Studies on DLC characterisation

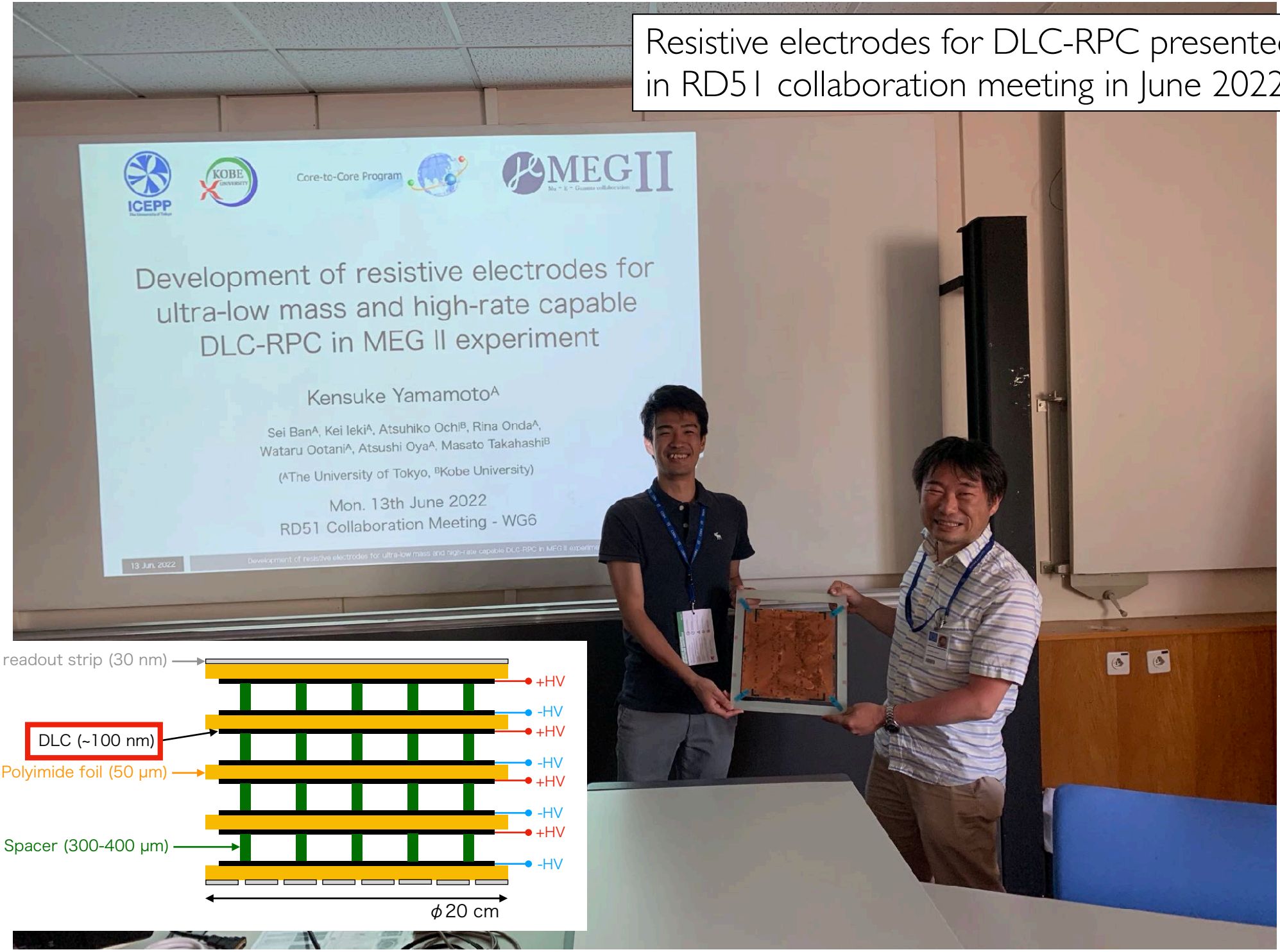
Kensuke Yamamoto[A]

S. Ban[A], W. Li[A], A. Ochi[B], W. Ootani[A], A. Oya[A], H. Suzuki[B], M. Takahashi[B]

([A]The University of Tokyo, [B]Kobe University)

RD51 Collaboration Meeting
4-8 December 2023

Resistive electrodes for DLC-RPC presented in RD51 collaboration meeting in June 2022



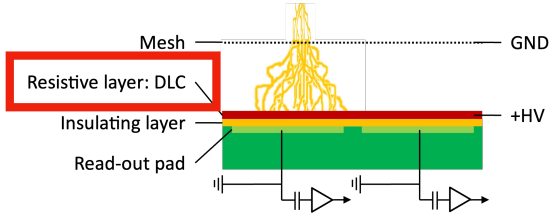
DLC in MPGD

- **Diamond-like carbon** is widely used in MPGD
  - As resistive material to prevent discharge

Resistive Micromegas

Advantages and requirements

- **Advantages of resistive Micromegas:**
  + limitation of the destructive effect of discharges
  + stable operation in intense pion beams
  + better position reconstruction, signal sharing
- **Objective:** profit from the advantages of the resistive Micromegas while maintaining good time resolution



Requirements for choosing the resistivity:

low enough to:

→ minimise the voltage drop during high rate beam

→ improve the position reconstruction

high eno

→ ensure stab

→ not affect the risin

MARTA LISOWSKA | MPGD2022 CONFERENCE | 11-16 DECEMBER 2022 TOWARDS ROBUST PICOSEC MICROMEGAS PRECISE TIMING DETECTORS

M. Lisowska

Cited from MPGD22

K.Gnanvo

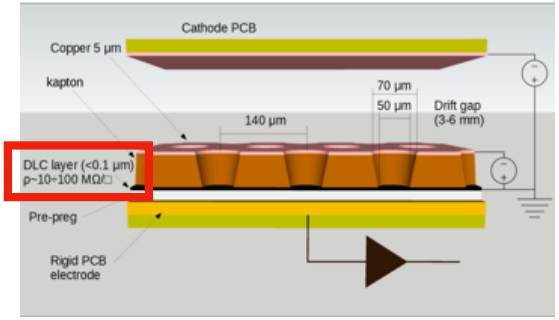
University of Virginia

CLAS12 Luminosity Upgrade: µRWELL Forward Tracker

Jefferson Lab Exploring the Nature of Matter

µRWELL is the chosen MPGD technology because of:

- ❖ Large area capability
- ❖ Low mass & compactness,
- ❖ Easy assembly, easy powering
- ❖ Robustness ➔ intrinsic spark quenching @ high gain → 10^4
- ❖ Excellent spatial resolution → < 100 µm
- ❖ Good time resolution → < 10 ns
- ❖ Rate capability for HR version of µRWELL →100 kHz/cm²

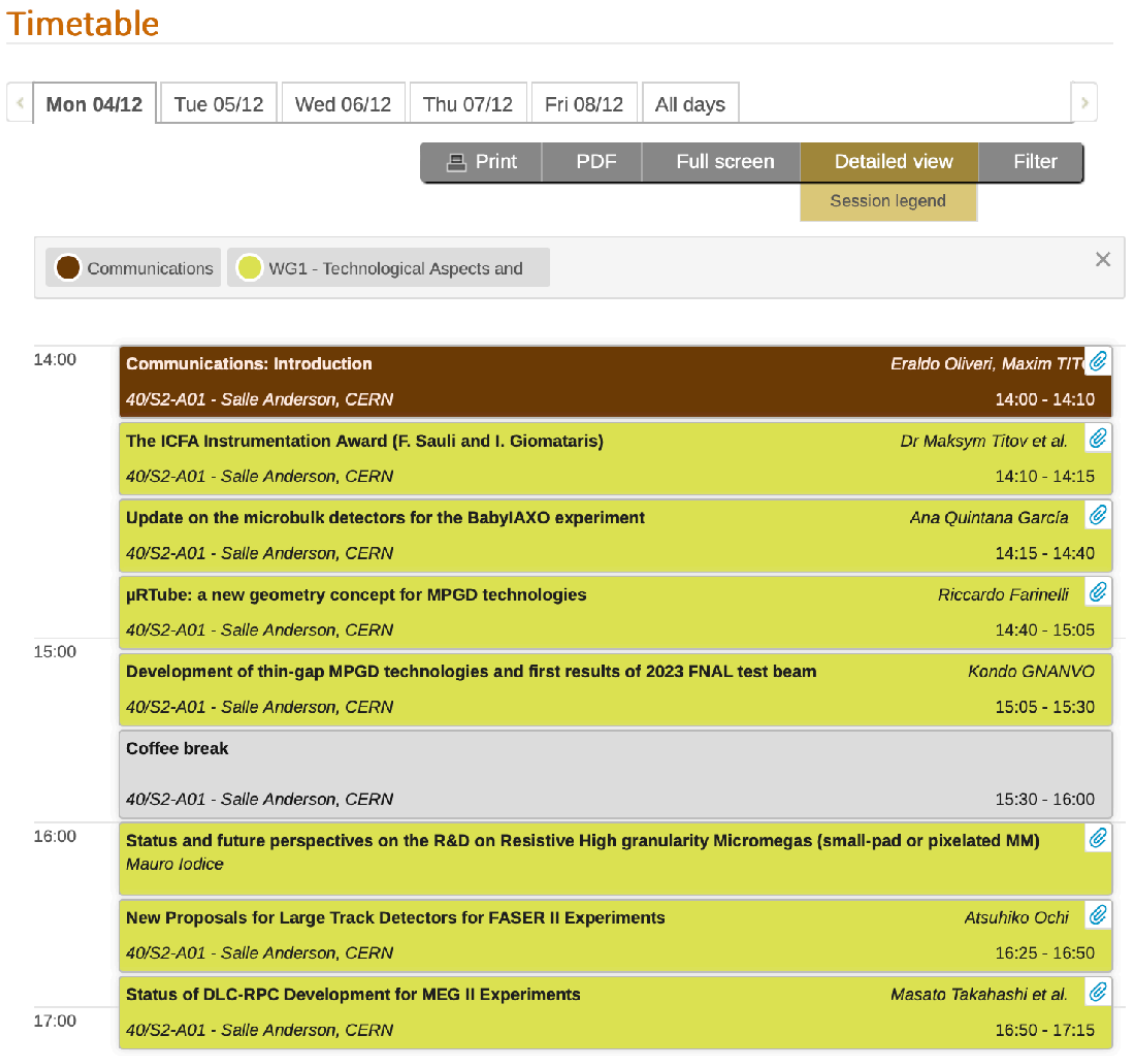


G. Bencivenni et al 2019 JINST 14 P05014

The 7th International Conference on Micro Pattern Gaseous Detector 2022 (MPGD2022), WIS, Rehovot, Israel - 12/12/2022 6

