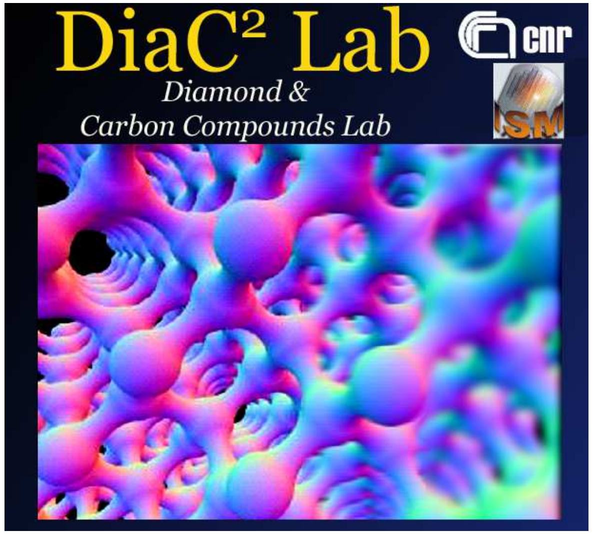

DiaC²Lab
Diamond and Carbon Compounds Laboratory

Activities on diamond-based devices and detectors at CNR-ISM

Marco Girolami

Istituto di Struttura della Materia (ISM)
Consiglio Nazionale delle Ricerche (CNR)
Roma, Italy

DiaC² Lab is inside the CNR Research Area of Roma 1
Via Salaria km. 29,300
00015 Monterotondo Scalo (Roma), Italy

A. Bellucci – PhD Student – Solar & Thermal Energy Converters
P. Calvani – Researcher – Device Technology & Surface Devices
M. Girolami – Researcher – Ionizing Radiation Detectors
D. M. Trucchi –Senior Researcher – Team Leader

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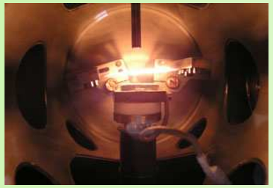
Consiglio Nazionale delle Ricerche

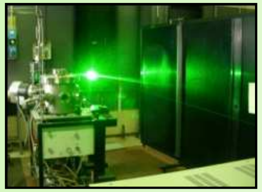
DiaC² Lab Facilities

Material Production

Microwave CVD ASTEX 1500 (2.45 GHz)
- Diamond film deposition (up to 4'') on Si, Mo, Carbides
- Nitrogen incorporation
- Hydrogen p-type surface doping



Hot Filament CVD
- Diamond film deposition (up to 4")
- Boron p-type doping



Pulsed Laser Deposition (PLD)
- Nanostructured thin-film deposition of carbon, carbides, refractory metals
- Excimer (ArF, KrF), Nd:YAG, Femtosecond Ti:Sapphire

Characterization

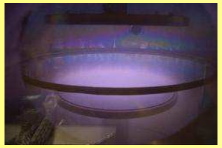
Raman & IR spectroscopy

Spectral Photometry (200-2000 nm)

SEM - EDS

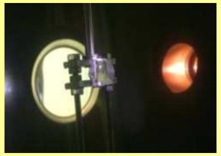
AFM

Technological Processes for Device Fabrication

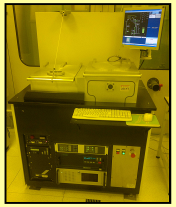


MW-CVD
- Surface Hydrogen Termination
- Thermal Annealing (up to 700°C)

RF & DC Sputtering
- Ti, Al, Cr, a-C, HOPG, WC, Ag, Au
- Up to 300°C
- Substrate biasing



Femtosecond Laser Treatments
- 3D Structures
- Cutting and Drilling
- Up to 600°C, RF Plasma Enhanced



Reactive Ion Etching (RIE)
- 3 D Structures & Micromachining
- 1000 W RF Power (13.56 MHz)
- Ar, O_2

Optical lithography
- 800 nm resolution
- Up to 4" masks
- Direct Writing System

Ultrasound Bonder
- 15 – 60 um wires
- Al/Si wires

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DiaC^2 Lab Facilities

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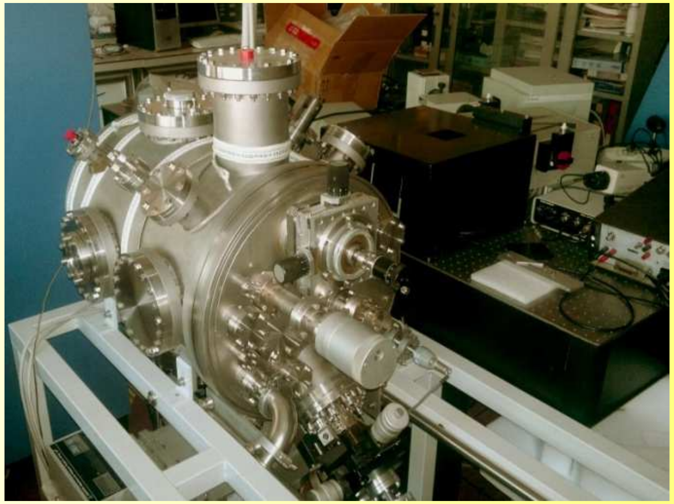
Characterization of Devices Performance



VTEC - Vacuum & Temperature Electronic Characterization

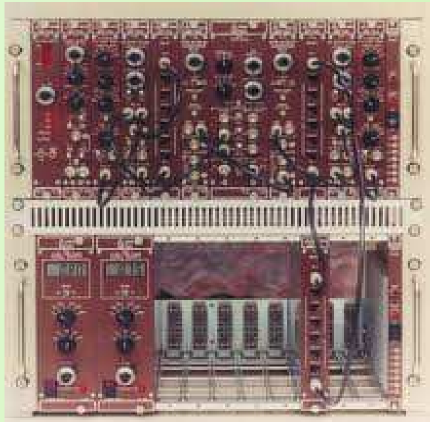
UHV 10^{-9} Torr
T = 77 - 1300 K

- Thermionic Emission
- UHV Field Emission
- Photoconductivity (T, λ = 200-1200 nm)
- Photo Emission Total Yield (T, λ = 200-1200 nm)
- I-V and C-V curves (Keithley and HP Instr.)
- Impedance Spectroscopy
- Four Point Probe

Detectors Characterization

X-Ray Photoconductivity
- Coolidge tube (Cu, Mo, W)
- Intensity modulation
- Frequency modulation

Climatic chamber analysis
- T from -40 to 180 °C
- Humidity value up to 98%
- Burn-in
- Ageing characterization



Time of Flight (TCT)
^{241}Am alpha particles in vacuum.

Alpha & Beta Spectroscopy
^{241}Am , ^{90}Sr
in collaboration with Univ. Roma Tre

Neutron Spectroscopy
14 MeV Frascati Neutron Generator
in collaboration with ENEA

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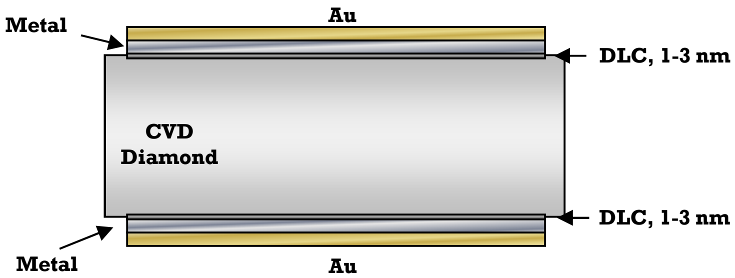
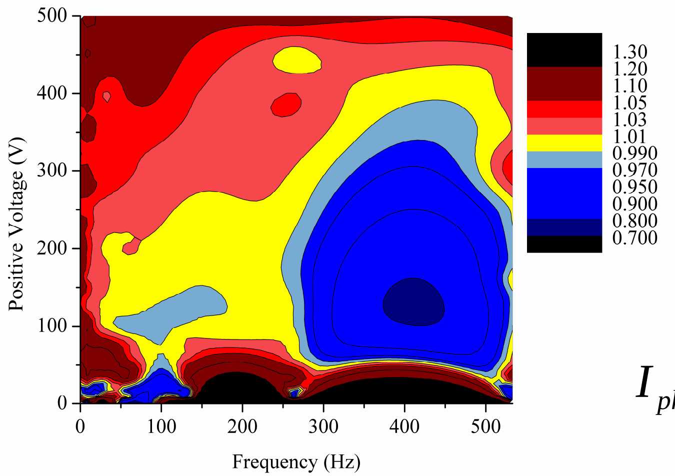
X-ray Dosimeters

