EAGLES
International Conference on
Rare-Earth Doped Glass Materials and Fibre Lasers
MPNS COST Action MP1401

cost EUROPEAN COOPERATION IN SCIENCE AND TECHNOLOGY

COST is supported by the EU Framework Programme Horizon 2020

October 18th – 19th, 2016 | Trento, Italy

CNR IFN Istituto di Fotonica e Nanotecnologie

POLITECNICO MILANO 1863

Local Surface Analysis and Laser Micromachining for Integrated Dielectric Platforms

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EAGLES Meeting

Oct 18th – 19°th, TRENTO, ITALY

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Local Surface Analysis and Laser Micromachining for Integrated Dielectric Platforms

Alberto Tagliaferri

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CNR – IFN @ PoliMI

POLITECNICO DI MILANO

http://www.polimi.it

- 20 staff researchers
- 36 associated researchers
- students, PhD students and post-doc

http://www.mi.ifn.cnr.it/

http://www.fisi.polimi.it

@ PoliMI Dept. of Physics

Research lines:

- Quantum optics and quantum information
- Femtosecond Laser Micromachining
- Nanotechnology and Microscopy
- Photonics for health, food and cultural heritage
- Solid-state lasers and amplifiers for metrology and spectroscopy
- Ultrashort light pulse generation and ultrafast phenomena in matter

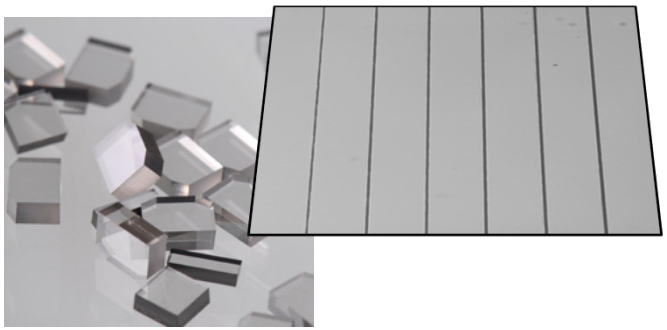
Outline:

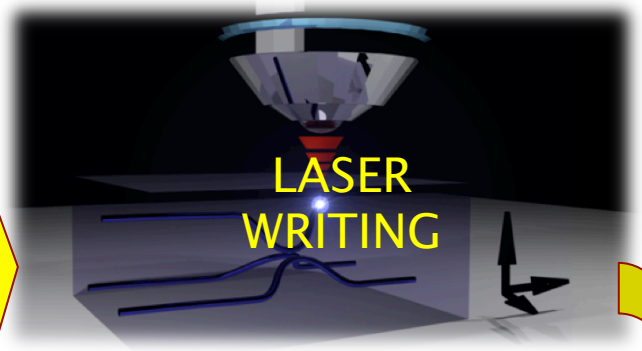
- Rationale: surface machining by fs-laser and surface inspection at the micro- and nano-scale
- Scanning Auger Microspectroscopy (SAM):
  - Local composition and oxidation assessment
  - Absolute thickness measurement: Thin film overlayers and 2D materials

- The case of diamond micromachining:
  - Surface and volume writing of waveguides by fs laser beams
  - Detailed analysis of nanoscale features by SEM

Rationale:

THE PROCESS OF fs-pulsed laser micro-machining of structures in "*optically trasparent*" platforms





- Morphology at micro-nano scale
- Metrology
- Local composition / state of oxidation
- Crystalline structure

- Absorption
- Refractive index
- …..

Rationale:

THE PROCESS OF
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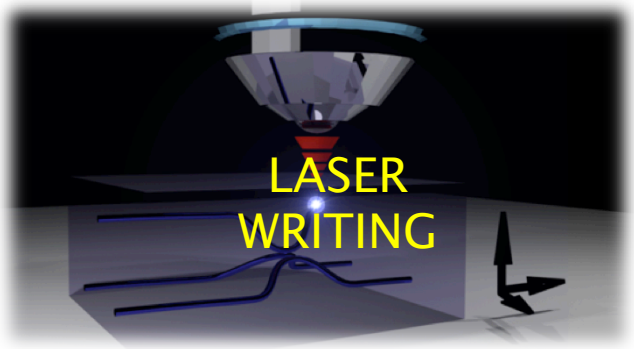
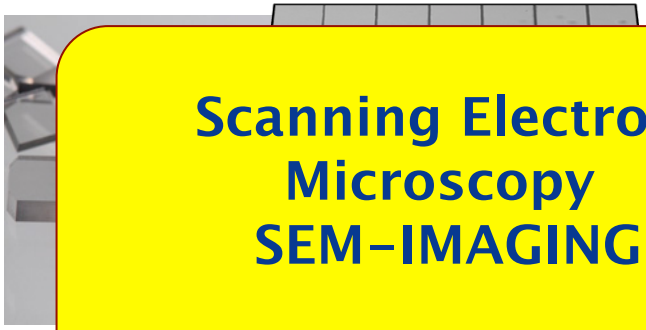




Scanning Electron Microscopy
SEM-IMAGING

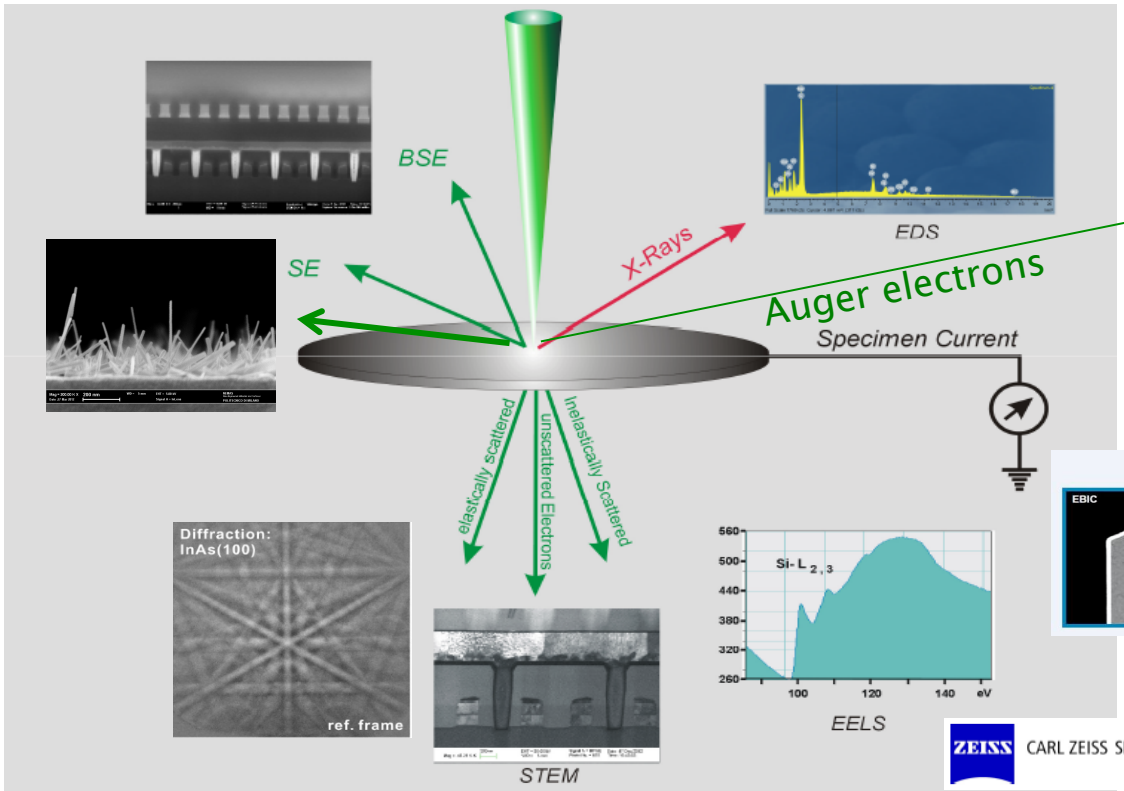
Scanning Auger Microspectroscopy
SAM

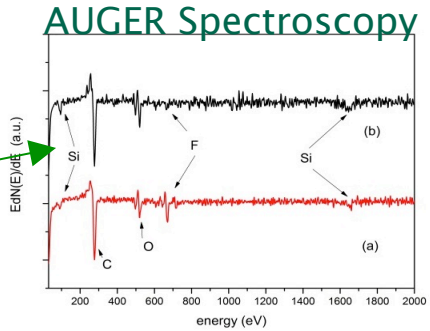
- **Morphology at micro-nano scale**
- **Metrology**
- **Local composition / state of oxidation**
- **Crystalline structure**

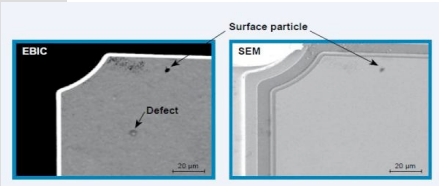
- Absorption
- Refractive index
- …..

SEM : versatile analytical tool

- Variable viewfield (nm ÷ mm) (*magnification range)*
- Variable depth sensitivity (nm ÷ ~100nm)*(primary e-beam Energy)*
- Operates directly on structures and devices
- Many probes / simultaneous acquisition of multiple information







EBIC

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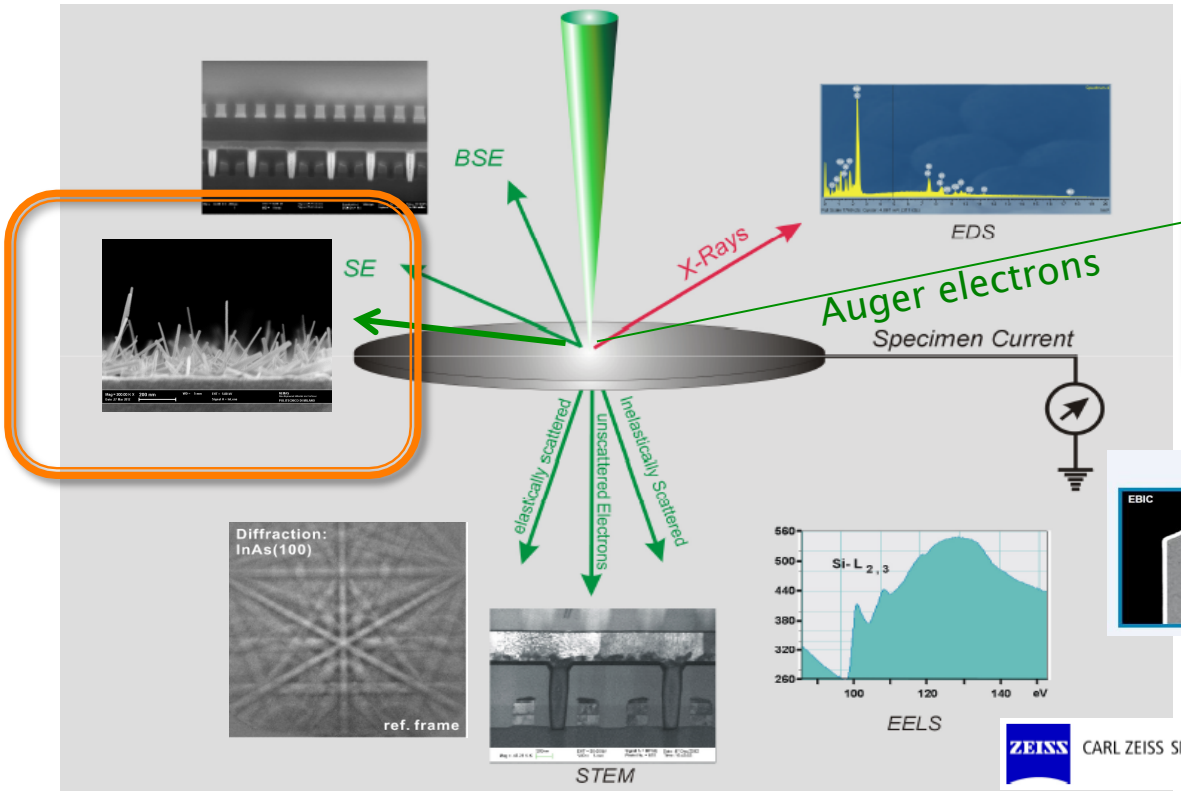

