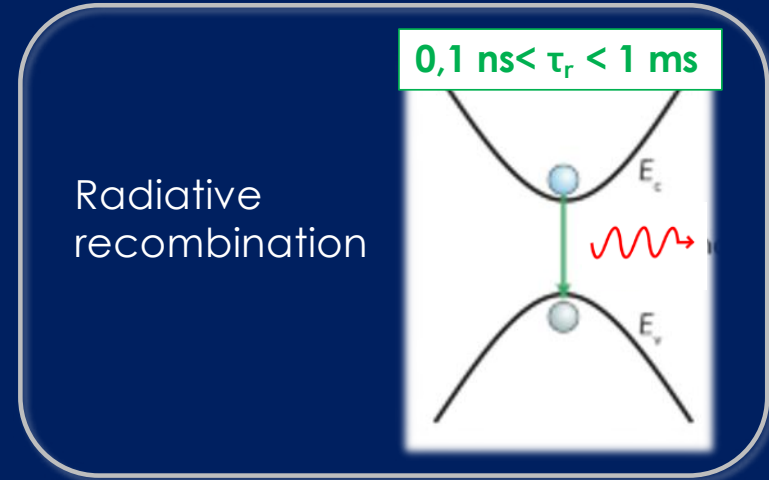
- o Understanding optical properties of materials = understanding time constants
- o Probability of a process to happen :

$$\tau \longleftrightarrow k$$

time constant (s) Process rate (s⁻¹)

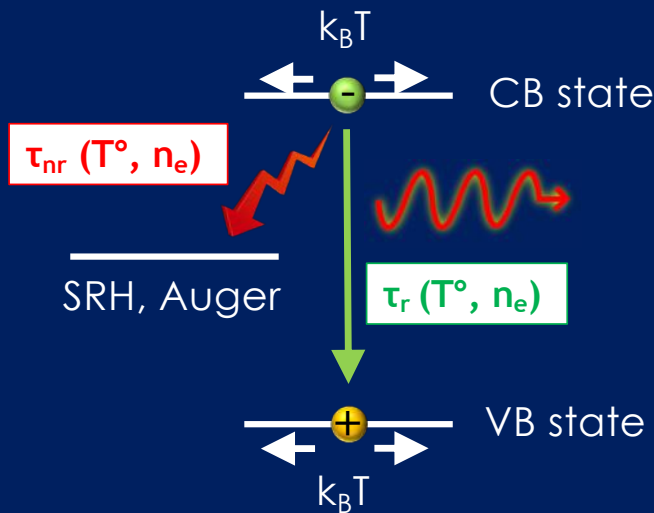


$$\tau_t \ll \tau_r, \tau_{nr}$$



The general picture

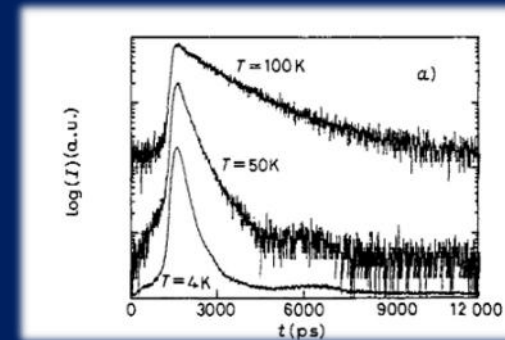
- Most of the PL processes can be understood with the following simplified picture :



- Global Lifetime of the luminescence:

$$\tau = \frac{1}{k_r + kn_r}$$

Measured in time-resolved PL

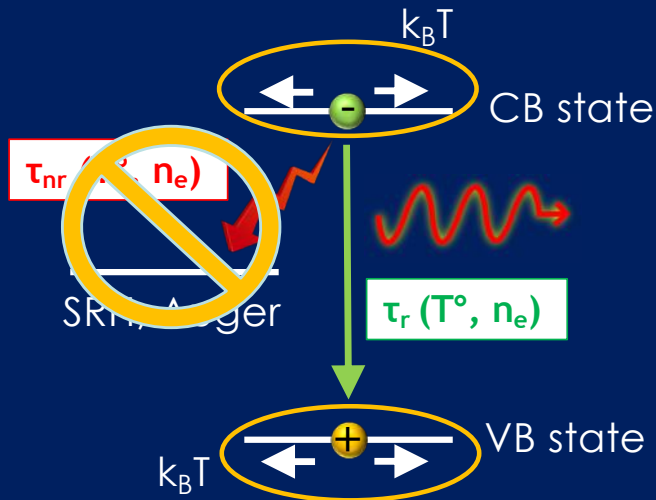


- Quantum yield (\propto PL intensity):

$$Q = \frac{k_r}{k_r + kn_r}$$

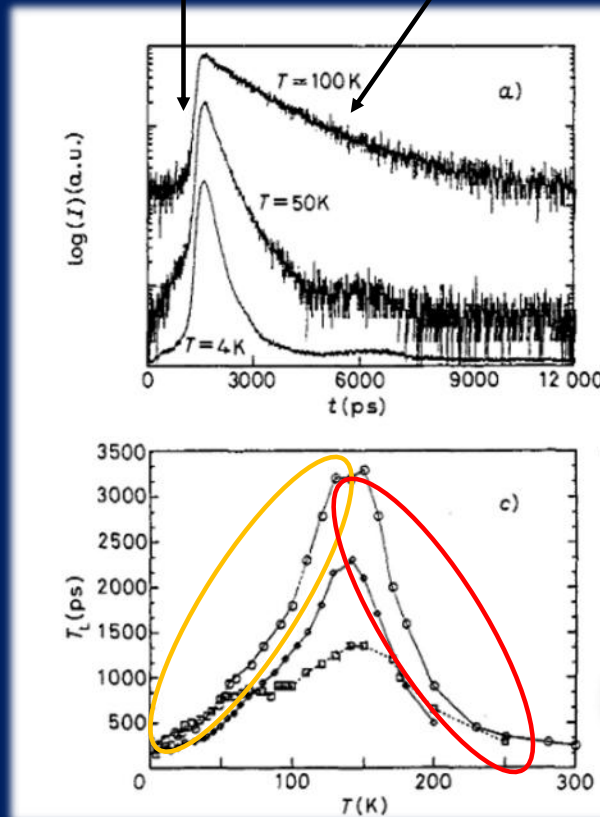
Influence of the temperature

Low T (<100 K)



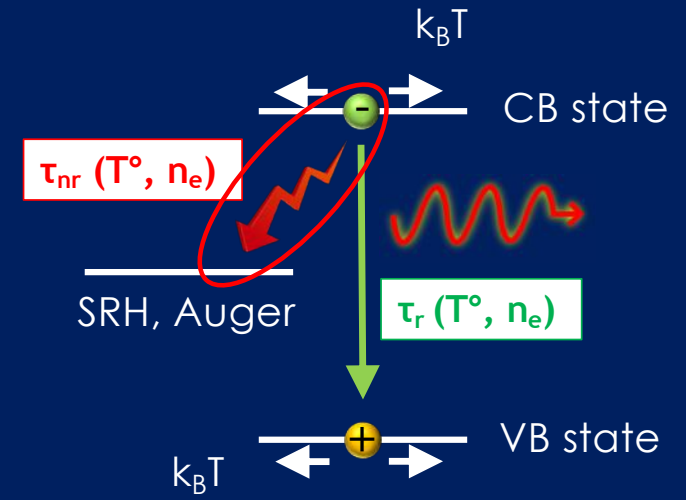
Low T measurements are a signature of pure radiative properties of the sample (w/o defects)

Laser pulse PL decay



Global T-dependent PL lifetimes (III-V SC QW)

high T (>100 K)



High T and RT measurements depend on NR and R competition

RT PL measurements are not sufficient to conclude on crystal defects or intrinsic efficiency

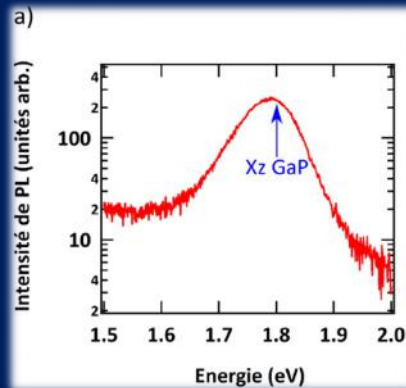


Simple Illustration with QDs PL

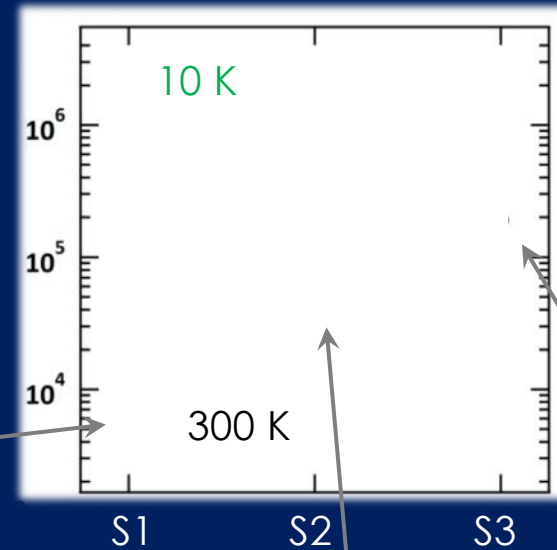
Ex: 3 samples with QDs grown on GaP with different growth conditions, analyzed in the same PL conditions

Sample S1

→ Low PL intensity at RT

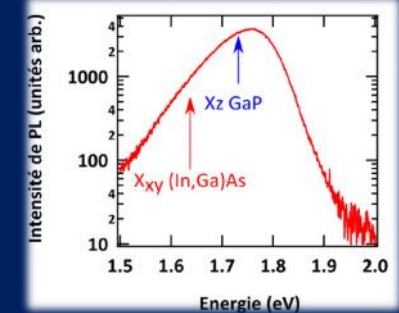


Integrated PL intensity



Sample S3

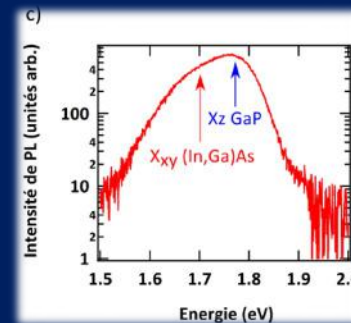
→ High PL intensity at RT



CRYSTAL DEFECTS ?

Sample S2

→ Intermediate PL intensity at RT



→ The RT alone can lead to erroneous conclusion !