Photoconductive (ohmic/diamond/ohmic)



- Best dosimetric performance for intermediate bias (1-3 kV/cm)
- Linearity with dose-rate (Δ=1) at high frequency needs a continuous increase of bias voltage



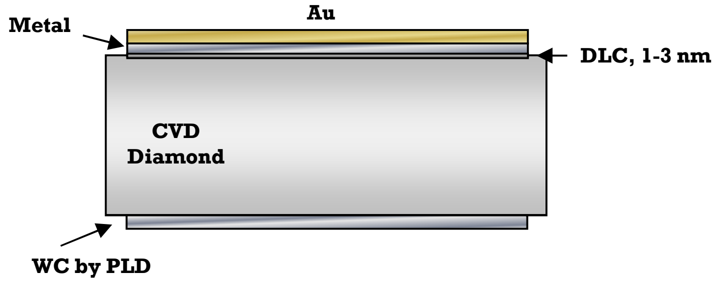
$$I_{ph} \propto DR^{\Delta}$$

Δ = 1.02 (slightly overlinear under CW operations)

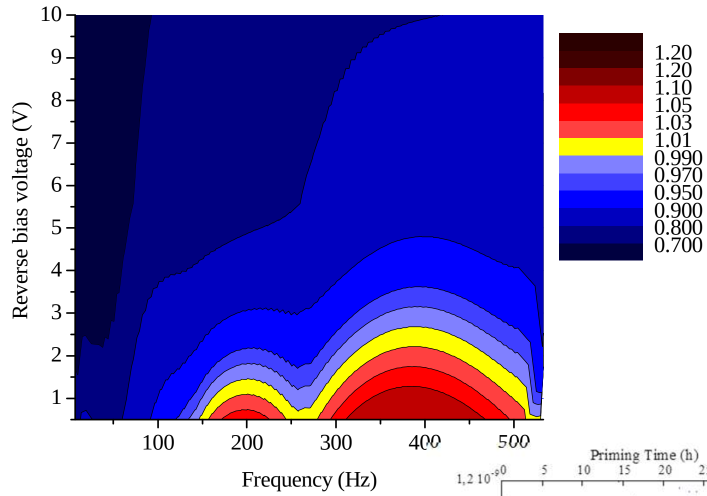
Specific sensitivity is about 60×10^{-3} C Gy^{-1} cm^{-3}

D.M. Trucchi et al., Nucl. Instrum. Meth. A 718 (2013) 373–375

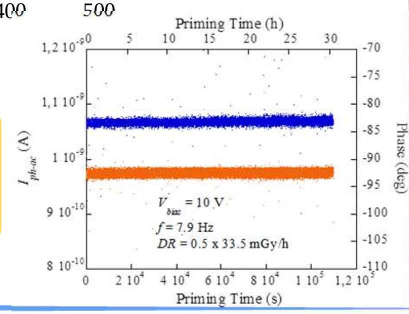
Photodiode (WC Schottky/diamond/ohmic)



- Fast response at low bias voltage and even in photovoltaic regime (@ 0V)
- Linearity with dose-rate (Δ=1) at low voltages and high frequency



Electronic-Grade plates do not suffer from priming



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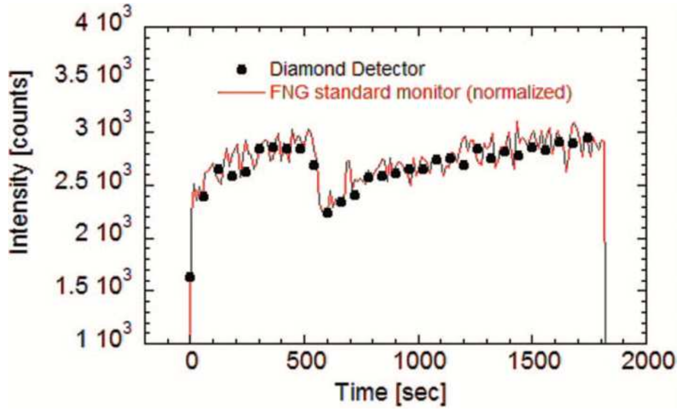
Single-pixel detectors for fast neutrons

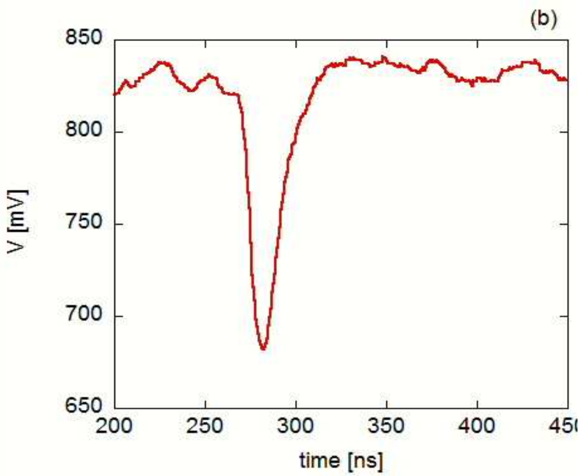
- 4.5 x 4.5 x 0.5 mm^3 Electronic Grade plates
- Ohmic contacts, alumina PCB, aluminum tracks

Installed at **FNG (Frascati Neutron Generator)** for flux monitoring of fast neutrons:

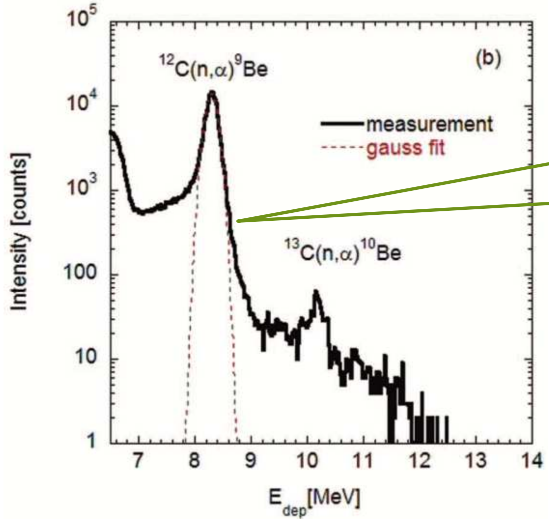
- **14 MeV neutrons**
- **2.5 MeV neutrons**

(relevant for neutron plasma diagnostics in nuclear fusion: tests ongoing at JET, UK)





Single pulse of a 2.5 MeV neutron
(fast amplifier Cividec C6)



Deposited energy spectrum of monoenergetic 14 MeV neutrons

2.5% (FWHM)

Good energy resolution for 14 MeV neutrons in DT plasmas at $E_n > 6$ MeV via $^{12}C(n,\alpha)^{9}Be$ reaction.

Inelastic neutron-carbon interaction $^{12}C(n,\alpha)$ ^{9}Be, followed by emission of an alpha particle from the ground state of ^{9}Be.

