Production of DLC in Italy

DLC Workshop
RD-51 Collaboration
Feb 13 2020

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Problems with etching of DLC coated PI

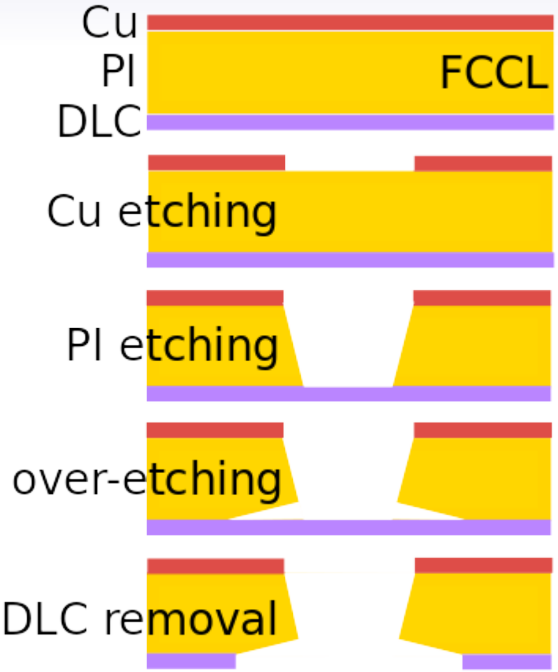
FTM amplification foil follows single-mask production process

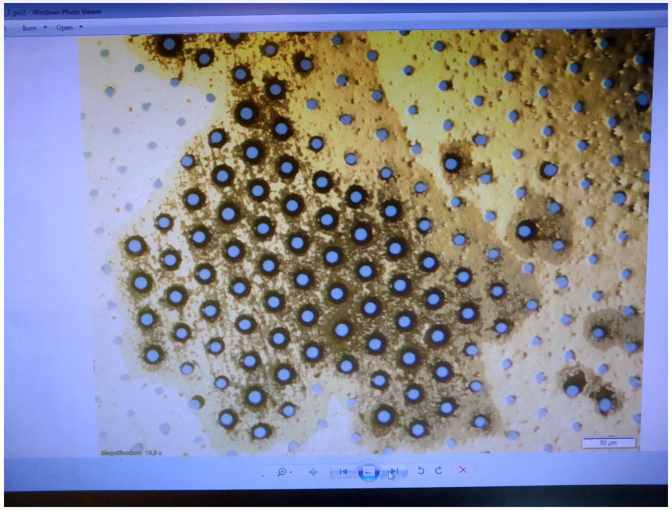
where Cu side of Cu/PI/DLC FCCL is used to start wet etching

chemical Polyimide etch reaches the DLC, DLC delaminates

chemical etch starts etching on DLC - Polyimide interface

upon removal of DLC on holes a very small DLC electrode remains

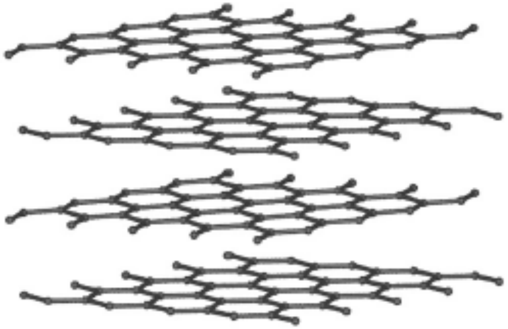




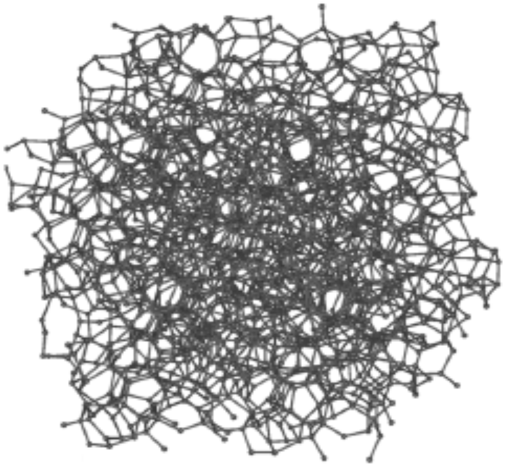
- **Problem with adhesion DLC to Polyimide**
- **Prompted Collaboration to investigate DLC**
  - INFN BA: Ion Beam Deposition
  - INFN LE: Pulsed Laser Deposition & Char

Introduction

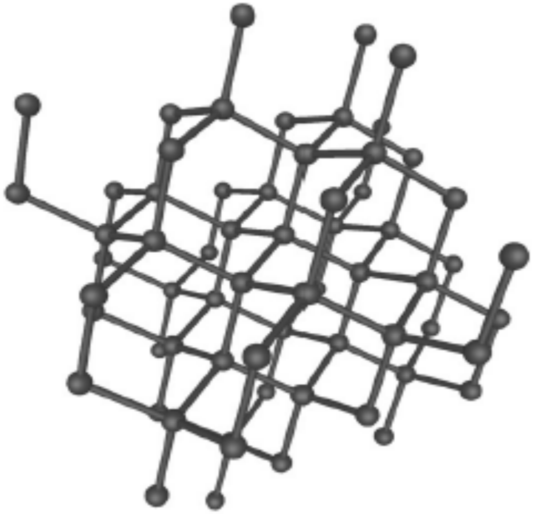
DLC is characterized by clusters of sp^2 and sp^3 bonded atoms in the material. The size and distribution of these clusters depend on the sp^3/sp^2 fraction.



Graphite. 100% sp^2 bonded carbon.



Amorphous carbon. Mix between sp^2 and sp^3 bonded carbon atoms

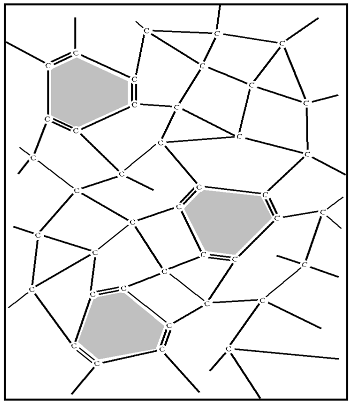


Diamond. 100% sp^3 bonded carbon.

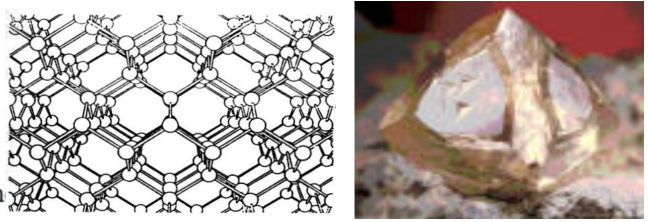
This bond configuration is such to confer to DLC particular properties intermediate between that ones of diamond and graphite which can be modulated by the sp^3/sp^2 fraction.

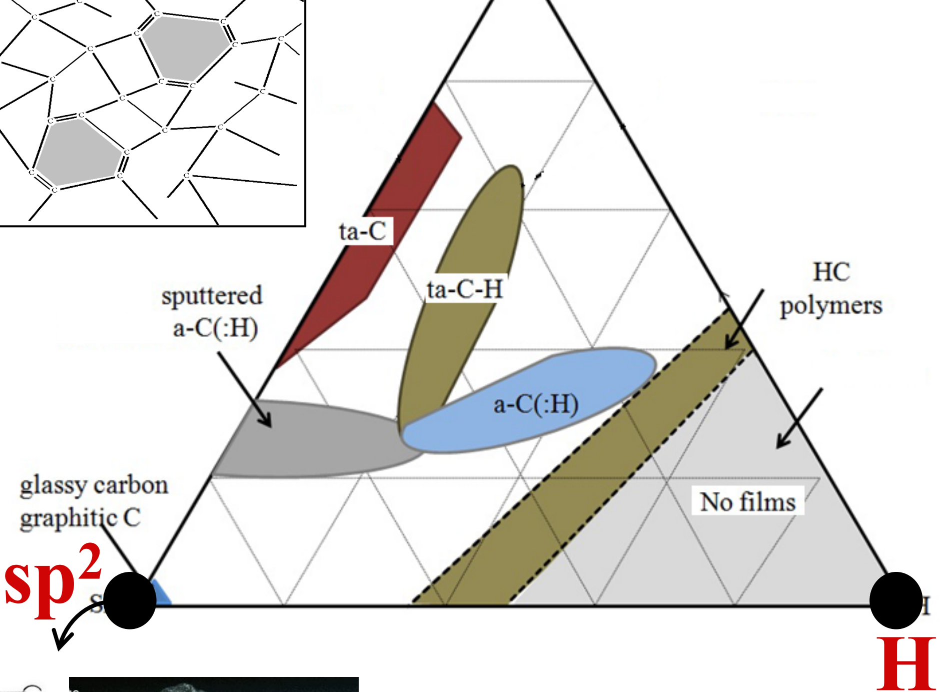
DLC main properties: high hardness, scratch resistance, smooth surface morphology, chemical inertness, good thermal conductivity, high electrical resistance, and optical transparency