→ The low-T PL enables to exclude impact of crystal defects

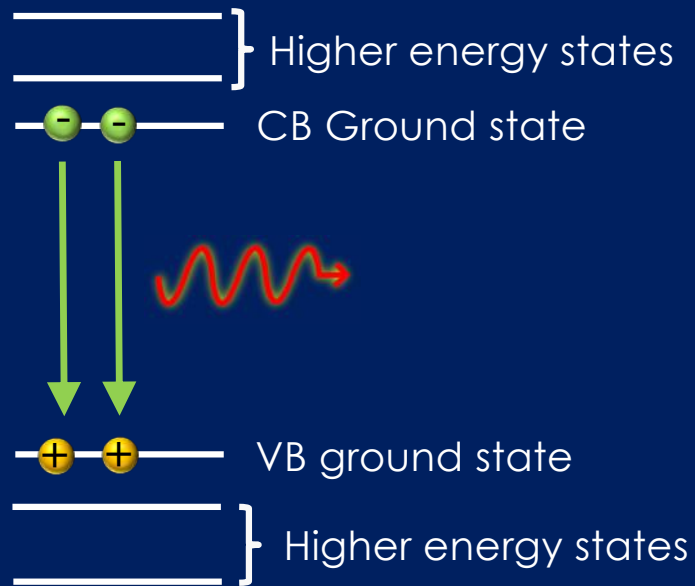


Influence of the incident power

Light emission properties

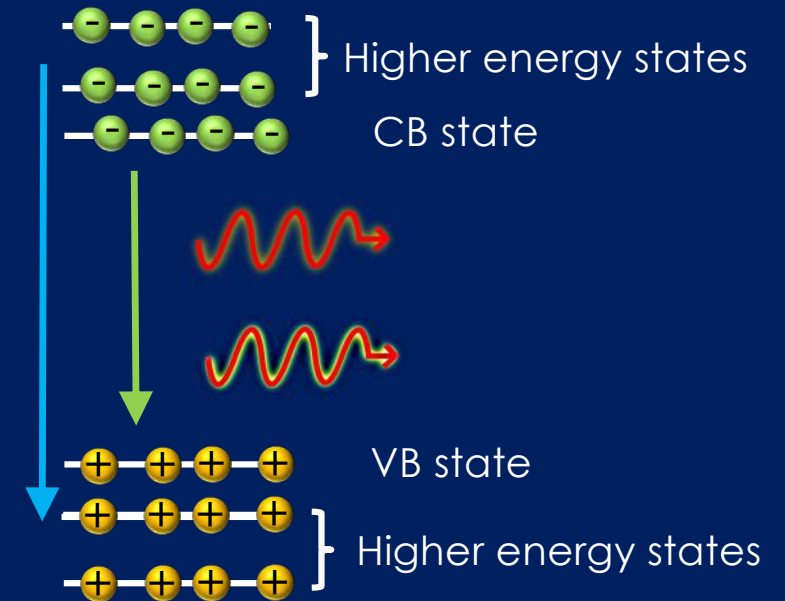
Photoluminescence

Low Excitation density D



Information on the ground state of the system

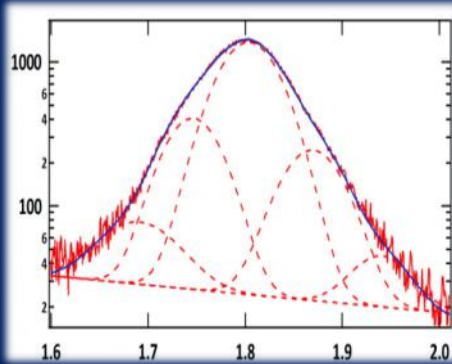
high Excitation density D



Information on higher energy states in the system



PL Intensity

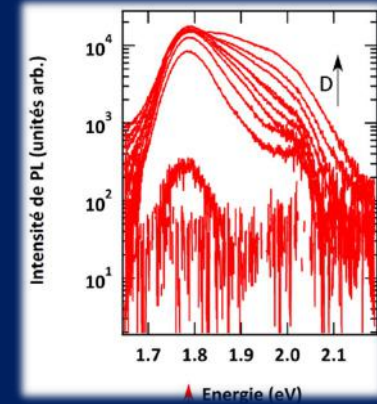
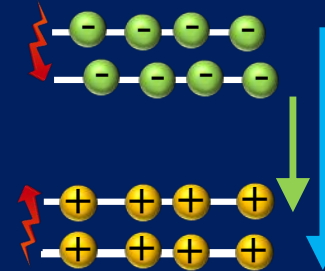


Energy (eV)

PL peak composed of multiple transitions ?

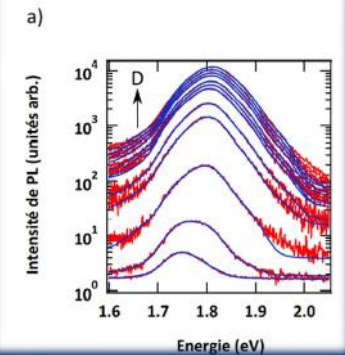
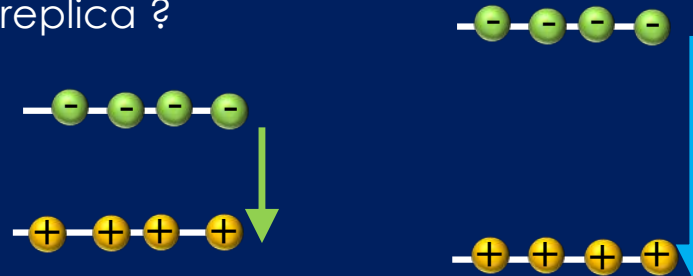
Excited states of the same object ?

-Quantum confined systems ?



Independent Higher energy states ?

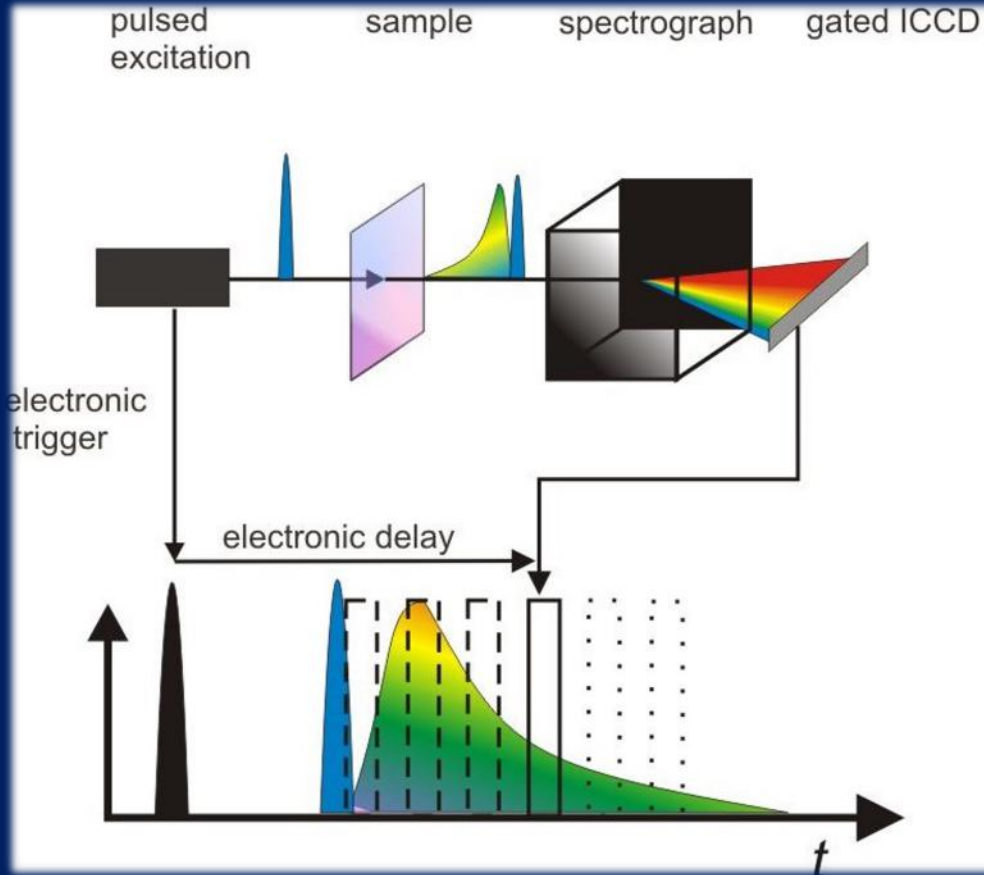
-Density of discrete states ?
-Phonon replica ?



Time-dependent measurement

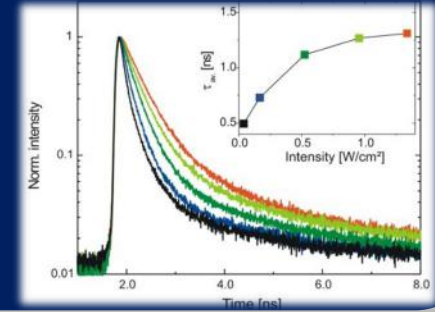
Light emission properties

Photoluminescence

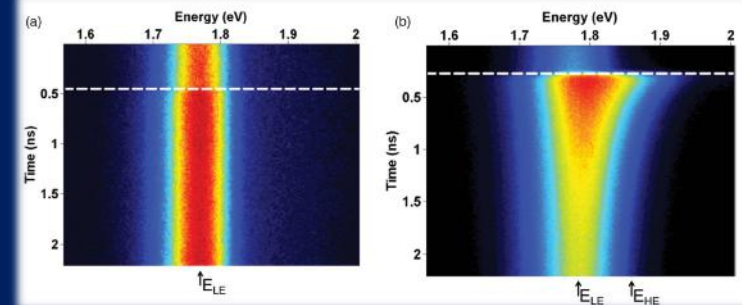


Conventional tr-PL

-Global Lifetime of the luminescence:



Streak camera spectroscopy:



10K; 70 $W.cm^{-2}$

10K; 4000 $W.cm^{-2}$

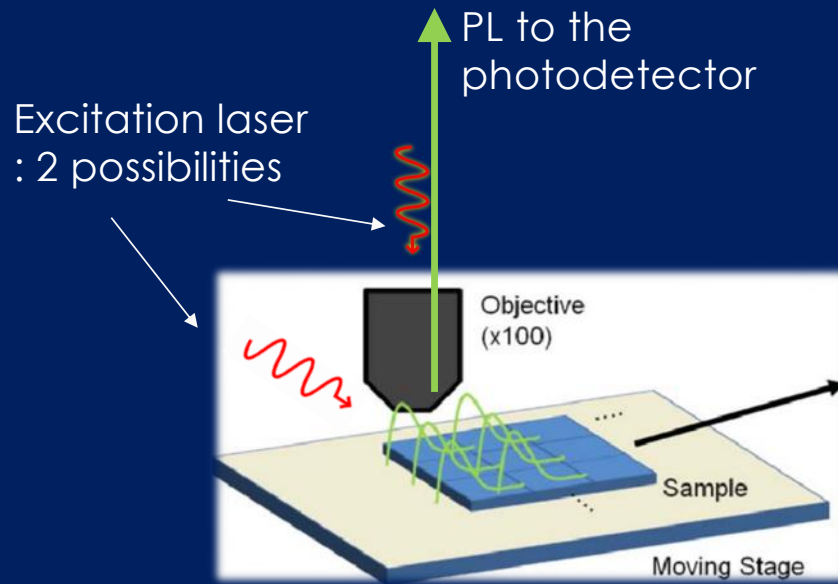
➔ Access to relaxation rates !



PL mapping

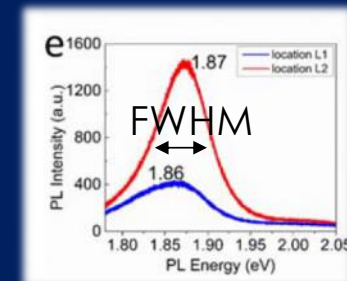
A powerful tool to check samples inhomogeneity

Principle

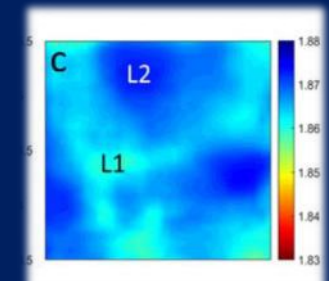


A powerful technique but sometimes misunderstood (absence of low T)

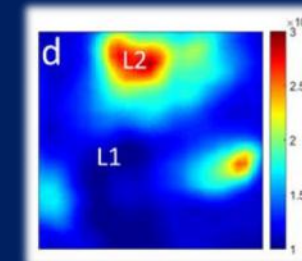
Samples & devices inhomogeneities



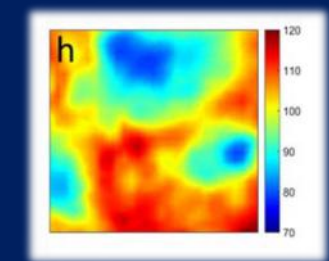
-PL spectrum at each pixel



-2D mapping for PL max energy



-2D mapping for PL intensity



-2D mapping for PL FWHM

Outline

I-Bandstructure of semiconductors, crystal defects and optical processes

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- Photoluminescence
- Electro- & Cathodo-luminescence