DLC in MPGD

- **Diamond-like carbon** is widely used in MPGD
  - As resistive material to prevent discharge



Demands for DLC get increasing

← μRTube

← **Resistive** MM

← μRWELL

← **DLC**-RPC

DLC in MPGD

- **Diamond-like carbon** is widely used in MPGD
  - As resistive material to prevent discharge

Supply of DLC starting

Cited from MPGD22

Sputtering machine at CERN started working

Title of project: *DLC based electrodes for future resistive MPGDs*

Contact person: *name: Yi Zhou*
address: Jinzhai Road No.96, Hefei, Anhui, P.R.China, 23
telephone number: +86-551-63607940
e-mail: zhouyi@mail.ustc.edu.cn

RD51 Institutes: **1.** *State Key Laboratory of Particle Detection and Electron*
University of Science and Technology of China,
contact person: Yi Zhou
e-mail: zhouyi@mail.ustc.edu.cn

2. *Kobe University,*
contact person: Atsuhiko Ochi
e-mail: ochi@kobe-u.ac.jp

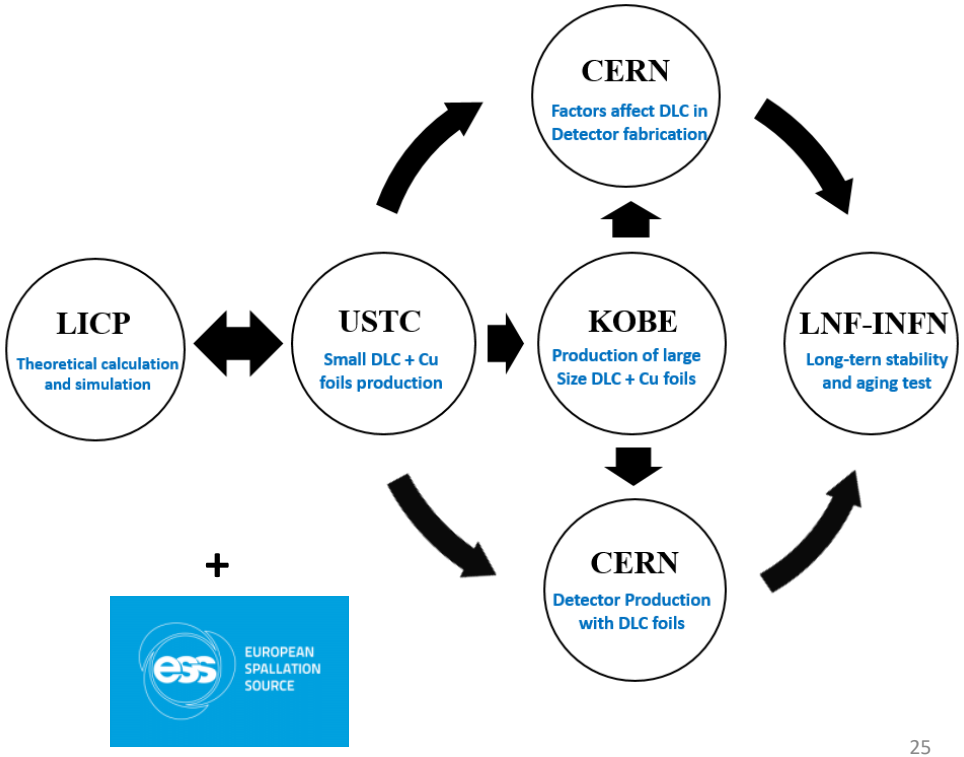
pictures

Machine in operation

70cm copper target installation

Drum unloading
after processing

DLC collaboration



R. De Oliveria

What we know about DLC

- **Used in various industries**, e.g. coating
  - Characteristics should be known in the field of material science
  - Few documentation on DLC deposited by physical sputtering method

- **Amorphous carbon**
  - Properties of both sp^2 and sp^3

- Attached well on **polyimide**

- **Resistivity can be controlled** by
  - Nitrogen doping for resistivity reduction
  - Thickness with an accuracy of 100%
  - Thermal annealing with an accuracy of 10%

What we **want to know** about DLC

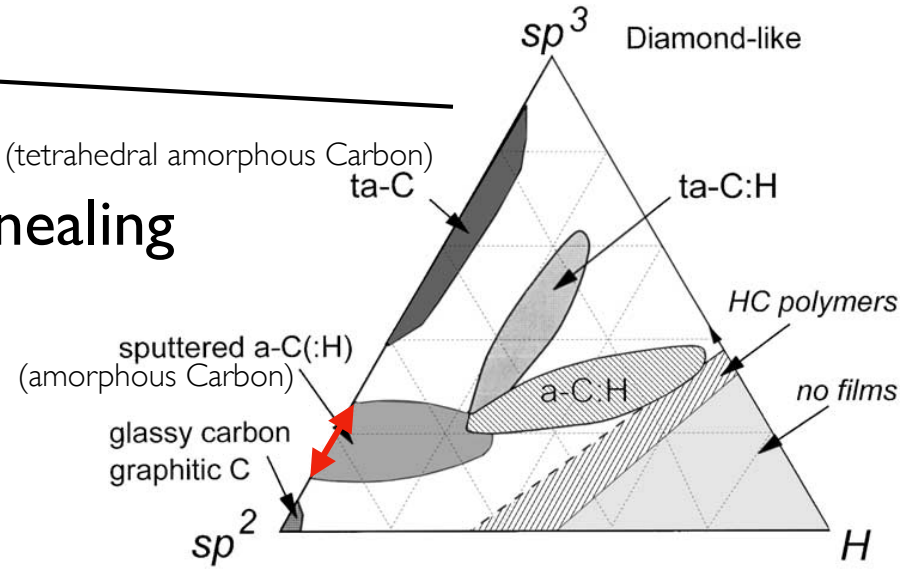
- **Used in various industries**, e.g. coating
  - Characteristics should be known in the field of material science
  - Few documentation on DLC deposited by physical sputtering method
  - ➡ Somehow import knowledge into gaseous detectors in HEP
- **Amorphous carbon**
  - Properties of both sp^2 and sp^3
  - ➡ What is the fraction between sp^2 and sp^3?
- Attached well on **polyimide**
  - ➡ Can DLC be deposited on other substrates?
- **Resistivity can be controlled** by
  - Nitrogen doping for resistivity reduction
  - Thickness with an accuracy of 100%
  - Thermal annealing with an accuracy of 10%
  - ➡ What is the mechanism of thermal annealing?

Today's topics

- Found a great review paper
  - J. Robertson, *Mater. Sci. Eng. R Rep.* **2002**, 37, 129–281
  - https://doi.org/10.1016/S0927-796X(02)00005-0

- **Molecular structure analysis (Raman spectroscopy)**
  - DLC tried to be sputtered on some substrates
  - sp^2 and sp^3 fraction
  - DLC categorisation

- **Resistivity control by thermal annealing**
  - Mechanism
  - Our measurement



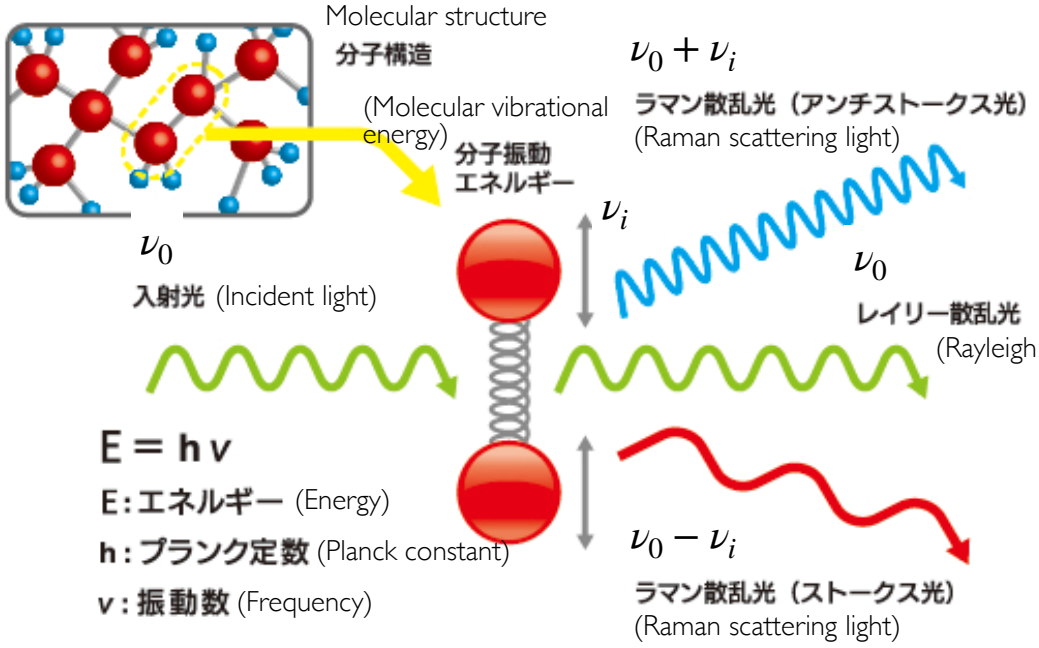
Ternary phase diagram

Raman scattering spectroscopy

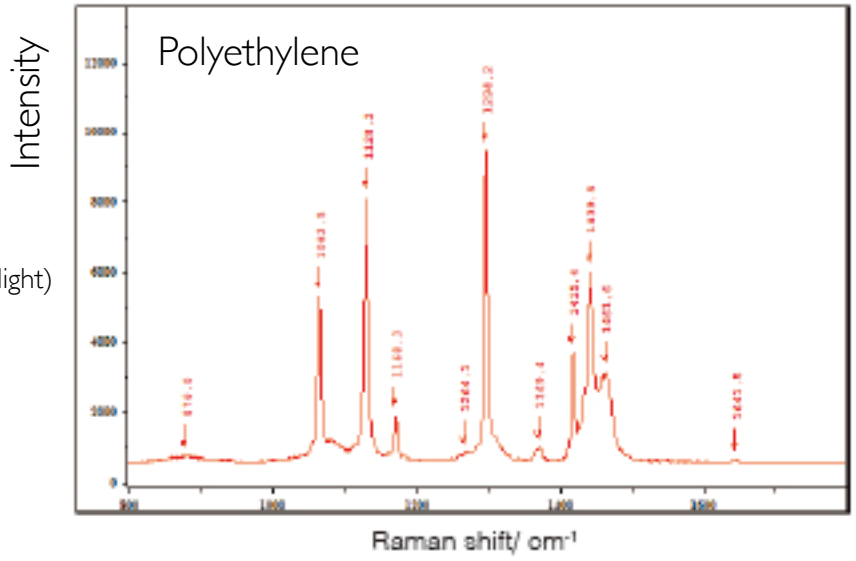
- **Measure molecular structure of DLC by Raman spectrum**