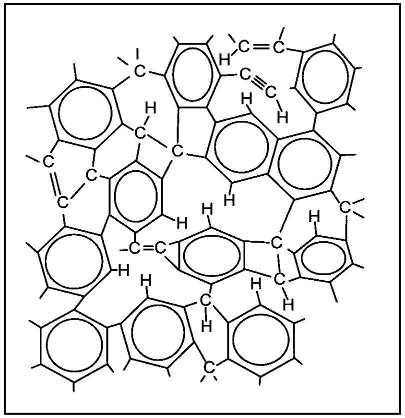
Introduction



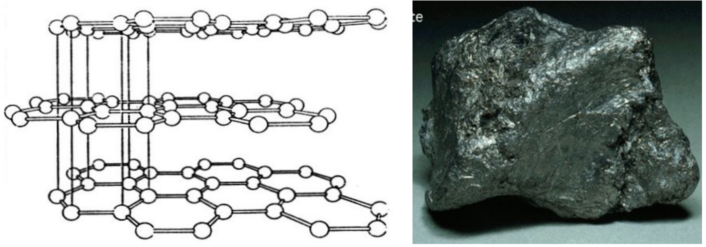
Diamond





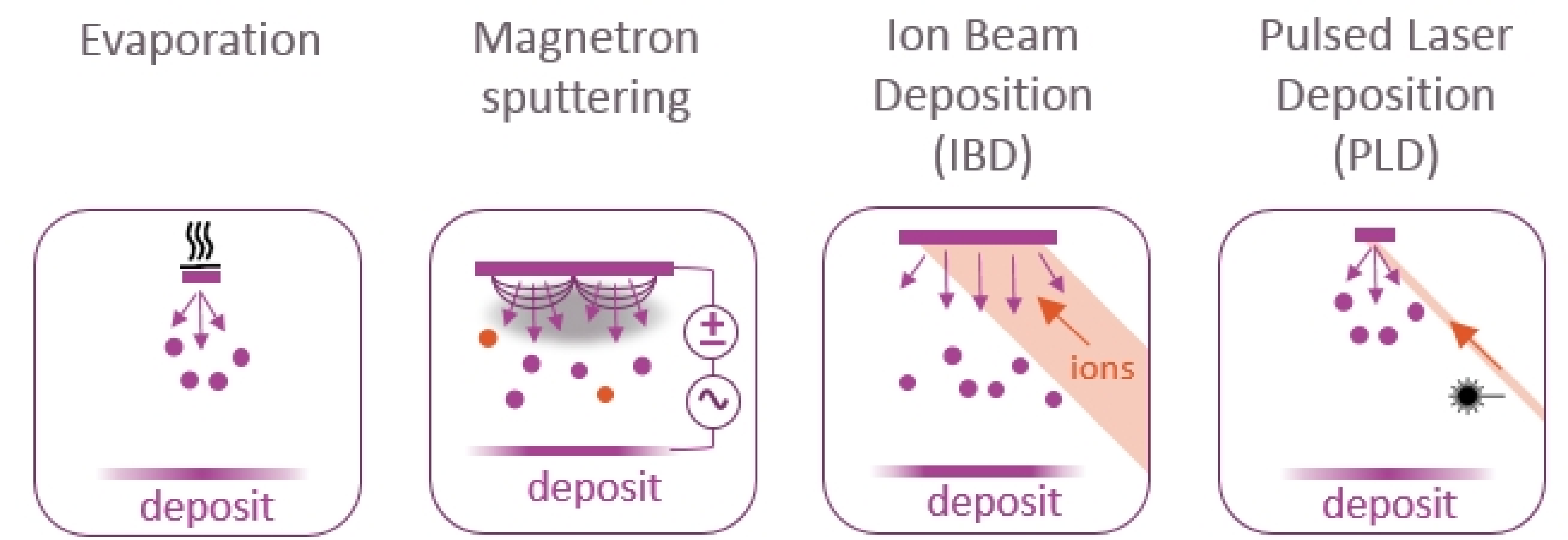


Graphite



Ternary phase diagram of bonding in amorphous C-H alloys: the physical properties of DLC films depend on H-concentration and the sp3/sp2 ratio

Physical Vapor Deposition (PVD)



- **Main difference: ion energy**
  - evaporation: $\mathcal{O}(0.1\,\mathrm{eV})$ sputter: $\mathcal{O}(0.1–10\,\mathrm{eV})$ ion beam $\mathcal{O}(10–100\,\mathrm{eV})$
- **advantages Magnetron Sputtering:**
  - fast process; large area; adopted by industry
- **advantages Ion Beam Sputtering:**
  - good quality film & good adhesion; can deposit many materials on many substrates (also non-conducting)
- **advantages Pulsed Laser Deposition:**
  - λ and J control sp_3/sp_2-ratio, many independent variables; precise control

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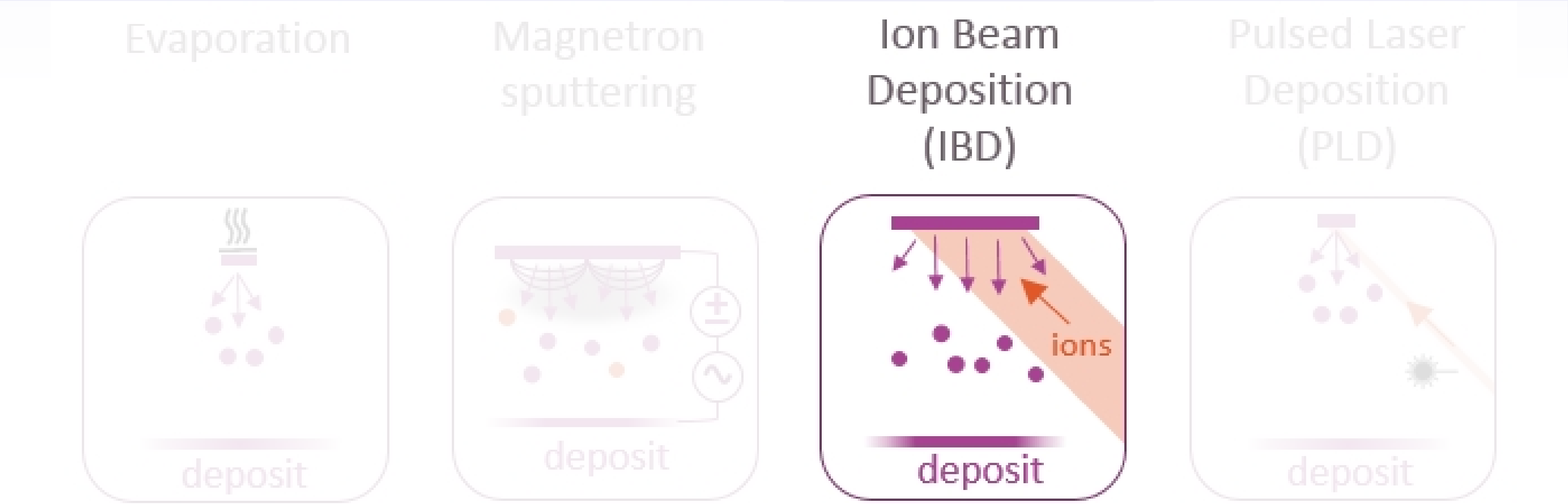