III-Characterizing light absorption properties

- Absorbance measurements
- Ellipsometry & Photo-current

IV-Toward single photons sources

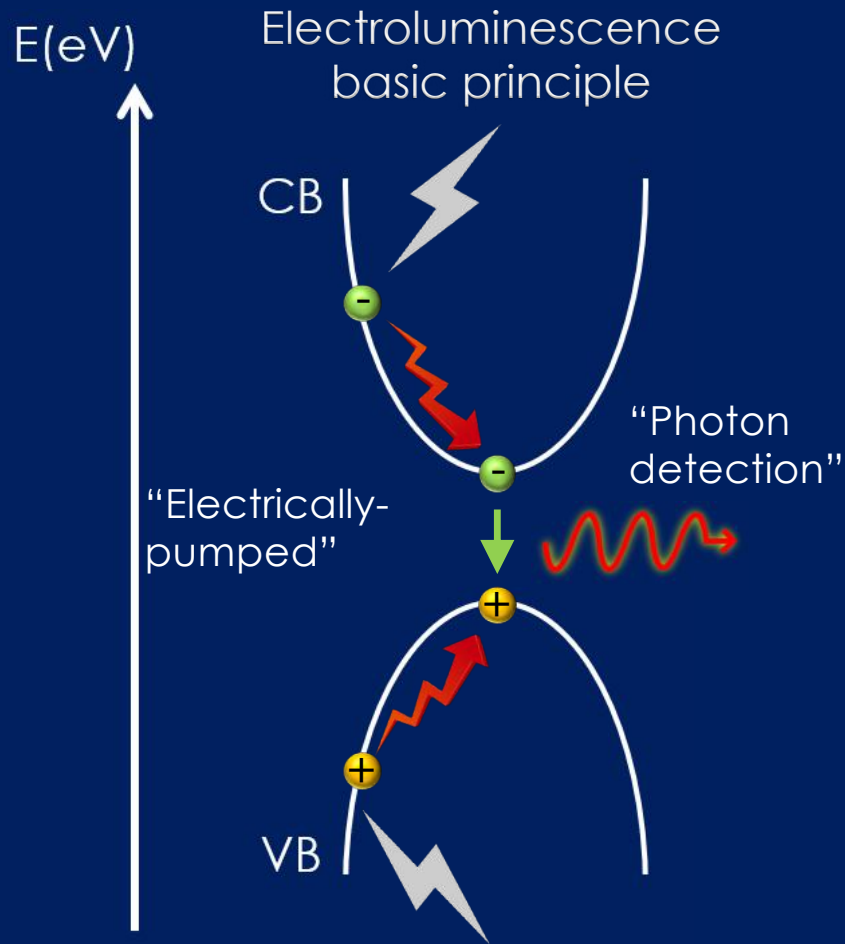
- micro-photoluminescence, $g(2)$



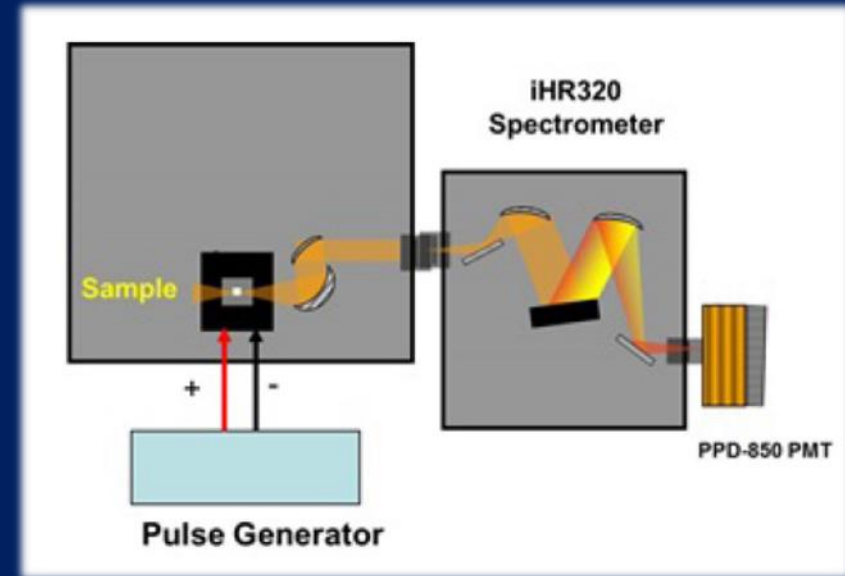
EL basic principle and setup

Light emission properties

Electroluminescence



Electroluminescence basic setup



Similar to PL, but the semiconductor is now excited by a pulse generator

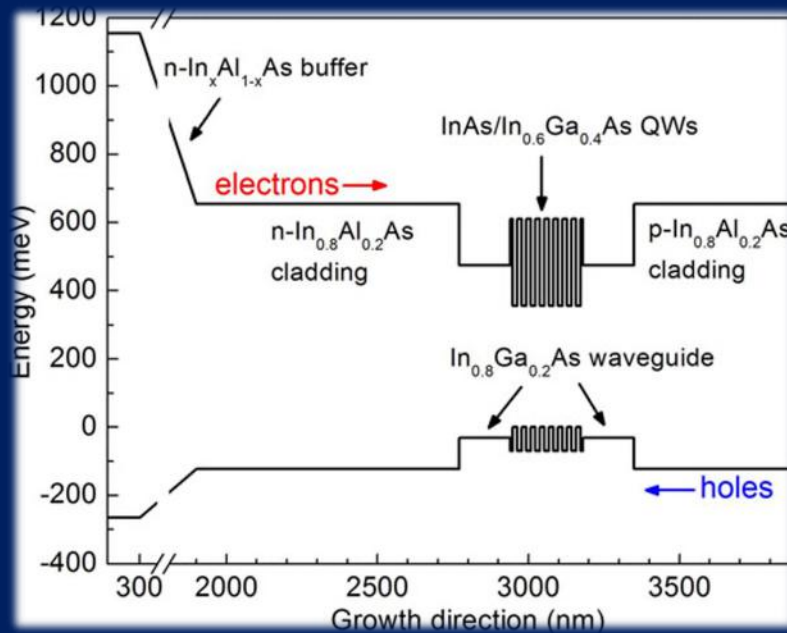
➔ A larger density of injected charge carriers



The issue of carriers injection

Choosing the good stacking of materials

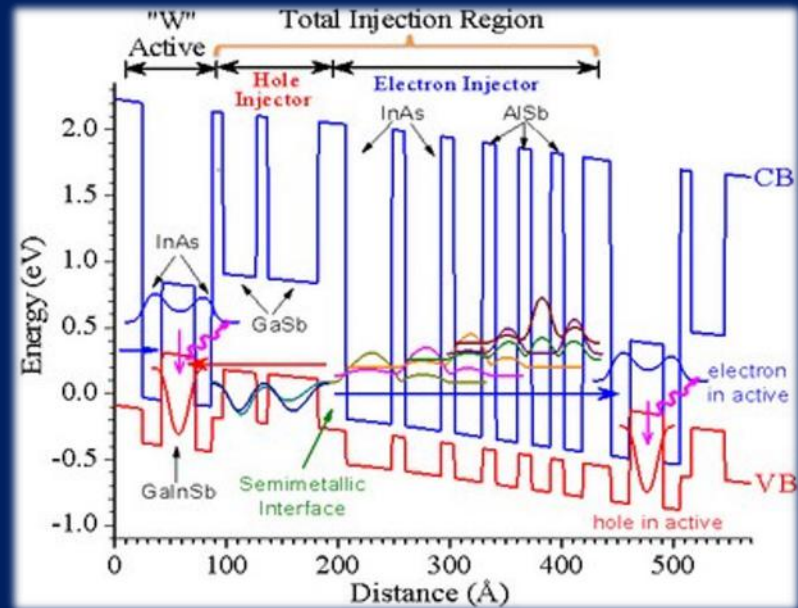
Ex : InP ridge laser



One should ensure that carriers are injected where the EL is expected

Managing the bias

Ex : QCL laser



Bias tends to make it even more complex



EL analysis is usually done on mature devices or materials stacks



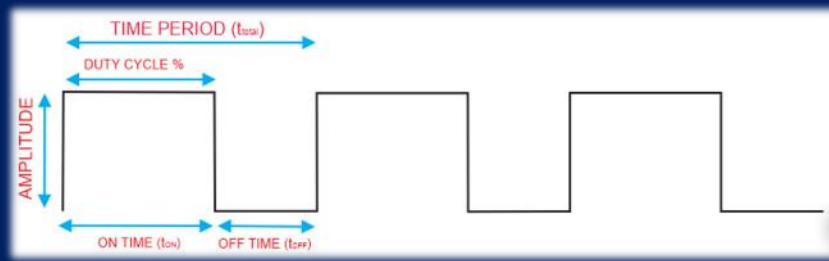
Importance of the duty cycle

The pulse generator

Continuous wave (cw) electrical pumping :



Pulsed electrical pumping :

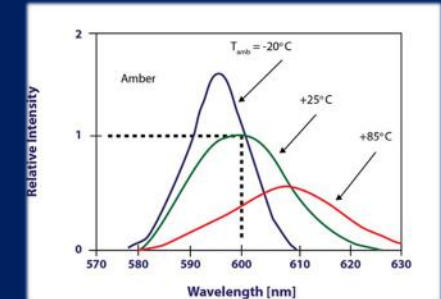


➔ The ultimate device should work in cw conditions

➔ The first step toward a laser device

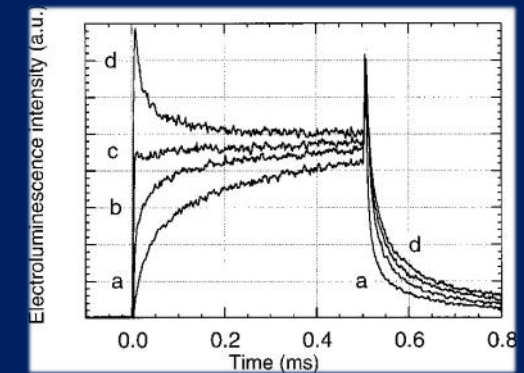
Thermal Management of the device

(Avoid degradation of devices due to heating)

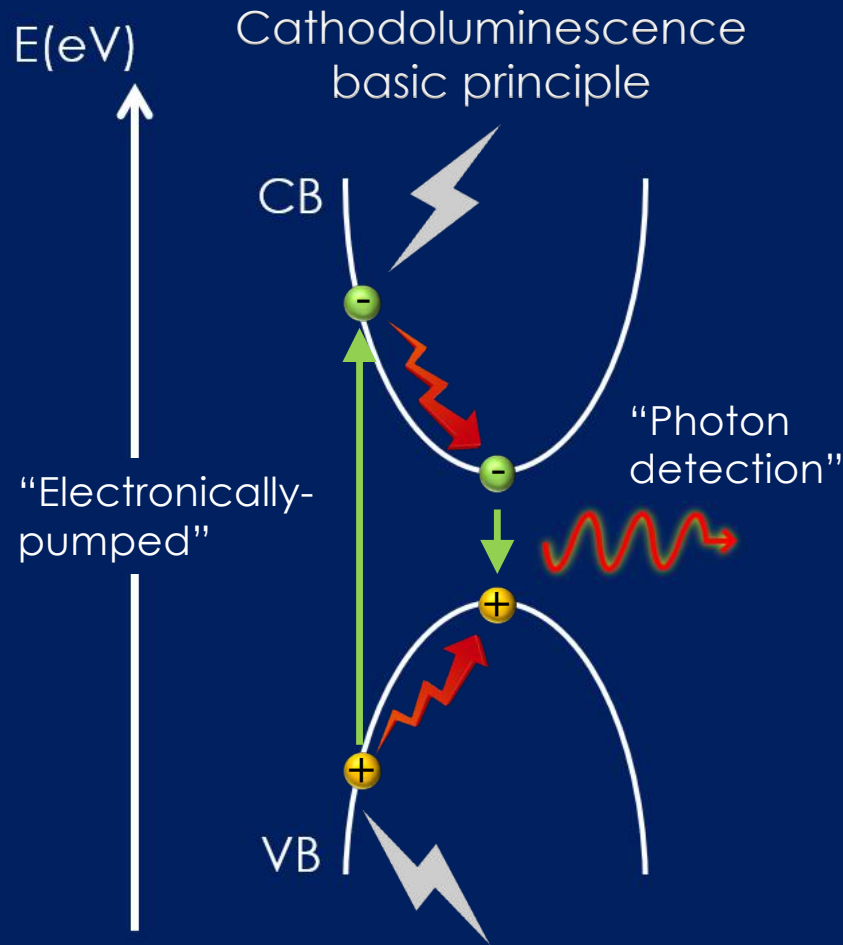


Time-resolved analysis

Transient EL analysis of carriers injection

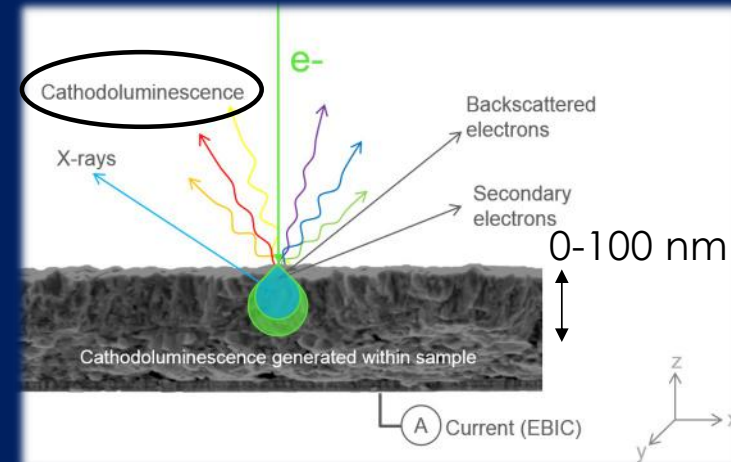


CL basic principle and setup



Electroluminescence basic setup

Cathode-Ray tubes e^- (1,5 to 25 kV)