- Raman spectrum information
  - Intensity ratio: material ratio
  - Raman shift: structure
  - …

- Polyimide is not proper for Raman spectroscopy
  - Consists of C



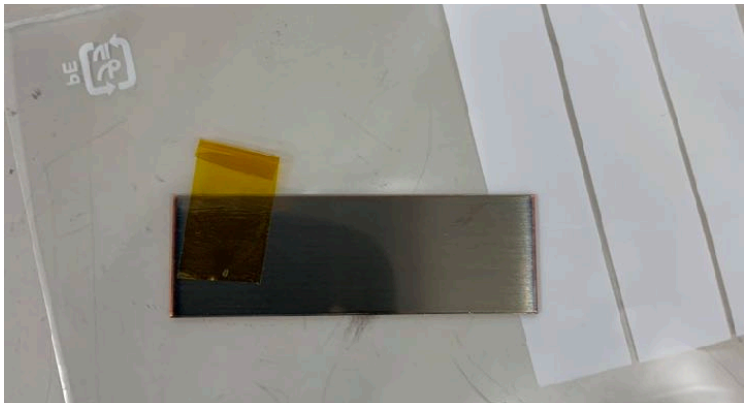
https://www.horiba.com/jp/scientific/products-jp/raman-spectroscopy/about-raman/1/



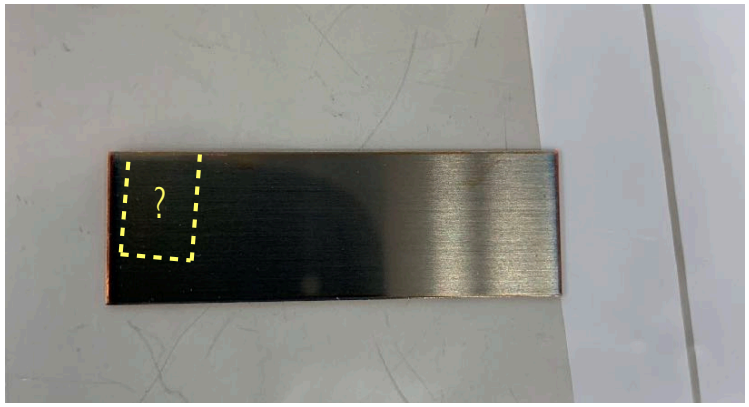
Wavenumber shift of Raman scattering light $1/\lambda_i = \nu_i/c$

DLC sputtering onto some materials

- DLC sputtered on polyimide so far



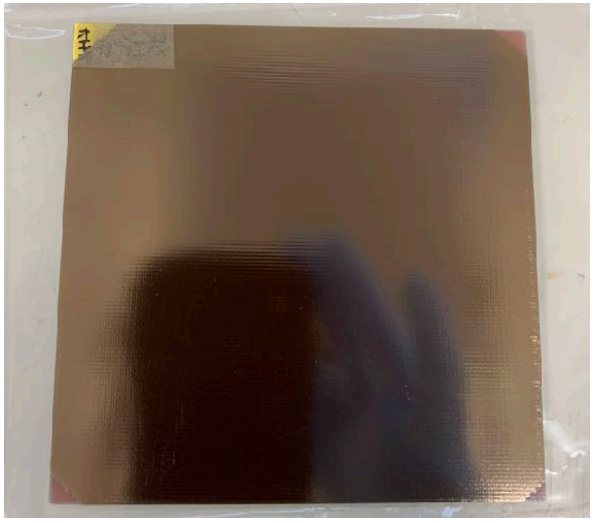
Kapton tape put



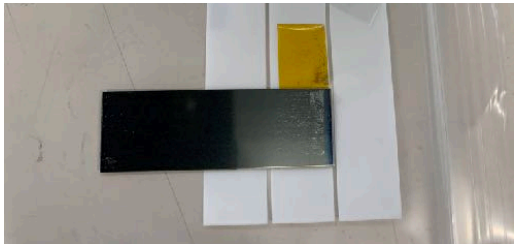
See the situation after removal
(Peeling test)

DLC on glasses

DLC on glass epoxy



DLC on 6 types of metal
(Zn, Ni, Pb, Cu, Fe, Al)



Peeling test result

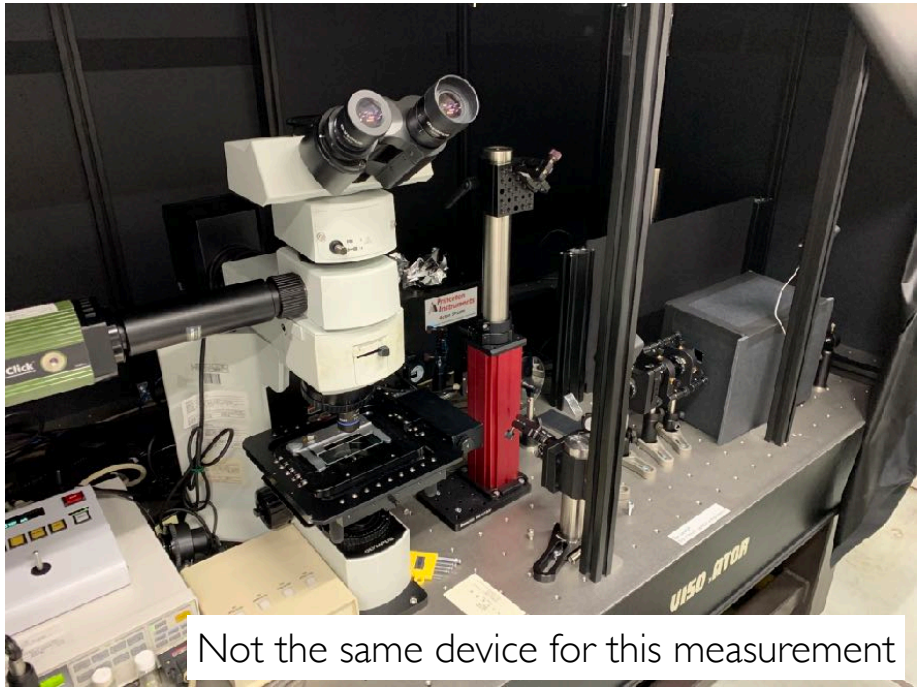
Material	Result	Material	Result
Polyimide	O	Zinc (Zn)	△
Glass-epoxy (FR4)	O	Nickel (Ni)	X
Heat-resistant glass	X	Lead (Pb)	△
Tempered glass	X	**Copper (Cu)**	O
Quartz glass	X	Iron (Fe)	X
		Aluminium (Al)	X

Used for
Raman spectrum measurement

Raman spectroscopy setup

- Measurement device
  - Thanks to S. Chiashi in *Dept. of Mechanical Engineering, UTokyo*
  - 4 lasers with different excitation wavelengths (488–785 nm)

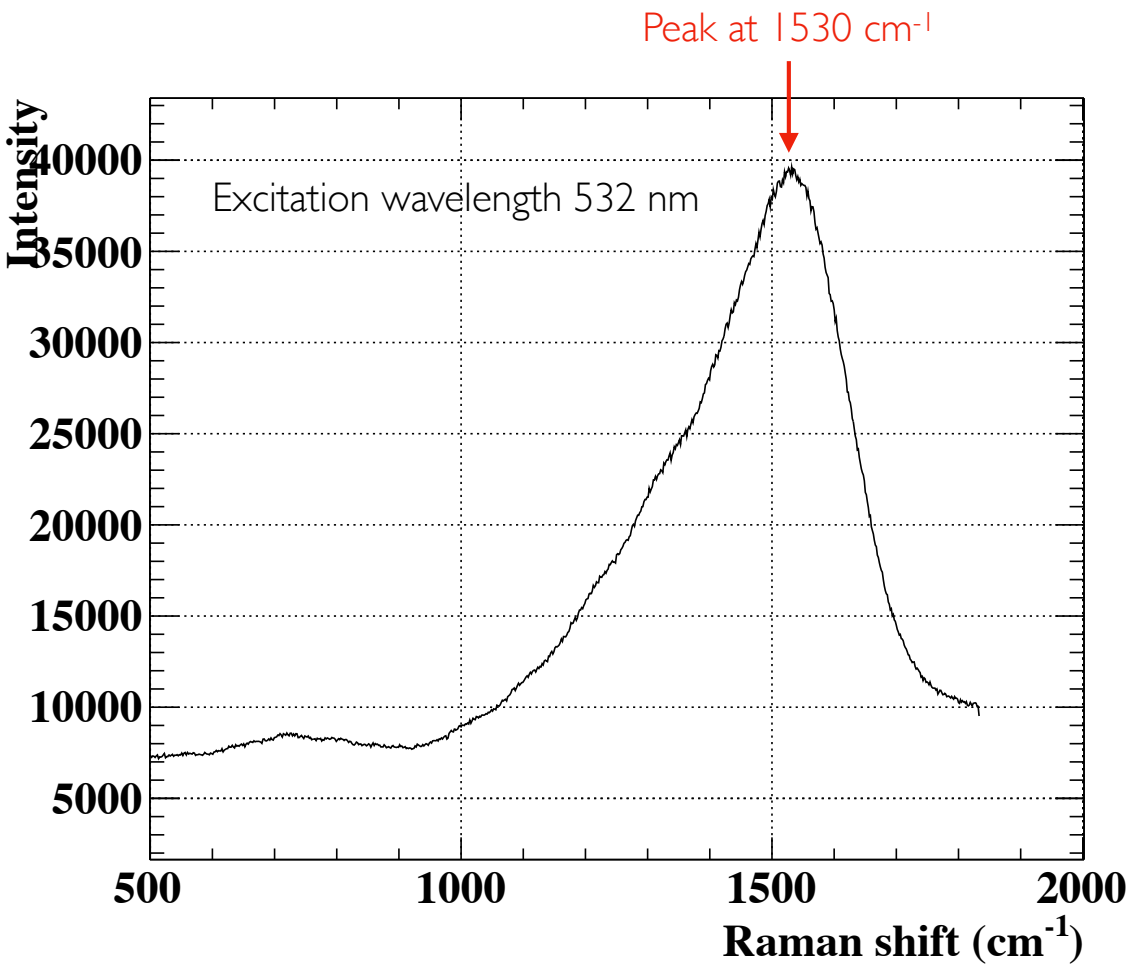
- Samples
  - DLC sputtered on Cu with and without thermal annealing
  - Annealing: 200°C for 30 minutes