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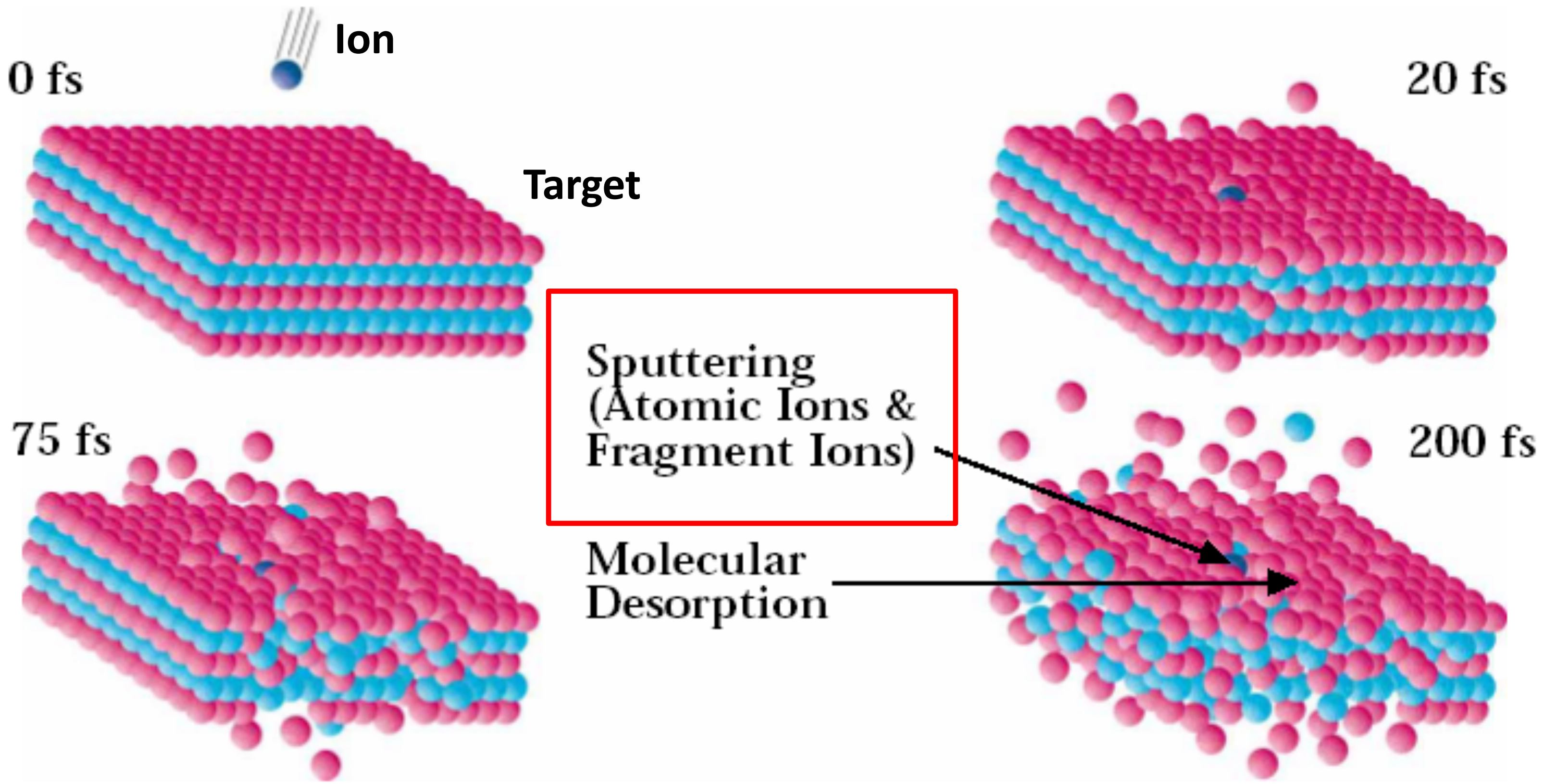
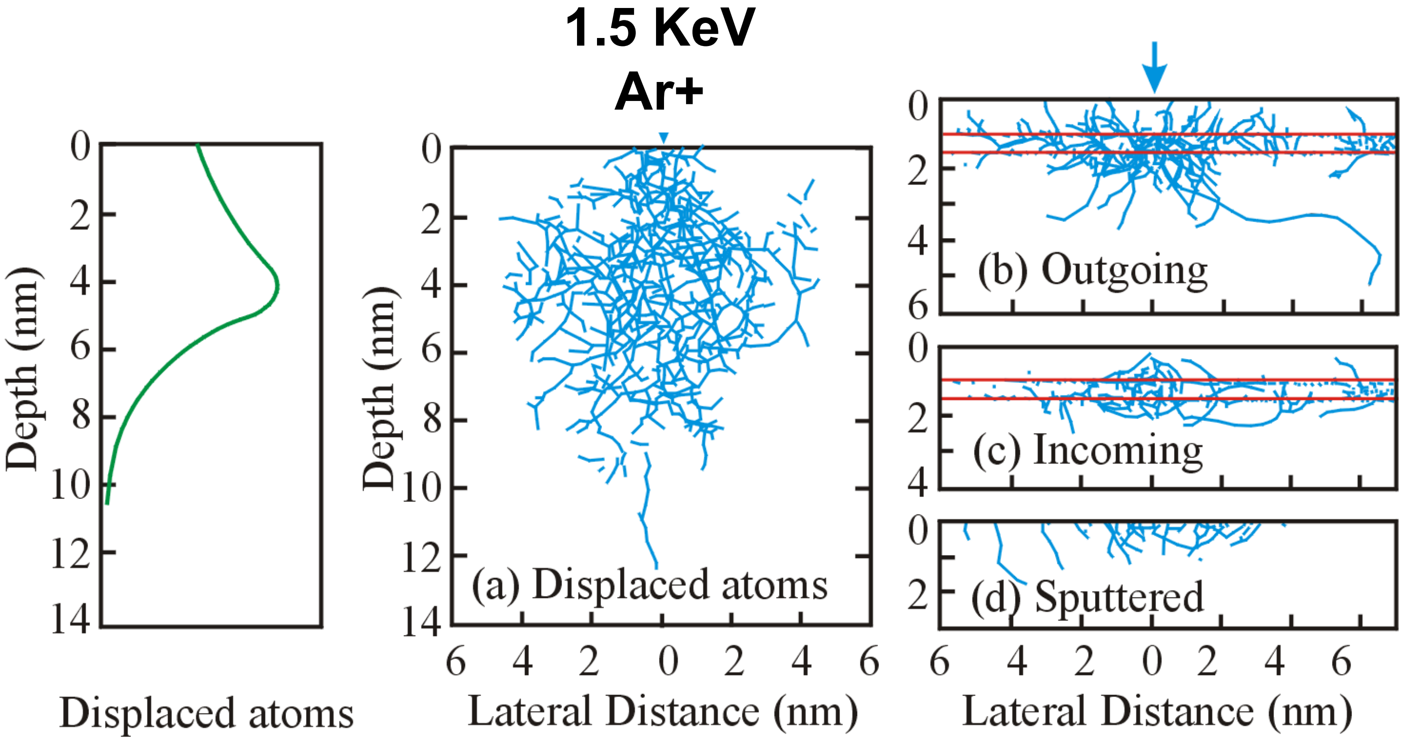
SPUTTERING PROCESS

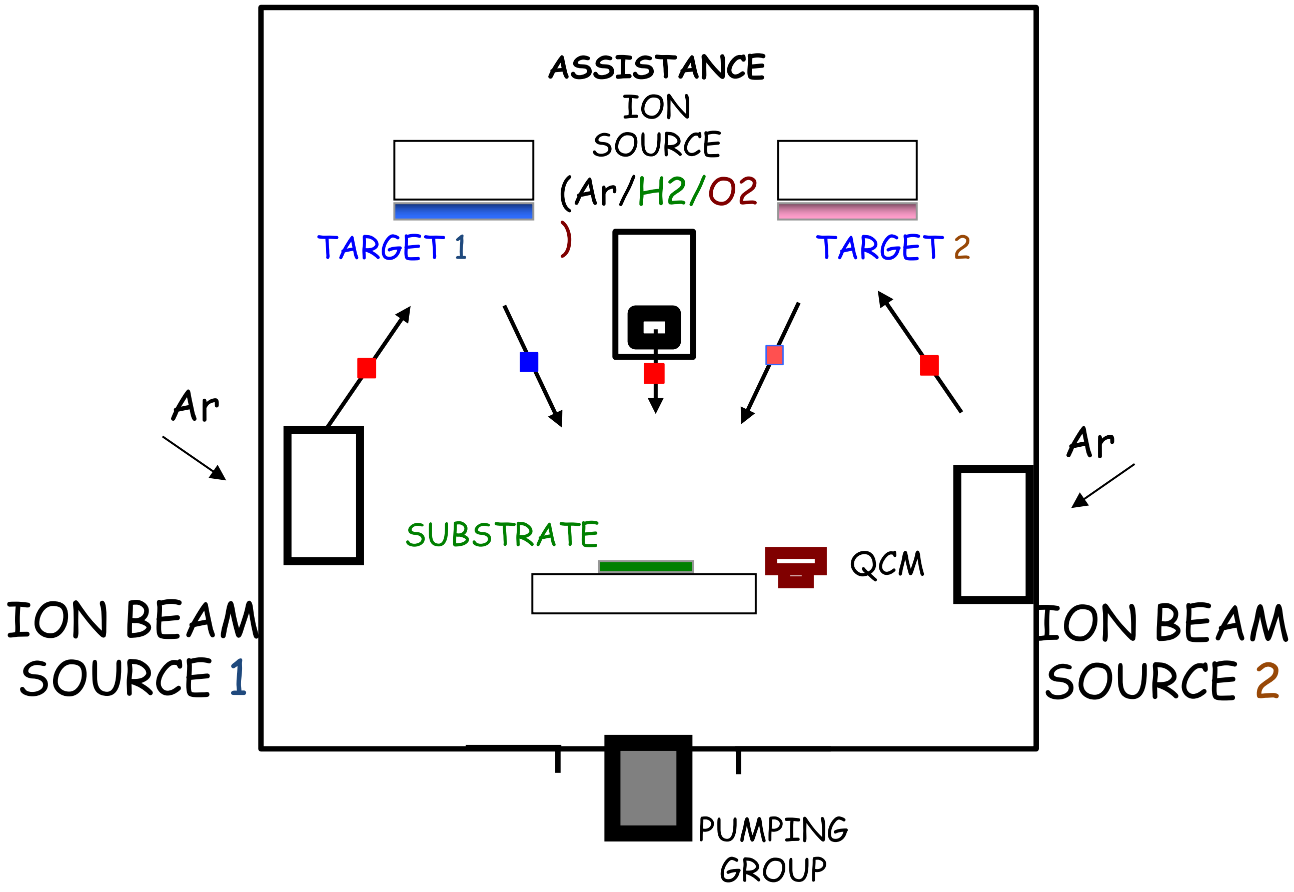


Simulated Trajectories



Computer simulation: Displacement of Cu atoms due to the impact of 1.5 keV Argon ions
(a) Trajectories within the entire volume of collision cascade for 10 incident particles
(b,c) Transport of target atoms out of and into the designated layer (20 incident particles)
(d) Trajectories of sputtered atoms (50 incident particles)