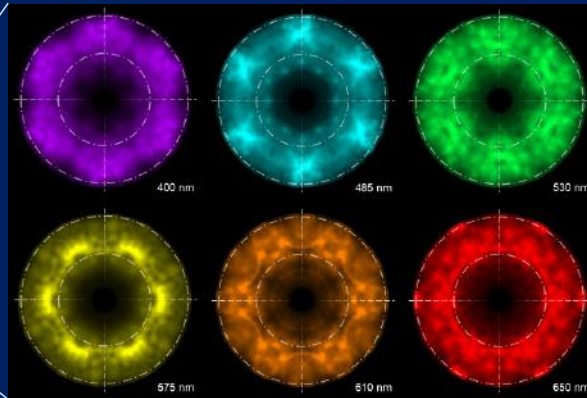
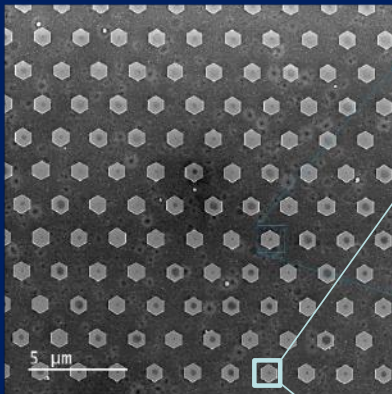


Similar to EL, but the semiconductor is now excited by an electron beam

➔ A much more complex experiment than EL and PL

Promises

Ex: III-N nanopillars

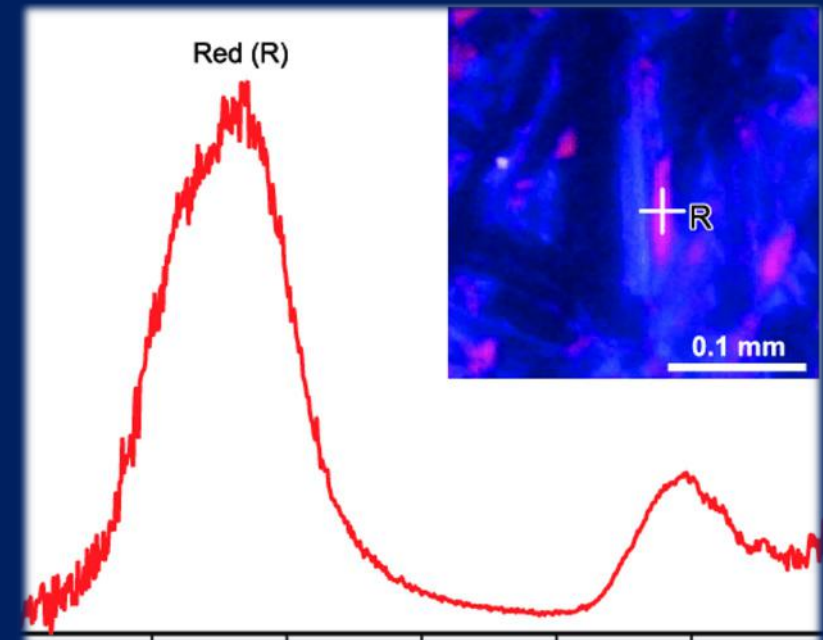


- Low-T, time-resolved, and mapping available, with a high spatial resolution, and variation of the depth



Interesting for localized defects, and sub-micronic nanostructures

Limit : An excitation spectroscopy



Mapping gives the coordinate of excitation, but the whole CL on the sample

Intermediate conclusion

Optical processes

- Understanding optical processes = understanding time constants

Photoluminescence

- Looks like a simple experiment, but requires deep understanding of the processes ($T^{\circ}\text{C}$, Power, tr-).
- Constant competition between radiative and non-radiative recombination channels

Electroluminescence

- A simple experiment, on mature samples/devices
- Importance of the pulse generator

Cathodoluminescence

- A complex experiment, adapted for localized spectroscopy
- An excitation spectroscopy

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III-Characterizing light absorption properties

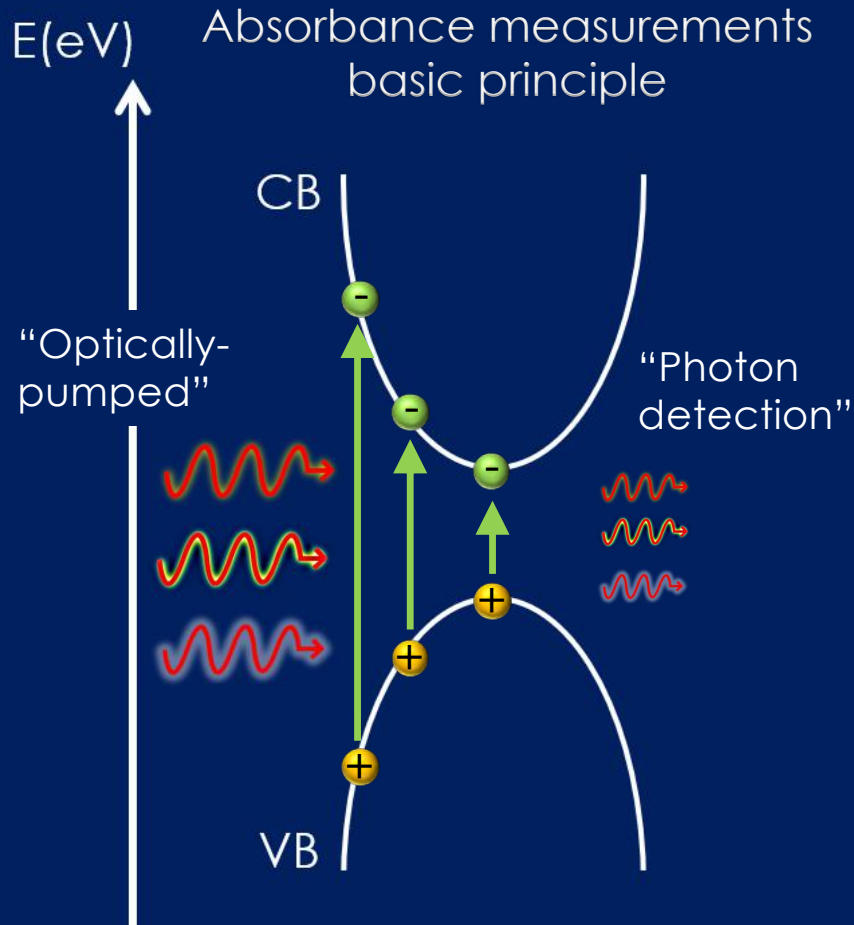
- Absorbance measurements
- Ellipsometry & Photo-current

IV-Toward single photons sources

- micro-photoluminescence, $g(2)$



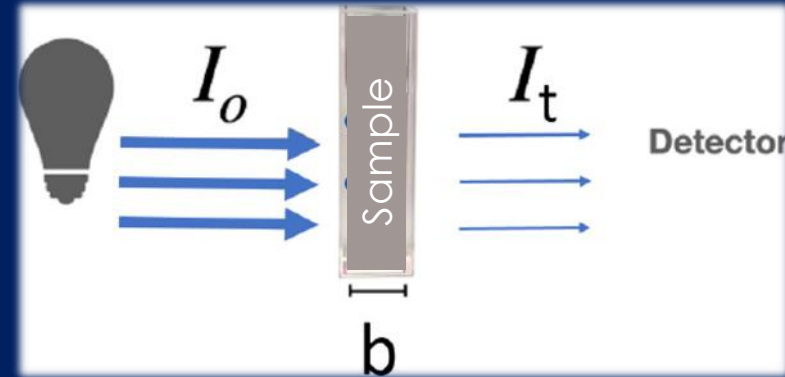
Absorbance basic principle and setup



Absorbance basic setup

- Light sources :
 - Broadband light sources
 - Tunable laser

- Detectors:
 - conv. photodetector



The measurement gives the Absorbance :

$$A = -\log_{10}\left(\frac{I_t}{I_0}\right)$$

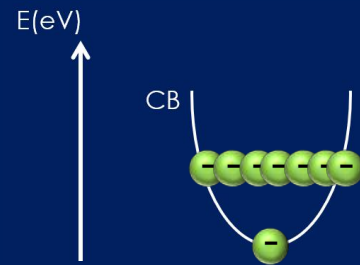
Absorption coefficient is then inferred :

$$\alpha = A/b \quad (\text{cm}^{-1})$$

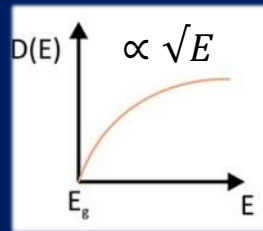
Useful informations about absorption

Joint density of states

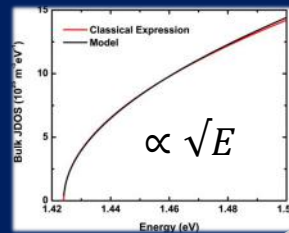
-Bandstructure : different possible states possible for each energy



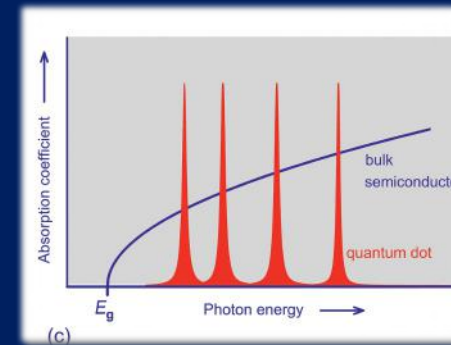
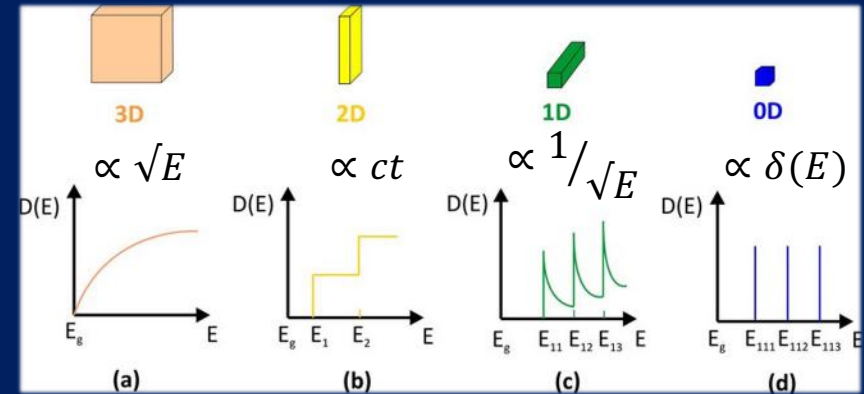
-Density of states DOS : density of possible states /energy /volume