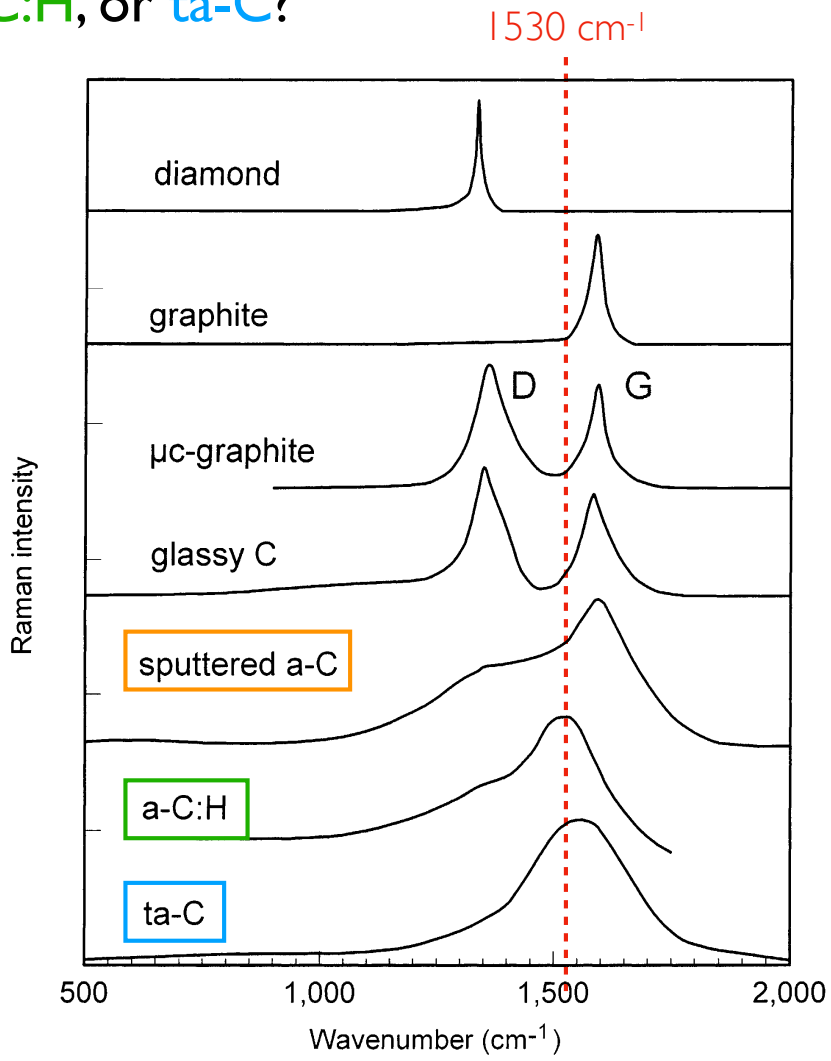


Not the same device for this measurement

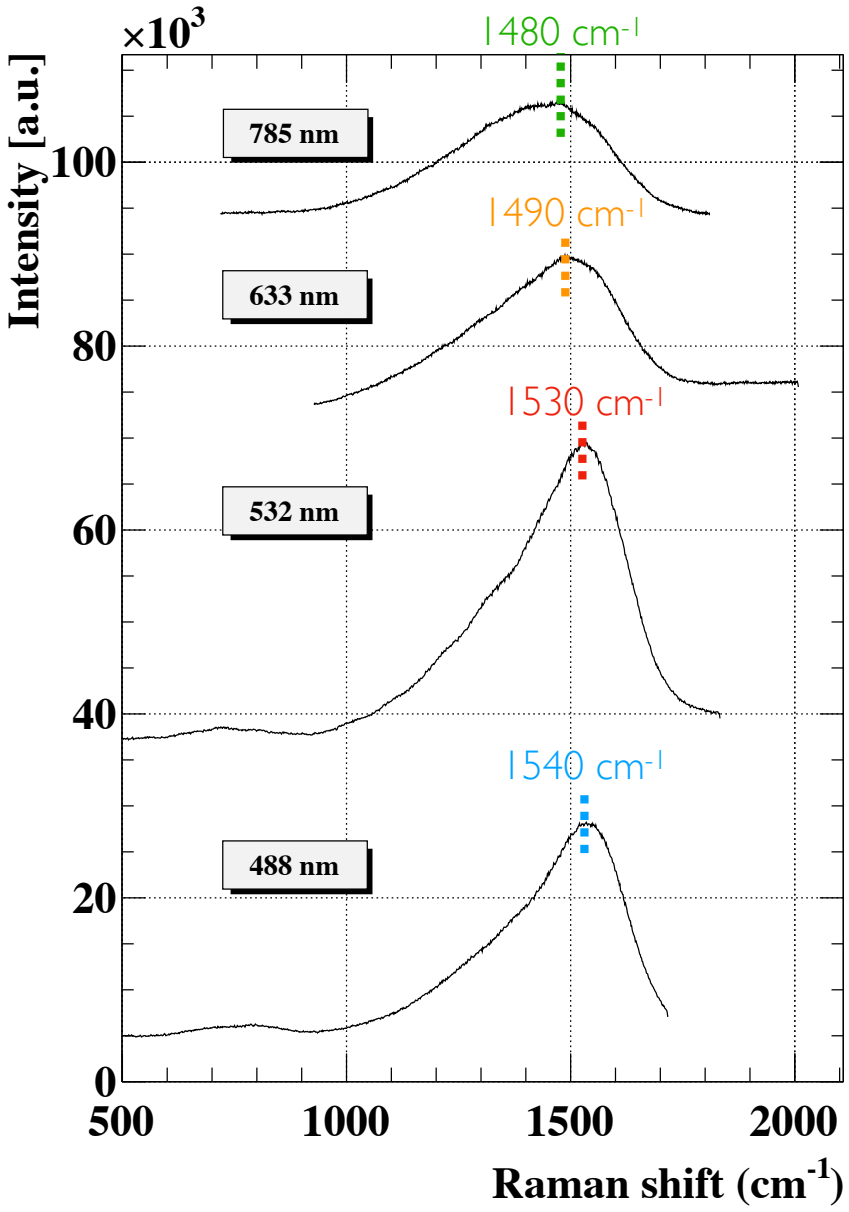
Raman spectrum of non-annealed sample

- Succeeded in measuring Raman spectrum
- Our DLC categorised as sputtered a-C, a-C:H, or ta-C?





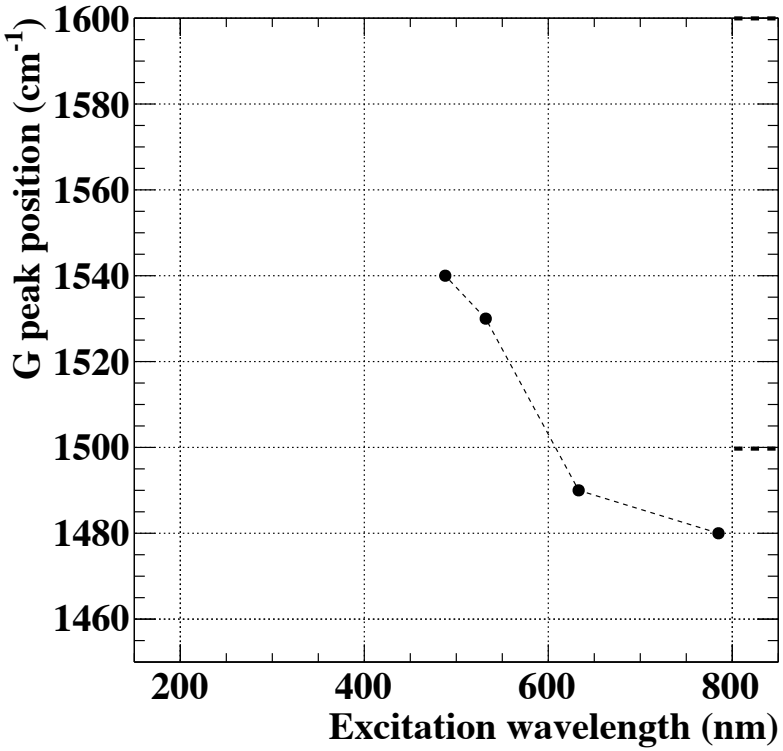
Excitation wavelength dependence

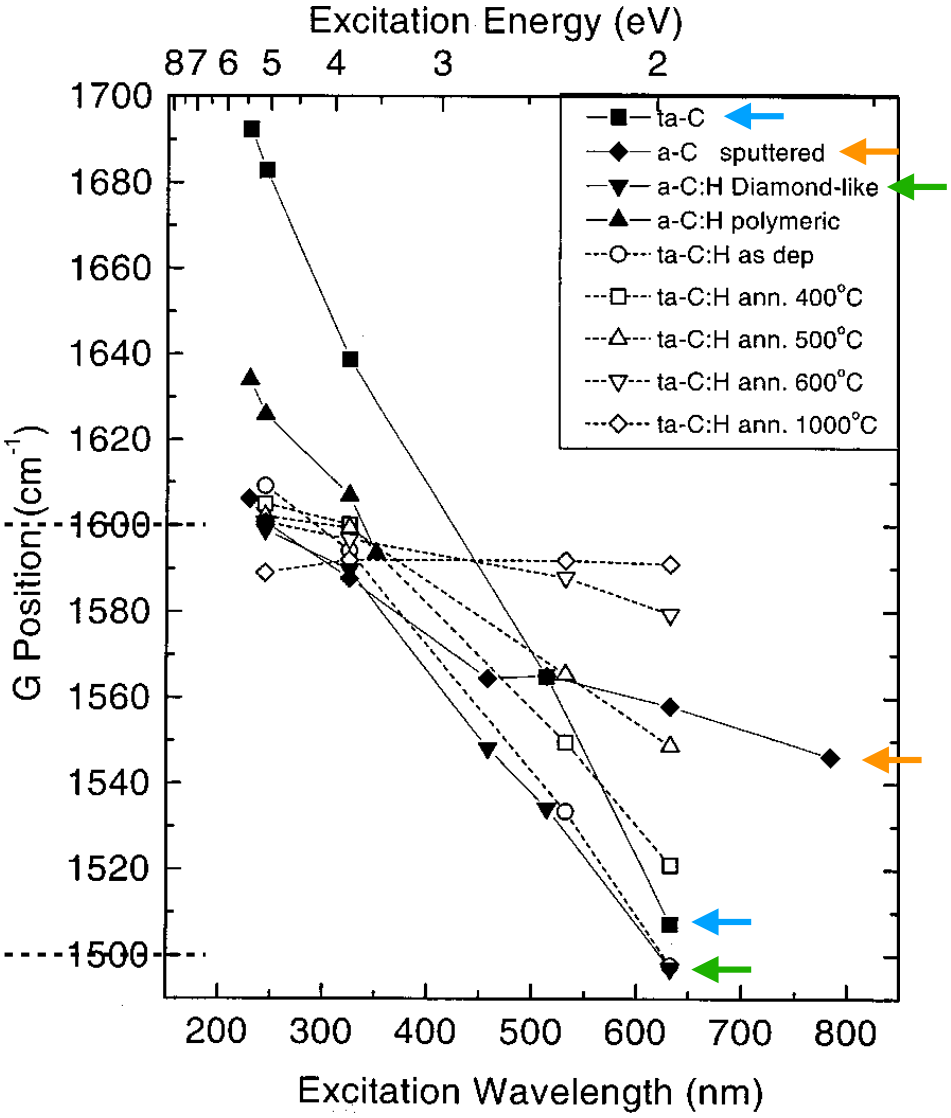


- Measured with different excitation wavelengths
  - Dependence on peak position observed
- Compare with the prior studies