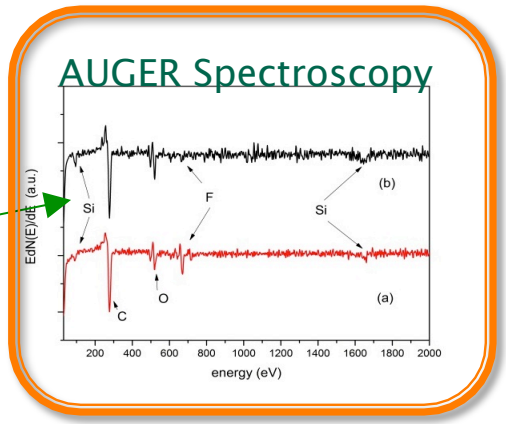

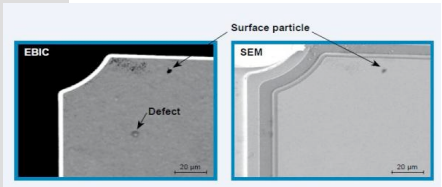

EBIC

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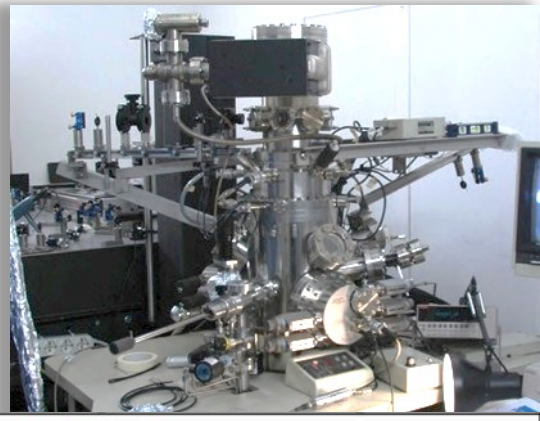
SAM-Scanning Auger Electron Microspectroscopy: The UHV case



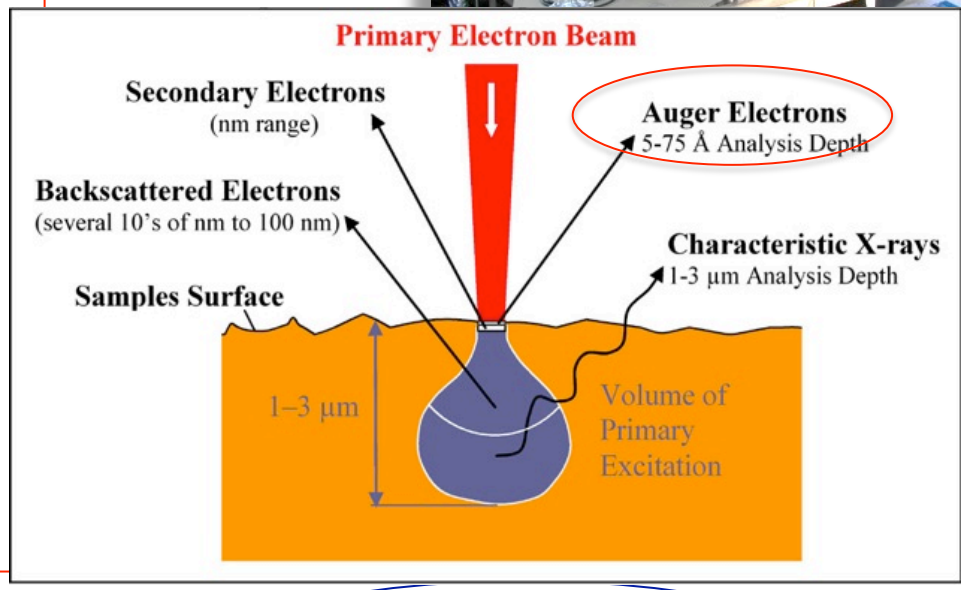
Scanning Auger Microscopy:

Auger Electron Spectroscopy

&

Scanning Electron Microscopy:

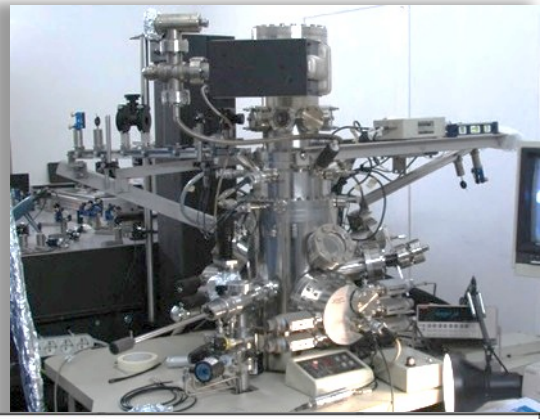
Local Surface Compositional Analysis



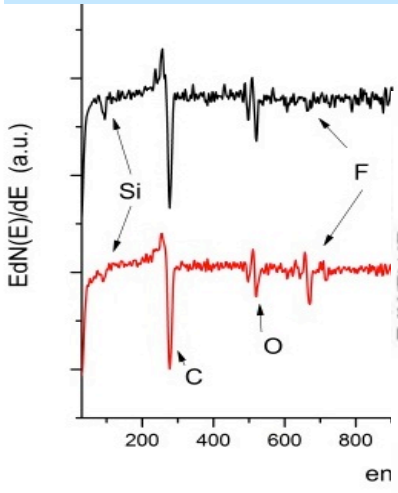
- **SURFACES**
- **INTERFACES**
- **DEPTH PROFILING**

PROBED VOLUME
$10 \div 100\ nm^3$

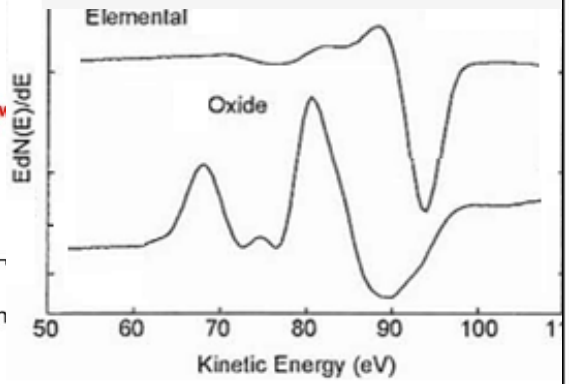
SAM-Scanning Auger Electron Microspectroscopy: The UHV case

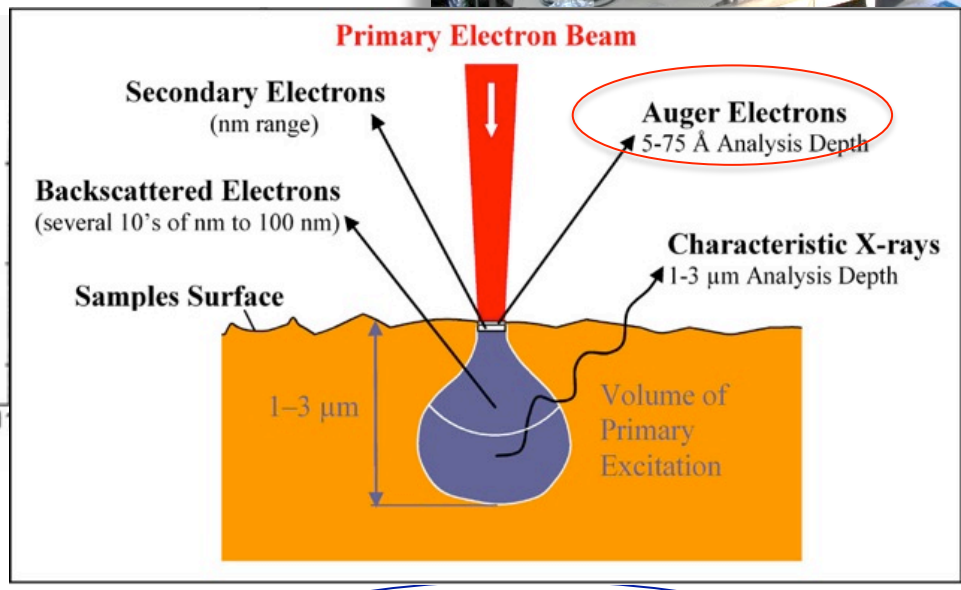


Composition



Oxidation





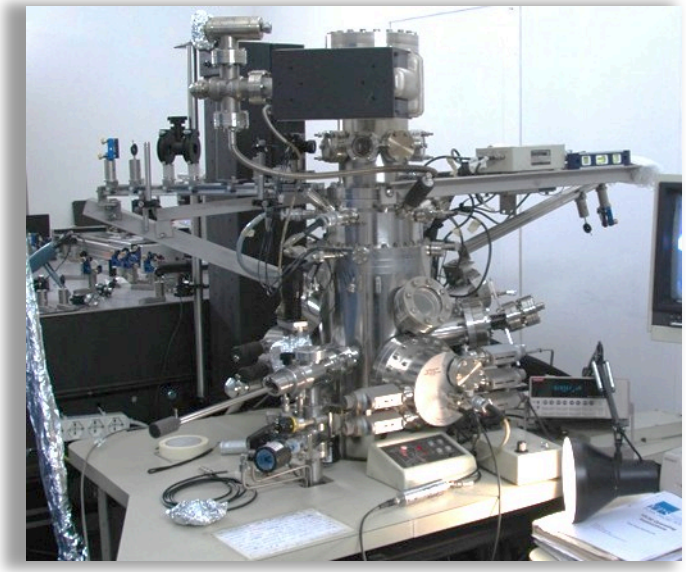
- SURFACES
- INTERFACES
- DEPTH PROFILING

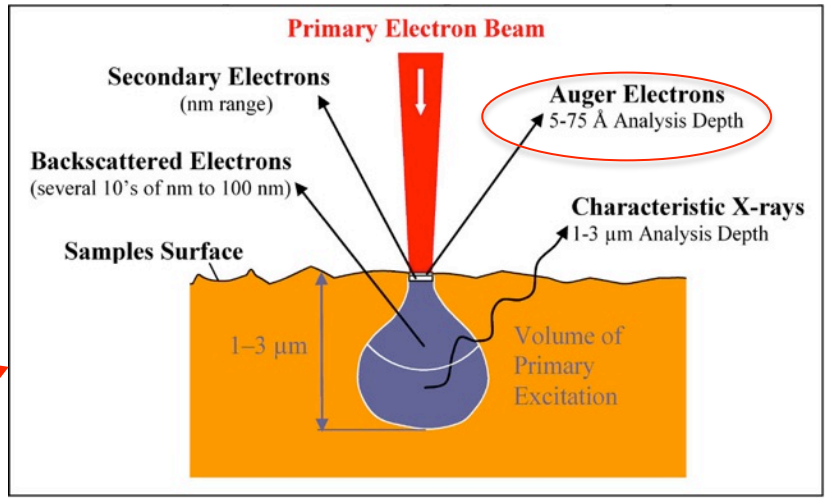
PROBED VOLUME
10÷ 100 nm^3

Scanning Auger Electron Microspectroscopy:

PHI-660 modified Field Effect Schottky Scanning Auger Microscope

- $E_{k,primary}$= 25kV
- e-Beam Current: 10pA÷ 100nA
- $\Delta E/E \approx 1\%$
- UHV (10^{-10} Torr)



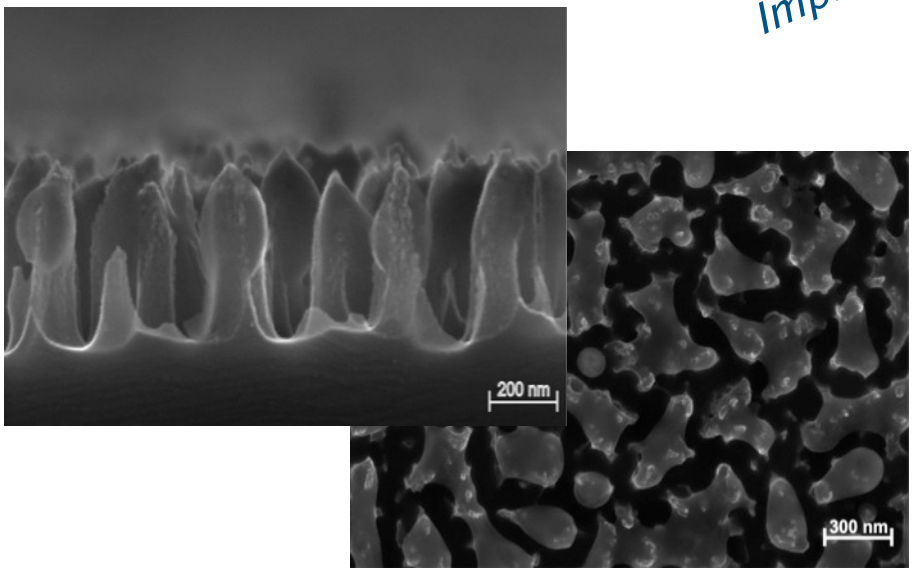


- Large range of field of view (μm ÷ cm)
- Locality (Φ = 10 ÷ 100 nm)
- Surface sensitivity (< 1nm in depth)
- Valuable on variety of substrates

SAM- local composition at surface

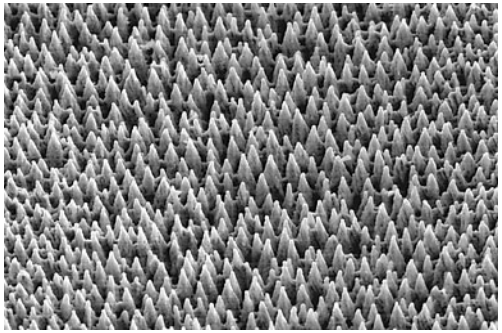
BLACK SILICON

Plasma etched
Surface nanotextured



Improving optical absorption in PV cells

Antibacterial surfaces

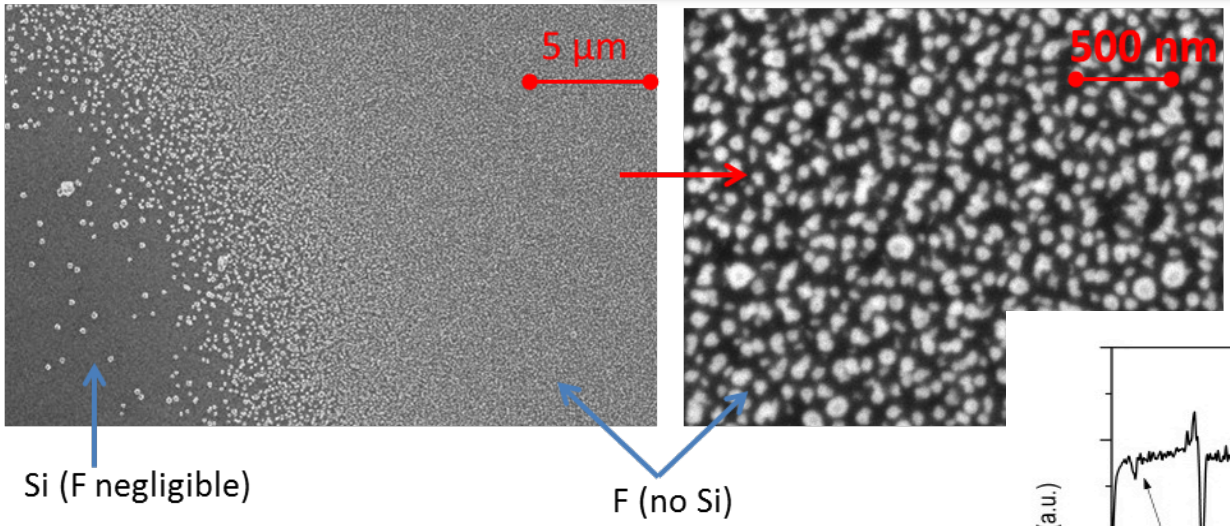


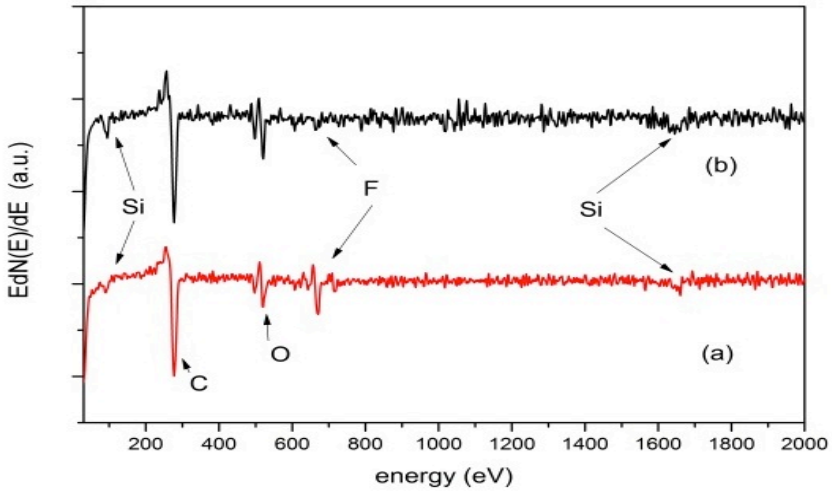
Thin Solid Films 603 (2016) 173–179

SAM- local composition at surface

BLACK SILICON

Assessment of local condition for surface passivation and pillar formation



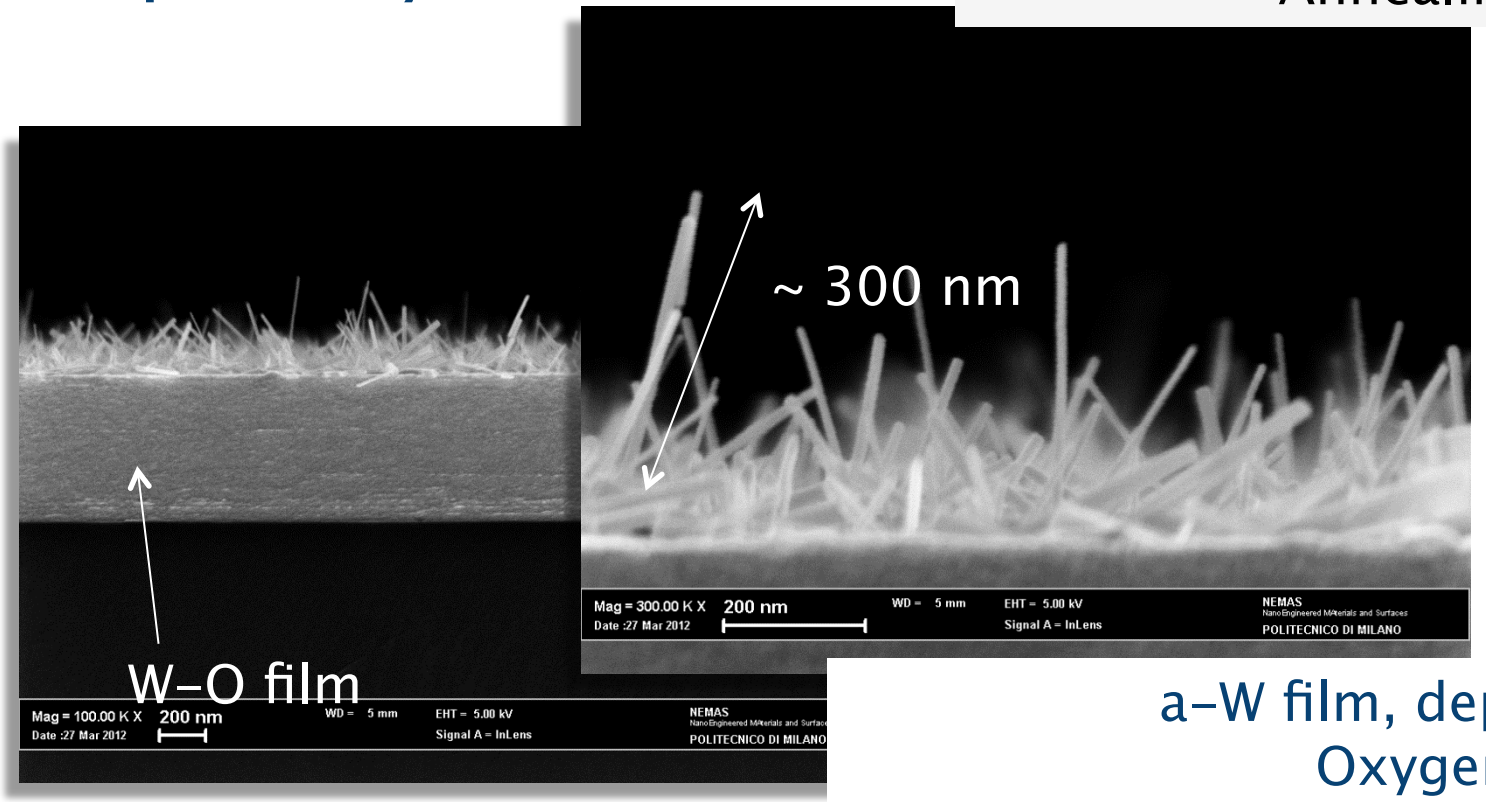


Thin Solid Films 603 (2016) 173–179

SAM- local oxidation at surface

WO_x NANORODS at surface of Metastable W -O thin films deposited by PLD

VACUUM THERMAL ANNEALING
(base pressure 10^{-5} Pa)
Temperature range: 200C ÷710C
Annealing time: 4 ÷10h