-Joint Density of states JDOS : density of possible optical transitions /energy /volume, at constant k



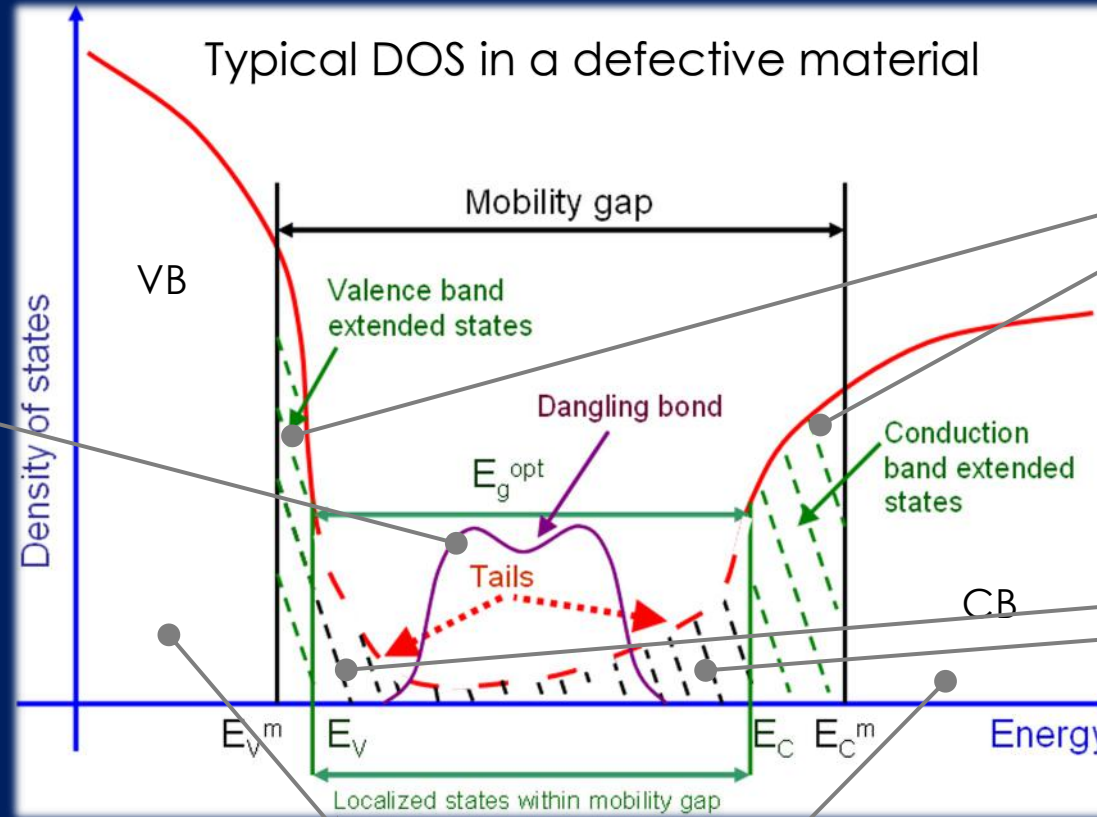
JDOS and absorption



Absorption ~ screenshot of the JDOS

Promises of absorbance measurements

Light absorption properties



Dangling bonds defects

Potential fluctuations (e.g. alloying)

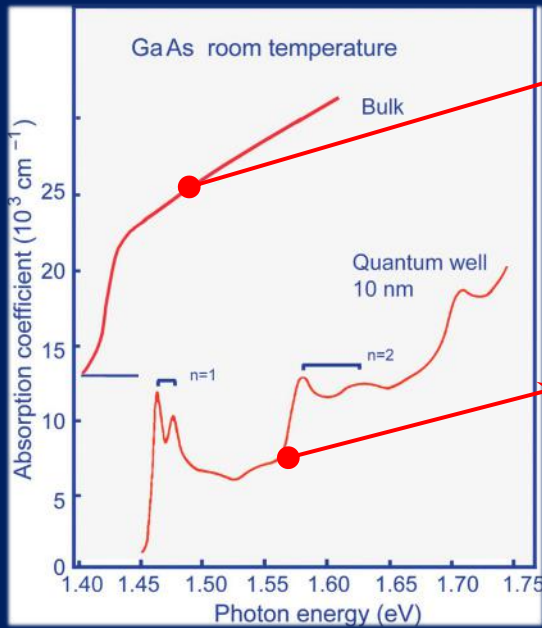
Localized states (traps) or quantum structures

Bulk material
In real conditions, mid-gap states are hardly accessible with absorbance (density, optical activity)



Absorption : a bunch of useful information

Ex : Quantum confinement



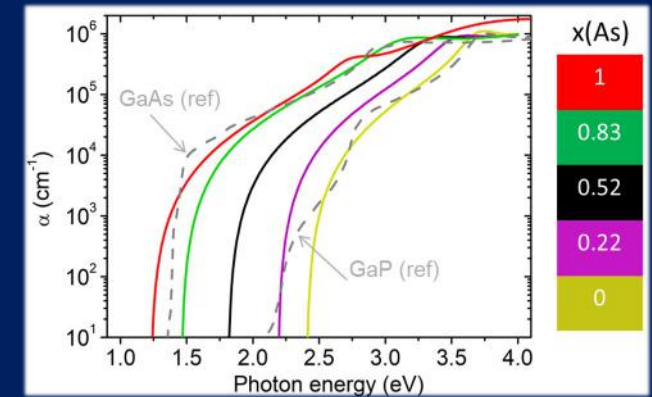
Typical \sqrt{E} bulk-like evolution

Typical step structure \rightarrow quantum confinement



Allows to identify quantum confined systems

Ex : alloying

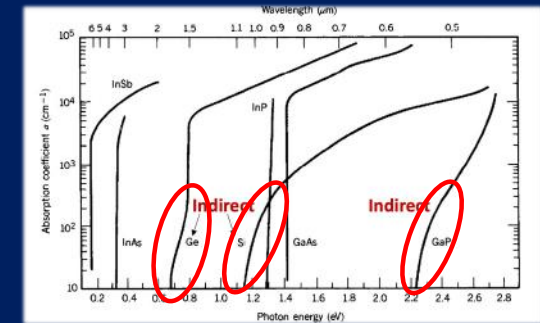


Ex : Direct/indirect bandgap

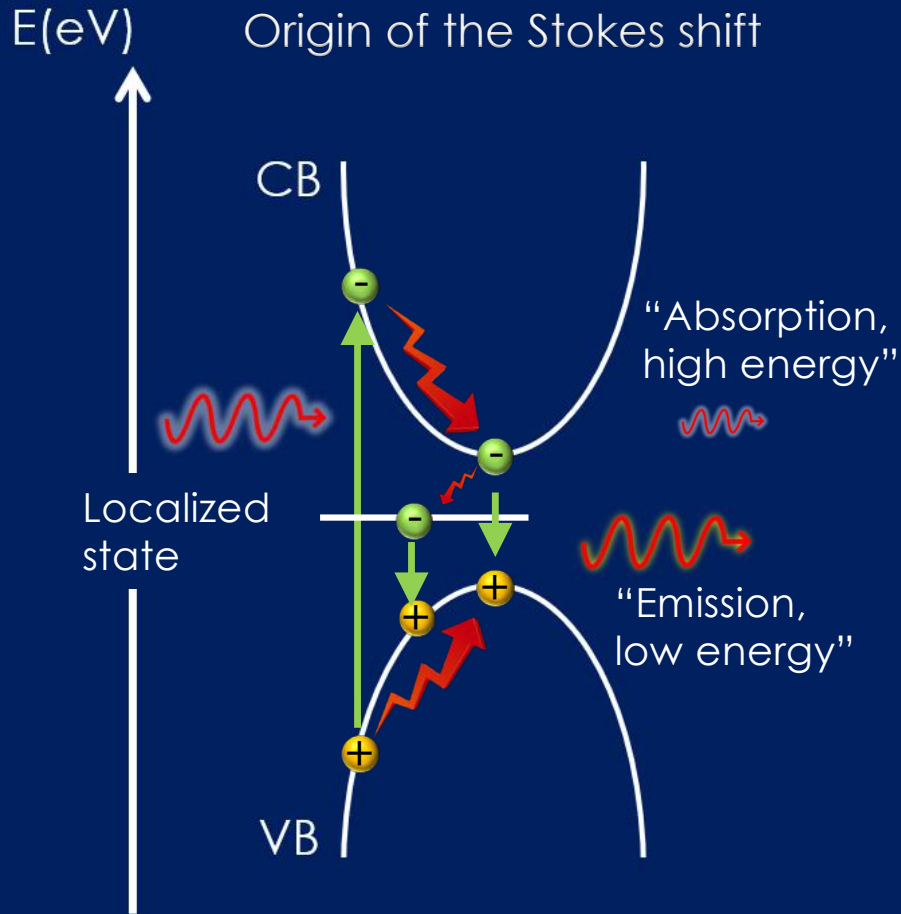
Absorption edge :

-sharp for a direct bandgap

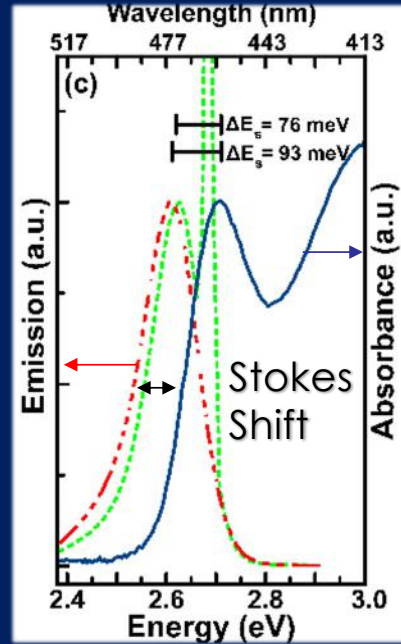
-smooth for an indirect bandgap



The Stokes Shift



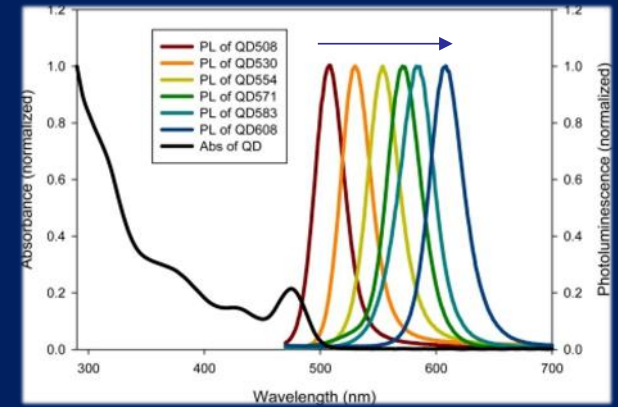
Usual situation



Max of the PL = absorption edge

Strong Localization

Increased localization



➔ Stokes shift is a measure of the localization

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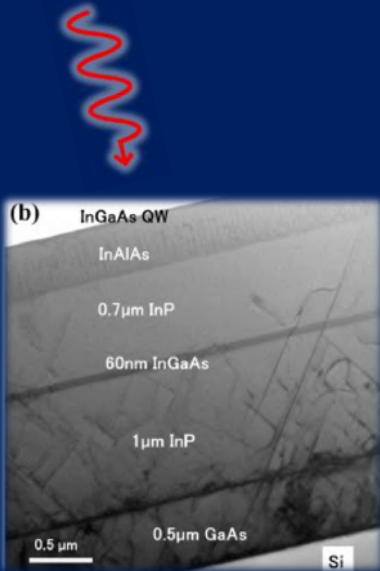
- Absorbance measurements
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IV-Toward single photons sources

- micro-photoluminescence, $g(2)$



The stacking issue



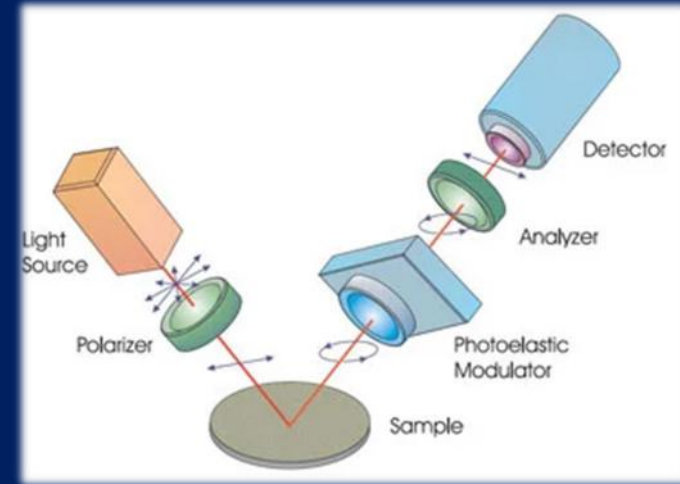
-Different layers with different bandgaps and n, k optical constants

-Optical constants (n, k) of the individual layers ?

