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The LASEC contribution to the Ptolemy project

LAboratorio di Spettroscopie Elettroniche e di Correlazione

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LAboratorio di Spettroscopie Elettroniche e di Correlazione (LASEC) Electronic and Correlation Spectroscopy LAb.

Short story of LASEC

- □ The LASEC lab has been active in the physics of surfaces and interfaces since the origins of ROMA TRE (1995)
- General Founded by G. Stefani and A. Ruocco
- □ Investigation of surface and interfaces at sub-nanoscale
- □ Exstensive use of electron spectroscopy (XPS, UPS, EELS)
- \Box Investigation of electron correlation by Coincidence spectroscopy (e,2e) and (γ ,2e)
- Determination of the surface structure of metal semiconductor and interfaces

LASEC nowadays

- extensive experience in the study of carbon-based nanostructures
- Transmission of electron through single and multi-layer graphene
- □ Hydrogenation of carbon-based nanostructures
- Electronic structure of organic molecule-metal interface

The LASEC Experimental Chamber



Al K α source:

✤ hv = 1486.7 eV Monochromatized beam ✤ XPS resolution = 0.46 eV

He discharge lamp:

* Spot diameter 300 μ m ✤ hv = 21.2 and 40.8 eV

Custom-made monochromatic

Continuous electron beam

Tuneable energy 30 - 900 eV

Resolution = 45 meV

Contribution to the Ptolemy project

Hydrogenation of several carbon nanostructures:

- Graphene
- VA-CNT
- NPG

Stability in time of hydrogenated samples

Determination of the trasmission of graphene to electron in low energy range

- Measure of the integrated current through a suspended single layer of graphene in the 30-900 eV range
- Extend the measure to 5 keV
- Measure the angle and energy resolved spectra of electron scattering through a graphene single layer
- Determine the total and the resolved cross-section of the electron single layer graphene interaction

Detection of electrons in the 0-1500 eV range

Determination of the absolute efficiency of not energy resolved electron detectors: APD, MCP

• Electron energy analyser: a candidate for the detection of electrons including their energy







The GREEAT project, see next talk by Alice Apponi



Hydrogenation of Graphene on TEM



Atomic hydrogen source (H-source):

- FOCUS EFM-H
- Hot tungsten capillary •
- H₂ thermal cracking into H *

H-source operating conditions:

- H₂ pressure 1.8 3.6 10⁻⁶ mbar *
- Power 8 40 W
- H dose 10 kL = $3.6 \cdot 10^{-6}$ mbar · hour

Graphene on TEM:

- Flat suspended graphene regions
- Controlled number of layer (possibly 1)
- 2D material! No "probed depth" issues

Two Graphene on TEM Samples From Same Growth





Different Starting Point, Different Saturation Point







Sample A result:

- ✤ Start with ~13% sp³
- ✤ 59% sp³ saturation after 320 kL dose



- ✤ Start with ~42% sp³

Sample B result:

✤ 100% sp³ saturation after 260 kL dose



* π -plasmon ~ completely quenched

✤ Ni has losses at ~6 eV and ~3.5 eV

* π -plasmon almost quenched despite 59% sp³ saturation

Quenching of π -Plasmon: Ni Losses Is What's Left



* π -plasmon ~completely quenched

✤ Ni has losses at ~6 eV and ~3.5 eV

* π -plasmon almost quenched

Hydrogenated graphene:

- $rac{1}{2}$ sp² to sp³ distortion
- Band gap opening
- Electronic transition onset $\propto (E E_g)^{1/2}$ for direct gap semicondutors
- EELS measurement² and fit with a straight line
- ✤ With this analysis E_g ~ 6.2 eV for both samples

HANDLE WITH CARE:

- Background
- Excitons

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Intensity² [arb. units]

Intensity² [arb. units]



Hydrogenation of NPG: Different Depth Sensitivity



Hydrogenated nanoporous graphene (NPG): Synchrotron micro-XPS experiment hv = 350 eV (C 1s E_K = 60 eV)

- sp³/(sp²+sp³) ≈ 90% achieved



What's new then?