a-W film, deposited at 40 Pa
Oxygen content 33.5%
Thermally Annealed : 4h @ 650C

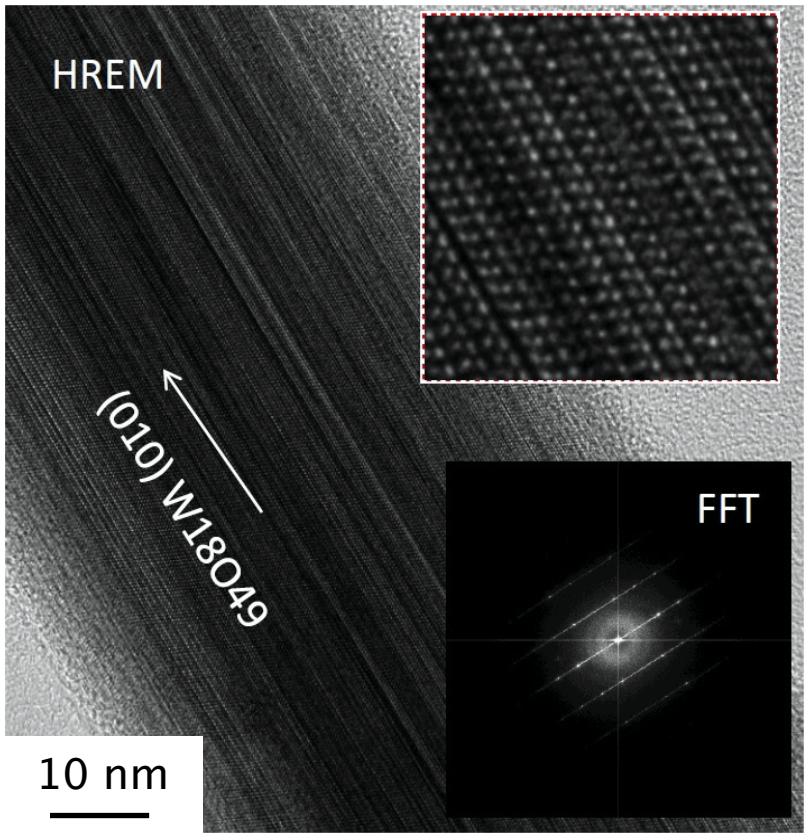
Nanotechnology, 26, 365601, (2015)

Hi-RES TEM: The role of Temperature on oxide crystallographic phase

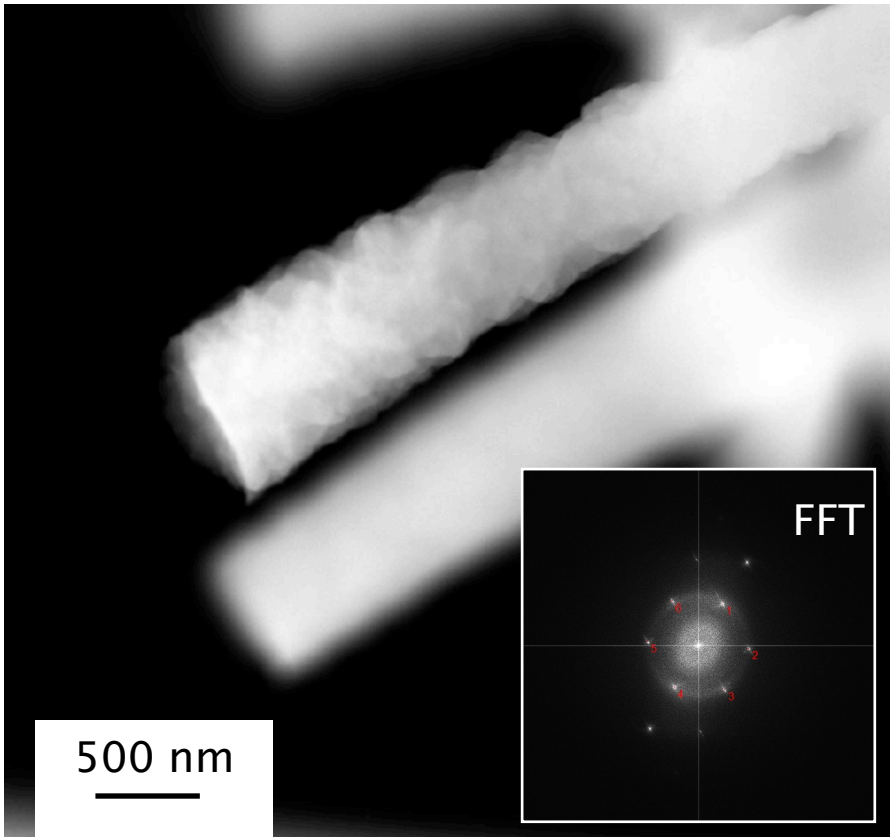
a-W film @40Pa He, O= 33%
Thermal annealing **4hrs**

Jeol 2200FS *field emission gun TEM @ 200 kV.*

500 °C



650 °C



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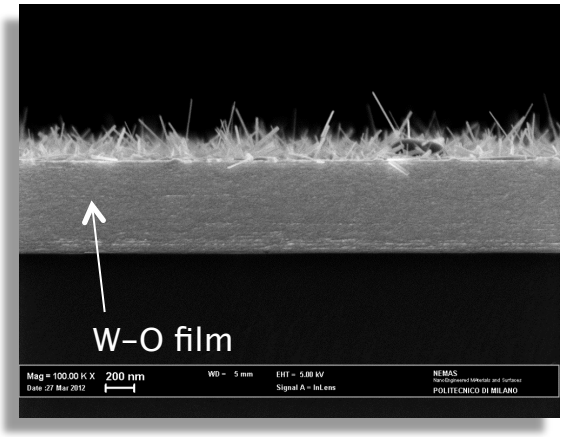
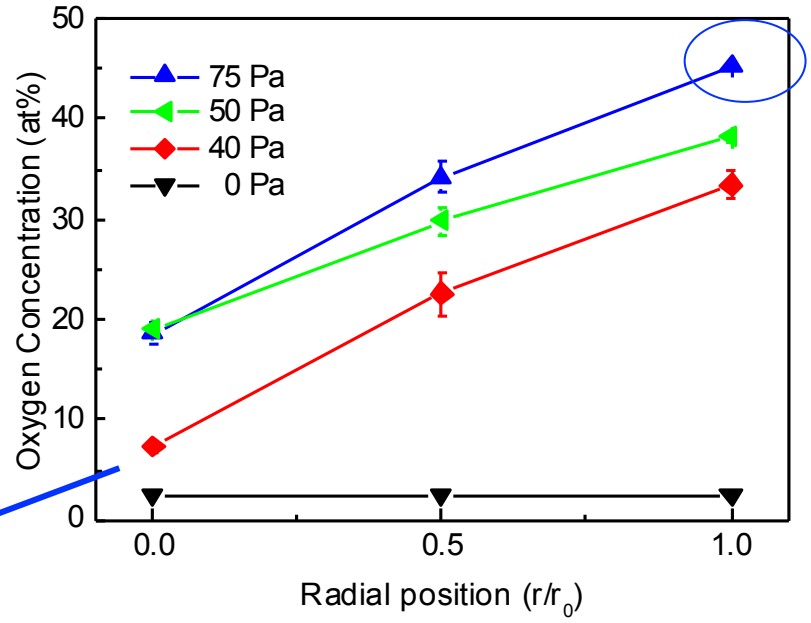
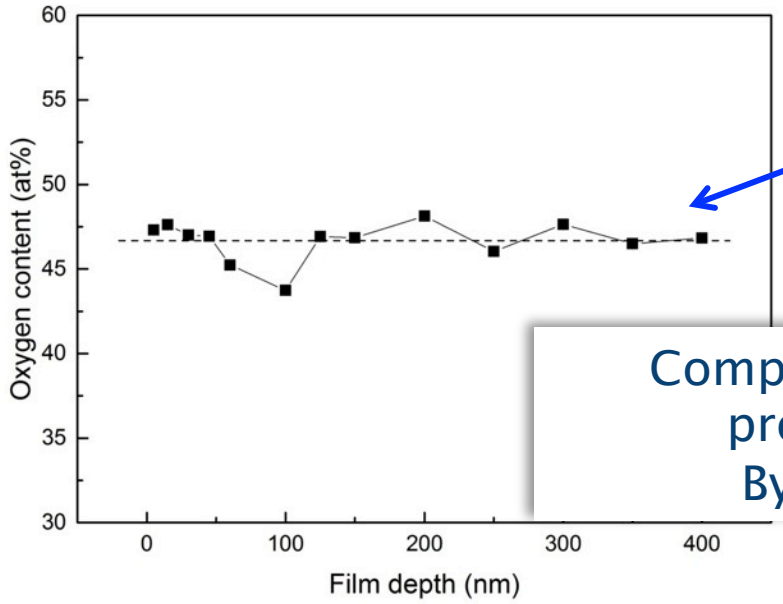
SAM- local oxidation at surface

Assessment of bulk composition of films
And local oxidation at surface







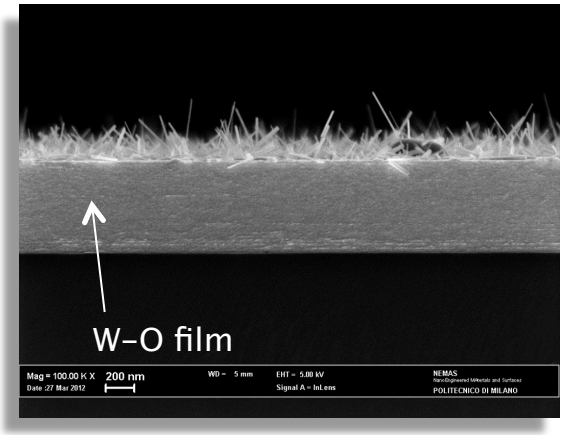
Compositional profiling
By SAM

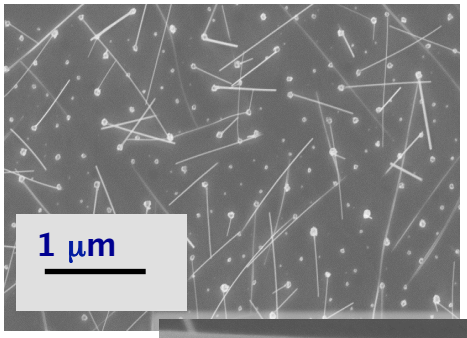
Compositional evaluation
By SEM – EDS

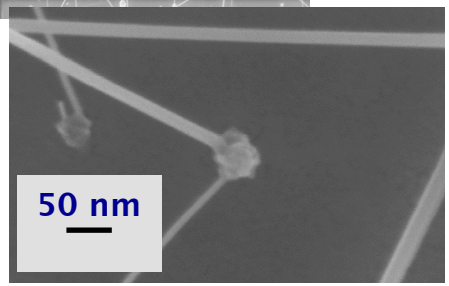
Nanotechnology, 26, 365601, (2015)