Global absorption of the whole sample can only be determined



Ellipsometry



Analysis of the change of the polarization of light during the reflection on the sample



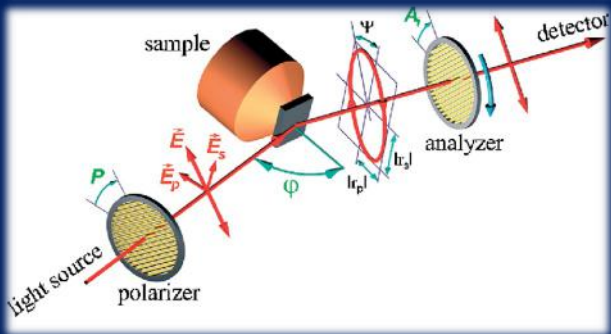
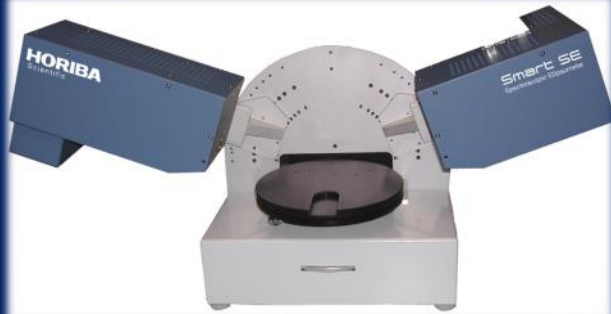
A very powerful technique

The method

Light absorption properties

Ellipsometry

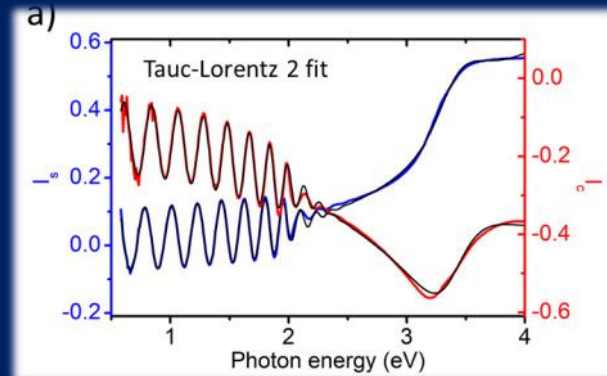
Measurement



➔ Measure of I_s and $I_c = f(\Psi, r_s, r_p)$

Fitting

- ➔ the trickiest part of the job
- ➔ Needs to assume a model

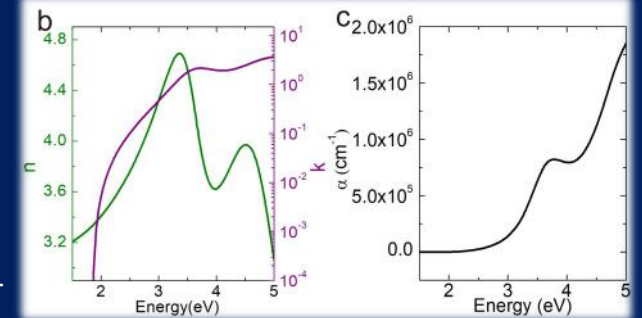


Optical constants

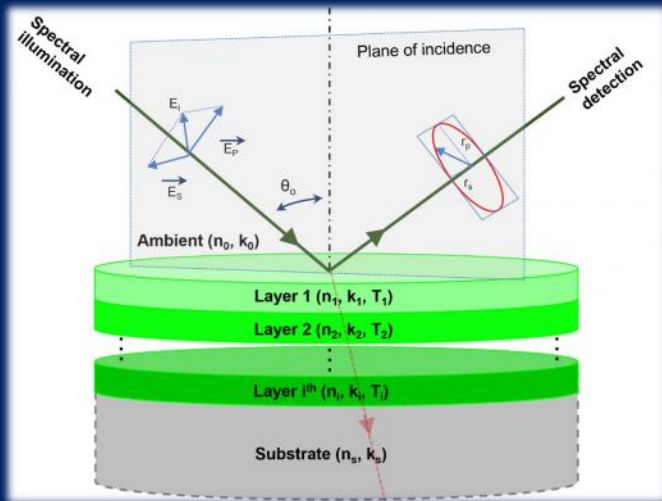
For each individual layer :

- n , optical index
- k , extinction coefficient

➔ α , absorption coefficient



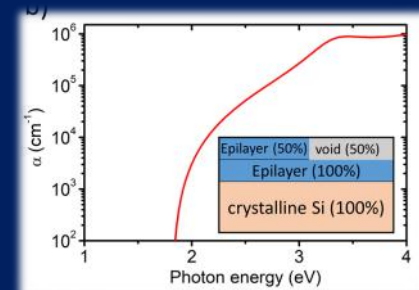
Optical constants and thicknesses



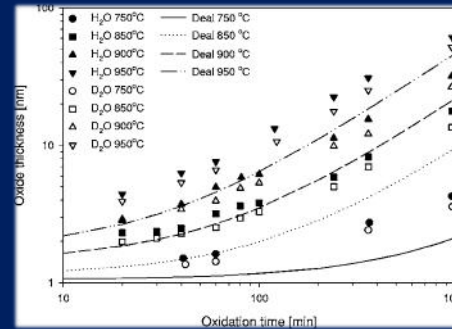
Systematic determination of optical constants and thicknesses

Roughness of surfaces and interfaces

→ Roughness of surfaces and interfaces is a fitting parameter

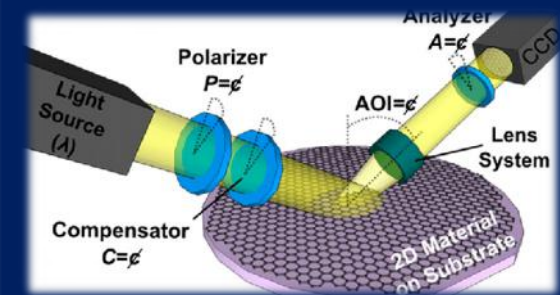


Oxidation of surfaces



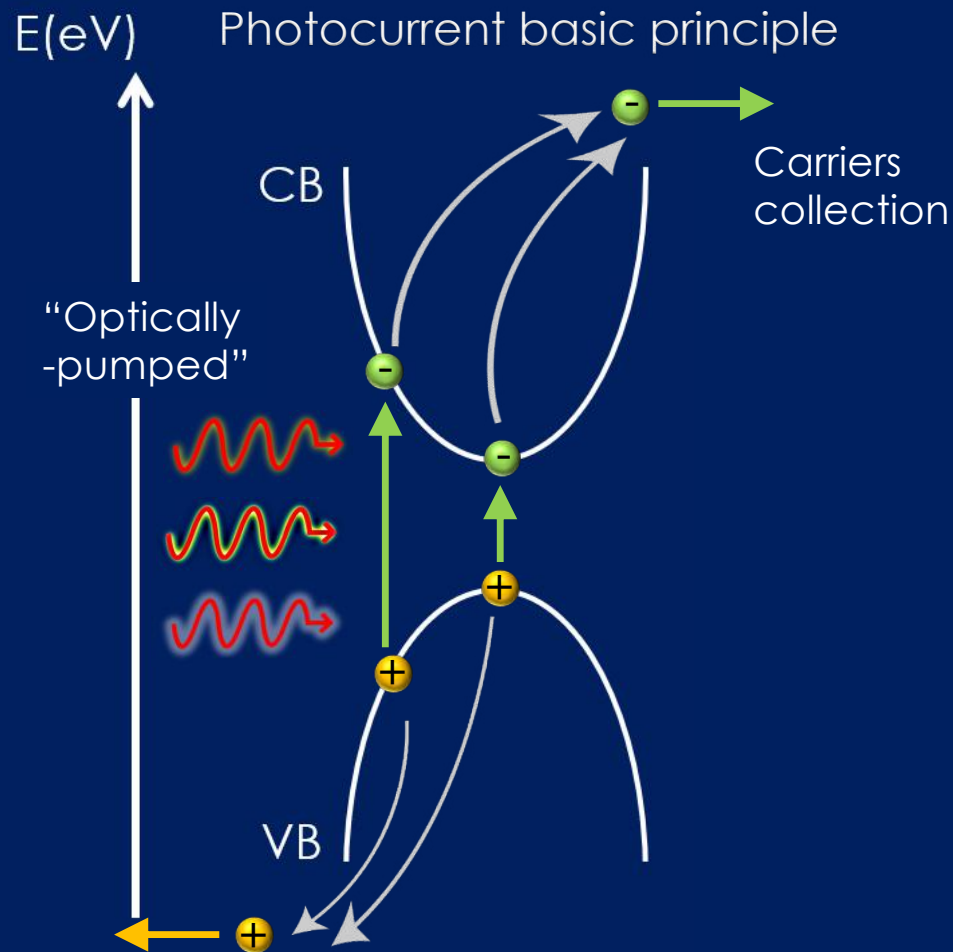
→ Thicknesses as low as 1 nm can be detected

Optical constants of 2D materials



→ Advanced applications (mapping)

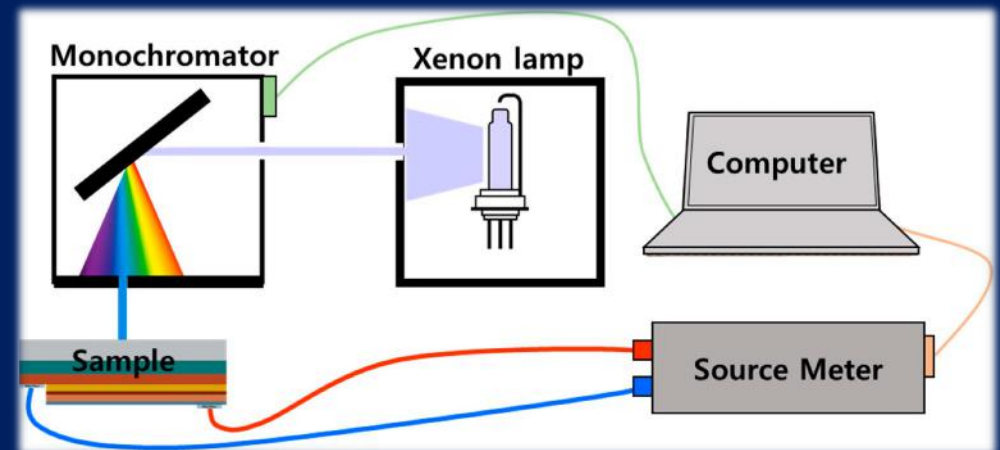
PC basic principle and setup



Photocurrent basic setup

Sources :