Excitation wavelength dependence

Excitation wavelength dependence of our DLC is similar to that of a-C:H



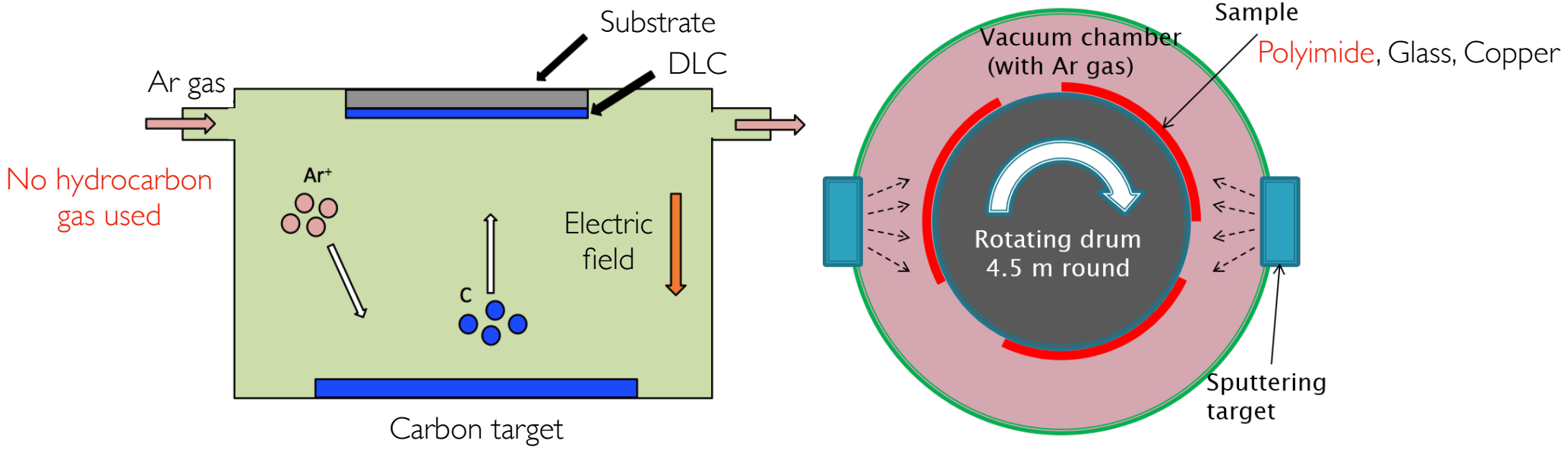


A. C. Ferrari and J. Robertson, Phys. Rev. B 64, 075414

Where hydrogen comes from?

- It is known DLC contains hydrogen if hydrocarbon gas flows in physical sputtering method

- **But, we do NOT use any hydrocarbon gas**
  - Outgas from polyimide could contain hydrogen?

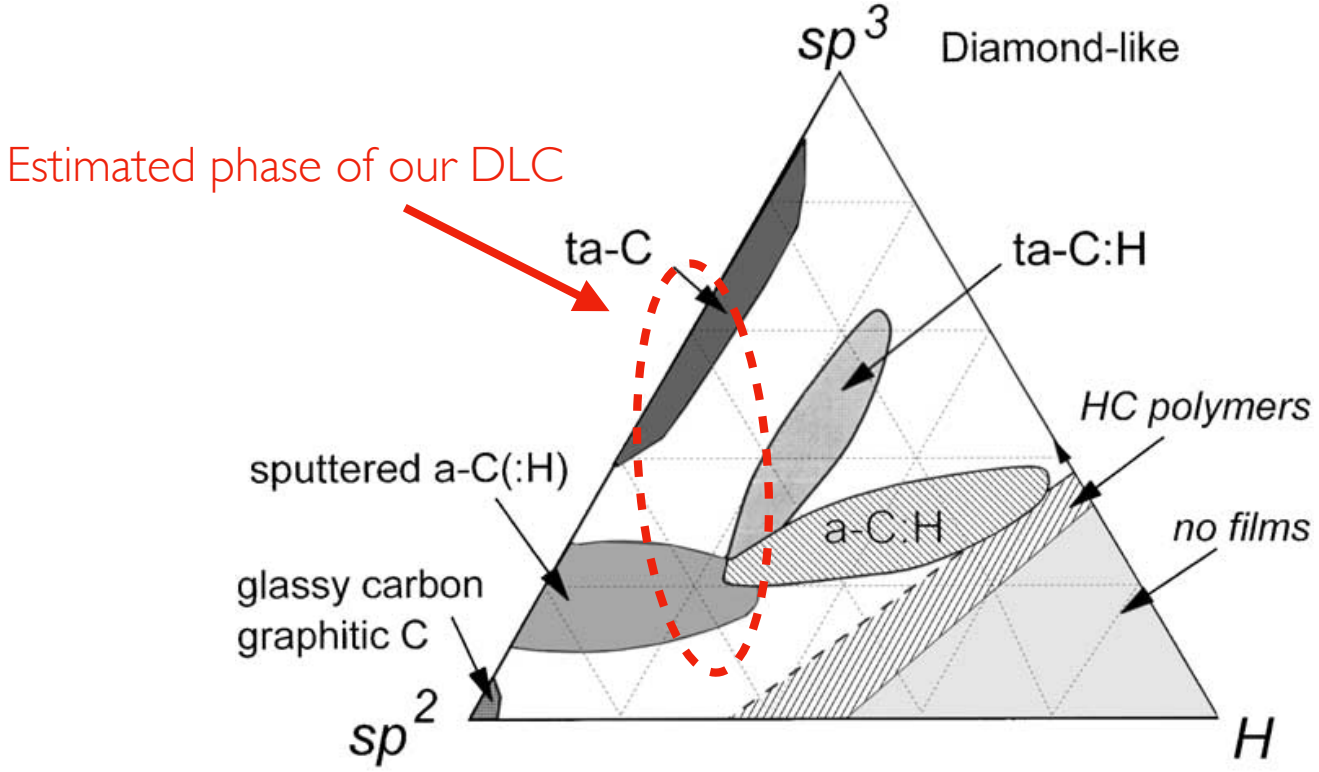


Categorisation in phase diagram

- Raman spectroscopy indicates our DLC categorised as a-C:H
- Hydrogen source not fully understood

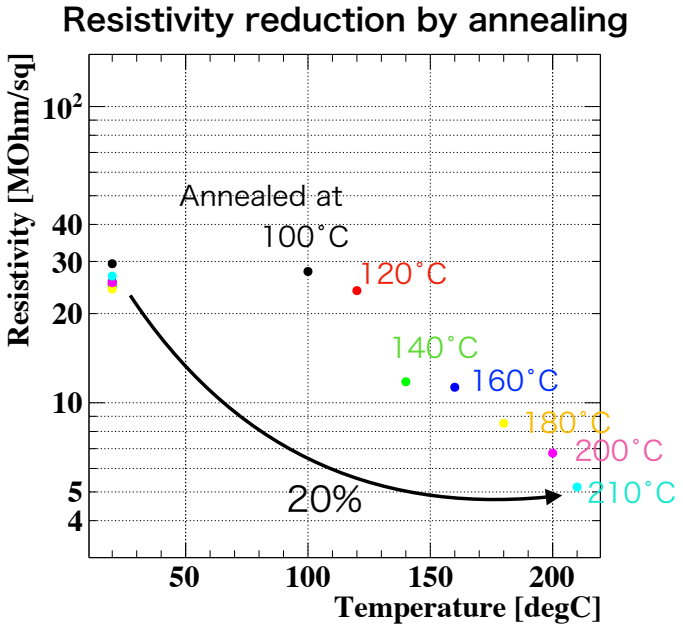
➡ **Estimate category with large uncertainty**

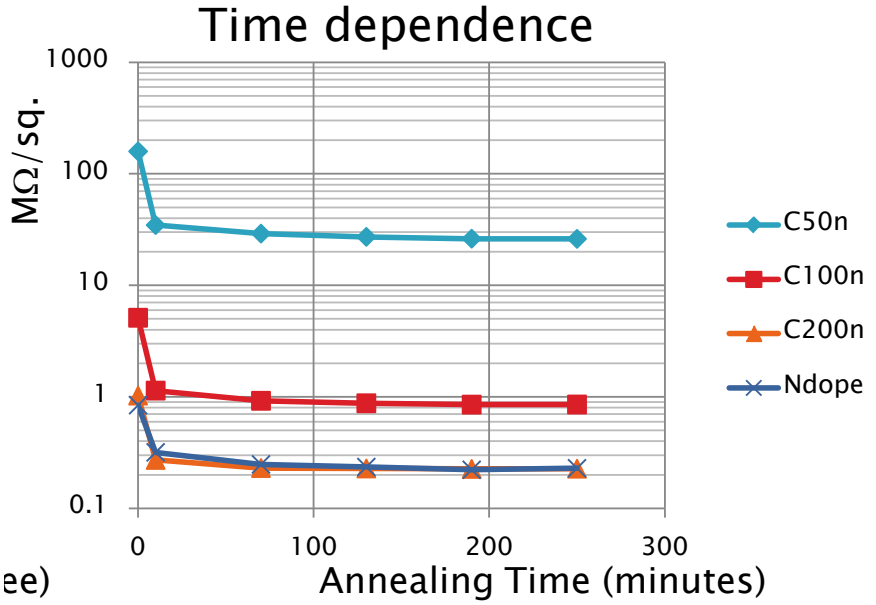
➡ **Look into properties of a-C:H and ta-C**



Thermal annealing observation

- Thermal annealing reduces resistivity
  - **Depends on temperature**
  - **NOT depends on time**
- Resistivity can be controlled with an accuracy of 10%
- **We did NOT know the mechanism**