SAM- local oxidation at surface

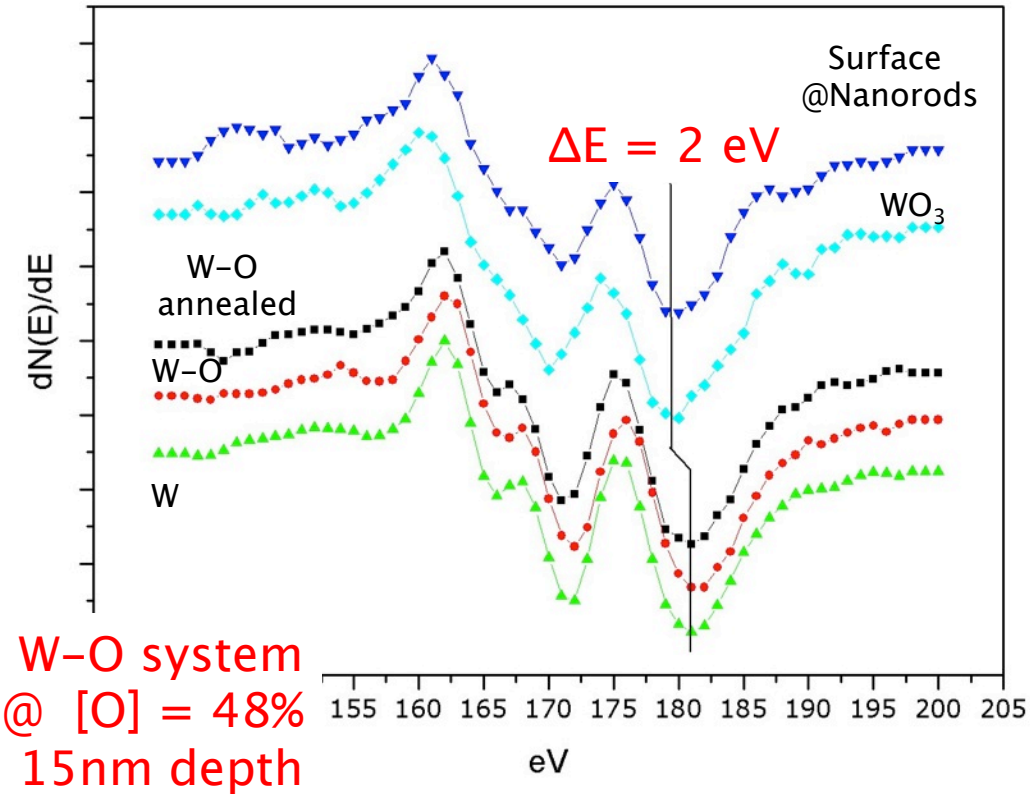
Assessment of bulk composition of films
And local oxidation at surface







W Auger transition peak

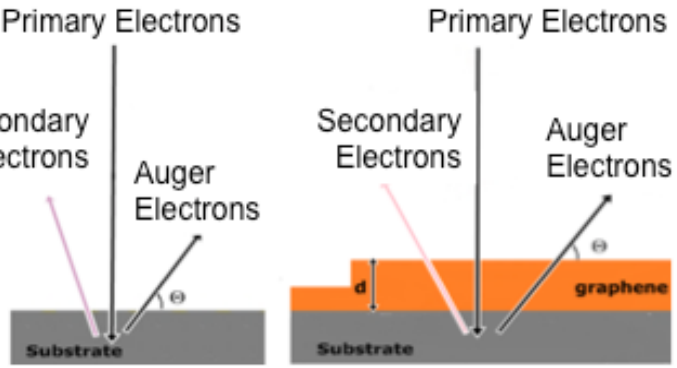


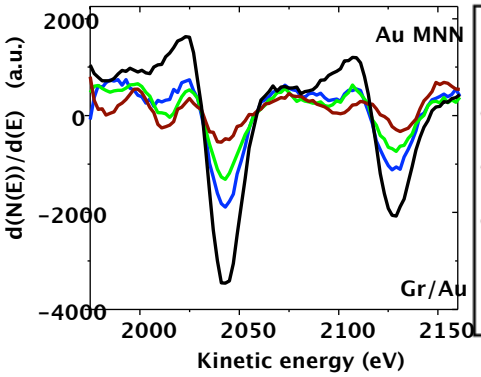
W-O system
@ [O] = 48%
- 15nm depth

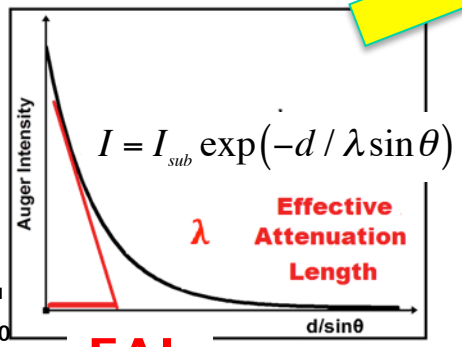
Nanotechnology, 26, 365601, (2015)

SAM – thickness metrology of sub-nm films

Auger electron attenuation by 2D overlayer on substrate

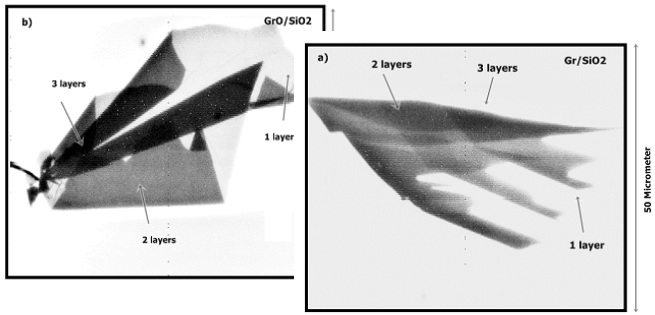






EAL

The case of GRAPHENE and GRAPHENE OXIDE



IF **EAL** is known
THEN **d** can be evaluated

NO EAL data
are available for 2D materials

Wide uncertainty
in thickness estimate

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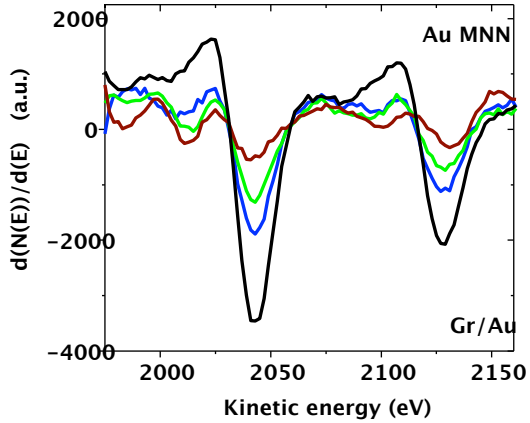
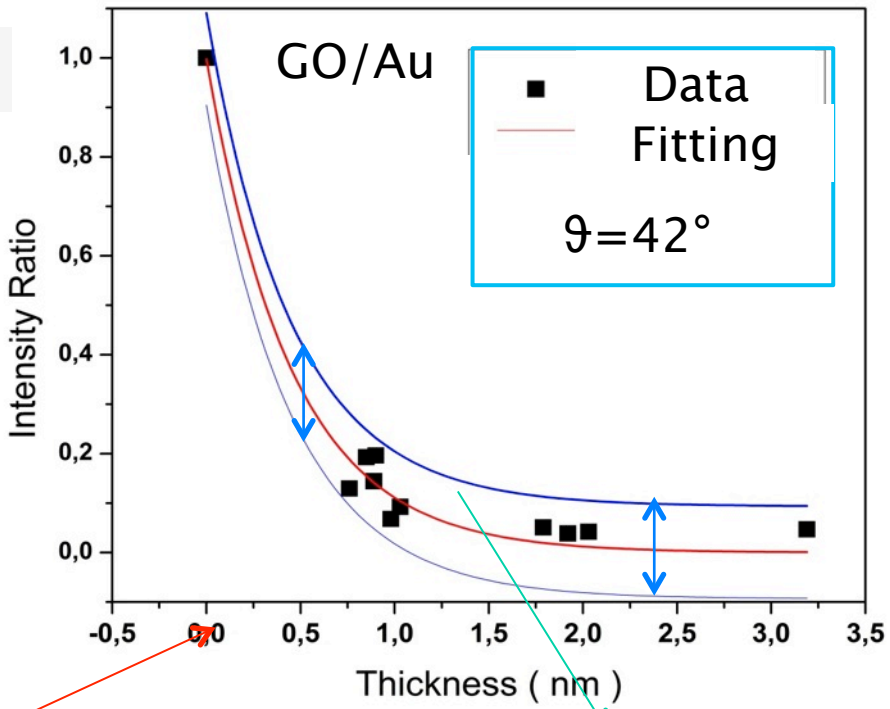
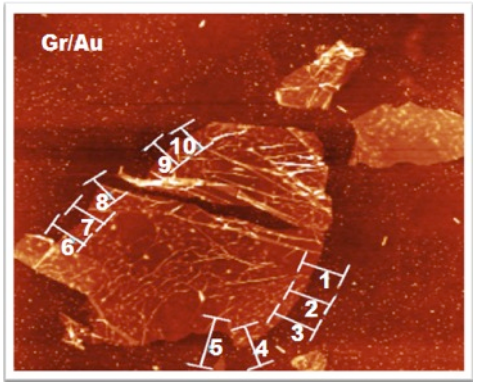
SAM – thickness metrology of sub-nm films

Calibration of Auger EAL by AFM



Sample /Substrate Auger signal ratio





Averaged AFM thickness assessment at borders

$$I = I_{sub} \exp\left(-d / \lambda \sin\theta\right)$$

From **d (averaged AFM)** values
=> **EAL (statistical)** can be evaluated

S. M. Pietralunga, *et al.*,,*in IEEE – Proc. of ICTON 2016*

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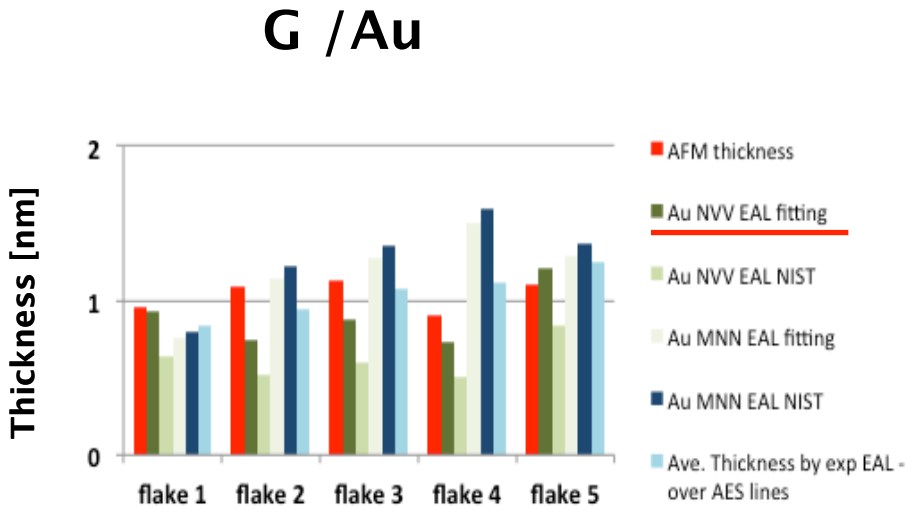
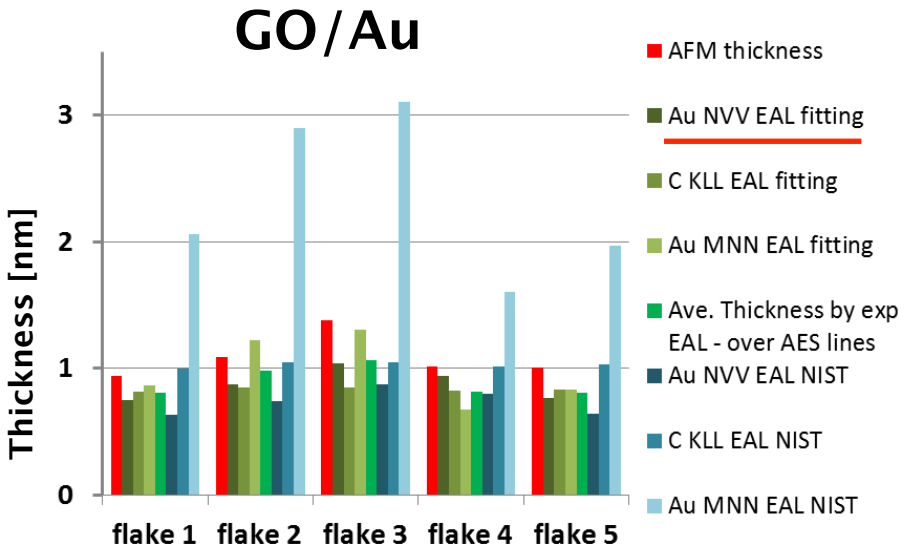
SAM – thickness metrology of sub-nm films