Dual Ion Beam Sputtering Scheme



Ion-beam sputtering Setup

- Room Temperature (23° C)
- Vacuum 10^{-5} mbar
- Pre-treatment with $O\bullet$ / $Ar\bullet$ radicals
- Ion Beam Source (1 keV, 100 mA)
- Assistant Source (100 eV, 1 A)
- Graphite Target: ∅10 cm
- Substrate: $6 \times 6\,\text{cm}^2$
- Quartz balance → film thickness
- Deposition speed ∼ 100 nm / hour

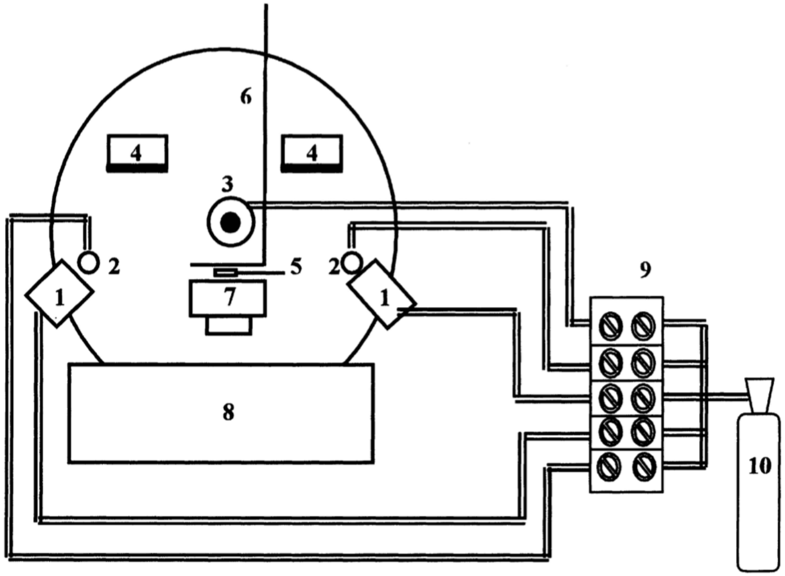


FIGURE 1 Schematic representation of the dual ion-beam sputtering deposition system used: (1) sputtering ion-beam sources, (2) plasma bridge neutralizers, (3) assistance ion-beam source, (4) targets, (5) quartz microbalance, (6) shutter, (7) substrate holder, (8) turbomolecular pump, (9) flow control unit, (10) argon supply

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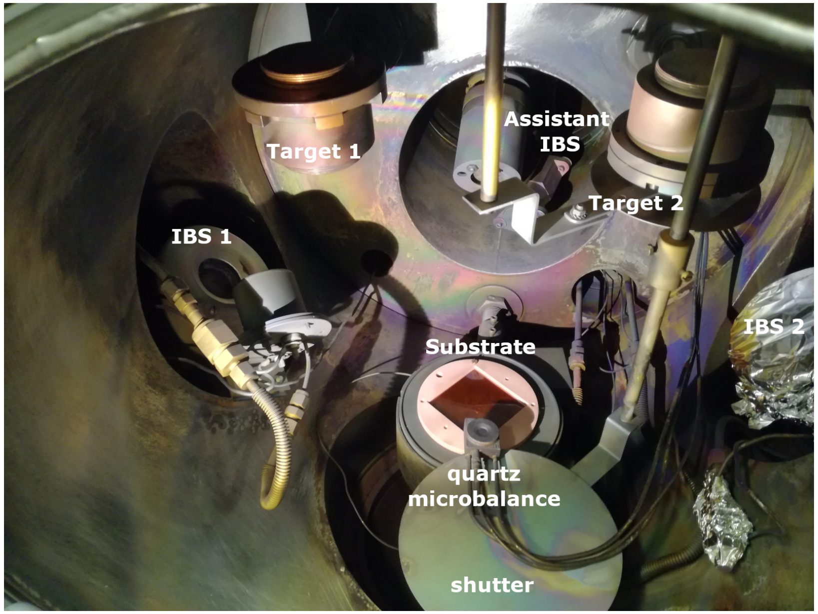
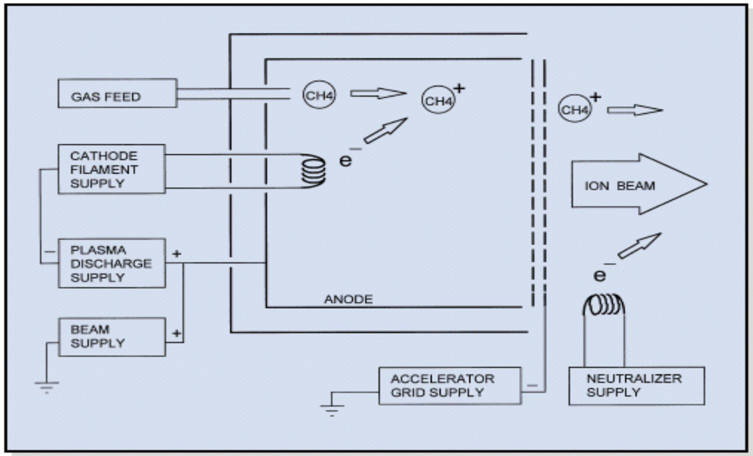
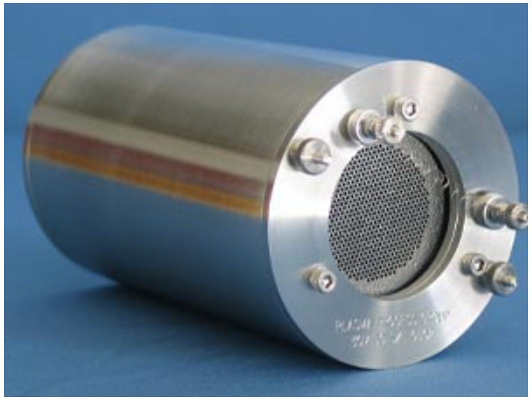
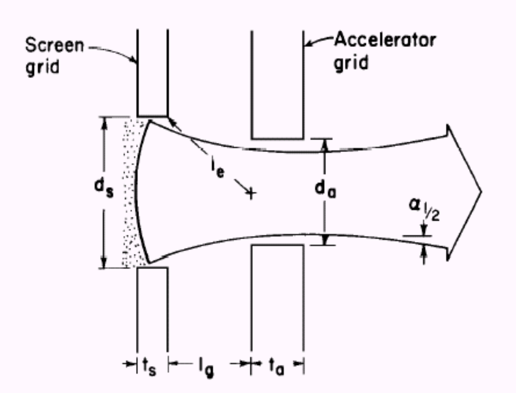


DUAL GRIDS ION SOURCE

High Energy

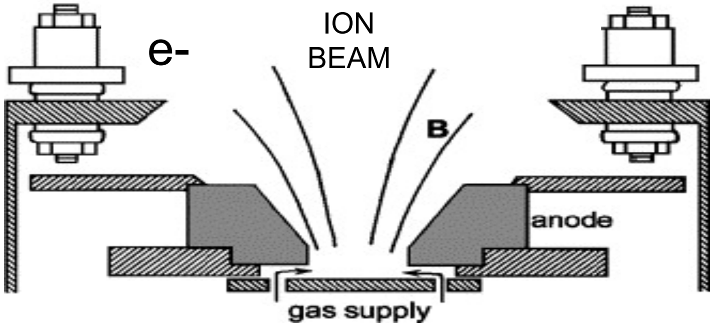






GRIDLESS ION SOURCE

High Current



Ion Beam Sputtering applied to DLC deposition on Kapton

The goals:

Good adhesion of DLC on Kapton
Sheet Resistance of about 100 MΩ

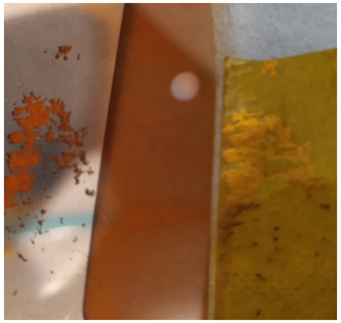
Adhesion tests of DLC on Kapton

To improve adhesion:

- Chemical surface cleaning
- ***Surface pretreatment*** with Ion Beam: **V_b=150V - I_b=1A - (5 sccm Ar + 4 sccm H_2)**

Scotch tape test results:

Copper on Kapton



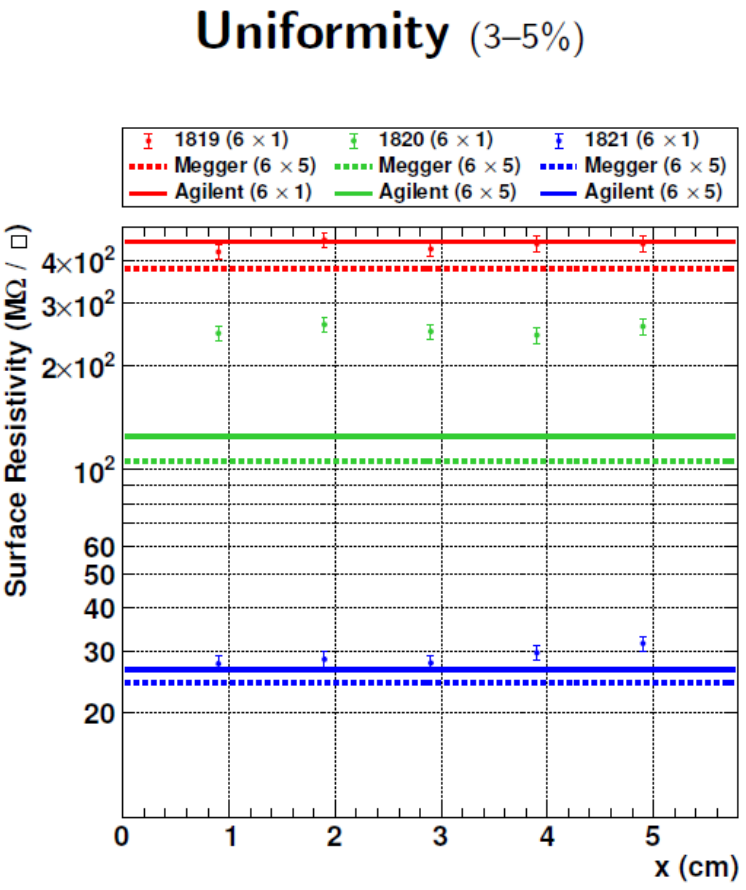
No Pretreatment

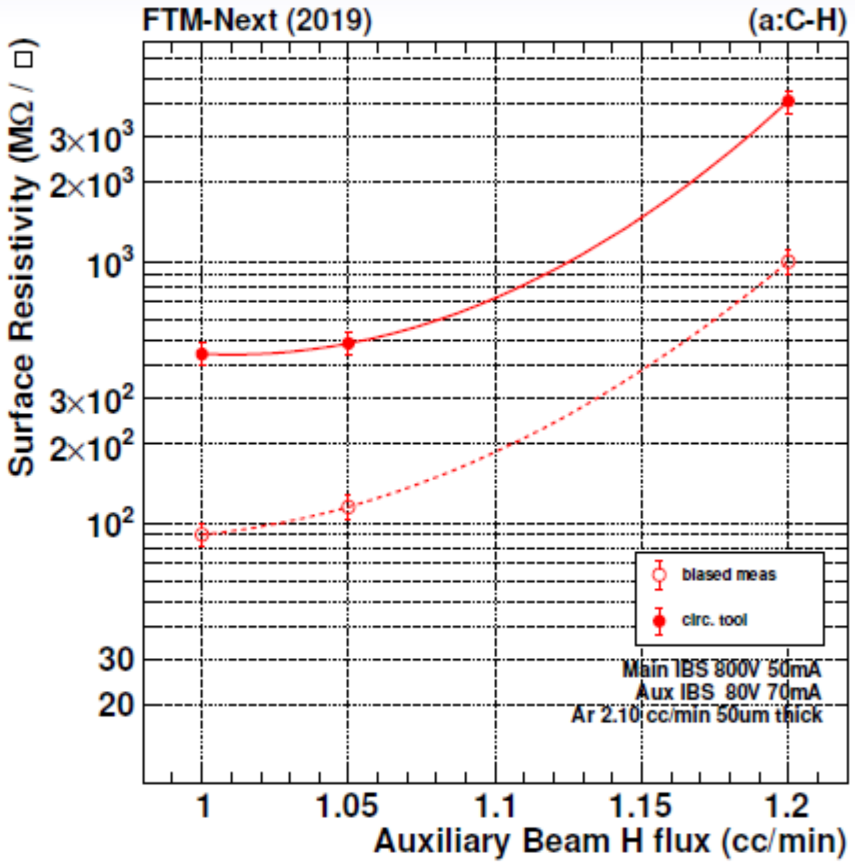
Pretreatment

DLC on Kapton

Pretreatment

Sheet Resistance Results


Uniformity (3–5%)

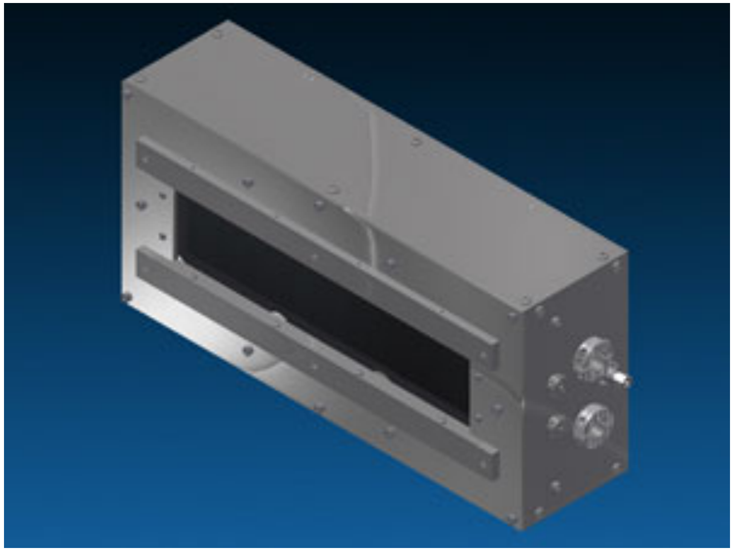

FTM-Next (2019)

Large Area Ion Beam Deposition System

400mm diameter *substrates*

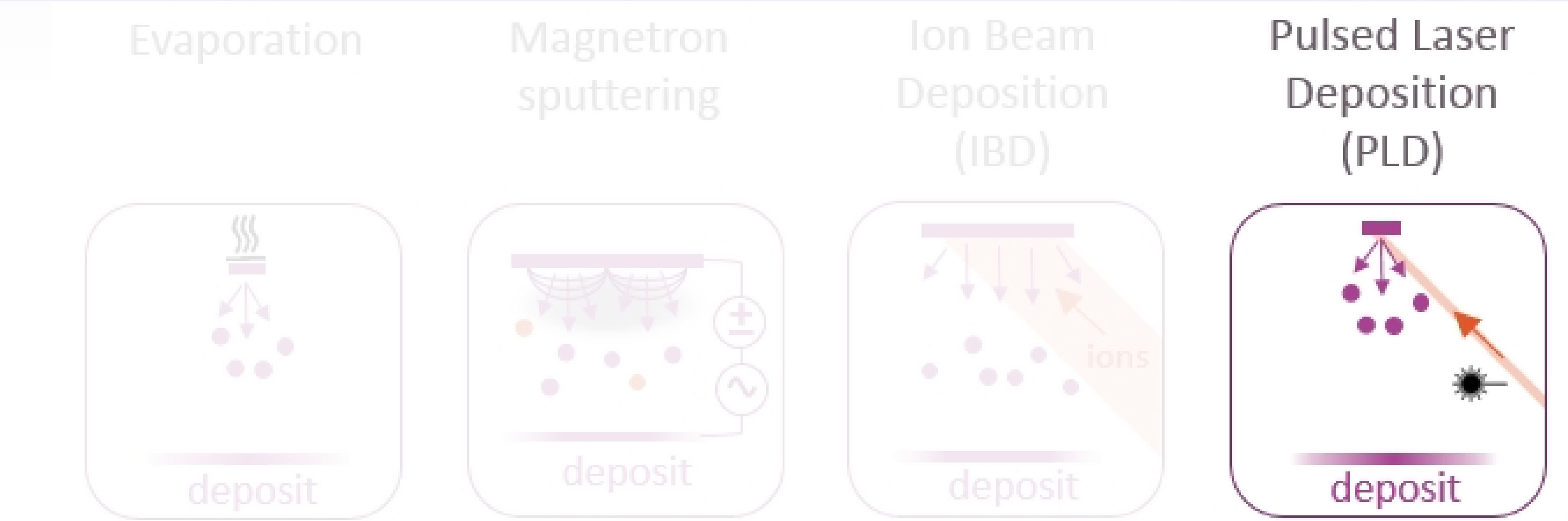
16cm RF *deposition source*
and
RF *neutralizers*

12cm RF *assist ion source.*



6 x 22/30 cm RF Ion Beam Source

Physical Vapor Deposition (PVD)