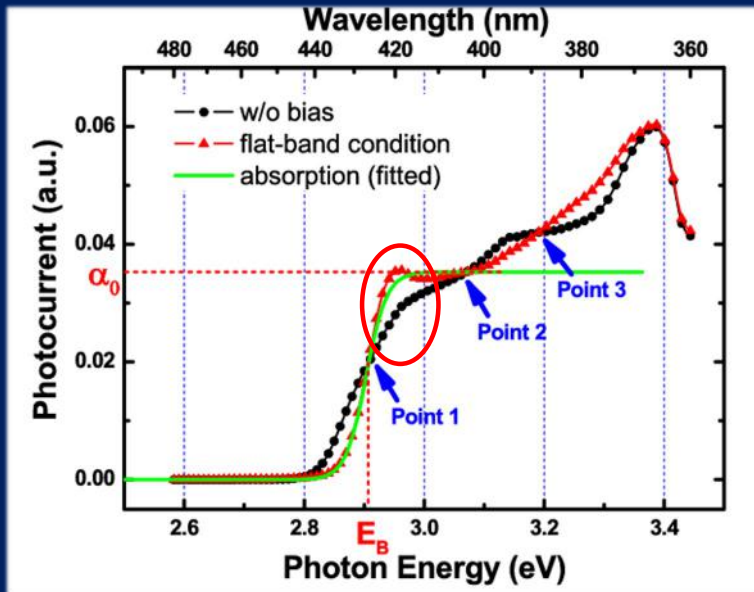
- Broad band light source + monochromator
- Tunable lasers



Collection of electrical signal

➔ Evaluation of the absorption + photo-generated carriers extraction efficiency

Analysis of carriers injection/collection

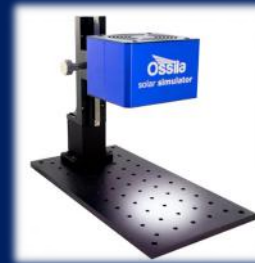


Ex : InGaN/GaN LED

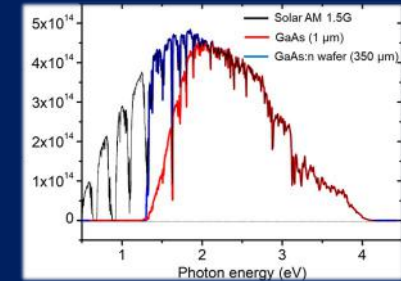
➔ Different carriers collection w or w/o bias, selective contacts, ...

Photovoltaic applications

- Solar AAA simulator

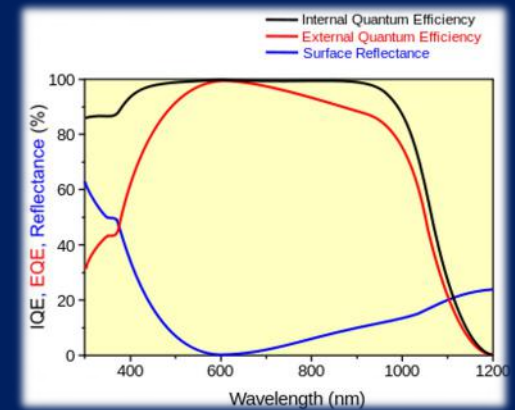


- Sample absorption



- Evaluation of the ability of the solar cell to generate photocurrent from the solar spectrum

➔ EQE, IQE



Absorbance measurements

- Measuring the absorption \approx measuring the JDOS
- Although defects theoretically contribute to absorption, not easy to detect experimentally
- Absorption useful for determining quantum confinement, alloying or band type (direct/indirect)
- Stokes Shift (PL & absorption) characterize the localization

Ellipsometry

- A powerful tool to determine optical constants of materials stacks
- Also interesting for roughnesses of surfaces/interfaces, oxidation, or 2D materials

Photo-current

- Allows to identify carriers collection issues
- Allows to identify specific features due to sunlight irradiation

Outline

I-Bandstructure of semiconductors, crystal defects and optical processes

- Bandstructures and semiconductors
- Crystal defects and their impact on optoelectronic properties

II-Characterizing light emission properties

- Photoluminescence
- Electro- & Cathodo-luminescence

III-Characterizing light absorption properties

- Absorbance measurements
- Ellipsometry & Photo-current

IV-Toward single photons sources

- micro-photoluminescence, $g(2)$



Single photon emitters

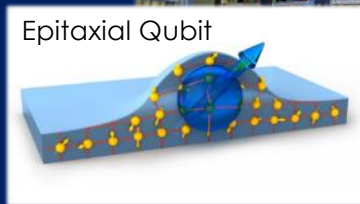
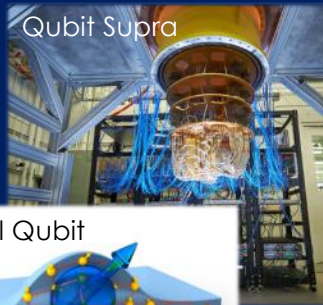
Quantum communications



- Quantum cryptography, Quantum key distribution

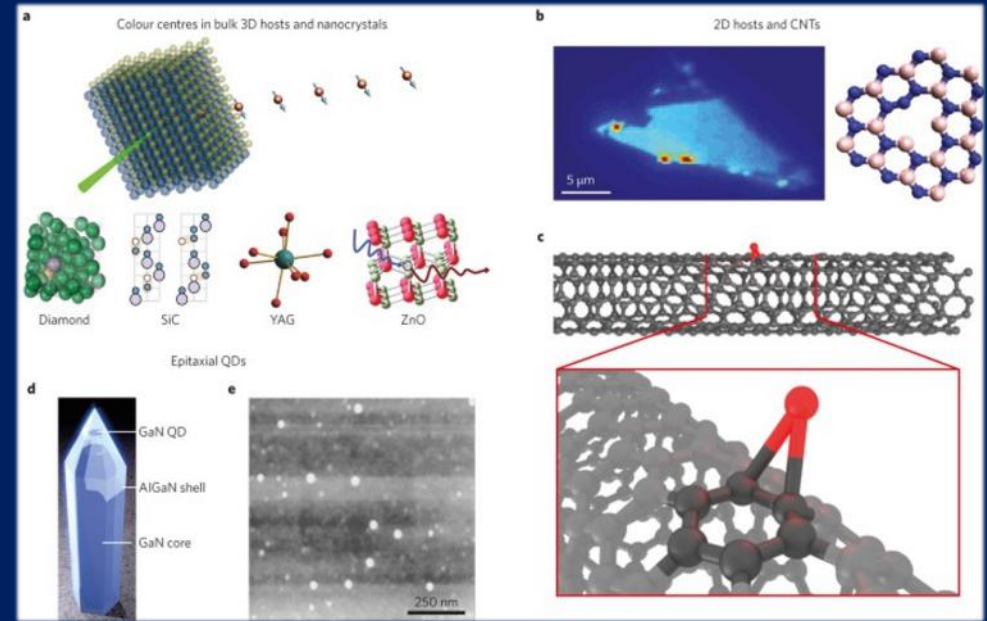
Quantum computing

- Qubit computing : fast, powerful



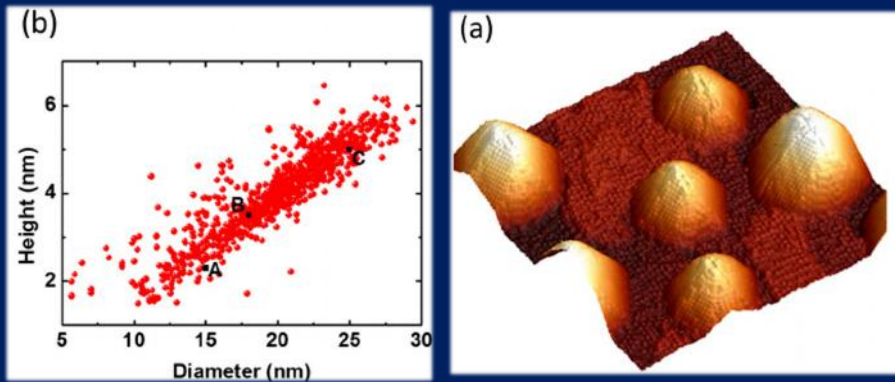
Single photon sources

- Quantum properties of single photons are of great interest for QT



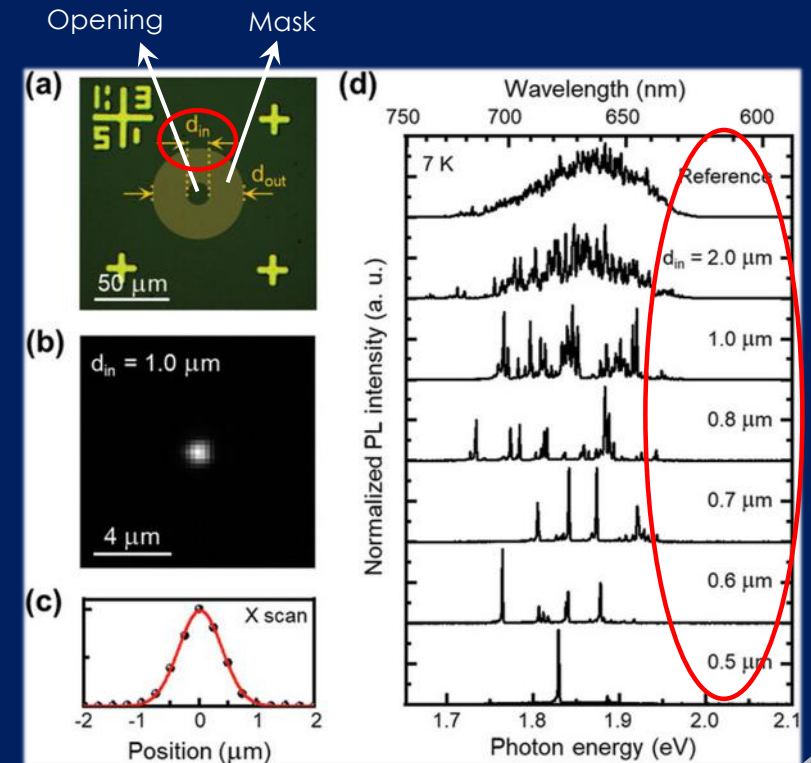
Many nanostructures were proposed for single photon emitters

Structural inhomogeneities of QDs



→ Structural inhomogeneities
→ inhomogeneities of Energy levels

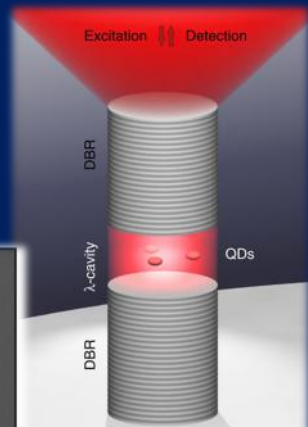
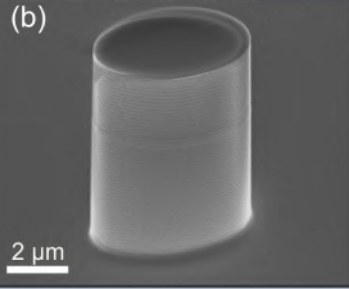
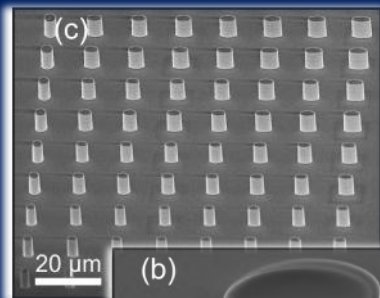
Inhomogeneous vs homogeneous broadening



→ Emitting window reduction to address single emitters

Individual emitters physical separation...