The case of Graphene and Graphene Oxide

from accurate **EAL** values ==> **d** can be evaluated



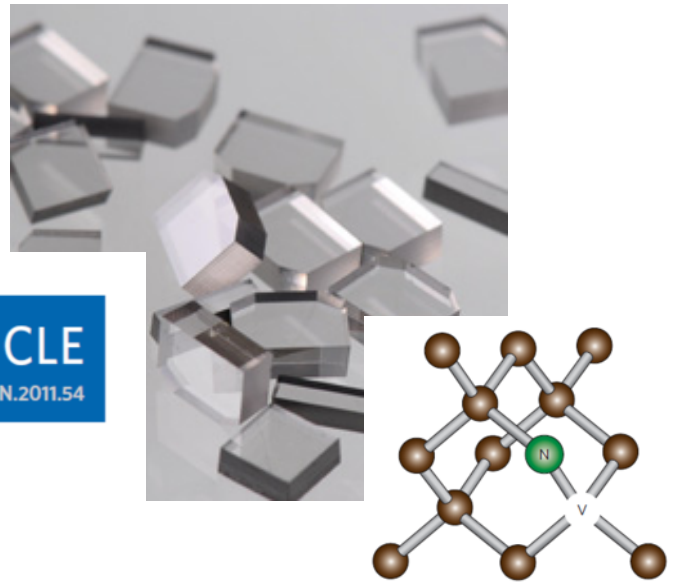


* *http://www.nist.gov/srd/nist82.cfm*

S. M. Pietralunga, *et al.*,,*in IEEE – Proc. of ICTON 2016*

Diamond photonic platform

⇒ Ideal platform for **quantum computing / magnetometry**



nature photonics

REVIEW ARTICLE

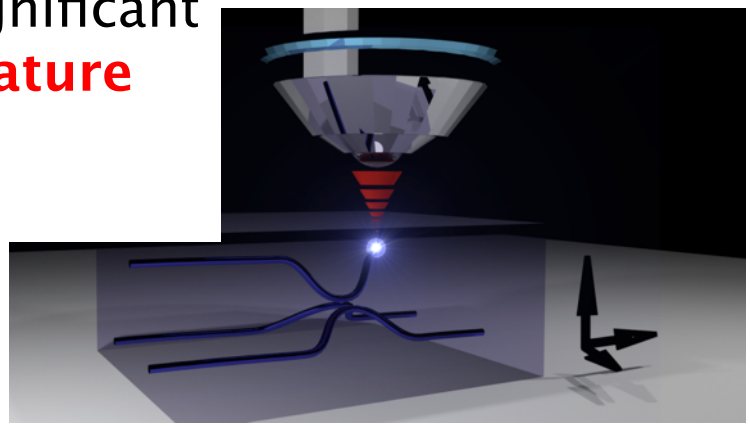
PUBLISHED ONLINE: 30 JUNE 2011 | DOI: 10.1038/NPHOTON.2011.54

Diamond photonics

Igor Aharonovich[1]†*, Andrew D. Greentree and Steven Prawer

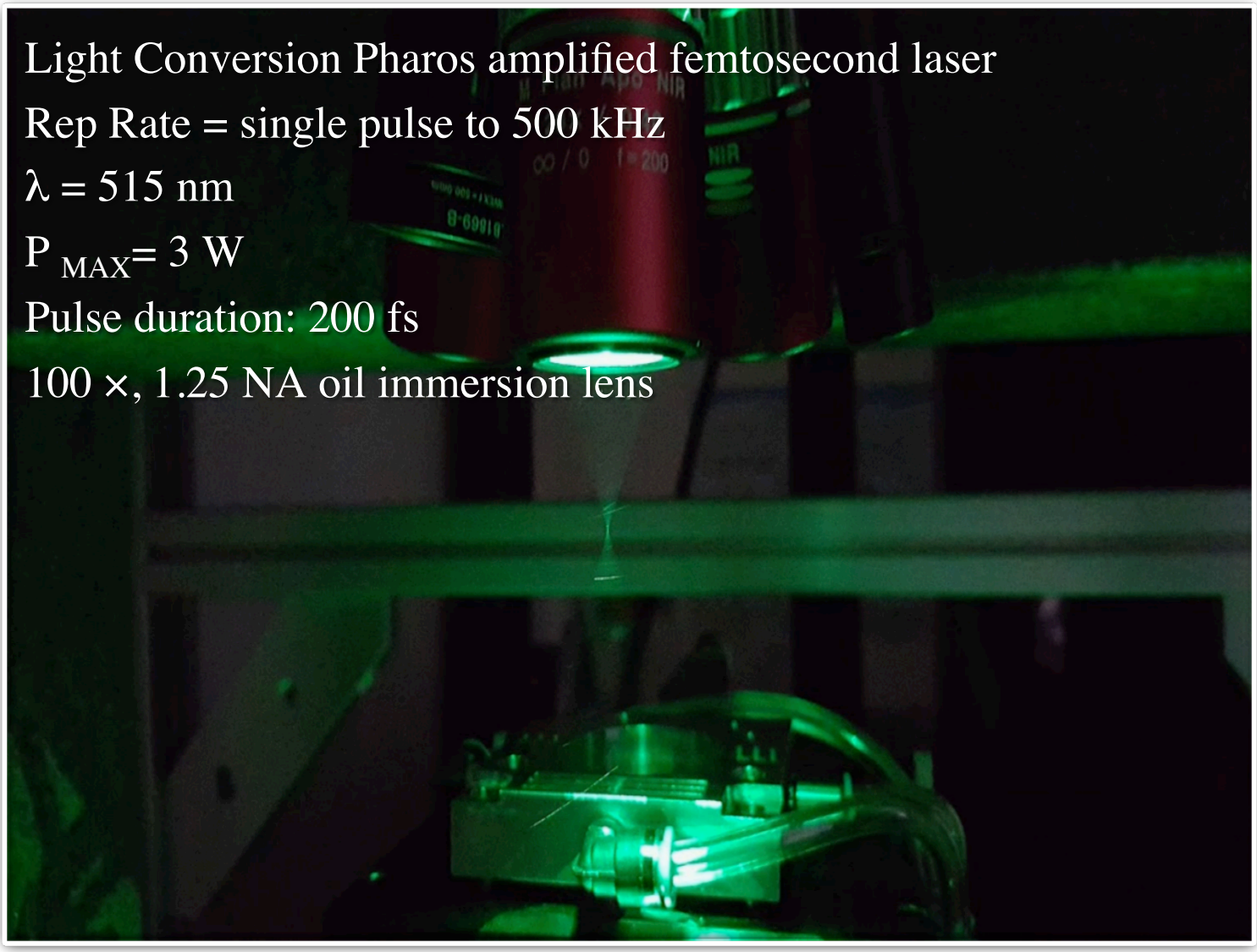
!

«The chemical inertness of diamond is a significant hurdle for diamond nanofabrication**...a mature fabrication toolkit has yet to be developed**»

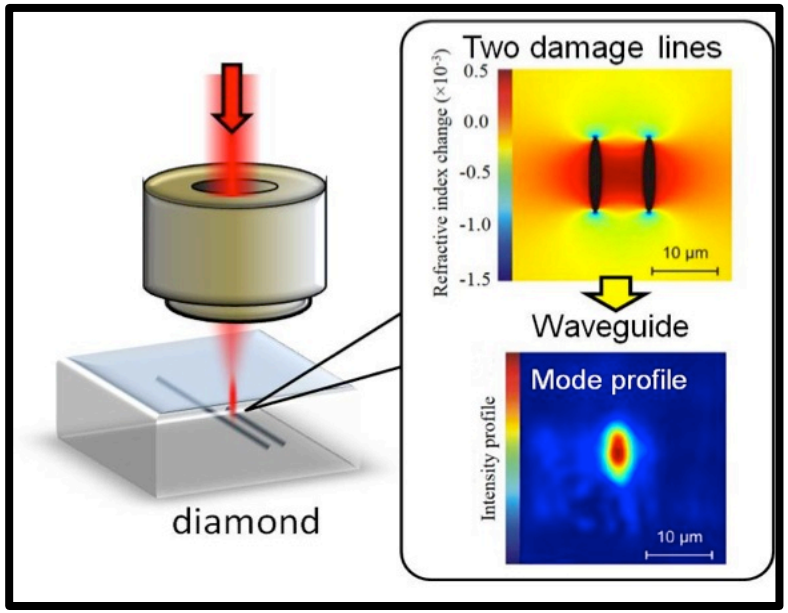


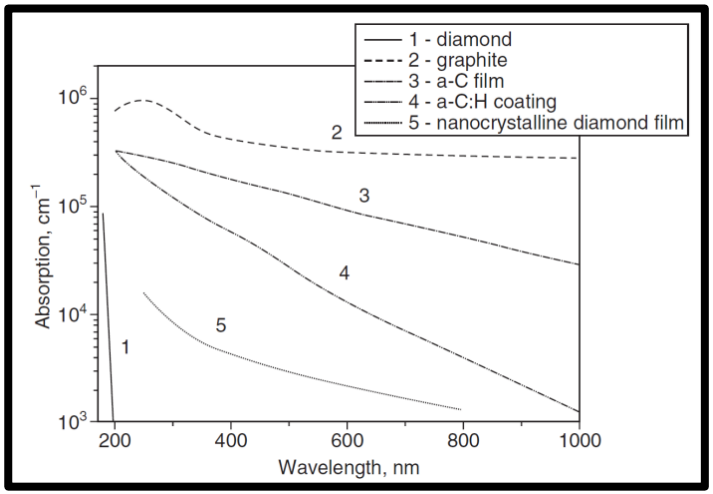
Solution => Focused femtosecond laser pulses to write 3D optical circuits