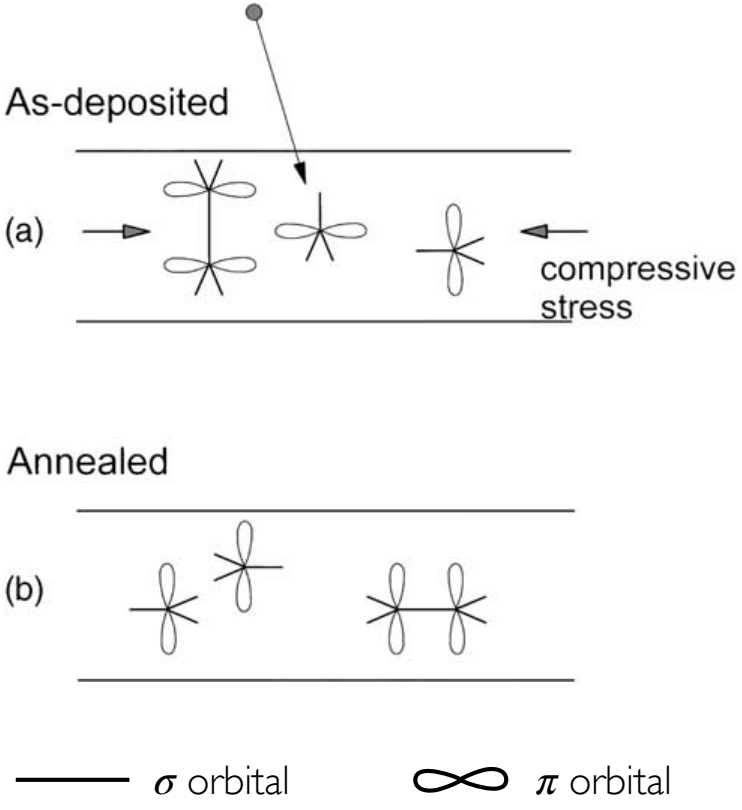


A.Ochi, Resistive DLC meeting 2020/3/26

Thermal annealing of ta-C

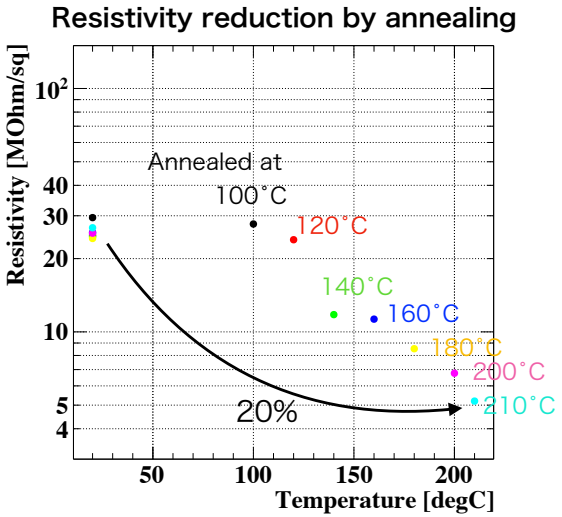
- **Thermal annealing of ta-C is well known**
  - a-C:H as well. But,
  - *"Thermal annealing of a-C:H also reduces the stress, as in ta-C. However, as the bonding in a-C:H is less stable during annealing, annealing is less useful in this case."*

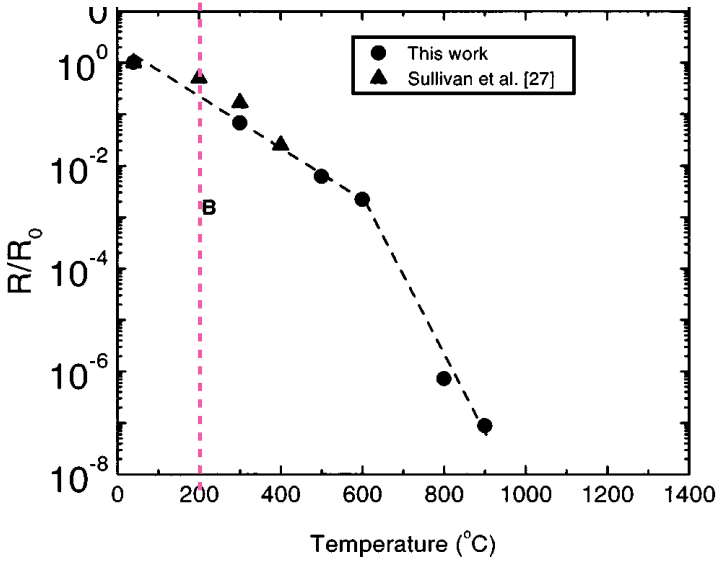
- Mechanism described
  - Thermal annealing converts a small fraction of sp^3 (2%) to sp^2
    - Distance between atoms is different between sp^2 and sp^3
    - New sp^2 structure has aligned electron orbitals
  - The conversion causes **exponential decrease** in resistivity
  - Compressive stress relieved by new sp^2 structure with electron orbitals aligned

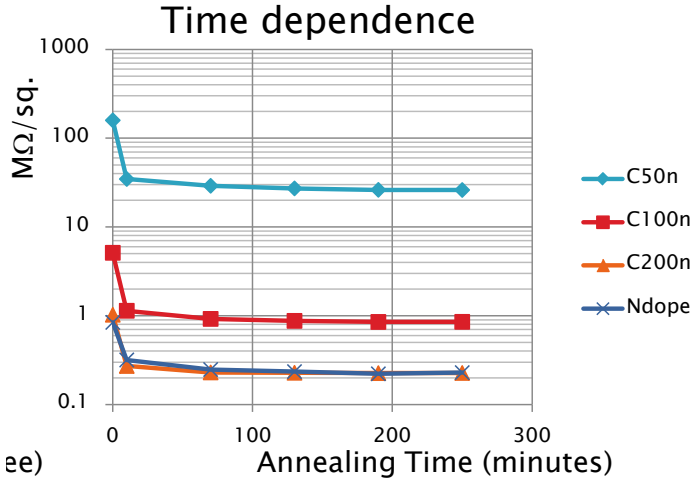


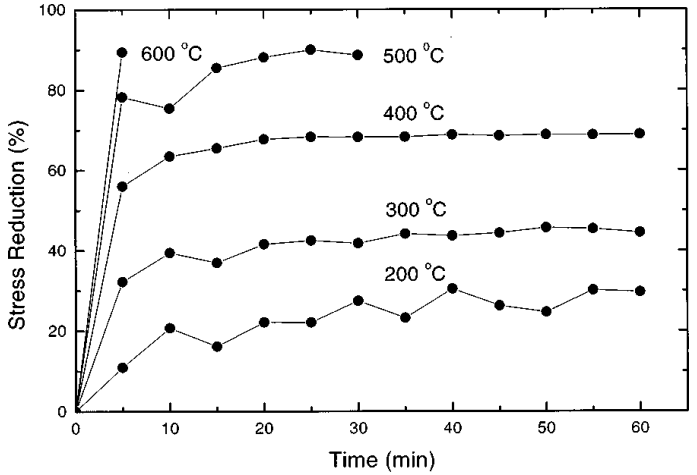
Change by annealing

- Our measurement consistent with previous works









A. C. Ferrari, et. al., *J. Appl. Phys.* **85**, 7191–7197 (1999)

A.Ochi, Resistive DLC meeting 2020/3/26

Conclusion

- **DLC attached well on FR4 and Cu as well as polyimide**

- Measure Raman spectroscopy for DLC
  - a-C:H indicated
    - Hydrogen source not understood
  - **Our DLC categorised as a-C:H and ta-C phases with large uncertainty**

- Discuss thermal annealing
  - **A small fraction of sp^3 converted to sp^2 with electron orbitals aligned**
  - Resistivity decrease correlated with compressive stress relief

- This study can **help our understanding and fabrication of DLC**
  - Especially when sputtering in our own sputtering facilities