μ -pillars array

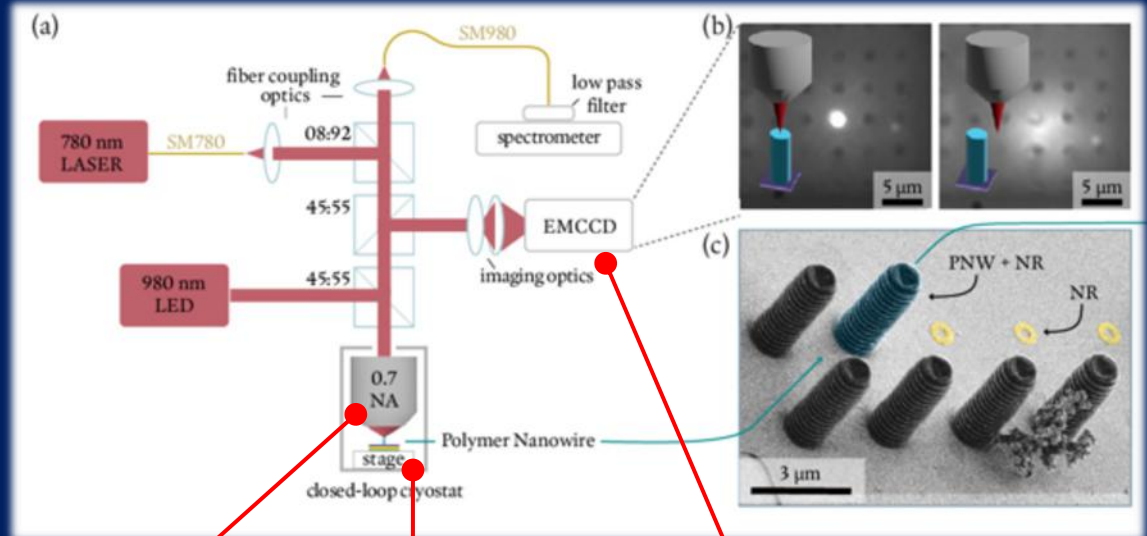


Individual QDs



Many different strategies to isolate individual emitters (pyramids, NWs...)

... and micro-Photoluminescence setup



High magnification lens

X-Y micro-positioning stage

Imaging optics for micro-positioning control

The single photon issue

About time statistics : How can we ensure that single photons are emitted ?

- o Temporal average is not sufficient :

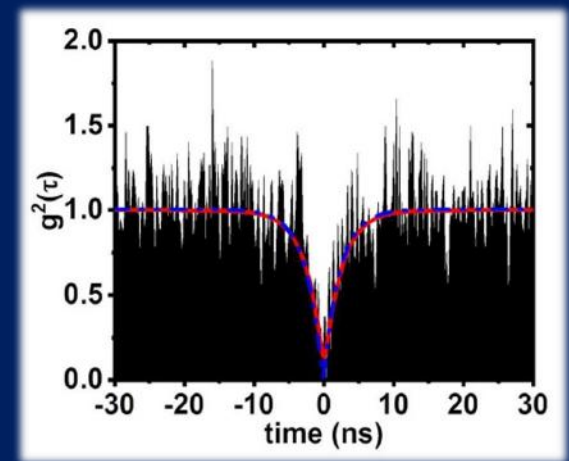
10 photons/s ~~↔~~ 1 photon every 0,1 s

➔ Autocorrelation in intensity $g^2(\tau)$:

-Probability of emitting a photon at time t , when a photon was emitted a time $t + \tau$

$$g^{(2)}(\tau) = \frac{\langle n(t)n(t+\tau) \rangle}{\langle n(t) \rangle^2}$$

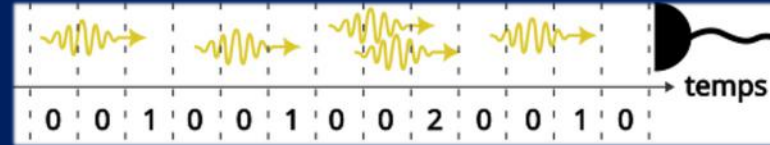
- For a single photon emitter, $g^2(0)=0$.



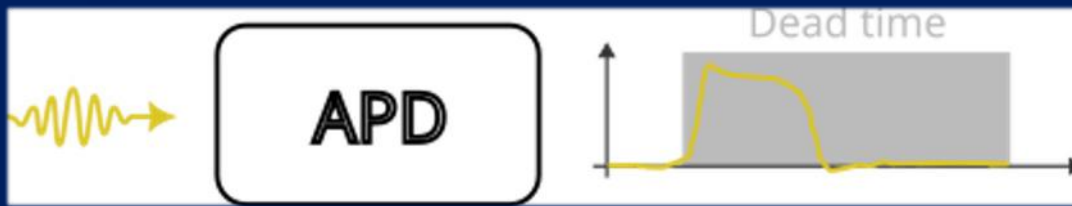
Measurement of the $g^{(2)}$

How can we measure experimentally the $g^{(2)}$?

- To measure $g^{(2)}$, one has to count photons



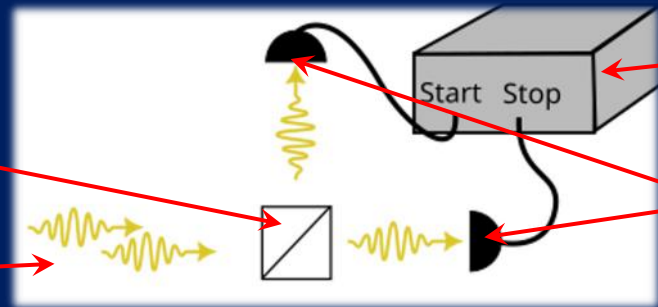
➔ Avalanche PhotoDiode (APD) can detect only one photon at a time, and convert it into electrical signal



- The experimental setup looks like :

Beam splitter

Photons Emitted from sample

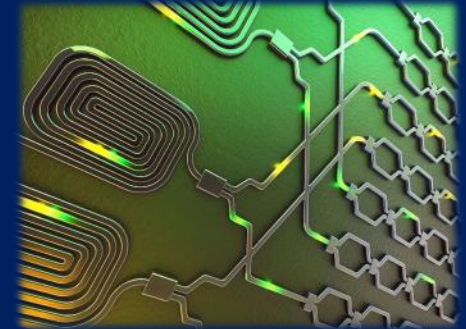


Correlator

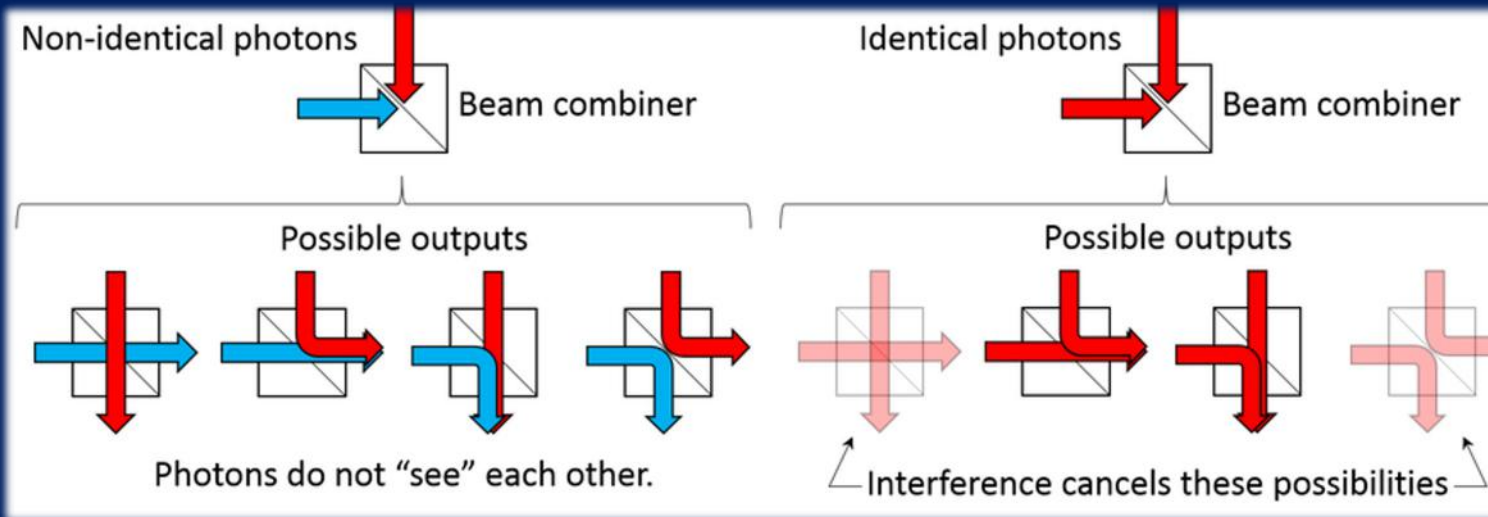
APDs

Motivations

- Quantum properties of 1 photon are interesting
- Quantum properties of a 2 photons - system are much more exciting
- Quantum states in a 2 photons-system : a fundamental step toward quantum technologies



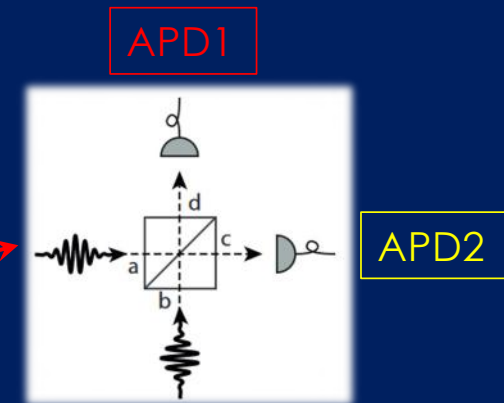
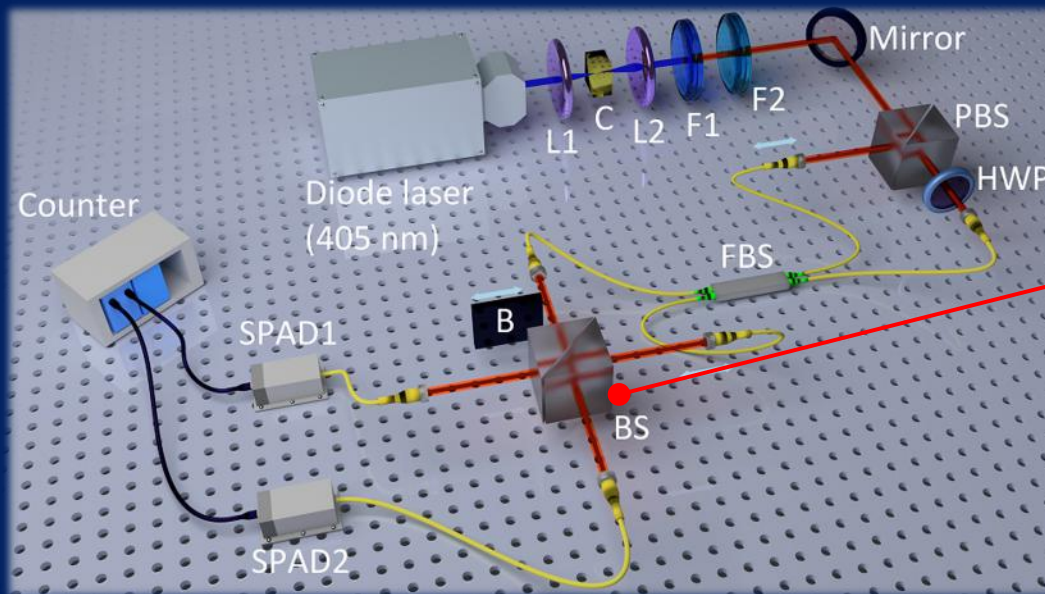
Properties of indistinguishable photons



- Hong-Ou-Mandel (HOM) effect = quantum interference

Measurements of the HOM effect

Similar setup than the $g^{(2)}$ but needs for two input signals



➔ If pairs of photons arrive always on the same photodetector, photons are indistinguishable

Single photons sources

- A large variety of nanostructures are now available for single photons sources, especially point defects
- $g^{(2)}$ experiments (correlation) are commonly implemented in Labs today.
- The control of quantum properties of 2-photons systems become a hot topic for the development of quantum technologies

General Conclusion

- Understanding optical processes in materials = understanding time constants and bandstructure
- Crystal defects in epitaxial materials have a huge impact on optoelectronic properties, not always detrimental
- (Electro-)optical characterizations of epitaxial materials provide number of useful information on crystal structure and bandstructure
- This information is crucial for the development of novel materials and devices, in the field of photonics, Energy applications or quantum technologies

For anyone wanting some bibliography about one of these topics,
please contact me directly at : charles.cornet@insa-rennes.fr

Thanks for your attention, questions ?