Fs-laser micromachining setup



Waveguide writing in bulk diamond



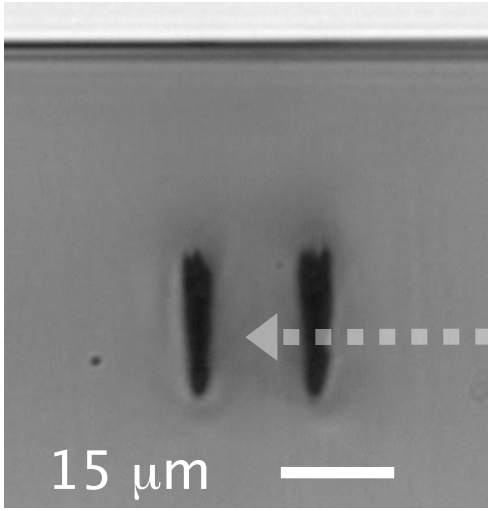


- Laser-write two closely spaced parallel lines
  => **stressors**
- Keep diamond between lines **unmodified** to **preserve NV centers** properties
- **Avoid formation** of highly absorptive **C- graphite** inside the lines

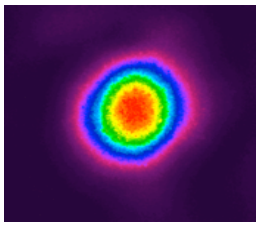
Waveguide writing in bulk diamond

515 nm, 500 kHz, 50 mW, 0.5 mm/s, 13 μm separation

along <110> crystallographic direction



λ = 808 nm

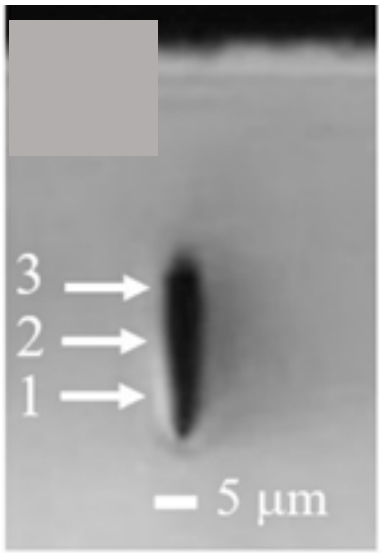


MFD = 8.5 μm × 10 μm
Insertion Loss = 14 dB

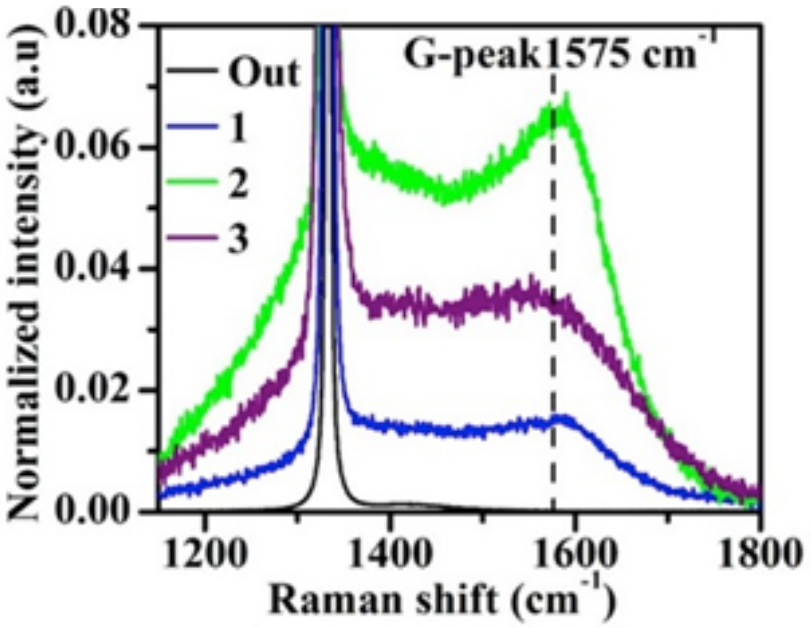
- single mode
- Similar properties @ 532 nm, 637 nm
- TM guiding only

Single stressor features

515 nm, 500 kHz, 50 mW, 0.5 mm/s



µ-Raman spectroscopy



Outside

- Diamond (sp^3 bonds) Raman peak @ 1331 cm^{-1}

(FWHM = 2.3 cm^{-1})

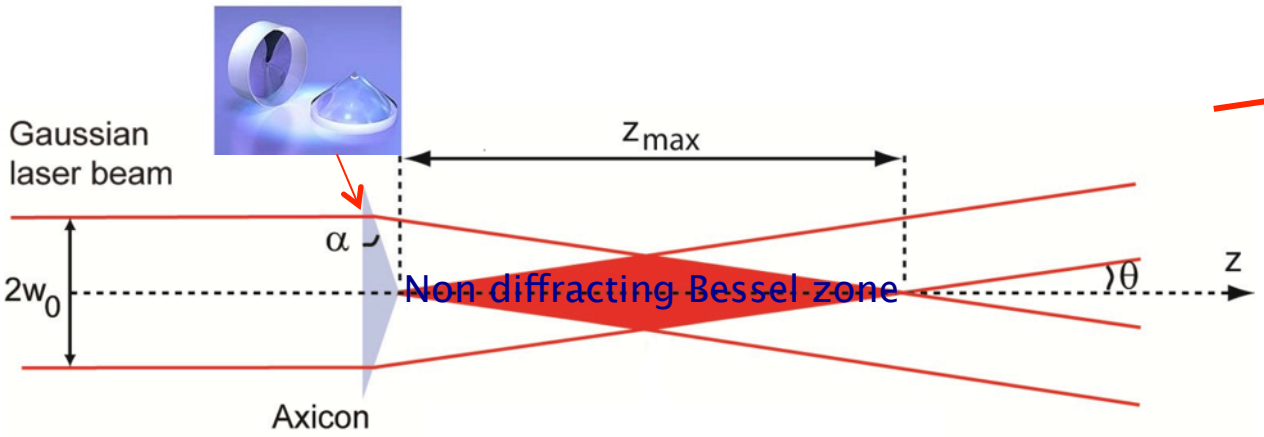
Inside

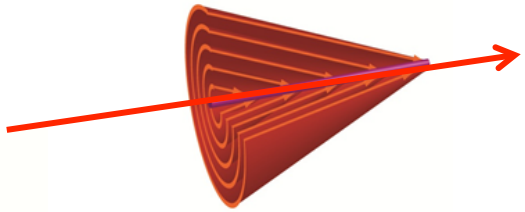
- Diamond peak decreases and widens
- Two new peaks:
- G-peak at 1575 cm^{-1}
- D-peak at 1360 cm^{-1}

} sp^2 bondings

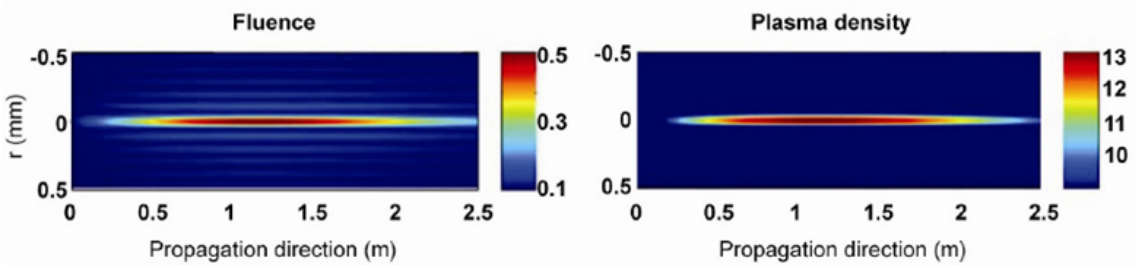
- Broad D and G=> amorphous carbon

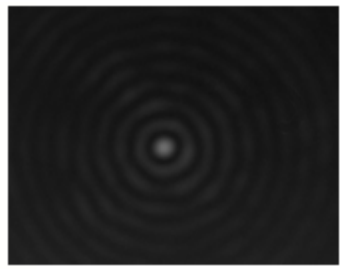
Bessel Beams in Laser micromachining





Equivalently
a superposition
of plane waves
distributed over a cone





Conical-flow energy
inwards
the beam central lobe

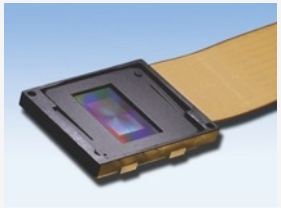
In transparent materials
the plasma track generated by the Bessel lobe
is the main support for
nonlinear absorption of laser energy

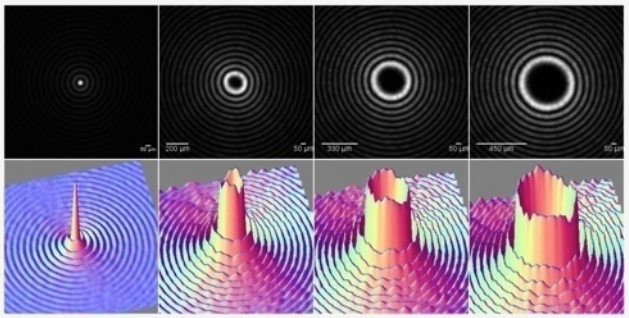
⇒ **high aspect-ratio material modification**

PRA 77, 043814 (2008)

Bessel Beams in Laser micromachining

Spatial light modulators (SLM)
to generate BB of any orders and features

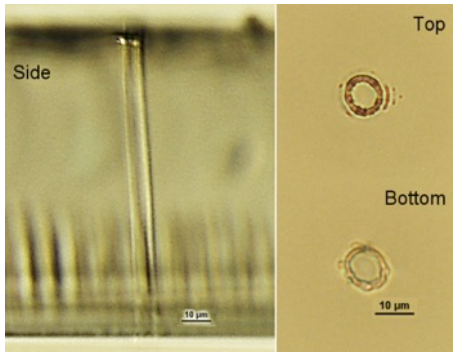




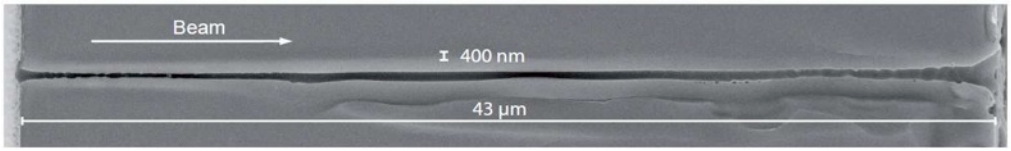
High order Bessels with azimutal phase

APPLICATIONS

Tubular microstructures

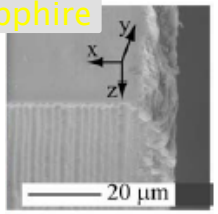


Void nanochannels for nanofluidics



APL 97, 081102 (2010) EJPD ST 199, 101 (2011)

sapphire



Fast single pass cutting

Appl. Phys. A 120, 385 (2015)
Appl. Phys. A 120, 443 (2015)
PATENT PCT/EP2013/003508

Ti:Sapphire @800nm
Pulse duration:40fs -ps
Low RepRate:20Hz (10mJ)
Single shot fabrication (40-100uJ/pulse)

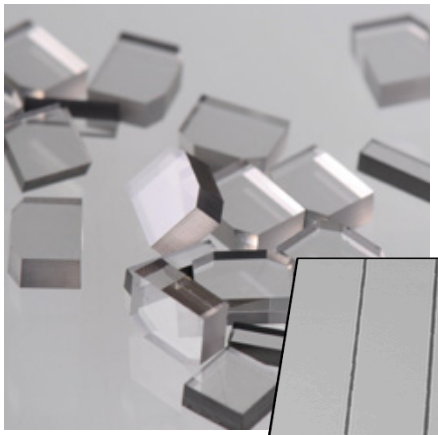
Single shot high aspect ratio guiding microstructures