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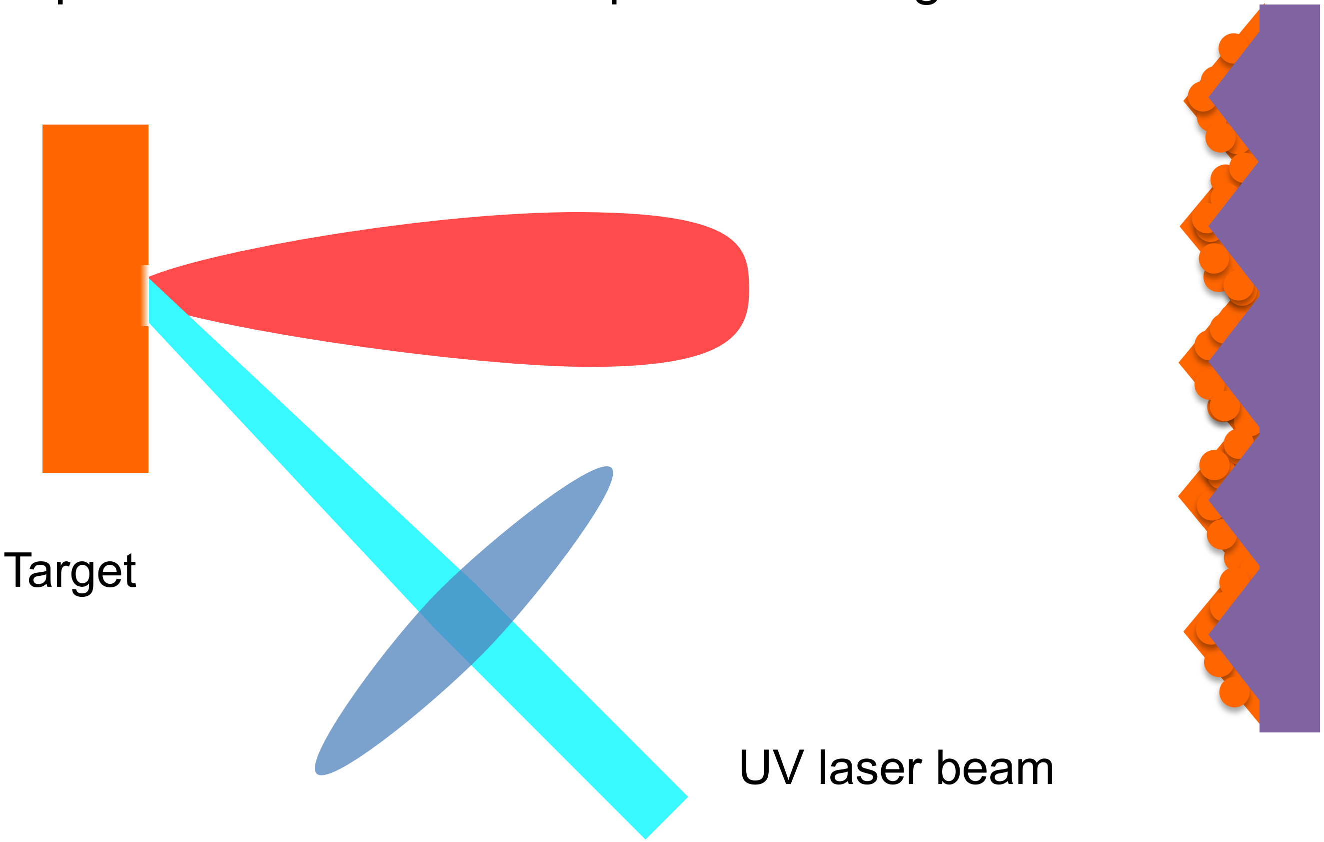
PLD for DLC films

Pulsed laser deposition is a “unique” technique for the deposition of hydrogen-free diamond-like carbon films.

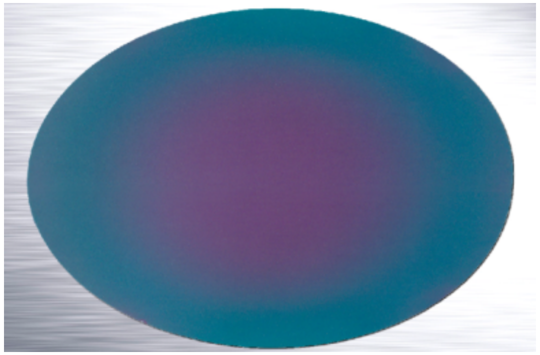
During deposition, amorphous carbon is evaporated from a solid target by a high-energy laser beam, ionized, and ejected as a plasma plume. The plume expands outwards and deposits the target material on a substrate.



PLD for DLC films

Advantages

- ✓ Stoichiometric transfer of material from target to substrate;
- ✓ Good control of the thickness (0.1 monolayer/pulse);
- ✓ Very few contaminants;
- ✓ High particles energies - Low substrate temperatures;
- ✓ Multilayer deposition in a single step;
- ✓ Deposition on flat and rough substrates;
- ✓ Many independent parameters



Drawbacks

- ✓ Low uniformity of the deposited film;
- ✓ Presence of droplets and particulates on the film surface.

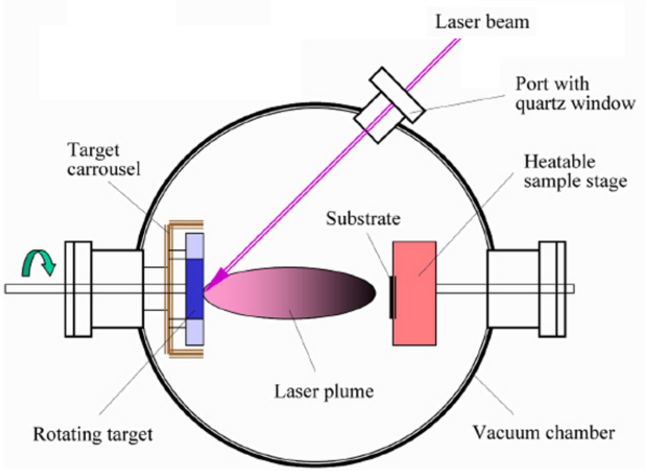
Pulsed Laser Deposition

Experimental Setup at INFN Lecce:

- multi-gas eximer laser
    - 248 nm - 193 nm
    - 1-20 Hz (20 ns)
    - 400 mJ → $1 - 6\,\mathrm{J/cm^2}$
- vacuum chamber with computer controlled movable substrate holder (can rotate)
- Atomic Force Microscopy (AFM - roughness)
- Four-Point Probe Station (VDP - Resistivity)
- Raman & X-ray spectroscopy (σ - sp^3/sp^2)
- Scanning Electron Micro (SEM - sp^3/sp^2)

First depositions:

- varying Fluence (J) to tune resistivity
- Raman to determine sp_3/sp_2 ratio



Experimental (first set of samples)

Target: pyrolytic graphite

KrF excimer laser: wavelength λ = 248 nm, pulse width τ = 20 ns, freq: f=10 Hz

Laser Fluence: 2,5 ÷ 5,5 J/cm^2

Target-substrate distance: d_{TS}: 55 ÷ 45 mm

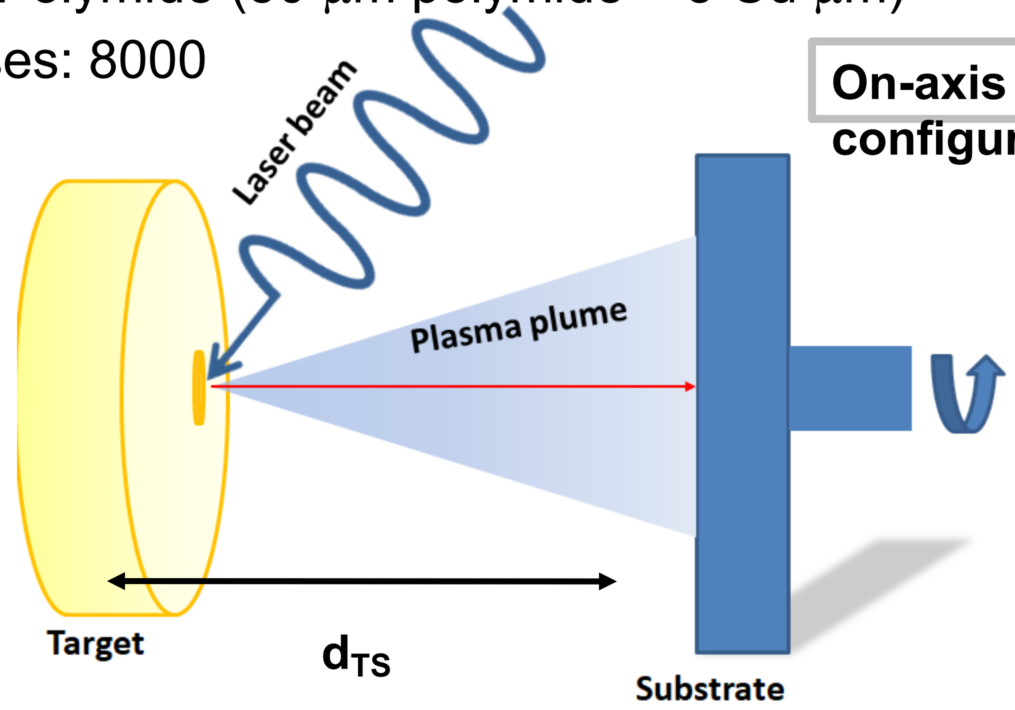
Background pressure: ~ 10^{-5} Pa

Laser spot area: ~ 4 mm^2

Substrates: Si/SiO$_2$, Polymide (50 μm polymide + 5 Cu μm)