C. Cazzaniga et al., Review of Scientific Instruments 85, 11E101 (2014)

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Single-pixel detectors – Alpha particle characterization

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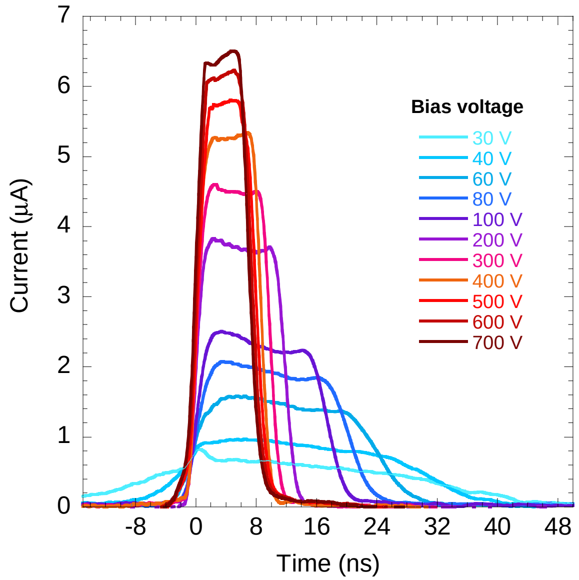
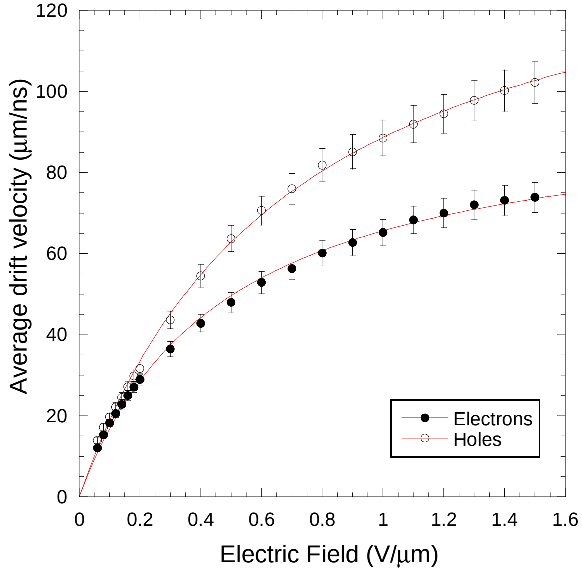
Transient-current-technique (TCT) with ^{241}Am (5.5 MeV)





Average drift velocity

$$\langle v_{dr} \rangle = \frac{\mu_0 F}{1 + \frac{\mu_0 F}{v_s}}$$

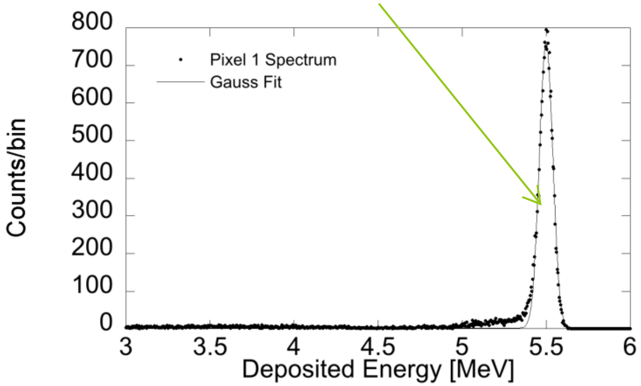
H. Pernegger et al., J. Appl. Phys. 97, 073704 (2004)

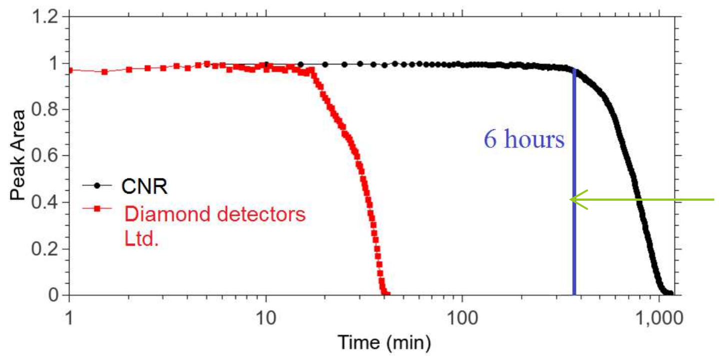
Electrons
μ_0 = 2050 cm^2/Vs
$v_s = 1 \cdot 10^7$ cm/s

Holes
μ_0 = 2170 cm^2/Vs
$v_s = 1.5 \cdot 10^7$ cm/s

Typical values of electronic grade samples

Energy resolution 1.73% (FWHM)





Detector stability: approx 6 hrs (alpha source with 37 kBq activity)

Signal recovered by inverting bias voltage

M. Rebai et al., Journal of Instrumentation 10, C03016 (2015)

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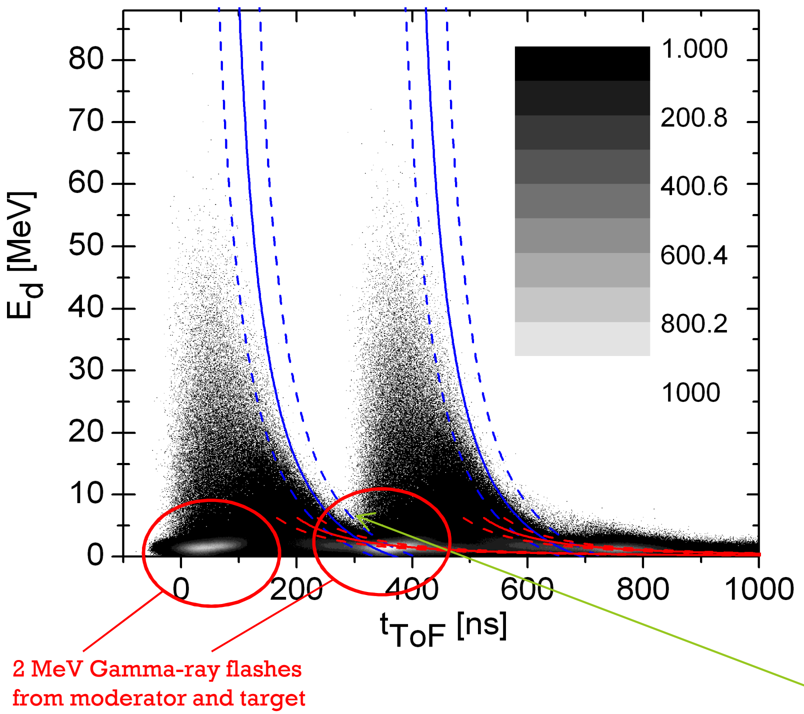
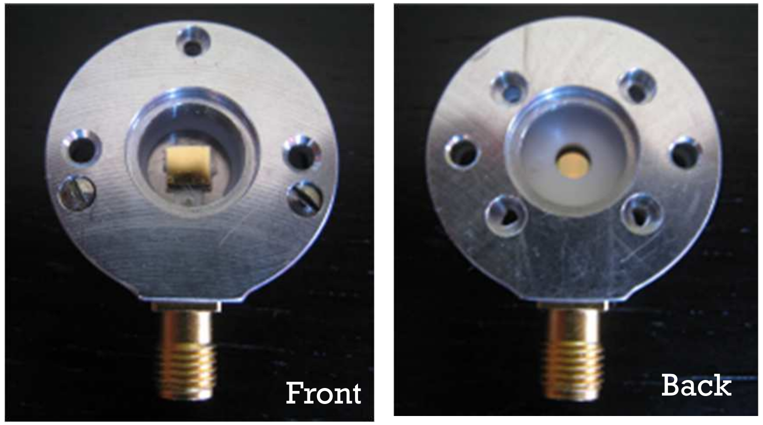
Single-pixel detectors at ISIS spallation neutron source

Biparametric measurements: Pulse-Height Analysis & Time-of-Flight





- Neutrons are produced by a 800 MeV proton beam with a double bunch fine structure (rep rate 50 Hz)
- The proton beam delivers an average current of 180 μA on a Ta-W target (15–20 neutrons per incident proton)
- The two proton bunches are about 70 ns wide (FWHM) and 322 ns apart.