- ✤ hv = 1486.7 eV (C 1s E_K = 1200 eV)
- Electron energy loss spectroscopy (plasmon investigation)

Different depth sensitivity Eκ = 60 eV depth ~ 9 Å $E_{\kappa} = 1200 \text{ eV} \text{ depth} \sim 90 \text{ Å}$

Betti, M.G. et al., Nano Letters (2022), https://doi.org/10.1021/acs.nanolett.2c00162



Quenching of π -Plasmon: EELS Footprint of Hydrogenation



- Excitation associated to sp²
- Quenching due to sp³ changing

NPG: We Reached the 64% Saturation



XPS at Synchrotron: Hydrogenation Only Superficial



Stability of Hydrogenated Graphene in Ultra High Vacuum and in Air



Hydorgenation Stability: Good in UHV, Oxidation in Air



Almost unchanged

- Significant oxidation

Hydrogenation Very Stable in UHV







Binding energy [eV]



Relative intensity



Measuring the Absolute efficiency of a Micro Channel Plate (MCP) at Roma Tre



Two-stage MCP

Hamamatsu MCP F1551-21S

- * Outer size \bigcirc = 27 mm
- * Effective diameter \bigcirc = 14.5 mm
- * Channel \bigcirc = 12 μ m
- * Open Area Ratio 60%
- * Two-stage chevron
- * Bias angle ~8°
- * Max MCP supply voltage 2.0 kV





MCP supply voltage 1800 V
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★ ∆Anode +100 V



▲Shape of channel entrance (SEM image)



We measure the absolute beam current or MCP output



$$\epsilon = \frac{eN_{out}}{I_{in}}$$

The efficiency is ~49% with no energy dependence





- * Linear fit from min current to ~50 fA (300 kCounts/s)
- * Efficiency ~49% with no evident energy dependence within 30-900 eV

Third information: the peak width



The key ingredients for measuring detector efficiency

Monogun at Roma Tre:



- max count rate for MCP in single electron count mode: 300 kHz
- minimum stable current from e-gun
- capability to measure the low current emitted from e-gun (picoamm. Keysight B2987A)



Monogun Main features

Energy range	30 – 900 eV
Energy resolution	45 meV
Current - range	10 fA – 100 nA
Current - resolution	200 aA
Spot size diam.	< 0.5mm

on count mode: 300 kHz 10 fA

nt B2987A) < 1fA

Measuring the Absolute efficiency of a Avalanche Photo Diode (APD) at Roma Tre



Windowless Silicon APDs by Hamamatsu

Ordered **two** models of windowless APDs

- **Both** have sensitive area: $\emptyset = 3 \text{ mm}$
- We tested S11625-30N (normal structure)
- Will test also S12435-30 (backlit)

There's still a SiO2 inert layer

- Unavoidable, even if windowless
- Unknown thickness
- Will electrons pass it?







We measure the absolute beam current or APD output













e-gun



APD response to electrons

Fixing $E_{gun} = 500 \text{ eV}$ and $V_{APD} = 355 \text{ V}$

500 eV **clear** linear dependence: IAPD = k Igun • Intercept consistent with zero • The angular coefficient related to the APD gain



Application of Electron Energy Analysers in the Ptolemy Project



Electrostatic Electron Analyser



- standard in photoemission spectroscopy

An electron analyser is made of three parts: Lens system: collect a specific solid angle, (de)accelerate e⁻ Energy selector (pass band filter) Detector (single or multichannel)

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- -

Electrostatic Electron Analyser: a possible alternative to TES



- hemispherical analyser at the e.m. filter end
- position sensitive detector for parallel acquisition

position sensitive detector



Two concentric hemispheres for the energy selection





- Analytical solution of the trajectory
- Electron with kinetic energy **Ep** travel along the central orbit
- select a band of energy around E_p

Applied potential to hemispheres

$$V_{in} = 2E_p \left(\frac{R_0}{R_{in}} - 1\right) > 0 \qquad V_{out} = 2E_p \left(\frac{R_0}{R_{out}} - 1\right) < 0$$

Energy resolution (bandpass energy) $\frac{\Delta E}{E_p} = \frac{x}{2R_0} + \frac{\vartheta^2}{4}$ $\begin{cases} \Delta E: \text{ energy resolution}\\ E_p: \text{ pass energy}^{34}\\ x: \text{ slit width}\\ R_0: \text{ mean radius}\\ \theta: \text{ accepted angle} \end{cases}$

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x₂ position at the exit slit as a function of:
x₁ position at entrance slit

E kinetic energy of incoming electron

 $- \theta$ incidence angle at entrance slit

-

linear energy dispersion at the exit slits entrance angle and position give a minor contribution with respect to the electron energy

Position sensitive detector for parallel acquisition



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Electrostatic lenses increase flux and resolution



Freedom to choose the pass energy

The Ptolemy exercise: input parameters



ested	50 meV
oint of e.m. filter	10 eV
at the exit of e.m.	(??)
o the end-point	10 eV

The Ptolemy exercise: the electrostatic lenses

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The LASEC group: Alice Apponi, Orlando Castellano, Daniele Paoloni, Martina Chirico, Simone Ritarossi, Francesco Offi, Alessandro Ruocco

Main collaborations:

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