Number of laser pulses: 8000

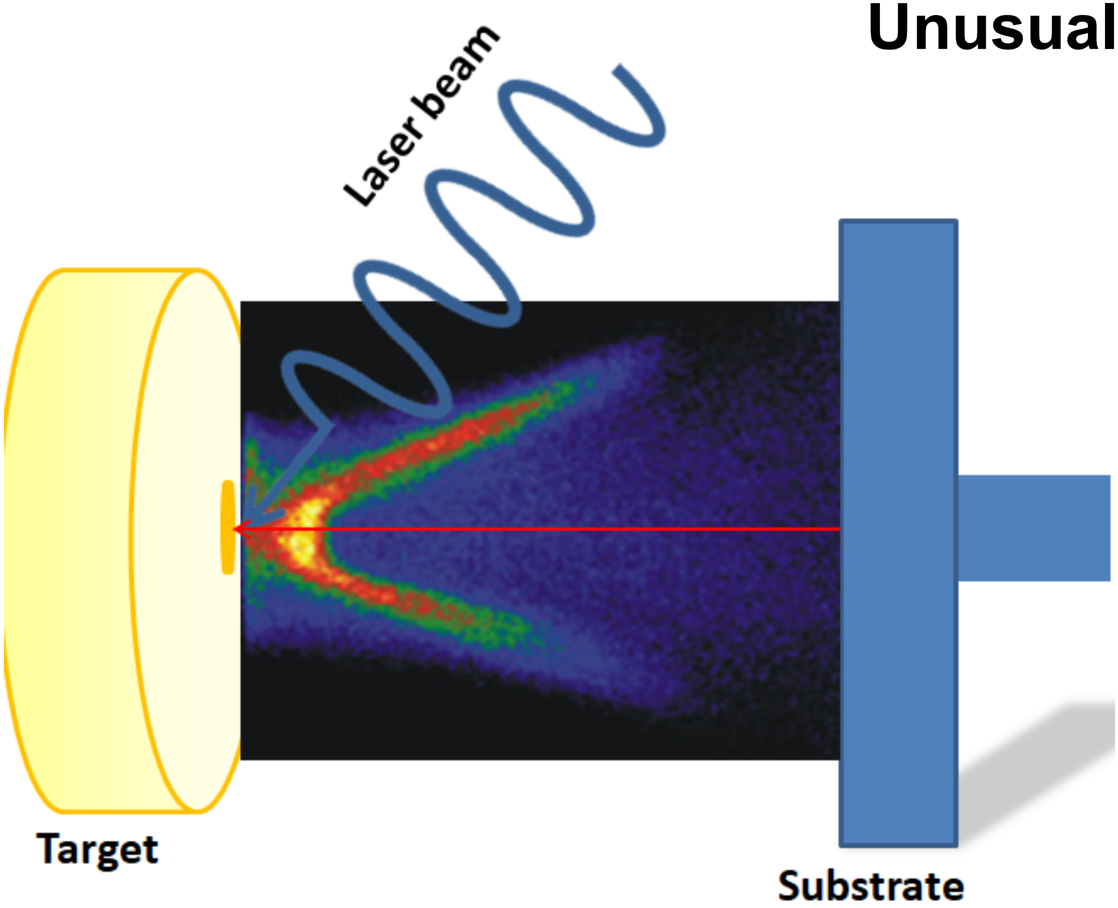
On-axis configuration

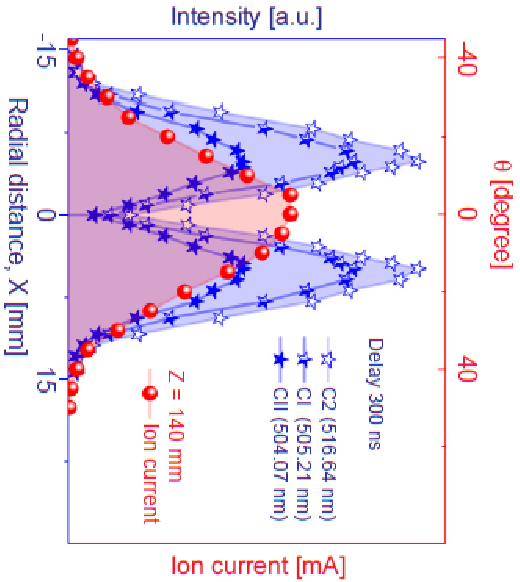


Second set of samples
(off-axis + substrate motion; big spot area)

Reason for non uniform films

Unusual plasma shape: V shape





C. Ursu, P. Nica, C. Focsa, Applied Surface Science 456 (2018) 717–725

DLC produced through PLD

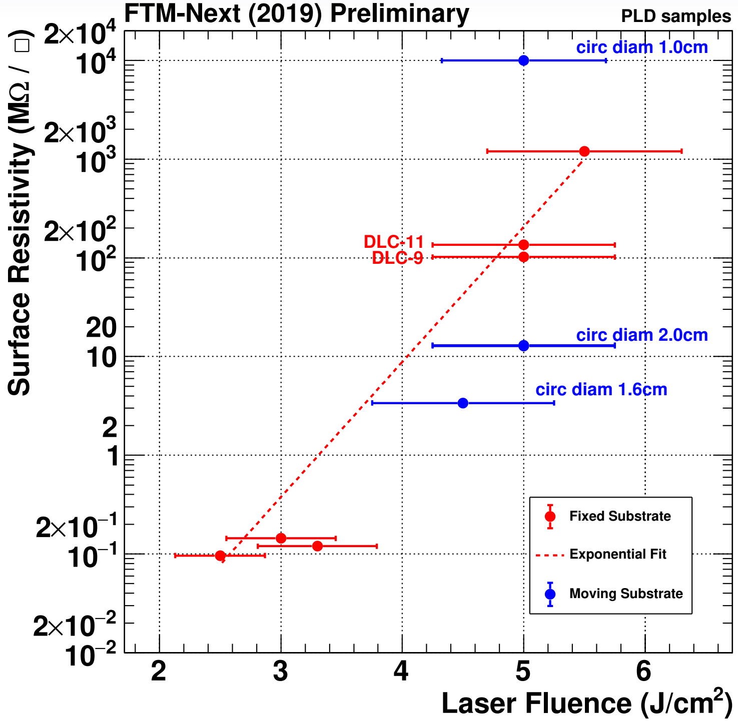
(top L) Resistivity tuned by Fluence

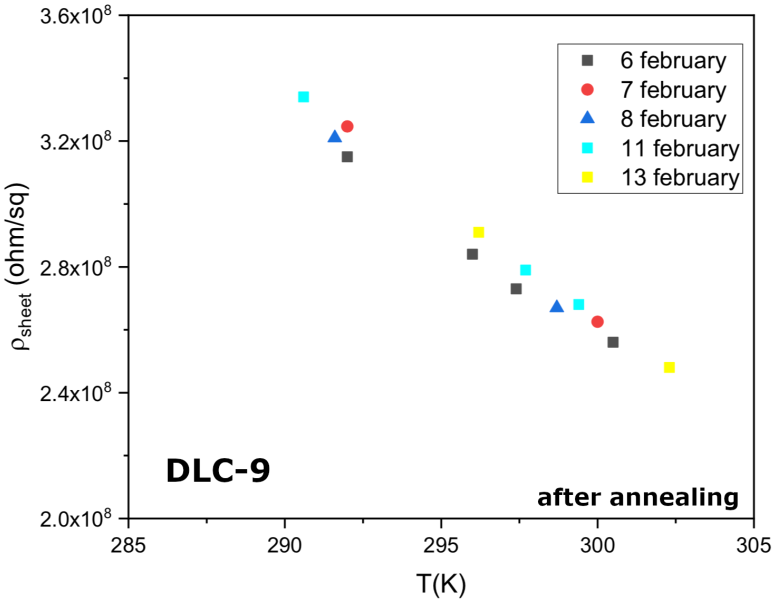
(top L) Target 100 MΩ/□ reached

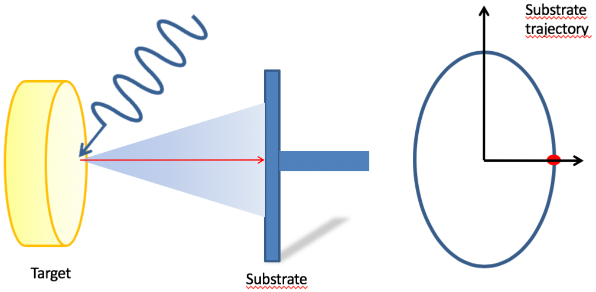
(bot L) PLD deposited samples stable in time

(bot R) Substrate rotation ⇒ Uniformity

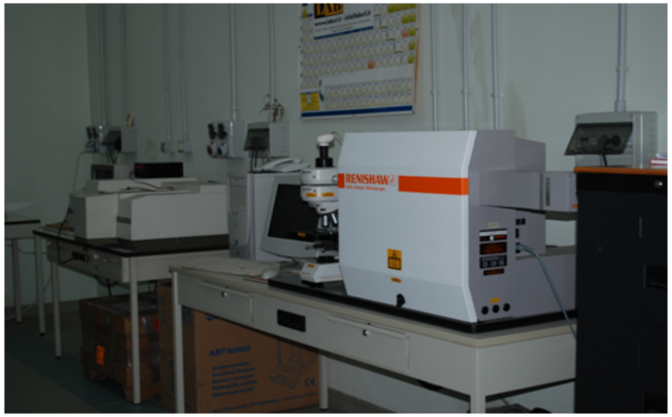
(future) Further characterization ongoing (Raman, XPS, AFM)