The structure of the event distribution in the contour plot reflects the time structure of the two bunches in the proton beam.

- Events from the two bunches are well separated in time only for deposited energies **E_d >6 MeV**. For lower E_d values the two bunches overlap.
- Blue lines reflect the maximum possible E_d for the n-alpha reactions ($^{12}C(n,\alpha)^9Be$ and $^{12}C(n,n')3\alpha$).
- Red lines reflect the maximum possible E_d for elastic scattering (carbon recoil after neutron collision).

Possible application as a high-flux fast-neutron beam monitor at ChipIr beamline at ISIS

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Mosaic Detectors

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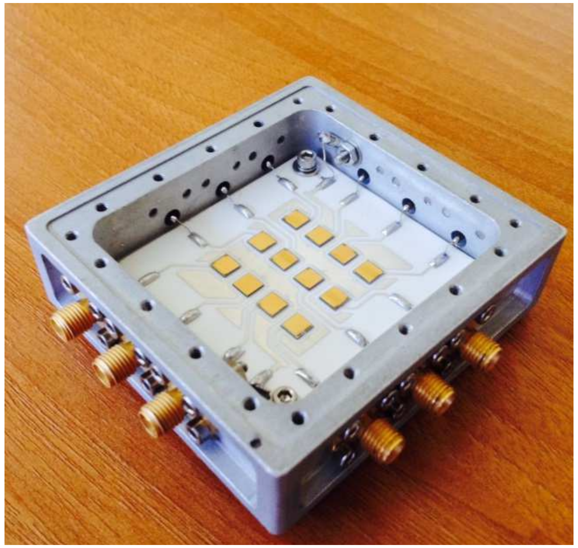
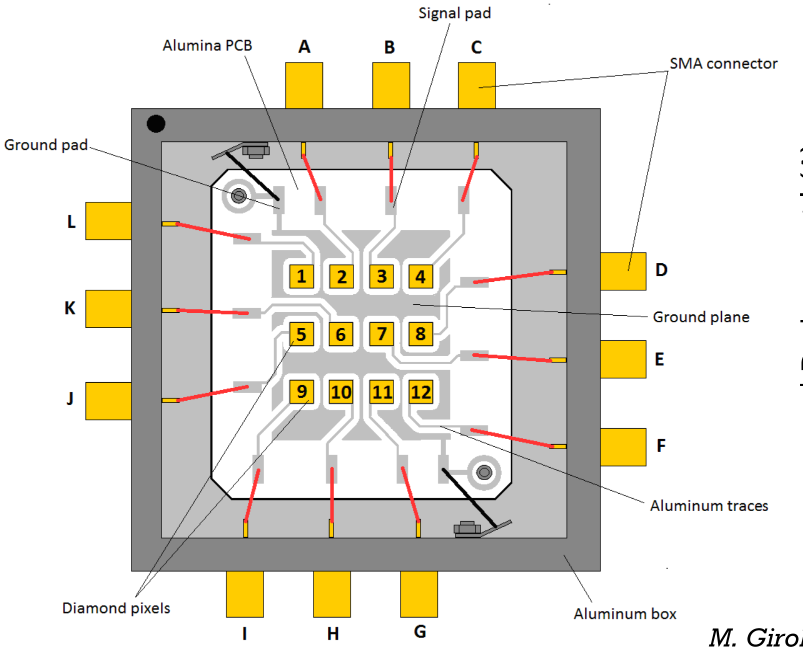
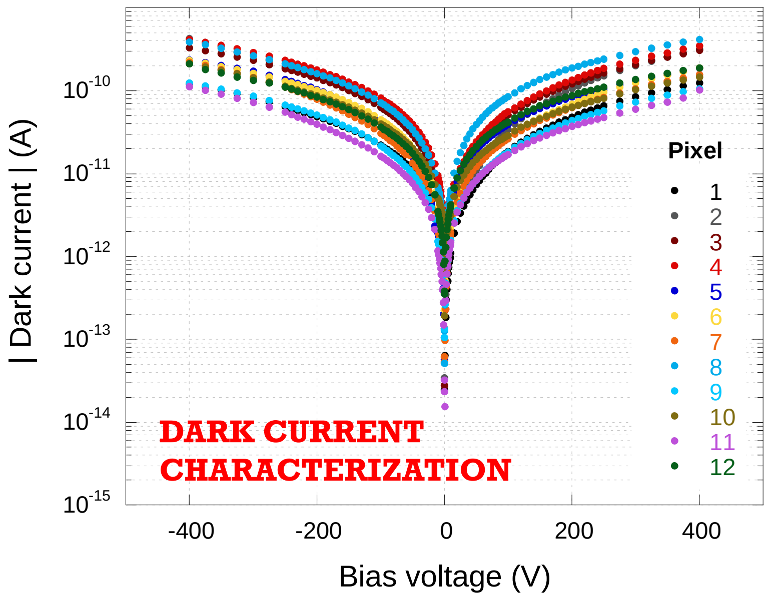


Main features of the first prototype:

- 12 single-crystal diamond pixels 4.5x4.5x0.5 mm^3
- Hydrogen-free 99.6% alumina PCB
- Aluminum tracks
- Ground plane to minimize cross-talk
- 2.5x2.5 cm^2 detection area (voids included)
- Operating voltage range: (- 400 V, +400 V)
- 12 standard SMA connectors

- Tested at CSNS (China Spallation Neutron Source)
- Recently installed at JET (UK) as part of the Vertical Neutron Spectrometer project.

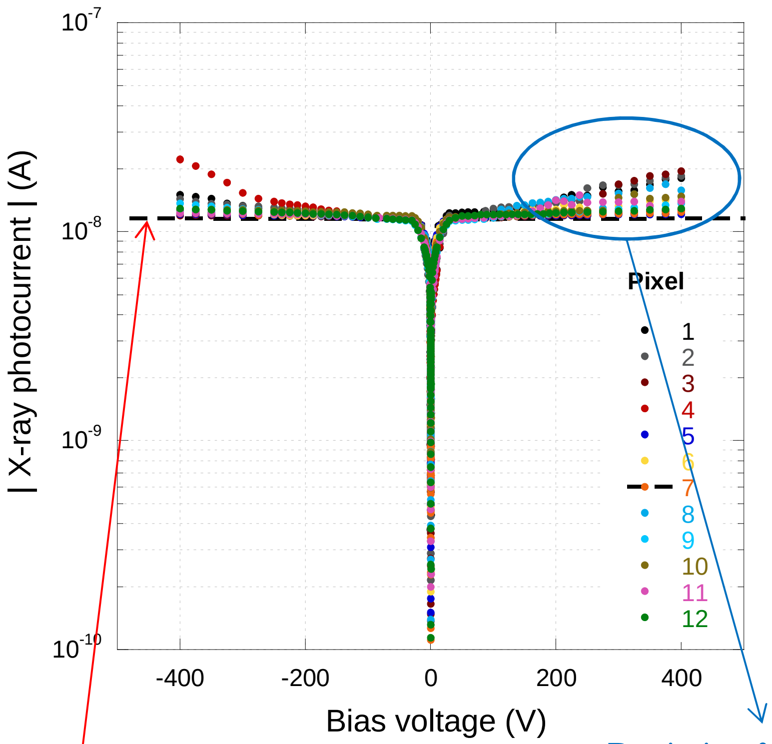




M. Girolami et al. ***"Mosaic diamond detectors for fast neutrons and large ionizing radiation fields"*** *– Physica Status Solidi A, Vol. 212, pp. 2424-2430, (2015).*

Mosaic Detectors – X-ray characterization

- Coolidge tube, filtered-Cu target (K_α @ 8.06 keV)
- Dose-rate 1 Gy/h (40 kV, 30 mA
- Circular 6mm-diameter spot