700 mm

JAP 120, 013102 (2016)

Waveguide laser writing at diamond surface

1 mm-long grooves written at diamond surface
Sub-threshold fluence

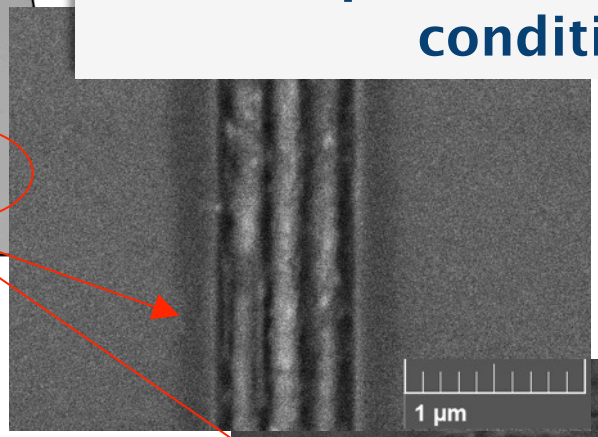


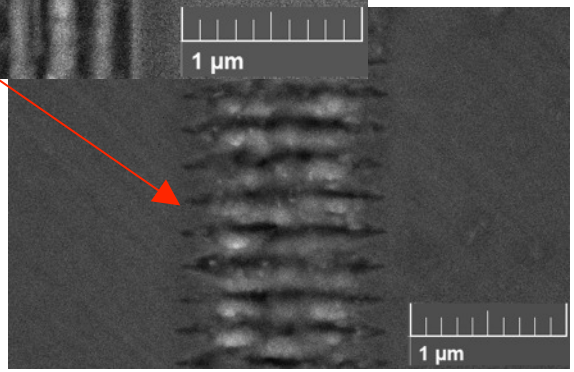
30 μm spacing

SEM imaging of nanoscale internal structure In dependence on writing conditions

No metallization

Polarization ⊥ or ∥





λ=1030nm 250 kHz, 0.42 NA (50×)
P = 15÷20 mW
v = 0.5, 1, 1.5, 2, 3, 3.5 mm/s

Summary:

- Scanning Auger Microscopy (SAM) locally assess elemental composition and state of oxidation on a wide range of materials, to the sub-micrometric range. Surface, interface and depth profiling analysis are possible
- SAM can provide absolute thickness measurement of ultra-thin film overlayers and 2D materials

- The case of diamond as a photonic dielectric platform:
  - fs laser beams write surface grooves and bulk waveguides for quantum optics chips
  - SEM imaging on diamond is carried on without metallization to reveal nanoscale morphological and structural details

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Thank you !

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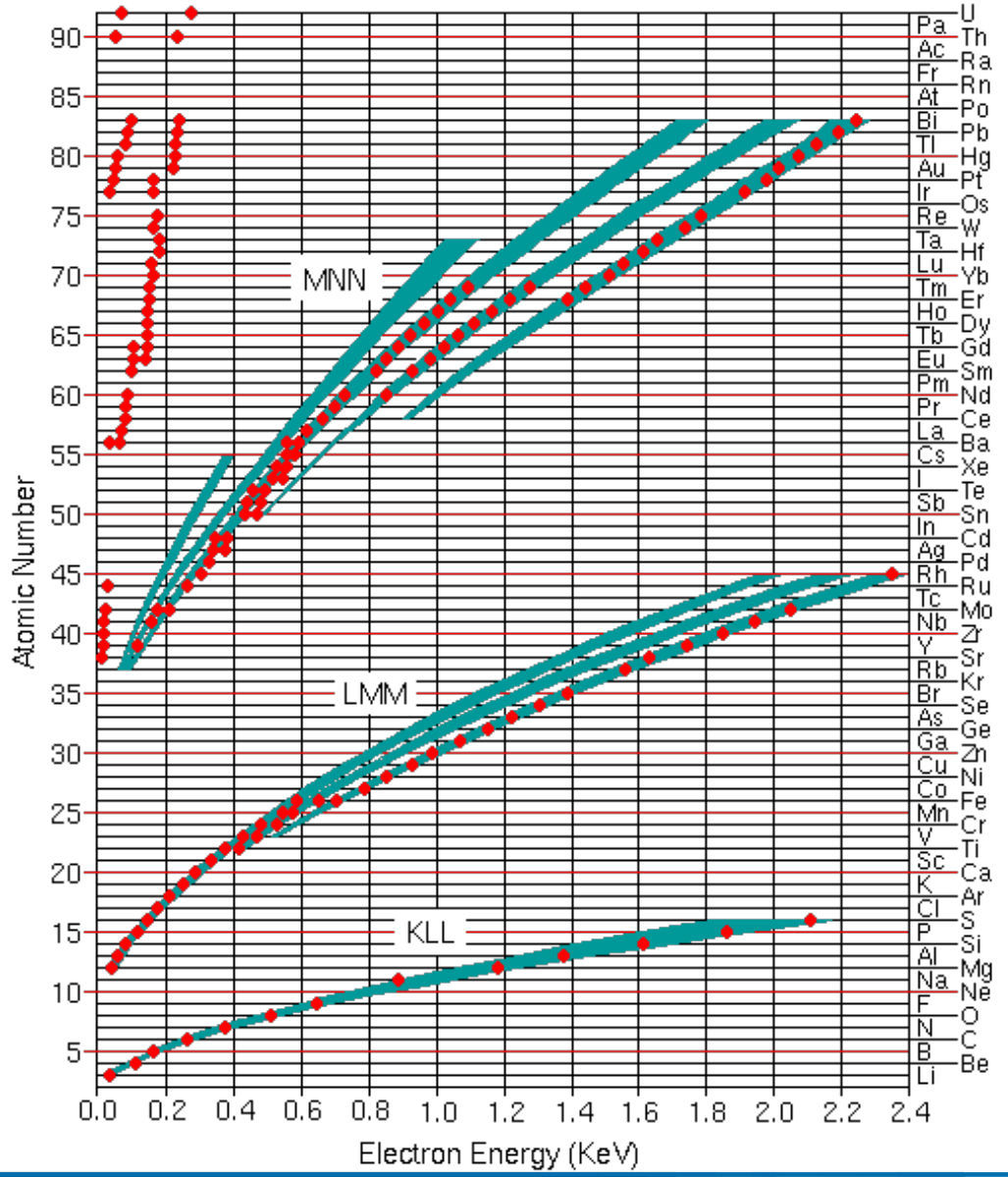
POLITECNICO MILANO 1863

CNR IFN Istituto di Fotonica e Nanotecnologie

AUGER Electron Energies



Quantum information with NV centers

Classical world

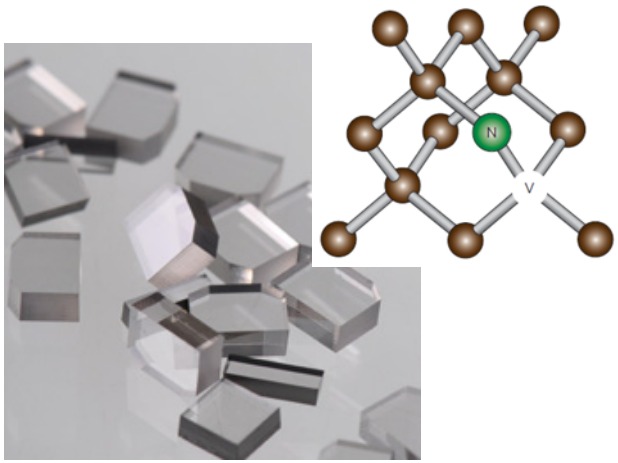
- 0 or 1 state

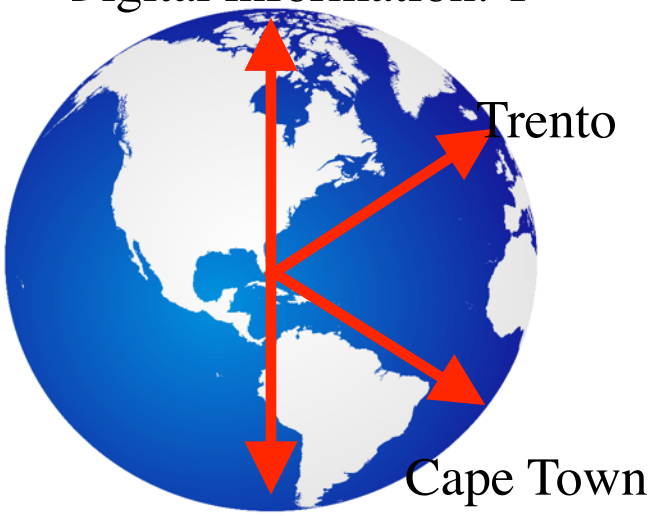
Quantum world

- Superposition of 0 and 1 states (qubit)
- Quantum computer can check many possibilities in **parallel**

- How to make qubits?

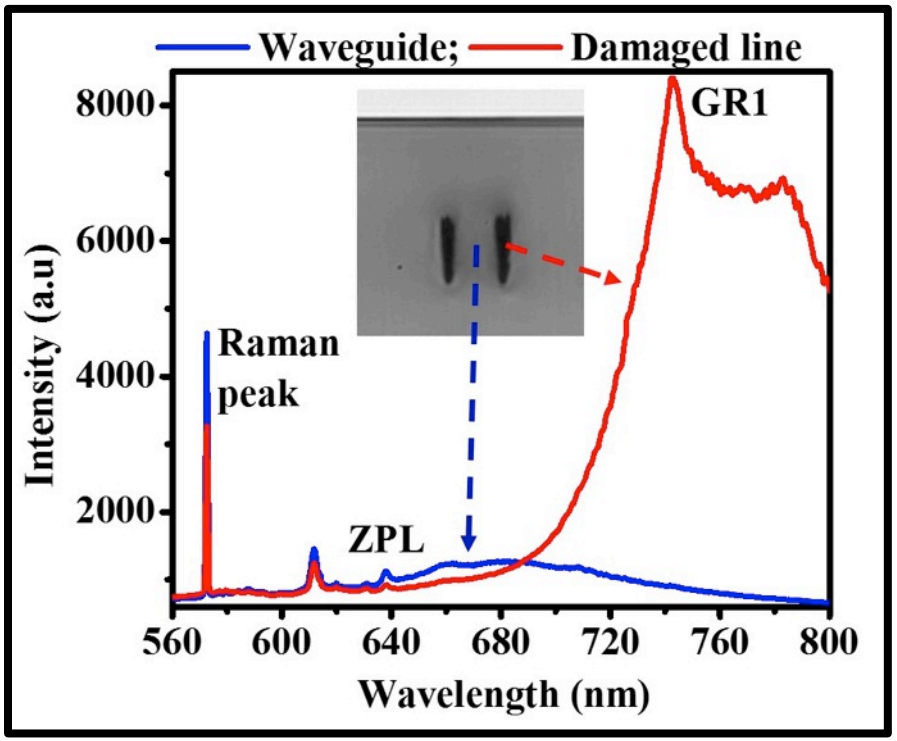
Diamond





Preservation of NV centers

PL measurements in confocal microscope (532 nm)



- Reduction in the intensity of the NV's ZPL
- Guiding region – same as pristine diamond
- Optically detected magnetic resonance (ODMR): hyperfine structure not altered
- Lifetime of the excited state transition preserved: (11.0 ± 1.5 ns)