Characterization Techniques



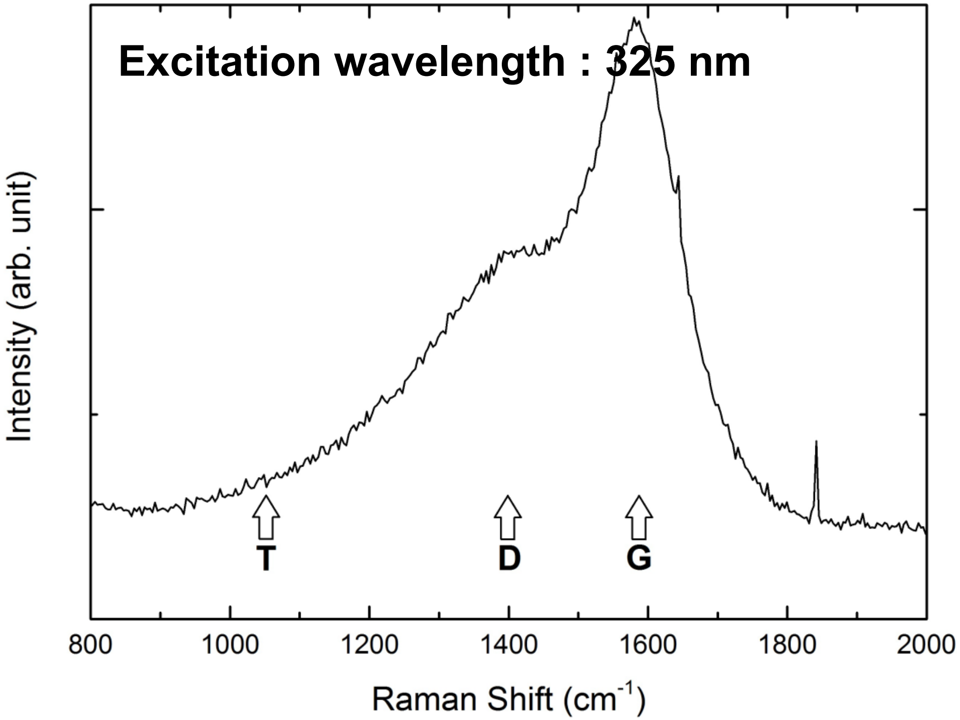
- Electrical measurements
- X-rays diffraction
- Transmission electron microscopy
- Scanning electron microscopy
- Scanning tunneling microscopy
- UV-Vis-NIR & Raman spectroscopy

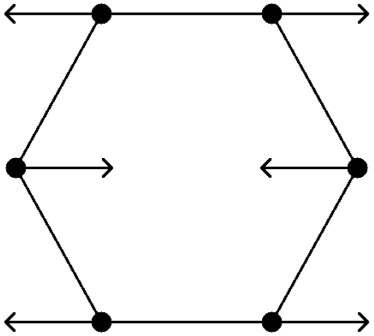
Raman spectroscopy

Under visible laser excitation
G peak (bond stretching of all pairs of sp^2 atoms in both rings and chains) → 1560 cm^{-1}

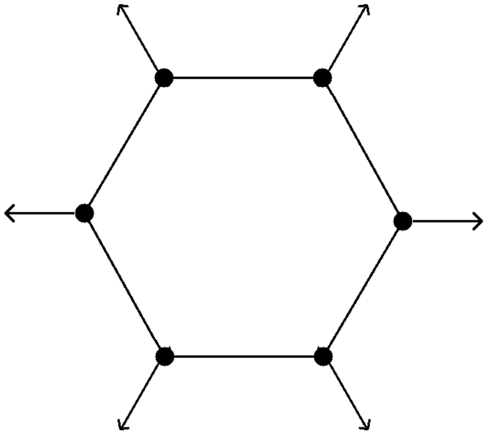
D peak (breathing modes of sp^2 atoms in rings) → 1360 cm^{-1}

Under UV laser excitation
T peak (C–C sp^3 vibrations)→1060 cm^{-1}





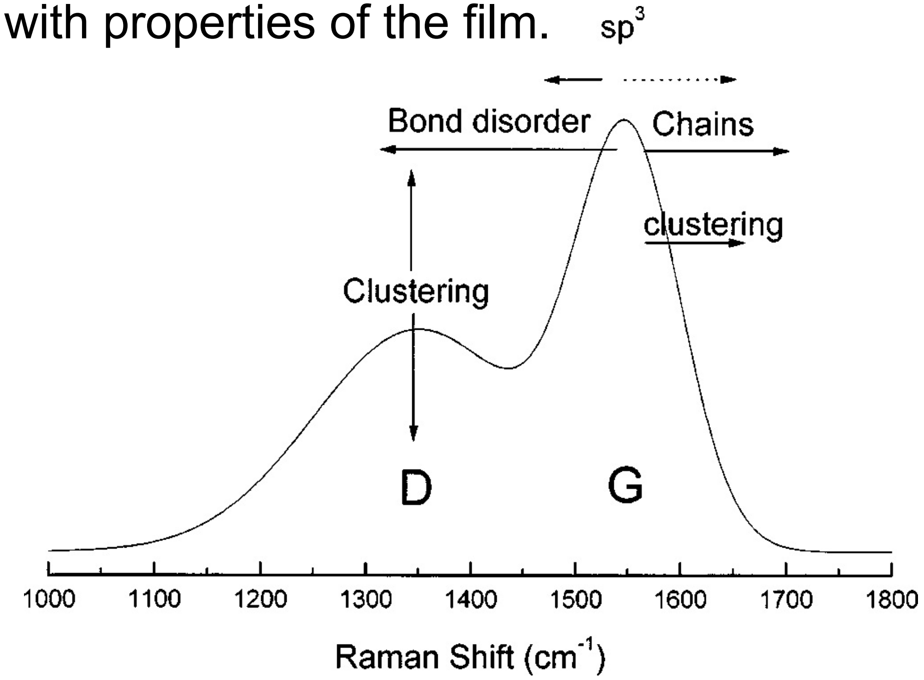
(A) E_{2g} , G peak mode



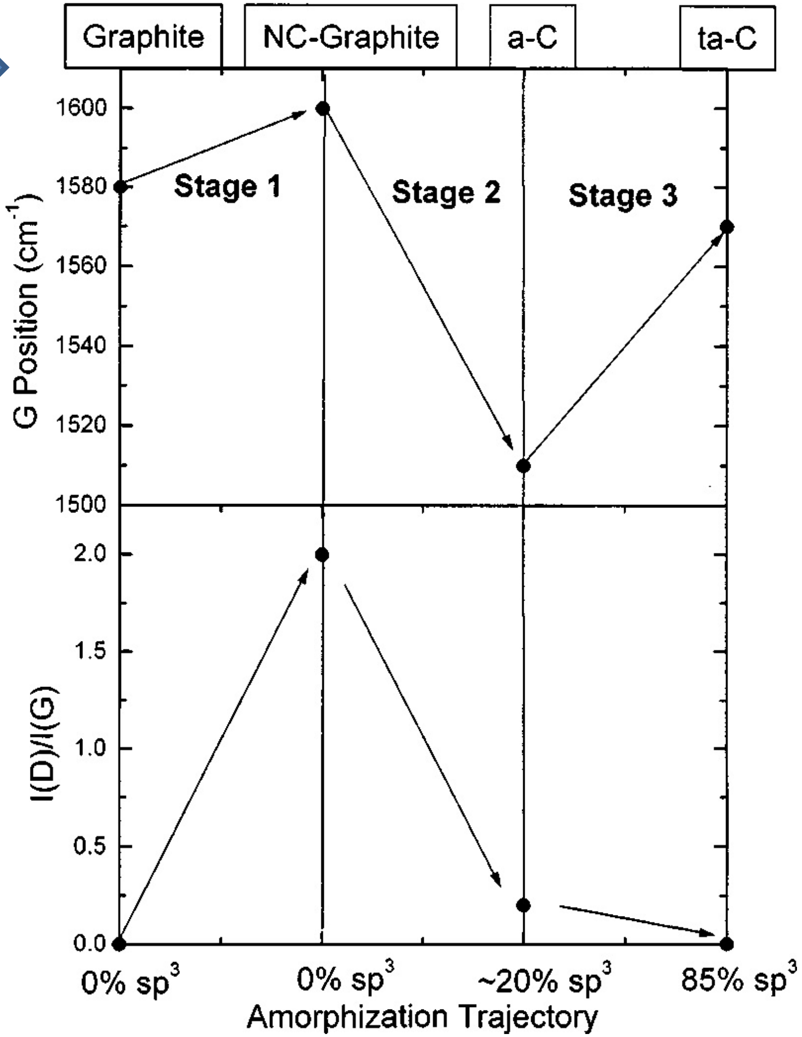
(B) A_{1g} , D peak mode

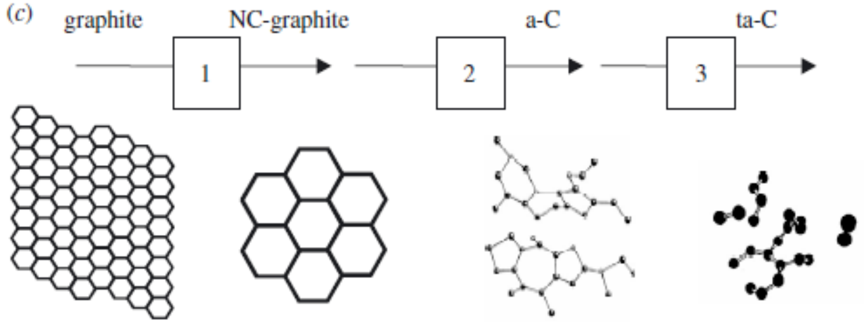
Three-stage model

Schematic model of how the D/G-peak cluster obtained with Raman spectroscopy changes
with properties of the film.



- sp^3 content
- sp^2 clusters size
- sp^2 cluster orientation





A. C. Ferrari and J. Robertson, Phil. Trans. R. Soc. Lond. A 2004 362, 2477-2512