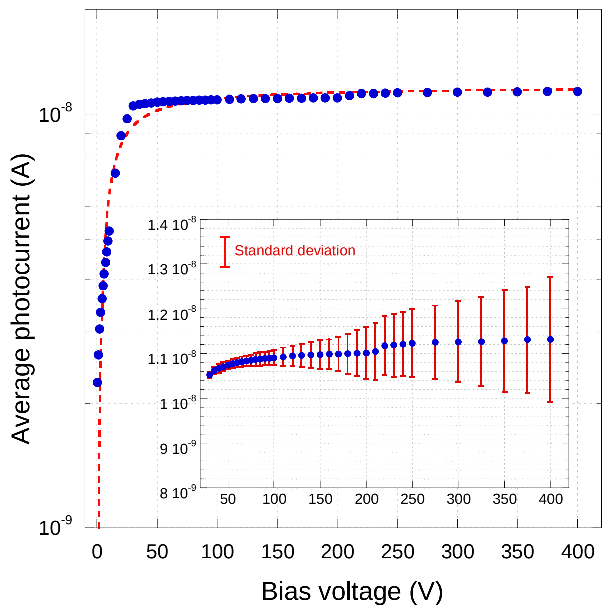


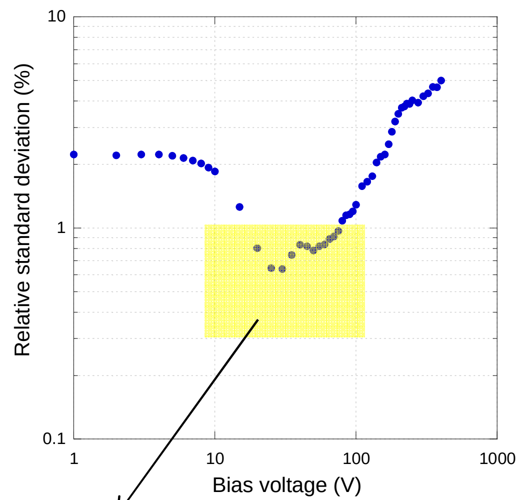
Deviation from Hecht at high bias voltage: **detrapping?**

$$|I_{ph}| \propto \frac{\mu\tau_R |V|}{d^2}\left[1-\exp\left(-\frac{d^2}{\mu\tau_R |V|}\right)\right]$$

Hecht's fit

Mobility-lifetime product
1×10^{-4} cm^2/V





RSD < 1% in the range 20-80 V : minimization of inter-pixel dispersion

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Mosaic Detectors – Beam profiling

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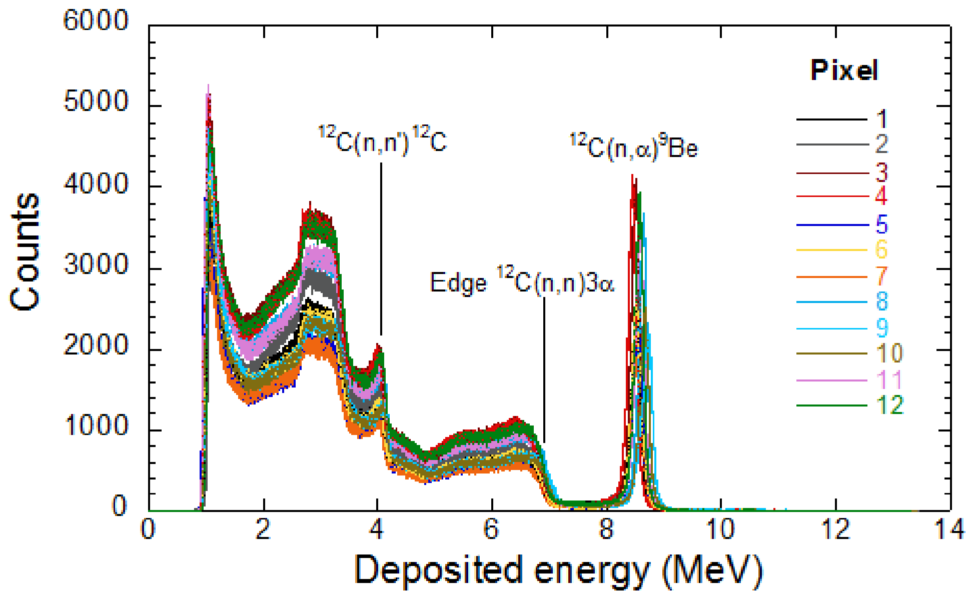
FAST NEUTRONS

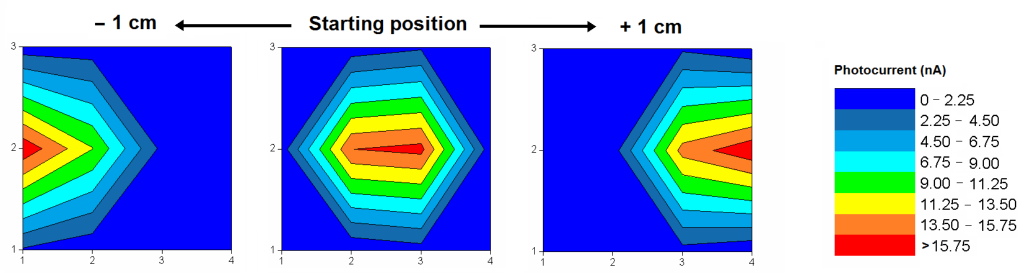


- FNG (Frascati Neutron Generator)
- 14 MeV neutrons (Deuterium ions accelerated on a tritiated-Ti target)
- Neutron flux: 5×10^{11} s^{-1}
- 12 simultaneous acquisitions with 12 fast charge pre-amplifiers (Cividec C6) + multichannel digitizer (CAEN V1730)

Pixel response non-uniformity could be due to:

- asymmetry of neutron yield at different neutron angles of incidence
- intrinsic broadening of incident neutron spectrum

X-RAYS



- X-ray (8 keV) beam profile
- Large (2cm) spot-size
- 12 simultaneous acquisitions with custom front-end electronics (integrator + ADC)

Resolution limited by the restricted number of pixels and voids in the active area (gaps between adjacent pixels)

M. Girolami et al. ***"Mosaic diamond detectors for fast neutrons and large ionizing radiation fields"*** *– Physica Status Solidi A, Vol. 212, pp. 2424-2430, (2015).*

AlN/Diamond Neutron Monochromators

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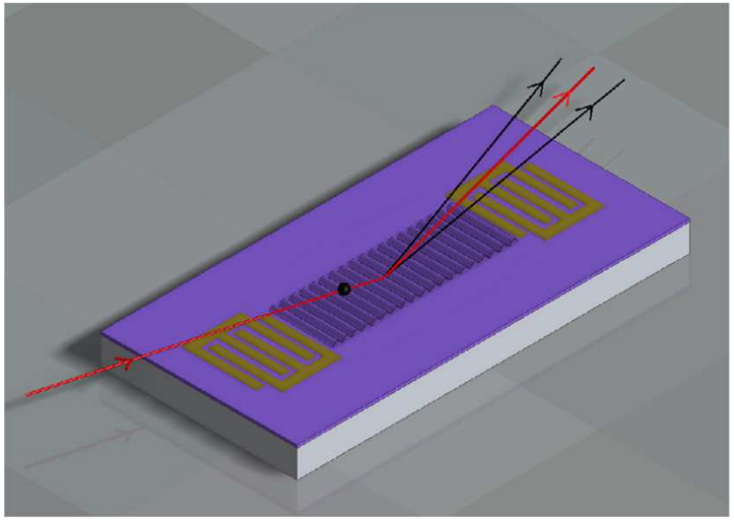
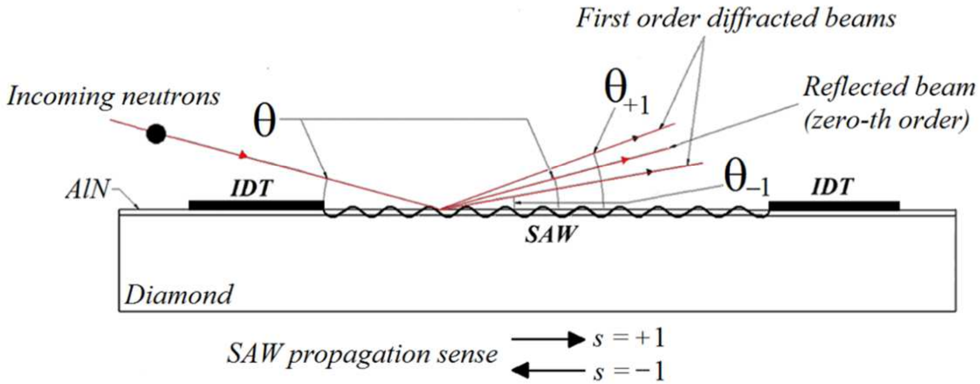
NEW CONCEPT DEVICE FOR NEUTRON MONOCHROMATIZATION