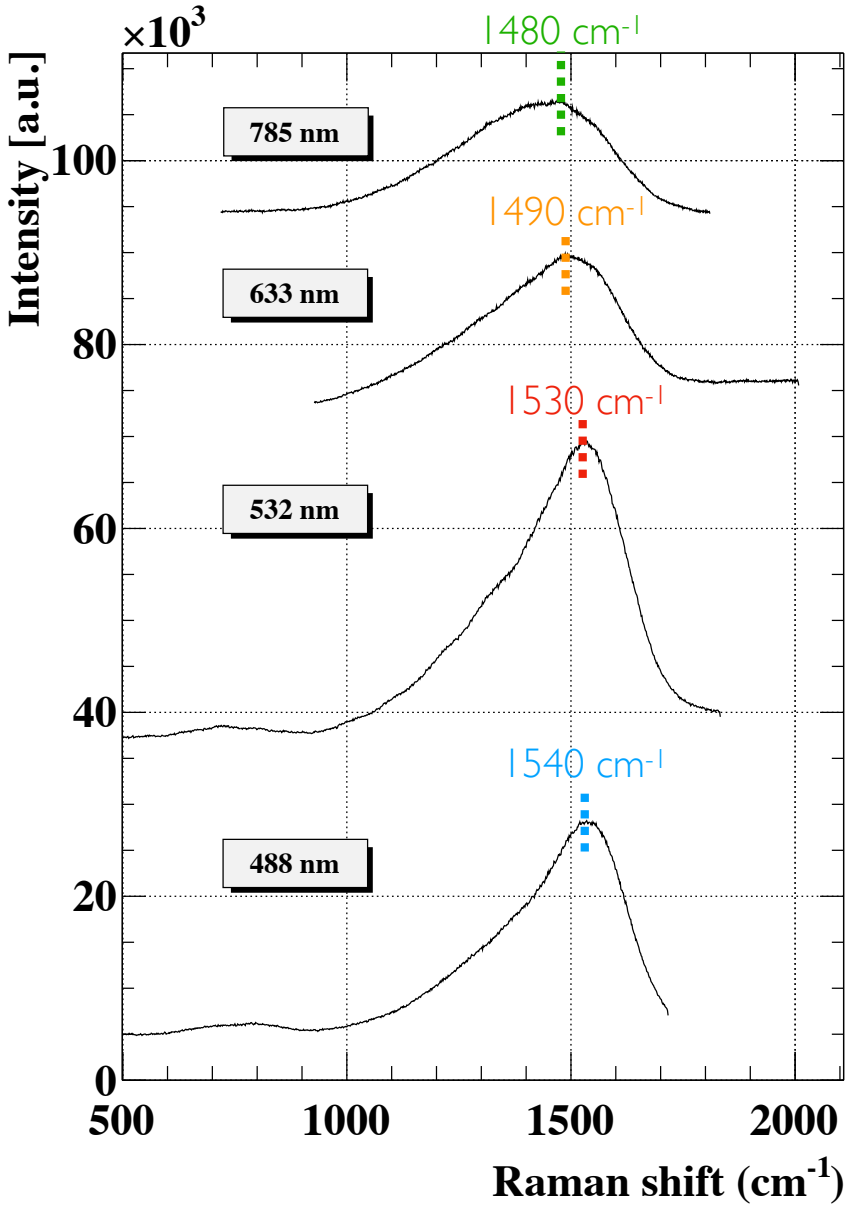
Reference & Acknowledgement

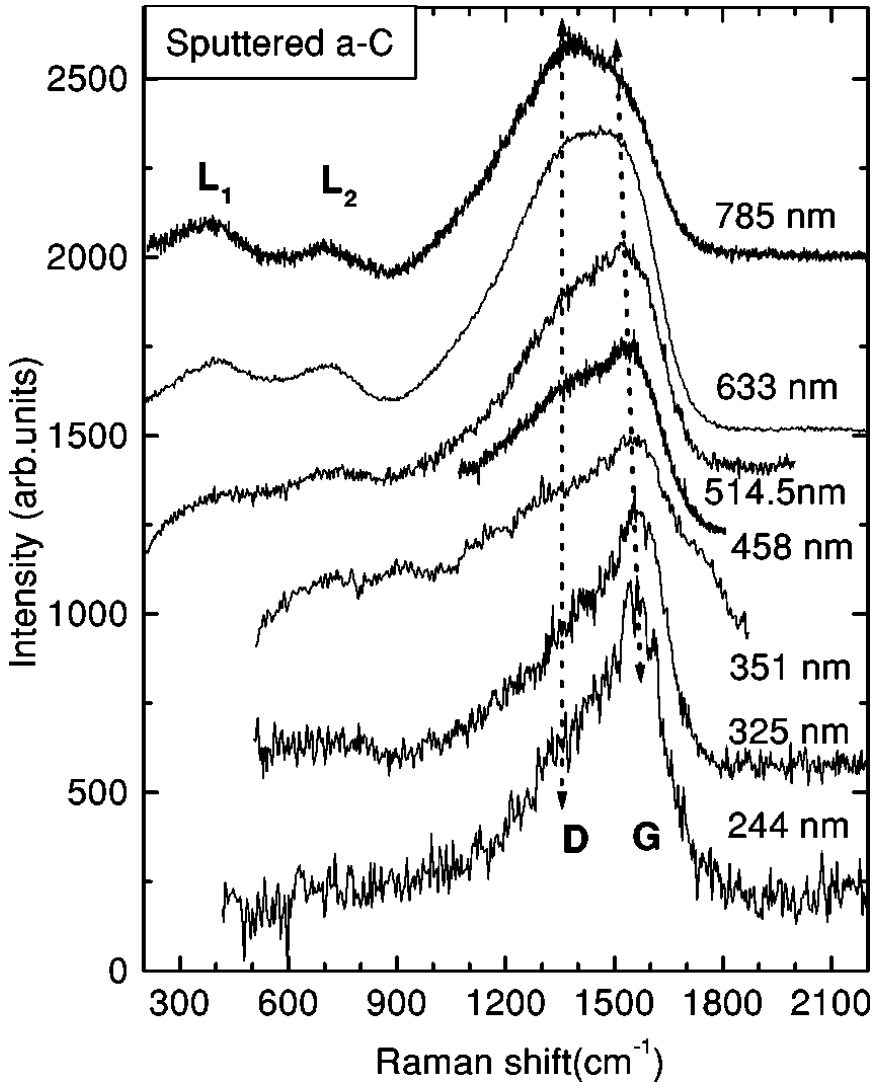
- Reference
  - J. Robertson, *Mater. Sci. Eng. R Rep.* **2002**, 37, 129–281
  - A. C. Ferrari and J. Robertson, *Phys. Rev. B* **64**, 075414
  - A. C. Ferrari, *et. al.*, *J. Appl. Phys.* **85**, 7191–7197

- Acknowledgement
  - Be-Sputter Co. Ltd.: Sputtering
  - S. Chiashi and R. Kaneda from *Dept. of Mechanical Engineering, the University of Tokyo*: Raman Spectroscopy