- **Thin (few hundreds of nm) film of aluminium nitride (AlN) deposited on a CVD diamond substrate.**
- **Surface acoustic wave (SAW) traveling between two metal IDT**





$$\theta_n^2 = \theta^2 + 2n(K/k)[\kappa/k - s]$$

Diffraction angles are greatly enhanced if SAW speed and frequency are high

$$K = \frac{2\pi\nu_s}{u_s}$$
SAW wavenumber

$$k = \frac{2\pi\nu_n}{u_n}$$
Neutron wavenumber

$$\kappa = \frac{m_n u_s}{\hbar}$$
Magnification factor for a traveling SAW

AlN/diamond resonator: u_s **> 10000 m/s**, ν_s **> 10 GHz**

DIFFRACTION EXTENDED TO EPITHERMAL NEUTRONS IN THE eV RANGE

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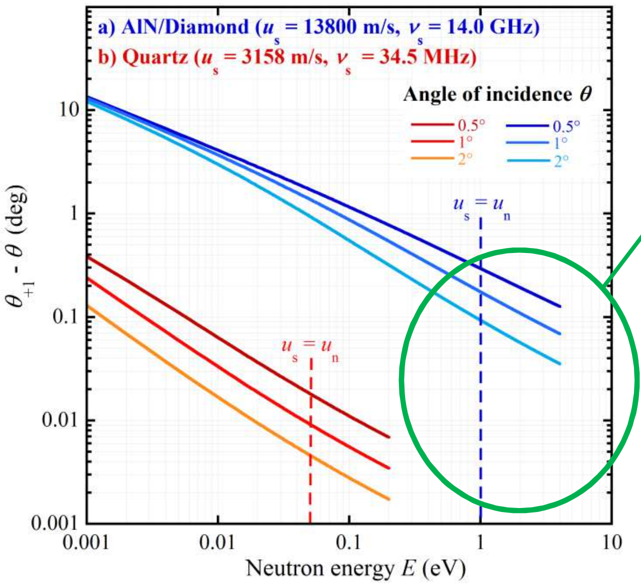
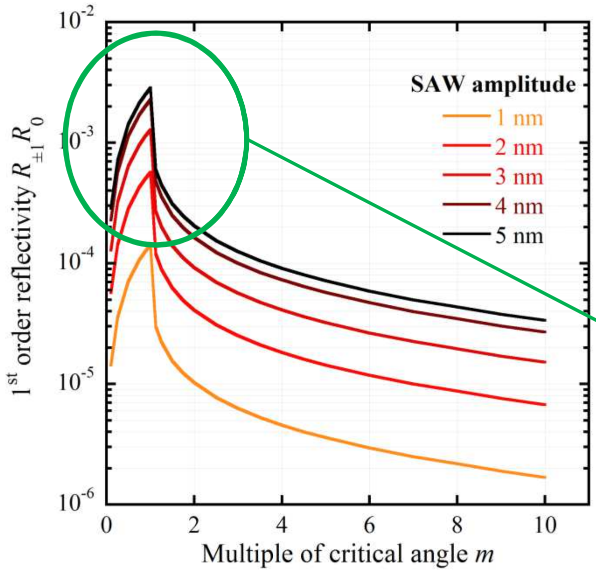
AlN/Diamond Neutron Monochromators

1° order diffraction angle



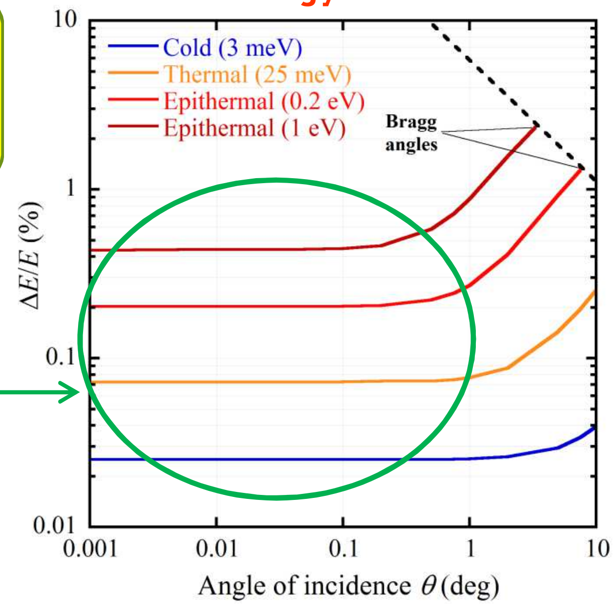


Neutron monochromatization effective up to eV range, where:

- **Crystal monochromators (Si, Ge, Be) have zero reflectivity**
- **Choppers have poor energy resolution (3 – 5%)**

Resolution < 1% in the eV range and < 0.1% for thermal neutrons

Energy resolution



Major drawback: low reflectivity (few ‰ at critical angle)

Solution: cascading devices ?

__M. Girolami__, A. Pietropaolo, A. Bellucci, P. Calvani, D. M. Trucchi
"High-resolution wide-range dynamic neutron monochromators"
EPL (Europhysics Letters), Vol. 109, Number 6, 66001 (2015).

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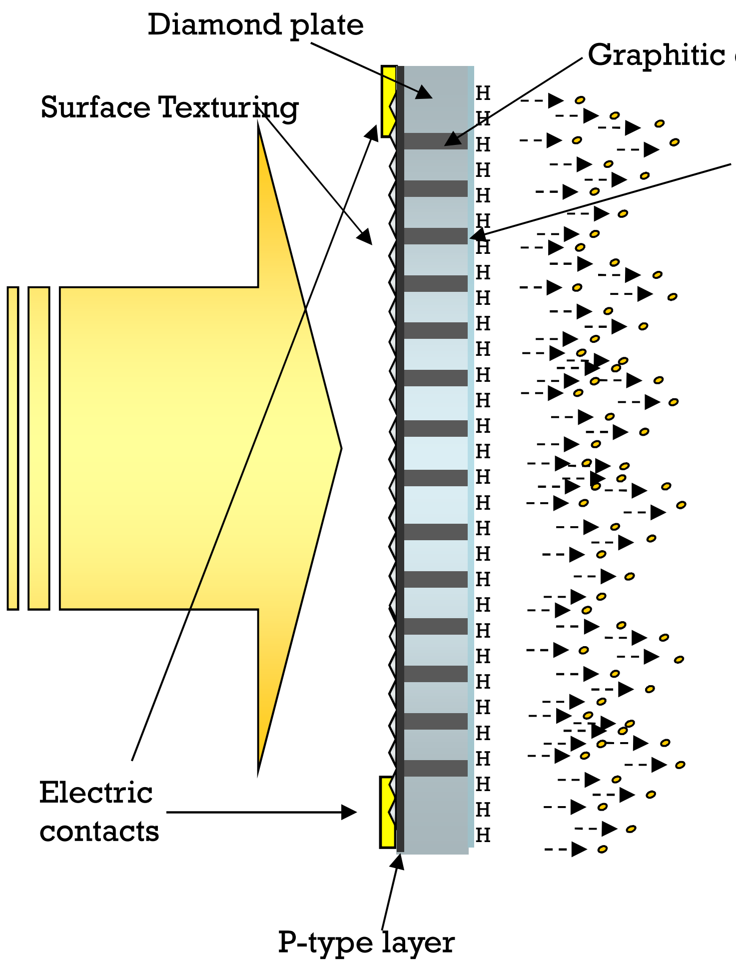
Diamond Treatment by Fs Laser for PETE cathodes