Backup

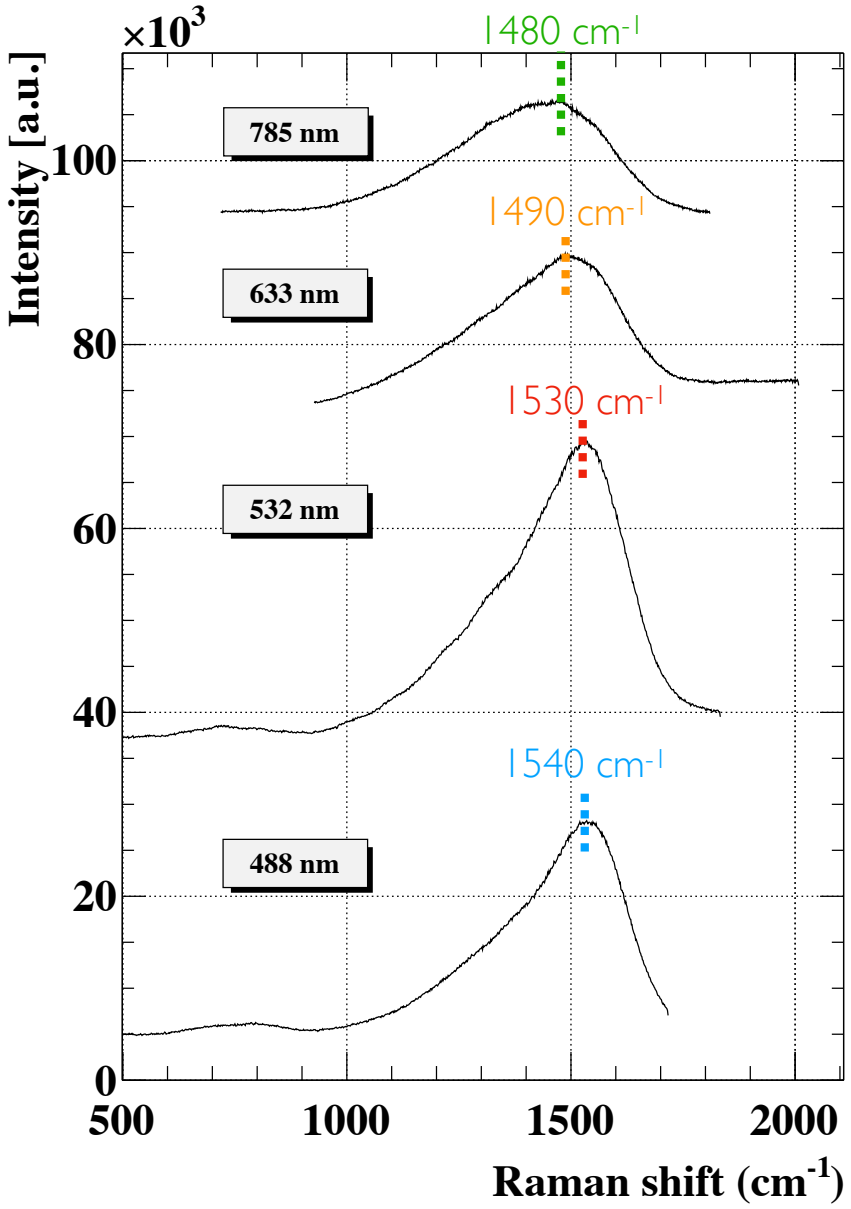
Wavelength dependence

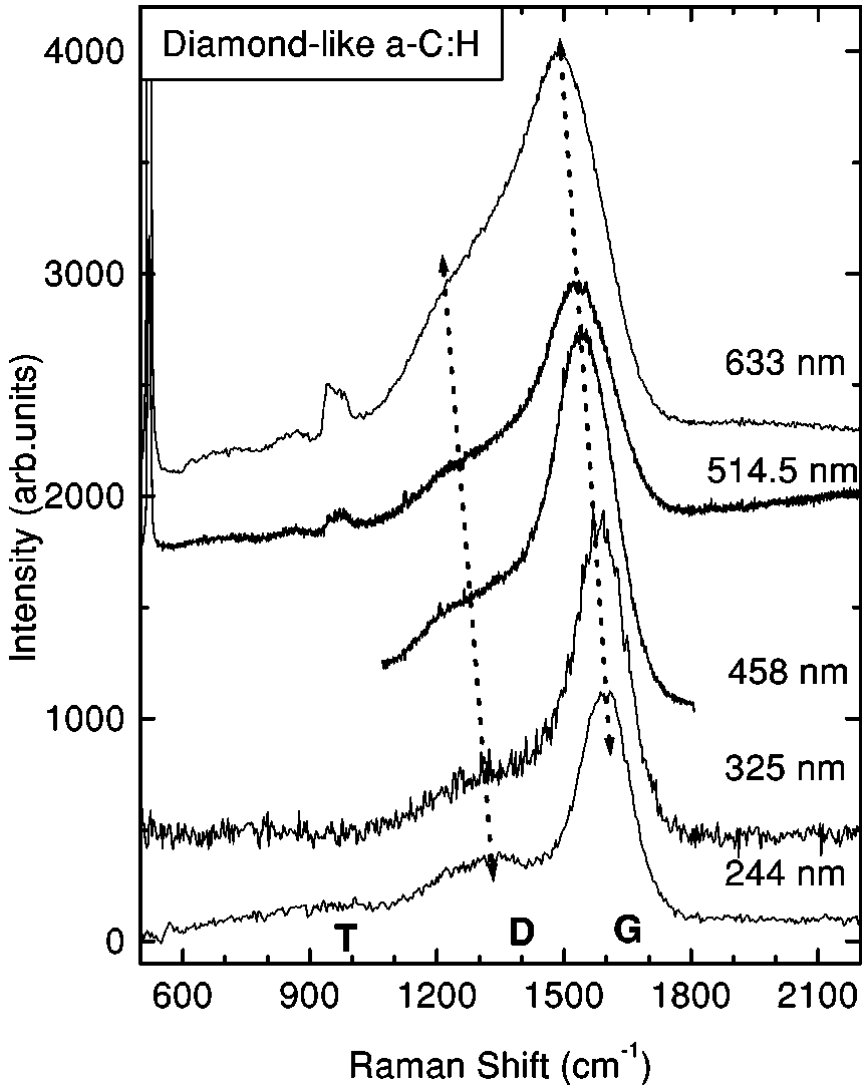




A. C. Ferrari and J. Robertson, Phys. Rev. B 64, 075414

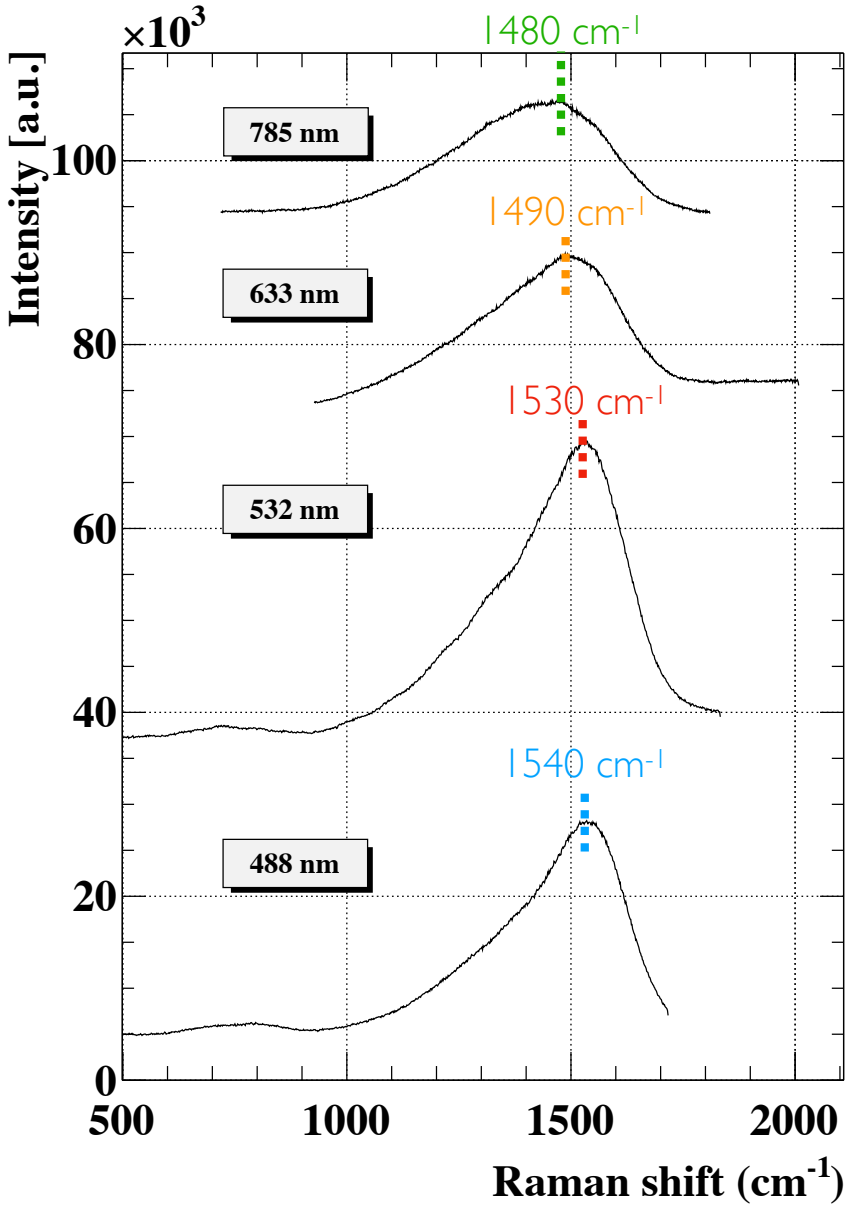
Wavelength dependence

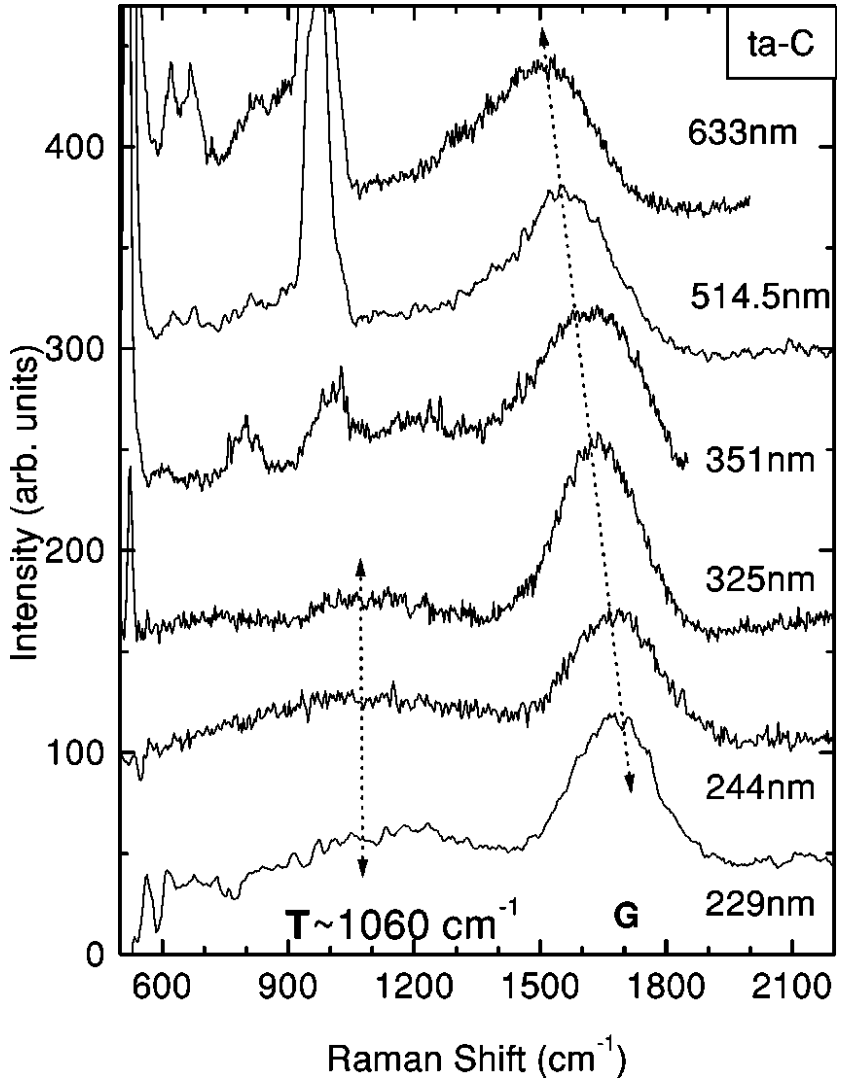




A. C. Ferrari and J. Robertson, Phys. Rev. B 64, 075414

Wavelength dependence

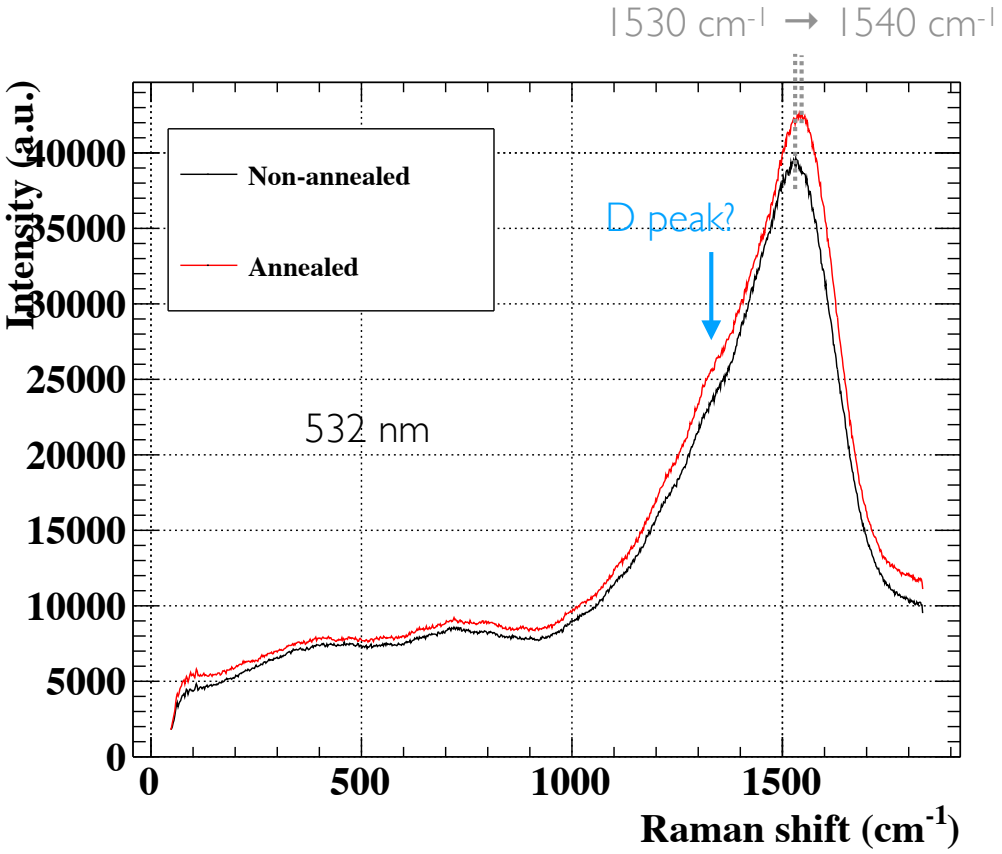




A. C. Ferrari and J. Robertson, Phys. Rev. B 64, 075414

Raman spectra w/(o) annealing

- No clear appearance of D peak by annealing at 200°C
  - However, observed a bit



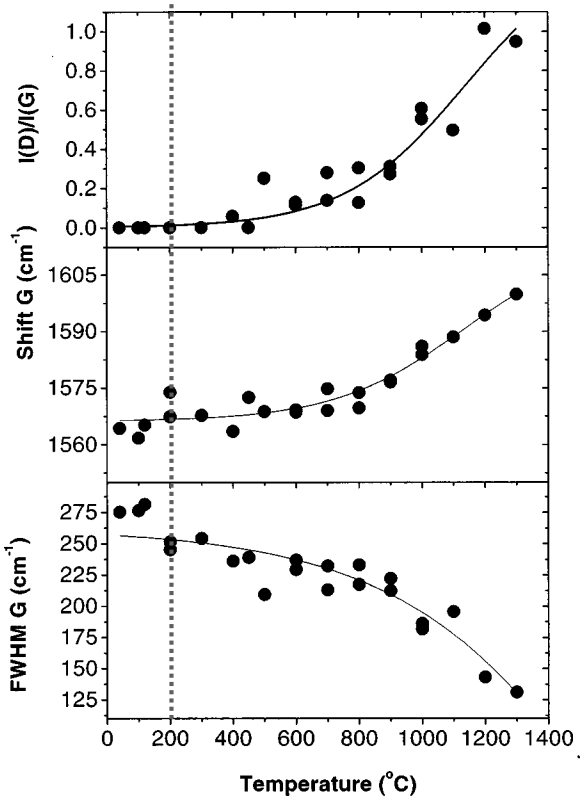


FIG. 9. Variation of G peak position, $I(D)/I(G)$ ratio and G peak position and G peak FWHM of the visible Raman spectra of Fig. 8, vs annealing temperature; the lines are guide to the eye.