Goal:

Increase absorptance and photoconductivity in the solar spectrum for efficient conversion modules based on PETE (Photon-Enhanced Thermionic Emission)



Technological steps:

- Implantation of B ions on front surface for a buried p-type layer a) acting as a back electric contact; b) increasing the photogeneration contribution; c) separating e-h for reducing recombination
- Fs laser-induced distributed contacts for reducing device series resistance
- Fs laser front surface nanoscale texturing for increasing optical absorption and photogeneration
- CVD deposition of n-doped thin diamond layer (100-500 nm)
- Emitting surface hydrogenation for NEA
- Deposition of electric contacts

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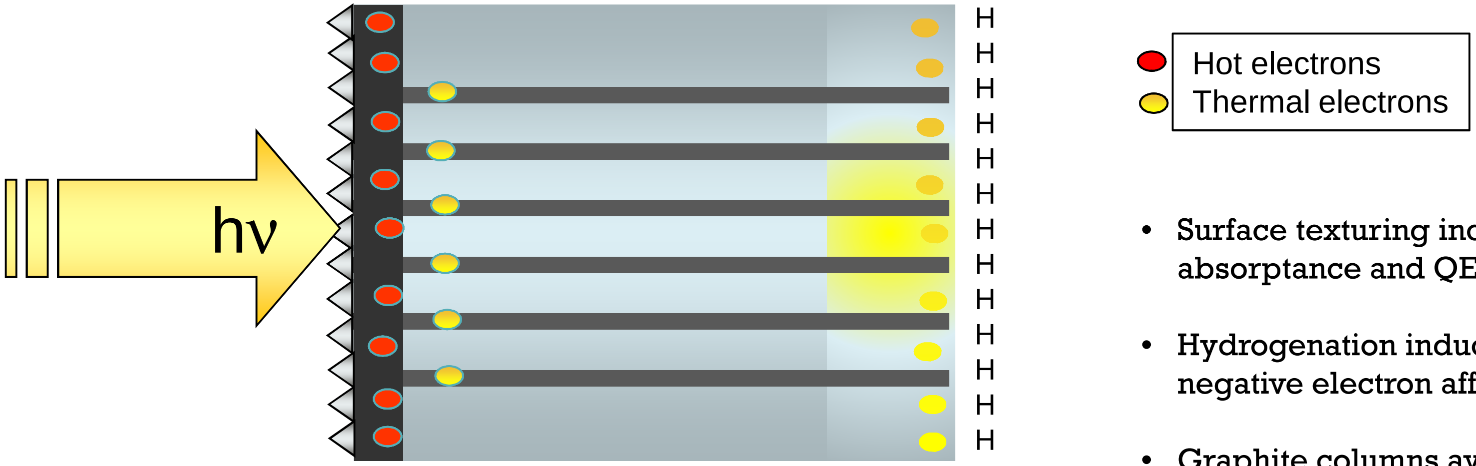
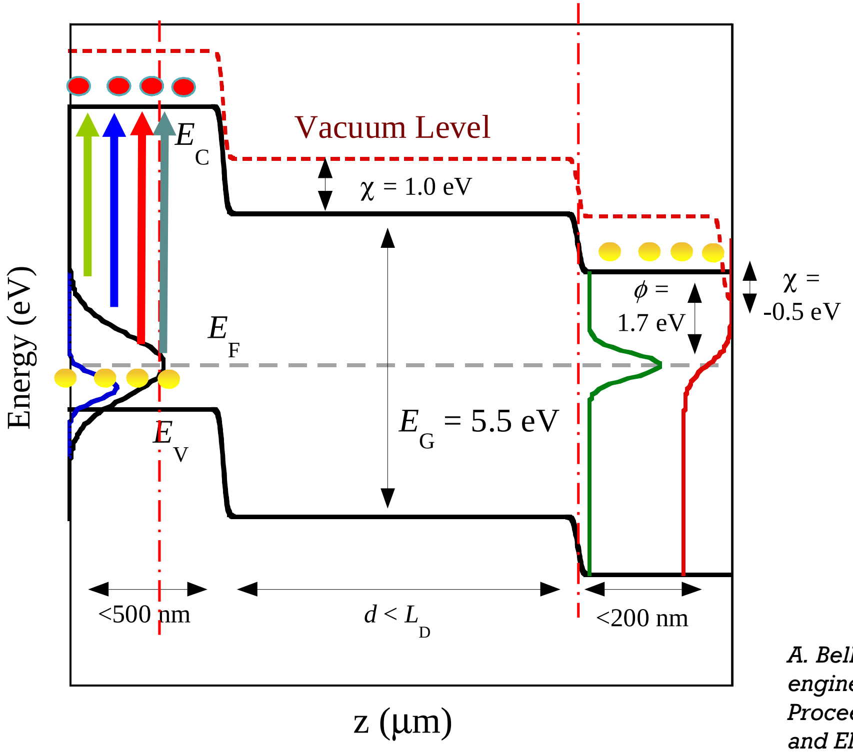
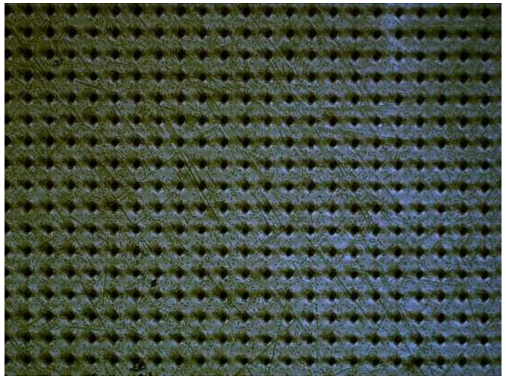
http://www.prometheus-energy.eu/

PETE – Band diagram





- Surface texturing increases absorptance and QE
- Hydrogenation induces negative electron affinity
- Graphite columns avoid bottlenecks in thermal electrons refilling



100x100 array of graphite columns (about 30 Ω total resistance)

A. Bellucci, P. Calvani, M. Girolami, D.M. Trucchi – "Defect engineering of diamond cathodes for high temperature solar cells" – Proceedings of IEEE 15th International Conference on Environment and Electrical Engineering (EEEIC), pp. 1616-1619 (2015).

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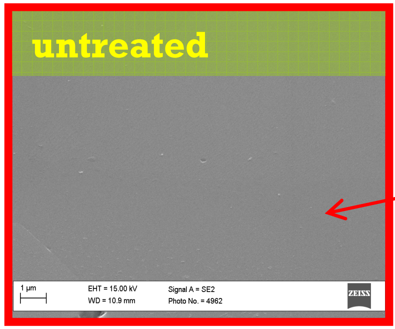
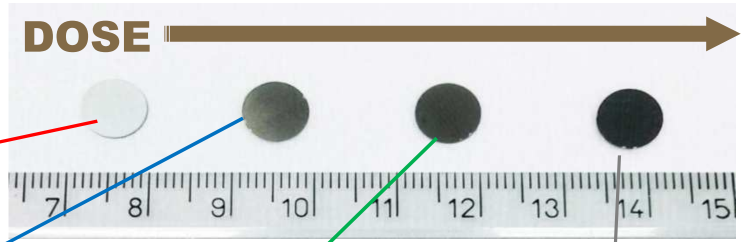
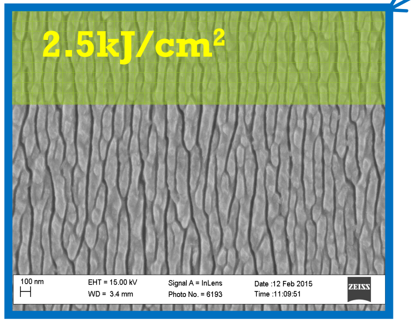
Black diamond

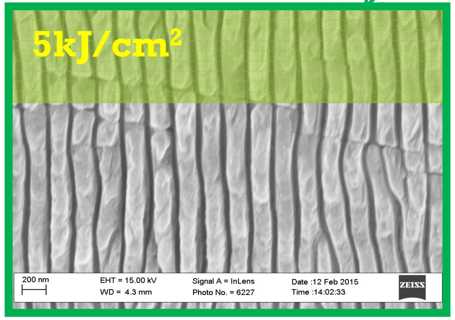
Marco Girolami- marco.girolami@ism.cnr.it

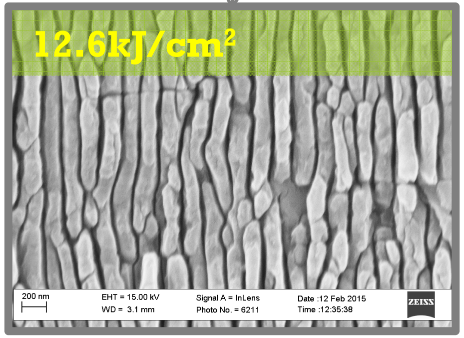




Femtosecond laser pulses are able to texture the surface of materials and dielectrics with periodic nanoscale structures without use of photolithographic steps

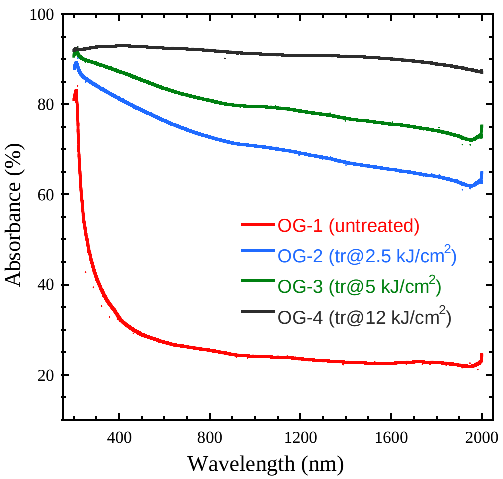


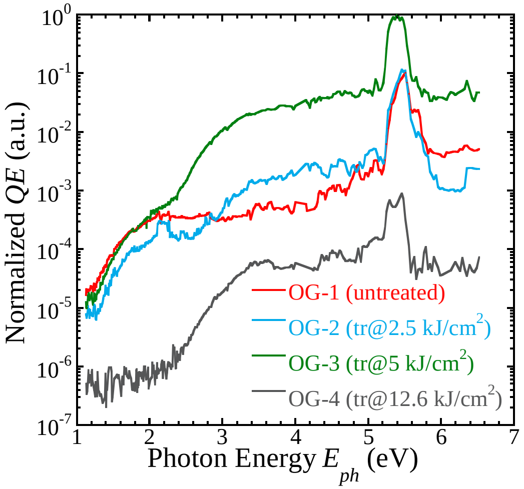




Surface structures with:

- Periodicity $\lambda/2n = 170$ nm
- Depth of 400 nm
- Length of several µm





Highest absorptance at the highest dose (grey curve)

Highest quantum yield at the intermediate dose (green curve)

↓

Trade-off between traps and recombination centers

P. Calvani et al. – "Absorptance enhancement in fs-laser-treated CVD diamond" – Physica Status Solidi A: applications and materials science, Vol. 212, pp. 2463-2467, (2015).

Consiglio Nazionale delle Ricerche

…just a clip

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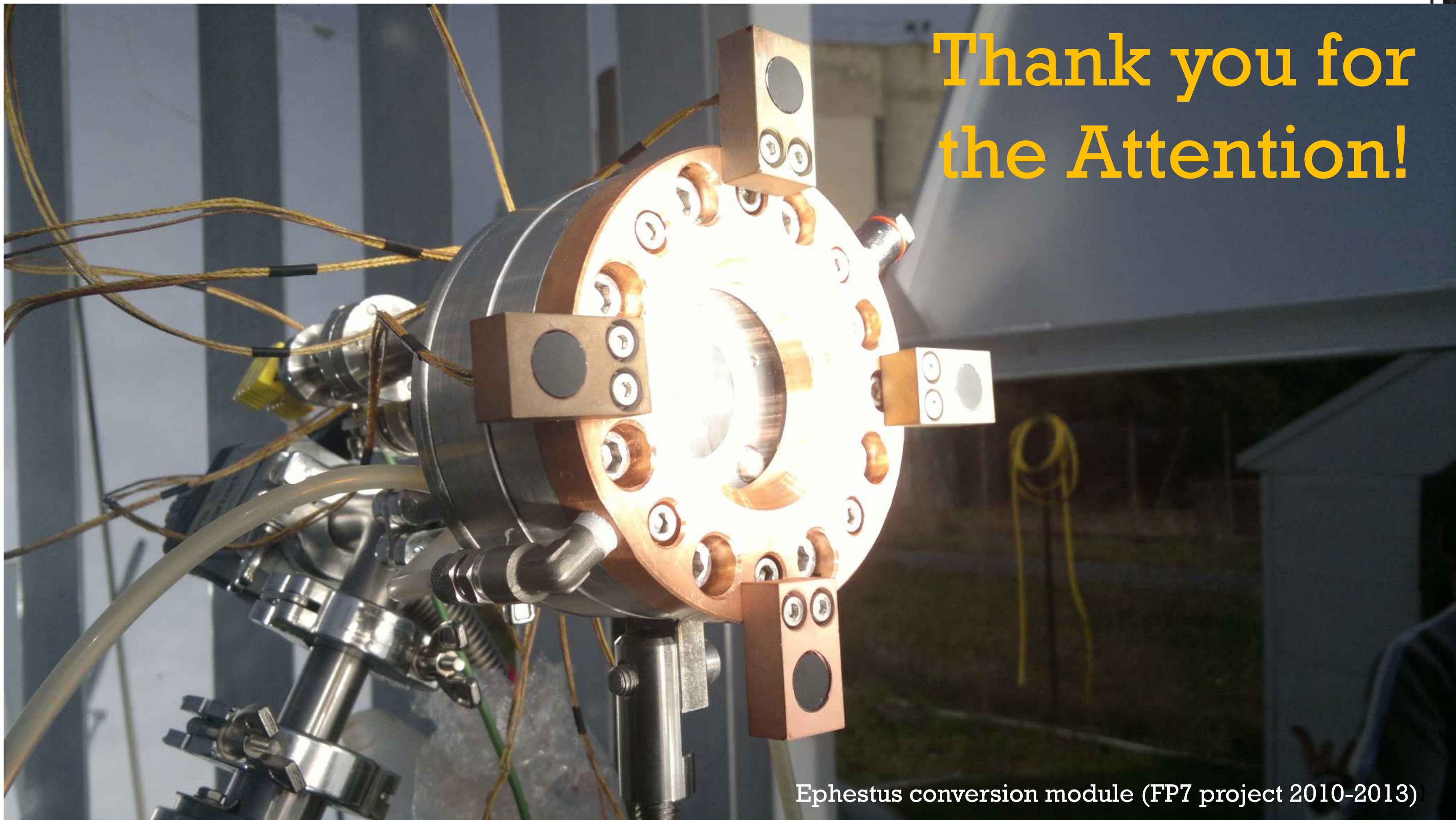
DiaC² Lab
Diamond & Carbon Compounds Lab

Thank you for the Attention!



Ephestus conversion module (FP7 project 2010-2013)